

EXHIBIT 6

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SOLID WASTE PERMIT APPLICATION
Medical Waste Treatment Facility | Nye County



MediWaste Disposal LLC

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1. INTRODUCTION

This sustainability permit application has been prepared for MediWaste Disposal LLC's (MediWaste) proposed medical waste facility in Pahrump, Nevada, in Nye County. MediWaste is seeking approval from the Nevada Division of Environmental Protection (NDEP) under Nevada Administrative Code (NAC) § 444.676, "System to process waste: Other methods," which states the following:

"Before any method of solid waste processing, not otherwise provided for in these regulations, is placed into operation, complete plans, specifications and design data must meet the approval of the solid waste management authority."

The MediWaste medical waste facility is designed to provide a cutting-edge pyrolysis process for treating medical waste. The facility's thermal destruction process will adequately treat wastes and result in the generation of a small amount of ash residue that can be disposed of in a Class I landfill. The MediWaste facility utilizes pyrolysis (endothermic gasification in the absence of oxygen) instead of incineration (combustion using oxygen) to treat the waste. Because of this approach to pyrolysis, the medical waste facility is unique and can be evaluated differently than landfills, traditional incinerators, or other solid waste facilities. Treating medical waste via pyrolysis aligns with the intent of NDEP's 2022 State of Nevada: Sustainable Materials Management Plan, where landfilling of wastes should be the last resort (NDEP 2022).

The medical waste will be loaded into the system (including packaging and pallets) in a batch process. Once the waste is loaded into the system, the door is closed and sealed, and the thermal destruction process begins. There are three separate unit operations to ensure pyrolysis of the waste, packaging, pallets, and wrapping. The first step heats the waste and packaging material until it is converted to smaller volatile fragments (vapors). The pyrolysis process also generates a small amount of ash residue (approximately 2-40% of the volume of the input material). Next, the vapors are sent to the second stage where they are formed into plasma, then oxidated to form carbon dioxide and water vapor. The third and final step will send the carbon dioxide and water vapor to a vent where the inert gasses are then exhausted from the stack. All steps occur in a fully enclosed system with no transfer points or fugitive emissions. This application is requesting the throughput capacity to install seven (7) pyrolysis units.

The only solid waste generated from the process is the hydrated ash which will be managed at an appropriately permitted and NDEP approved disposal site (expected to be a Class I landfill). The ash will be appropriately characterized using the Toxicity Characteristic Leaching Procedure (TCLP) to ensure no RCRA-toxics are present above threshold levels. The waste hydrated ash will also be tested for dioxins and furans per EPA Method 8290A. Should the ash fail the TCLP for any toxic constituents, it will be managed according to all state and federal requirements.

Information presented in this application is based on process information provided by Vulcan Systems, the pyrolysis equipment manufacturer.

2. PROPERTY DESCRIPTION

MediWaste is seeking to establish a medical waste treatment facility at 1850 E. Basin Avenue, Pahrump, Nye County (Parcel 035-191-52). The parcel is approximately 4.7 acres in size, currently zoned "HI" for heavy industrial use, and has recently been operated by Pinnacle Propane. The facility location and land use zoning for a 1-mile radius around the facility is shown on Figure 1 in Appendix D. The Pahrump Regional Planning Commission approved a Conditional Use Permit for the medical waste treatment facility during their meeting on April 10, 2024.

The property contains two, one-story metal sided buildings suitable for offices, housing the pyrolysis process, general storage, and storing medical waste containers prior to processing. The existing buildings on-site are Building 1 (8,650 square feet [sq ft]) and Building 2 (3,764 sq ft), and these buildings are not anticipated to be expanded by MediWaste for their planned operations. An aerial image of the site with pertinent site features marked is provided in Figure 2 in Appendix D.

2.1 Proof of Ownership

MediWaste has purchased the property located at 1850 E. Basin Avenue, Pahrump, Nye County (Parcel 035-191-52). Proof of ownership and leasing of the property is provided in Appendix A.

2.2 Zoning

Approval is being sought for this facility under NAC 444.676, and therefore the facility is not considered a Class I or Class II disposal site, so it is not subject to location restrictions ("set backs") under NAC 444.678.

The adjacent properties to the north, east, and west are zoned for heavy industrial use, and properties to the south are vacant and zoned for general commercial use. The property is contained within a "Zone X" for Federal Emergency Management Agency (FEMA) floodplain hazards, meaning it is in an area of minimal flood risk.

Properties and land zoning within a 1-mile radius of the site are shown on Figure 1 in Appendix D.

2.3 Security

The facility is secured with fencing, gates, and doors to control access to the buildings and the outside staging area behind the buildings. The buildings will be outfitted with an alarm system that is activated when not occupied. The parking areas and staging areas surrounding site buildings are outfitted with lights to illuminate the areas day and night. The facility will also be equipped with a closed-circuit television camera system to monitor and identify unapproved personnel or activities onsite.

The gates and surrounding fences will be marked with warning signs in English that are worded:

"CAUTION - BIOHAZARDOUS WASTE STORAGE AREA - UNAUTHORIZED PERSONS KEEP OUT"

and in Spanish:

"CUIDADO - ZONA DE RESIDUOS BIOLOGICOS PELIGROSOS - PROHIBIDA LA ENTRADA A PERSONAS NO AUTORIZADAS."

The fencing that faces E. Basin Avenue is a block wall that prevents public viewing of waste handling operations. The remaining three sides of the property are secured by chain link fencing with barbed wire to discourage unauthorized individuals from accessing the property.

2.4 Traffic

Tractor trailer loads of medical waste are expected to arrive for processing each week. Other vehicles coming to the site are those used by site workers or waste transporters. At full operation, the facility expects to receive five to six truckloads of medical waste processing per day.

The preferred method of ingress and egress from the facility will be along E. Basin Avenue, and trucks will depart Pahrump via State Highway 160, approximately 0.75 miles west of the facility. Per the Nevada Department of Transportation (NDOT) Annual Average Daily Traffic Count station located on State Highway 160 just outside of Pahrump (Station 0230005), the station logged an average of 24,100 vehicles per year in 2024. This annual value averages out to 66 vehicles per day. The additional five to six trucks per day due to the proposed MediWaste operations is a minor increase in traffic may be noticed along E. Basin Avenue and State Highway 160. The NDOT traffic data is included as Appendix I.

2.5 Stormwater

The property contains existing stormwater management features that will be utilized and maintained by MediWaste for facility operations. A grading plan for the site provided by Pinnacle Propane is included in the drawing set for the Nye County Conditional Use Permit in Appendix B. The site is relatively flat topographically, and stormwater runoff within the fence line generally flows to the north along the asphalt surface, on to native ground surface cover in the rear of the facility and eventually to the stormwater detention basin in the northwest corner of the property, identified on Figure 2 in Appendix D. MediWaste will also install a culvert around the property that will ensure all stormwater is diverted to the detention basin and no stormwater leave the site. The culvert will be designed by an architect.

MediWaste will employ best management practices to keep unprocessed waste and pyrolysis outputs from coming into contact with stormwater runoff. Unprocessed medical waste will be stored interior to Building 1, as shown on Figure 3 in Appendix D, or staged in the delivery trailers. Waste will only be stored in the delivery trailers until the containers can be unloaded and recorded in the operating record. Hydrated ash from the pyrolysis process will be stored in closed or covered containers to prevent contact with rainfall.

3. FACILITY AND PROCESS DESCRIPTION

MediWaste is not planning to significantly improve the existing facility prior to commencing operations. The pyrolysis processes can be housed within Building 2, and medical waste will be stored in Building 1 prior to processing. The medical waste storage area, pyrolysis process, and directions of waste flow for the facility are shown in Figures 2 and 3 in Appendix D. The facility layouts provided in this application are considered preliminary and may be re-evaluated and revised by MediWaste for up to the first 2 years of operations. Prior to any changes in the placement of the feedstock or hydrated ash waste, MediWaste will notify NDEP and follow the Division's required process to capture the updates.

The only waste acceptable to bring to the facility will be medical waste as defined by the following:

- ▶ NAC 444.589 which has the meaning ascribed to it in 49 Code of Federal Regulations (CFR) Part 173, Appendix G: "Definition of Regulated Medical Waste"
- ▶ NAC 444.00745: "Sharps" defined. (NRS 439.200) "Sharps" means an object which has been or may have been contaminated with a pathogen through handling or during transportation and which is also capable of cutting or penetrating the skin, tissue, cartilage or mucosa of a person or a packaging material. The term includes, without limitation, needles, syringes, scalpels, broken glass, culture slides, culture dishes, broken capillary tubes, broken rigid plastic, Pasteur pipettes and similar items that have a point or sharp edge or which may break during transportation resulting in a point or sharp edge. (Added to NAC by Bd. of Health by R112-16, eff. 12-19-2017)
- ▶ United States Department of Transportation (USDOT) according to 49 CFR 173.134

MediWaste will only accept a subset of the wastes outlined in these definitions (as noted in Section 4.2 and the Waste Acceptance Policy in Appendix F). The verbiage of the above stated definitions is provided in Appendix G.

The facility is designed with the ability to process up to 2,520,000 pounds (approximately 1,260 tons, 630 cubic yards) of medical waste per month with all seven pyrolysis lines operating. Building 1 is arranged to store up to 500,000 pounds (approximately 250 tons, 125 cubic yards) of medical waste at any time. In addition, MediWaste anticipates storing up to ten (10) trailer loads (400,000 pounds, 200 tons) within enclosed tractors in the secured paved lot behind Buildings 1 and 2 in the areas designated on Figure 2 in Appendix D. Therefore, 900,000 pounds (approximately 450 tons, 225 cubic yards) is the maximum amount of unprocessed medical waste that will be onsite at any time. Medical waste will not be onsite for more than 30 days. There will be a maximum of five to six trailer loads with medical waste arriving for processing per day, 42 trailer loads per week, 168 trailer loads per month, and 2,016 trailer loads per year. There may be occasional exceedance in the event a large customer order comes in, but this will be corrected for in subsequent days (i.e., a large incoming shipment would be scheduled such that other shipments would not be received until there is staging and processing capacity). Total deliveries will be bounded by the physical process capacity of the facility. Payment for services is generally obtained upon destruction, not upon receipt of the shipment, so there is no incentive for MediWaste to accumulate materials.

The pyrolysis process is a batch process where the waste container (gaylord box or polypropylene bag) and pallet will be loaded into the system and processed without any disassembly or segregation required. A detailed description of the pyrolysis process is provided in Section 3.2.

MediWaste will maintain records of the weights and/or volumes of wastes entering the facility using and outputs leaving the facility using medical waste manifests and shipping papers. Each container of

unprocessed medical waste will be weighed prior to processing, and the production outputs will be tracked daily.

3.1 Waste Storage and Handling

Medical waste transported to and stored at the facility, including waste transported from other states, will meet the requirements of NAC § 444.662, "Storage of solid wastes before collection," specifically Section 6, which states the following:

"Medical wastes must be stored in watertight, tightly covered and clearly labeled containers that are resistant to corrosion and are in a safe location, inaccessible to the public. In addition, medical wastes must be stored in cleanable containers with liners or in a manner approved by the solid waste management authority. Medical wastes must not be deposited in containers with other solid wastes. Medical wastes must be transported separately from other solid wastes to an approved disposal site and handled in accordance with a method approved by the solid waste management authority."

Medical waste will be picked up directly from hospitals, medical waste generating facilities, or transfer stations and be transported to the facility using enclosed tractor trailers in accordance with USDOT regulations for the transport of medical waste (49 CFR 173.134). Medical waste may be pre-processed at a off-site transfer station, where the personnel will inspect and consolidate medical waste into appropriate containers prior to shipping to MediWaste's facility for final processing. MediWaste uses USDOT approved, packing group II packaging materials which are leakproof and rodent proof to store and transport medical waste. Medical waste is transported and stored largely in rigid plastic containers with sealed lids. In turn, these sealed plastic containers will be stored in plastic lined one cubic yard container boxes or one ton-nominal bulk bags made of polypropylene. As such, the overall packaging is inherently watertight and rodent proof. In addition, MediWaste will engage the services of a pest control company to prevent rodent activity near or around the facility.

Trucks will enter the facility via the eastern or western gates facing E. Basin Avenue, and the containers with medical waste will be unloaded using a forklift and staged in the larger building for weighing and screening before processing. MediWaste utilizes a customer relationship management (CRM) tool and associated software to track orders, provide delivery schedules, and control inventory in the building for all incoming materials. MediWaste will take their current practices in other states (which have already been audited and approved) and transpose them to the Pahrump facility. Upon receipt, each container of medical waste will have a label that will be scanned upon acceptance into the CRM software. The label will be used to track the container while at the facility and will tie to the trailer the container is staged in. The label will be scanned again prior to destruction to close the loop and generate an invoice to the customer. A sample container label and inbound shipping paperwork packet are included in Appendix H.

Prior to unloading into Building 1, tractor trailers with unprocessed medical waste will be staged onsite within the secured, fenced area. Waste material will only be staged in the tractor trailers until the material can be unloaded and logged into the facility record (no more than 30 days). Tractor trailers will be maintained in drivable condition (i.e., no flat tires). In the event a trailer is not drivable, the medical waste will be relocated to another trailer if it cannot be processed. The unprocessed medical waste is non-putrescible and does not require refrigeration under all federal and state laws in both California and Nevada. Putrescible wastes are explicitly excluded by the revised Waste Acceptance Policy found in Appendix F and are covered in the Medical Waste Screening. The waste will undergo an inspection process of BOL's and manifests when it arrives to screen out putrescible wastes. There are no solvents of high vapor pressure materials, and the cycle time to receive and unload a truck is generally short. Due to the material being

shipped and staged in closed containers, storage of the material would not result in emissions. The containers are sealed when they leave the hospital, are then consolidated into closed gaylord containers, and are then placed in a closed trailer. Waste flows within the facility boundary are shown on the site on Figure 2 in Appendix D.

Unprocessed medical waste will be in sealed, gaylord containers or polypropylene bags in Building 1 and moved to Building 2, which will hold the pyrolysis units, using forklifts or pallet jacks. The waste is never removed from the final container it is received in. Medical waste is initially sorted at the point of generation at the medical facility into colored containers based on the requirements shown in the summary of the California Medical Waste Management Act (MWMA) attached in Appendix F. MediWaste will only accept the red containers which are sealed to prevent exposure to potentially infectious waste. Hospitals are legally and contractually required to sort waste materials (including medical waste) correctly. Since the small waste containers are sealed at the point of generation, MediWaste will rely on the quality control measures in place at the medical-facility level to ensure non-conforming waste is not sent to the facility. MediWaste will inspect the outside of the containers to ensure only red containers are received and to ensure that containers are in good condition. From there, the waste container (including packaging and the pallet) will be placed in the pyrolysis units for processing. The container and pallet are all placed in the pyro chamber and destroyed. By law, pallets used to hold medical waste cannot be reused and must be destroyed. The layout of Buildings 1 and 2, including the proposed location of the pyrolysis units, storage areas for hydrated ash, offices, unprocessed medical waste storage are shown on Figure 3 in Appendix D. The layout of equipment and specification sheets are shown on Figures 4 and 5 in Appendix D.

MediWaste has developed a Waste Acceptance Plan (attached as Appendix F) that clearly outlines to customers what waste can be accepted by the facility and the procedure when non-conforming material or waste is received. Waste that is not identified prior to unloading as not suitable or approved for pyrolysis will be removed using handheld picking tools and placed aside for characterization and offsite disposal.

3.2 Pyrolysis Process

Pyrolysis is a process that converts solid material, in this case medical waste, into inert byproducts and hydrated ash using high temperatures. The hydrated ash, char, and inert byproducts are the residue from thermal breakdown. The residue is from non-combustibles like metals, sharps, tweezers, surgical instruments, fillers in plastics and paper like Al_2CO_3 , CaCO_3 , CaO , clays in cardboard, glass bottles, glass components, and ceramics. The pyrolysis process will be contained within the existing on-site structures and is not anticipated to pose a noise or nuisance concern to the community or surrounding properties. There is no water required for the pyrolysis process, nor is there any wastewater generated from the process. The proposed layout of the pyrolysis process and the process flow in Building 2 are shown on Figure 3 of Appendix D.

The overall pyrolysis process for treatment of medical waste consists of three sequential processing steps listed below:

The first unit operation is carried out in the primary pyrolytic chamber in which waste is heated with an external heat source to the point where molecular torsion and cracking takes place. The materials are generally heated for a period of time based on water content, but the average processing time is eight (8) hours per cycle at high temperature (over 1,400 degrees F). Sustained exposure at 1400°F for minutes delivers an extreme thermal load, sufficient to sterilize all biological waste and fully decompose thermally stable pharmaceutical compounds. Likewise, a lower temperature for extended times can deliver the same of greater thermal loads. This heat induced torsion breaks large molecules down to smaller volatile

fragments (vapors). This process repeats itself until all the paper, plastic, and all other components of the waste are converted to vapors. The system is fully enclosed, with massive stainless-steel piping conveying the vapors out of the pyro system.

The vapors are then received by the second stage – where a plasma forms. Under similar conditions as the pyrolytic chamber, this section of the system takes the volatile organic compounds (VOCs) - the volatile molecules - and further reduces them in size. Following this point in the process, excess air is quantitatively introduced to begin the oxidation process converting the VOCs to carbon dioxide and water vapor.

The final step in the process is the vent. The vent provides increased residence time to allow the remaining vapors and solid particulates to react. The benign and inert exhaust gases from the vent are then exhausted through the stack.

These steps take place inside a fully closed system. No leaks are possible nor fugitive emissions. There are no transfer points or removal of streams.

The utilities required for operation are natural gas or liquid propane and 480-volt electrical power supply. When processing the solid waste, a 2-40 % volume of hydrated ash residue is expected to be recovered and sent to an appropriately permitted and NDEP approved disposal site (expected to be a Class I landfill) for final disposal. MediWaste expects the ash generation will be on the lower end of the range. The estimated volume range of ash to be generated is:

- ▶ 1,680-33,600 lbs daily;
- ▶ 11,760-235,200 lbs weekly;
- ▶ 50,400-1,008,000 lbs monthly; and
- ▶ 604,800-12,096,000 lbs yearly.

The ash is captured in the pyrolytic chamber (the first processing step) and is removed periodically based on accumulation. The ash will be placed in end dump tractor trailers or tipper transfer trailers. All containers would have lids or a tarp system to contain the ash, ensuring the ash is contained and does not pose a risk to the environment. End dump tractor trailers (roll-offs) are sent out as they are filled. MediWaste anticipates one roll-off bin will be generated per week, up to 10 days. In the event of extended facility downtime, the roll-off bin would be shipped within 90 days of generation. The maximum onsite storage capacity of ash is one 40-yard roll-off container. There is a possibility of two 40-yard roll-off containers while one is being scheduled for pickup. The only other variance may be a delay in pickup by landfill.

The generation of hydrated ash will depend on many variables. The amount of cardboard, glass, paper, fillers, surgical instruments, etc. will determine how much ash is generated per run. MediWaste estimates that seven machines will generate one 40-yard roll-off container per 7 to 10 days. Before the ash is sent to the disposal facility, full TCLP tests are carried out to evaluate the suitability of sending that ash to the correct landfill (e.g., if the ash material exhibits any toxic hazardous waste characteristics). A retention sample is kept per pyro batch and will be sent off for periodic testing. These samples will be kept separate but the combined "load", which is a roll-off container, will be managed in accordance with the most conservative sampling result (e.g., if a sample comes back as hazardous then the entire roll-off bin will be managed as hazardous). All retention samples are required to be analyzed by a Nevada certified laboratory per NRS 459. 500. The TCLP test is always requested by the disposal facility operator as well. The hydrated ash will also be tested for dioxin and furans per EPA test Method 8290A. Should the operator deem that the reaction can be terminated early (as determined by spontaneous cooling), some additional inert char may be present in the ash.

3.3 Prevention of Hazards, Nuisances, and Impairments to the Environment (NAC 444.668)

MediWaste will mitigate any hazards, nuisances, and impairments to human health or the environment through the use of operating plans, system and facility design, as well as employee training. The facility will be fenced, gated and entry control will be implemented on the doors to buildings to preclude public access. Incoming medical waste will be transported within closed semi-trailers and contained within the closed plastic containers it is initially accumulated in, within a closed supersack or gaylord box. Prior to processing, the medical waste will be stored in the same closed containers from transit, and will be stored indoors to prevent any potential release to the environment. The pyrolysis process occurs indoors within enclosed equipment outfitted with pollution control devices. No noise nuisance or pollution exposure is anticipated due to the design of the process. Any noise is limited to work inside the facility, and all OSHA noise level standards are met. Maximum noise levels inside the building are less than 80 dB, much less than 80 dB at the building wall, and even less than that on the fence line. Vehicle noise will be minimal, and far less than neighboring industrial activities. No odors will emit from vehicles delivering the waste for processing. No "added" noise will be created above threshold dB values. Traffic will not be significantly changed as discussed in Section 2.4. There is no visual impact since the surrounding businesses are also industrial in nature due to the Heavy Industrial zoning of the area.

The only waste product from the pyrolysis process is hydrated ash that is contained in the pyrolytic chamber of the process equipment. Once full, the chamber is emptied and transferred into a trailer with a tarp or lid for storage until transported for disposal. The ash transfer and storage will be conducted using best management practices to mitigate risk of stormwater or groundwater exposure. The entire medical waste treatment facility is designed to reduce the hazards, nuisances, and the risk of any impairment to the environment or surrounding public.

Nuisance issues are dealt with through the county permits (see the attached Conditional Use Permit (CUP) approval letter). The CUP is only approved in the event all concerns and issues (including nuisance issues) have been addressed to the satisfaction of the county. In addition, MediWaste has ensured that there are no visual impairments from the facility. No odors emit from the facility since there are no putrescible wastes on the premises. There are also no solvents, or high vapor pressure materials. The facility is fenced and screened from public view. The site is zoned for heavy industrial and is surrounded by other heavy industry operations. As such, the proposed facility would have no impact to the overall nuisance level of the area.

4. OPERATING PLAN

Only loads from MediWaste collection vehicles or approved third-party transporters will be accepted. Third-party transporters must have prior arrangements with MediWaste and meet criteria established in agreements between the transporter and MediWaste. Third party transporters must remain in good standing with applicable permits issued by USDOT and NDEP that are current and have not expired. Medical waste will be collected from hospitals and other medical waste generating facilities.

The facility is designed to operate up to 24 hours a day, 7 days a week. Operation of the pyrolysis unit(s) will be variable at the start of operations and may only operate for a fraction of the day as staff become familiar with the systems. The goal is to ramp up operations and pyrolysis activities over time with the ability to have continuous operations at the facility. This application is requesting the maximum throughput for the facility operating 24 hours a day, 7 days a week.

The facility will be staffed by 5 to 15 full-time employees responsible for managing incoming wastes and outputs and for operating the up to seven (7) pyrolysis units.

4.1 Incoming Medical Waste Storage

Medical waste will be stored in watertight, closed, and clearly labeled containers that are resistant to corrosion, water, and rodents (e.g., waste is placed in rigid plastic containers, placed inside polyethylene bags, placed inside polypropylene bags or cardboard boxes). No putrescible or bulk liquid waste is accepted per the Waste Acceptance Policy and Medical Waste Screening. Medical waste will be trucked to the facility in enclosed 53-foot-long tractor trailers, which will remain fully enclosed until the waste is offloaded into Building 1. Within the trailers, waste will be contained in closed, gaylord containers or polypropylene bags with two layers of additional packaging.

Waste will be accepted at the facility on a predetermined, scheduled basis. Unprocessed medical waste will remain in the delivery trailers in the secured, fenced portion of the facility until it can be offloaded, in the area designated on Figure 2 in Appendix D. See Section 3.1 for a full discussion of the mitigation of odors and emissions associated with the storage of the incoming waste.

The trailers will be surrounded by a reusable diking system to ensure any spills of material are contained. The trucks are also sealed and leak proof for washing. Once offloaded, the waste containers will be staged in Building 1 until treatment in the pyrolysis process. Staging the medical waste within the trailers will limit the movement of the containers and reduce the risk of any incidents during container transfers. Received medical waste will not be onsite for more than 30 days prior to processing. The amount of time waste material has been onsite can be verified with the CRM records discussed above in Section 3.1.

4.2 Incoming Medical Waste Screening

Loads of waste entering the facility will be screened for radiation and inspected for potentially hazardous substances or hazardous waste. The facility will only accept medical waste and will not accept any items outlined as unacceptable in the Waste Acceptance Policy in Appendix F. Additionally, the facility will not accept household wastes (NRS 459.432 / NAC 444.581) or industrial wastes (NAC 444.585) – wastes that meet these definitions will not be accepted and will be rejected at the gate. It is unlikely household waste would be sent to the facility since a household waste generator would not want to pay ten (10) times the disposal costs to dispose of household waste at this facility.

Waste containers will be scanned for radiation using a handheld radiation detector, and scanning will be conducted within 6 inches from the sides of the container. The facility will not accept radioactive waste. MediWaste defines "radioactive waste" as any reading of the radiation detector that is three times the background levels as defined by the Community Environmental Monitoring Program (CEMP) station summary for Pahrump, NV, maintained by the Desert Research Institute. Based on an average reading, MediWaste defines radioactive as 36 μ R/h. Radioactive material will not be accepted at the facility and will be sent back to the customer for proper management within 24 hours. If the waste contains radioactive elements like Iodine or Barium, the detection levels will be far above our limit. However, the pyrolysis system is not impacted by radioactivity. Radioactivity is a process in the nucleus of atoms. Pyrolysis is a process in the electrons of atom, thus these processes are not related.

Containers will be visually inspected for evidence of potentially hazardous substances or hazardous waste. Evidence of potentially hazardous substances includes containers with federal and state-only hazardous waste labelling (including containers marked as Hazardous Waste Pharmaceuticals) or containers known to be used to contain federal or state-only hazardous waste, the presence of liquids other than water accumulating in the bottom of the container, and potentially olfactory cues (e.g., petroleum hydrocarbon or solvent-like odors). The shipping paperwork the waste arrived on will also be inspected to confirm conformance with the Waste Acceptance Policy. Any material that arrives marked as federal, state-only listed, or RCRA Subpart P hazardous waste (coded PHRM or PHARMS) on a hazardous waste manifest will be considered hazardous waste and will be rejected. If a container is found to contain a hazardous substance, including polychlorinated biphenyl (PCB) waste, then all of the waste contained in that container will be considered hazardous and will be rejected at the gate.

Per the Waste Acceptance Policy in Appendix F, the following items cannot be accepted at the MediWaste facility:

- Untreated Category A Infectious Substances (per DOT Division 6.2)
- Putrescible Wastes as defined in NAC 444.608
- RCRA Hazardous Waste as defined under NAC 444.580
 - This includes drums or other containers with a hazard warning symbol, batteries and other heavy metals.
- RCRA Hazardous Pharmaceutical Waste as defined by 40 CFR 266 Subpart P
 - Nevada has not adopted 40 CFR 266 Subpart P. Pharmaceutical waste from other states managed under Subpart P is considered fully regulated RCRA hazardous waste once it crosses the border into Nevada.
- DEA controlled drugs, including controlled substances
- Radioactive wastes (above 36 μ R/h)
- Chemicals including formaldehyde, formalin, acids, alcohol, waste solvents reagents, fixer developer
- Lead-containing materials
- Complete Human Remains (including heads, full torsos and fetuses)
- Bulk Chemotherapy Waste
- Compressed Glass Cylinders, Canister, Inhalers and Aerosol Cans
- Any Mercury-Containing Material or Devices
 - Any mercury thermometers, Sphygmomanometers, lab or medical devices.
- Mercury-Containing Dental Waste
 - Non-contact and contact amalgam and products, chairside, amalgam sludge or vacuum pump filters, extracted teeth with mercury filings and empty amalgam capsules.

Per the Waste Acceptance Policy in Appendix F, the following items can be accepted at the MediWaste Facility:

- Sharps
 - Any object contaminated with a pathogen or that may become contaminated with a pathogen through handling or during transportation and also capable of cutting or penetrating skin or packaging material. Sharps includes needles, syringes, scalpels, broken glass, culture slides, culture dishes, broken capillary tubes, broken rigid plastic and exposed ends of dental wires.
- Regulated Medical Waste or Clinical Waste or (Bio) Medical Waste as defined in NAC 444.589
- Trace Chemotherapy Contaminated Waste
 - RCRA empty drug vials, syringes and needles, split kits, IV tubing and bags, contaminated gloves and gowns, and regulated materials as defined in applicable laws, rules, regulations or guidelines.
- Pathological Waste
 - Human or animal body parts, organs, tissues and surgical specimen (decanted of formaldehyde, formalin or other preservatives, as required per hazardous waste rules).
- Non-RCRA Pharmaceuticals
 - Must be characterized and certified as non-RCRA hazardous waste by the generator. Excludes all DEA scheduled drugs, including controlled substances.
 - Does not include pharmaceuticals managed under 40 CFR 266 Subpart P in other states. Nevada has not adopted the alternative management standards and pharmaceutical waste from other states managed under Subpart P is considered fully regulated RCRA hazardous waste once it crosses the border into Nevada.
- California Only – Solidified Canisters
 - Suction canisters that have been injected with solidifier materials to control liquids or suction canisters made of high heat resistant plastics such as polysulfide.

4.3 Handling and Storage of Hydrated Ash

The treatment of medical waste via pyrolysis will generate hydrated ash. Samples of the accumulated hydrated ash will be collected for characterization and disposal. The anticipated sampling program to characterize the hydrated ash for disposal is presented in Section 4.4. The filled, covered, trailers containing ash will be moved to a designated storage area (noted as the "Ash Loading Area" in Figure 3 of Appendix D) in the asphalt area in the rear of the facility pending characterization and disposal. The ash is a solid and will not contain any free liquids. The material will be held in a fully enclosed 40-yard roll-off container. The container loading will be completed by trained operators. In addition, there will be a full recording of additions to the container that includes the day, time, weight, and initial waste code (if applicable). This recording process will also require a visual inspection of the container and surrounding area to ensure any material spills are noted and cleaned up immediately. The ash will be collected until the facility has enough volume for a full load (approximately one end dump trailer or transfer trailer which can hold 23 tons) and will then be shipped to the appropriately permitted and NDEP approved disposal facility.

4.4 Characterization of Hydrated Ash for Disposal

The hydrated ash will be sampled for waste characterization prior to final disposal at an appropriately permitted and NDEP approved disposal facility. Laboratory samples will be analyzed for toxicity using the Resource Conservation and Recovery Act (RCRA) Hazardous Waste Test Methods (SW-846) Toxicity Characteristic Leaching Procedure (TCLP) by Method SW-846 1311 which is used to quantify the mobility of organic and inorganic constituents in potentially hazardous waste. The following analyses will be performed on the samples:

- TCLP Volatiles by SW-846 8260

- ▶ TCLP Semivolatiles by SW-846 8270
- ▶ TCLP Metals by SW-846 6010B/7470A

EPA Method 8290A will be used to test for dioxins and furans. In addition, samples will be analyzed for the hazardous characteristics of ignitability, corrosivity (if any free liquids are present), and reactivity. The ash generated at the other facilities that utilize Vulcan's pyrolysis equipment to process medical waste have not had any instances of the ash analytical coming back as hazardous waste. The ash sampling reports are included in Appendix J. In the unlikely event the results indicate the waste is hazardous, then the entirety of the parent material from which the sample was taken and any ash it was stored with will be considered hazardous and disposed of at a permitted hazardous waste landfill. Nonhazardous waste will be disposed of at an appropriately permitted and NDEP approved disposal facility. Sample results will be shared with the disposal facility for the purposes of developing waste profiles for acceptance.

4.4.1 Sampling Frequency

MediWaste will employ a phased sampling approach to characterizing the ash as described in the Sampling and Analysis Plan provided in Appendix K.

5. DISPOSAL FACILITIES

The hydrated ash will only be shipped to appropriately permitted and NDEP approved disposal sites. MediWaste may use multiple facilities depending on what each landfill is accepting at the time of disposal. MediWaste has existing service contracts with 24-hour HazMat response contractors listed in Section 5.3 to provide immediate action in the event of a spill or release.

5.1 Municipal Solid Waste Facilities

MediWaste will validate that any selected disposal facility has the appropriate permits and approvals to accept the waste. The disposal facility will likely require testing results to verify the characteristics of the waste. For context, this business practice and pyrolysis equipment is in use in two medical waste processing facilities, one in California and one in Texas. Neither facility has experienced issues finding a compliant disposal option for the hydrated ash or rejected medical waste.

MediWaste does not have any written agreements to accept the hydrated ash or medical waste. Typically, a landfill will not accept a waste unless they have received sampling data detailing the characteristics of a waste which cannot be done until the facility begins operations. In addition to requiring sampling data, landfills may periodically not accept a certain waste stream due to other volumes received at the facility and the current activities at a landfill. All of these variables mean that each load would receive a "spot price" based on the status of the landfill.

The process typically goes as follows:

- ▶ A relationship is generally created with multiple landfills (local, in-state, out of state) which are contacted when a load of hydrated ash needs to be disposed of.
- ▶ Each load is tested by the landfill before they accept it.
- ▶ Each load will vary in cost depending on "current" activities in the landfill.
- ▶ All prices are "spot prices".
- ▶ An overarching agreement can exist, but loads are treated as unique and discrete events.

5.2 Hazardous Waste Facilities

In the unlikely event the hydrated ash is determined to be hazardous waste, the ash would be disposed of at a permitted hazardous waste disposal facility. MediWaste would comply with the applicable federal hazardous waste management and disposal requirements detailed in 40 CFR 262. If greater than 220 pounds of non-acute hazardous waste is generated, the facility would obtain an EPA ID number. If the material can be managed under the episodic waste provisions found at 40 CFR 262 Subpart L, a notification would be sent to NDEP and all applicable management and disposal requirements followed.

5.3 24-hour Hazardous Material Response

- ▶ Clean Harbors Environmental Services, 4500 N Walnut Rd, North Las Vegas, Nevada 89081
 - Phone: 702-258-0109
- ▶ Patriot Environmental Services, 647 Cape Horn Drive, Henderson, Nevada 89011
 - Phone: 800-624-9136

6. TRAINING PLAN

MediWaste has a robust internal training program to train all employees on systems, equipment, and health and safety protocols. All new employees receive the Occupational Safety and Health Administration (OSHA) required HazCom training and Bloodborne Pathogen training before being allowed to work in the portion of the facility that contains waste. All employees receive annual refresher training, where mandated, that includes the above topics.

A training course will be provided to all employees involved in the handling and tracking of medical waste. MediWaste will provide all necessary personal protective equipment (PPE) to employees for the handling and processing of medical waste.

MediWaste has a "Safety 52" program where it covers a topic every week, including, but not limited to, confined space, lock out/tag out, heat illness, fire prevention, emergency action plan, injury and illness prevention program (IIPP), PPE use, and Autoclave 101. Training sessions are documented using sign in sheets, and the data is stored in the company computer system. Site specific training will also be conducted that includes location of emergency equipment, what to do in case of a spill, operation of the pyrolysis process, waste management, and storage locations of medical waste at the facility.

7. EMERGENCY PLAN

MediWaste is in contact with representatives from Pahrump and Nye County, including first responders and emergency response organizations. MediWaste has made local authorities aware of their planned operations and implications for emergency responders. Emergency response organizations are contacted and made aware of operations as a part of the Conditional Use Permit planning to be issued by Nye County.

7.1 Emergency Action Plan

MediWaste has prepared an emergency action plan for the facility in the unlikely event that MediWaste would experience a shutdown of operations, precautions are in place in the event of natural disasters or other unforeseeable circumstances (e.g., power outages, nuclear attack, or other event which may restrict access to the facility). These contingencies include use of an alternate approved medical waste haulers that have agreements in place (and can provide upon request), as well as contracts in place with secondary (in-state) disposal facility and a tertiary (out-of-state) treatment facility should conditions require their use. The facilities are listed in Sections 5.1 and 5.2.

Nevada regulations do not require the treatment of medical waste prior to disposal in a permitted landfill, therefore, the Apex Regional Landfill will be the first choice for disposal of unprocessed medical waste during a facility shutdown given their proximity to the facility. See the Section 5.1 for a discussion on how MediWaste would validate other disposal facilities how the acceptance process would work.

MediWaste uses a cloud-based communications system (VOIP), in the event that its location is compromised, calls will be routed to cell phones through the regular phone numbers. Operations also work off a cloud-based system so that teams will be able to access our data and system as long as they have power and internet access.

In the event any of fleet damage, MediWaste has contracts in place to access rentals to service our clients. In the event of an on-site emergency need such as small spills, MediWaste has the required spill kits on each of its fleet vehicles and is trained in proper cleanup and reporting. Larger spills will be managed in accordance with the planned facility spill prevention, control, and countermeasures (SPCC) and stormwater pollution prevention (SWPP) plans.

7.2 Fire Suppression

Building 2 is outfitted with a sprinkler system that is automatically activated if fire or smoke is detected. The fire suppression system is already constructed. The facility will also be outfitted with fire extinguishers throughout the facility. The locations of fire suppression systems including valves, control points, and fire extinguishers will be clearly displayed on placards throughout the facility.

7.3 NDEP Reporting Requirements

In the event of an emergency involving a release of hazardous waste or hazardous constituents, any fire or explosion at the facility, or any condition which may endanger human health or the environment, the following reporting requirements must be followed:

Information such as any imminent or existing hazard from a release of waste or hazardous constituents, any fire or explosion at the facility, or any condition which may endanger human health or the environment shall be reported to the Division within 24 hours from the time the facility becomes aware of the circumstances by any of the following methods:

- ▶ Emergency Response Commission (Spill Hotline) telephone at 888.331.6337
- ▶ Online: <https://nevadaenvironmentalactivities.ndep.nv.gov/Spill/ReportForm.aspx>

8. FACILITY CLOSURE PLAN

In the event that MediWaste decides to cease operations and close the facility, MediWaste will notify NDEP and the local solid waste management authority in writing at least 90 days before beginning final closure of the facility. Beginning final closure is defined as the date when the facility is no longer accepting medical waste.

Within 30 days after the receipt of the final shipment of medical waste, MediWaste will remove all remaining solid waste, litter, recovered materials and equipment from the facility. Operable equipment which may be utilized by the next facility owner will be left on-site in acceptable working condition along with a written agreement from the next owner for transfer of the specific equipment. If the next owner will not utilize the equipment, MediWaste will dismantle and remove the equipment from the facility. The facility will be cleaned to verify that no unprocessed waste or residual materials are remaining at the facility or on the equipment.

An engineering cost estimate was developed to evaluate the cost of closing the facility if MediWaste ceases operations and the financial assurance method (Section 9) will be used to cover the cost of closure. A table of costs that comprise the estimate is presented in the Closure Plan in Appendix E. The closure cost assumes that the facility will be storing the maximum amount of medical waste allowable at time of closure, that the pyrolysis units and other equipment will be removed, and that the facility will be cleaned. The cost estimate was developed to be a conservative estimate of closure costs and a 15% contingency factor was applied to all costs.

9. FINANCIAL ASSURANCE

MediWaste has arranged for the appropriate level of financial assurance via their insurance policy to cover the cost of closure in accordance with NAC 444.6855 ("Financial assurance: Insurance"). The policy of insurance will state that the insurer may not cancel, terminate, or fail to renew the policy except for a failure to pay the premium in accordance with NAC 444.6855. A copy of the certificate of liability insurance (COI) for the property is provided in Appendix C. The COI provides up to two-million dollars in general aggregate coverage which is more than adequate to cover costs associated with implementing the closure plan.

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10. REFERENCES

NDEP. 2022. *2022 State of Nevada: Sustainable Materials Management Plan - Aligning Solid Waste, Hazardous Waste, and Other Materials Management Processes with Sustainable Practices*. Revised December 14, 2022.

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**APPENDIX A. PROOF OF PROPERTY OWNERSHIP & EVIDENCE OF
PROPERTY INSURANCE**

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NEVADA

STANDARD INDUSTRIAL/COMMERCIAL SINGLE-TENANT LEASE - NET (DO NOT USE THIS FORM FOR MULTI-TENANT BUILDINGS)

1. Basic Provisions ("Basic Provisions").

1.1 **Parties.** This Lease ("Lease"), dated for reference purposes only 01/25/2024, is made by and between Basin Ave Properties LLC ("Lessor") and MediWaste Disposal LLC ("Lessee"), (collectively the "Parties", or individually a "Party").

1.2 **Premises:** That certain real property, including all improvements therein or to be provided by Lessor under the terms of this Lease, commonly known as (street address, city, state, zip): 1850 East Basin Avenue, Pahrump, NV 89060 ("Premises"). The Premises are located in the County of Nye, and are generally described as (describe briefly the nature of the Premises and, if applicable, the "Project"): an approximate 4.70 acre industrial zoned lot with two steel framed warehouse buildings totaling an approximate 12,113 square feet. (See also Paragraph 2)

1.3 **Term:** 15 years and 0 months ("Original Term") commencing the day following the Lessor's purchase of the Premises ("Commencement Date") and ending the fifteenth anniversary of the Commencement Date ("Expiration Date"). (See also Paragraph 3)

1.4 **Early Possession:** If the Premises are available Lessee may have non-exclusive possession of the Premises commencing N/A ("Early Possession Date"). (See also Paragraphs 3.2 and 3.3)

1.5 **Base Rent:** [REDACTED] per month ("Base Rent"), payable on the 1 day of each month commencing upon the Commencement Date. (See also Paragraph 4)

☒ If this box is checked, there are provisions in this Lease for the Base Rent to be adjusted. See Paragraph See Addendum 1.

1.6 Base Rent and Other Monies Paid Upon Execution:

- (a) **Base Rent:** _____ for the period _____.
- (b) **Security Deposit:** _____ ("Security Deposit"). (See also Paragraph 5)
- (c) **Association Fees:** _____: for the period _____.
- (d) **Other:** _____ for _____.
- (e) **Total Due Upon Execution of this Lease:** \$0.

1.7 **Agreed Use:** General industrial use and any other legal use. (See also Paragraph 6)

1.8 **Insuring Party.** Lessor is the "Insuring Party" unless otherwise stated herein. (See also Paragraph 8)

1.9 **Real Estate Brokers.** (See also Paragraph 15 and 25)

(a) **Representation.** The following real estate broker(s) ("Broker(s)") and brokerage relationships exist in this transaction (check the applicable boxes):

- ☐ _____ represents Lessor exclusively ("Lessor's Broker");
- ☐ _____ represents Lessee exclusively ("Lessee's Broker"); or
- ☐ _____ represents both Lessor and Lessee ("Dual Agency").

(b) **Payment to Brokers.** Upon execution and delivery of this Lease by both Parties, Lessor shall pay to the Brokers the brokerage fee agreed to in a separate written agreement (or if there is no such agreement, the sum of _____ or _____ % of the total Base Rent) for the brokerage services rendered by the Brokers.

1.10 **Guarantor.** The obligations of the Lessee under this Lease are to be guaranteed by MediWaste Disposal LLC ("Guarantor"). (See also Paragraph 37)

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45. Conflict. Any conflict between the printed provisions of this Lease and the typewritten or handwritten provisions shall be controlled by the typewritten or handwritten provisions.

46. Offer. Preparation of this Lease by either party or their agent and submission of same to the other Party shall not be deemed an offer to lease to the other Party. This Lease is not intended to be binding until executed and delivered by all Parties hereto.

47. Amendments. This Lease may be modified only in writing, signed by the Parties in interest at the time of the modification. As long as they do not materially change Lessee's obligations hereunder, Lessee agrees to make such reasonable non-monetary modifications to this Lease as may be reasonably required by a Lender in connection with the obtaining of normal financing or refinancing of the Premises.

48. Waiver of Jury Trial. THE PARTIES HEREBY WAIVE THEIR RESPECTIVE RIGHTS TO TRIAL BY JURY IN ANY ACTION OR PROCEEDING INVOLVING THE PROPERTY OR ARISING OUT OF THIS LEASE.

49. Arbitration of Disputes. An Addendum requiring the Arbitration of all disputes between the Parties and/or Brokers arising out of this Lease ☐ is ☒ is not attached to this Lease.

50. Accessibility; Americans with Disabilities Act. Since compliance with the Americans with Disabilities Act (ADA) and other state and local accessibility statutes are dependent upon Lessee's specific use of the Premises, Lessor makes no warranty or representation as to whether or not the Premises comply with ADA or any similar legislation. In the event that Lessee's use of the Premises requires modifications or additions to the Premises in order to be in compliance with ADA or other accessibility statutes, Lessee agrees to make any such necessary modifications and/or additions at Lessee's expense.

LESSOR AND LESSEE HAVE CAREFULLY READ AND REVIEWED THIS LEASE AND EACH TERM AND PROVISION CONTAINED HEREIN, AND BY THE EXECUTION OF THIS LEASE SHOW THEIR INFORMED AND VOLUNTARY CONSENT THERETO. THE PARTIES HEREBY AGREE THAT, AT THE TIME THIS LEASE IS EXECUTED, THE TERMS OF THIS LEASE ARE COMMERCIALY REASONABLE AND EFFECTUATE THE INTENT AND PURPOSE OF LESSOR AND LESSEE WITH RESPECT TO THE PREMISES.

ATTENTION: NO REPRESENTATION OR RECOMMENDATION IS MADE BY AIR CRE OR BY ANY BROKER AS TO THE LEGAL SUFFICIENCY, LEGAL EFFECT, OR TAX CONSEQUENCES OF THIS LEASE OR THE TRANSACTION TO WHICH IT RELATES. THE PARTIES ARE URGED TO:

1. SEEK ADVICE OF COUNSEL AS TO THE LEGAL AND TAX CONSEQUENCES OF THIS LEASE.
2. RETAIN APPROPRIATE CONSULTANTS TO REVIEW AND INVESTIGATE THE CONDITION OF THE PREMISES. SAID INVESTIGATION SHOULD INCLUDE BUT NOT BE LIMITED TO: THE POSSIBLE PRESENCE OF HAZARDOUS SUBSTANCES, THE ZONING OF THE PREMISES, THE STRUCTURAL INTEGRITY, THE CONDITION OF THE ROOF AND OPERATING SYSTEMS, COMPLIANCE WITH THE AMERICANS WITH DISABILITIES ACT AND THE SUITABILITY OF THE PREMISES FOR LESSEE'S INTENDED USE.

WARNING: IF THE PREMISES ARE LOCATED IN A STATE OTHER THAN NEVADA, CERTAIN PROVISIONS OF THE LEASE MAY NEED TO BE REVISED TO COMPLY WITH THE LAWS OF THE STATE IN WHICH THE PREMISES ARE LOCATED.

The parties hereto have executed this Lease at the place and on the dates specified above their respective signatures.

Executed at: Corona, CA

On: 1/25/2024

By LESSOR:

Basin Ave Properties LLC

By: Ryan Oganesian

Name Printed: Ryan Oganesian

DS
RO

INITIALS

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STNNV-1.00, Revised 10-22-2020

Executed at: Corona, CA

On: 1/25/2024

By LESSEE:

MediWaste Disposal LLC

By: Ryan Oganesian

Name Printed: Ryan Oganesian

DS
RO

INITIALS

Last Edited: 1/25/2024 4:34 PM

Page 26 of 27

DOC #1027029

Official Records Nye County NV
Deborah Beatty - Recorder
03/13/2024 12:59:38 PM
Requested By: FIRST AMERICAN TITLE
Recorded By: sb RPTT: \$7,020.00
Recording Fee: \$37.00
Page 1 of 4

A.P.N.: 035-191-52

File No: NCS-1188644-HHLV (KR)

R.P.T.T.: \$7,020.00

When Recorded Mail To: Mail Tax Statements To:
Basin Ave Properties LLC
PO Box 6579
Corona, CA 92878

GRANT, BARGAIN and SALE DEED

FOR A VALUABLE CONSIDERATION, receipt of which is hereby acknowledged,

Pinnacle Propane Express, LLC, a Delaware limited liability company

do(es) hereby *GRANT, BARGAIN and SELL* to

Basin Ave Properties LLC, a California limited liability company

the real property situate in the County of Nye, State of Nevada, described as follows:

A PORTION OF THE SOUTHEAST QUARTER (SE ¼) OF THE SOUTHEAST QUARTER (SE ¼) OF SECTION 11, TOWNSHIP 20 SOUTH, RANGE 53 EAST, M.D.M., MORE PARTICULARLY DESCRIBED AS

PARCEL 2 AS SHOWN BY PARCEL MAP RECORDED FEBRUARY 9, 2000 AS FILE # 487360 OF OFFICIAL RECORDS, NYE COUNTY, NEVADA.

TOGETHER with all tenements, hereditaments and appurtenances, including easements and water rights, if any, thereto belonging or appertaining, and any reversions, remainders, rents, issues or profits thereof.

Subject to

1. All general and special taxes for the current fiscal year.
2. Covenants, Conditions, Restrictions, Reservations, Rights, Rights of Way and Easements now of record.

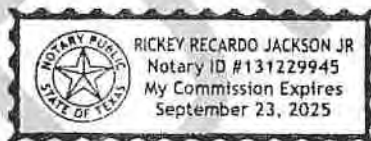
Pinnacle Propane Express, LLC, a Delaware limited liability company

By: [Signature]
Name: Barbara Vecchio
Title: Chief Financial Officer

STATE OF NEVADA TEXAS ss.
COUNTY OF CLARK DALLAS

This instrument was acknowledged before me on MARCH 7, 2024 by
Barabara Vecchio as Chief Financial Officer of Pinnacle Propane Express, LLC.

[Signature]
Notary Public
(My commission expires: Sept 23, 2025)



**STATE OF NEVADA
DECLARATION OF VALUE**

1. Assessor Parcel Number(s)

- a) 035-191-52
b) _____
c) _____
d) _____

2. Type of Property

- a) ☐ Vacant Land b) ☐ Single Fam. Res.
c) ☐ Condo/Twnhse d) ☐ 2-4 Plex
e) ☐ Apt. Bldg. f) ☒ Comm'l/Ind'l
g) ☐ Agricultural h) ☐ Mobile Home
i) ☐ Other _____

FOR RECORDERS OPTIONAL USE ONLY

Book _____ Page: _____
Date of Recording: _____
Notes: _____

3. a) Total Value/Sales Price of Property: \$1,800,000.00
b) Deed in Lieu of Foreclosure Only (value of property) (\$ _____)
c) Transfer Tax Value: \$1,800,000.00
d) Real Property Transfer Tax Due \$7,020.00

4. **If Exemption Claimed:**

- a. Transfer Tax Exemption, per 375.090, Section: _____
b. Explain reason for exemption: _____

5. Partial Interest: Percentage being transferred: 100 %

The undersigned declares and acknowledges, under penalty of perjury, pursuant to NRS 375.060 and NRS 375.110, that the information provided is correct to the best of their information and belief, and can be supported by documentation if called upon to substantiate the information provided herein. Furthermore, the parties agree that disallowance of any claimed exemption, or other determination of additional tax due, may result in a penalty of 10% of the tax due plus interest at 1% per month. Pursuant to NRS 375.030, the Buyer and Seller shall be jointly and severally liable for any additional amount owed.

Signature: _____ Capacity: Grantor
Signature: _____ Capacity: Grantee

**SELLER (GRANTOR) INFORMATION
(REQUIRED)**

Print Name: Pinnacle Propane Express, LLC
Address: 600 E. LAS COLINAS BLVD STE 1650
City: IRVING
State: TX Zip: 75039

**BUYER (GRANTEE) INFORMATION
(REQUIRED)**

Print Name: Basln Ave Properties LLC
Address: PO Box 6579
City: Corona
State: CA Zip: 92878

COMPANY/PERSON REQUESTING RECORDING (required if not seller or buyer)

First American Title Insurance
Company National Commercial
Print Name: Services File Number: NCS-1188644-HHLV KR/ LP
Address 8311 W. Sunset Road, Suite 100
City: Las Vegas State: NV Zip: 89113

(AS A PUBLIC RECORD THIS FORM MAY BE RECORDED/MICROFILMED)

**STATE OF NEVADA
DECLARATION OF VALUE**

1. Assessor Parcel Number(s)

a) 035-191-52

b)

c)

d)

2. Type of Property

a) ☐ Vacant Land b) ☐ Single Fam. Res.c) ☐ Condo/Twnhse d) ☐ 2-4 Plexe) ☐ Apt. Bldg. f) ☒ Comm'l/Ind'lg) ☐ Agricultural h) ☐ Mobile Homei) ☐ Other**FOR RECORDERS OPTIONAL USE ONLY**

Book _____ Page: _____

Date of Recording: _____

Notes: **sb- nv**

3. a) Total Value/Sales Price of Property:

\$1,800,000.00

b) Deed in Lieu of Foreclosure Only (value of property) (\$)

c) Transfer Tax Value:

\$1,800,000.00

d) Real Property Transfer Tax Due

\$7,020.00

4. **If Exemption Claimed:**

a. Transfer Tax Exemption, per 375.090, Section: _____

b. Explain reason for exemption: _____

5. Partial Interest: Percentage being transferred: 100 %

The undersigned declares and acknowledges, under penalty of perjury, pursuant to NRS 375.060 and NRS 375.110, that the information provided is correct to the best of their information and belief, and can be supported by documentation if called upon to substantiate the information provided herein. Furthermore, the parties agree that disallowance of any claimed exemption, or other determination of additional tax due, may result in a penalty of 10% of the tax due plus interest at 1% per month. Pursuant to NRS 375.030, the Buyer and Seller shall be jointly and severally liable for any additional amount owed.

Signature: _____

Capacity: Grantor

Signature: _____

Capacity: Grantee

**SELLER (GRANTOR) INFORMATION
(REQUIRED)**

Print Name: Pinnacle Propane Express, LLC

Address: 600 E Las Colinas Blvd #1650

City: Irving

State: CA

Zip: 75039

**BUYER (GRANTEE) INFORMATION
(REQUIRED)**

Print Name: Basin Ave Properties LLC

Address: PO Box 6579

City: Corona, CA

State: CA

Zip: 92878

COMPANY/PERSON REQUESTING RECORDING (required if not seller or buyer)First American Title Insurance
Company National Commercial

Print Name: Services

Address: 8311 W. Sunset Road, Suite 100

City: Las Vegas

File Number: NCS-1188644-HHLV KR/ BA

State: NV

Zip: 89113

(AS A PUBLIC RECORD THIS FORM MAY BE RECORDED/MICROFILMED)

APPENDIX B. SITE PLANS – PINNACLE PROPANE

DRAFT

APN: 035-191-52
4.71 ACRES

PARCEL 2 PER PARCEL MAP 487360, BEING EAST HALF (E 1/2) OF THE
OF THE SOUTHWEST QUARTER (SW 1/4) OF THE SOUTHEAST QUARTER (SE 1/4)
OF THE SOUTHEAST QUARTER (SE 1/4) OF SECTION 11,
TOWNSHIP 20 SOUTH, RANGE 63 EAST, M.D.M. NYE COUNTY, NEVADA.

APN: 035-191-52
4.71 ACRES

1. COVER SHEET
2. GENERAL NOTES SHEET
3. UTILITIES INC. NOTES SHEET
4. SECTION SHEET
5. DETAIL SHEET
6. GRADING PLAN
7. UTILITY / TRAFFIC PLAN
8. OPPOSITE UTILITY PLAN

A.C.	ASPHALTIC CONCRETE	NO.	NUMBER
B.C.	BEGINNING OF CURB OR BACK OF CURB	O.C.	ON CENTER
B.C.R.	BEGINNING OF CURB RETURN	P.	POWER LINE
B.D.	BENT	P.C.	POINT OF CURVATURE
B.G.	BENT	P.C.C.	POINT OF COMPOUND CURVE
B.M.	BENCHMARK	P.I.	POINT OF INTERSECTION
B.M.E.	BACK UP MEASURES	P.L.	PROPERTY LINE
B.V.	BEGINNING OF VERTICAL CURVE	P.P.	POWER LINE
C.B.	CURB AND GUTTER	P.S.C.	POINT OF CONNECTION
C.C.	CENTLINE	P.S.C.	POINT OF ADVERSE CURVE
C.C.S.	CENTERLINE	PROP.	PROPOSED
C.C.S.	CONSTRUCTIVE CONSTRUCTION	P.T.	POINT OF TANGENT
D.C.	DOWN HILL	P.V.C.	POLYVINYL CHLORIDE PIPE
D.A.	DIAMETER	P.V.I.	POINT OF VERTICAL INTERSECTION
E.	EAST	P.W.T.	PAVEMENT
E.C.	END OF CURVE	R.	RATE OF FLOW
E.C.R.	END OF CURB RETURN	R.	RADIUS
E.L.	ELEVATION	R.T.	RIGHT
E.P.	EDGE OF PAVING	R/W.	RIGHT OF WAY
E.V.C.	END OF VERTICAL CURVE	S.	SOUTH
E.V.C.	EXISTING	S.S./B.W.	SEWER SANITARY SEWER
F.H. P.L.	FINISHED FLOOR	S.D.	STORM DRAIN
F.H.	FINISHED GRADE	SHT.	SHEET
F.H.	FIRE HYDRANT	SP. MH.	SPECIAL MANHOLE
F.L.	FLOWLINE	STA.	STATION
G.	GAS	STD.	STANDARD
GB	GRADE BREAK	SV.	SEWER
G.V.	GATE VALVE	T.	TRAVEL DISTANCE
HORIZ.	HORIZONTAL	TEL.	TELEPHONE
H.P.	HIGH POINT	T.C.	TOP OF CONCRETE
INT.	INVERT	T.O.C.	TOP OF CURB
L.S.	LANDSCAPE	T.M.H.	TOP OF MANHOLE
L.V.	LEV. VERTICAL	T.M.H.	TOP OF MANHOLE
L.F.	LINEAL FEET	T.W.	TOP OF WALL
L.F.	LEFT	TRAMS.	TRAMWAY
L.F.	LOW POINT	T.S.W.	TOP OF SIDEWALK
L.P.	LINE POINT	V.C.	VERTICAL CURVE
M.	MANHOLE	VOT.	VOLUME
M.H.	MANHOLE	V.S.	VALLEY SITTER
M.H.	MANHOLE	W.	WEST
N.	NORTH	W.P.T.	WATER
N.C.	N.Y.C. COUNTY		



PER NYC COUNTY STANDARDS, DETAILS AND SPECIFICATION FOR THE PAMPUNG REGIONAL PLANNING DISTRICT, ADOPTED JUNE 1, 1989

WAREHOUSING, 24 EMPLOYEES
WAREHOUSING, 7700 SF
ADT=3.88 * 24 = 94
ADT=4.88 * 7700 = 36
ATTN: SA

	PUBLIC	PRIVATE
TOTAL ADVERTISING REVENUE	- \$7	\$8,000.00
TITLE & ADDRESS RATES	- \$2	\$266.67
COPY & PASTE CHARGES (SEE SCHEDULE)	- \$5	\$75.00
TITLE & PASTE CHARGE CONT.	- \$5	\$66.67
NEW STAFFS	- \$5	\$0.00
* CONTRACTS RECEIVED	- \$5	\$433.33

DOUGLAS W. CONSTRUCTION
2070 S. CORTINA AVE.
PAIDEN, AK 99804
PHONE: 773-537-8767
FAX: 773-537-8778

OWNERS:
POINACILLI PRODUCTIONS
3225 STATE HWY 142
FALLS CH, KY 40368
MICHEL 775-677-6767
FAX 775-633-6778

NORTH 89°27'47" EAST, BEING THE BEARING OF THE SOUTH LINE OF PARCEL 3" AS SHOWN BY PARCEL MAP, DOCUMENT NUMBER 487,390 IN THE NYS COUNTY RECORDER'S OFFICE, NYORA.

3 KILOMETERS SOUTHWEST OF THE
INTERSECTION OF SR-180 AND
SR-372

HAD 83
FEET = 2741.14
METERS = 835.5

<i>[Signature]</i>	7/10/13	DATE
ONE CANTON PARK, SUITE 100		
<i>[Signature]</i>	7/10/13	DATE
FARMINGTON VALLEY FIRE DEPARTMENT		
<i>[Signature]</i>	7/10/13	DATE
2000 N. MAIN STREET, SUITE 100, FARMINGTON, CT 06030		
THE CANTON PLANNING		
AMERICAN ELECTRIC ASSOCIATION, INC.		DATE
<i>[Signature]</i>	7/10/13	DATE
VALLEY INC. OF CENTRAL MICHIGAN		
DATE		DATE

NOTE: IT SHALL BE THE RESPONSIBILITY OF THE CONTRACTOR TO LOCATE AND PROTECT IN PLACE ANY AND ALL EXISTING UTILITIES. ANY DAMAGE TO EXISTING UTILITIES SHALL BE REPAIRED AT NO COST TO THE OWNER.

EXISTING UTILITY LINES AND OTHER SERVICES ARE APPROXIMATELY 10% OF THE TOTAL PROJECT COST. THE CONTRACTOR IS RESPONSIBLE FOR OBTAINING THE NECESSARY PERMITS AND EXISTING UTILITY LOCATIONS OF ALL EXISTING UTILITIES ARE TO BE MAINTAINED. THE CONTRACTOR IS RESPONSIBLE FOR OBTAINING THE NECESSARY PERMITS AND EXISTING UTILITY LOCATIONS OF ALL EXISTING UTILITIES ARE TO BE MAINTAINED. THE CONTRACTOR IS RESPONSIBLE FOR OBTAINING THE NECESSARY PERMITS AND EXISTING UTILITY LOCATIONS OF ALL EXISTING UTILITIES ARE TO BE MAINTAINED.

Call
before you
Overhead

1-800-742-3330
UNILEVER WATER PURIFICATION, INC.

Call
before you
Dio

1-800-227-2900
COUNSELLING SERVICE 1-800-227-2900

NOTE:
DO NOT REUSE

100

COVER SHEET

**PINNACLE PROPANE
TOWN OF PAHRUMP, NEVADA**

[illegible]

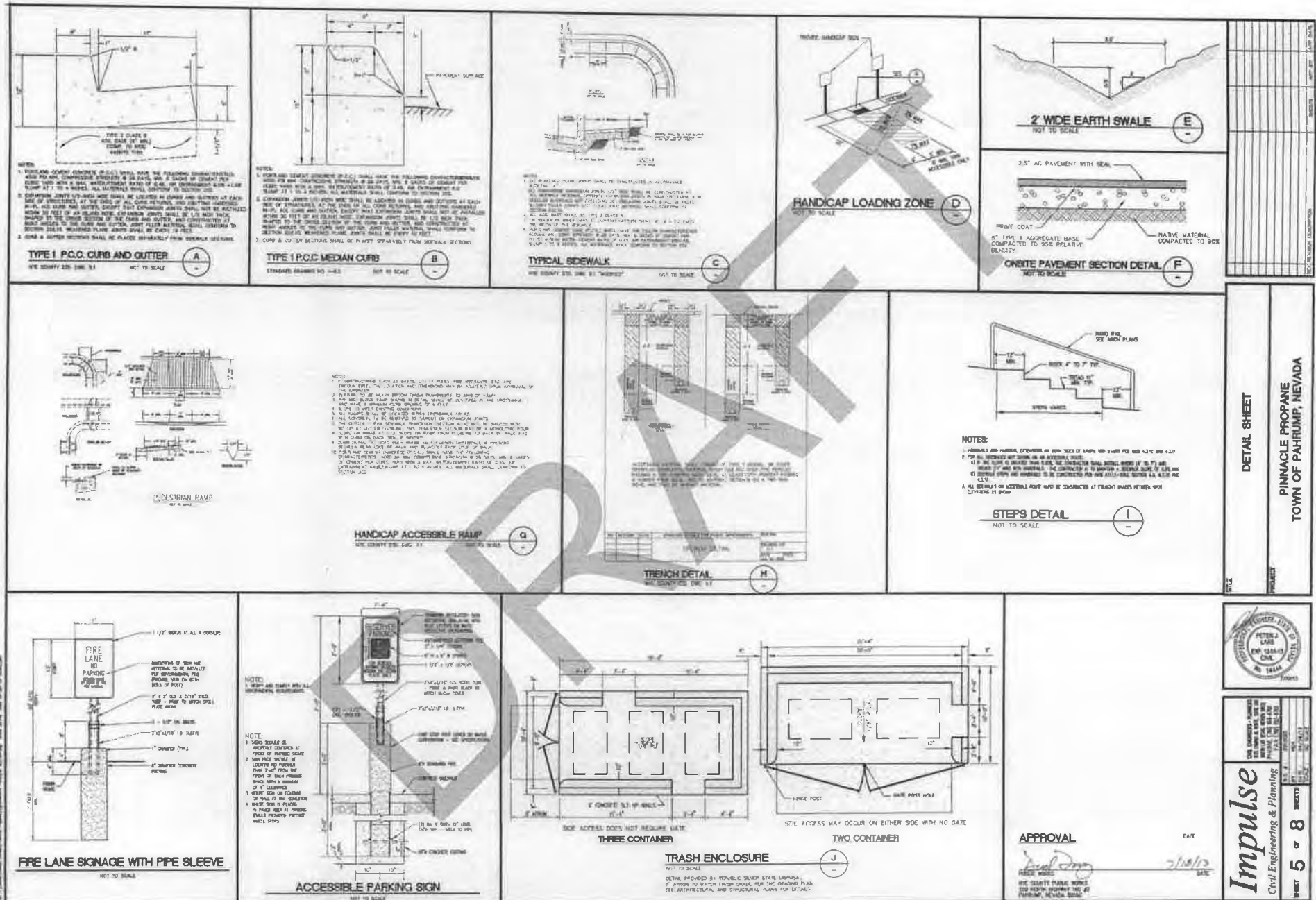
NDEP 191

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1-800-27-2800

[illegible]

**PINNACLE PROPANE
TOWN OF PAHRUMP, NEVADA**



FOR CONTINUATION SEE SHEET 8

STA 14+00 MATCHLINE



BASIN AVENUE PROFILE

LEGEND		
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APPENDIX C. CERTIFICATE OF INSURANCE

DRAFT



CERTIFICATE OF LIABILITY INSURANCE

DATE (MM/DD/YYYY)

4/15/2024

THIS CERTIFICATE IS ISSUED AS A MATTER OF INFORMATION ONLY AND CONFERS NO RIGHTS UPON THE CERTIFICATE HOLDER. THIS CERTIFICATE DOES NOT AFFIRMATIVELY OR NEGATIVELY AMEND, EXTEND OR ALTER THE COVERAGE AFFORDED BY THE POLICIES BELOW. THIS CERTIFICATE OF INSURANCE DOES NOT CONSTITUTE A CONTRACT BETWEEN THE ISSUING INSURER(S), AUTHORIZED REPRESENTATIVE OR PRODUCER, AND THE CERTIFICATE HOLDER.

IMPORTANT: If the certificate holder is an ADDITIONAL INSURED, the policy(ies) must have ADDITIONAL INSURED provisions or be endorsed. If SUBROGATION IS WAIVED, subject to the terms and conditions of the policy, certain policies may require an endorsement. A statement on this certificate does not confer rights to the certificate holder in lieu of such endorsement(s).

PRODUCER Edgewood Partners Ins Center 115 North El Molino Ave Pasadena CA 91101	CONTACT NAME: PHONE (A/C, No, Ext): (626) 795-9000 E-MAIL ADDRESS:	FAX (A/C, No):
INSURED Basin Ave Properties LLC 1850 E. Basin Ave. Pahrump, NV 89048	COAGENT	INSURER(S) AFFORDING COVERAGE INSURER A : Greenwich Insurance Company INSURER B : INSURER C : INSURER D : INSURER E : INSURER F :
		NAIC # 22322

COVERAGES

CERTIFICATE NUMBER: 251383185

REVISION NUMBER:

THIS IS TO CERTIFY THAT THE POLICIES OF INSURANCE LISTED BELOW HAVE BEEN ISSUED TO THE INSURED NAMED ABOVE FOR THE POLICY PERIOD INDICATED. NOTWITHSTANDING ANY REQUIREMENT, TERM OR CONDITION OF ANY CONTRACT OR OTHER DOCUMENT WITH RESPECT TO WHICH THIS CERTIFICATE MAY BE ISSUED OR MAY PERTAIN, THE INSURANCE AFFORDED BY THE POLICIES DESCRIBED HEREIN IS SUBJECT TO ALL THE TERMS, EXCLUSIONS AND CONDITIONS OF SUCH POLICIES. LIMITS SHOWN MAY HAVE BEEN REDUCED BY PAID CLAIMS.

INSR LTR	TYPE OF INSURANCE	ADDL INSD	SUBR WVD	POLICY NUMBER	POLICY EFF (MM/DD/YYYY)	POLICY EXP (MM/DD/YYYY)	LIMITS
A	☒ COMMERCIAL GENERAL LIABILITY ☐ CLAIMS-MADE ☒ OCCUR GEN'L AGGREGATE LIMIT APPLIES PER: ☒ POLICY ☐ PRO-JECT ☐ LOC OTHER:			GEC300089008	7/20/2023	7/20/2024	EACH OCCURRENCE \$ 1,000,000 DAMAGE TO RENTED PREMISES (Ea occurrence) \$ 100,000 MED EXP (Any one person) \$ 5,000 PERSONAL & ADV INJURY \$ 1,000,000 GENERAL AGGREGATE \$ 2,000,000 PRODUCTS - COMP/OP AGG \$ 2,000,000 \$
	AUTOMOBILE LIABILITY ☐ ANY AUTO ☐ OWNED AUTOS ONLY ☐ SCHEDULED AUTOS ☐ HIRED AUTOS ONLY ☐ NON-OWNED AUTOS ONLY						COMBINED SINGLE LIMIT (Ea accident) \$ BODILY INJURY (Per person) \$ BODILY INJURY (Per accident) \$ PROPERTY DAMAGE (Per accident) \$ \$
	UMBRELLA LIAB ☐ OCCUR EXCESS LIAB ☐ CLAIMS-MADE DED ☐ RETENTION \$						EACH OCCURRENCE \$ AGGREGATE \$ \$
	WORKERS COMPENSATION AND EMPLOYERS' LIABILITY ANY PROPRIETOR/PARTNER/EXECUTIVE OFFICER/MEMBER EXCLUDED? (Mandatory in NH) If yes, describe under DESCRIPTION OF OPERATIONS below	☐ Y ☒ N	☐ N/A				PER STATUTE ☐ OTH-ER ☐ E.L. EACH ACCIDENT \$ E.L. DISEASE - EA EMPLOYEE \$ E.L. DISEASE - POLICY LIMIT \$

DESCRIPTION OF OPERATIONS / LOCATIONS / VEHICLES (ACORD 101, Additional Remarks Schedule, may be attached if more space is required)
COMMON POLICY CONDITIONS-IL00171198

CERTIFICATE HOLDER**CANCELLATION**

Proof of Insurance

SHOULD ANY OF THE ABOVE DESCRIBED POLICIES BE CANCELLED BEFORE THE EXPIRATION DATE THEREOF, NOTICE WILL BE DELIVERED IN ACCORDANCE WITH THE POLICY PROVISIONS.

AUTHORIZED REPRESENTATIVE

COMMON POLICY CONDITIONS

All Coverage Parts included in this policy are subject to the following conditions.

A. Cancellation

1. The first Named Insured shown in the Declarations may cancel this policy by mailing or delivering to us advance written notice of cancellation.
2. We may cancel this policy by mailing or delivering to the first Named Insured written notice of cancellation at least:
 - a. 10 days before the effective date of cancellation if we cancel for nonpayment of premium; or
 - b. 30 days before the effective date of cancellation if we cancel for any other reason.
3. We will mail or deliver our notice to the first Named Insured's last mailing address known to us.
4. Notice of cancellation will state the effective date of cancellation. The policy period will end on that date.
5. If this policy is cancelled, we will send the first Named Insured any premium refund due. If we cancel, the refund will be pro rata. If the first Named Insured cancels, the refund may be less than pro rata. The cancellation will be effective even if we have not made or offered a refund.
6. If notice is mailed, proof of mailing will be sufficient proof of notice.

B. Changes

This policy contains all the agreements between you and us concerning the insurance afforded. The first Named Insured shown in the Declarations is authorized to make changes in the terms of this policy with our consent. This policy's terms can be amended or waived only by endorsement issued by us and made a part of this policy.

C. Examination Of Your Books And Records

We may examine and audit your books and records as they relate to this policy at any time during the policy period and up to three years afterward.

D. Inspections And Surveys

1. We have the right to:
 - a. Make inspections and surveys at any time;

- b. Give you reports on the conditions we find; and

- c. Recommend changes.

2. We are not obligated to make any inspections, surveys, reports or recommendations and any such actions we do undertake relate only to insurability and the premiums to be charged. We do not make safety inspections. We do not undertake to perform the duty of any person or organization to provide for the health or safety of workers or the public. And we do not warrant that conditions:
 - a. Are safe or healthful; or
 - b. Comply with laws, regulations, codes or standards.

- a. Are safe or healthful; or
 - b. Comply with laws, regulations, codes or standards.

3. Paragraphs 1. and 2. of this condition apply not only to us, but also to any rating, advisory, rate service or similar organization which makes insurance inspections, surveys, reports or recommendations.

4. Paragraph 2. of this condition does not apply to any inspections, surveys, reports or recommendations we may make relative to certification, under state or municipal statutes, ordinances or regulations, of boilers, pressure vessels or elevators.

E. Premiums

The first Named Insured shown in the Declarations:

1. Is responsible for the payment of all premiums; and
2. Will be the payee for any return premiums we pay.

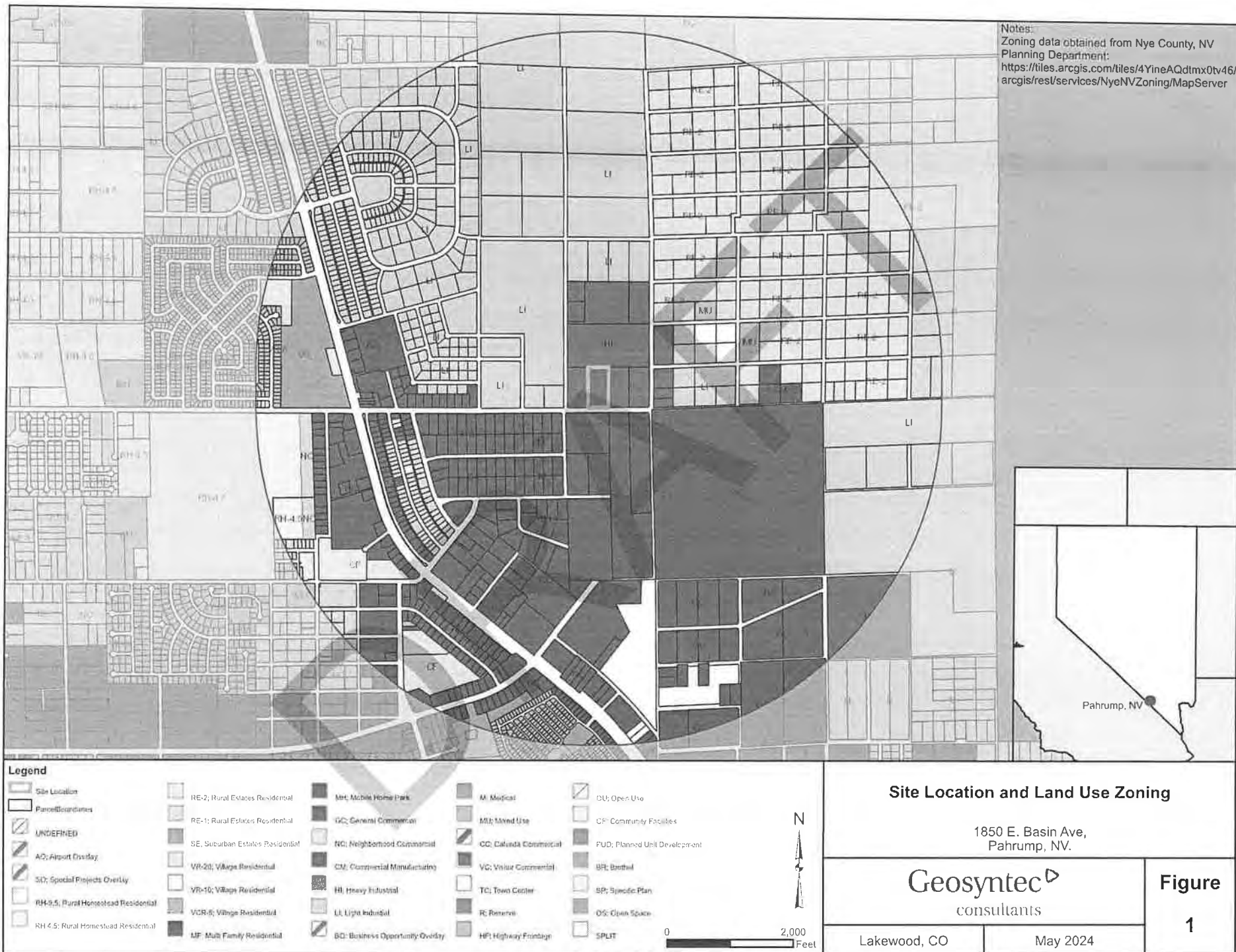
F. Transfer Of Your Rights And Duties Under This Policy

Your rights and duties under this policy may not be transferred without our written consent except in the case of death of an individual named insured.

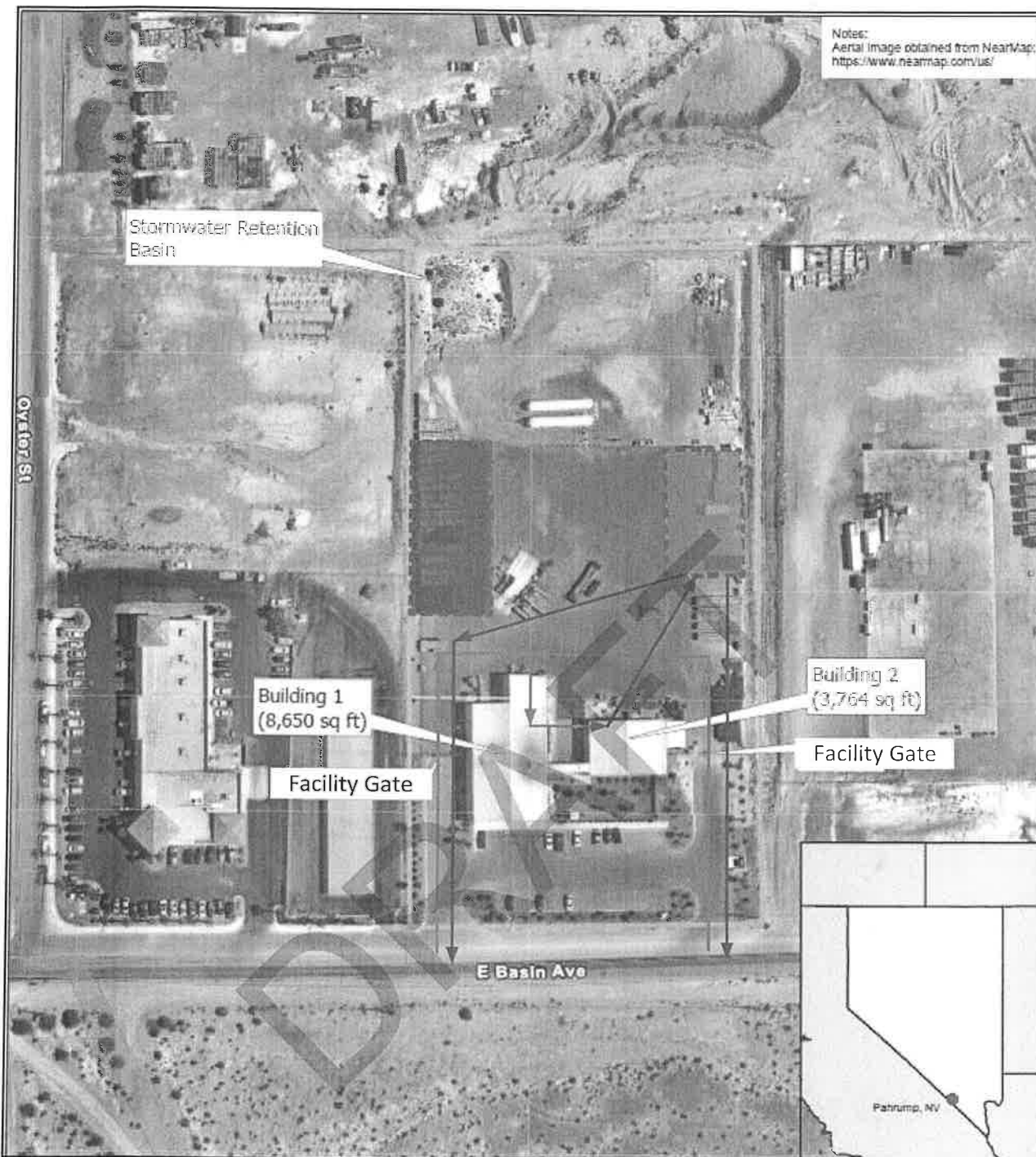
If you die, your rights and duties will be transferred to your legal representative but only while acting within the scope of duties as your legal representative. Until your legal representative is appointed, anyone having proper temporary custody of your property will have your rights and duties but only with respect to that property.

APPENDIX D. SITE MAPS AND FIGURES

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Q:\GISProjects\1850_BasinAve_Nevada\APRX\SitePlan.aprx\F01 - Site Location Land Use 4/15/2024 10:31 AM (finnian.ofarrell)



Legend

-  Site Location
-  Trailer Parking and Storage
-  Trailer Bin Storage
-  Flow of Incoming Material
-  Flow of Outgoing Ash



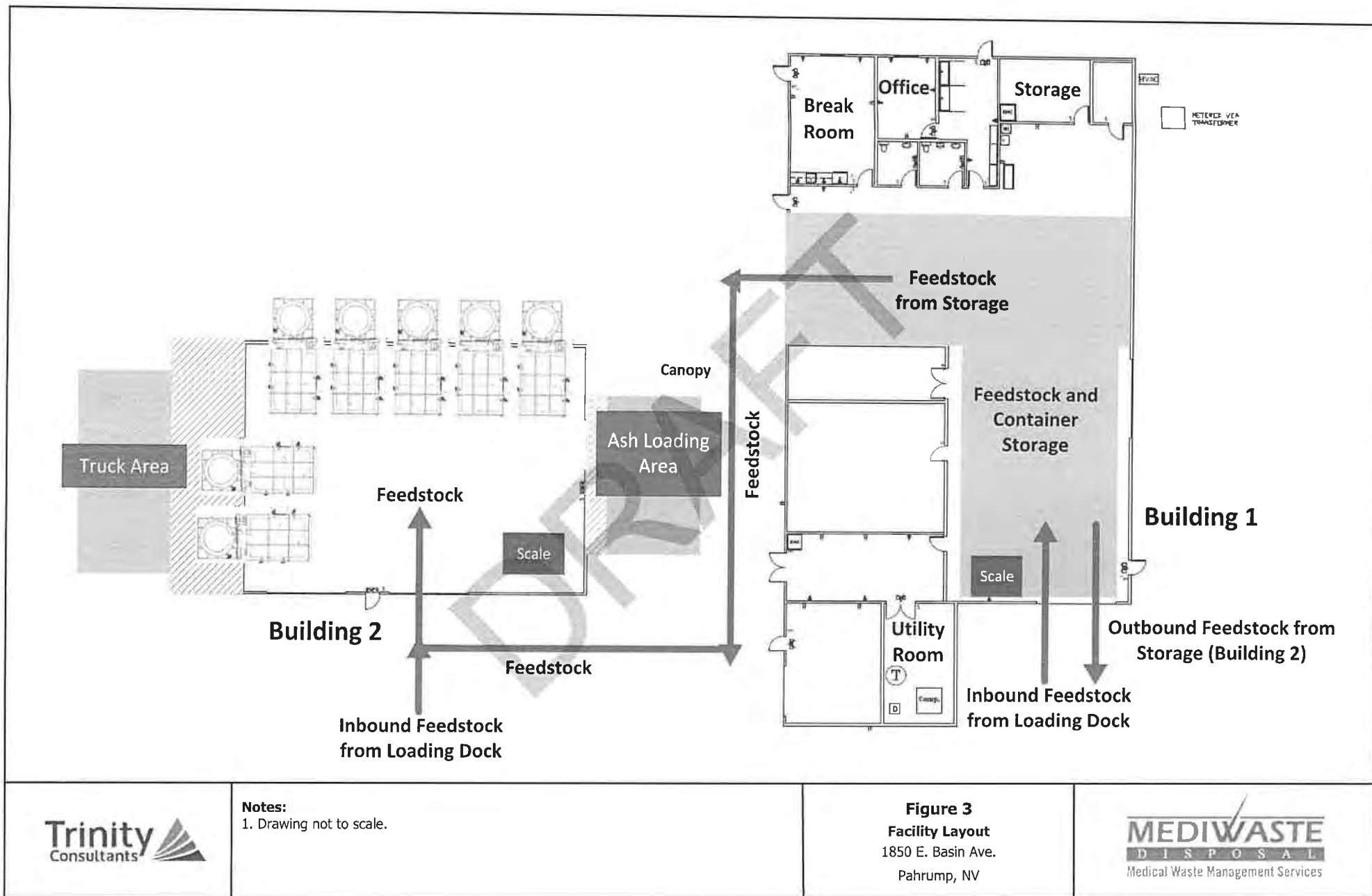
Notes:

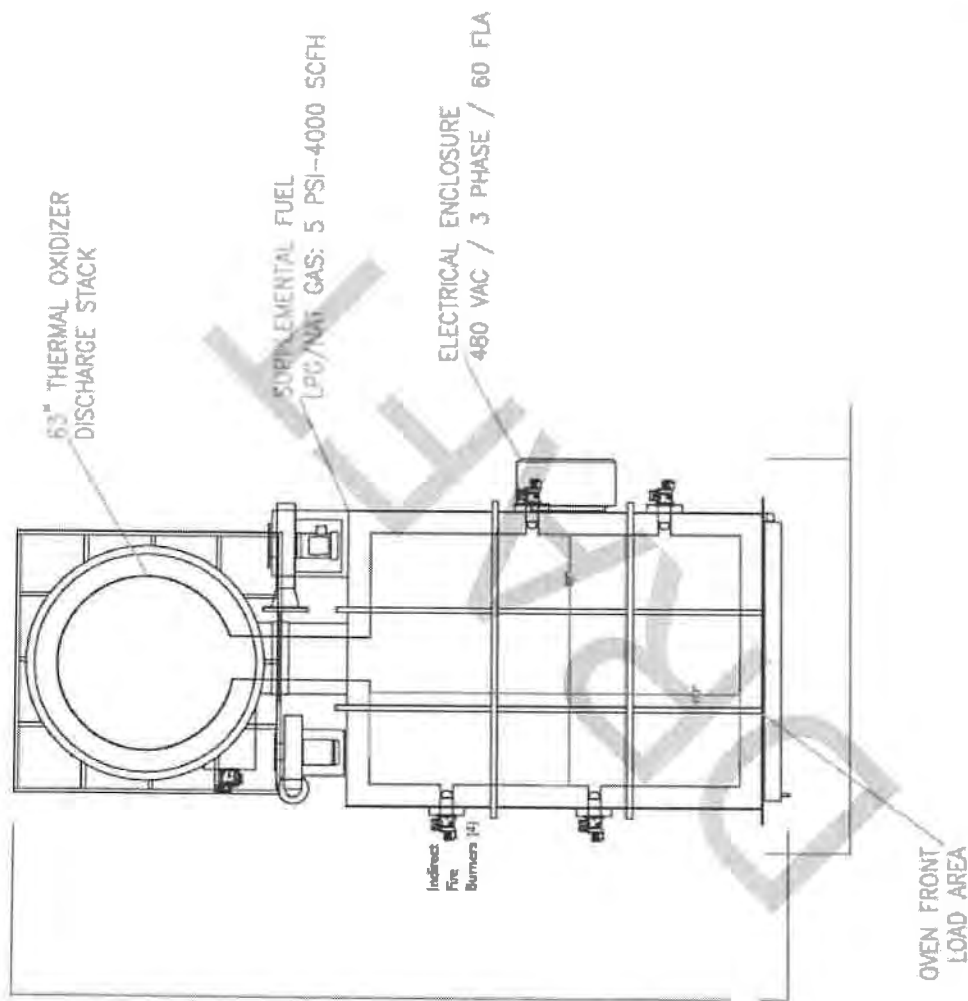
1. Drawing not to scale.

Figure 2
Site Features
1850 E. Basin Ave.
Pahrump, NV

Trinity
Consultants

MEDIWASTE
DISPOSAL
Medical Waste Management Services





Notes:
1. Drawing not to scale.



Figure 5
Pyrolysis Equipment Drawing
(Top View)
1850 E. Basin Ave.
Pahrump, NV

APPENDIX E. CLOSURE PLAN & COST ESTIMATE

DRAFT

SITE CLOSURE PLAN
Medical Waste Treatment Facility



MediWaste Disposal LLC / Pahrump, NV

Prepared By:

Matt Tarnoff – Managing Consultant
Jenna Kube – Senior Consultant

TRINITY CONSULTANTS

1575 Delucchi Lane
Suite 203
Reno, NV 89502
775-242-3200

February 2025
Revised October 2025

Project 0242901.0052



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1. SITE CLOSURE PLAN AND FINANCIAL ASSURANCE

1.1 Introduction

This Closure Plan is for the clean closure of the MediWaste LLC (MediWaste) medical waste treatment facility. MediWaste is located in Pahrump, Nevada. The facility will operate under NAC 444.676 (System to process waste: Other methods), but will adhere to the closure requirements for material recovery facilities, as required under NAC 444.74771 and 444.74747.

1.1.1 Facility Contact Information

The facility contact is Ryan Oganessian, who is the authorized agent for MediWaste.
Telephone: 714-401-3327.
Mailing address: 1850 E. Basin Avenue, Pahrump, NV, 89060.
Email: ryan@medi-waste.com

1.1.2 Facility Description

MediWaste is proposing to construct and operate a medical waste treatment facility. MediWaste's proposed treatment facility will accept medical waste to be disposed of via a pyrolysis process. MediWaste is proposing to process up to 84,000 pounds per day of medical waste on a 24-hour, 7-day per week schedule with seven (7) pyrolysis lines.

Unprocessed medical waste will be stored internally to the process buildings or in the enclosed tractor trailers in which the waste will be delivered. Either storage option will ensure the unprocessed waste is covered and protected from the elements and any potential release to the environment. The only waste to be generated from the pyrolysis process is hydrated ash. The ash will be transferred from the catch bin on the pyrolysis equipment to end dump tractor trailers or tipper transfer trailers. All containers will have lids or a tarp system to contain the ash. The pyrolysis process is designed to treat and destroy all related packaging materials, eliminating the need for any additional waste streams.

The facility is graded and paved with asphalt. The property contains two, one-story metal sided buildings suitable for offices, housing the pyrolysis process equipment, general storage, and storing medical waste containers prior to processing. The site is located on E Basin Avenue and is bounded by Oyster Street to the west and Panorama Road to the east. The site is less than a mile east of NV-160.

1.1.3 Facility History, Function, Location and Layout

MediWaste's Pahrump facility is located at 1850 E Basin Ave, Pahrump, Nevada 89060 (Nye County). The property is owned and operated by MediWaste.

The facility is secured with fencing, gates, and doors to control access to the buildings and the outside staging area behind the buildings. The facility will be equipped with a closed-circuit television camera system to monitor for unapproved personnel or activities onsite. The buildings will be outfitted with an alarm system that is activated when not occupied. The parking areas and staging areas surrounding site buildings are outfitted with lights to illuminate the areas day and night. The access road is designated as a public roadway, E Basin Avenue, with no proposed alterations. Trucks will enter the property from E Basin Avenue to deliver the medical waste or to drop the trailers for storage. The facility will be staffed by 5 to 15 full-time employees, with anticipated 24/7 operations.

1.1.4 Facility Operations

MediWaste's proposed medical waste treatment facility will accept medical waste to be treated using a pyrolysis process. Incoming medical waste for processing will be stored in enclosed tractor trailers or interior to the process buildings in lined cubic yard cardboard boxes or polyethylene bags. Medical waste treatment operations will occur inside the process buildings.

The medical waste treatment will produce hydrated ash from the pyrolysis process. The hydrated ash will be stored outside in end dump tractor trailers or tipper transfer trailers prior to shipment offsite for disposal. The ash will be stored until a full trailer bin is accumulated (approximately 23 tons of ash). MediWaste will not be accepting any waste other than medical waste.

1.1.5 Maximum Waste Inventory

The facility is planning to process approximately 84,000 pounds of medical waste per day and will receive weekly shipments of medical waste for treatment. MediWaste will keep approximately 500,000 pounds of medical waste inside the processing building and ten (10) trailer loads within enclosed tractors in the secured paved lot. Each trailer can hold a maximum of 40,000 pounds. Therefore, 900,000 pounds is the maximum amount of unprocessed medical waste that will be stored onsite at any time. Additionally, MediWaste anticipates the need to dispose of general trash associated with facility operations and closure activities.

1.2 Closure Performance Standard - NAC 444.74771

The medical waste treatment facility will be closed in a manner that complies with the requirements of NAC 444.74771 and 444.74747, and therefore achieves clean closure. The requirements of closure activities are as follows:

- ▶ A written closure plan must be compiled and shall include a detailed written estimate of the cost to hire a person to close the facility in accordance with the plan.
- ▶ The facility must notify NDEP in writing at least 90 days before the facility is expected to close.
- ▶ The facility cannot accept waste for treatment after the expected closure date.
- ▶ The facility must remove all waste, litter, and inoperable equipment within 30 days of receiving the final shipment of waste.
- ▶ No putrescible waste will be accepted at the facility, therefore removal is not necessary.

1.3 Closure Activities

This section addresses activities that will be completed during closure of the facility. The following activities are described:

- ▶ Removal of wastes and waste residues,
- ▶ Equipment dismantling and removal,
- ▶ Facility cleaning,
- ▶ Identifying and managing contaminated environmental media, and
- ▶ Confirming clean closure.

1.3.1 Removal of Wastes and Waste Residues

MediWaste will remove all unprocessed medical waste and hydrated ash residue from the facility in a staged process. The facility will determine if they can process the remaining amount of medical waste. If the

medical waste cannot be processed for any reason, it will be picked up and disposed of via a licensed disposal facility. General trash will be collected in a dumpster and hauled by a contracted third-party vendor.

1.3.2 Equipment Dismantling and Removal

The pyrolysis equipment will be dismantled and removed following the final processing of medical waste. MediWaste has estimated that the equipment dismantling and packing will require two days. The hauling is then estimated to be three days.

1.3.3 Facility Cleaning

The facility will be cleaned following the removal of the equipment and waste materials. Due to the medical waste being processed within its shipping packaging, MediWaste does not anticipate significant clean up due to the pyrolysis process. MediWaste has developed a closure cost estimate based on an average commercial cleaning fee based on square footage.

1.3.4 Identifying and Managing Contaminated Environmental Media

The medical waste treatment is located indoors and all medical waste staged for processing is fully contained. MediWaste does not anticipate contamination of soil, ground water, or other environmental media from the proposed operations. Unless there is evidence of a spill of medical waste or hydrated ash, or some other release to the environment, MediWaste does not plan to call out soil sampling.

1.3.5 Confirming Clean Closure

When closure activities are complete, MediWaste anticipates that all medical waste for treatment will be processed or removed, all hydrated ash will be disposed of, equipment will be removed, facility will be cleaned, and any affected environmental media will be removed or decontaminated. A third-party certified environmental manager (CEM) will conduct a site visit during and post site closure to ensure all appropriate clean up measures have been completed. A closure report will be completed to document all actions taken.

1.4 Closure Schedule and Timeframe

The notification of intent to close the facility will be sent, in writing, at least 90 days before beginning final closure of the recycling facility. MediWaste will remove all non-putrescible waste from the site within 30 days and will not accept any putrescible waste while operating. MediWaste will confirm the facility has been closed out following the completion of closure activities.

1.5 Cost of Closure

1.5.1 Closure Cost Estimate

The information presented in this section for implementing the Closure Plan has been prepared in accordance with NAC 444.74771 and 444.74747. The following assumptions were made:

- ▶ A third party will be used to conduct closure activities.
- ▶ The maximum waste volume will be present on site and managed during closure.
- ▶ Costs will be incurred for management of waste handled during closure.

The closure cost estimate will be adjusted annually for inflation as specified in NAC 444.74771 and 444.74747. Costs for closure labor, equipment, and analytical services are based on currently available

rates. At the time of implementation of this Closure Plan, MediWaste reserves the option to use any other appropriately permitted facility for disposal or recycling of wastes. The cost estimate for closure of the MediWaste facility is presented in Appendix A. A contingency factor has been applied to the cost estimate to address uncertainty and variability in estimating future costs.

1.5.2 Financial Assurance for Closure

Financial Assurance for closure will be procured upon the approval of the Closure Cost amount stated in Appendix A. A copy of the financial assurance mechanism will be provided in Appendix B.

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APPENDIX A. CLOSURE COST ESTIMATE

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Table 1. Closure Cost Estimate

Item	Vendor	Details	Cost
Removal of Medical Waste	Vulcan Processing LLC	900,000 pounds of medical waste at \$0.29/lb	\$261,000.00
Full Plant Decommissioning and Associated Waste Material Removal	Energy Reconnaissance, Inc.	See quote for details	\$117,206.25
Third Party Environmental Consultant Cost	Trinity Consultants	See quote for details	\$10,000.00
Contingency Factor	N/A	15% of total estimated cost (\$388,206.25)	\$58,230.94
Total Cost			\$446,437.19



1575 Delucchi Lane, Suite 203, Reno, NV 89502 / P 775.242.3200 / F 480.222.4561 / trinityconsultants.com

VIA E-MAIL: ryan@medi-waste.com

November 11, 2025

Mr. Ryan Oganessian
MediWaste
1850 E. Basin Ave.
Pahrump, NV 89061

RE: Trinity Consultants Proposal for MediWaste Site Closure Certification

Dear Mr. Oganessian,

Trinity Consultants, Inc. (Trinity) appreciates the opportunity to provide MediWaste with this proposal for completing all necessary tasks associated with site closure certification in the event the proposed medical waste treatment facility must close.

Project Background

The Site is proposing to accept regulated medical waste for treatment using a pyrolysis process. The pyrolysis process will generate a hydrated ash waste material for disposal. No other waste products will be generated during normal operation of the facility.

Scope of Work

Trinity will prepare all applicable site closure documents and transmit the documents to NDEP in the event the facility must close.

► **Site Closure Documents and Actions**

- ◆ Send written notification to NDEP 90-days prior to intended closure date
- ◆ Conduct site visit pre-closure to determine level of clean-up required
- ◆ Complete and develop Phase 1 Environmental Site Assessment (ESA) report
- ◆ Conduct final site visit to ensure all clean-up activities have been completed
- ◆ Send written notification to NDEP that site closure has been completed

Project Assumptions

This proposal was developed based on the following assumptions to arrive at a cost estimate:

- ◆ MediWaste will promptly notify Trinity of the intent to close the site.
- ◆ MediWaste will promptly provide all information as requested.
- ◆ Trinity will not be responsible for conducting closure activities (decontamination, sampling, etc.).
- ◆ Two site visits will be required for this scope of work.

HEADQUARTERS

12700 Park Central Dr, Ste 2100, Dallas, TX 75251 / P 800.229.6655 / P 972.661.8100 / F 972.385.9203

Project Costs

The budgetary estimate for completing the proposed site closure is **\$10,000**. MediWaste will be invoiced on a time-and-materials basis using Trinity's current Price Schedule. Trinity can begin work on the task via email approval or issuance of a Purchase Order when and if the facility intends to close. If MediWaste requests additional assistance beyond the scope of work identified in this proposal, Trinity can prepare a separate proposal for those additional items.

Thank you again for the opportunity to present this proposal. If you have any questions or require any additional information, please feel free to call me at (720) 879-1670.

Sincerely,

TRINITY CONSULTANTS



Jenna Kube
Senior Consultant

Energy Reconnaissance, Inc.
 1270 N. Red Gum Street
 Anaheim, CA 92806
 Phone # (714) 630-4491

Invoice

Date	Invoice #
10/27/2025	12

Bill To

MediWaste
 1850 E. Basin Ave.
 Parhump, NV 89061

P.O. Number		Terms	Rep	Ship	Via	F.O.B.	Project
TBD		Due Now			Best Method	Anaheim, CA	ERM2161
Quantity	Item Code	Description				Price Each	Amount
1	N/A	Full Plant Decommissioning - All Units Labor				88,906.25	88,906.25
2	N/A	Freight - Market Value + 10%				Current Estimate	17,500.00
		Destination - Anaheim, CA (OEM)					
3	N/A	Full Decontamination & Wash - Market Value 3 Workers - 3 Days				6 Trucks - Flat Bed Current Estimate	10,800.00
		Payment Terms: * 100% Payment With PO					117,206.25
						Subtotal	117,206.25
						Sales Tax (0.0%)	\$0.00
Thank you for your business.						Total	117,206.25
						Payments/Credits	\$0.00
						Balance Due	117,206.25

DISPOSAL AGREEMENT

This Treatment and Disposal Agreement (the "Agreement") is entered into as of this 30th day of **January 2025** (the "Effective Date") between **Medical Waste Services, LLC**, a California limited liability company with its principal office located at 7321 Quimby Street, Paramount, California 90723 (**MWS**) and **Vulcan Processing LLC** a Nevada business with its principal office located at **10805 Bernini Drive Las Vegas, NV 89141**.

WHEREAS, MWS operates a regulated medical waste and Regulated/APHIS waste treatment facility at **7321 Quimby Street, Paramount, CA 90723** (the "Facility") at which it receives regulated medical waste and Regulated/APHIS waste, treats the regulated medical waste and Regulated/APHIS waste and transports, or arranges for the transportation of, the treated waste to a landfill for disposal; and

WHEREAS Disposer is in the business of collecting regulated medical waste and/or Regulated/APHIS waste from its customers; and

WHEREAS Disposer desires to engage MWS to treat the regulated medical waste and/or Regulated/APHIS waste collected by Disposer and delivered to the Facility and properly dispose, or arrange for the proper disposal, of the waste once treated, all as set forth herein.

NOW, THEREFORE, in consideration of the mutual benefits to be derived from this Agreement and of the representations, warranties, covenants, conditions, and provisions hereinafter set forth, the parties hereto hereby agree as follows:

1. Definitions.

(a) **"Regulated medical waste"** means: isolation waste, cultures and stocks of infectious agents and associated biological human blood and blood products, pathological waste, trace chemotherapy waste, contaminated sharps, waste from surgery and autopsy, miscellaneous laboratory waste, dialysis waste or disposable linen substitutes, waste contaminated with blood or blood-borne pathogens, waste that has come into contact with infectious or pathological agents and any other waste defined as "infectious waste", "medical waste", "bio-medical waste", bio-hazardous waste" or any similar terminology used under applicable laws or regulations.

(b) **"Regulated/APHIS waste"** is defined in the regulations promulgated under 7 CFR 330.400 and 9 CFR 94.5, including, without limitation, all waste material derived in whole or in part from fruits, vegetables, meats or other plant or animal (including poultry) material and other refuse of any character whatsoever that has been associated or commingled with any such material on board any vessel or other means of conveyance (including aircraft) which has been in any port outside the United States and Canada within the previous two (2) year period. Regulated Garbage ("APHIS") also includes meals and other foods that were available for consumption by passengers and crew on an aircraft but were not consumed, and any other waste, substance or material regulated by the Animal and Plant Health Inspection Service of the United States Department of Agriculture and not described or defined herein.

(b) **"Non-conforming Waste"** means any waste, substance or other material not falling within the above definition of Regulated medical waste and Regulated/APHIS waste, including, without limitation, toxic chemicals, substances or materials, research or productive mycotoxins, heavy metals (lead or mercury), bulk chemicals or reagents, volatiles, explosives, corrosives, pressurized containers, fetal and anatomical remains, animal carcasses, bulk chemotherapy waste, radioactive waste, pharmaceuticals of all types, whether RCRA or non-RCRA, all controlled substances as defined under applicable federal or state laws and all hazardous waste or hazardous substances as defined in any applicable federal, state or local laws or regulations. In the event that MWS at any time during the term of this Agreement specifically agrees, in writing, to accept any of the foregoing waste types as part of the services to be provided to Disposer under this Agreement, such waste type shall then be deemed to be included in the definition of Regulated medical waste and Regulated/APHIS waste and shall not be considered Non-conforming Waste. "Non-conforming Waste" shall also include improperly classified and/or improperly packaged Regulated medical waste and/or Regulated/APHIS waste, any Regulated medical waste and/or Regulated/APHIS waste in containers that are damaged, broken, punctured, wet or leaking and any waste identified as unacceptable under any MWS waste acceptance protocol in affect during the term of this Agreement.

2. Duties and Obligations of the Parties.

(a) MWS shall accept and treat Regulated medical waste and Regulated/APHIS waste delivered to the Facility by Disposer to render it non-infectious and shall thereafter transport, or arrange for the transportation of, the treated waste from the Facility to a permitted sanitary landfill or other permitted facility (the "Disposal Facility") selected by MWS, in its sole discretion, for disposal (the "Services"). MWS shall provide all personnel and equipment necessary to perform the Services at MWS's sole cost and expense.

(b) Disposer shall be solely responsible for the proper packaging and labeling of the Regulated medical waste and Regulated/APHIS waste and the safe and proper transportation of such waste from the collection point to the Facility, at its sole cost and expense. All vehicles and containers shall be in good condition, be secured against spillage and leakage, comply with applicable laws and regulations and be acceptable to MWS. At all times while at the Facility, Disposer, and its employees, agents and representatives, shall abide by MWS's work, safety and other policies, procedures, rules and regulations, shall follow the directions and instructions of MWS's personnel and otherwise conduct themselves in a safe and professional manner.

(c) Regulated medical waste and/or Regulated/APHIS waste shall be delivered to the Facility by Disposer only during normal working hours, which are between 8:30 a.m. and 4:30 p.m., Monday through Friday, except holidays, unless otherwise approved, in writing by MWS. MWS shall use reasonable business efforts to promptly notify Disposer in the event the Facility becomes unavailable for any reason; provided, however, that MWS shall have no obligation or liability to Disposer in connection with the unavailability of the Facility or any deficiency in or failure to notify Disposer, nor shall MWS be responsible for securing an alternate treatment facility.

(d) Title to the Regulated medical waste and/or Regulated/APHIS waste shall pass from Disposer to MWS once the Regulated medical waste and/or Regulated/APHIS waste is fully offloaded from Disposer's vehicles at the Facility and Disposer's vehicles have left the Facility. Title to Nonconforming Waste shall at all times remain with Disposer.

(e) MWS shall have the right to inspect the contents of any vehicle to determine the presence of Non-conforming Waste, including the right to require the person operating such vehicle to unload the contents as directed by MWS. MWS shall have the right to reject any Non-conforming Waste. At MWS's request, Disposer shall promptly remove any Non-conforming Waste delivered by Disposer to the Facility, transporting and disposing of it at a permitted disposal facility. Disposer acknowledges that delivery of Non-conforming Waste could have a material adverse effect on MWS and the operation of the Facility and be grounds for barring of any future deliveries, termination of this Agreement and recovery of damages.

(f) Each party shall perform its obligations and duties hereunder in compliance with all applicable federal, state and local licenses, permits and approvals and all federal, state and local laws, ordinances, requirements, orders, directives, rules and regulations including without limitation those established by OSHA, and all material agreements relating to any of the foregoing to which it is a party or by which its assets are affected.

3. Term/Renewal.

(a) The initial term of this Agreement shall be twelve (12) months, commencing on the "Effective Date" of this agreement.

(b) Upon the expiration of the initial term, this Agreement shall renew automatically for a like term of one (1) year, and thereafter for additional successive one (1) year terms, unless terminated by either party giving written notice to the other party no less than sixty (60) days in advance of the expiration of the then current term.

4. Exclusivity. Disposer agrees to use MWS exclusively for treatment and disposal of biohazardous waste collected in California throughout the term of this agreement, and any extensions.

5. Termination. Either party may terminate this Agreement "for cause" by giving the other party written notice, by U.S. certified mail, return receipt requested. Such notice shall state, with specificity, the cause for the termination and give the other party a period of thirty (30) days from the date of such notice in which to satisfactorily cure the cause (the "Cure Period"). In the absence of such a cure to the satisfaction of the notifying party, termination will occur at the expiration of the Cure Period.

6. Rates/Invoicing.

(a) Disposer shall pay MWS for the services at the rates set forth in Exhibit A, attached hereto and incorporated herein (the "Service Fees"). The Service Fees shall be inclusive of all costs incurred by MWS, including the costs of transportation and disposal, except as otherwise provided on Exhibit A.

(b) MWS reserves the right to require Disposer to submit a security deposit to ensure that Disposer faithfully performs its obligations under this Agreement (the "Security Deposit"). The amount of the Security Deposit shall be determined by MWS, in its reasonable judgment, but shall be based on Disposer's actual or estimated monthly billing amount, past payment history, if any, and general creditworthiness. Upon the expiration or termination of this Agreement, if Disposer has fully performed all its obligations, MWS shall refund the Security Deposit to disposer. If Disposer has failed to perform or comply with any of its obligations under this Agreement, MWS may deduct any damages incurred by MWS as a result thereof or any amounts owed by Disposer in connection therewith from the Security Deposit. In lieu of the Security Deposit, Disposer may provide MWS with a performance bond.

(c) Weekly, MWS shall send an invoice to the Disposer for all Service Fees due and owing for the preceding week. Each invoice shall be accompanied by appropriate documentation verifying the Service Fees included on such an invoice. Disposer agrees to pay all invoices on or before the thirtieth (30th) day following the date of receipt of such invoice. MWS reserves the right to impose a service charge of 1.5% per month (18% annual rate) on accounts 30 days past due. In addition, MWS shall be entitled to recover reasonable attorney's fees and court costs if suit is necessary to recover past due amounts.

(d) Because fuel, transportation and landfill (or other) disposal costs constitute the major portion of the cost of the Services provided hereunder by MWS, Disposer agrees that MWS may increase the Service Fees proportionally to adjust for any increase in such costs. Disposer further agrees that MWS may increase the Service Fees (i) from time to time to proportionally pass through to Disposer any increases in taxes, fees or other governmental charges assessed against the Facility or the acceptance and treatment of Regulated medical waste and Regulated/APHIS waste at the Facility and (ii) from time to time, but no more often than annually, to adjust for increases in operating costs.

7. Independent Contractor. This Agreement reflects an arms-length transaction, creates no fiduciary, partnership, joint venture or employment or other agency relationship, and is not entered into for the benefit of (nor are any rights granted to) any third party except as specifically provided herein. The services contemplated by this Agreement shall be performed and furnished by MWS as an independent contractor under the sole supervision, management, direction, and control of MWS in accordance with the terms and conditions of this Agreement. All Services will be performed in a good and efficient manner.

8. Insurance. Each party shall maintain (i) workers compensation insurance as required by law; (ii) employer's liability insurance having a minimum limit of \$1,000,000 per occurrence; (iii) commercial general liability insurance (Broad Form) including employment, personal injury and blanket contractual liability coverage with a minimum combined single limit of \$1,000,000 per occurrence; (iv) comprehensive automobile liability insurance applicable to all owned, non-owned and hired vehicles with a minimum combined single limit of \$1,000,000 per occurrence; (v) pollution liability insurance having a minimum limit of \$2,000,000 per occurrence and (vi) excess of (ii), (iii) and (iv) above, umbrella liability insurance with a minimum limit of \$1,000,000 per occurrence. Each party will provide the other party, upon request, a certificate of insurance, evidencing such insurance.

9. Confidentiality. Each party recognizes and acknowledges that it will have access to confidential information of the other party of a special and unique value which may include, without limitation, the composition, volume and generators of the Regulated medical waste and/or Regulated/APHIS waste, the operation of the Facility, service requirements, costs of providing service and equipment, customer identity and vendor information and similar matters. Each party therefore agrees that it will not at any time, either during or after the term of this Agreement, make any independent use of, or disclose to any other person or organization, other than their respective employees or subcontractors, and then only as required in connection with the performance of this Agreement, any confidential information of the other party, except as specifically authorized by the other party in writing. In the event of a breach or threatened breach by either party of the provisions of this Agreement, the other party shall be entitled to an injunction restraining the disclosure, in whole or in part, of such confidential information. Nothing herein shall be construed as prohibiting either party from pursuing any other remedies available to it for such breach or threatened breach, including the recovery of damages from the breaching party.

10. Books and Records. Each party shall maintain and retain adequate books, records and other documentation relating to the services to be performed by it under this Agreement (including without limitation, copies of all weight records, manifests and tracking records) and any other records, reports, information or documentation reasonably

necessary to verify such party's compliance with its obligations hereunder ("Records"). Each party shall retain such Records consistent with and for the periods required by applicable regulatory requirements and guidelines. Each party shall have the right to inspect and audit, at any reasonable time and at its own expense, the other party's Records.

11. Indemnification.

(a) MWS agrees to defend, indemnify and hold Disposer harmless from any and all claims, loss, damages, suits, judgment, amount in contribution or settlement, penalties, costs, liabilities and expenses whatsoever, including attorneys' fees (including, without limitation, reasonable attorneys fees) (collectively "Damages") caused by or resulting from MWS's or its employees', agents', subcontractors', officers' or directors' acts or omissions in the performance of its obligations under this Agreement or by MWS's breach of this Agreement.

(b) Disposer agrees to defend, indemnify and hold MWS and its subcontractors harmless from any and all Damages caused by or resulting from Disposer's or its employees', agents', other contractors', officers' or directors' acts or omissions in the performance of its obligations under this Agreement, or by Disposer's breach of this Agreement, including, without limitation, by the delivery of Non-conforming Waste to the Facility.

12. Force Majeure. Except for the obligation to pay for services rendered, neither party hereto shall be liable for its failure to perform hereunder due to contingencies beyond its reasonable control, including, but not limited to, strikes, riots, war, fire, acts of God, compliance with any law, regulation or order, whether valid or invalid, of the United States of America or any other governmental body or any instrumentality thereof, whether now existing or hereafter created.

13. Miscellaneous.

(a) Counterparts. This Agreement may be executed in several counterparts, each of which shall be an original but all of which together shall constitute one and the same instrument. Facsimile transmission of executed signature pages of this Agreement shall be deemed as execution and delivery of this Agreement.

(b) Amendment and Waiver. This Agreement shall not be modified or amended except by a writing executed by the party against whom the modified or amended term or provision is sought to be enforced. Neither a failure by either party hereto to exercise any of its options hereunder, nor failure to enforce its rights or seek its remedies upon any default shall effect or constitute a waiver of the respective party's right to enforce such right, or to seek remedy with respect to that default or to any prior or subsequent default. The remedies provided in this Agreement shall be cumulative and shall not in any way abridge, modify, or preclude any other rights or remedies to which either party may be entitled either at law or in equity.

(c) Dispute Resolution. The parties agree to the following mechanism to obtain prompt and expeditious resolution of all disputes under or related to this Disposal Agreement:

(a) any dispute seeking damages, possession and/or any dispute seeking specific enforcement of any provision herein shall be heard and determined by a Referee pursuant to California Civil Procedure Section 638 in effect as of the date hereof. The venue of any proceeding hereunder shall be in Orange County, California (unless changed by order of the Referee).

(i) The party seeking to resolve the dispute shall serve a complaint or statement of claim on the other party, describing the matters in dispute in the manner prescribed for giving of notice hereunder. Within five (5) business days after the service of the complaint or statement of claim, the party seeking relief shall make a written request for the specific designation of a Referee to try the dispute. The Referee shall be chosen from the Orange County panel of retired jurists operating as **Judicial Arbitration and Mediation Service** ("JAMS"). Thereafter the parties shall use their best efforts to agree upon the selection of a Referee. If the parties are unable to agree upon a Referee within ten (10) business days after a written request to do so by any party, then either party may petition the Presiding Judge of the Orange County Superior Court to appoint a Referee from the panel maintained in Orange County by said JAMS.

(ii) The provisions of California Code of Civil Procedure Section 640, 642, 643, 644, and 645 shall be applicable to dispute resolution by a Referee hereunder. In an effort to clarify and amplify the provisions of California Code of Civil Procedure Sections 644 and 645, the parties agree that the Referee shall decide the dispute submitted by the parties for decision in the same manner as required for a trial by court as set forth in California Code of Civil Procedure Section 631.8 and 632, and Rule 3.1590 as the California Rules of Court. The Referee shall try and decide the dispute according to and based on all the substantive and procedural

statutory and decisional law of the State of California, unless the parties stipulate to the contrary. When the Referee has decided on the dispute, the Referee shall also cause the preparation of a judgment based on said decision. The Judgment (or other order) to be entered by the Superior Court, based upon the decision of the Referee, shall be appealable in the same manner as if the Judge signing the Judgment (or other order) had tried the case.

(b) The parties shall diligently cooperate with one another and the person appointed to resolve the dispute, and shall perform such acts as may be necessary to obtain a prompt and expeditious resolution of the dispute. If any party refuses to diligently cooperate, and any other party, after first giving notice of its intent to rely on the provisions of this Section 12, incurs additional expenses or attorneys' fees solely as a result of such failure to diligently cooperate, the Referee may award such additional expenses and attorneys' fees to the party giving such notice, even if such party is not the prevailing party in the dispute.

(c) The cost of the proceeding shall initially be borne equally by the parties to the dispute, but the prevailing party in such proceeding shall be entitled to recover, in addition to reasonable attorneys' fees and all other costs, its contribution for the cost of the Referee as an item of recoverable costs. If either party refuses to pay his share of the costs of the proceeding, at the time(s) required, the other party may do so, in which event that party will be entitled to recover (or offset) the amount advanced, with interest, even if that party is not the prevailing party. The Referee shall include such costs in his judgment or order.

(d) Entire Agreement; Assignment; Binding Effect. This Agreement constitutes the entire agreement and understanding between the parties hereto with respect to the subject matter hereof and supersedes any prior agreement, representation or understanding with respect thereto. Neither party may assign this Agreement in whole or in part without the prior, express written consent of the other party; provided, however, either party may assign this Agreement to any entity which is owned or controlled by or under common control with that party. Any permitted assignment shall not release any party from any of its obligations contained in this Agreement. This Agreement is and shall be binding upon and inure to the benefit of the parties hereto and their respective legal representatives, successors, and permitted assigns.

(e) Savings Clause. If any one or more of the provisions contained in this Agreement shall, for any reason, be held to be invalid, illegal or unenforceable in any respect, such invalidity, illegality or unenforceability shall not affect any other provisions of this Agreement but this Agreement shall be construed as if such invalid, illegal or unenforceable provision had never been contained herein.

IN WITNESS WHEREOF, each of the parties hereto has caused this Agreement to be executed and delivered by the duly authorized officers of each such party as of the date first above written.

Medical Waste Services, LLC:

By: _____



By: _____

Erik T. Nelson
VP of Operations

1/30/2025

Fortunato Villamagna
Managing Member

1/30/2025

Exhibit A Service Fees

All waste containers brought to the MWS facility are limited to a maximum gross weight of seventy-five (75) pounds each. Containers exceeding the 75-pound weight limit may be subject to rejection, unless adequate arrangements have been agreed to between MWS and Disposer. Waste packaged in gaylord boxes will be the exception to these weight standards.

	<u>Rate Per Container</u>	<u>Rate Per Pound</u>
Biohazardous Waste Treated via Autoclave:	NA	.29

	<u>Rate Per Container</u>	<u>Rate Per Pound</u>
Chemotherapy Waste, Pathology Waste, Non-RCRA Pharmaceutical Waste Treated Via Incineration:	NA	.75

	<u>Rate Per Container</u>	<u>Rate Per Pound</u>
Regulated/APHIS Waste, Treated via Autoclave:	NA	.48

	<u>Rate Per Container</u>	<u>Rate Per Pound</u>
Other Waste (specify type/treatment)	NA	.0127
State Fee _____		

Minimum Transaction Charge:	\$5000 per transaction.
Labor/materials:	\$250 per truck/trailer load
Storage Fees:	\$10 per pallet

Pricing acknowledged by MWS (initials): EN

Pricing acknowledged by Disposer (initials): _____

APPENDIX B. FINANCIAL ASSURANCE FOR CLOSURE

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APPENDIX F. WASTE ACCEPTANCE POLICY

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REGULATED MEDICAL WASTE ACCEPTANCE POLICY

MediWaste Disposal, LLC, a California limited liability company ("MediWaste"), policy requires compliance with all applicable regulations regarding the collection, transportation and treatment of regulated medical waste. Federal Department of Transportation (DOT) Regulations require the generator of regulated medical waste to certify that the packaging and documentation of transported regulated medical waste complies with DOT regulations regarding waste classification, packaging, labeling and shipping documentation. To ensure that neither MediWaste nor the generator of regulated medical waste violates applicable regulations, it is imperative that all parties understand the rules regarding proper identification, classification, segregation and packaging of regulated medical waste. The purpose of this policy is to summarize the minimum requirements for preparing your medical waste collection, transportation and treatment. Additional facility or state-specific waste acceptance policies may apply based on permit specifications. Please contact your local representative for further information.

REGULATED MEDICAL WASTE

MediWaste accepts medical waste generated in a broad range of medical, diagnostic, therapeutic and research activities. The term "medical waste" includes biohazardous, biomedical, medical, infectious or regulated medical, pharmaceuticals and related products, as well as crime scene wastes, law enforcement confiscated waste, and APHS waste as defined by the FDA and federal, state or local laws, rules, regulations and guidelines. Except as defined by specific state regulations, this excludes Resource Conservation and Recovery Act (RCRA) waste, including pharmaceutical waste managed under 40 CFR 266 Subpart P in other states. In addition, MediWaste cannot accept bulk liquids, radioactive materials, or complete human remains (including heads, full torsos and family planning related products). MediWaste cannot accept these excluded materials packaged as regulated medical waste. All lab wastes or materials which contain or have the potential to contain infectious substances arising from those agents listed under 42 CFR 72.3 are strictly prohibited from medical waste by federal law and must be pretreated prior to disposal. Separate protocol and packaging requirements apply for the disposal of non-hazardous pharmaceuticals. Hazardous waste transportation services may be offered in certain geographical locations, under separate contract. Please contact your local representative for details and packaging specifications.

WASTE SEGREGATION AND PACKAGING

The generator is solely responsible for properly segregating packaging and labeling of regulated medical waste. Proper segregation and packaging reduce the potential for accidental release of the contents and exposure to employees and the general public. DOT regulations require (49 CFR 173.197) that all packages of regulated medical waste be prepared to transport in containers meeting the following requirements: (1) rigid; (2) leak resistant; (3) impervious to moisture; (4) of sufficient strength to prevent tearing or bursting under normal conditions of use and handling; (5) sealed to prevent leakage during transport, and (6) puncture resistant for sharps. All regulated medical waste must be accompanied by a properly completed shipping document (See 49 CFR 172.202).

MANAGEMENT OF NON-CONFORMING WASTE

As required by regulations and company policy, MediWaste employees may refuse containers that are non-conforming because of their contents or are improperly packaged, leaking, damaged or likely to create a risk of exposure to employees or the general public. Any non-conforming waste identified en route to or at a MediWaste location may be returned to the generator for proper packaging or disposal. Proper segregation and packaging are essential to ensure compliant and safe handling, collection, transportation and treatment of regulated medical waste.

MEDIWASTE REGULATED MEDICAL WASTE ACCEPTANCE POLICY CHECKLIST

ACCEPTED REGULATED MEDICAL WASTE

- Sharps – Means any object contaminated with a pathogen or that may become contaminated with a pathogen through handling or during transportation and also capable of cutting or penetrating skin or packaging material. Sharps includes needles, syringes, scalpels, broken glass, culture slides, culture dishes, broken capillary tubes, broken rigid plastic and exposed ends of dental wires.
- Regulated Medical Waste or Clinical Waste or (Bio) Medical Waste – Means a waste or reusable material derived from the medical treatment of an animal or human, which includes diagnosis and immunization or from biomedical research, which includes the production and testing of biological products.

ACCEPTED REGULATED MEDICAL WASTE WHICH MUST BE IDENTIFIED AND SEGREGATED FOR INCINERATION

- Trace Chemotherapy Contaminated Waste – RCRA empty drug vials, syringes and needles, split kits, IV tubing and bags, contaminated gloves and gowns, and regulated materials as defined in applicable laws, rules, regulations or guidelines.
- Pathological Waste – Human or animal body parts, organs, tissues and surgical specimen (decanted of formaldehyde, formalin or other preservatives, as required per hazardous waste rules).
- Non-RCRA Pharmaceuticals – Must be characterized and certified as non-RCRA hazardous materials by the generator. This does not include pharmaceutical waste managed under 40 CFR 266 Subpart P in other states. Excludes all DEA scheduled drugs, including controlled substances.
- California Only – Solidified Canisters – Suction canisters that have been injected with solidifier materials to control liquids or suction canisters made of high heat resistant plastics such as polysulfide.

REGULATED MEDICAL WASTE NOT ACCEPTED BY MEDIWASTE

- Untreated Category A Infectious Substances. Putrescible wastes are not accepted.
 - RCRA Hazardous Pharmaceutical Waste and all DEA controlled drugs, including controlled substances. RCRA Subpart P Pharmaceuticals are not accepted.
 - Radioactive wastes are not accepted at any level of radiation (3xs the local background level).
 - Chemicals – Formaldehyde, formalin, acids, alcohol, waste solvents reagents, fixer developer.
 - Hazardous Waste – Drums or other containers with a hazard warning symbol, batteries and other heavy metals.
 - Radioactive Waste – Any container with a radioactive level that exceeds regulatory or permitted limits; lead-containing materials.
 - Complete Human Remains (including heads, full torsos and fetuses).
 - Bulk Chemotherapy Waste.
 - Compressed Glass Cylinders, Canister, Inhalers and Aerosol Cans.
 - Any Mercury-Containing Material or Devices – Any mercury thermometers, Sphygmomanometers, lab or medical devices.
 - Mercury-Containing Dental Waste – Non-contact and contact amalgam and products, chairside, amalgam sludge or vacuum pump filters, extracted teeth with mercury fillings and empty amalgam capsules.
- Additional waste acceptance policies may apply based on state or permit specific requirements. Hazardous waste transportation services may be offered in certain geographic locations, under separate contract. Please refer to your local MediWaste Representative for additional information and options for possible hazardous waste handling or container and labeling requirements.

* Consult a MediWaste Representative for specific requirements

Additional waste acceptance policies may apply based on state or permit specific requirements. Hazardous waste transportation services may be offered in certain geographic locations, under separate contract. Please refer to your local MediWaste Representative for additional information and options for possible hazardous waste handling or container and labeling requirements.

Under California's Medical Waste Management Act (MWMA), the responsibility for the proper packaging of medical waste lies with the medical waste generator. This includes ensuring that waste is correctly contained, labeled, and prepared for storage, transport, and disposal in compliance with state and federal regulations.(cdph.ca.gov)

Key Legal References

1. Definition of Medical Waste Generator

According to California Health and Safety Code §117705, a "medical waste generator" is defined as:(cdph.ca.gov)

“Any person whose act or process produces medical waste.”(cdph.ca.gov)

This definition encompasses healthcare providers, laboratories, clinics, and other facilities that produce medical waste.

2. Packaging and Handling Requirements

While the MWMA outlines general responsibilities, specific packaging standards are influenced by federal regulations. The MWMA acknowledges that:

“California statute provides for slightly more stringent standards in packaging than federal regulations, as California law governs the accumulation, storage, and treatment of waste, rather than just its transport.” (cdph.ca.gov)

This means that medical waste generators must adhere to both state and federal requirements, ensuring that waste is packaged securely to prevent leaks, spills, or exposure.

3. Transportation Regulations

For the transportation of medical waste, the MWMA references federal standards:

“Sections 173.196 and 173.197 of Title 49 of the Code of Federal Regulations impose standards for the transportation of medical waste on public roads and highways while in transport.” (cdph.ca.gov)

Medical waste generators must ensure that packaged waste complies with these transportation standards when being moved off-site.

4. Training and Compliance

Generators are also responsible for training staff in proper waste handling procedures. The MWMA requires that:(cdph.ca.gov)

“Training shall comply with applicable federal Occupational Safety and Health Administration regulations, including those found in Section 1910 of Title 29 of the Code of Federal Regulations.” (cdph.ca.gov)

This ensures that all personnel involved in the handling of medical waste are knowledgeable about safety protocols and regulatory compliance.

Summary

In summary, under California law, medical waste generators are responsible for: (cdph.ca.gov)

- Properly identifying and segregating medical waste.
- Packaging waste in accordance with state and federal regulations.
- Ensuring secure and compliant transportation of waste.
- Training staff on safe handling and compliance requirements. (cdph.ca.gov)

For a comprehensive understanding of these responsibilities, refer to the Medical Waste Management Act provided by the California Department of Public Health. (cdph.ca.gov)

APPENDIX G. REGULATED MEDICAL WASTE DEFINITIONS

DRAFT

This content is from the eCFR and is authoritative but unofficial.

Title 49 —Transportation

Subtitle B —Other Regulations Relating to Transportation

Chapter I —Pipeline and Hazardous Materials Safety Administration, Department of Transportation

Subchapter C —Hazardous Materials Regulations

Part 173 —Shippers—General Requirements for Shipments and Packagings

Subpart D —Definitions Classification, Packing Group Assignments and Exceptions for Hazardous Materials Other Than Class 1 and Class 7

Source: Amdt. 173-224, 55 FR 52634 Dec. 21, 1990, unless otherwise noted.

Authority: 49 U.S.C. 5101-5128, 44701; 49 CFR 1.81, 1.96 and 1.97.

Editorial Note: Nomenclature changes to part 173 appear at 70 FR 56098, Sept. 23, 2005.

§ 173.134 Class 6, Division 6.2—Definitions and exceptions.

- (a) *Definitions and classification criteria.* For the purposes of this subchapter, the following definitions and classification criteria apply to Division 6.2 materials.
- (1) *Division 6.2 (Infectious substance)* means a material known or reasonably expected to contain a pathogen. A pathogen is a microorganism (including bacteria, viruses, parasites, and fungi) or other agent, such as a proteinaceous infectious particle (prion) that can cause disease in humans or animals. An infectious substance must be assigned the identification number UN2814, UN2900, UN3291, UN3373, or UN3549 as appropriate, and must be assigned to one of the following categories:
- (i) *Category A:* An infectious substance in a form capable of causing permanent disability or life-threatening or fatal disease in otherwise healthy humans or animals when exposure to it occurs. An exposure occurs when an infectious substance is released outside of its protective packaging, resulting in physical contact with humans or animals. A Category A infectious substance must be assigned to identification number UN2814, UN2900, or UN3549, as appropriate. Assignment to UN2814, UN2900, or UN3549 must be based on the known medical history or symptoms of the source patient or animal, endemic local conditions, or professional judgment concerning the individual circumstances of the source human or animal.
- (ii) *Category B:* An infectious substance that is not in a form generally capable of causing permanent disability or life-threatening or fatal disease in otherwise healthy humans or animals when exposure to it occurs. This includes Category B infectious substances transported for diagnostic or investigational purposes. A Category B infectious substance must be described as "Biological substance, Category B" and assigned identification number UN3373. This does not include regulated medical waste, which must be assigned identification number UN3291.
- (2) *Biological product* means a virus, therapeutic serum, toxin, antitoxin, vaccine, blood, blood component or derivative, allergenic product, or analogous product, or arsphenamine or derivative of arsphenamine (or any other trivalent arsenic compound) applicable to the prevention, treatment, or cure of a disease or condition of human beings or animals. A *biological product* includes a material subject to regulation under 42 U.S.C. 262 or 21 U.S.C. 151-159. Unless otherwise excepted, a

biological product known or reasonably expected to contain a pathogen that meets the definition of a Category A or B infectious substance must be assigned the identification number UN 2814, UN 2900, or UN 3373, as appropriate.

- (3) **Culture** means an infectious substance containing a pathogen that is intentionally propagated. *Culture* does not include a human or animal patient specimen as defined in paragraph (a)(4) of this section.
 - (4) **Patient specimens** means those collected directly from humans or animals and transported for research, diagnosis, investigational activities, or disease treatment or prevention. *Patient specimens* includes excreta, secretions, blood and its components, tissue and tissue swabs, body parts, and specimens in transport media (e.g., transwabs, culture media, and blood culture bottles).
 - (5) **Regulated medical waste or clinical waste or (bio) medical waste** means a waste or reusable material derived from the medical treatment of an animal or human, which includes diagnosis and immunization, or from biomedical research, which includes the production and testing of biological products. Regulated medical waste or clinical waste or (bio) medical waste containing a Category A infectious substance must be classed as an infectious substance, and assigned to UN2814, UN2900, or UN3549, as appropriate.
 - (6) **Sharps** means any object contaminated with a pathogen or that may become contaminated with a pathogen through handling or during transportation and also capable of cutting or penetrating skin or a packaging material. *Sharps* includes needles, syringes, scalpels, broken glass, culture slides, culture dishes, broken capillary tubes, broken rigid plastic, and exposed ends of dental wires.
 - (7) **Toxin** means a Division 6.1 material from a plant, animal, or bacterial source. A *toxin* containing an infectious substance or a *toxin* contained in an infectious substance must be classed as Division 6.2, described as an infectious substance, and assigned to UN 2814 or UN 2900, as appropriate.
 - (8) **Used health care product** means a medical, diagnostic, or research device or piece of equipment, or a personal care product used by consumers, medical professionals, or pharmaceutical providers that does not meet the definition of a patient specimen, biological product, or regulated medical waste, is contaminated with potentially infectious body fluids or materials, and is not decontaminated or disinfected to remove or mitigate the infectious hazard prior to transportation.
- (b) **Exceptions.** The following are not subject to the requirements of this subchapter as Division 6.2 materials:
- (1) A material that does not contain an infectious substance or that is unlikely to cause disease in humans or animals.
 - (2) Non-infectious biological materials from humans, animals, or plants. Examples include non-infectious cells, tissue cultures, blood or plasma from individuals not suspected of having an infectious disease, DNA, RNA or other non-infectious genetic elements.
 - (3) A material containing micro-organisms that are non-pathogenic to humans or animals.
 - (4) A material containing pathogens that have been neutralized or inactivated such that they no longer pose a health risk.
 - (5) A material with a low probability of containing an infectious substance, or where the concentration of the infectious substance is at a level naturally occurring in the environment so it cannot cause disease when exposure to it occurs. Examples of these materials include: Foodstuffs; environmental samples, such as water or a sample of dust or mold; and substances that have been treated so that

the pathogens have been neutralized or deactivated, such as a material treated by steam sterilization, chemical disinfection, or other appropriate method, so it no longer meets the definition of an infectious substance.

- (6) A biological product, including an experimental or investigational product or component of a product, subject to Federal approval, permit, review, or licensing requirements, such as those required by the Food and Drug Administration of the U.S. Department of Health and Human Services or the U.S. Department of Agriculture.
- (7) Blood collected for the purpose of blood transfusion or the preparation of blood products; blood products; plasma; plasma derivatives; blood components; tissues or organs intended for use in transplant operations; and human cell, tissues, and cellular and tissue-based products regulated under authority of the Public Health Service Act (42 U.S.C. 264-272) and/or the Food, Drug, and Cosmetic Act (21 U.S.C. 301 et seq.).
- (8) Blood, blood plasma, and blood components collected for the purpose of blood transfusion or the preparation of blood products and sent for testing as part of the collection process, except where the person collecting the blood has reason to believe it contains an infectious substance, in which case the test sample must be shipped as a Category A or Category B infectious substance in accordance with § 173.196 or § 173.199, as appropriate.
- (9) Dried blood spots or specimens for fecal occult blood detection placed on absorbent filter paper or other material.
- (10) A Division 6.2 material, other than a Category A infectious substance, contained in a patient sample being transported for research, diagnosis, investigational activities, or disease treatment or prevention, or a biological product, when such materials are transported by a private or contract carrier in a motor vehicle used exclusively to transport such materials. Medical or clinical equipment and laboratory products may be transported aboard the same vehicle provided they are properly packaged and secured against exposure or contamination. If the human or animal sample or biological product meets the definition of regulated medical waste in paragraph (a)(5) of this section, it must be offered for transportation and transported in conformance with the appropriate requirements for regulated medical waste.
- (11) A human or animal sample (including, but not limited to, secret, excreta, blood and its components, tissue and tissue fluids, and body parts) being transported for routine testing not related to the diagnosis of an infectious disease, such as for drug/alcohol testing, cholesterol testing, blood glucose level testing, prostate specific antibody testing, testing to monitor kidney or liver function, or pregnancy testing, or for tests for diagnosis of non-infectious diseases, such as cancer biopsies, and for which there is a low probability the sample is infectious.
- (12) Laundry and medical equipment and used health care products, as follows:
 - (i) Laundry or medical equipment conforming to the regulations of the Occupational Safety and Health Administration of the Department of Labor in 29 CFR 1910.1030. This exception includes medical equipment intended for use, cleaning, or refurbishment, such as reusable surgical equipment, or equipment used for testing where the components within which the equipment is contained essentially function as packaging. This exception does not apply to medical equipment being transported for disposal.

- (ii) Used health care products not conforming to the requirements in 29 CFR 1910.1030 and being returned to the manufacturer or the manufacturer's designee are excepted from the requirements of this subchapter when offered for transportation or transported in accordance with this paragraph (b)(12). For purposes of this paragraph, a health care product is used when it has been removed from its original packaging. Used health care products contaminated with or suspected of contamination with a Category A infectious substance may not be transported under the provisions of this paragraph.
 - (A) Each used health care product must be drained of free liquid to the extent practicable and placed in a watertight primary container designed and constructed to assure that it remains intact under conditions normally incident to transportation. For a used health care product capable of cutting or penetrating skin or packaging material, the primary container must be capable of retaining the product without puncture of the packaging under normal conditions of transport. Each primary container must be marked with a BIOHAZARD marking conforming to 29 CFR 1910.1030(g)(1)(i).
 - (B) Each primary container must be placed inside a watertight secondary container designed and constructed to assure that it remains intact under conditions normally incident to transportation. The secondary container must be marked with a BIOHAZARD marking conforming to 29 CFR 1910.1030(g)(1)(i).
 - (C) The secondary container must be placed inside an outer packaging with sufficient cushioning material to prevent shifting between the secondary container and the outer packaging. An itemized list of the contents of the primary container and information concerning possible contamination with a Division 6.2 material, including its possible location on the product, must be placed between the secondary container and the outside packaging.
 - (D) Each person who offers or transports a used health care product under the provisions of this paragraph must know about the requirements of this paragraph.
- (13) Any waste or recyclable material, other than regulated medical waste, including—
 - (i) Household waste as defined in § 171.8, when transported in accordance with applicable state, local, or tribal requirements.
 - (ii) Sanitary waste or sewage;
 - (iii) Sewage sludge or compost;
 - (iv) Animal waste generated in animal husbandry or food production; or
 - (v) Medical waste generated from households and transported in accordance with applicable state, local, or tribal requirements.
- (14) Corpses, remains, and anatomical parts intended for interment, cremation, or medical research at a college, hospital, or laboratory.
- (15) Forensic material transported on behalf of a U.S. Government, state, local or Indian tribal government agency, except that—
 - (i) Forensic material known or suspected to contain a Category B infectious substance must be shipped in a packaging conforming to the provisions of § 173.24.

- (ii) Forensic material known or suspected to contain a Category A infectious substance or an infectious substance listed as a select agent in 42 CFR part 73 must be transported in packaging capable of meeting the test standards in § 178.609 of this subchapter. The secondary packaging must be marked with a BIOHAZARD symbol conforming to specifications in 29 CFR 1910.1030(g)(1)(i). An itemized list of contents must be enclosed between the secondary packaging and the outer packaging.
- (16) A raw agricultural commodity as defined in the Federal Food, Drug, and Cosmetics Act (21 U.S.C. 301 et seq.).
- (c) **Exceptions for regulated medical waste.** The following provisions apply to the transportation of regulated medical waste:
 - (1) A regulated medical waste transported by a private or contract carrier is excepted from—
 - (i) The requirement for an "INFECTIOUS SUBSTANCE" label if the outer packaging is marked with a "BIOHAZARD" marking in accordance with 29 CFR 1910.1030; and
 - (ii) The specific packaging requirements of § 173.197, if packaged in a rigid non-bulk packaging conforming to the general packaging requirements of §§ 173.24 and 173.24a and packaging requirements specified in 29 CFR 1910.1030, provided the material does not include a waste concentrated stock culture of an infectious substance. Sharps containers must be securely closed to prevent leaks or punctures.
 - (2) The following materials may be offered for transportation and transported as a regulated medical waste when packaged in a rigid non-bulk packaging conforming to the general packaging requirements of §§ 173.24 and 173.24a and packaging requirements specified in 29 CFR 1910.1030 and transported by a private or contract carrier in a vehicle used exclusively to transport regulated medical waste:
 - (i) Waste stock or culture of a Category B infectious substance;
 - (ii) Plant and animal waste regulated by the Animal and Plant Health Inspection Service (APHIS);
 - (iii) Waste pharmaceutical materials;
 - (iv) Laboratory and recyclable wastes;
 - (v) Infectious substances that have been treated to eliminate or neutralize pathogens;
 - (vi) Forensic materials being transported for final destruction;
 - (vii) Rejected or recalled health care products;
 - (viii) Documents intended for destruction in accordance with the Health Insurance Portability and Accountability Act of 1996 (HIPAA) requirements;
 - (ix) Medical or clinical equipment and laboratory products provided they are properly packaged and secured against exposure or contamination; or
 - (x) Sharps in sharp containers provided the containers are securely closed to prevent leaks or punctures; do not exceed 18 gallons capacity; registered under the Medical Device Regulations of FDA; made of puncture resistant plastic that meets ASTM Standard F2132-01, Standard Specification for Puncture Resistance of Materials Used in Containers for Discarded Medical Needles and Other Sharps; and are securely fitted into wheeled racks that hold them in an

upright position. The wheeled racks must contain full rows of sharps containers secured in place by a moveable bar; and must be securely held in place on the motor vehicle by straps or load bars during transportation. No shelf in any wheeled rack may exceed the manufacturer's recommended load capacity.

- (d) If an item listed in paragraph (b) or (c) of this section meets the definition of another hazard class or if it is a hazardous substance, hazardous waste, or marine pollutant, it must be offered for transportation and transported in accordance with applicable requirements of this subchapter.
- (e) **Transitional provisions.** The authorization for continued use of the criteria for packing group assignments in effect on December 31, 2006 ended on January 1, 2012.

[67 FR 53138, Aug. 14, 2002, as amended at 68 FR 57632, Oct. 6, 2003; 70 FR 56098, Sept. 23, 2005; 71 FR 32258, June 2, 2006; 71 FR 78631, Dec. 29, 2006; 72 FR 55692, Oct. 1, 2007; 73 FR 4718, Jan. 28, 2008; 74 FR 2257, Jan. 14, 2009; 76 FR 43530, July 20, 2011; 77 FR 60942, Oct. 5, 2012; 78 FR 15327, Mar. 11, 2013; 85 FR 27880, May 11, 2020; 85 FR 83397, Dec. 21, 2020; 87 FR 44992, July 26, 2022]

49 CFR Ch. 1 (10-1-93 Edition)
Pt. 173, App. G

APPENDIX G TO PART 173—DEFINITION OF
REGULATED MEDICAL WASTE

1. GENERAL

A. *Regulated medical waste* is a waste material listed in the following table that is—

- (a) Generated in the diagnosis, treatment (e.g., provision of medical services), or immunization of human beings or animals;
- (b) In research pertaining thereto; or
- (c) In the production or testing of biologicals.

TABLE—REGULATED MEDICAL WASTE

Waste class	Description
(1) Cultures and Stocks.	Cultures and stocks of infectious agents and associated biologicals, including: cultures from medical and pathological laboratories; cultures and stocks of infectious agents from research and industrial laboratories; wastes from the production of biologicals; discarded live and attenuated vaccines; and culture dishes and devices used to transfer, inoculate, and mix cultures.
(2) Pathological Wastes.	Human pathological wastes, including tissues, organs, and body parts and body fluids that are removed during surgery or autopsy, or other medical procedures, and specimens of body fluids and their containers. (1) Liquid waste human blood; (2) products of blood; (3) items saturated and/or dripping with human blood; or (4) items that were saturated and/or dripping with human blood that are now caked with dried human blood; including serum, plasma, and other blood components, and their containers, which were used or intended to use in either patient care, testing and laboratory analysis or the development of pharmaceuticals. Intravenous bags are also included in this category.
(3) Human Blood and Blood Products.....	
(4) Sharps.....	

Waste class	Description
(5) Animal Waste	Contaminated animal carcasses, body parts, and bedding of animals that were known to have been exposed to infectious agents during research (including research in veterinary hospitals), production of biologicals, or testing of pharmaceuticals.
(6) Isolation Waste.	Biological waste and discarded materials contaminated with blood, excretion, exudates, or secretions from humans who are isolated to protect others from certain highly communicable disease, or isolated animals known to be infected with highly communicable diseases.
(7) Unused Sharps.	The following unused, discarded sharps: hypodermic needles, suture needles, syringes, and scalpel blades.

NOTE: The term *solid waste* includes solid, semisolid, or liquid materials, but does not include domestic sewage materials identified in the requirements of the Environmental Protection Agency contained in 40 CFR 261.4.

2. EXCEPTIONS

(a) The following wastes are excepted from the requirements for regulated medical waste:

(i) Hazardous waste identified or listed under the regulation in 40 CFR part 261;

(ii) Household waste, as defined in 40 CFR 261.4(b)(1);

(iii) Ash from incineration of regulated medical waste once the incineration process has been completed;

(iv) Residues from treatment and destruction processes of regulated medical waste once the waste has been both treated and destroyed; or

(v) Human corpses, remains, and anatomical parts that are intended for interment or cremation.

(b) Samples of regulated medical waste transported off-site by EPA or State-designated enforcement personnel for enforcement purposes are excepted from the requirements of this subchapter during the enforcement proceeding.

(c) Until October 1, 1992, cultures of etiologic agents of 50 milliliters (1,666 fluid ounces) or less total quantity in one outside package are not subject to any requirements of this subchapter if the items as packaged do not contain any material otherwise subject to the requirements of parts 171 through 180 of this subchapter.

[Amdt. 173-224, 56 FR 66280, Dec., 20 1991]

APPENDIX H. WASTE TRACKING DOCUMENTS

DRAFT

www.medi-waste.com

Manifest # 1612559

Date Oct 8, 2025

GENERATOR	Generator Service Name & Address		Telephone # (888) 728-2264		Customer ID # 2009260001	
	Acme Dialysis 1234 Main St Montebello, CA 90640					
	Description of Waste					
	UN3291 Regulated Medical Waste, n.o.s., 6.2, PGII		Quantity	Weight or Volume (lbs) (gal)	Transfer	
	8 Gal RX	Pharmaceutical Waste	1	8	Yes	
	Generator's Certification: I hereby declare that the contents of this consignment are fully and accurately described above and are classified, packaged, marked and labeled / placarded, and are in all respects in proper condition for transport by the highway according to applicable international and national government regulators including applicable state regulations.					
	Priscilla Lopez				Oct 08, 2025	
	Print/Typed Name		Signature		Date	
TRANSPORTER	Transporter Name and Address		State Permit ID # 6300		Telephone # (855) 449-6334	
	MediWaste Disposal, LLC 235 Deiningir Cir Corona, CA 92880-1707					
	Transporter Certification: I certify, under penalty of criminal and/or civil prosecution for making or submission of false statements, representations or omissions that I have read, understood and will comply with the applicable State and Federal Regulations.					
	Francisco Ayala				Oct 08, 2025	
	Print/Typed Name		Signature		Date	
	Transfer Facility Name and Address		State Permit ID # TS/OST- 136		Telephone # (323) 680-0012	
	MediWaste Disposal 235 Deiningir Cir Corona, CA 92880-1707					
	Arturo Pineda				Oct 8, 2025	
	Print/Typed Name		Signature		Date	
DESTINATION	Destination Facility		State Permit ID #		Alternate Destination Facility	
	MW Nevada DF		XXXXXXXX			
	1850 E. Basin Ave		Telephone #		State Permit ID #	
	Pahrump, NV 89060		855-449-6334		Telephone #	
	Certificate of Destruction		Reference Load Number		Certificate of Destruction	
	Date Received 2025-10-08				Date Received	
	Print/Typed Name		Signature		Print/Typed Name	
					Signature	
	Destruction Certification: The above documented waste was received, treated and disposed of in accordance with all local, State, and Federal Regulations and following all conditions within specified facility's permit, on dates documented.					
	Discrepancy Box					

Emergency Spill Response: Phone: (888) 703-2810

Manifest Consolidation Batch Id	1908	Source	MediWaste Disposal
External Id	1,908	Transporter	MediWaste Disposal, LLC.
Waste From Date	2025-10-07	Destination	MW Nevada DF
Ship Date	2025-10-08	Count	2846

Material Type	Container Count	Net Weight
Pharmaceutical Waste	2846	30057

Date	Site Id	Manifest	Label	Description	Material Type	Weight
2025-10-07	2042690001	1595000	1595000-1604687-001	17 Gal RX	Pharmaceutical Waste	11.00
2025-10-07	2030670001	1595368	1595368-1605800-031	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605800-032	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605800-033	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605800-034	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605800-035	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605800-036	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605795-089	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605795-090	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605795-091	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	53.00
2025-10-07	2030670001	1595368	1595368-1605795-092	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605795-093	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605795-083	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605795-084	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605795-085	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
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2025-10-07	2030670001	1595368	1595368-1605795-087	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605795-088	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2030670001	1595368	1595368-1605795-077	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00

Date	Site Id	Manifest	Label	Description	Material Type	Weight
2025-10-08	2042990001	1610766	1610766-1661876-010	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-08	2042990001	1610766	1610766-1661876-011	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-08	2042990001	1610766	1610766-1661876-012	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	1.00
2025-10-07	2013870001	1611998	1611998-1665931-001	2 Gal RX	Pharmaceutical Waste	18.00
2025-10-08	2018630001	1612001	1612001-1665941-001	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	178.00
2025-10-08	2018630001	1612001	1612001-1665941-002	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	14.00
2025-10-08	2018630001	1612001	1612001-1665941-003	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	13.00
2025-10-08	2018630001	1612001	1612001-1665941-004	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	4.00
2025-10-08	2018630001	1612001	1612001-1665941-005	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	11.00
2025-10-08	2018630001	1612001	1612001-1665941-006	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	13.00
2025-10-08	2018630001	1612001	1612001-1665941-007	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	10.00
2025-10-08	2018630001	1612001	1612001-1665941-008	17 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	6.00
2025-10-08	2018630001	1612001	1612001-1665943-001	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	3.00
2025-10-08	2018630001	1612001	1612001-1665943-002	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	4.00
2025-10-08	2018630001	1612001	1612001-1665943-003	4.5 Gal Reusable (RX-Commingled)	Pharmaceutical Waste	6.00
2025-10-07	206202001	1612426	1612426-1667674-001	17 Gal RX	Pharmaceutical Waste	25.00
2025-10-07	2014130001	1612428	1612428-1667686-001	31 Gal RX	Pharmaceutical Waste	13.00
2025-10-07	2034970001	1612483	1612483-1667880-001	17 Gal RX	Pharmaceutical Waste	6.00
2025-10-08	2009260001	1612559	1612559-1668269-001	8 Gal RX	Pharmaceutical Waste	22.00
Total						30,057.00



**REGULATED MEDICAL WASTE
TRACKING AND SHIPPING DOCUMENT**



Manifest ID # 1908

Date 2025-10-08

GENERATOR

Generator Service Name and Address

MediWaste Disposal
235 Deiningen Cir
Corona, CA 92880-1707

Telephone # (323) 680-0012

Customer ID # 204423001

TS/OST- 136

Description of Waste

UN 3291 Regulated Medical Waste, n.o.s., 6.2, pg II

Quantity Weight or Volume (lbs) (gal) Transfer

Count

Material Type

Net Weight

Gross Weight

2846

Pharmaceutical Waste

30057

31616

All waste treated at Destination Facility unless marked as Alt. Destination above.

Generator's Certification: This is to certify that the above materials are properly classified, described, packaged, marked and labeled, and are in proper condition for transportation according to the applicable regulations of the Department of Transportation.

Print / Typed Name

Signature

Date

TRANSFER / TRANSPORTATION

Transporter Name and Address

MediWaste Disposal, LLC.
235 Deiningen Circle
Corona, CA 92878

State Permit ID # 6300

Telephone # (855) 927-8321

Transporter Certification: I certify, under penalty of criminal and/or civil prosecution for making or submission of false statements, representations, or omissions that I have read, understood, and will comply with the applicable State and Federal Regulations.

Print / Typed Name

Signature

Date

Transfer Facility Name and Address

State Permit ID #

Telephone #

Print / Typed Name

Signature

Date Received

DESTINATION

Destination Facility

MW Nevada DF
1850 E. Basin Ave
Pahrump, NV 89060

State Permit ID #

xxxxxxxxxx

Telephone #

855-449-6334

Alternate Destination Facility

State Permit ID #

Telephone #

Consolidated Tracking ID #

Certificate of Destruction

Date

Certificate of Destruction

Date

Print / Typed Name

Signature

Print / Typed Name

Signature

Discrepancy

Emergency Spill Response:

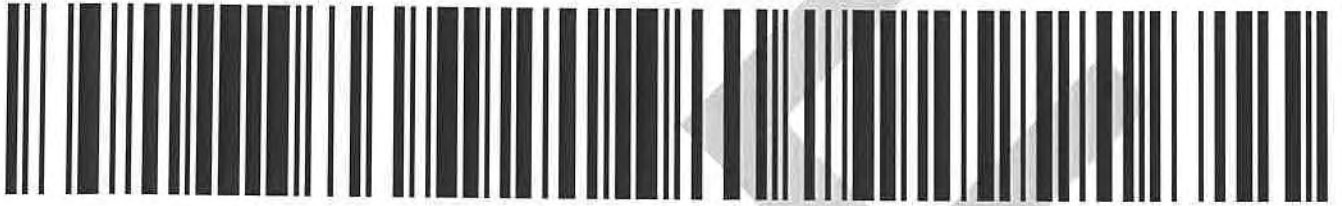
REV. 08212020

NDEP 242

STRAIGHT BILL OF LADING and Medical Waste Tracking Document

Date: 10/08/2025		BOL.No. 1908		LOAD MW-INC-900		Page 1 of 1	
Generator: MediWaste Disposal 235 Deininger Circle Corona, CA 92880 Permit: TS/OST-136 855-449-6334				Treatment Facility: MW Nevada DF 1850 E. Basin Ave Pahrump, NV 89060 Permit:			
		Trailer No.		Received By:		Date:	
Description		Container Type		Pallets	Weight LBS		
UN3291, Regulated Medical Waste, N.O.S., 6.2, PGII		Pharmaceutical Waste Pallets		50	30057		
		Total		50	30057		
Loose Pieces	Pallets said to contain	Cartons	COD Amount		Freight Charges: ☐ Prepaid ☐ Collect		
Bill Freight Charge to:			COD Fee: ☐ Prepaid ☐ Collect				
Mailing Address:			Is Customer Check Acceptable for COD?				
			☐ Yes ☐ No Check No.				
This is to certify that the above named materials are properly classified, described, packaged, marked and labeled, and are in proper condition for transportation according to the applicable regulations of the Department of Transportation.							
Received subject to classifications and lawfully filed tariffs in effect on the date of issue of this Straight Bill of Lading, the property described above, in apparent good order, except as noted (contents and condition of the contents of packages unknown), marked, consigned and destined as shown above which said company (the word company being understood throughout this contract as meaning any person or corporation in possession of the property under the contract) agrees to carry to its usual place of delivery at said destination, if on its own railroad, water line, highway route or routes, or within the territory of its highway operations, otherwise to deliver to another carrier on the route to said destination. It is mutually agreed as to each carrier of all or any of said property, over all or any portion of said route to destination and as to each party at any time interested in all or any of said property, that every service to be performed hereunder shall be subject to all the Straight Bill of Lading terms and conditions in the governing classification of the date of shipment.							
Shipper hereby certifies that he is familiar with all the Bill of Lading terms and conditions in the governing classification and the said terms and conditions are hereby agreed to by the shipper and accepted for himself and his assigns.							
Shipper: MediWaste Disposal 235 Deininger Circle Corona, CA 92880 Permit: TS/OST-136 855-449-6334				Transporter: MediWaste Disposal 235 Deininger Circle Corona, CA 92880 Permit: 6300 855-449-6334			
Authorized Signature:		Date: 10/08/2025		Authorized Signature:		Date: 10/08/2025	
Print Name: ARTURO PINEDA				Printed Name:			

8 GALLON PHARMACEUTICAL



GENERATOR

11612559-1605795-077

Acme Dialysis
1234 Main St
Montebello, CA 90640

2025-10-08

TRANSPORTER

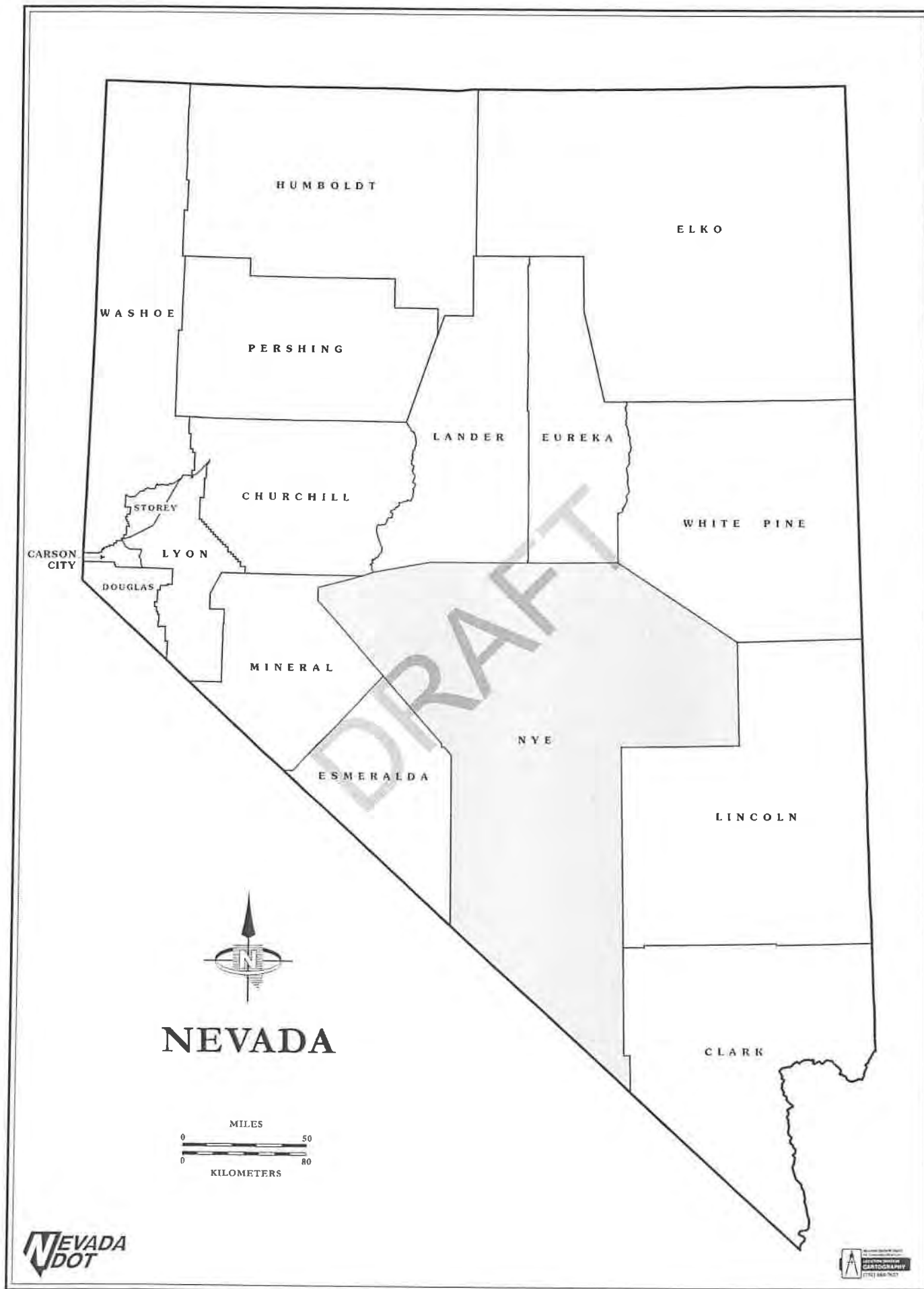
Mediowaste Disposal, Llc.
235 Deininger Cir
Corona, CA 92880-1707

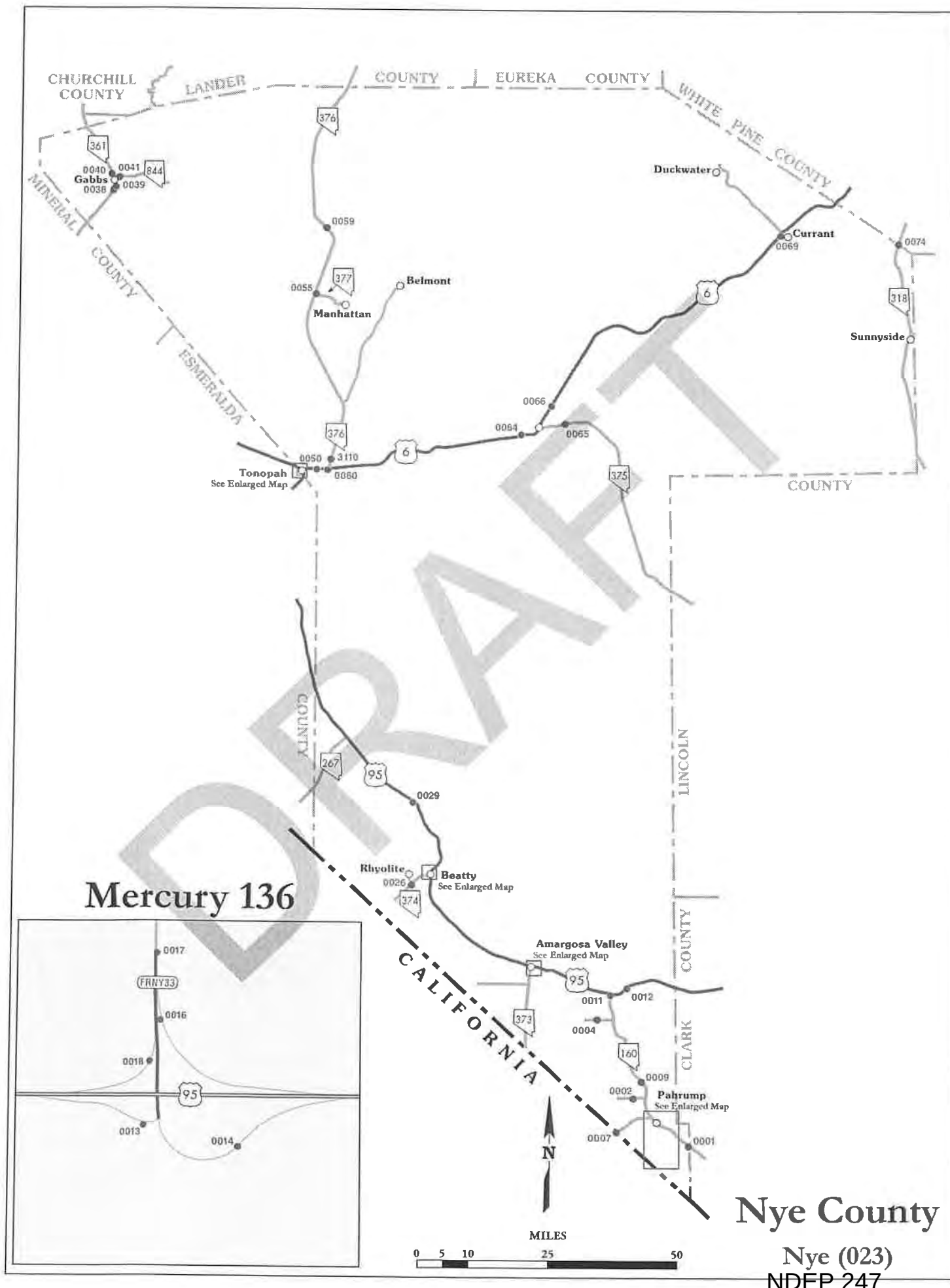
TREATMENT

MW Nevada DF
1850 E. Basin Ave
Pahrump, NV 89060

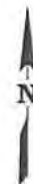
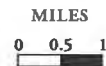
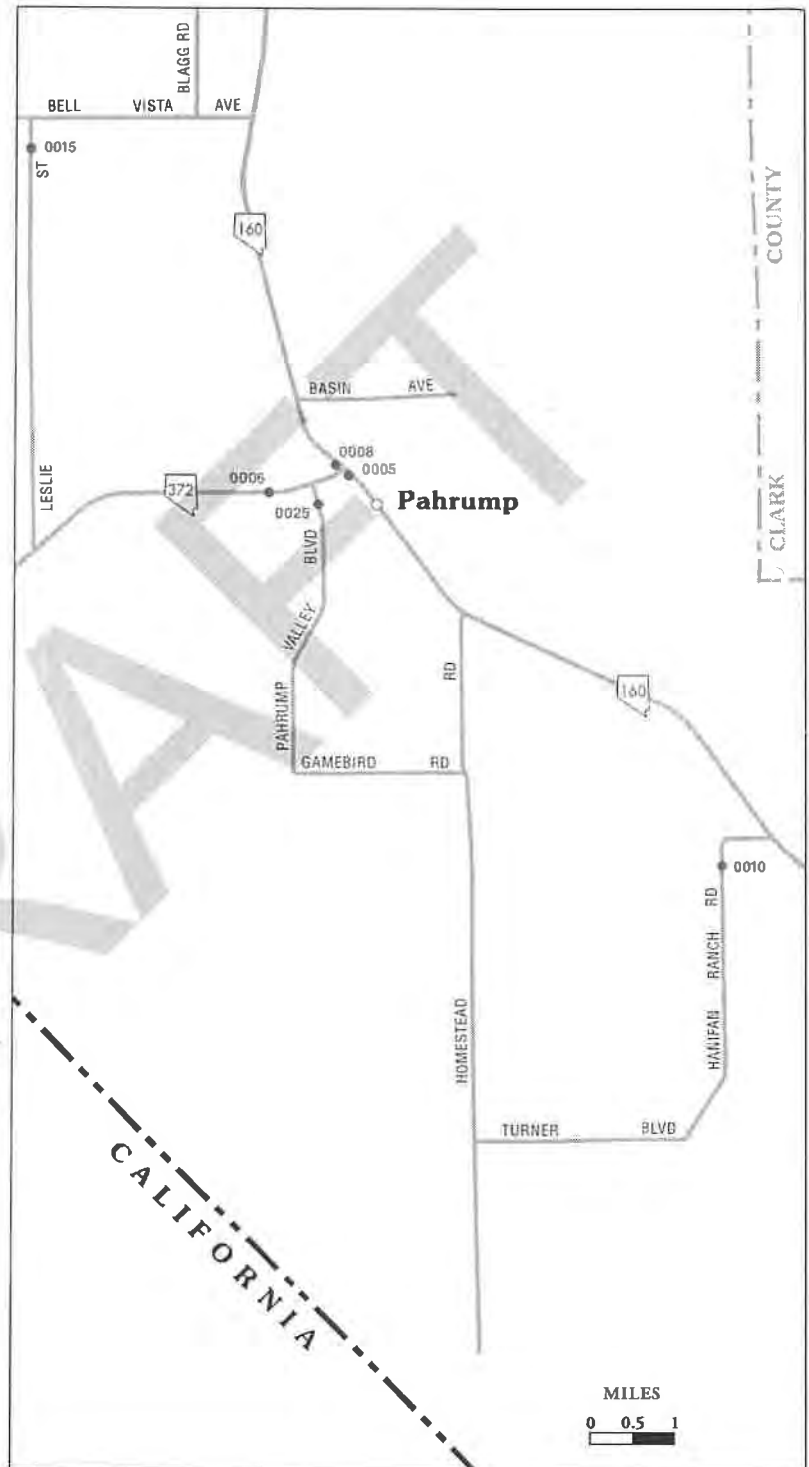
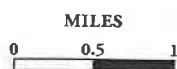
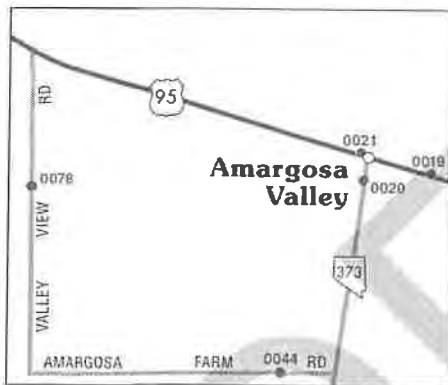
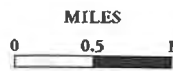
APPENDIX I. NDOT TRAFFIC DATA

DRAFT





Nye County
Nye (023)
NDEP 247



Nye County
Nye (23)
NDEP/248

State of Nevada Department of Transportation
Annual Average Daily Traffic Count Stations
County Name: Nye

		2015	2016	2017	2018	2019	2020	2021	2022	2023	2024
Station	Route/Location	AADT	AADT	AADT	AADT	AADT	AADT	AADT	AADT	AADT	AADT
0230001	SR160, .3 mi N of the CL/NY County Line	8250	8850	9200	10100	10200	9600	11200	11000	12100	12100
0230002	Bell Vista Ave, 400ft E of Leslie St	2250	2350	2100	2600	2500	2200	2300	2550	2700	3000
0230004	Crystal Rd, 320ft W of SR160	100	110	120	120	120	110	100	90	120	130
0230005	SR160, 585ft S of SR372 (Charles Brown Hwy)	21200	21800	23000	23600	24000	23700	23600	23400	24200	24100
0230006	SR372 (Charles Brown Hwy), 205ft W of West St	11300	10900	9100	10500	11200	10700	10800	10500	12400	11800
0230007	SR372 (Charles Brown Hwy), 580ft E of NV/CA Stateline	930	970	1100	1100	1150	1650	1300	1000	1350	980
0230008	SR160, 510ft N of SR372 (Charles Brown Hwy)	21900	22000	22000	23300	23200	22300	22400	22800	23200	23600
0230009	SR160, 245ft S of Leslie St	1550	1650	1700	1650	1950	1600	2000	1950	2000	2050
0230010	Haffen Ranch Rd, 315ft N of Manse Rd	1050	1100	1200	1300	1300	1050	1150	1350	1250	1450
0230011	SR160, .2 mi S of US95	930	1050	1000	1450	1200	1000	1100	1200	1200	1300
0230012	US95, 4 mi N of the Mercury Intch	3050	3150	3800	3150	3850	3250	3850	3750	4450	3850
0230013	US95, S/B off-ramp of the Mercury Intch 'Exit 136'	120	110	100	120	130	120	140	150	150	140
0230014	US95, S/B on-ramp of the Mercury Intch 'Exit 136'	360	370	350	400	460	410	440	490	520	510
0230015	Leslie St, 425ft S of Bell Vista Rd	1500	1500	1600	1750	1750	1550	1550	1700	1750	1700
0230016	US95, N/B off-ramp of the Mercury Intch 'Exit 136'	350	380	390	420	420	400	450	510	570	560
0230017	D.O.E Rd to Mercury Test Site, .2 mi N of the Mercury Intch 'Exit 136'	750	830	840	860	1000	930	1100	1250	1250	1400

Color indicates that this AADT value is estimated

		2015	2016	2017	2018	2019	2020	2021	2022	2023	2024
Station	Route/Location	AADT	AADT	AADT	AADT	AADT	AADT	AADT	AADT	AADT	AADT
0230018	US95, N/B on-ramp of the Mercury Intch 'Exit 136'	110	110	100	120	130	120	130	150	150	140
0230019	US95, 1.5 mi S of SR373 (Death Valley Jct Rd)	3000	3200	3200	3550	3600	3200	3650	3800	3500	3600
0230020	SR373 (Death Valley Junction Rd), .6 mi S of US95	690	750	810	810	1000	550	610	770	740	850
0230021	US95, 640ft N of SR373	3150	2850	3500	3050	3100	3000	3450	3600	3850	3300
0230022	US95, 315ft N of Cottonwood Ln	3900	4200	4100	4550	4150	4350	5000	4700	4950	4600
0230024	SR374 (Death Valley Rd), 375ft E of 'E' Ave	410	450	460	480	510	400	420	450	410	560
0230025	Pahrump Valley Rd, 495ft S of SR372	6400	6600	5500	6600	6650	6100	7850	7750	8150	8800
0230026	SR374 (Death Valley Rd), .2 mi W of Rhyolite Rd	260	290	300	300	370	310	230	220	260	350
0230028	US95, 1 mi N of SR374 (Death Valley Rd)	3050	3250	3400	4000	3600	3750	4300	4250	3550	4050
0230029	US95, 7 mi N of Springdale Rest Stop at mp 77	2600	2650	2600	2900	2700	2450	2750	4100	2900	2700
0230031	US95, .2 mi S of US6 in Tonopah	5250	5100	5400	5300	5300	4950	5450	5150	5200	5250
0230033	US95, 100ft S of Bryan Ave in Tonopah	7300	7800	6500	6550	6450	6200	6700	6700	6600	6650
0230034	US95, 500ft N of Cemetery Rd N of Tonopah	5600	6000	6100	4650	4500	4400	4850	4950	4750	4850
0230038	SR361 (Gabbs Valley Rd), 200ft N of 3rd St	170	170	200	210	290	230	360	240	220	300
0230039	County Rd, .15 mi E of SR361	160	140	170	180	210	170	240	180	170	120
0230040	SR361 (Gabbs Valley Rd), .2 mi N of SR844 (Ione Rd)	210	180	270	290	370	350	380	420	270	260
0230041	SR844 (Ione Rd), .2 mi E of SR361 (Gabbs Valley Rd)	110	110	110	120	70	80	50	70	60	50
0230044	Amargosa Farm Rd, .2 mi W of SR373	160	190	200	200	310	280	250	290	290	320
0230049	US6, .25 mi E of US95 in Tonopah	1700	1800	1600	1650	1850	1700	1800	1800	1650	1800

Color indicates that this AADT value is estimated

		2015	2016	2017	2018	2019	2020	2021	2022	2023	2024
Station	Route/Location	AADT	AADT	AADT	AADT	AADT	AADT	AADT	AADT	AADT	AADT
0230050	US6, .2 mi W of SR376 (Tonopah/Austin Rd)	1250	1200	1200	1350	1350	950	1350	1350	1250	1500
0230055	SR377 (Manhattan Rd), 400ft E of SR376 to Manhattan	100	120	110	110	110	140	150	100	80	100
0230059	SR376 (Tonopah-Austin Rd), 2.4 mi S of Carvers Rest Area	1200	1200	1200	1200	1350	1300	1200	1300	1100	1150
0230060	US6, .2 mi E of SR376 (Tonopah-Austin Rd)	620	730	590	680	670	610	680	670	590	750
0230064	US6, 200ft W of SR375 (Warm Springs Rd)	370	390	400	420	390	370	450	450	400	310
0230065	SR375 (Warm Springs Rd), 200ft E of US6	150	190	170	200	200	180	210	250	190	220
0230066	US6, 760ft E of SR375 (Warm Springs Rd)	230	240	190	250	250	170	220	250	220	160
0230069	US6, .6 mi W of SR379 (Duckwater Rd)	260	300	260	230	290	210	290	290	300	240
0230074	SR318 (Sunnyside Rd), N of Sunnyside at the NY-WP County Line	1350	1250	1600	1600	1600	1650	1650	1600	1550	1600
0230078	Valley View Rd, .2 mi W of US95	50	90	90	100	110	140	120	110	140	160

Color indicates that this AADT value is estimated

APPENDIX J. ASH SAMPLING DATA

DRAFT



SGS Silver State Analytical Laboratories
1135 Financial Blvd
Reno, NV 89502
(775) 857-2400
www.ssalabs.com

July 09, 2025
Workorder 25060929

F. Villamanga
MediWaste
235 Deininger Circle
Corona, CA 92880

Project: Hydrated Ash 6/10/25

Dear F. Villamanga:

It is the policy of SGS Silver State Analytical Laboratory - Reno to strictly adhere to a comprehensive Quality Assurance Plan that ensures the data presented in this report are both accurate and precise. SGS Silver State Analytical Laboratory - Reno maintains accreditation in the State of Nevada (NV-00015) and the State of California (ELAP 2990).

The data presented in this report was obtained from the analysis of samples received under a chain of custody. Unless otherwise noted below, samples were received in good condition, properly preserved and within the hold time for the requested analyses. Any anomalies associated with the analysis of the samples have been flagged in the Analytical Report with an appropriate explanation in the Definitions & Qualifiers.

25060929
Sample -01 Sub Contracted

Sincerely,

Carly Wood
Laboratory Director
1135 Financial Blvd
Reno, NV 89502



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Analytical Report

Workorder#: 25060929
Date Reported: 7/9/2025

Client: MediWaste
Project Name: Hydrated Ash 6/10/25
PO #:

Sampled By: F. Villamagna

Laboratory Accreditation Number: NV930/CA3029

Laboratory ID	Client Sample ID	Date/Time Sampled	Date Received
25060929-01	Hydrated Ash 6/10/25	06/10/2025 10:00	6/18/2025

Parameter	Method	Result	Units	PQL	Analyst	Date/Time Analyzed	Data Flag
Arsenic	EPA 6020B	1.2	µg/L	10	JG	06/30/2025 17:49	
Barium	EPA 6020B	328	µg/L	170	JG	06/30/2025 17:49	
Cadmium	EPA 6020B	< 1.00	µg/L	10	JG	06/30/2025 17:49	
Chromium	EPA 6020B	< 1.00	µg/L	10	JG	06/30/2025 17:49	
Lead	EPA 6020B	< 1.00	µg/L	10	JG	06/30/2025 17:49	
Mercury	EPA 7470	< 0.32	µg/L	0.32	JG	07/01/2025 15:31	S
Selenium	EPA 6020B	< 1.00	µg/L	10	JG	06/30/2025 17:49	
Silver	EPA 6020B	< 1.00	µg/L	10	JG	06/30/2025 17:49	

Laboratory Accreditation Number: NV015/CA2990

Laboratory ID	Client Sample ID	Date/Time Sampled	Date Received
25060929-02	Hydrated Ash 6/10/25	06/10/2025 10:00	6/18/2025

Parameter	Method	Result	Units	PQL	Analyst	Date/Time Analyzed	Data Flag
Antimony	EPA 6020	2.2	mg/Kg	2	AL	06/27/2025 15:53	
Arsenic	EPA 6020	<2	mg/Kg	2	AL	06/27/2025 15:53	
Barium	EPA 6020	2100	mg/Kg	492	AL	07/01/2025 17:31	
Beryllium	EPA 6020	<2	mg/Kg	2	AL	06/27/2025 15:53	
Cadmium	EPA 6020	<2	mg/Kg	2	AL	06/27/2025 15:53	
Chromium	EPA 6020	2.5	mg/Kg	2	AL	06/27/2025 15:53	
Cobalt	EPA 6020	1.1	mg/Kg	6	AL	06/27/2025 15:53	
Copper	EPA 6020	70.0	mg/Kg	10	AL	07/01/2025 15:23	
Lead	EPA 6020	1.2	mg/Kg	2	AL	06/27/2025 15:53	
Mercury	SW-846 7471B	< 0.2	mg/Kg	2	AL	07/04/2025 17:39	
Molybdenum	EPA 6020	6.4	mg/Kg	6	AL	06/27/2025 15:53	
Nickel	EPA 6020	3.4	mg/Kg	2	AL	06/27/2025 15:53	
Selenium	EPA 6020	<6	mg/Kg	6	AL	06/27/2025 15:53	
Silver	EPA 6020	0.2	mg/Kg	2	AL	06/27/2025 15:53	
Vanadium	EPA 6020	<2.0	mg/Kg	20	AL	06/27/2025 15:53	
Zinc	EPA 6020	3800	mg/Kg	1966	AL	07/01/2025 17:31	



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Quality Control Report

WO#: 25060929
7/9/2025

Analysis: Metals 6020 Solid
Method: EPA 6020

Batch ID: R102515

Method Blank

RunID: 102515 SeqNo 2808204 Units: mg/Kg
Analysis Date: 6/27/2025 3:31:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Antimony	< 0.0010	0.0010	
Arsenic	< 0.0010	0.0010	
Beryllium	< 0.0010	0.0010	
Cadmium	< 0.0010	0.0010	
Chromium	< 0.0010	0.0010	
Cobalt	< 0.0050	0.0050	
Lead	< 0.0010	0.0010	
Molybdenum	< 0.0050	0.0050	
Nickel	< 0.0010	0.0010	
Selenium	< 0.0050	0.0050	
Silver	< 0.0010	0.0010	
Vanadium	< 0.020	0.020	

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Quality Control Report

WO#: 25060929
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Method Blank

RunID: 102515 SeqNo 2808205 Units: mg/Kg
Analysis Date: 6/27/2025 3:33:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Antimony	< 0.0010	0.0010	
Arsenic	< 0.0010	0.0010	
Beryllium	< 0.0010	0.0010	
Cadmium	< 0.0010	0.0010	
Chromium	< 0.0010	0.0010	
Cobalt	< 0.0050	0.0050	
Lead	< 0.0010	0.0010	
Molybdenum	< 0.0050	0.0050	
Nickel	< 0.0010	0.0010	
Selenium	< 0.0050	0.0050	
Silver	< 0.0010	0.0010	
Vanadium	< 0.020	0.020	

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Quality Control Report

WO#: 25060929
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Method Blank

RunID: 102515 SeqNo 2808217 Units: mg/Kg
Analysis Date: 6/27/2025 3:49:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Antimony	< 1.0	1.0	
Arsenic	< 1.0	1.0	
Beryllium	< 1.0	1.0	
Cadmium	< 1.0	1.0	
Chromium	< 1.0	1.0	
Cobalt	< 5.0	5.0	
Lead	< 1.0	1.0	B
Molybdenum	< 5.0	5.0	
Nickel	< 1.0	1.0	B
Selenium	< 5.0	5.0	
Silver	< 1.0	1.0	
Vanadium	< 20	20	

Laboratory Control Sample (LCS)

RunID: 102515 SeqNo 2808203 Units: mg/L
Analysis Date: 6/27/2025 3:29:00 PM Analyst: AL

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Antimony	0.02500	0.026	104								
Arsenic	0.02500	0.026	104								
Beryllium	0.02500	0.026	103								
Cadmium	0.02500	0.026	106								
Chromium	0.02500	0.026	105								
Cobalt	0.02500	0.026	106								
Lead	0.02500	0.026	105								
Molybdenum	0.02500	0.026	105								
Nickel	0.02500	0.027	108								
Selenium	0.1250	0.13	106								
Silver	0.02500	0.027	108								
Vanadium	0.02500	0.026	103								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060930-02A

RunID: 102515 SeqNo 2808224 Units: mg/Kg
Analysis Date: 6/27/2025 3:58:00 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Antimony	19.37	50.00	73	107	50.00	73	107	0.0675	20	70	130	
Arsenic	0.8251	50.00	51	100	50.00	51	101	0.619	20	70	130	
Beryllium	0.1734	50.00	44	88.6	50.00	44	88.3	0.350	20	70	130	
Cadmium	1.055	50.00	50	97.6	50.00	51	100	2.79	20	70	130	
Chromium	32.08	50.00	82	100	50.00	83	101	0.425	20	70	130	
Cobalt	13.22	50.00	61	95.0	50.00	62	98.3	2.63	20	70	130	

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Lead	12.63	50.00	61	97.6	50.00	61	96.1	1.19	20	70	130	
Molybdenum	9.677	50.00	61	103	50.00	63	106	2.78	20	70	130	
Nickel	45.04	50.00	91	92.6	50.00	93	96.1	1.90	20	70	130	
Selenium	3.568	250.0	250	99.8	250.0	260	101	1.60	20	70	130	
Silver	4.272	50.00	54	99.2	50.00	55	101	2.07	20	70	130	
Vanadium	8.590	50.00	58	98.2	50.00	58	99.2	0.878	20	70	130	

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060930-02A

RunID: 102515 SeqNo 2808225 Units: mg/Kg

Analysis Date: 6/27/2025 3:59:00 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Antimony	19.37	50.00	73	107								
Arsenic	0.8251	50.00	51	101								
Beryllium	0.1734	50.00	44	88.3								
Cadmium	1.055	50.00	51	100								
Chromium	32.08	50.00	83	101								
Cobalt	13.22	50.00	62	98.3								
Lead	12.63	50.00	61	96.1								
Molybdenum	9.677	50.00	63	106								
Nickel	45.04	50.00	93	96.1								
Selenium	3.568	250.0	260	101								
Silver	4.272	50.00	55	101								
Vanadium	8.590	50.00	58	99.2								

Analysis: Metals, EPA6020

Method: EPA 6020B

Batch ID: R102596

Method Blank

RunID: 102596 SeqNo 2811796 Units: µg/L

Analysis Date: 6/30/2025 5:16:17 PM Analyst: JG

Analyte	Result	Rep Limit	Rep Qual
Arsenic	< 1.00	1.00	
Barium	< 1.00	1.00	
Cadmium	< 1.00	1.00	
Chromium	< 1.00	1.00	
Lead	< 1.00	1.00	
Selenium	< 1.00	1.00	
Silver	< 1.00	1.00	

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Quality Control Report

WO#: 25060929

7/9/2025

Method Blank

RunID: 102596 SeqNo 2811801 Units: µg/L

Analysis Date: 6/30/2025 5:44:01 PM Analyst: JG

Analyte	Result	Rep Limit	Rep Qual
Arsenic	< 10.0	10.0	
Barium	< 170	170	
Cadmium	< 10.0	10.0	
Chromium	< 10.0	10.0	
Lead	< 10.0	10.0	
Selenium	< 10.0	10.0	
Silver	< 10.0	10.0	

Laboratory Control Sample (LCS)

RunID: 102596 SeqNo 2811794 Units: µg/L

Analysis Date: 6/30/2025 5:05:10 PM Analyst: JG

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Arsenic	40.00	41.6	104								
Barium	40.00	41.8	104								
Cadmium	40.00	41.4	104								
Chromium	40.00	42.9	107								
Lead	40.00	42.0	105								
Selenium	200.0	208	104								
Silver	40.00	42.9	107								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060929-01A

RunID: 102596 SeqNo 2811803 Units: µg/L

Analysis Date: 6/30/2025 5:55:09 PM Analyst: JG

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Arsenic	11.90	400.0	388	93.9	400.0	380	92.0	2.01	20	75	125	
Barium	328.5	400.0	715	96.6	400.0	716	96.8	0.140	20	75	125	
Cadmium	1.500	400.0	373	93.0	400.0	371	92.4	0.672	20	75	125	
Chromium	7.000	400.0	395	97.0	400.0	385	94.4	2.72	20	75	125	
Lead	1.700	400.0	386	96.0	400.0	382	95.0	1.04	20	75	125	
Selenium	0	2000	1900	95.1	2000	1890	94.3	0.813	20	75	125	
Silver	0	400.0	383	95.8	400.0	379	94.8	1.08	20	75	125	

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060929-01A

RunID: 102596 SeqNo 2811804 Units: µg/L

Analysis Date: 6/30/2025 6:00:37 PM Analyst: JG

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual

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Arsenic	11.90	400.0	380	92.0
Barium	328.5	400.0	716	96.8
Cadmium	1.500	400.0	371	92.4
Chromium	7.000	400.0	385	94.4
Lead	1.700	400.0	382	95.0
Selenium	0	2000	1890	94.3
Silver	0	400.0	379	94.8

Analysis: Mercury - RCRA
Method: EPA 7470

Batch ID: R102598

Method Blank

RunID: 102598 SeqNo 2811086 Units: µg/L
Analysis Date: 7/1/2025 2:47:00 PM Analyst: JG

Analyte	Result	Rep Limit	Rep Qual
Mercury	< 0.16	0.16	

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Quality Control Report

WO#: 25060929

7/9/2025

Method Blank

RunID: 102598 SeqNo 2811092 Units: µg/L

Analysis Date: 7/1/2025 3:17:00 PM Analyst: JG

Analyte	Result	Rep Limit	Rep Qual
Mercury	< 0.32	0.32	

Laboratory Control Sample (LCS)

RunID: 102598 SeqNo 2811085 Units: µg/L

Analysis Date: 7/1/2025 2:44:00 PM Analyst: JG

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	5.000	5.0	99.5								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061233-01A

RunID: 102598 SeqNo 2811090 Units: µg/L

Analysis Date: 7/1/2025 3:03:00 PM Analyst: JG

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0	10.00	8.3	82.9	10.00	8.0	79.9	3.68	20	80	120	S

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061233-01A

RunID: 102598 SeqNo 2811091 Units: µg/L

Analysis Date: 7/1/2025 3:05:00 PM Analyst: JG

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0	10.00	8.0	79.9								

Analysis: Metals 6020 Solid

Method: EPA 6020

Batch ID: R102605

Method Blank

RunID: 102605 SeqNo 2811216 Units: mg/Kg

Analysis Date: 7/1/2025 3:17:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Barium	< 0.0050	0.0050	
Copper	< 0.0010	0.0010	
Zinc	< 0.020	0.020	

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Quality Control Report

WO#: 25060929
7/9/2025

Method Blank

RunID: 102605 SeqNo 2811217 Units: mg/Kg

Analysis Date: 7/1/2025 3:18:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Barium	< 0.0050	0.0050	
Copper	< 0.0010	0.0010	
Zinc	< 0.020	0.020	

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Quality Control Report

WO#: 25060929
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Method Blank

RunID: 102605 SeqNo 2811218 Units: mg/Kg
Analysis Date: 7/1/2025 3:19:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Barium	< 0.010	0.010	
Copper	< 0.0020	0.0020	
Zinc	< 0.040	0.040	

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Quality Control Report

WO#: 25060929
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Method Blank

RunID: 102605 SeqNo 2811229 Units: mg/Kg
Analysis Date: 7/1/2025 3:36:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Barium	< 0.010	0.010	
Copper	< 0.0020	0.0020	
Zinc	< 0.040	0.040	

Laboratory Control Sample (LCS)

RunID: 102605 SeqNo 2811214 Units: mg/L
Analysis Date: 7/1/2025 3:14:00 PM Analyst: AL

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Barium	0.02500	0.023	93.2								
Copper	0.02500	0.026	105								
Zinc	0.02500	0.024	94.4								

Laboratory Control Sample (LCS)

RunID: 102605 SeqNo 2811215 Units: mg/L
Analysis Date: 7/1/2025 3:15:00 PM Analyst: AL

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Barium	0.02500	0.023	93.1								
Copper	0.02500	0.026	105								
Zinc	0.02500	0.024	96.5								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060930-02A

RunID: 102605 SeqNo 2811227 Units: mg/Kg
Analysis Date: 7/1/2025 3:33:00 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Barium	1748	50.00	1800	41.4	50.00	1700	2.62	1.10	20	70	130	S
Copper	622.9	50.00	670	84.5	50.00	670	91.2	0.503	20	70	130	
Zinc	3876	50.00	3800	-52.8	50.00	3900	30.2	1.07	20	70	130	S

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060930-02A

RunID: 102605 SeqNo 2811228 Units: mg/Kg
Analysis Date: 7/1/2025 3:35:00 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Barium	1748	50.00	1700	2.62								
Copper	622.9	50.00	670	91.2								

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WO#: 25060929
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Zinc	3876	50.00	3900	30.2
------	------	-------	------	------

Analysis: Mercury, RCRA Solid
Method: SW-846 7471B

Batch ID: R102714

Method Blank

RunID: 102714 SeqNo 2813843 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Mercury	< 0.00010	0.00010	

Laboratory Control Sample (LCS)

RunID: 102714 SeqNo 2813842 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.006000	0.0060	99.8								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061035-03A

RunID: 102714 SeqNo 2813854 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.06280	1.000	< 2.0	108	1.000	< 2.0	110	0	20	80	120	

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061035-03A

RunID: 102714 SeqNo 2813855 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.06280	1.000	< 2.0	110								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061035-08A

RunID: 102714 SeqNo 2813861 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.07810	1.000	< 2.0	111	1.000	< 2.0	107	0	20	80	120	

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Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061035-08A

RunID: 102714 SeqNo 2813862 Units: mg/Kg

Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.07810	1.000	< 2.0	107								

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CHAIN-OF-CUSTODY-RECORD

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[illegible]



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Definitions & Qualifiers

WO#: 25060929
Date: 7/9/2025

Definitions:

LCS: Laboratory Control Sample; prepared by adding a known mass of target analytes to a specified amount of de-ionized water and prepared with the batch of samples, used to calculate Accuracy (%REC).

LCSD: LCS Duplicate; used to calculate both Accuracy (%REC) and Precision (%RPD)

MBLK: Method Blank; a sample of similar matrix that is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

MS: Matrix Spike; prepared by adding a known mass of target analytes to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available, used to calculate Accuracy (%REC)

MSD: Matrix Spike Duplicate; used to calculate both Accuracy (%REC) and Precision (%RPD)

RPD: Relative Percent Difference; comparison between sample and duplicate and/or MS and MSD.

PQL: Practical Quantitation Limit; the limit to which data is quantitated for reporting.

MDL: Method Detection Limit; the limit to which the instrument can reliably detect.

MCL: Maximum Contaminant Level; value set according to EPA guidelines.

TNTC: Too Numerous to Count; colony density is too thick to be individually counted or greater than method reporting requirements.

Qualifiers:

* - Analyte exceeds Safe Drinking Water Act MCL, does not meet drinking water standards.

- Laboratory not accredited for this analyte.

C - Analyte value below Safe Drinking Water Act MCL, does not meet drinking water standards.

B - Analyte found above the PQL in associated method blank.

G - Calibration blank analyte detected above PQL.

H - Sample analyzed beyond holding time for this parameter.

J - Estimated Value; Analyte found between MDL and PQL limits.

L - Sample concentration is at least 5 times greater than spike contribution. Spike recovery criteria do not apply.

R - RPD between sample and duplicate sample outside the RPD acceptance limits.

S - Batch MS and/or MSD were outside acceptance limits, batch LCS was acceptable.

W - Sample temperature when received was out of limit as specified by method.

Z - Batch LCS and/or LCSD were outside acceptance limits.



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July 09, 2025
Workorder 25060930

F. Villamanga
MediWaste
235 Deininger Circle
Corona, CA 92880

Project: Hydrated Ash 6/9/25

Dear F. Villamanga:

It is the policy of SGS Silver State Analytical Laboratory - Reno to strictly adhere to a comprehensive Quality Assurance Plan that ensures the data presented in this report are both accurate and precise. SGS Silver State Analytical Laboratory - Reno maintains accreditation in the State of Nevada (NV-00015) and the State of California (ELAP 2990).

The data presented in this report was obtained from the analysis of samples received under a chain of custody. Unless otherwise noted below, samples were received in good condition, properly preserved and within the hold time for the requested analyses. Any anomalies associated with the analysis of the samples have been flagged in the Analytical Report with an appropriate explanation in the Definitions & Qualifiers.

25060930
Sample -01 Sub Contracted.

Sincerely,

Carly Wood
Laboratory Director
1135 Financial Blvd
Reno, NV 89502



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Analytical Report

Workorder#: 25060930

Date Reported: 7/9/2025

Client: MediWaste

Project Name: Hydrated Ash 6/9/25

PO #:

Sampled By: F. Villamanga

Laboratory Accreditation Number: NV930/CA3029

Laboratory ID	Client Sample ID	Date/Time Sampled	Date Received
25060930-01	Hydrated Ash 6/9/25	06/09/2025 10:00	6/18/2025

Parameter	Method	Result	Units	PQL	Analyst	Date/Time Analyzed	Data Flag
Arsenic	EPA 6020B	0.28	µg/L	10	JG	06/30/2025 18:06	
Barium	EPA 6020B	518	µg/L	170	JG	06/30/2025 18:06	
Cadmium	EPA 6020B	< 1.00	µg/L	10	JG	06/30/2025 18:06	
Chromium	EPA 6020B	< 1.00	µg/L	10	JG	06/30/2025 18:06	
Lead	EPA 6020B	2.0	µg/L	10	JG	06/30/2025 18:06	
Mercury	EPA 7470	< 0.32	µg/L	0.32	JG	07/01/2025 15:28	S
Selenium	EPA 6020B	< 1.00	µg/L	10	JG	06/30/2025 18:06	
Silver	EPA 6020B	< 1.00	µg/L	10	JG	06/30/2025 18:06	

Laboratory Accreditation Number: NV015/CA2990

Laboratory ID	Client Sample ID	Date/Time Sampled	Date Received
25060930-02	Hydrated Ash 6/9/25	06/09/2025 10:00	6/18/2025

Parameter	Method	Result	Units	PQL	Analyst	Date/Time Analyzed	Data Flag
Antimony	EPA 6020	1.9	mg/Kg	2	AL	06/27/2025 15:54	
Arsenic	EPA 6020	<2	mg/Kg	2	AL	06/27/2025 15:54	
Barium	EPA 6020	1900	mg/Kg	494	AL	07/01/2025 17:32	
Beryllium	EPA 6020	<2	mg/Kg	2	AL	06/27/2025 15:54	
Cadmium	EPA 6020	< 2.0	mg/Kg	2	AL	06/27/2025 15:54	
Chromium	EPA 6020	3.2	mg/Kg	2	AL	06/27/2025 15:54	
Cobalt	EPA 6020	1.3	mg/Kg	6	AL	06/27/2025 15:54	
Copper	EPA 6020	79.0	mg/Kg	10	AL	07/01/2025 15:29	
Lead	EPA 6020	1.3	mg/Kg	2	AL	06/27/2025 15:54	
Mercury	SW-846 7471B	< 0.2	mg/Kg	2	AL	07/04/2025 17:39	
Molybdenum	EPA 6020	0.7	mg/Kg	6	AL	06/27/2025 15:54	
Nickel	EPA 6020	4.5	mg/Kg	2	AL	06/27/2025 15:54	
Selenium	EPA 6020	<6	mg/Kg	6	AL	06/27/2025 15:54	
Silver	EPA 6020	4.3	mg/Kg	2	AL	06/27/2025 15:54	
Vanadium	EPA 6020	<20	mg/Kg	20	AL	06/27/2025 15:54	
Zinc	EPA 6020	4800	mg/Kg	1972	AL	07/01/2025 17:32	



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Quality Control Report

WO#: 25060930
7/9/2025

Analysis: Metals 6020 Solid
Method: EPA 6020

Batch ID: R102515

Method Blank

RunID: 102515 SeqNo 2808204 Units: mg/Kg
Analysis Date: 6/27/2025 3:31:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Antimony	< 0.0010	0.0010	
Arsenic	< 0.0010	0.0010	
Beryllium	< 0.0010	0.0010	
Cadmium	< 0.0010	0.0010	
Chromium	< 0.0010	0.0010	
Cobalt	< 0.0050	0.0050	
Lead	< 0.0010	0.0010	
Molybdenum	< 0.0050	0.0050	
Nickel	< 0.0010	0.0010	
Selenium	< 0.0050	0.0050	
Silver	< 0.0010	0.0010	
Vanadium	< 0.020	0.020	

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Quality Control Report

WO#: 25060930
7/9/2025

Method Blank

RunID: 102515 SeqNo 2808205 Units: mg/Kg
Analysis Date: 6/27/2025 3:33:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Antimony	< 0.0010	0.0010	
Arsenic	< 0.0010	0.0010	
Beryllium	< 0.0010	0.0010	
Cadmium	< 0.0010	0.0010	
Chromium	< 0.0010	0.0010	
Cobalt	< 0.0050	0.0050	
Lead	< 0.0010	0.0010	
Molybdenum	< 0.0050	0.0050	
Nickel	< 0.0010	0.0010	
Selenium	< 0.0050	0.0050	
Silver	< 0.0010	0.0010	
Vanadium	< 0.020	0.020	

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Quality Control Report

WO#: 25060930
7/9/2025

Method Blank

RunID: 102515 SeqNo 2808217 Units: mg/Kg
Analysis Date: 6/27/2025 3:49:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Antimony	< 1.0	1.0	
Arsenic	< 1.0	1.0	
Beryllium	< 1.0	1.0	
Cadmium	< 1.0	1.0	
Chromium	< 1.0	1.0	
Cobalt	< 5.0	5.0	
Lead	< 1.0	1.0	B
Molybdenum	< 5.0	5.0	
Nickel	< 1.0	1.0	B
Selenium	< 5.0	5.0	
Silver	< 1.0	1.0	
Vanadium	< 20	20	

Laboratory Control Sample (LCS)

RunID: 102515 SeqNo 2808203 Units: mg/L
Analysis Date: 6/27/2025 3:29:00 PM Analyst: AL

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Antimony	0.02500	0.026	104								
Arsenic	0.02500	0.026	104								
Beryllium	0.02500	0.026	103								
Cadmium	0.02500	0.026	106								
Chromium	0.02500	0.026	105								
Cobalt	0.02500	0.026	106								
Lead	0.02500	0.026	105								
Molybdenum	0.02500	0.026	105								
Nickel	0.02500	0.027	108								
Selenium	0.1250	0.13	106								
Silver	0.02500	0.027	108								
Vanadium	0.02500	0.026	103								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060930-02A

RunID: 102515 SeqNo 2808224 Units: mg/Kg
Analysis Date: 6/27/2025 3:58:00 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Antimony	19.37	50.00	73	107	50.00	73	107	0.0675	20	70	130	
Arsenic	0.8251	50.00	51	100	50.00	51	101	0.619	20	70	130	
Beryllium	0.1734	50.00	44	88.6	50.00	44	88.3	0.350	20	70	130	
Cadmium	1.055	50.00	50	97.6	50.00	51	100	2.79	20	70	130	
Chromium	32.08	50.00	82	100	50.00	83	101	0.425	20	70	130	
Cobalt	13.22	50.00	61	95.0	50.00	62	98.3	2.63	20	70	130	

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Lead	12.63	50.00	61	97.6	50.00	61	96.1	1.19	20	70	130	
Molybdenum	9.677	50.00	61	103	50.00	63	106	2.78	20	70	130	
Nickel	45.04	50.00	91	92.6	50.00	93	96.1	1.90	20	70	130	
Selenium	3.568	250.0	250	99.8	250.0	260	101	1.60	20	70	130	
Silver	4.272	50.00	54	99.2	50.00	55	101	2.07	20	70	130	
Vanadium	8.590	50.00	58	98.2	50.00	58	99.2	0.878	20	70	130	

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060930-02A

RunID: 102515 SeqNo 2808225 Units: mg/Kg

Analysis Date: 6/27/2025 3:59:00 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Antimony	19.37	50.00	73	107								
Arsenic	0.8251	50.00	51	101								
Beryllium	0.1734	50.00	44	88.3								
Cadmium	1.055	50.00	51	100								
Chromium	32.08	50.00	83	101								
Cobalt	13.22	50.00	62	98.3								
Lead	12.63	50.00	61	96.1								
Molybdenum	9.677	50.00	63	106								
Nickel	45.04	50.00	93	96.1								
Selenium	3.568	250.0	260	101								
Silver	4.272	50.00	55	101								
Vanadium	8.590	50.00	58	99.2								

Analysis: Metals, EPA6020

Method: EPA 6020B

Batch ID: R102596

Method Blank

RunID: 102596 SeqNo 2811796 Units: µg/L

Analysis Date: 6/30/2025 5:16:17 PM Analyst: JG

Analyte	Result	Rep Limit	Rep Qual
Arsenic	< 1.00	1.00	
Barium	< 1.00	1.00	
Cadmium	< 1.00	1.00	
Chromium	< 1.00	1.00	
Lead	< 1.00	1.00	
Selenium	< 1.00	1.00	
Silver	< 1.00	1.00	

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Quality Control Report

WO#: 25060930
7/9/2025

Method Blank

RunID: 102596 SeqNo 2811801 Units: µg/L
Analysis Date: 6/30/2025 5:44:01 PM Analyst: JG

Analyte	Result	Rep Limit	Rep Qual
Arsenic	< 10.0	10.0	
Barium	< 170	170	
Cadmium	< 10.0	10.0	
Chromium	< 10.0	10.0	
Lead	< 10.0	10.0	
Selenium	< 10.0	10.0	
Silver	< 10.0	10.0	

Laboratory Control Sample (LCS)

RunID: 102596 SeqNo 2811794 Units: µg/L
Analysis Date: 6/30/2025 5:05:10 PM Analyst: JG

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Arsenic	40.00	41.6	104								
Barium	40.00	41.8	104								
Cadmium	40.00	41.4	104								
Chromium	40.00	42.9	107								
Lead	40.00	42.0	105								
Selenium	200.0	208	104								
Silver	40.00	42.9	107								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060929-01A

RunID: 102596 SeqNo 2811803 Units: µg/L
Analysis Date: 6/30/2025 5:55:09 PM Analyst: JG

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Arsenic	11.90	400.0	388	93.9	400.0	380	92.0	2.01	20	75	125	
Barium	328.5	400.0	715	96.6	400.0	716	96.8	0.140	20	75	125	
Cadmium	1.500	400.0	373	93.0	400.0	371	92.4	0.672	20	75	125	
Chromium	7.000	400.0	395	97.0	400.0	385	94.4	2.72	20	75	125	
Lead	1.700	400.0	386	96.0	400.0	382	95.0	1.04	20	75	125	
Selenium	0	2000	1900	95.1	2000	1890	94.3	0.813	20	75	125	
Silver	0	400.0	383	95.8	400.0	379	94.8	1.08	20	75	125	

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060929-01A

RunID: 102596 SeqNo 2811804 Units: µg/L
Analysis Date: 6/30/2025 6:00:37 PM Analyst: JG

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual

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Arsenic	11.90	400.0	380	92.0
Barium	328.5	400.0	716	96.8
Cadmium	1.500	400.0	371	92.4
Chromium	7.000	400.0	385	94.4
Lead	1.700	400.0	382	95.0
Selenium	0	2000	1890	94.3
Silver	0	400.0	379	94.8

Analysis: Mercury - RCRA
Method: EPA 7470

Batch ID: R102598

Method Blank

RunID: 102598 SeqNo 2811086 Units: µg/L
Analysis Date: 7/1/2025 2:47:00 PM Analyst: JG

Analyte	Result	Rep Limit	Rep Qual
Mercury	< 0.16	0.16	

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WO#: 25060930
7/9/2025

Method Blank

RunID: 102598 SeqNo 2811092 Units: µg/L
Analysis Date: 7/1/2025 3:17:00 PM Analyst: JG

Analyte	Result	Rep Limit	Rep Qual
Mercury	< 0.32	0.32	

Laboratory Control Sample (LCS)

RunID: 102598 SeqNo 2811085 Units: µg/L
Analysis Date: 7/1/2025 2:44:00 PM Analyst: JG

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	5.000	5.0	99.5								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061233-01A

RunID: 102598 SeqNo 2811090 Units: µg/L
Analysis Date: 7/1/2025 3:03:00 PM Analyst: JG

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0	10.00	8.3	82.9	10.00	8.0	79.9	3.68	20	80	120	S

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061233-01A

RunID: 102598 SeqNo 2811091 Units: µg/L
Analysis Date: 7/1/2025 3:05:00 PM Analyst: JG

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0	10.00	8.0	79.9								

Analysis: Metals 6020 Solid
Method: EPA 6020

Batch ID: R102605

Method Blank

RunID: 102605 SeqNo 2811216 Units: mg/Kg
Analysis Date: 7/1/2025 3:17:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Barium	< 0.0050	0.0050	
Copper	< 0.0010	0.0010	
Zinc	< 0.020	0.020	

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WO#: 25060930
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Method Blank

RunID: 102605 SeqNo 2811217 Units: mg/Kg
Analysis Date: 7/1/2025 3:18:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Barium	< 0.0050	0.0050	
Copper	< 0.0010	0.0010	
Zinc	< 0.020	0.020	

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WO#: 25060930
7/9/2025

Method Blank

RunID: 102605 SeqNo 2811218 Units: mg/Kg
Analysis Date: 7/1/2025 3:19:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Barium	< 0.010	0.010	
Copper	< 0.0020	0.0020	
Zinc	< 0.040	0.040	

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WO#: 25060930
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Method Blank

RunID: 102605 SeqNo 2811229 Units: mg/Kg
Analysis Date: 7/1/2025 3:36:00 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Barium	< 0.010	0.010	
Copper	< 0.0020	0.0020	
Zinc	< 0.040	0.040	

Laboratory Control Sample (LCS)

RunID: 102605 SeqNo 2811214 Units: mg/L
Analysis Date: 7/1/2025 3:14:00 PM Analyst: AL

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Barium	0.02500	0.023	93.2								
Copper	0.02500	0.026	105								
Zinc	0.02500	0.024	94.4								

Laboratory Control Sample (LCS)

RunID: 102605 SeqNo 2811215 Units: mg/L
Analysis Date: 7/1/2025 3:15:00 PM Analyst: AL

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Barium	0.02500	0.023	93.1								
Copper	0.02500	0.026	105								
Zinc	0.02500	0.024	96.5								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060930-02A
RunID: 102605 SeqNo 2811227 Units: mg/Kg
Analysis Date: 7/1/2025 3:33:00 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Barium	1748	50.00	1800	41.4	50.00	1700	2.62	1.10	20	70	130	S
Copper	622.9	50.00	670	84.5	50.00	670	91.2	0.503	20	70	130	
Zinc	3876	50.00	3800	-52.8	50.00	3900	30.2	1.07	20	70	130	S

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25060930-02A
RunID: 102605 SeqNo 2811228 Units: mg/Kg
Analysis Date: 7/1/2025 3:35:00 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Barium	1748	50.00	1700	2.62								
Copper	622.9	50.00	670	91.2								

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Zinc	3876	50.00	3900	30.2
------	------	-------	------	------

Analysis: Mercury, RCRA Solid
Method: SW-846 7471B

Batch ID: R102714

Method Blank

RunID: 102714 SeqNo 2813843 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Result	Rep Limit	Rep Qual
Mercury	< 0.00010	0.00010	

Laboratory Control Sample (LCS)

RunID: 102714 SeqNo 2813842 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	LCS Spike Added	LCS Result	LCS % Recovery	LCSD Spike Added	LCSD Result	LCSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.006000	0.0060	99.8								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061035-03A

RunID: 102714 SeqNo 2813854 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.06280	1.000	< 2.0	108	1.000	< 2.0	110	0	20	80	120	

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061035-03A

RunID: 102714 SeqNo 2813855 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.06280	1.000	< 2.0	110								

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Sample Spiked: 25061035-08A

RunID: 102714 SeqNo 2813861 Units: mg/Kg
Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.07810	1.000	< 2.0	111	1.000	< 2.0	107	0	20	80	120	

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7/9/2025

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

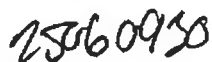
Sample Spiked: 25061035-08A

RunID: 102714 SeqNo 2813862 Units: mg/Kg

Analysis Date: 7/4/2025 5:39:58 PM Analyst: AL

Analyte	Sample Result	MS Spike Added	MS Result	MS % Recovery	MSD Spike Added	MSD Result	MSD % Recovery	RPD	RPD Limit	Low Limit	High Limit	Qual
Mercury	0.07810	1.000	< 2.0	107								

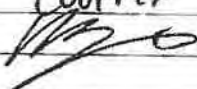
Original



CHAIN-OF-CUSTODY-RECOF

☐ 1135 FINANCIAL BOULEVARD, RENO, NV 89502
Phone (775) 857-2400 Fax: (888) 398-7002 (EPA#: NV00015, CA2526)

Page 1 of 1

Report Results To:	Report Attention:	Ryan Oganesian	Project Number:	Pahrump	Send Invoice To:	Invoice Attention:	Ryan Oganesian	PO#	Quote #	COMPLIANCE MONITORING? Yes ☐ No ☐	NEW ADDRESS? Yes ☐ No ☐	Results: Invoice: ☐
	Company:	MediWaste				Company:	MediWaste					
	Mailing Address:	235 Deininger Circle				Mailing Address:	235 Deininger Circle			Applicable Program SDWA ☐ CWA ☐ RCRA ☐ Mining ☐ Other ☐		
	City, State, Zip:	Corona, CA 92880				City, State, Zip:	Corona, CA 92880					
Phone:	714-401-3327	Email / Fax:	fvillamagna@msn.com		Phone:	714-401-3327	Email / Fax:	fvillamagna@msn.com	QC Level Report ① ② ③ ④ NOTE: Surcharges apply to Level II, III and IV only			
Sampled by: F. Villamagna Signature: 					ANALYSES REQUESTED					Send Results Via: Mail: ☐ Email: ☐ Fax: ☐		
I attest to the validity and authenticity of the sample. I am aware that tampering with or intentionally mislabeling the sample location, data or time is considered fraud and may be grounds for legal action.					Other Pertinent Information / Special Instructions					Send Invoice Via: Mail: ☐ Email: ☐ Fax: ☐		
Standard: ☐ Standard TAT 7-10 Business Days. Note that some tests vary. Rush Same Day: ☐ 3 Day: ☐ Other (specify): _____ 1 Day: ☐ 4 Day: ☐ Rush results will be issued after 4:00 p.m. 2 Day: ☐ 5 Day: ☐ NOTE: A Rush Surcharge is applied for rush samples										Field Measurements On-Site pH _____ Chlorine: _____ Temperature _____ Other: _____		
Date Sampled	Time Sampled	Sample Identification	SSAL - SEM Lab No.	Comp. Grail	Mult	Preservative	TCLP 8	TTL				
05/09/25	10:00 AM	Hydrated Ash 6/9/2025					X	X				
COMMENTS												
Relinquished By: 			Print Name F. Villamagna			Company Vulcan			Date 6/10/2025		Time 12:00	
Received By:			Carrier									
Relinquished By:			Carrier									
Received By:									6-18-25		12:30	
Relinquished By:												
Received By:												
Authorized By:												
Authorization is required to process samples. This obligates your organization for service fees. SSAL Standard T & C's or other written agreement applies. If collections or legal services are required to recover said fees, your organization will be responsible for all fees and costs in addition to service fees.												
Samples are discarded 30 days after results are reported unless other arrangements are made and storage fees may apply. The analytical results associated with this COC apply only to those samples as they are received by the laboratory.												

Matrix* DW-Drinking Water, WW-Waste Water, GW-Ground Water, SW-Surface Water, SS-Soil, S-Solid, OT-Other
Preservative** 1=H₂SO₄, 2=HNO₃, 3=HCl, 4=NaOH, 5=Na₂S₂O₃, 6=None, 7=Other

Samples are discarded 30 days after results are reported unless other arrangements are made and storage fees may apply.
The analytical results associated with this COC apply only to these samples as they are received by the laboratory.
The liability of the laboratory is limited to the amount paid for the report.

Container*** P-Plastic, G-Glass, V-Voa Vial, OT-4

NDEP 283



SGS Silver State Analytical Laboratories
1135 Financial Blvd
Reno, NV 89502
(775) 857-2400
www.ssalabs.com

Definitions & Qualifiers

WO#: 25060930

Date: 7/9/2025

Definitions:

LCS: Laboratory Control Sample; prepared by adding a known mass of target analytes to a specified amount of de-ionized water and prepared with the batch of samples, used to calculate Accuracy (%REC).

LCSD: LCS Duplicate; used to calculate both Accuracy (%REC) and Precision (%RPD)

MBLK: Method Blank; a sample of similar matrix that is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

MS: Matrix Spike; prepared by adding a known mass of target analytes to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available, used to calculate Accuracy (%REC)

MSD: Matrix Spike Duplicate; used to calculate both Accuracy (%REC) and Precision (%RPD)

RPD: Relative Percent Difference; comparison between sample and duplicate and/or MS and MSD.

PQL: Practical Quantitation Limit; the limit to which data is quantitated for reporting.

MDL: Method Detection Limit; the limit to which the instrument can reliably detect.

MCL: Maximum Contaminant Level; value set according to EPA guidelines.

TNTC: Too Numerous to Count; colony density is too thick to be individually counted or greater than method reporting requirements.

Qualifiers:

* - Analyte exceeds Safe Drinking Water Act MCL, does not meet drinking water standards.

- Laboratory not accredited for this analyte.

C - Analyte value below Safe Drinking Water Act MCL, does not meet drinking water standards.

B - Analyte found above the PQL in associated method blank.

G - Calibration blank analyte detected above PQL.

H - Sample analyzed beyond holding time for this parameter.

J - Estimated Value; Analyte found between MDL and PQL limits.

L - Sample concentration is at least 5 times greater than spike contribution. Spike recovery criteria do not apply.

R - RPD between sample and duplicate sample outside the RPD acceptance limits.

S - Batch MS and/or MSD were outside acceptance limits, batch LCS was acceptable.

W - Sample temperature when received was out of limit as specified by method.

Z - Batch LCS and/or LCSD were outside acceptance limits.

APPENDIX K. ASH SAMPLING AND ANALYSIS PLAN

DRAFT

ASH SAMPLING AND ANALYSIS PLAN

Medical Waste Treatment Facility | Nye County



MediWaste Disposal LLC

Prepared By:

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April 2026

Project 0242901.0052



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1. PURPOSE

The MediWaste medical waste pyrolysis facility generates a hydrated ash from the treatment of medical waste. The objective of this Sampling and Analysis Plan (SAP) is to determine whether the ash exhibits the metal EP toxicity hazardous waste characteristics and if the ash contains dioxins and furans. This plan was developed using guidance from EPA's SW-846 Chapter 9 and the RCRA Waste Sampling Draft Technical guidance. The results of this SAP will support the determination on whether the ash waste stream is a RCRA hazardous waste. This SAP is consistent with EPA's guidance using clearly defined decision units and objectives, random composite sampling to address heterogeneity, a reasonable number of samples to address temporal variability, and document procedures and QA/QC to support reproducibility and regulatory review.

The hydrated ash is generated from the pyrolysis treatment of medical waste. At full capacity, the MediWaste facility will operate seven pyrolysis machines with the maximum processing capacity of 2,520,600 pounds of medical waste per month. The treatment process generates approximately 2-40% hydrated ash residue from the pyrolysis process, depending on the solid content in the medical waste. The facility estimates that approximately one 40-yard roll-off container per week of hydrated ash will be generated when operating at full capacity.

Due to the process inputs (medical waste), the ash is being evaluated for the RCRA toxicity characteristics for metals and dioxins and furans.

This SAP contains the following sections:

- ▶ Purpose
- ▶ RCRA and Dioxin and Furan Criteria
- ▶ Analytical Methods
- ▶ Sample Collection Methodology
- ▶ Sampling Procedures
- ▶ Quality Assurance and Quality Control
- ▶ Data Evaluation and Decision Rule
- ▶ References

2. SELECTION OF CONSTITUENTS TO BE ANALYZED

The approach provided in this SAP focuses on whether the hydrated ash produced at the MediWaste facility exhibits the metal toxicity characteristics of hazardous waste under 40 CFR Part 261 Subpart C (adopted by reference in Nevada at NAC 444.8632) and contains dioxins and furans.

2.1 RCRA Toxicity Characteristic

Per 40 CFR §261.24, a solid waste exhibits the characteristic of toxicity if, using the Toxicity Characteristic Leaching Procedure (TCLP) per EPA Test Method 1311 in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," in the EPA Publication SW-846, the extract from a representative sample of the waste contains any of the contaminants at a concentration above its regulatory level listed in Table 1 of §261.24, which includes eight RCRA metals, volatile organics, semi-volatile organics, and pesticides. Table 2-1 below summarizes these TCLP contaminants and limits for the toxicity characteristic. This SAP is only concerned with the metals listed in the table below.

Table 2-1. RCRA Toxicity Thresholds

Metals			Volatile Organic Compounds		
Contaminant	EPA HW #	Regulatory Level	Contaminant	EPA HW #	Regulatory Level
Arsenic	D004	5.0 mg/L	Benzene	D018	0.5 mg/L
Barium	D005	100.0 mg/L	Carbon tetrachloride	D019	0.5 mg/L
Cadmium	D006	1.0 mg/L	Chlorobenzene	D021	100.0 mg/L
Chromium	D007	5.0 mg/L	Chloroform	D022	6.0 mg/L
Lead	D008	5.0 mg/L	1,2-Dichloroethane	D028	0.5 mg/L
Mercury	D009	0.2 mg/L	1,1-Dichloroethylene	D029	0.7 mg/L
Selenium	D010	1.0 mg/L	Methyl ethyl ketone	D035	200.0 mg/L
Silver	D011	5.0 mg/L	Tetrachloroethylene	D039	0.7 mg/L
			Trichloroethylene	D040	0.5 mg/L
			Vinyl chloride	D043	0.2 mg/L
Pesticides			Semi-Volatile Organic Compounds		
Contaminant	EPA HW #	Regulatory Level	Contaminant	EPA HW #	Regulatory Level
Chlordane	D020	0.03 mg/L	o-Cresol	D023	200.0 mg/L
Endrin	D012	0.02 mg/L	m-Cresol	D024	200.0 mg/L
Heptachlor (and its epoxide)	D031	0.008 mg/L	p-Cresol	D025	200.0 mg/L
Lindane	D013	0.4 mg/L	Cresol	D026	200.0 mg/L
Methoxychlor	D014	10.0 mg/L	1,4-Dichlorobenzene	D027	7.5 mg/L
Toxaphene	D015	0.5 mg/L	2,4-Dinitrotoluene	D030	0.13 mg/L
			Hexachlorobenzene	D032	0.13 mg/L
			Hexachlorobutadiene	D033	0.5 mg/L
			Hexachloroethane	D034	3.0 mg/L
			Nitrobenzene	D036	2.0 mg/L
Herbicides			Pentachlorophenol	D037	100.0 mg/L
Contaminant	EPA HW #	Regulatory Level	Pyridine	D038	5.0 mg/L
2,4-D	D016	10.0 mg/L	2,4,5-Trichlorophenol	D041	400.0 mg/L
2,4,5-TP (Silvex)	D017	1.0 mg/L	2,4,6-Trichlorophenol	D042	2.0 mg/L

2.2 Dioxin and Furans

The Nevada Division of Environmental Protection, Bureau of Sustainable Materials (NDEP-BSMM) has requested that MediWaste conduct sampling of the hydrated ash to determine the presence of dioxins and furans. There is no threshold for dioxins and furans in the state or federal hazardous waste regulations, but there are no disposal facilities in Nevada that can accept waste with dioxins and furans. Per a meeting with Jeffrey Kinder, Deputy Administrator of NDEP, NDEP is undergoing internal discussions regarding dioxin and

uran testing limits. As such, a limit to compare analytical testing values against has not been provided and is not included in this sampling plan at this time. This plan will be updated when a dioxin and furan limit has been determined.

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3. ANALYTICAL METHODS

This section provides an overview of the analytical methods employed by this SAP and the criteria to be used. The full analytical methods can be found in [Appendix C](#).

3.1 EPA Method 1311: Toxicity Characteristic Leaching Procedure (TCLP)

The TCLP method is used for the determination of the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes and is meant to mimic the leachate in a landfill. The TCLP is a standard EPA method employed to determine the toxicity characteristic of waste.

EPA Method 1311 states that liquid wastes (wastes with less than 0.5% dry solids) the TCLP extract is what is left after filtering through a 0.6 to 0.8 μm glass fiber filter. If the waste is a solid, the solid phase is extracted with extraction fluid equal to 20 times the weight of the solid phase of the waste. An extractor vessel is used to test for volatile analytes. For a material with multiple phases, the liquid phase of the waste and the liquid extract are combined and they are analyzed together for TCLP.

If total analysis demonstrates the individual analytes are not present or are below 20 times their respective regulatory levels (i.e., the rule of 20), TCLP testing does not need to be completed. If the regulatory thresholds shown in Table 2-1 are exceeded for any of the RCRA metals, then the sample is a hazardous waste.

3.2 EPA Test Method 8290A: Dioxins and Furans

EPA Method 8290A is used for highly sensitive detection of dioxins (PCDDs) and furans (PCDFs) using High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS). The method allows for dioxin and furan detection down to the part-per-trillion (ppt) to parts-per-quadrillion (ppq) concentrations.

EPA Method 8290A uses matrix-specific extraction, analyte-specific cleanup, and HRGC/HRMS analytical methods. A designated amount (per Table 1 of the Method) of the waste is spiked with a solution contain specified amounts of each of the nine isotopically labeled dioxins and furans listed in Table 2 of the Method. The sample is then extracted according to a matrix-specific extraction procedure. Aqueous samples that contain one percent or more solids and solid samples that show an aqueous phase are filtered, the solid phase (including the filter) and the aqueous phase extracted separately, and the extracts combined before extract cleanup.

The extracts undergo an acid-base washing treatment and are dried. The final extract for HRGC/HRMS analysis is prepared by adding a nonane solution containing the recovery standards. The concentrated extract is then injected into the HRGC/HRMS system for analysis.

4. SAMPLE COLLECTION METHODOLOGY

4.1 Population Definition

The ash waste is a solid or semi-solid depending on water content. There are a maximum of seven pyrolysis machines that discharge into separate collection bins that are all accumulated together in one roll-off container prior to disposal. The facility estimates they will generate an average of one roll-off container per week during maximum operation (i.e., all seven machines are operating). One week of ash production is assumed to represent typical operations for the facility that week. Ash from the seven machines is expected to be similar in composition and reasonably well mixed in the roll-off container; any variability will be managed through simplified random composite sampling. MediWaste believes composite sampling is appropriate to characterize the ash waste stream since the ash stream from each machine will be collected in one roll-off container, and a composite sample would accurately reflect the composition of the entire roll-off container. Despite variation in the medical waste accepted at the facility, the pyrolysis process results in a generally homogeneous ash stream.

4.2 Design Overview

This SAP will utilize simplified random composite sampling within each roll-off container. The facility will replicate weekly composites over time to address the potential variation over time.

4.3 Sampling Size and Frequency

For each full roll-off container, the facility will collect at least 14 increments per roll-off container which will be collected from different locations/depths within the container using a random pattern. All 14 increments will be combined into one sample to represent the roll-off container. Fourteen (14) increments were chosen such that there would be two samples per pyrolysis machine (following EPA's guidance that there must be at least two samples to validate results). The sample will be thoroughly homogenized into one composite sample to be submitted to the lab for analysis. This follows the SW-846 principles to obtain a representative sample of a heterogeneous solid waste using multiple increments and compositing. A representative sample is defined as exhibiting average properties of the whole waste stream.

The facility will sample four consecutive roll-off containers to estimate the variability and central tendency of the ash waste stream ($n=4$). This scheme aligns with EPA's emphasis on balancing decision error rates, variability, and resource constraints. If all four composite samples are below the hazardous waste TCLP limits for metals and operations are stable, the facility will rely on this initial dataset to complete waste determinations on the ash moving forward. If any single composite sample is above the hazardous waste TCLP limits for metals, then that roll-off will be managed as RCRA hazardous waste. The next roll-off for sampling will restart the four consecutive roll-offs sampling plan. The decision to sample four consecutive samples was modeled after the EPA's sampling requirements to de-list a hazardous waste. EPA requires that a waste stream subject to a de-listing petition have at least four samples to represent temporal variability of waste.

Quarterly confirmation sampling will be required for the first five years of operation. MediWaste determined four consecutive samples would be sufficient to determine the toxicity of the hydrated ash based on previous sample results from a similar medical waste pyrolysis facility that show RCRA toxicity results either as non-detect or two to four orders of magnitude below the hazardous thresholds for RCRA metals.

5. SAMPLING PROCEDURES

5.1 Field Increment Collection

Samples will be collected when the roll-off container is filled. The roll-offs will be covered unless adding or removing waste. A clean scoop, auger, or sampling thief suitable for ash will be required. The roll-off container will be divided into zones (top/middle/bottom; quadrants) and 14 locations for sampling will be randomly selected across the container. An equal sample will be collected at each location. All increments will be placed into a clean composite container.

5.2 Composite Preparation

The composite sample will be homogenized thoroughly. Sample will then be subsampled into lab containers following SW-846 guidance for solids. Each container will be labeled with a unique sample ID, date, time, and name of sampler.

5.3 Sampling Documentation

A sample identification label that identifies the sample number, date and time of sampling, matrix, and initials of sampling personnel will be completed and affixed to each sample container. An example of a sample label is provided in Figure 2-1. The sample will be sealed in a resealable plastic bag and stored in a cooler with ice or frozen reusable ice packs prior to lab delivery.

A Sampling Log will be completed for each sample collected. Information regarding the sampling will be recorded on the Sampling Log and will include the following:

- ▶ Date
- ▶ Weather conditions
- ▶ Unique sample identification number
- ▶ Location of sample collection including QA/QC blanks
- ▶ Times of sample collection
- ▶ Documentation of all sample collection method and equipment
- ▶ Description of material sampled
- ▶ Laboratory analyses to be performed on sample
- ▶ Names of sampling personnel
- ▶ Odd or unusual occurrences

6. QUALITY ASSURANCE AND QUALITY CONTROL

For field quality control, at least one field duplicate composite will be collected during the 4-roll off container sampling campaign to assess sampling variability. To ensure lab QAQC, SW-846 method QC requirements, including blanks, LCS, spikes, etc., will be followed. The facility will document sample IDs, sampling times, sampling personnel, and transfers using a chain of custody. Upon receipt of analytical results, the facility will verify the holding times, QC results, and method performance prior to using the data to characterize the ash waste.

6.1 Sample Preservation and Storage

A summary of the sample handling procedures, including types of containers and preservatives required for each analysis, is provided in Table 2-1. All ash samples will be stored in a cooler with ice or frozen reusable ice packs immediately after collection. The cooler of filled sample containers will be transported by courier or hand-delivered to the laboratory for analysis.

6.2 Chain of Custody Procedures

Samples will remain in the custody of the sampling personnel during each sampling event. At the end of each sampling event and prior to the transfer of the samples to the courier, chain-of-custody (COC) entries will be made for all samples using a COC record (Figure 2-2). One COC form will be completed for each cooler of samples. All information on the COC form and the sample container labels will be checked against the sampling log entries, and samples will be recounted before transferring custody. Upon transfer of custody to the courier or to the laboratory, the COC form will be signed by a member of the MediWaste team, sealed in plastic, and taped to the inside lid of the cooler. All COC forms received by the laboratory must be signed and dated by the laboratory's sample custodian.

A signed, dated custody seal (Figure 2-3) will be placed over the lid opening of the sample cooler to indicate if the cooler is opened during shipment.

The laboratory will note the condition of each sample received as well as questions or observations concerning sample integrity. If the integrity of the custody seal is damaged or broken, the laboratory staff receiving the container must indicate on the chain of custody that the custody seal was damaged or broken. The samples in the cooler with the damaged or broken custody seal will be analyzed and the results flagged. The sample custodian will also maintain a sample-tracking record that follows each sample through all stages of laboratory processing. The sample tracking records must show the date of sample extraction, if extraction was performed, and sample analysis. These records will be used to determine compliance with holding time limits during laboratory audits and data validation.

6.3 Lab Quality Control

All samples will be analyzed by a Nevada Certified laboratory. The quality of analytical data generated is controlled by the frequency and type of internal QC checks developed for each analysis type. Internal laboratory QC checks will include the following:

- ▶ Method Blanks of laboratory reagent-grade water treated in the same manner as the sample (i.e., digested, extracted, distilled, etc.) then analyzed and reported as a standard sample. These blank

samples are prepared in the laboratory and are analyzed to assess possible laboratory contamination. Matrix spikes consist of an aliquot of a field sample that is fortified (spiked) with the analytes of interest and analyzed with an associated sample batch to monitor the effects of the field sample matrix (matrix effects) on the analytical method. Surrogates consist of spiking the sample, method blank, blank spike and duplicate; and matrix spike and duplicate with the (preferably deuterated) analytes of interest to monitor method performance and possible bias from matrix effects. Accuracy of the laboratory analyses is determined by the recoveries of surrogates and the matrix spike compound.

- Laboratory Control Standard for Inorganics is a standard solution with a certified concentration that is analyzed as a sample and is used to monitor analytical accuracy.

An example of the laboratory internal QC forms will be available once the laboratory contractor(s) have been selected to perform analytical work associated with closure activities. The following guidance will be used for data validation as applicable to the individual methods used: Laboratory Data Validation Functional Guidelines for Evaluating Organic Analysis, (1994) and Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis, (1994); prepared by USEPA Data Review Work Group.

7. DATA EVALUATION AND DECISION RULE

The facility will compare the analytical results from each composite sample to the RCRA toxic characteristics thresholds for metals listed in Table 2-1. As noted above in Section 2.2, NDEP has not provided a limit for dioxins and furans at this time, and therefore there is not a value to compare analytical results against. If all four container composite samples are below the applicable thresholds and QC is acceptable, the ash is not a RCRA hazardous waste based on the data set and current operating conditions. If any composite sample exceeds a threshold, then that roll-off of ash is considered a RCRA hazardous waste unless further investigation and resampling (with a revised design) demonstrate otherwise.

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8. REFERENCES

USEPA, 1986. SW-846 Compendium, Chapter 9: Sampling Plan. U.S. Environmental Protection Agency. September 1986.

USEPA, 2002. RCRA Waste Sampling Draft Technical Guidance; Planning, Implementation, and Assessment. Office of Solid Waste, U.S. Environmental Protection Agency. August 2002.

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Table A-1. Summary of Sampling and Analysis Requirements

Analyte	EPA Method	Container*	Preservative	Holding Time
TCLP Metals	6010C	4 oz, P or G	Cool 4 C	180 days
Mercury	7471B	4 oz, P or G	Cool 4 C	28 days
Dioxins and Furans	8290A	4 oz, G	Cool 6 C	30 days

* Volume listed is minimum
volume

C Degrees Celsius

P Plastic

G Glass

oz Ounce

Reference: Hazardous Waste Test Methods / SW-846

Table A-2. Quality Assurance/Quality Control Samples

Control Sample	Frequency, Criteria, Corrective Action
Method Blank	Laboratory contamination check: 1 per 12-hour period. Criterion: no target compounds in blank above detection limit except common solvents, which can be 5 times detection limit. Corrective action: reanalyze.
Instrument Blank	Contamination check: at beginning of run.
Laboratory Control Sample	Method accuracy: 1 per sample set. Criteria: Selected laboratory will have its own internal criteria for acceptability, however, laboratory control sample are typically acceptable if they are below a 20% relative percent difference. Corrective action: check standards, recalibrate if necessary.
Continuing Calibration	Instrument performance: 1 per 12-hour period. System performance check compounds Criteria: same as initial calibration. Continuous calibration check: percent difference must be < 20%. Corrective action: prepare and analyze another standard; if they still fail, recalibrate instrument.
Internal Standards	Quantitation: added to all samples and blanks. Criterion: must not vary more than a factor of 2 from the continuing calibration standard. Corrective action: reanalyze, report reanalysis; if it fails, report original with comment.
Surrogate Spikes	Performance check on individual samples, added to all samples and blanks. Criterion: Selected laboratory will have its own internal criteria for acceptability; however, surrogates are typically acceptable in the 70-130% recovery range. Corrective action: if any surrogates are out low, re-prep and reanalyze. If any surrogates are out high and sample is below detection limit, report sample. If surrogates are out high and sample is not below detection limit, re-prep and reanalyze.
Matrix Spike/Matrix Spike Duplicate	Accuracy/Precision: 1 per 20 samples of similar matrix. Criterion: Selected laboratory will have its own internal criteria for acceptability; however, matrix spike/matrix spike duplicates are typically acceptable in the 70-130% recovery range. Corrective action: if any analytes fail analysis of the extracted Laboratory Control Sample is required. If LCS fails, reanalysis of the run is required.

Control Sample	Frequency, Criteria, Corrective Action
Instrument Performance Check	GC/MS for mass resolution. Identification: 1 per 12-hour period. Criterion: must meet criteria specified by the method and corresponding laboratory SOP. Corrective action: continue tuning to meet ion profile.

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APPENDIX B. FIGURES

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Figure B-1. Example Sample Label

ESS

ENVIRONMENTAL SAMPLING SUPPLY

LOT #	
SAMPLE	
ID	
SAMPLED BY	DATE
	TIME
LOCATION	PRESERVATIVE
ANALYSIS	CLIENT

Figure B-2. Example Chain of Custody Form

SEND REPORT TO:

Project Number _____

Project Name _____

Project Manager _____

Project Phone _____

Project Fax _____

Req'd Report Date _____

Lab Contact _____

Lab Phone _____

Sample ID #	TYPE	CONTAINER	VOLUME	PRESERVATIVE	ANALYSIS REQ'D	DATE	NOTES	LAB ID #
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								

Sample TAT Req'd: _____ Notes: _____

SAMPLE CHARACTERISTICS

Flammable ☐
 Corrosive ☐
 Hazardous ☐
 Radioactive ☐
 Gas ☐
 Solid ☐
 Liquid ☐
 Sludge ☐
 BipPhase ☐
 TriPhase ☐
 Sp. Grav. _____
 Color _____
 Flash Pt. _____
 Odor _____

Initial: _____

CUSTODY TRACKING

1) Relinquished By: _____ Date: _____ Time: _____ Received By: _____ Date: _____ Time: _____

2) Relinquished By: _____ Date: _____ Time: _____ Received By: _____ Date: _____ Time: _____

3) Relinquished By: _____ Date: _____ Time: _____ Received By: _____ Date: _____ Time: _____

Figure B-3. Example Chain of Custody Seal

CUSTODY SEAL	
Person Collecting Sample _____ (signature)	Sample No. _____
Date Collected _____	Time Collected _____

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APPENDIX C. ANALYTICAL METHODS

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METHOD 1311

TOXICITY CHARACTERISTIC LEACHING PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (*i.e.*, those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

2.3 If compatible (*i.e.*, multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE). This device is for use only when the waste is being tested for the mobility of volatile analytes (*i.e.*, those listed in Table 1). The ZHE (depicted in Figure 2) allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (see Section 4.3.1). The vessels shall have an internal volume of 500-600 mL, and be equipped to accommodate a 90-110 mm filter. The devices contain VITON[®] O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in Table 3.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Section 7.3) refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

¹ VITON[®] is a trademark of Du Pont.

4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste (see Section 4.3.3). It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Section 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel described in Section 4.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the waste is evaluated for other than volatile analytes, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered (see Section 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended). Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration. Suitable filter holders known to EPA are shown in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals. Borosili-

cate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 μm , or equivalent. Filters known to EPA which meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: The meter should be accurate to ± 0.05 units at 25 °C.

4.6 ZHE Extract Collection Devices: TEDLAR^{®2} bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1% of total waste), the TEDLAR[®] bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1% of total waste), the syringe or the TEDLAR[®] bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR[®] bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit (see Section 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Any laboratory balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, 500 mL.

² TEDLAR[®] is a registered trademark of Du Pont.

4.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

4.11 Magnetic stirrer.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent Water. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.2.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.2.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 90 ± 5 degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

5.4 Nitric acid (1N), HNO₃, made from ACS reagent grade.

5.5 Sodium hydroxide (1N), NaOH, made from ACS reagent grade.

5.6 Glacial acetic acid, CH₃CH₂OOH, ACS reagent grade.

5.7 Extraction fluid.

5.7.1 Extraction fluid # 1: Add 5.7 mL glacial CH₃CH₂OOH to 500 mL of reagent water (See Section 5.2), add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05 .

5.7.2 Extraction fluid # 2: Dilute 5.7 mL glacial $\text{CH}_3\text{CH}_2\text{OOH}$ with reagent water (See Section 5.2) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05 .

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.8 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction (see Section 1.4 concerning the use of this extract for volatile organics). If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4 °C. Samples should be opened only immediately prior to extraction).

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Section 8.0 (QA requirements) for acceptable sample and extract holding times.

7.0 PROCEDURE

7.1 Preliminary Evaluations

Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids (Section 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 7.1.2); (3) determination of whether the solid portion of the waste requires particle size reduction (Section 7.1.3); and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 7.1.4).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Section 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.1.1.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (*i.e.*, filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Section 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Section 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Section 7.1.1.9)}}{\text{Total weight of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2 If the percent solids determined in Section 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Section 7.1.3 to

determine whether the solid material requires particle size reduction or to Section 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Section 7.1.1.9 is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed and to Section 7.3 with a fresh portion of the waste if the volatile TCLP is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at 100 ± 20 °C until two successive weighing yield the same value within $\pm 1\%$. Record the final weight.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{(\text{Wt. of dry waste + filter}) - \text{tared wt. of filter}}{\text{Initial wt. of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed, and to Section 7.3 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed, return to the beginning of this Section (7.1) and, with a fresh portion of waste, determine whether particle size reduction is necessary (Section 7.1.3) and determine the appropriate extraction fluid (Section 7.1.4). If only the volatile TCLP is to be performed, see the note in Section 7.1.4.

7.1.3 Determination of whether the waste requires particle size reduction (particle size is reduced during this step): Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2 , or is smaller than 1 cm in its narrowest dimension (*i.e.*, is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Section 7.3.6).

NOTE: Surface area criteria are meant for filamentous (*e.g.*, paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet

the criteria, sample specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents (Section 7.2), determine the appropriate fluid (Section 5.7) for the nonvolatiles extraction as follows:

NOTE: TCLP extraction for volatile constituents uses only extraction fluid #1 (Section 5.7.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 7.3.

7.1.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 7.2.

7.1.4.3 If the pH from Section 7.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50 °C, and hold at 50 °C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 7.2.

7.1.5 If the aliquot of the waste used for the preliminary evaluation (Sections 7.1.1 - 7.1.4) was determined to be 100% solid at Section 7.1.1.1, then it can be used for the Section 7.2 extraction (assuming at least 100 grams remain), and the Section 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Section 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 7.3. The aliquot of the waste subjected to the procedure in Section 7.1.1.7 might be appropriate for use for the Section 7.2 extraction if an adequate amount of solid (as determined by Section 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Section 7.2.10 of the nonvolatile TCLP extraction.

7.2 Procedure When Volatiles are not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the

solids content of the waste sample (percent solids, See Section 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see Section 7.1.1), weigh out a subsample of the waste (100 gram minimum) and proceed to Section 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 4.4).

NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids (Section 7.1.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids (Sections 7.1.1 or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Section 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at

room temperature, then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.2.5, to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (*i.e.*, filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (See Section 7.2.12) or stored at 4 °C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the waste contains <0.5% dry solids (see Section 7.1.2), proceed to Section 7.2.13. If the waste contains >0.5% dry solids (see Section 7.1.1 or 7.1.2), and if particle size reduction of the solid was needed in Section 7.1.3, proceed to Section 7.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 7.2.11.

7.2.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid

material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Section 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 ± 2 °C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Section 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the TCLP extract as follows:

7.2.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.2.12 is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.2.12 with the initial liquid phase of the waste obtained in Section 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Section 7.2.7, is not or may not be compatible with the filtered liquid resulting from Section 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.2.14.

7.2.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4 °C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to $\pm 0.5\%$), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).

C_1 = The concentration of the analyte of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

7.3 Procedure When Volatiles are Involved

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of nonvolatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a

sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4 °C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (See Section 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Section 4.6 are recommended for use under the conditions stated in Sections 4.6.1 - 4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 7.3, Section 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the waste is 100% solid (see Section 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Section 7.3.5.

7.3.4 If the waste contains < 0.5% dry solids (Section 7.1.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing $\geq$ 0.5% dry solids (Sections 7.1.1 and/or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For wastes containing < 5% solids (see Section 7.1.1), weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing $\geq$ 5% solids (see Section 7.1.1), determine the amount of waste to charge into the ZHE as follows:

Weight of waste to charge ZHE = $\frac{\text{25}}{\text{percent solids (Section 7.1.1)}} \times 100$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle size reduction of the solid portion of the waste was required in Section 7.1.3, proceed to Section 7.3.6. If particle size reduction was not required in Section 7.1.3, proceed to Section 7.3.7.

7.3.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 7.1.3. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4 °C prior to particle size reduction. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to Section 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace

slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 7.1.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Section 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% dry solids (see Section 7.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Section 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately (See Sections 7.3.13 through 7.3.15) or stored at 4 °C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.3.4 or 7.3.8)}}{100}$$

7.3.12 The following Sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (See Section 5.7).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (*i.e.*, temperature of room in which extraction occurs) shall be maintained at 23 ± 2 °C during agitation.

7.3.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (*i.e.*, no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (*i.e.*, TEDLAR[®] bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 7.3.9. All extract shall be filtered and collected if the

TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Sections 4.6 and 7.3.1).

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

7.3.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.3.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Section 7.3.13 and the initial liquid phase (Section 7.3.9) are collectively defined as the TCLP extract.

7.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4 °C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (*i.e.*, are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1) (C_1) + (V_2) (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).

C_1 = The concentration of the analyte of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

8.0 QUALITY ASSURANCE

8.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

8.2 A matrix spike shall be performed for each waste type (*e.g.*, wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.2.2 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R (\% \text{Recovery}) = 100 (X_s - X_u)/K$$

where:

X_s = measured value for the spiked sample,

X_u = measured value for the unspiked sample, and

K = known value of the spike in the sample.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1. The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

8.4.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The fourth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and

150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

8.4.4 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependant variable versus the independent variable. Derive concentrations for unknowns using the internal calibration curve as if it were an external calibration curve.

8.5 Samples must undergo TCLP extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES [DAYS]				
	From: Field collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

9.0 METHOD PERFORMANCE

9.1 Ruggedness. Two ruggedness studies have been performed to determine the effect of various perturbations on specific elements of the TCLP protocol. Ruggedness testing determines the sensitivity of small procedural variations which might be expected to occur during routine laboratory application.

9.1.1 Metals - The following conditions were used when leaching a waste for metals analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Extraction time	16 hours vs. 18 hours
Headspace	20% vs. 60%
Buffer #2 acidity	190 meq vs. 210 meq
Acid-washed filters	yes vs. no
Filter type	0.7 μ m glass fiber vs. 0.45 μ m vs. polycarbonate
Bottle type	borosilicate vs. flint glass

Of the seven method variations examined, acidity of the extraction fluid had the greatest impact on the results. Four of 13 metals from an API separator sludge/electroplating waste (API/EW) mixture and two of three metals from an ammonia lime still bottom waste were extracted at higher levels by the more acidic buffer. Because of the sensitivity to pH changes, the method requires that the extraction fluids be prepared so that the final pH is within ± 0.05 units as specified.

9.1.2 Volatile Organic Compounds - The following conditions were used when leaching a waste for VOC analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Headspace	0% vs. 5%
Buffer #1 acidity	60 meq vs. 80 meq
Method of storing extract	Syringe vs. Tedlar [®] bag
Aliquotting	yes vs. no
Pressure behind piston	0 psi vs. 20 psi

None of the parameters had a significant effect on the results of the ruggedness test.

9.2 Precision. Many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP toxicity test and that method precision is adequate. One of the more significant contributions to poor precision appears to be related to sample homogeneity and inter-laboratory variation (due to the nature of waste materials).

9.2.1 Metals - The results of a multi-laboratory study are shown in Table 6, and indicate that a single analysis of a waste may not be adequate for waste characterization and identification requirements.

9.2.2 Semi-Volatile Organic Compounds - The results of two studies are shown in Tables 7 and 8. Single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual compounds in the multi-laboratory study fell in the RSD range of 20 - 120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not present problems (*i.e.*, column degradation of the gas chromatograph) for the analytical conditions used.

9.2.3 Volatile Organic Compounds - Eleven laboratories participated in a collaborative study of the use of the ZHE with two waste types which were fortified with a mixture of VOCs. The results of the collaborative study are shown in Table 9. Precision results for VOCs tend to occur over a considerable range. However, the range and mean RSD compared very closely to the same collaborative study metals results in Table 6. Blackburn and Show concluded that at the 95% level of significance: 1) recoveries among laboratories were statistically similar, 2) recoveries did not vary significantly between the two sample types, and 3) each laboratory showed the same pattern of recovery for each of the two samples.

10.0 REFERENCES

1. Blackburn, W.B. and Show, I. "Collaborative Study of the Toxicity Characteristics Leaching Procedure (TCLP)." Draft Final Report, Contract No. 68-03-1958, S-Cubed, November 1986.
2. Newcomer, L.R., Blackburn, W.B., Kimmell, T.A. "Performance of the Toxicity Characteristic Leaching Procedure." Wilson Laboratories, S-Cubed, U.S. EPA, December 1986.
3. Williams, L.R., Francis, C.W.; Maskarinec, M.P., Taylor D.R., and Rothman, N. "Single-Laboratory Evaluation of Mobility Procedure for Solid Waste." EMSL, ORNL, S-Cubed, ENSECO.

Table 1.
Volatile Analytes^{1,2}

Compound	CAS No.
Acetone	67-64-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1,-Trichloroethane	71-55-6
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

¹ When testing for any or all of these analytes, the zero-headspace extractor vessel shall be used instead of the bottle extractor.

² Benzene, carbon tetrachloride, chlorobenzene, chloroform, 1,2-dichloroethane, 1,1-dichloroethylene, methyl ethyl ketone, tetrachloroethylene, and vinyl chloride are toxicity characteristic constituents.

Table 2.
Suitable Rotary Agitation Apparatus¹

Company	Location	Model No.
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel extractor (DC20S) 8-vessel extractor (DC20) 12-vessel extractor (DC20B) 24-vessel extractor (DC24C)
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	2-vessel (3740-2-BRE) 4-vessel (3740-4-BRE) 6-vessel (3740-6-BRE) 8-vessel (3740-8-BRE) 12-vessel (3740-12-BRE) 24-vessel (3740-24-BRE)
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	8-vessel (08-00-00) 4-vessel (04-00-00)
IRA Machine Shop and Laboratory	Santurce, PR (809) 752-4004	8-vessel (011001)
Lars Lande Manufacturing	Whitmore Lake, MI (313) 449-4116	10-vessel (10VRE) 5-vessel (5VRE) 6-vessel (6VRE)
Millipore Corp.	Bedford, MA (800) 225-3384	4-ZHE or 4 2-liter bottle extractor (YT310RAHW)

¹ Any device that rotates the extraction vessel in an end-over-end fashion at 30 ± 2 rpm is acceptable.

Table 3.
Suitable Zero-Headspace Extractor Vessels¹

Company	Location	Model No.
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	3745-ZHE, Gas Pressure Device
Lars Lande Manufacturing ²	Whitmore Lake, MI (313) 449-4116	ZHE-11, Gas Pressure Device
Millipore Corporation	Bedford, MA (800) 225-3384	YT30090HW, Gas Pressure Device
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	VOLA-TOX1, Gas Pressure Device
Gelman Science	Ann Arbor, MI (800) 521-1520	15400 Gas Pressure Device

¹ Any device that meets the specifications listed in Section 4.2.1 of the method is suitable.

² This device uses a 110 mm filter.

Table 4.
Suitable Filter Holders¹

Company	Location	Model/ Catalogue No.	Size
Nucleopore Corporation	Pleasanton, CA (800) 882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	302400 311400	142 mm 47 mm
Millipore Corporation	Bedford, MA (800) 225-3384	YT30142HW XX1004700	142 mm 47 mm

¹ Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic analytes are of concern. The 142 mm size filter holder is recommended.

Table 5.
Suitable Filter Media¹

Company	Location	Model	Pore Size (µm)
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	GF75	0.7
Gelman Science	Ann Arbor, MI (800) 521-1520	66256 (90mm) 66257 (142mm)	0.7

¹ Any filter that meets the specifications in Section 4.4 of the Method is suitable.

Table 6. Multi-Laboratory TCLP Metals, Precision

Waste	Extraction Fluid	Metal	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms	#1	Cadmium	0.053	0.031	60
	#2		0.023	0.017	76
	#1	Chromium	0.015	0.0014	93
	#2		0.0032	0.0037	118
	#1	Lead	0.0030	0.0027	90
	#2		0.0032	0.0028	87
API/EW Mixture	#1	Cadmium	0.0046	0.0028	61
	#2		0.0005	0.0004	77
	#1	Chromium	0.0561	0.0227	40
	#2		0.105	0.018	17
	#1	Lead	0.0031	0.0031	100
	#2		0.0124	0.0136	110
Fossil Fuel Fly Ash	#1	Cadmium	0.080	0.069	86
	#2		0.093	0.067	72
	#1	Chromium	0.017	0.014	85
	#2		0.070	0.040	57
	#1	Lead	0.0087	0.0074	85
	#2		0.0457	0.0083	18
					%RSD Range = 17 - 118
					Mean %RSD = 74

NOTE: $\bar{X}$ = Mean results from 6 - 12 different laboratories
Units = mg/L
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 7. Single-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms	Phenol	#1	19000	2230	11.6
		#2	19400	929	4.8
	2-Methylphenol	#1	2000	297	14.9
		#2	1860	52.9	2.8
	4-Methylphenol	#1	7940	1380	17.4
		#2	7490	200	2.7
	2,4-Dimethylphenol	#1	321	46.8	14.6
		#2	307	45.8	14.9
	Naphthalene	#1	3920	413	10.5
		#2	3827	176	4.6
	2-Methylnaphthalene	#1	290	44.8	15.5
		#2	273	19.3	7.1
	Dibenzofuran	#1	187	22.7	12.1
		#2	187	7.2	3.9
	Acenaphthylene	#1	703	89.2	12.7
		#2	663	20.1	3.0
	Fluorene	#1	151	17.6	11.7
		#2	156	2.1	1.3
	Phenanthrene	#1	241	22.7	9.4
		#2	243	7.9	3.3
	Anthracene	#1	33.2	6.19	18.6
		#2	34.6	1.55	4.5
	Fluoranthrene	#1	25.3	1.8	7.1
		#2	26.0	1.8	7.1
API/EW Mixture	Phenol	#1	40.7	13.5	33.0
		#2	19.0	1.76	9.3
	2,4-Dimethylphenol	#1	33.0	9.35	28.3
		#2	43.3	8.61	19.9
	Naphthalene	#1	185	29.4	15.8
		#2	165	24.8	15.0
	2-Methylnaphthalene	#1	265	61.2	23.1
		#2	200	18.9	9.5
%RSD Range = 1 - 33					
Mean %RSD = 12					

NOTE: Units = $\mu\text{g/L}$

Extractions were performed in triplicate

All results were at least 2x the detection limit

Extraction Fluid #1 = pH 4.9

#2 = pH 2.9

Table 8. Multi-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime	BNAs	#1	10043	7680	76.5
Still Bottoms (A)		#2	10376	6552	63.1
API/EW	BNAs	#1	1624	675	41.6
Mixture (B)		#2	2074	1463	70.5
Fossil Fuel	BNAs	#1	750	175	23.4
Fly Ash (C)		#2	739	342	46.3
					Mean %RSD = 54

NOTE: Units = $\mu\text{g/L}$

$\bar{X}$ = Mean results from 3 - 10 labs

Extraction Fluid #1 = pH 4.9

#2 = pH 2.9

%RSD Range for Individual Compounds

A, #1 0 - 113

A, #2 28 - 108

B, #1 20 - 156

B, #2 49 - 128

C, #1 36 - 143

C, #2 61 - 164

Table 9. Multi-Laboratory (11 Labs) VOCs, Precision

Waste	Compound	$\bar{X}$	S	%RSD
Mine Tailings	Vinyl chloride	6.36	6.36	100
	Methylene chloride	12.1	11.8	98
	Carbon disulfide	5.57	2.83	51
	1,1-Dichloroethene	21.9	27.7	127
	1,1-Dichloroethane	31.4	25.4	81
	Chloroform	46.6	29.2	63
	1,2-Dichloroethane	47.8	33.6	70
	2-Butanone	43.5	36.9	85
	1,1,1-Trichloroethane	20.9	20.9	100
	Carbon tetrachloride	12.0	8.2	68
	Trichloroethene	24.7	21.2	86
	1,1,2-Trichloroethene	19.6	10.9	56
	Benzene	37.9	28.7	76
	1,1,2,2-Tetrachloroethane	34.9	25.6	73
	Toluene	29.3	11.2	38
	Chlorobenzene	35.6	19.3	54
	Ethylbenzene	4.27	2.80	66
	Trichlorofluoromethane	3.82	4.40	115
	Acrylonitrile	76.7	110.8	144
Ammonia Lime Still Bottoms	Vinyl chloride	5.00	4.71	94
	Methylene chloride	14.3	13.1	92
	Carbon disulfide	3.37	2.07	61
	1,1-Dichloroethene	52.1	38.8	75
	1,1-Dichloroethane	52.8	25.6	49
	Chloroform	64.7	28.4	44
	1,2-Dichloroethane	43.1	31.5	73
	2-Butanone	59.0	39.6	67
	1,1,1-Trichloroethane	53.6	40.9	76
	Carbon tetrachloride	7.10	6.1	86
	Trichloroethene	57.3	34.2	60
	1,1,2-Trichloroethene	6.7	4.7	70
	Benzene	61.3	26.8	44
	1,1,2,2-Tetrachloroethane	3.16	2.1	66
	Toluene	69.0	18.5	27
	Chlorobenzene	71.8	12.0	17
	Ethylbenzene	3.70	2.2	58
	Trichlorofluoromethane	4.05	4.8	119
	Acrylonitrile	29.4	34.8	118
%RSD Range = 17 - 144				
Mean %RSD = 75				

NOTE: Units = $\mu\text{g/L}$

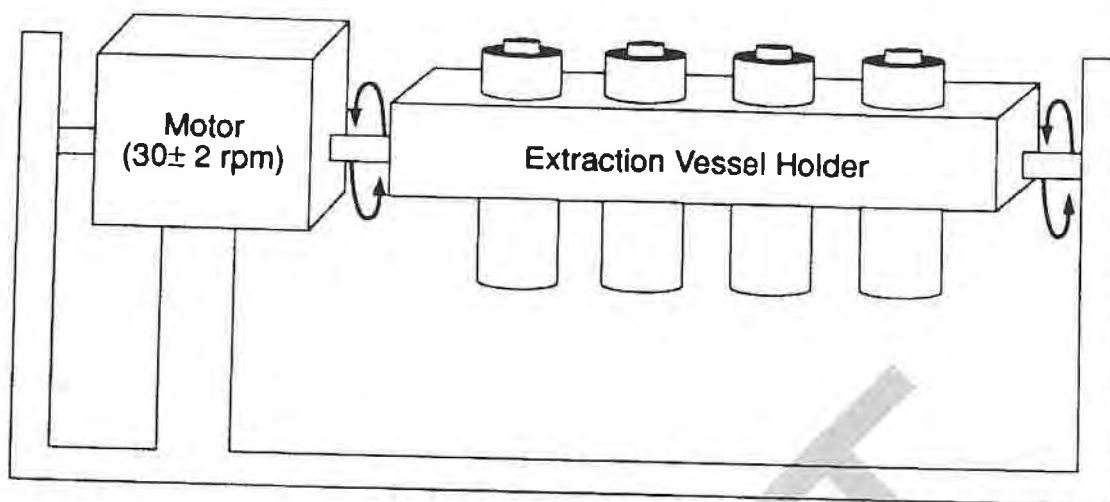


Figure 1. Rotary Agitation Apparatus

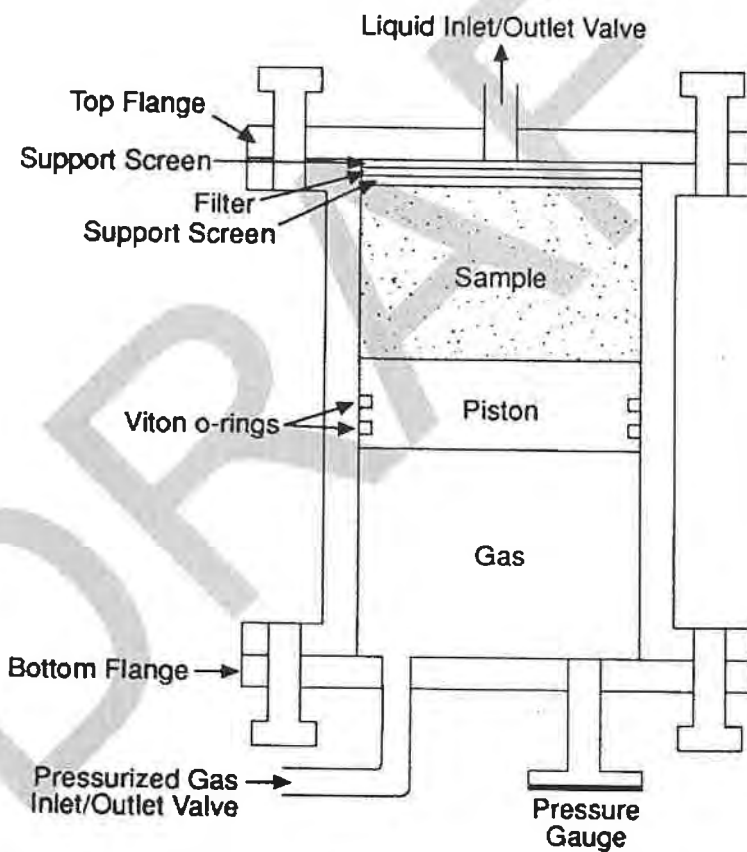
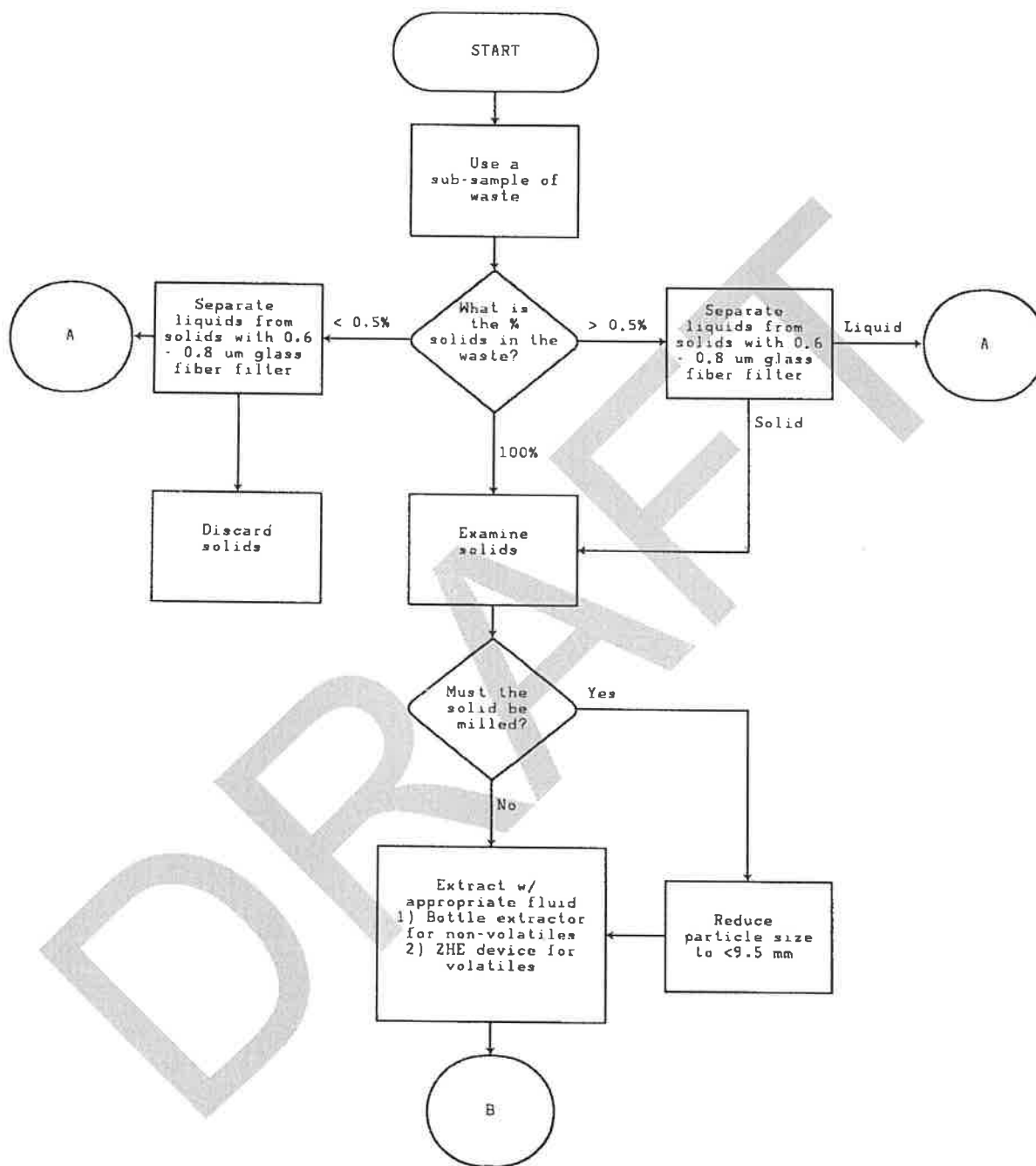


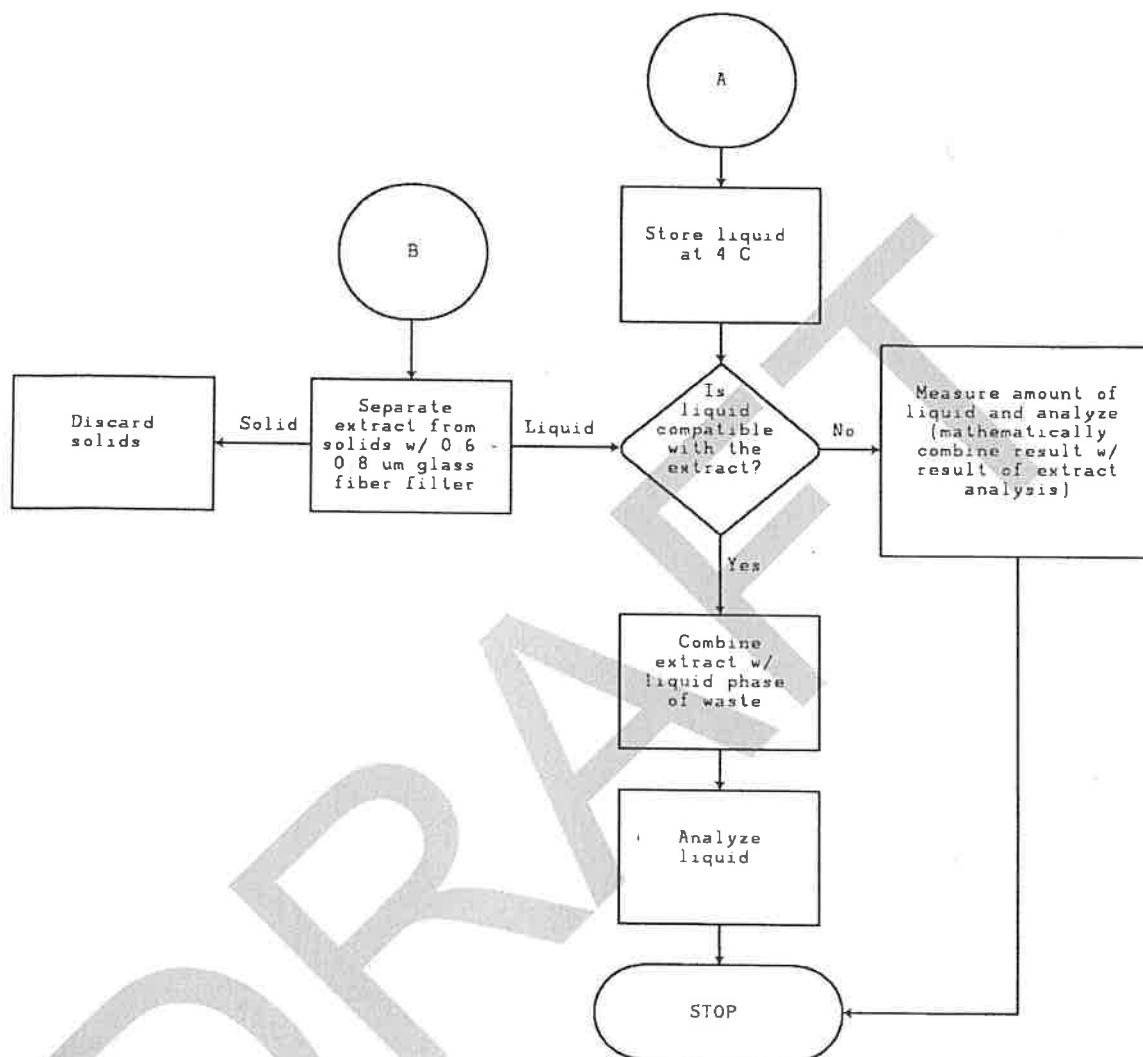
Figure 2. Zero-Headspace Extractor (ZHE)

METHOD 1311

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



METHOD 1311 (CONTINUED)
TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



METHOD 8290A

POLYCHLORINATED DIBENZO-*p*-DIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs) BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH- RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method provides procedures for the detection and quantitative measurement of polychlorinated dibenzo-*p*-dioxins (tetra- through octachlorinated homologues; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologues; PCDFs) in a variety of environmental matrices and at part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations. The following compounds have been determined by this method:

Compound	CAS Registry No. ^a
2,3,7,8-Tetrachlorodibenzo- <i>p</i> -dioxin (TCDD)	1746-01-6
1,2,3,7,8-Pentachlorodibenzo- <i>p</i> -dioxin (PeCDD)	40321-76-4
1,2,3,4,7,8-Hexachlorodibenzo- <i>p</i> -dioxin (HxCDD)	39227-28-6
1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin (HxCDD)	57653-85-7
1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin (HxCDD)	19408-74-3
1,2,3,4,6,7,8-Heptachlorodibenzo- <i>p</i> -dioxin (HpCDD)	35822-46-9
1,2,3,4,5,6,7,8-Octachlorodibenzo- <i>p</i> -dioxin (OCDD)	3268-87-9
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7
1,2,3,4,5,6,7,8-Octachlorodibenzofuran (OCDF)	39001-02-0

Total Tetrachlorodibenzo- <i>p</i> -dioxin (TCDD)	41903-57-5
Total Pentachlorodibenzo- <i>p</i> -dioxin (PeCDD)	36088-22-9
Total Hexachlorodibenzo- <i>p</i> -dioxin (HxCDD)	34465-46-8
Total Heptachlorodibenzo- <i>p</i> -dioxin (HpCDD)	37871-00-4
Total Tetrachlorodibenzofuran (TCDF)	55722-27-5
Total Pentachlorodibenzofuran (PeCDF)	30402-15-4
Total Hexachlorodibenzofuran (HxCDF)	55684-94-1
Total Heptachlorodibenzofuran (HpCDF)	38998-75-3

^a Chemical Abstract Service Registry Number

1.2 This method requires the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits (MCLs), and other pertinent information. Samples containing concentrations of specific congeneric analytes (PCDDs and PCDFs) considered within the scope of this method that are greater than ten times the upper MCLs must be analyzed by a protocol designed for such concentration levels, e.g., Method 8280. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is described.

1.3 The sensitivity of this method is dependent upon the level of interferences within a given matrix. The calibration range of the method for a 1-L water sample is 10 to 2000 ppq for TCDD/TCDF and PeCDD/PeCDF, and 1.0 to 200 ppt for a 10-g soil, sediment, fly ash, or tissue sample for the same analytes (Table 1). Analysis of a one-tenth aliquot of the sample permits measurement of concentrations up to 10 times the upper MCL. The actual limits of quantitation will differ from the lower MCL, depending on the complexity of the matrix.

1.4 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, 5000, and 8000) for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced in residue analysis and skilled in HRGC/HRMS. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.6 Because of the extreme toxicity of these compounds, the analyst must take necessary precautions to prevent the exposure of laboratory personnel or others to materials known or believed to contain PCDDs or PCDFs. Typical infectious waste incinerators are not satisfactory devices for disposal of materials highly contaminated with PCDDs or PCDFs. A

laboratory planning to use these compounds should prepare a disposal plan. Additional safety instructions are outlined in Secs. 5.0.

2.0 SUMMARY OF METHOD

2.1 This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) analytical techniques. See Method 3500 for guidance on other appropriate extraction techniques.

2.2 If interferences are encountered, the method provides selected cleanup procedures to aid the analyst in their elimination.

2.3 A designated amount (see Table 1) of soil, sediment, fly ash, water, sludge (including paper pulp), still bottom, fuel oil, chemical reactor residue, fish tissue, or human adipose tissue is spiked with a solution containing specified amounts of each of the nine isotopically ($^{13}\text{C}_{12}$) labeled PCDDs/PCDFs listed in the far left column of Table 2. The sample is then extracted according to a matrix-specific extraction procedure. Aqueous samples that contain 1 percent or more solids and solid samples that show an aqueous phase are filtered, the solid phase (including the filter) and the aqueous phase extracted separately, and the extracts combined before extract cleanup. The extraction procedures are:

- (a) Toluene -- Soxhlet extraction for soil, sediment, fly ash, and paper pulp samples
- (b) Methylene chloride -- liquid-liquid extraction for water samples
- (c) Toluene -- Dean-Stark extraction for fuel oil, and aqueous sludge samples
- (d) Toluene extraction for still bottom samples
- (e) Hexane/methylene chloride or methylene chloride -- Soxhlet extraction for fish tissue samples
- (f) Methylene chloride extraction for human adipose tissue samples
- (g) As another option, all solid samples (wet or dry) may be extracted with toluene using a Soxhlet/Dean Stark extraction system or using pressurized fluid extraction (PFE) (Method 3545) or microwave extraction (Method 3546).
- (h) Water samples may also be extracted using solid-phase extraction (SPE) by Method 3535.

The decision for the selection of an extraction procedure for chemical reactor residue samples is based on the appearance (consistency, viscosity) of the samples. Generally, the samples can be handled according to the procedure used for still bottom (or chemical sludge) samples.

2.4 The extracts are submitted to an acid-base washing treatment and dried. Following a solvent exchange step, the extracts are cleaned up by column chromatography on alumina, silica gel, and activated carbon.

2.4.1 The extracts from adipose tissue samples are treated with silica gel impregnated with sulfuric acid before chromatography on acidic silica gel, neutral alumina, and activated carbon.

2.4.2 Fish tissue and paper pulp extracts are subjected to an acid wash treatment only, prior to chromatography on alumina and activated carbon.

2.5 The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 10 to 50 μL (depending on the matrix) of a nonane solution containing 50 $\text{pg}/\mu\text{L}$ of the

recovery standards $^{13}\text{C}_{12}$ -1,2,3,4-TCDD and $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD (Table 2). The former is used to determine the percent recoveries of tetra- and pentachlorinated PCDD/PCDF congeners, while the latter is used to determine the percent recoveries of the hexa-, hepta- and octachlorinated PCDD/PCDF congeners.

2.6 An aliquot of the concentrated extract is injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving power of at least 10,000 (10 percent valley definition).

2.7 The identification of OCDD and nine of the fifteen 2,3,7,8-substituted congeners (Table 3), for which ^{13}C -labeled standards are available in the sample fortification and recovery standard solutions (Table 2), is based on their elution at their exact retention time (within 0.005 retention time units measured in the routine calibration) and the simultaneous quantitation of the two most abundant ions in the molecular ion region. The remaining six 2,3,7,8-substituted congeners (i.e., 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF, and 1,2,3,4,7,8,9-HpCDF), for which no carbon-labeled internal standards are available in the sample fortification solution, and all other PCDD/PCDF congeners are identified when their relative retention times fall within their respective PCDD/PCDF retention time windows, as established from the routine calibration data, and the simultaneous quantitation of the two most abundant ions in the molecular ion region. The identification of OCDF is based on its retention time relative to $^{13}\text{C}_{12}$ -OCDD and the simultaneous quantitation of the two most abundant ions in the molecular ion region. Identification also is based on a comparison of the ratios of the integrated ion abundance of the molecular ion species to their theoretical abundance ratios.

2.8 Quantitation of the individual congeners, total PCDDs, and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homologue, during which each calibration solution is analyzed once.

3.0 DEFINITIONS

3.1 Method calibration limits (MCL)

3.1.1 Lower method calibration limit (LCML) is the low standard on the calibration curve.

3.1.2 Upper method calibration limit (UMCL) is the high standard on the calibration curve.

NOTE: The LCML and UCML values in Table 1 were those used during the method development process. Other calibration ranges may be used based on project data requirements.

3.2 Refer to Chapter One and the manufacturer's instructions for other definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data (see Refs. 1 and 2). All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific

selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Four for general guidance on the cleaning of glassware. In addition, analysts should avoid using PVC gloves. Also refer to Method 8000 for a discussion of interferences.

4.2 The use of high-purity reagents and pesticide-grade solvents helps to minimize interference problems. Purification of solvents by distillation, in all glass systems, may be required.

4.3 Interferants coextracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated alkyldibenzofurans, that may be found at concentrations several orders of magnitude higher than that of the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established in Sec. 9.3.1.3. While cleanup techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve the sensitivity described in this method.

4.4 A high-resolution capillary column (60-m DB-5, J&W Scientific, or equivalent) is used in this method. However, no single column is known to resolve all 210 isomers. The 60-m DB-5 GC column is capable of 2,3,7,8-TCDD isomer specificity (Sec. 6.2.1). In order to determine the concentration of the 2,3,7,8-TCDF (if detected on the DB-5 column), the sample extract must be reanalyzed on a column capable of 2,3,7,8-TCDF isomer specificity (e.g., DB-225, SP-2330, SP-2331, or equivalent).

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in the chemical analysis of samples suspected to contain PCDDs/PCDFs. Additional information on laboratory safety is given in Refs. 3, 4 and 5.

5.2 Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCDDs or PCDFs. It is the responsibility of the laboratory personnel to ensure that safe handling procedures are employed.

5.3 The following safety practices are excerpts from EPA Method 1613, Sec. 5 (October 1994 version), amended as necessary for use in conjunction with this method. The 2,3,7,8-TCDD isomer has been found to be acnegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in positions 2,3,7,8 are known to have toxicities comparable to that of 2,3,7,8-TCDD. The analyst should note that finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. It is recommended that such samples be processed in a confined environment, such as a hood or a glove box. Laboratory personnel handling these types of samples should wear masks fitted with charcoal filters to prevent inhalation of dust.

5.4 The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.

5.5 Each laboratory must develop a strict safety program for the handling of PCDDs and PCDFs. The laboratory practices listed below are recommended.

5.5.1 Contamination of the laboratory will be minimized by conducting most of the manipulations in a hood, or in a separate containment facility away from the main laboratory.

5.5.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high boiling alcohols.

5.5.3 Liquid waste should be dissolved in methanol or ethanol and irradiated with ultraviolet light at a wavelength less than 290 nm for several days (use F 40 BL lamps, or equivalent). Using this analytical method, analyze the irradiated liquid wastes and dispose of the solutions when 2,3,7,8-TCDD and 2,3,7,8-TCDF congeners can no longer be detected.

5.6 The following precautions for safe handling of 2,3,7,8-TCDD in the laboratory were issued by Dow Chemical U.S.A. (revised 11/78) and were amended for use in conjunction with this method. The following statements on safe handling are as complete as possible on the basis of available toxicological information. The precautions for safe handling and use are necessarily general in nature since detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual use. Assistance in evaluating the health hazards of particular plant conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. The 2,3,7,8-TCDD isomer is extremely toxic to certain kinds of laboratory animals. However, it has been handled for years without injury in analytical and biological laboratories. Many techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD.

5.6.1 Protective equipment -- Disposable plastic gloves, apron or lab coat, safety glasses and laboratory hood adequate for radioactive work. However, PVC gloves should not be used.

5.6.2 Training -- Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

5.6.3 Personal hygiene -- Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift).

5.6.4 Confinement -- Isolated work area, posted with signs, segregated glassware and tools, plastic-backed absorbent paper on benchtops.

5.6.5 Waste -- Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.

5.6.6 Disposal of hazardous wastes -- Refer to the November 7, 1986 issue of the Federal Register on Land Ban Rulings for details concerning the handling of dioxin-containing wastes.

5.6.7 Decontamination of personnel -- Apply a mild soap with plenty of scrubbing action.

5.6.8 Glassware, tools and surfaces -- Chlorothene NU Solvent™ (Dow Chemical Company) is the least toxic solvent shown to be effective. Satisfactory cleaning may be accomplished by rinsing with chlorothene, then washing with a detergent and water. Dishwater may be disposed to the sewer after percolation through a charcoal bed filter. It is prudent to minimize solvent wastes because they require costly special disposal through commercial services.

5.6.9 Laundry -- Clothing known to be contaminated should be disposed according to the precautions of the source described under Sec. 5.6.6, "Disposal of hazardous wastes." Laboratory coats or other clothing worn in 2,3,7,8-TCDD work area may be laundered. Clothing should be collected in plastic bags. Persons who convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows the problem. The washer should be run through one full cycle before being used again for other clothing.

5.6.10 Wipe tests -- A useful method for determining cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper, extract the filter paper and analyze the extract.

NOTE: A procedure for the collection, handling, analysis, and reporting requirements of wipe tests performed within the laboratory is described in Appendix A of this method. The results and decision-making processes are based on the presence of 2,3,7,8-substituted PCDDs/PCDFs.

5.6.11 Inhalation -- Any procedure that may generate airborne contamination must be carried out with good ventilation. Gross losses to a ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no significant inhalation hazards except in case of an accident.

5.6.12 Accidents -- Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

5.7 It is recommended that personnel working in laboratories where PCDDs/PCDFs are handled be given periodic physical examinations (at least annually). Such examinations should include specialized tests, such as those for urinary porphyrins and for certain blood parameters which, based upon published clinical observations, are appropriate for persons who may be exposed to PCDDs/PCDFs. Periodic facial photographs to document the onset of dermatologic problems are also advisable.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 High-resolution gas chromatograph/high-resolution mass spectrometer/data system (HRGC/HRMS/DS) -- The GC must be equipped for temperature programming, and all required accessories must be available, such as syringes, gases, and capillary columns.

6.1.1 GC injection port -- The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. On-column 1- μ L injections can be used on the 60-m DB-5 column. The use of a moving needle injection port is also acceptable. When using the method described in this protocol, a 2- μ L injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2 μ L). Also, 1- μ L injections may be used; however, laboratories must remain consistent throughout the analyses by using the same injection volume at all times.

6.1.2 GC/MS interface -- The GC/MS interface components should withstand 350 °C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel™, or equivalent, ferrules are recommended.

6.1.3 Mass spectrometer -- The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley).

6.1.4 Data system -- A dedicated data system is employed to control the rapid multiple-ion monitoring process and to acquire the data. Quantitation data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantitations may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data at a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass spectral peak profiles (Sec. 9.3.2.3 and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should permit the measurement of noise on the base line.

NOTE: The detector ADC zero setting must allow peak-to-peak measurement of the noise on the base line of every monitored channel and allow for good estimation of the instrument resolving power. The effect of different zero settings on the measured resolving power is shown in Figure 2.

6.2 GC columns

Fused-silica capillary columns are needed. The columns must demonstrate the required separation of all 2,3,7,8-specific isomers whether a dual-column or a single-column analysis is chosen. Chromatographic performance must be demonstrated and documented (Sec. 9.3.1) at the beginning of each 12-hr period (after mass resolution and GC resolution are demonstrated) during which sample extracts or concentration calibration solutions will be analyzed. Recommended operating conditions for the recommended columns are shown in Sec. 11.6.

The columns listed in this section were the columns used in developing the method. The listing of these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Laboratories may use these columns or other columns provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

6.2.1 60-m DB-5 (J&W Scientific) or equivalent fused-silica capillary column

In order to have an isomer-specific determination of 2,3,7,8-TCDD and to allow the quantitation of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, use of this column is recommended. Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60-m DB-5 column. Problems have been associated with the separation of 2,3,7,8-TCDD from 1,2,3,7-TCDD and 1,2,6,8-TCDD, and with the separation of 2,3,7,8-TCDF from 1,2,4,9-, 1,2,7,9-, 2,3,4,6-, 2,3,4,7-, and 2,3,4,8-TCDF. Because of the toxicologic concern associated with 2,3,7,8-TCDD and 2,3,7,8-TCDF, additional analyses may be necessary for some samples.

6.2.2 30-m DB-225 (J&W Scientific) or equivalent fused-silica capillary column

For the DB-225 column, problems are associated with the separation of 2,3,7,8-TCDF from 2,3,4,7-TCDF and a combination of 1,2,3,9- and 2,3,4,8-TCDF.

6.3 Miscellaneous equipment and materials -- The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.3.1 Nitrogen evaporation apparatus with variable flow rate.

6.3.2 Balances capable of accurately weighing to 0.01 g and 0.0001 g.

6.3.3 Centrifuge.

6.3.4 Water bath -- Equipped with concentric ring covers and capable of being temperature controlled within ± 2 °C.

6.3.5 Stainless steel or glass container large enough to hold contents of one-pint sample containers.

6.3.6 Glove box.

6.3.7 Drying oven.

6.3.8 Stainless steel spoons and spatulas.

6.3.9 Laboratory hoods.

6.3.10 Pipets -- Disposable, Pasteur, 150 mm long x 5 mm ID.

6.3.11 Pipets -- Disposable, serological, 10-mL, for the preparation of the carbon columns described in Sec. 11.5.3.

6.3.12 Reaction vial -- 2-mL, silanized amber glass (Reacti-vial, or equivalent).

6.3.13 Stainless steel meat grinder with a 3 to 5 mm hole size inner plate.

6.3.14 Separatory funnels -- 125-mL and 2000-mL.

6.3.15 Kuderna-Danish concentrator -- 500-mL, fitted with 10-mL concentrator tube and three-ball Snyder column. Other evaporation devices such as a Turbovap and Nevap may be used as appropriate for a given application. Users should consult the manufacturers guidelines for operational requirements and instructions.

NOTE: The use of a solvent vapor recovery system (Kontes K-545000-1006 or K-547300-0000, Ace Glass 6614-30, or equivalent) is recommended for use in methods that use Kuderna-Danish or other evaporative concentrators. Incorporation of this apparatus may be required by Federal, State, or local municipality regulations that govern air emissions of volatile organics. EPA recommends the incorporation of this type of reclamation system as a method to implement an emissions reduction program. Solvent recovery is a means to conform with waste minimization and pollution prevention initiatives.

6.3.16 PTFE or Carborundum™ (silicon carbide) boiling chips (or equivalent), washed with hexane before use.

CAUTION: PTFE boiling chips may float in methylene chloride, may not work in the presence of any water phase, and may be penetrated by nonpolar organic compounds.

6.3.17 Chromatographic columns -- Glass, 300 mm x 10.5 mm, fitted with PTFE stopcock.

6.3.18 Adapters for concentrator tubes.

6.3.19 Glass fiber filters -- 0.70- μ m, Whatman GFF, or equivalent.

6.3.20 Dean-Stark trap -- 5- or 10-mL, with T-joints, condenser and 125-mL flask.

6.3.21 Continuous liquid-liquid extractor -- 1-L sample capacity, suitable for use with heavier than water solvents.

6.3.22 All glass Soxhlet apparatus with 500-mL flask.

6.3.23 Soxhlet/Dean-Stark extractor (optional) -- All glass, 500-mL flask.

6.3.24 Glass funnels -- Sized to hold 170 mL of liquid.

6.3.25 Desiccator.

6.3.26 Solvent reservoir (125-mL) -- Compatible with gravity carbon column.

6.3.27 Rotary evaporator with a temperature-controlled water bath.

6.3.28 High speed tissue homogenizer -- Equipped with an EN-8 probe, or equivalent.

6.3.29 Glass wool -- Extract with methylene chloride, dry, and store in a glass jar.

6.3.30 Extraction jars -- Glass, 250-mL, equipped with PTFE-lined screw caps.

6.3.31 Volumetric flasks -- Class A, 10-mL to 1000-mL.

6.3.32 Glass vials -- 1-dram (or metric equivalent).

6.3.33 See Methods 3535, 3545, and 3546 for the supplies specific to their particular preparation procedures.

6.4 Laboratory glassware cleaning procedures -- Reuse of glassware should be minimized to avoid the risk of using contaminated glassware. All glassware that is reused must be scrupulously cleaned as soon as possible after use, applying the following procedure.

6.4.1 Rinse glassware with the last solvent used in it.

6.4.2 Wash with hot water containing detergent.

6.4.3 Rinse with copious amounts of tap water and several portions of organic-free reagent water. Drain dry.

6.4.4 Rinse with pesticide-grade acetone and hexane.

6.4.5 After glassware is dry, store inverted or capped with aluminum foil in a clean environment.

6.4.6 Do not bake reusable glassware as a routine part of cleaning. Baking may be warranted after particularly dirty samples are encountered, but it should be minimized, because repeated baking may cause active sites on the glass surface that will irreversibly adsorb PCDDs/PCDFs.

CAUTION: The analysis for PCDDs/PCDFs in water samples is for much lower concentrations than in soil/sediment, fly ash, or chemical waste samples. Extreme care must be taken to prevent cross-contamination between soil/sediment, fly ash, chemical waste and water samples. Therefore, it is strongly recommended that separate glassware be reserved for analyzing water samples.

6.5 Pre-extraction of glassware -- All glassware should be rinsed or pre-extracted with solvent immediately before use. Soxhlet-Dean-Stark (SDS) apparatus and continuous liquid-liquid extractors should be pre-extracted for approximately 3 hr immediately prior to use, using the same solvent and extraction conditions that will be employed for sample extractions. The pooled waste solvent for a set of extractions may be concentrated and analyzed as a method of demonstrating that the glassware was free of contamination.

It is recommended that each piece of reusable glassware be numbered in such a fashion that the laboratory can associate all reusable glassware with the processing of a particular sample. This will assist the laboratory in:

- (1) Tracking down possible sources of contamination for individual samples,
- (2) Identifying glassware associated with highly contaminated samples that may require extra cleaning, and
- (3) Determining when glassware should be discarded.

7.0 REAGENTS AND STANDARDS

7.1 Reagent-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

7.2 Organic-free reagent water -- All references to water in this method refer to organic-free reagent water.

7.3 Column chromatography reagents

7.3.1 Alumina -- neutral, 80/200 mesh (Super 1, Woelm®, or equivalent). Store in a sealed container at room temperature, in a desiccator, over self-indicating silica gel.

7.3.2 Alumina -- acidic AG4 (Bio Rad Laboratories catalog #132-1240, or equivalent). Soxhlet extract with methylene chloride for 24 hr if blanks show contamination, and activate by heating in a foil-covered glass container for 24 hr at 190 °C. Store in a glass bottle sealed equipped with a PTFE-lined screw cap.

7.3.3 Silica gel -- high-purity grade, type 60, 70-230 mesh. Soxhlet extract with methylene chloride for 24 hr if blanks show contamination, and activate by heating in a foil covered glass container for 24 hr at 190 °C. Store in a glass bottle sealed equipped with a PTFE-lined screw cap.

7.3.4 Silica gel impregnated with sodium hydroxide. Add one part (by weight) of 1 M NaOH solution to two parts (by weight) silica gel (extracted and activated) in a screw cap bottle and mix with a glass rod until free of lumps. Store in a glass bottle sealed with a PTFE-lined screw cap.

7.3.5 Silica gel impregnated with 40 percent (by weight) sulfuric acid. Add two parts (by weight) concentrated sulfuric acid to three parts (by weight) silica gel (extracted and activated), mix with a glass rod until free of lumps, and store in a screw capped glass bottle. Store in a glass bottle sealed with a PTFE-lined screw cap.

7.3.6 Celite 545® (Supelco), or equivalent.

7.3.7 Charcoal carbon -- Activated carbon, Carbopak C (Supelco) or equivalent, prewashed with methanol and dried *in vacuo* at 110 °C. Store in a glass bottle sealed with a PTFE-lined screw cap. (Note: AX-21 [Anderson Development Company] carbon is no longer available, but existing stocks may be utilized.)

7.4 Reagents

7.4.1 Sulfuric acid, H₂SO₄, concentrated, ACS grade, specific gravity 1.84.

7.4.2 Potassium hydroxide, KOH, ACS grade, 20 percent (w/v) in organic-free reagent water.

7.4.3 Sodium chloride, NaCl, analytical reagent, 5 percent (w/v) in organic-free reagent water.

7.4.4 Potassium carbonate, K_2CO_3 , anhydrous, analytical reagent.

7.5 Sodium sulfate (powder, anhydrous), Na_2SO_4 -- Purify by heating at 400 °C for 4 hr in a shallow tray. If, after heating, the sodium sulfate develops a noticeable grayish cast (due to the presence of carbon in the crystal matrix), that batch of sodium sulfate is not suitable for use and should be discarded. Extraction with methylene chloride may produce sodium sulfate that is suitable for use in such instances, but following extraction, a reagent blank must be analyzed to demonstrate that there is no interference from the sodium sulfate.

7.6 Solvents -- All solvents should be (at a minimum) pesticide quality or equivalent, distilled-in-glass. Solvents may be degassed prior to use.

Samples should be extracted using a solvent system that gives optimum, reproducible recovery of the analytes of interest from the sample matrix, at the concentrations of interest. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed, *including* those specifically listed in this method, the analyst *must* demonstrate adequate performance for the analytes of interest, at the desired project-specific concentration levels. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations.

7.6.1 Methylene chloride, CH_2Cl_2 .

7.6.2 Hexane, C_6H_{14} .

7.6.3 Methanol, CH_3OH .

7.6.4 Nonane, C_9H_{20} .

7.6.5 Toluene, $C_6H_5CH_3$.

7.6.6 Cyclohexane, C_6H_{12} .

7.6.7 Acetone, CH_3COCH_3 .

7.7 High-resolution concentration calibration (HRCC) solutions (Table 5) -- Five nonane solutions containing 17 unlabeled and 11 carbon-labeled PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homologue-dependent, with the lowest values for the tetrachlorinated dioxin and furan (1.0 pg/ μ L) and the highest values for the octachlorinated congeners (1000 pg/ μ L). Standards containing more carbon-labeled PCDDs and PCDFs may also be employed. All standards should be stored away from any light source at ≤ 6 °C when not in use (-10 °C is recommended), and should be freshly prepared once a year, or sooner if check standards indicate a problem. The calibration verification (GC column performance check) standard should be prepared, as necessary, and stored at ≤ 6 °C. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

7.8 GC column performance check (evaluation) solution -- This solution contains the first and last eluting isomers for each homologous series from tetra- through heptachlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The $^{13}C_{12}$ -2,3,7,8-TCDD is also present. The laboratory is required to use nonane as the solvent and adjust the volume so that the final

concentration does not exceed 100 pg/μL per congener. Table 7 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution.

7.9 Sample fortification solution -- This nonane solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that $^{13}\text{C}_{12}$ -OCDF is not present in the solution.) Standards containing more carbon-labeled PCDDs and PCDFs may also be employed, provided that the same labeled compounds are contained in the calibration standards in Sec. 7.7.

7.10 Recovery standard solution -- This nonane solution contains two recovery standards, $^{13}\text{C}_{12}$ -1,2,3,4-TCDD and $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD, at a nominal concentration of 50 pg/μL per compound. 10 to 50 μL (depending on the matrix) of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample collection

8.1.1 Sample collection personnel should, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly not homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.1.2 Grab and composite samples must be collected in glass containers. Conventional sampling practices must be followed. The bottle must not be prewashed with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.2 Grinding or blending of fish samples -- If not otherwise specified in a project plan, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3 to 5 mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested. If so requested, the above whole fish requirement is superseded.

8.3 Recommended sample storage and holding times -- All samples should be stored at $\leq 6^\circ\text{C}$, or lower, in the dark, extracted within 30 days and completely analyzed within 45 days of extraction. Fish and adipose tissue samples should be stored at $< -10^\circ\text{C}$ in the dark, and should be extracted within 30 days and completely analyzed within 45 days of collection.

8.4 Unused portions of samples and sample extracts should be stored at $\leq 6^\circ\text{C}$ in the dark for six months after sample receipt to allow further analyses if necessary.

NOTE: The holding times listed in this method are recommendations. PCDDs and PCDFs are very stable in a variety of matrices, and holding times under the conditions listed in this section may be as long as a year for certain matrices.

8.5 Phase separation

This is a guideline for phase separation for very wet (>25 percent water) soil, sediment and paper pulp samples. Place a 50-g portion in a suitable centrifuge bottle and centrifuge for 30 min at 2,000 rpm. Remove the bottle and mark the interface level on the bottle. Estimate the relative volume of each phase. With a disposable pipet, transfer the liquid layer into a clean bottle. Mix the solid with a stainless steel spatula and remove a portion to be weighed and analyzed (percent dry weight determination, extraction). Return the remaining solid portion to the original sample bottle (empty) or to a clean sample bottle that is properly labeled, and store it as appropriate. Analyze the solid phase by using only the soil, sediment and paper pulp method. Take note of, and report, the estimated volume of liquid before disposing of the liquid as a liquid waste.

8.6 Soil, sediment, or paper sludge (pulp) percent dry weight determination

When results are to be reported on a dry-weight basis, the percent dry weight of soil, sediment or paper pulp samples may be determined according to the following procedure. Weigh a 10-g portion of the soil or sediment sample (± 0.5 g) to three significant figures. Dry it to constant weight at 110 °C in an adequately ventilated oven. Allow the sample to cool in a desiccator. Weigh the dried solid to three significant figures. Calculate and report the percent dry weight. Do not use this solid portion of the sample for extraction, but instead dispose of it as hazardous waste.

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

WARNING: Finely divided soils and sediments contaminated with PCDDs/PCDFs are hazardous because of the potential for inhalation or ingestion of particles containing PCDDs/PCDFs (including 2,3,7,8-TCDD). Such samples should be handled in a confined environment (i.e., a closed hood or a glove box).

8.7 Lipid content determination

8.7.1 Fish tissue -- To determine the lipid content of fish tissue, concentrate 125 mL of the fish tissue extract (Sec. 11.2.2), in a tared 200-mL round-bottom flask, on a rotary evaporator until a constant weight is achieved.

$$\% \text{ lipid} = \frac{\text{weight of residue} \times 2}{\text{weight of sample}} \times 100$$

The factor of 2 accounts for the use of half of the extract (e.g., 125 mL of 250 mL total volume) for the lipid determination.

Dispose of the lipid residue as a hazardous waste if the results of the analysis indicate the presence of PCDDs or PCDFs.

Other procedures and other extract volumes may be employed for the lipid determination, provided that they are clearly described and documented. Adjustments to the amount of internal standards spiked in Sec. 11.1 will be required if different volumes are employed.

8.7.2 Adipose tissue -- Details for the determination of the adipose tissue lipid content are provided in Sec. 11.3.3.

8.8 Also see the introductory material to Chapter Four, "Organic Analytes," for general information related to sample collection, preservation, and storage.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Refer to Method 8000 for specific determinative method QC procedures. Refer to Method 3500 for QC procedures to validate sample extraction and ensure the proper operation of the various sample preparation techniques. If an extract cleanup procedure is performed, refer to Method 3600 for the appropriate QC procedures. Any more specific QC procedures provided in this method will supersede those noted in Methods 8000, 3500, or 3600.

9.3 System performance criteria -- System performance criteria are presented below. The laboratory may use the recommended GC column described in Sec. 6.2. The laboratory must document that all applicable system performance criteria (described in Sec. 9.3) were met before it analyses any sample. Sec. 11.6.1 provides recommended GC conditions that can be used to satisfy the required criteria. Figure 3 provides a typical 12-hr analysis sequence, whereby the response factors and mass spectrometer resolving power checks must be performed at the beginning and the end of each 12-hr period of operation. A GC column performance check is only required at the beginning of each 12-hr period during which samples are analyzed. An HRGC/HRMS method blank run is required between a calibration run and the first sample run. The same method blank extract may thus be analyzed more than once if the number of samples within a batch requires more than 12 hr of analyses.

9.3.1 GC column performance

9.3.1.1 Inject 2 μ L of an aliquot (Sec. 6.1.1) of the column performance check solution (Sec. 7.8) and acquire selected ion monitoring (SIM) data as described in Sec. 11.6.2 within a total cycle time of ≤ 1 sec (Sec. 11.6.3.1).

9.3.1.2 The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of ≤ 25 percent (Figure 4), where:

$$\text{Valley percent} = (x/y) \times (100)$$

- x = measured as in Figure 4 from the 2,3,7,8-closest TCDD eluting isomer
 y = the peak height of 2,3,7,8-TCDD

It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions described in this protocol. Their retention times are used to determine the five homologue retention time windows that are used for qualitative (Sec. 11.8.4.1) and quantitative purposes. All peaks (that includes $^{13}\text{C}_{12}$ -2,3,7,8-TCDD) should be labeled and identified on the chromatograms. Furthermore, all first eluters of a homologous series should be labeled with the letter "F," and all last eluters of a homologous series should be labeled with the letter "L" (Figure 4 shows an example of peak labeling for TCDD isomers). Any individual selected ion current profile (SICP) (for the tetras, this would be the SICP for m/z 322 and m/z 304) or the reconstructed homologue ion current (for the tetras, this would correspond to m/z 320 + m/z 322 + m/z 304 + m/z 306) constitutes an acceptable form of data presentation. An SICP for the labeled compounds (e.g., m/z 334 for labeled TCDD) is also required.

9.3.1.3 The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds. Allowable tolerance on the daily verification with the GC performance check solution should be better than 10 sec for the absolute retention times of all the components of the mixture. Particular caution should be exercised for the switching time between the last tetra-chlorinated congener (i.e., 1,2,8,9-TCDD) and the first pentachlorinated congener (i.e., 1,3,4,6,8-PeCDF), as these two compounds elute within 15 sec of each other on the 60-m DB-5 column. A laboratory with a GC/MS system that is not capable of detecting both congeners (1,2,8,9-TCDD and 1,3,4,6,8-PeCDF) within one analysis must take corrective action. If the recommended column is not used, then the first- and last-eluting isomer of each homologue must be determined experimentally on the column which is used, and the appropriate isomers must then be used for window definition and switching times.

9.3.2 Mass spectrometer performance

9.3.2.1 The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed (Sec. 11.8). Static resolving power checks must be performed at the beginning and at the end of each 12-hr period of operation. However, it is recommended that a check of the static resolution be made and documented before and after each analysis. Corrective action must be implemented whenever the resolving power does not meet the requirement.

9.3.2.2 Chromatography time for PCDDs and PCDFs exceeds the long term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a

mass drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in an increase in downtime for source cleaning.

9.3.2.3 Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760) obtained during the above peak matching experiment by using the low-mass PFK ion at m/z 304.9824 as a reference. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 5) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10 percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

9.4 Initial demonstration of proficiency

Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000 for information on how to accomplish a demonstration of proficiency.

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis.

9.6 Sample quality control for preparation and analysis

9.6.1 Performance evaluation samples -- When available, performance evaluation (PE) samples containing known amounts of unlabeled 2,3,7,8-substituted

PCDDs/PCDFs or other PCDD/PCDF congeners should be analyzed alongside routine field samples.

9.6.2 Performance check solutions

9.6.2.1 At the beginning of each 12-hr period during which samples are to be analyzed, an aliquot of the (1) GC column performance check solution and (2) high-resolution concentration calibration solution No. 3 (HRCC-3; see Table 5) must be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. A mass resolution check must also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed. Alternatively, these check sample analyses may be combined into one run using a commercially prepared standard.

9.6.2.2 To validate positive sample data, the routine or continuing calibration (HRCC-3; Table 5) and the mass resolution check must be performed also at the end of each 12-hr period during which samples are analyzed. Furthermore, an HRGC/HRMS method blank run should be recorded following a calibration run and prior to the first sample run.

9.6.2.2.1 If the laboratory operates only during one period (shift) each day of 12 hr or less, the GC performance check solution must be analyzed only once (at the beginning of the period) to validate the data acquired during the period. However, the mass resolution and continuing calibration checks must be performed at the beginning as well as at the end of the period.

9.6.2.2.2 If the laboratory operates during consecutive 12-hr periods (shifts), analysis of the GC performance check solution must be performed at the beginning of each 12-hr period. The mass resolution and continuing calibration checks from the previous period can be used for the beginning of the next period.

9.6.2.3 Results of at least one analysis of the GC column performance check solution and of two mass resolution and continuing calibration checks must be reported with the sample data collected during a 12-hr period.

9.6.2.4 Deviations from criteria specified for the GC performance check or for the mass resolution check invalidate all positive sample data collected between analyses of the performance check solution, and the extracts from those positive samples must be reanalyzed.

If the routine calibration run fails at the beginning of a 12-hr shift, the instructions in Sec. 11.7.4.4 must be followed. If the continuing calibration check performed at the end of a 12 hr period fails by no more than 25 percent RPD for the 17 unlabeled compounds and 35 percent RPD for the 9 labeled reference compounds, use the mean to the two "daily" RF values from the two daily routine calibration runs to compute the analyte concentrations, instead of the $\overline{RF}$ values obtained from the initial calibration. A new initial calibration (new $\overline{RF}$ s) is required immediately (within 2 hr) following the analysis of the samples, whenever the RPD from the end-of-shift routine calibration exceeds 25 percent or 35 percent,

respectively. Failure to perform a new initial calibration immediately following the analysis of the samples will automatically require reanalysis of all positive sample extracts analyzed before the failed end-of-shift continuing calibration check.

9.6.3 The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from commercial sources.

9.6.4 Field blanks -- Batches of field samples may contain a field blank sample of uncontaminated soil, sediment or water that is to be fortified before analysis according to Sec. 9.6.4.1. In addition to this field blank, a batch of samples may include a rinsate, which is a portion of the solvent that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

9.6.4.1 Fortified field blank (OPTIONAL)

9.6.4.1.1 Weigh a 10-g portion or use 1-L (for aqueous samples) of the designated field blank sample and add 100 μ L of the solution containing the nine internal standards (Table 2) diluted with 1.0 mL acetone (Sec. 11.1).

9.6.4.1.2 Extract by using the procedures beginning in Secs. 11.4.5 or 11.4.6, as applicable, add 10 μ L of the recovery standard solution (Sec. 7.10) and analyze a 2- μ L aliquot of the concentrated extract.

9.6.4.1.3 Calculate the concentration (Sec. 11.9.1) of 2,3,7,8-substituted PCDDs/PCDFs and the percent recovery of the internal standards (Sec. 11.9.2).

9.6.4.2 Rinsate sample (OPTIONAL)

9.6.4.2.1 Take a 100-mL ($\pm$ 0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add 100 μ L of the solution containing the nine internal standards (Table 2).

9.6.4.2.2 Using a K-D apparatus, concentrate to about 5 mL.

NOTE: As an option, a rotary evaporator may be used in place of the K-D apparatus for the concentration of the rinsate.

9.6.4.2.3 Transfer the 5 mL concentrate from the K-D concentrator tube in 1-mL portions to a 1-mL minivial, reducing the volume in the minivial as necessary with a gentle stream of dry nitrogen.

9.6.4.2.4 Rinse the K-D concentrator tube with two 0.5 mL portions of hexane and transfer the rinses to the 1 mL minivial. Concentrate with dry nitrogen, as necessary.

9.6.4.2.5 Just before analysis, add 10 μ L of the recovery standard solution (Table 2) and reduce the volume to its final volume, as necessary (Sec. 11.8.1). No column chromatography is required.

9.6.4.2.6 Analyze an aliquot of the solution following the same procedures used to analyze samples.

9.6.4.2.7 Report percent recovery of the internal standard and the presence of any PCDD/PCDF compounds in µg/L of rinsate solvent.

9.6.5 Duplicate analyses (OPTIONAL)

In each batch of samples, locate the sample designated for duplicate analysis (the sample may be labeled "double volume"), and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration.

9.6.5.1 The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference (difference expressed as percentage of the mean). Report all results.

9.6.5.2 Recommended actions to help locate problems

- (1) Verify satisfactory instrument performance (Secs. 9.3 and 9.6).
- (2) If possible, verify that no error was made while weighing the sample portions.
- (3) Review the analytical procedures with the performing laboratory personnel.

9.7 Percent recovery of the internal standards -- For each sample, method blank and rinsate, calculate the percent recovery (Sec. 11.9.2). The percent recovery should be between 40 percent and 135 percent for all 2,3,7,8-substituted internal standards.

NOTE: A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data.

9.8 Identification criteria

9.8.1 If either one of the identification criteria appearing in Secs. 11.8.4.1.1 through 11.8.4.1.4 is not met for an homologous series, it is reported that the sample does not contain unlabeled 2,3,7,8-substituted PCDD/PCDF isomers for that homologous series at the calculated quantitation limit (Sec. 11.9.5)

9.8.2 If the first initial identification criteria (Secs. 11.8.4.1.1 through 11.8.4.1.4) are met, but the criteria appearing in Secs. 11.8.4.1.5 and 11.8.4.2 are not met, that sample is presumed to contain interfering contaminants. This must be noted on the analytical report form, and the sample should be rerun or the extract reanalyzed.

9.9 The analysis of method blanks is critical to the provision of meaningful sample results.

9.9.1 Method blanks should be prepared at a frequency of at least 5%, that is, one method blank for each group of up to 20 samples prepared at the same time, by the same procedures.

9.9.2 When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

9.9.3 If the method blank results do not meet the project-specific acceptance criteria, then the laboratory should take corrective action to locate and reduce the source of the contamination and to re-extract and reanalyze any samples associated with the contaminated method blank.

9.9.4 The laboratory should not subtract the results of the method blank from those of any associated samples. Such "blank subtraction" is inappropriate and often leads to negative sample results. If the method blank results do not meet the acceptance criteria in 9.9.3 and reanalysis is not practical, then the data user should be provided with the sample results, the method blank results, and a discussion of the corrective actions undertaken by the laboratory.

9.10 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

10.0 CALIBRATION AND STANDARDIZATION

See Sec 11.7 for information on calibration and standardization.

11.0 PROCEDURE

11.1 Internal standard addition

The sample fortification solution (Sec. 7.9) containing the carbon-labeled internal standards is added to each sample prior to extraction.

11.1.1 Select an appropriately sized sample aliquot. Typical sample size requirements for different matrices are given in Sec. 11.4 and in Table 1. Transfer the sample portion to a tared flask and determine its weight.

11.1.2 Except for adipose tissue, add an appropriate quantity of the sample fortification mixture (Sec. 7.9) to the sample. All samples should be spiked with 100 μL of the sample fortification mixture to give internal standard concentrations as indicated in Table 1. As an example, for $^{13}\text{C}_{12}$ -2,3,7,8-TCDD, a 10-g soil sample requires the addition of 1000 pg of $^{13}\text{C}_{12}$ -2,3,7,8-TCDD to give the required 100 ppt fortification level. The fish tissue sample (20 g) must be spiked with 200 μL of the internal standard solution, because half of the extract will be used to determine the lipid content (Sec. 8.7.1).

11.1.2.1 For the fortification of soil, sediment, fly ash, water, fish tissue, paper pulp and wet sludge samples, mix the sample fortification solution with 1.0 mL acetone.

11.1.2.2 Do not dilute the nonane solution for the other matrices.

11.1.2.3 The fortification of adipose tissue is carried out at the time of homogenization (Sec. 11.3.2.3).

11.2 Extraction and purification of fish and paper pulp samples

11.2.1 Add 60 g of anhydrous sodium sulfate to a 20-g portion of a homogeneous fish sample (Sec. 8.2) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the fish/sodium sulfate mixture in the Soxhlet apparatus on top of a glass wool plug. Add 250 mL of methylene chloride or hexane/methylene chloride (1:1) to the Soxhlet apparatus and reflux for 16 hr. The solvent must cycle completely through the system five to ten times per hour. Follow the same procedure for the partially dewatered paper pulp sample (using a 10-g sample, 30 g of anhydrous sodium sulfate and 200 mL of toluene).

NOTE: As an option, a Soxhlet/Dean-Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.2.2 Transfer the fish extract from Sec. 11.2.1 to a 250-mL volumetric flask and fill to the mark with methylene chloride. Mix well, then remove 125 mL for the determination of the lipid content (Sec. 8.7.1). Transfer the remaining 125 mL of the extract, plus two 15-mL hexane/methylene chloride rinses of the volumetric flask, to a K-D apparatus equipped with a Snyder column. Quantitatively transfer all of the paper pulp extract to a K-D apparatus equipped with a Snyder column.

NOTE: As an option, a rotary evaporator may be used in place of the K-D apparatus for the concentration of the extracts.

11.2.3 Add a PTFE (or equivalent) boiling chip. Concentrate the extract in a water bath to an apparent volume of 10 mL. Remove the apparatus from the water bath and allow to cool for 5 minutes.

11.2.4 Add 50 mL of hexane and a new boiling chip to the K-D flask. Concentrate in a water bath to an apparent volume of 5 mL. Remove the apparatus from the water bath and allow to cool for 5 minutes.

NOTE: The methylene chloride must have been completely removed before proceeding with the next step.

11.2.5 Remove and invert the Snyder column and rinse it into the K-D apparatus with two 1-mL portions of hexane. Decant the contents of the K-D apparatus and concentrator tube into a 125-mL separatory funnel. Rinse the K-D apparatus with two additional 5-mL portions of hexane and add the rinses to the funnel. Proceed with the appropriate cleanup according to the instructions in Sec. 11.5.

11.3 Extraction and purification of human adipose tissue

11.3.1 Human adipose tissue samples must be stored at a temperature of -20 °C or lower from the time of collection until the time of analysis. The use of chlorinated materials during the collection of the samples must be avoided. Samples are handled with stainless steel forceps, spatulas, or scissors. All sample bottles (glass) are cleaned as indicated in Sec. 6.4. PTFE-lined caps should be used.

NOTE: The storage temperature of -20 °C in Sec. 11.3.1 is the maximum storage temperature permissible for adipose tissue samples. Lower storage temperatures are recommended.

11.3.2 Adipose tissue extraction

11.3.2.1 Weigh a 10-g portion of a frozen adipose tissue sample to the nearest 0.01 g, into a culture tube (2.2 x 15 cm).

NOTE: The sample size may be smaller, depending on availability. In such situations, the analyst is required to adjust the volume of the internal standard solution added to the sample to meet the fortification level listed in Table 2.

11.3.2.2 Allow the adipose tissue specimen to reach room temperature (up to 2 hr).

11.3.2.3 Add 10 mL of methylene chloride and 100 μ L of the sample fortification solution. (Sec. 7.9) Homogenize the mixture for approximately 1 minute with a tissue homogenizer.

11.3.2.4 Allow the mixture to separate, then remove the methylene chloride extract from the residual solid material with a disposable pipet. Percolate the methylene chloride through a filter funnel containing a clean glass wool plug and 10 g of anhydrous sodium sulfate. Collect the dried extract in a graduated 100-mL volumetric flask.

11.3.2.5 Add a second 10 mL portion of methylene chloride to the sample and homogenize for 1 min. Decant the solvent, dry it, and transfer it to the 100-mL volumetric flask (Sec. 11.3.2.4).

11.3.2.6 Rinse the culture tube with at least two additional portions of methylene chloride (10-mL each), and transfer the entire contents to the filter funnel containing the anhydrous sodium sulfate. Rinse the filter funnel and the anhydrous sodium sulfate contents with additional methylene chloride (20 to 40 mL) into the 100-mL flask. Discard the sodium sulfate.

11.3.2.7 Adjust the volume to the 100-mL mark with methylene chloride.

11.3.3 Adipose tissue lipid content determination

11.3.3.1 Preweigh a clean 1-dram (or metric equivalent) glass vial to the nearest 0.0001 g on an analytical balance tared to zero.

11.3.3.2 Accurately transfer 1.0 mL of the final extract (100 mL) from Sec. 11.3.2.7 to the vial. Reduce the volume of the extract on a water bath (50-60 °C) by a gentle stream of purified nitrogen until an oily residue remains. Nitrogen evaporation is continued until a constant weight is achieved.

NOTE: When the sample size of the adipose tissue is smaller than 10 g, then the analyst may use a larger portion (up to 10 percent) of the extract defined in Sec. 11.3.2.7 for the lipid determination.

11.3.3.3 Accurately weigh the vial with the residue to the nearest 0.0001 g and calculate the weight of the lipid present in the vial based on the difference of the weights.

11.3.3.4 Calculate the percent lipid content of the original sample to the nearest 0.1 percent as shown below:

$$\% \text{ Lipid} = \frac{W_{lr} \times V_{ext}}{W_{at} \times V_{al}} \times 100$$

where:

W_{lr} = weight of the lipid residue to the nearest 0.0001 g calculated from Sec. 11.3.3.3,

V_{ext} = total volume (100 mL) of the extract in mL from Sec. 11.3.2.7,

W_{at} = weight of the original adipose tissue sample to the nearest 0.01 g from Sec. 11.3.2.1, and

V_{al} = volume of the aliquot of the final extract in mL used for the quantitative measure of the lipid residue (1.0 mL) from Sec. 11.3.3.2.

11.3.3.5 Record the weight of the lipid residue measured in Sec. 11.3.3.3 and the percent lipid content from Sec. 11.3.3.4.

11.3.4 Adipose tissue extract concentration

11.3.4.1 Quantitatively transfer the remaining extract from Sec. 11.3.3.2 (99.0 mL) to a 500-mL Erlenmeyer flask. Rinse the volumetric flask with 20 to 30 mL of additional methylene chloride to ensure quantitative transfer.

11.3.4.2 Concentrate the extract on a rotary evaporator and a water bath at 40 °C until an oily residue remains.

11.3.5 Adipose tissue extract cleanup

11.3.5.1 Add 200 mL of hexane to the lipid residue in the 500-mL Erlenmeyer flask and swirl the flask to dissolve the residue.

11.3.5.2 Slowly add, with stirring, 100 g of 40 percent (w/w) sulfuric acid-impregnated silica gel. Stir with a magnetic stirrer for 2 hr at room temperature.

11.3.5.3 Allow the solid phase to settle, and decant the liquid through a filter funnel containing 10 g of anhydrous sodium sulfate on a glass wool plug, into another 500-mL Erlenmeyer flask.

11.3.5.4 Rinse the solid phase with two 50-mL portions of hexane. Stir each rinse for 15 minutes, decant, and dry as described under Sec. 11.3.5.3. Combine the hexane extracts from Sec. 11.3.5.3 with the rinses.

11.3.5.5 Rinse the sodium sulfate in the filter funnel with an additional 25 mL of hexane and combine this rinse with the hexane extracts from Sec. 11.3.5.4.

11.3.5.6 Prepare an acidic silica column as follows: Pack a 2 cm x 10 cm chromatographic column with a glass wool plug, add approximately 20 mL of hexane, add 1 g of silica gel and allow to settle, then add 4 g of 40 percent (w/w) sulfuric acid-impregnated silica gel and allow to settle. Elute the excess hexane from the column until the solvent level reaches the top of the chromatographic packing. Verify that the column does not have any air bubbles and channels.

11.3.5.7 Quantitatively transfer the hexane extract from the Erlenmeyer flask (Secs. 11.3.5.3 through 11.3.5.5) to the silica gel column reservoir. Allow the hexane extract to percolate through the column and collect the eluate in a 500-mL K-D apparatus.

11.3.5.8 Complete the elution by percolating 50 mL of hexane through the column into the K-D apparatus. Concentrate the eluate on a steam bath to about 5 mL. Use nitrogen evaporation to bring the final volume to about 100 μ L.

CAUTION: If the silica gel impregnated with 40 percent sulfuric acid is highly discolored throughout the length of the adsorbent bed, the cleaning procedure must be repeated beginning with Sec. 11.3.5.1.

11.3.5.9 The extract is ready for the column cleanups described in Secs. 11.5.2 and 11.5.3.

11.4 Extraction and purification of environmental and waste samples

11.4.1 Sludge/wet fuel oil

11.4.1.1 Extract aqueous sludge or wet fuel oil samples by refluxing a sample (e.g., 2 g) with 50 mL of toluene in a 125-mL flask fitted with a Dean-Stark water separator. Continue refluxing the sample until all the water is removed.

NOTE: If the sludge or fuel oil sample dissolves in toluene, treat it according to the instructions in Sec. 11.4.2 below. If the sludge sample originates from pulp (paper mills), treat it according to the instructions starting in Sec. 11.2, but without the addition of sodium sulfate.

11.4.1.2 Cool the sample, filter the toluene extract through a glass fiber filter, or equivalent, into a 100-mL round-bottom flask.

11.4.1.3 Rinse the filter with 10 mL of toluene and combine the extract with the rinse.

11.4.1.4 Concentrate the combined solutions to near dryness on a rotary evaporator at 50 °C or using nitrogen evaporation. Proceed with Sec. 11.4.4.

11.4.2 Still bottom/oil

11.4.2.1 Extract still bottom or oil samples by mixing a sample portion (e.g., 1.0 g) with 10 mL of toluene in a small beaker and filtering the solution

through a glass fiber filter (or equivalent) into a 50-mL round-bottom flask. Rinse the beaker and filter with 10 mL of toluene.

11.4.2.2 Concentrate the combined toluene solutions to near dryness on a rotary evaporator at 50 °C or using nitrogen evaporation. Proceed with Sec. 11.4.4.

11.4.3 Fly ash

CAUTION: Because of the tendency of fly ash to "fly," all handling steps should be performed in a hood in order to minimize contamination.

11.4.3.1 Weigh about 10 g of fly ash to two decimal places and transfer to an extraction jar. Add 100 µL of the sample fortification solution (Sec. 7.9), diluted to 1 mL with acetone, to the sample. Add 150 mL of 1 M HCl to the fly ash sample. Seal the jar with the PTFE-lined screw cap and shake for 3 hr at room temperature.

11.4.3.2 Rinse a glass fiber filter with toluene, and filter the sample through the filter paper, placed in a Buchner funnel, into a 1-L flask. Wash the fly ash cake with approximately 500 mL of organic-free reagent water and dry the filter cake overnight at room temperature in a desiccator.

11.4.3.3 Add 10 g of anhydrous powdered sodium sulfate, mix thoroughly, let sit in a closed container for 1 hr, mix again, let sit for another hr, and mix again.

11.4.3.4 Place the sample and the filter paper into an extraction thimble, and extract in a Soxhlet extraction apparatus charged with 200 mL of toluene for 16 hr using a five cycle/hour schedule.

NOTE: As an option, a Soxhlet/Dean-Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.4.3.5 Cool and filter the toluene extract through a glass fiber filter into a 500-mL round-bottom flask. Rinse the filter with 10 mL of toluene. Add the rinse to the extract and concentrate the combined toluene solutions to near dryness on a rotary evaporator at 50 °C or using nitrogen evaporation. Proceed with Sec. 11.4.4.

11.4.3.6 Alternatively, fly ash samples may be extracted with a toluene/acetic acid mixture using pressurized fluid extraction (PFE), as described in Method 3545. When using PFE, the HCl pretreatment in Sec. 11.4.3.1 may be omitted.

11.4.4 Transfer the concentrate to a 125-mL separatory funnel using 15 mL of hexane. Rinse the flask with two 5-mL portions of hexane and add the rinses to the funnel. Shake the combined solutions in the separatory funnel for two minutes with 50 mL of 5 percent sodium chloride solution, discard the aqueous layer, and proceed with Sec. 11.5.

11.4.5 Aqueous samples

11.4.5.1 Allow the sample to come to ambient temperature, then mark the water meniscus on the side of the 1-L sample bottle for later determination of the exact sample volume. Add the required acetone diluted sample fortification solution (Sec. 7.9).

11.4.5.2 When the sample is judged to contain 1 percent or more solids, the sample must be filtered through a glass fiber filter that has been rinsed with toluene. If the suspended solids content is too great to filter through the 0.45- μ m filter, centrifuge the sample, decant, and then filter the aqueous phase.

NOTE: Paper mill effluent samples normally contain 0.02%-0.2% solids, and would not require filtration. However, for optimum analytical results, all paper mill effluent samples should be filtered, the isolated solids and filtrate extracted separately, and the extracts recombined.

11.4.5.3 Combine the solids from the centrifuge bottle(s) with the particulates on the filter and with the filter itself and proceed with the Soxhlet extraction as indicated in Secs. 11.4.6.1 through 11.4.6.4.

NOTE: Pressurized fluid extraction has *not* been evaluated for the extraction of the particulate fraction.

Remove and invert the Snyder column and rinse it down into the K-D apparatus with two 1-mL portions of hexane.

11.4.5.4 Pour the aqueous filtrate into a 2-L separatory funnel. Add 60 mL of methylene chloride to the sample bottle, seal and shake for 30 sec to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting.

11.4.5.5 Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation (e.g., glass stirring rod).

11.4.5.6 Collect the methylene chloride in a K-D apparatus (mounted with a 10-mL concentrator tube) by passing the sample extracts through a filter funnel packed with a glass wool plug and 5 g of anhydrous sodium sulfate.

NOTE: As an option, a rotary evaporator may be used in place of the K-D apparatus for the concentration of the extracts.

11.4.5.7 Repeat the extraction twice with fresh 60-mL portions of methylene chloride. After the third extraction, rinse the sodium sulfate with an additional 30 mL of methylene chloride to ensure quantitative transfer. Combine all extracts and the rinse in the K-D apparatus.

NOTE: A continuous liquid-liquid extractor may be used in place of a separatory funnel when experience with a sample from a given source indicates that a serious emulsion problem will result or an emulsion is encountered when using a separatory funnel. Add 60 mL of methylene chloride to the sample bottle, seal, and shake for 30 sec to rinse the inner surface.

Transfer the solvent to the extractor. Repeat the rinse of the sample bottle with an additional 50- to 100-mL portion of methylene chloride and add the rinse to the extractor. Add 200 to 500 mL of methylene chloride to the distilling flask, add sufficient organic-free reagent water (Sec. 7.2) to ensure proper operation, and extract for 24 hr. Allow to cool, then detach the distilling flask. Dry and concentrate the extract as described in Secs. 11.4.5.6 and 11.4.5.8 through 11.4.5.10. Proceed with Sec. 11.4.5.11.

11.4.5.8 Attach a Snyder column and concentrate the extract on a water bath until the apparent volume of the liquid is 5 mL. Remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

11.4.5.9 Remove the Snyder column, add 50 mL of hexane, add the concentrate obtained from the Soxhlet extraction of the suspended solids (Sec. 11.4.5.3), if applicable, re-attach the Snyder column, and concentrate to approximately 5 mL. Add a new boiling chip to the K-D apparatus before proceeding with the second concentration step.

11.4.5.10 Rinse the flask and the lower joint with two 5-mL portions of hexane and combine the rinses with the extract to give a final volume of about 15 mL.

11.4.5.11 Determine the original sample volume by filling the sample bottle to the mark with water and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL. Proceed with Sec. 11.5.

11.4.6 Soil/sediment

11.4.6.1 Add 10 g of anhydrous powdered sodium sulfate to the sample aliquot (10 g or less) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the soil/sodium sulfate mixture in the Soxhlet apparatus on top of a glass wool plug (the use of an extraction thimble is optional).

NOTE: As an option, a Soxhlet/Dean-Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.4.6.2 Add 200 to 250 mL of toluene to the Soxhlet apparatus and reflux for 16 hr. The solvent must cycle completely through the system 5 times per hr.

NOTE: If the dried sample is not of free flowing consistency, more sodium sulfate must be added.

11.4.6.3 Cool and filter the extract through a glass fiber filter into a 500-mL round-bottom flask for evaporation of the toluene. Rinse the filter with 10 mL of toluene, and concentrate the combined fractions to near dryness on a rotary evaporator at 50 °C. Remove the flask from the water bath and allow to cool for 5 min.

11.4.6.4 Transfer the residue to a 125-mL separatory funnel, using 15 mL of hexane. Rinse the flask with two additional portions of hexane, and add the rinses to the funnel. Proceed with Sec. 11.5.

11.4.6.5 Alternatively, soil/sediment samples may be extracted with toluene using pressurized fluid extraction (PFE), as described in Method 3545 or by microwave extraction, as described in Method 3546.

11.5 Cleanup

11.5.1 Acid-base washing

11.5.1.1 Partition the hexane extract against 40 mL of concentrated sulfuric acid. Shake for 2 min. Remove and discard the sulfuric acid layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).

11.5.1.2 Omit this step for the fish sample extract. Partition the extract against 40 mL of 5 percent (w/v) aqueous sodium chloride. Shake for 2 min. Remove and discard the aqueous layer (bottom).

11.5.1.3 Omit this step for the fish sample extract. Partition the extract against 40 mL of 20 percent (w/v) aqueous potassium hydroxide (KOH). Shake for 2 min. Remove and discard the aqueous layer (bottom). Repeat the base washing until no color is visible in the bottom layer (perform a maximum of four base washings). Strong base (KOH) is known to degrade certain PCDDs/PCDFs, so contact time must be minimized.

11.5.1.4 Partition the extract against 40 mL of 5 percent (w/v) aqueous sodium chloride. Shake for 2 min. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a filter funnel containing anhydrous sodium sulfate on a glass wool plug, and collect it in a 50-mL round-bottom flask. Rinse the funnel with the sodium sulfate with two 15-mL portions of hexane, add the rinses to the 50-mL flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35 °C water bath) or nitrogen evaporation, making sure all traces of toluene (when applicable) are removed.

11.5.2 Silica/alumina column cleanup

11.5.2.1 Pack a gravity column (glass, 30 cm x 10.5 mm), fitted with a PTFE stopcock, with silica gel as follows: Insert a glass wool plug into the bottom of the column. Place 1 g of silica gel in the column and tap the column gently to settle the silica gel. Add 2 g of sodium hydroxide-impregnated silica gel, 4 g of sulfuric acid-impregnated silica gel, and 2 g of silica gel. (Secs. 7.3.4, 7.3.5, and 7.3.3) Tap the column gently after each addition. A small positive pressure (5 psi) of clean nitrogen may be used if needed. Elute with 10 mL of hexane and close the stopcock just before exposure of the top layer of silica gel to air. Discard the eluate. Check the column for channeling. If channeling is observed, discard the column. Do not tap the wetted column.

11.5.2.2 Pack a gravity column (glass, 300 mm x 10.5 mm), fitted with a PTFE stopcock, with alumina as follows: Insert a glass wool plug into the bottom of the column. Add a 4 g layer of sodium sulfate. (Sec 7.5) Add a 4 g layer of Woelm® Super 1 neutral alumina. (Sec. 7.3.1) Tap the top of the column gently. Woelm® Super 1 neutral alumina need not be activated or cleaned before use, but it should be stored in a sealed desiccator. Add a 4 g layer of anhydrous sodium sulfate to cover the alumina. Elute with 10 mL hexane and close the stopcock just before exposure of the sodium sulfate layer to air. Discard the eluate. Check the

column for channeling. If channeling is observed, discard the column. Do not tap a wetted column.

NOTE: Alternatively, acidic alumina (Sec. 7.3.2) may be used in place of neutral alumina.

11.5.2.3 Dissolve the residue from Sec. 11.5.1.4 in 2 mL of hexane and apply the hexane solution to the top of the silica gel column. Rinse the flask with enough hexane (3-4 mL) to quantitatively transfer the sample to the surface of the silica gel.

11.5.2.4 Elute the silica gel column with 90 mL of hexane, concentrate the eluate on a rotary evaporator (35 °C water bath) to approximately 1 mL, and apply the concentrate to the top of the alumina column (Sec. 11.5.2.2). Rinse the rotary evaporator flask twice with 2 mL of hexane, and add the rinses to the top of the alumina column.

11.5.2.5 Add 20 mL of hexane to the alumina column and elute until the hexane level is just below the top of the sodium sulfate. Do not discard the eluted hexane, but collect it in a separate flask and store it for later use, as it may be useful in determining where the labeled analytes are being lost if recoveries are not satisfactory.

11.5.2.6 Add 15 mL of 60 percent methylene chloride in hexane (v/v) to the alumina column and collect the eluate in a conical-shaped (15-mL) concentration tube. With a carefully regulated stream of nitrogen, concentrate the 60 percent methylene chloride/hexane fraction to about 2 mL.

11.5.3 Carbon column cleanup

11.5.3.1 Thoroughly mix 9.0 g of activated carbon (Sec. 7.3.7) and 41.0 g of Celite 545® to produce an 18% w/w mixture. Activate the mixture at 130 °C for 6 hr, and store in a desiccator.

NOTE: Check each new batch of the carbon/Celite mixture by adding 50 µL of the calibration verification solution to 950 µL of hexane. Take this solution through the carbon column cleanup step, concentrate to 50 µL and analyze. If the recovery of any of the analytes is less than 80%, this batch of carbon/Celite mixture may not be used.

11.5.3.2 Prepare a 4-inch long glass column by cutting off each end of a 10-mL disposable serological pipet. Fire polish both ends and flare if desired. Insert a glass wool plug at one end of the column, and pack it with 1 g of the carbon/Celite mixture. Insert an additional glass wool plug in the other end.

CAUTION: It is very important that the column be packed properly to ensure that carbon fines are not carried into the eluate. PCDDs/PCDFs will adhere to the carbon fines and greatly reduce recovery. If carbon fines are carried into the eluate, filter the eluate, using a 0.7-µm filter (pre-rinsed with toluene), then proceed to Sec. 11.5.3.6.

11.5.3.3 Rinse the column with:

- 4 mL of toluene
- 2 mL of methylene chloride/methanol/toluene (75:20:5 v/v)
- 4 mL of cyclohexane/methylene chloride (50:50 v/v)

The flow rate should be less than 0.5 mL/min. Discard all the column rinsates.

11.5.3.4 While the column is still wet, transfer the concentrated eluate from Sec. 11.5.2.6 to the prepared carbon column. Rinse the eluate container with two 0.5-mL portions of hexane and transfer the rinses to the carbon column. Elute the column with the following sequence of solvents:

- 10 mL of cyclohexane/methylene chloride (50:50 v/v).
- 5 mL of methylene chloride/methanol/toluene (75:20:5 v/v).

NOTE: The above two eluates may be collected and combined, and used as a check on column efficiency.

11.5.3.5 Once the solvents have eluted through the column, turn the column over, and elute the PCDD/PCDF fraction with 20 mL of toluene, and collect the eluate.

11.5.3.6 Concentrate the toluene fraction to about 1 mL on a rotary evaporator by using a water bath at 50 °C or with nitrogen evaporation. Carefully transfer the concentrate into a 1-mL minivial and, again at elevated temperature (50 °C), reduce the volume to about 100 µL using a stream of nitrogen and a sand bath. Rinse the rotary evaporator flask three times with 300 µL of a solution of 1 percent toluene in methylene chloride, and add the rinses to the concentrate. Add 10 µL of the nonane recovery standard solution (Sec. 7.10) for soil, sediment, water, fish, paper pulp and adipose tissue samples, or 50 µL of the recovery standard solution for sludge, still bottom and fly ash samples. Store the sample at room temperature in the dark.

11.6 Chromatographic/mass spectrometric conditions and data acquisition parameters

11.6.1 Gas chromatograph operating conditions

Column coating:	DB-5
Film thickness:	0.25 µm
Column dimension:	60-m x 0.32 mm
Injector temperature:	270 °C
Splitless valve time:	45 s
Interface temperature:	Function of the final temperature
Temperature program	
Initial temperature:	200 °C
Initial hold time:	2 min
1st temp. ramp:	5 °C/min to 220 °C, hold for 16 min
2nd temp. ramp:	5 °C/min to 235 °C, hold for 7 min
3rd temp. ramp:	5 °C/min to 330 °C, hold for 5 min
Total time:	60 min

11.6.2 Mass spectrometer

11.6.2.1 The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less (Sec. 11.6.3.1). At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. Note that with the exception of the last descriptor (OCDD/OCDF), all descriptors contain 10 ions. The selection (Table 6) of the molecular ions M and M+2 for ^{13}C -HxCDF and ^{13}C -HpCDF rather than M+2 and M+4 (for consistency) was made to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The selection of the lock-mass ion is left to the performing laboratory.

NOTE: At the option of the analyst, the tetra- and pentachlorinated dioxins and furans may be combined into a single descriptor.

11.6.2.2 The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 6. By using a PFK molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). By using peak matching conditions and the aforementioned PFK reference peak, verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 6).

11.6.3 Data acquisition

11.6.3.1 The total cycle time for data acquisition must be ≤ 1 sec. The total cycle time includes the sum of all the dwell times and voltage reset times.

11.6.3.2 Acquire SIM data for all of the ions in the descriptors in Table 6.

11.7 Calibration

11.7.1 Initial calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs and must meet the acceptance criteria in Sec. 11.7.2. Initial calibration is also required if any routine calibration (Sec. 11.7.3) does not meet the required criteria listed in Sec. 11.7.4.

11.7.1.1 At a minimum, all five high-resolution concentration calibration solutions listed in Table 5 must be used for the initial calibration.

11.7.1.2 Tune the instrument with PFK, as described in Sec. 11.6.2.2.

11.7.1.3 Inject 2 μL of the GC column performance check solution (Sec. 7.8) and acquire SIM mass spectral data as described earlier in Sec. 11.6.2. The total cycle time must be ≤ 1 sec. The laboratory must not perform any further analysis until it is demonstrated and documented that the criteria listed in Sec. 9.6.1 were met.

11.7.1.4 By using the same GC (Sec. 11.6.1) and MS (Sec. 11.6.2) conditions that produced acceptable results with the column performance check solution, analyze a 2-μL portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameters.

11.7.1.4.1 The ratio of integrated ion current for the ions appearing in Table 8 (homologous series quantitation ions) must be within the indicated control limits (set for each homologous series) for all unlabeled calibration standards in Table 5.

11.7.1.4.2 The ratio of integrated ion current for the ions belonging to the carbon-labeled internal and recovery standards (Table 5) must be within the control limits stipulated in Table 8.

NOTE: Secs. 11.7.1.4.1 and 11.7.1.4.2 require that 17 ion ratios from Sec. 11.7.1.4.1 and 11 ion ratios from Sec. 11.7.1.4.2 be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.

11.7.1.4.3 For each selected ion current profile (SICP) and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. Measurement of S/N is required for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

11.7.1.4.4 Referring to Table 9, calculate the 17 relative response factors (RF) for unlabeled target analytes [RF(n); n = 1 to 17] relative to their appropriate internal standards (Table 5) and the nine RFs for the ¹³C₁₂-labeled internal standards [RF(is); is = 18 to 26] relative to the two recovery standards (Table 5) according to the following formulae:

$$RF_n = \frac{(A_n^1 + A_n^2) \times Q_{is}}{(A_{is}^1 + A_{is}^2) \times Q_n}$$

$$RF_{is} = \frac{(A_{is}^1 + A_{is}^2) \times Q_{rs}}{(A_{rs}^1 + A_{rs}^2) \times Q_{is}}$$

where:

A_n^1 and A_n^2 = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for unlabeled PCDDs/PCDFs,

A_{is}^1 and A_{is}^2 = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for the labeled internal standards,

A_{rs}^1 and A_{rs}^2 = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for the labeled recovery standards,

Q_{is} = quantity of the internal standard injected (pg),

Q_{rs} = quantity of the recovery standard injected (pg), and

Q_n = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RF_n and RF_{is} are dimensionless quantities; the units used to express Q_{is} , Q_{rs} and Q_n must be the same.

11.7.1.4.5 Calculate the RF values and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RF}_n = \frac{\sum_{j=1}^5 RF_{n(j)}}{5}$$

where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener ($n = 1$ to 17; Table 9), and j is the injection number (or calibration solution number; $j = 1$ to 5).

11.7.1.4.6 The relative response factors to be used for the determination of the concentration of total isomers in a homologous series (Table 9) are calculated as follows:

For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the $\overline{RF}$ used will be the same as the RF determined in Sec. 11.7.1.4.5.

NOTE: The calibration solutions do not contain $^{13}C_{12}$ -OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the $[M+6]^+$ ion of $^{13}C_{12}$ -OCDF from the $[M+2]^+$ ion of OCDD (and $[M+4]^+$ from $^{13}C_{12}$ -OCDF with $[M]^+$ of OCDD). Therefore, the $\overline{RF}$ of OCDF is calculated relative to $^{13}C_{12}$ -OCDD.

For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the $\overline{RF}$ used for those homologous series will be the mean of the RF s calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RF}_k = \frac{\sum_{n=1}^t RF_n}{t}$$

where:

- k = 27 to 30 (Table 9), with 27 = PeCDF; 28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,
- t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution pattern are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

11.7.1.4.7 Relative response factors (RF_m) to be used for the determination of the percent recoveries for the nine internal standards are calculated as follows:

$$RF_m = \frac{A_{is}^m \times Q_{rs}}{Q_{is}^m \times A_{rs}}$$

$$\overline{RF}_m = \frac{\sum_{j=1}^5 RF_{m(j)}}{5}$$

where:

- m = 18 to 26 (congener type) and j = 1 to 5 (injection number),
- A_{is}^m = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for a given internal standard (m = 18 to 26),
- A_{rs} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for the appropriate recovery standard (see Table 5, footnotes),

Q_{rs}, Q_{is}^m = quantities of, respectively, the recovery standard (rs) and a particular internal standard (is = m) injected (pg),

RF_m = relative response factor of a particular internal standard (m) relative to an appropriate recovery standard, as determined from one injection, and

$\overline{RF}_m$ = calculated mean relative response factor of a particular internal standard (m) relative to an appropriate recovery standard, as determined from the five initial calibration injections (j).

11.7.2 Criteria for acceptable calibration -- The criteria listed below for acceptable calibration must be met before sample analyses are performed.

11.7.2.1 The percent relative standard deviations for the mean response factors (RF_n and $\overline{RF}_m$) from the 17 unlabeled standards must not exceed ± 20 percent, and those for the nine labeled reference compounds must not exceed ± 20 percent.

11.7.2.2 The S/N for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10 .

11.7.2.3 The ion abundance ratios (Table 8) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Sec. 11.7.2.1 is met, the analyte-specific RF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RFs will be used for all calculations until the routine calibration criteria (Sec. 11.7.4) are no longer met. At such time, new RF values will be calculated from a new set of injections of the calibration solutions.

11.7.3 Routine calibration (continuing calibration check) -- Routine calibrations must be performed at the beginning of a 12-hr period, after successful mass resolution and GC resolution performance checks. A routine calibration is also required at the end of a 12-hr shift. Inject 2 μ L of the concentration calibration solution HRCC-3 standard (Table 5). By using the same HRGC/HRMS conditions as used in Secs. 11.6.1 and 11.6.2, determine and document an acceptable calibration as provided in Sec. 11.7.4.

11.7.4 Criteria for acceptable routine calibration -- The following criteria must be met before further analysis is performed.

11.7.4.1 The measured RFs [RF_n for the unlabeled standards] obtained during the routine calibration runs must be within ± 20 percent of the mean values established during the initial calibration (Sec. 11.7.1.4.5).

11.7.4.2 The measured RFs [RF_m for the labeled standards] obtained during the routine calibration runs must be within ± 30 percent of the mean values established during the initial calibration (Sec. 11.7.1.4.7).

11.7.4.3 The ion abundance ratios (Table 8) must be within the allowed control limits.

11.7.4.4 If either one of the criteria in Secs. 11.7.4.1 and 11.7.4.2 is not satisfied, repeat one more time. If these criteria are still not satisfied, the entire routine calibration process (Sec. 11.7.3) must be reviewed. If the ion abundance ratio criterion (Sec. 11.7.4.3) is not satisfied, refer to the note in Sec. 11.7.1.4.2 for resolution.

NOTE: An initial calibration must be carried out whenever the HRCC-3, the sample fortification, or the recovery standard solution is replaced by a new solution from a different lot.

11.8 Analysis

11.8.1 Remove the sample or blank extract (from Sec. 11.5.3.6) from storage. With a stream of dry, purified nitrogen, reduce the extract volume to 10 μ L to 50 μ L.

NOTE: A final volume of 20 μ L or more should be used whenever possible. A 10- μ L final volume is difficult to handle, and injection of 2 μ L out of 10 μ L leaves little sample for confirmations and repeat injections, and for archiving.

11.8.2 Inject a 2- μ L aliquot of the extract into the GC, operated under the conditions that have been established to produce acceptable results with the performance check solution (Secs. 9.1.6.1 and 9.6.2).

11.8.3 Acquire SIM data according to Secs. 11.6.2 and 11.6.3. Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (Secs. 11.7.1.4.4 through 11.7.1.4.7). Ions characteristic of polychlorinated diphenyl ethers are included in the descriptors listed in Table 6.

NOTE: The acquisition period must at least encompass the PCDD/PCDF overall retention time window previously determined (Sec. 9.3.1.3). Selected ion current profiles (SICP) for the lock-mass ions (one per mass descriptor) must also be recorded and included in the data package. These SICPs must be true representations of the evolution of the lock-mass ions amplitudes during the HRGC/HRMS run (see Sec. 9.3.2.2 for the proper level of reference compound to be metered into the ion chamber). The analyst may be required to monitor a PFK ion, not as a lock-mass, but as a regular ion, in order to meet this requirement. It is recommended to examine the lock-mass ion SICP for obvious basic sensitivity and stability changes of the instrument during the GC/MS run that could affect the measurements.

11.8.4 Identification criteria -- For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

11.8.4.1 Retention times

11.8.4.1.1 For 2,3,7,8-substituted congeners, which have an isotopically-labeled internal or recovery standard present in the sample extract (this represents a total of 10 congeners including OCDD; Tables 2 and 3), the retention time (RRT; at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 to +3 sec of the isotopically-labeled standard.

11.8.4.1.2 For 2,3,7,8-substituted compounds that do not have an isotopically-labeled internal standard present in the sample

extract (this represents a total of six congeners; Table 3), the retention time must fall within 0.005 retention time units of the relative retention times measured in the routine calibration. Identification of OCDF is based on its retention time relative to $^{13}\text{C}_{12}$ -OCDD as determined from the daily routine calibration results.

11.8.4.1.3 For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution (Sec. 9.6.2).

11.8.4.1.4 The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach maximum simultaneously (± 2 sec).

11.8.4.1.5 The ion current responses for both ions used for the labeled standards (e.g., for $^{13}\text{C}_{12}$ -TCDD: m/z 331.9368 and m/z 333.9339) must reach maximum simultaneously (± 2 sec).

NOTE: The analyst is required to verify the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF (Sec. 9.3.1.3) in the SICPs of the daily performance checks. Should either compound be missing, the analyst must take corrective action as it may indicate a potential problem with the ability to detect all of the PCDDs/PCDFs.

11.8.4.2 Ion abundance ratios

The integrated ion currents for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Secs. 11.7.1.4.1 and 11.7.1.4.2 and Table 8 for details.

11.8.4.3 Signal-to-noise ratio

All ion current intensities must be ≤ 2.5 times noise level for positive identification of an unlabeled PCDD/PCDF compound or a group of coeluting isomers. Figure 6 describes the procedure to be followed for the determination of the S/N. Labeled analytes must have an $S/N \geq 10$.

11.8.4.4 Polychlorinated diphenyl ether interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a $S/N \leq 2.5$ is detected at the same retention time (± 2 sec) in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

11.9 Calculations

11.9.1 For gas chromatographic peaks that have met the criteria outlined in Sec. 11.8.4, calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times W \times \overline{RF}_n}$$

where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) in pg/g,

A_x = sum of the integrated ion abundances of the quantitation ions (Table 6) for unlabeled PCDDs/PCDFs,

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standards,

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

W = weight, in g, of the sample (solid or organic liquid), or volume in mL of an aqueous sample, and

$\overline{RF}_n$ = calculated mean relative response factor for the analyte ($\overline{RF}_n$ with $n = 1$ to 17; Sec. 11.7.1.4.5).

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs, $\overline{RF}_n$ is the value calculated using the equation in Sec. 11.7.1.4.5. However, if it is a non-2,3,7,8-substituted congener, the $RF(k)$ value is the one calculated using the equation in Sec. 11.7.1.4.6 (RF_k , for $k = 27$ to 30).

11.9.2 Calculate the percent recovery of the nine internal standards measured in the sample extract, using the formula:

$$\text{percent recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times \overline{RF}_m} \times 100$$

where:

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (see Table 5, footnotes),

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

Q_{rs} = quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$\overline{RF}_m$ = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 5, footnotes) recovery standard. This represents the mean obtained in Sec. 11.7.1.4.7 ($\overline{RF}_m$ with $m = 18$ to 26).

NOTE: For human adipose tissue, adjust the percent recoveries by adding 1 percent to the calculated value to compensate for the 1 percent of the extract diverted for the lipid determination.

11.9.3 If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limits (MCL) listed in Table 1 (e.g., 200 pg/ μ L for TCDD in soil), the linear range of response versus concentration may have been exceeded, and a second analysis of the sample (using a one-tenth aliquot) should be undertaken. The volumes of the internal and recovery standard solutions should remain the same as described for the sample preparation (Secs. 11.0).

If a smaller sample size would not be representative of the entire sample, one of the following options is recommended:

- (1) Re-extract an additional aliquot of sufficient size to insure that it is representative of the entire sample. Spike it with a higher concentration of internal standard. Prior to GC/MS analysis, dilute the sample so that it has a concentration of internal standard equivalent to that present in the calibration standard. Then, analyze the diluted extract.
- (2) Re-extract an additional aliquot of sufficient size to insure that it is representative of the entire sample. Spike it with a higher concentration of internal standard. Immediately following extraction, transfer the sample to a volumetric flask and dilute to known volume. Remove an appropriate aliquot and proceed with cleanup and analysis.
- (3) Use the original analysis data to quantitate the internal standard recoveries. Respike the original extract (note that no additional cleanup is necessary) with 100 times the usual quantity of internal standards. Dilute the re-spiked extract by a factor of 100. Reanalyze the diluted sample using the internal standard recoveries calculated from the initial analysis to correct the results for losses during isolation and cleanup.

11.9.4 The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value must be specified in the report. If an isomer is not detected, use zero (0) in this calculation.

11.9.5 Sample specific estimated quantitation limit -- The sample specific estimated quantitation limit (EQL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EQL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

11.9.5.1 Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

Use the expression below to calculate an EDL for each 2,3,7,8-substituted PCDD/PCDF that does not have a response with $S/N \geq 2.5$. The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the internal standard (if the congener possesses an internal standard) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a ^{13}C -labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that peak height. Use the formula:

$$\text{EQL} = \frac{2.5 \times H_x \times Q_{is}}{H_{is} \times W \times \overline{RF}_n}$$

where:

EQL = estimated quantitation limit for homologous 2,3,7,8-substituted PCDDs/PCDFs.

H_x = sum of the height of the noise level for each quantitation ion (Table 6) for the unlabeled PCDDs/PCDFs, measured as shown in Figure 6.

H_{is} = sum of the height of the noise level for each quantitation ion (Table 6) for the labeled internal standard, measured as shown in Figure 6.

W , $\overline{RF}_n$, and Q_{is} retain the same meanings as defined in Sec. 11.9.1.

11.9.5.2 Estimated maximum possible concentration -- An estimated maximum possible concentration (EMPC) is calculated for 2,3,7,8-substituted isomers that are characterized by a response with an S/N of at least 2.5 for both the quantitation ions, and meet all of the identification criteria in Sec. 11.8.4 except the ion abundance ratio criteria or when a peak representing a PCDPE has been detected. An EMPC is a worst-case estimate of the concentration. Calculate the EMPC according to the expression shown in Sec. 11.9.1.

11.9.6 The relative percent difference (RPD) of any duplicate sample results is calculated as follows:

$$\text{RPD} = \frac{|S_1 - S_2|}{\frac{S_1 + S_2}{2}} \times 100$$

where S_1 and S_2 represent sample and duplicate sample results.

11.9.7 The 2,3,7,8-TCDD toxicity equivalents (TE) of PCDDs and PCDFs present in the sample are calculated, if requested by the data user, according to the method recommended by the Chlorinated Dioxins Workgroup (CDWG) of the EPA and the Center for Disease Control (CDC). This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the fifteen 2,3,7,8-substituted PCDDs and PCDFs (Table 3) and to OCDD and OCDF, as shown in Table 10. The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 10. The exclusion of other homologous series such as mono-, di-, and tri- chlorinated dibenzodioxins and dibenzofurans does not mean that they are non-toxic. However, their toxicity, as known at this time, is much lower than the toxicity of the compounds listed in Table 10. The above procedure for calculating the 2,3,7,8-TCDD toxicity equivalents is not claimed by the CDWG to be based on a thoroughly established scientific foundation. The procedure, rather, represents a "consensus recommendation on science policy." Since the procedure may be changed in the future, reporting requirements for PCDD and PCDF data would still include the reporting of the analyte concentrations of the PCDD/PCDF congener as calculated in Secs. 11.9.1 and 11.9.4.

11.9.8 Two GC column TEF determination

11.9.8.1 The concentration of 2,3,7,8-TCDD (see note below), is calculated from the analysis of the sample extract on the 60-m DB-5 (or equivalent) fused-silica capillary column. The experimental conditions remain the same as the conditions described previously in Sec. 11.8, and the calculations are performed as outlined in Sec. 11.9. The chromatographic separation between the 2,3,7,8-TCDD and its close eluters (1,2,3,7/1,2,3,8-TCDD and 1,2,3,9-TCDD) must be equal or less than 25 percent valley.

11.9.8.2 The concentration of the 2,3,7,8-TCDF is obtained from the analysis of the sample extract on the 30-m DB-225 (or equivalent) fused-silica capillary column. However, the GC/MS conditions must be altered so that: (1) only the first three descriptors (i.e., tetra-, penta-, and hexachlorinated congeners) of Table 6 are used; and (2) the switching time between descriptor 2 (pentachlorinated congeners) and descriptor 3 (hexachlorinated congeners) takes place following the elution of $^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDD. The concentration calculations are performed as outlined in Sec. 11.9. The chromatographic separation between the 2,3,7,8-TCDF and its close eluters (2,3,4,7-TCDF and 1,2,3,9-TCDF) must be equal or less than 25 percent valley.

NOTE: The confirmation and quantitation of 2,3,7,8-TCDD (Sec. 11.9.8.1) may be accomplished on the SP-2330 GC column instead of the DB-5 column, provided the criteria listed in Sec. 9.3.1 are met and the requirements described in Sec. 9.6.2 are followed.

11.9.8.3 For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance and signal-to-noise ratio criteria described in Secs. 11.8.4.2 and 11.8.4.3, respectively. In addition, the retention time identification criterion described in Sec. 11.8.4.1.1 applies here for congeners for which a carbon-labeled analogue is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogues are available must fall within 0.006 units of the carbon-labeled standard RRT. Experimentally,

this is accomplished by using the attributions described in Table 11 and the results from the routine calibration run on the SP-2330 column.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 See Sec. 11.9 and Method 8000 for information on data analysis and calculations.

12.2 Results need to be reported in units commensurate with their intended use and all dilutions need to be taken into account when computing final results.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance goals for users of the methods. Instead, performance goals should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 Table 12 provides data from a comparison of the Soxhlet extraction and pressurized fluid extraction of samples of ground chimney brick. The data are taken from Reference 8 and are provided for guidance purposes only.

13.3 Table 13 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction of samples of urban dust. The data are taken from Reference 8 and are provided for guidance purposes only.

13.4 Table 14 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction of samples of fly ash. PFE data are provided for samples that were pretreated with an HCl wash and for samples that were not pretreated, but were extracted with a mixture of toluene and acetic acid. The data are taken from Reference 8 and are provided for guidance purposes only.

13.5 Table 15 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction of a soil sample (EC-2) from the National Water Research Institute (Burlington, Ontario, Canada) that contained high levels of PCDDs and PCDFs. The data are taken from Reference 8 and are provided for guidance purposes only.

13.6 Table 16 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction of a sediment sample (HS-2) from the National Research Council Institute for Marine Biosciences (Halifax, Nova Scotia, Canada) that contained low levels of PCDDs and PCDFs. The data are taken from Reference 8 and are provided for guidance purposes only.

13.7 Table 17 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction for two field-contaminated sediment samples. The data are taken from Reference 8 and are provided for guidance purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution

prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, <http://www.acs.org>.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. D. G. Patterson, J. S. Holler, D. F. Grote, L. R. Alexander, C. R. Lapeza, R. C. O'Connor and J. A. Liddle, "Control of Interferences in the Analysis of Human Adipose Tissue for 2,3,7,8-Tetrachlorodibenzo-*p*-dioxin," *Environ. Toxicol. Chem.* 5, 355-360 (1986).
2. Y. Tondeur and W. F. Beckert, "Method 8290: Analytical Procedures and Quality Assurance for Multimedia Analysis of Polychlorinated Dibenzo-*p*-Dioxins and Dibenzofurans by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry," U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Las Vegas, NV.
3. "Carcinogens -- Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.
4. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).
5. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)
6. Y. Tondeur, W. J. Niederhut, S. R. Missler, and J. E. Campana, "Hybrid HRGC/MS/MS Method for the Characterization of Tetrachlorinated Dibenzo-*p*-dioxins in Environmental Samples," *Mass Spectrom*, 14, 449-456 (1987).
7. USEPA National Dioxin Study -- Phase II, "Analytical Procedures and Quality Assurance Plan for the Determination of PCDD/PCDF in Fish," EPA-Duluth, October 26, 1987.

8. B. E. Richter, J. L. Ezzell, D. E. Knowles, and F. Hoefler, "Extraction of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Environmental Samples Using Accelerated Solvent Extraction (ASE)," *Chemosphere*, 34 (5-7), 975-987, 1997.

17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

DRAFT

TABLE 1
TYPES OF MATRICES, SAMPLE SIZES, AND 2,3,7,8-TCDD-BASED
METHOD CALIBRATION LIMITS (PARTS PER TRILLION)

	Water	Soil Sediment Paper Pulp ^b	Fly Ash	Fish Tissue ^c	Human Adipose Tissue	Sludge Fuel Oil	Still Bottom
Lower MCL ^a	0.01	1.0	1.0	1.0	1.0	5.0	10
Upper MCL ^a	2	200	200	200	200	1000	2000
Sample Weight (g)	1000	10	10	20	10	2	1
IS Spiking Level (ppt)	1	100	100	100	100	500	1000
Final Ext. Vol. (µL) ^d	10-50	10-50	50	10-50	10-50	50	50

^a For other congeners multiply the values by 1 for TCDF/PeCDD/PeCDF, by 2.5 for HxCDD/HxCDF/HpCDD/HpCDF, and by 5 for OCDD/OCDF.

^b Sample dewatered according to Sec. 8.5.

^c One half of the extract from the 20 g sample is used for determination of lipid content (Sec. 11.2.2).

^d See Sec. 11.8.1.

NOTE: Chemical reactor residues are treated as still bottoms, if their appearances so suggest.

TABLE 2
COMPOSITION OF THE SAMPLE FORTIFICATION
AND RECOVERY STANDARD SOLUTIONS^a

Analyte	Sample Fortification Solution Concentration (pg/ μ L)	Recovery Standard Solution Concentration (pg/ μ L)
¹³ C ₁₂ -2,3,7,8-TCDD	10	--
¹³ C ₁₂ -2,3,7,8-TCDF	10	--
¹³ C ₁₂ -1,2,3,4-TCDD	--	50
¹³ C ₁₂ -1,2,3,7,8-PeCDD	10	--
¹³ C ₁₂ -1,2,3,7,8-PeCDF	10	--
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	25	--
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	25	--
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD	--	50
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	25	--
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	25	--
¹³ C ₁₂ -OCDD	50	--

^aThese solutions should be made freshly every day in nonane or other appropriate solvent because of the possibility of adsorptive losses to glassware. If these solutions are to be kept for more than one day, then the sample fortification solution concentrations should be increased ten fold, and the recovery standard solution concentrations should be doubled. Corresponding adjustments of the spiking volumes must then be made.

TABLE 3
THE FIFTEEN 2,3,7,8-SUBSTITUTED PCDD AND PCDF CONGENERS

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDF(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF

* The ¹³C-labeled analogue is used as an internal standard.

+ The ¹³C-labeled analogue is used as a recovery standard.

TABLE 4

ISOMERS OF CHLORINATED DIOXINS AND FURANS AS A FUNCTION
OF THE NUMBER OF CHLORINE ATOMS

Number of Chlorine Atoms	Number of Dioxin Isomers	Number of 2,3,7,8-Dioxins	Number of Furan Isomers	Number of 2,3,7,8-Furans
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	75	7	135	10

TABLE 5
HIGH-RESOLUTION CONCENTRATION CALIBRATION SOLUTIONS

Analyte	Concentration (pg/μL)				
Unlabeled Analytes	5	4	3	2	1
2,3,7,8-TCDD	200	50	10	2.5	1
2,3,7,8-TCDF	200	50	10	2.5	1
1,2,3,7,8-PeCDD	500	125	25	6.25	2.5
1,2,3,7,8-PeCDF	500	125	25	6.25	2.5
2,3,4,7,8-PeCDF	500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDD	500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDD	500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDD	500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDF	500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDF	500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDF	500	125	25	6.25	2.5
2,3,4,6,7,8-HxCDF	500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDD	500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDF	500	125	25	6.25	2.5
1,2,3,4,7,8,9-HpCDF	500	125	25	6.25	2.5
OCDD	1,000	250	50	12.5	5
OCDF	1,000	250	50	12.5	5
<u>Internal Standards</u>					
¹³ C ₁₂ -2,3,7,8-TCDD	50	50	50	50	50
¹³ C ₁₂ -2,3,7,8-TCDF	50	50	50	50	50
¹³ C ₁₂ -1,2,3,7,8-PeCDD	50	50	50	50	50
¹³ C ₁₂ -1,2,3,7,8-PeCDF	50	50	50	50	50
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	125	125	125	125	125
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	125	125	125	125	125
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	125	125	125	125	125
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	125	125	125	125	125
¹³ C ₁₂ -OCDD	250	250	250	250	250
<u>Recovery Standards</u>					
¹³ C ₁₂ -1,2,3,4-TCDD ^(a)	50	50	50	50	50
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD ^(b)	125	125	125	125	125

(a) Used for recovery determinations of TCDD, TCDF, PeCDD and PeCDF internal standards.

(b) Used for recovery determinations of HxCDD, HxCDF, HpCDD, HpCDF and OCDD internal standards.

TABLE 6

IONS MONITORED FOR HRGC/HRMS ANALYSIS OF PCDDS/PCDFS

Descriptor	Accurate Mass ^a	Ion ID	Elemental Composition	Analyte
1	303.9016	M	C ₁₂ H ₄ ³⁵ Cl ₄ O	TCDF
	305.8987	M+2	C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO	TCDF
	315.9419	M	¹³ C ₁₂ H ₄ ³⁵ Cl ₄ O	TCDF (S)
	317.9389	M+2	¹³ C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO	TCDF (S)
	319.8965	M	C ₁₂ H ₄ ³⁵ Cl ₄ O ₂	TCDD
	321.8936	M+2	C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO ₂	TCDD
	331.9368	M	¹³ C ₁₂ H ₄ ³⁵ Cl ₄ O ₂	TCDD (S)
	333.9338	M+2	¹³ C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO ₂	TCDD (S)
	375.8364	M+2	C ₁₂ H ₄ ³⁵ Cl ₅ ³⁷ ClO	HxCDFE
2	[354.9792]	LOCK	C ₉ F ₁₃	PFK
	339.8597	M+2	C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO	PeCDF
	341.8567	M+4	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O	PeCDF
	351.9000	M+2	¹³ C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO	PeCDF (S)
	353.8970	M+4	¹³ C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O	PeCDF (S)
	355.8546	M+2	C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO ₂	PeCDD
	357.8516	M+4	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O ₂	PeCDD
	367.8949	M+2	¹³ C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO ₂	PeCDD (S)
	369.8919	M+4	¹³ C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O ₂	PeCDD (S)
3	409.7974	M+2	C ₁₂ H ₃ ³⁵ Cl ₆ ³⁷ ClO	HpCDFE
	[354.9792]	LOCK	C ₉ F ₁₃	PFK
	373.8208	M+2	C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO	HxCDF
	375.8178	M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O	HxCDF
	383.8639	M	¹³ C ₁₂ H ₂ ³⁵ Cl ₆ O	HxCDF (S)
	385.8610	M+2	¹³ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO	HxCDF (S)
	389.8156	M+2	C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO ₂	HxCDD
	391.8127	M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O ₂	HxCDD
	401.8559	M+2	¹³ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO ₂	HxCDD (S)
	403.8529	M+4	¹³ C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O ₂	HxCDD (S)

TABLE 6 (Continued)

Descriptor	Accurate Mass ^a	Ion ID	Elemental Composition	Analyte
4	445.7555	M+4	C ₁₂ H ₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O	OCDPE
	[430.9728]	LOCK	C ₉ F ₁₇	PFK
	407.7818	M+2	C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO	HpCDF
	409.7788	M+4	C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O	HpCDF
	417.8250	M	¹³ C ₁₂ H ³⁵ Cl ₇ O	HpCDF (S)
	419.8220	M+2	¹³ C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO	HpCDF
	423.7767	M+2	C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO ₂	HpCDD
	425.7737	M+4	C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O ₂	HpCDD
	435.8169	M+2	¹³ C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO ₂	HpCDD (S)
	437.8140	M+4	¹³ C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O ₂	HpCDD (S)
	479.7165	M+4	C ₁₂ H ³⁵ Cl ₇ ³⁷ Cl ₂ O	NCDPE
	[430.9728]	LOCK	C ₉ F ₁₇	PFK
5	441.7428	M+2	C ₁₂ ³⁵ Cl ₇ ³⁷ ClO	OCDF
	443.7399	M+4	C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O	OCDF
	457.7377	M+2	C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂	OCDD
	459.7348	M+4	C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂	OCDD
	469.7780	M+2	¹³ C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂	OCDD (S)
	471.7750	M+4	¹³ C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂	OCDD (S)
	513.6775	M+4	C ₁₂ ³⁵ Cl ₈ ³⁷ Cl ₂ O	DCDPE
	[442.9728]	LOCK	C ₁₀ F ₁₇	PFK

S = internal/recovery standard

^a The following nuclidic masses were used:

H	=	1.007825	O	=	15.994915
C	=	12.000000	³⁵ Cl	=	34.968853
¹³ C	=	13.003355	³⁷ Cl	=	36.965903
F	=	18.9984			

TABLE 7

PCDD AND PCDF CONGENERS PRESENT IN THE GC PERFORMANCE EVALUATION
SOLUTION AND USED FOR DEFINING THE HOMOLOGUE GC RETENTION TIME
WINDOWS ON A 60-M DB-5 COLUMN

# Chlorine Atoms	PCDD Positional Isomer		PCDF Positional Isomer	
	First Eluter	Last Eluter	First Eluter	Last Eluter
4 ^a	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,4,6,7,9/1,2,4,6,8,9	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,8	1,2,3,4,7,8,9
8	1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9	

^aIn addition to these two TCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, ¹³C₁₂-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present as a check of column resolution.

TABLE 8
THEORETICAL ION ABUNDANCE RATIOS AND THEIR CONTROL LIMITS
FOR PCDDS AND PCDFS

# Chlorine Atoms	Ion Type	Theoretical Abundance Ratio	Control Limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6 ^(a)	M/M+2	0.51	0.43	0.59
7 ^(b)	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

^aUsed only for ¹³C-HxCDF (IS).

^bUsed only for ¹³C-HpCDF (IS).

TABLE 9

RELATIVE RESPONSE FACTOR [RF (NUMBER)] ATTRIBUTIONS

Number	Specific Congener Name
1	2,3,7,8-TCDD (and total TCDDs)
2	2,3,7,8-TCDF (and total TCDFs)
3	1,2,3,7,8-PeCDD (and total PeCDDs)
4	1,2,3,7,8-PeCDF
5	2,3,4,7,8-PeCDF
6	1,2,3,4,7,8-HxCDD
7	1,2,3,6,7,8-HxCDD
8	1,2,3,7,8,9-HxCDD
9	1,2,3,4,7,8-HxCDF
10	1,2,3,6,7,8-HxCDF
11	1,2,3,7,8,9-HxCDF
12	2,3,4,6,7,8-HxCDF
13	1,2,3,4,6,7,8-HpCDD (and total HpCDDs)
14	1,2,3,4,6,7,8-HpCDF
15	1,2,3,4,7,8,9-HpCDF
16	OCDD
17	OCDF
18	¹³ C ₁₂ -2,3,7,8-TCDD
19	¹³ C ₁₂ -2,3,7,8-TCDF
20	¹³ C ₁₂ -1,2,3,7,8-PeCDD
21	¹³ C ₁₂ -1,2,3,7,8-PeCDF
22	¹³ C ₁₂ -1,2,3,6,7,8-HxCDD
23	¹³ C ₁₂ -1,2,3,4,7,8-HxCDF
24	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD
25	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF
26	¹³ C ₁₂ -OCDD
27	Total PeCDFs
28	Total HxCDFs
29	Total HxCDDs
30	Total HpCDFs

TABLE 10

2,3,7,8-TCDD TOXICITY EQUIVALENCY FACTORS (TEFs) FOR THE POLYCHLORINATED
DIBENZODIOXINS AND DIBENZOFURANS

Analyte	TEF ^a
2,3,7,8-TCDD	1.00
1,2,3,7,8-PeCDD	0.50
1,2,3,6,7,8-HxCDD	0.10
1,2,3,7,8,9-HxCDD	0.10
1,2,3,4,7,8-HxCDD	0.10
1,2,3,4,6,7,8-HpCDD	0.01
1,2,3,4,6,7,8,9-OCDD	0.001
2,3,7,8-TCDF	0.1
1,2,3,7,8-PeCDF	0.05
2,3,4,7,8-PeCDF	0.5
1,2,3,6,7,8-HxCDF	0.1
1,2,3,7,8,9-HxCDF	0.1
1,2,3,4,7,8-HxCDF	0.1
2,3,4,6,7,8-HxCDF	0.1
1,2,3,4,6,7,8-HpCDF	0.01
1,2,3,4,7,8,9-HpCDF	0.01
1,2,3,4,6,7,8,9-OCDF	0.001

^aTaken from "Interim Procedures for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-*p*-Dioxin and -Dibenzofurans (CDDs and CDFs) and 1989 Update", (EPA/625/3-89/016, March 1989).

TABLE 11

ANALYTE RELATIVE RETENTION TIME REFERENCE ATTRIBUTIONS

Analyte	Analyte RRT Reference ^a
1,2,3,4,7,8-HxCDD	¹³ C ₁₂ -1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	¹³ C ₁₂ -1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	¹³ C ₁₂ -1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	¹³ C ₁₂ -1,2,3,4,7,8-HxCDF

^aThe retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to ¹³C₁₂-1,2,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to ¹³C₁₂-1,2,3,4,6,7,8-HpCDF.

TABLE 12

COMPARISON OF SOXHLET AND PRESSURIZED FLUID EXTRACTION (PFE)
FOR EXTRACTION OF GROUND CHIMNEY BRICK

Analyte	Soxhlet (n=1) (ng/kg)	PFE (n=2)* (ng/kg)
2,3,7,8-TCDD	6	6
1,2,3,7,8-PeCDD	52	57
1,2,3,4,7,8-HxCDD	46	52
1,2,3,6,7,8-HxCDD	120	130
1,2,3,7,9,9-HxCDD	97	1000
1,2,3,4,6,7,8-HpCDD	1000	820
OCDD	2900	2600
2,3,7,8-TCDF	160	180
1,2,3,7,8 (+ 1,2,3,4,8)-PeCDF	430	470
2,3,4,7,9-PeCDF	390	390
1,2,3,4,7,8 (+ 1,2,3,4,7,9)-HxCDF	1100	1100
1,2,3,6,7,8-HxCDF	540	570
2,3,4,6,7,8-HxCDF	400	360
1,2,3,7,8,9-HxCDF	42	42
1,2,3,4,6,7,8-HpCDF	2100	2000
1,2,3,4,7,8,9-HpCDF	140	120
OCDF	2000	2000
Total TCDD	440	530
Total PeCDD	900	940
Total HxCDD	1800	2000
Total HpCDD	2000	2100
Total TCDF	2300	2600
Total PeCDF	4100	4300
Total HxCDF	4700	4700
Total HpCDF	2800	2600

* Sum of two extractions of each sample
Data from Reference 8

TABLE 13

COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF URBAN DUST

Analyte	Soxhlet (n=1) (ng/kg)	PFE (n=2)* (ng/kg)
2,3,7,8-TCDD	3.3	3.2
1,2,3,7,8-PeCDD	11.8	13.1
1,2,3,4,7,8-HxCDD	9.8	8.0
1,2,3,6,7,8-HxCDD	11.5	9.5
1,2,3,7,9,9-HxCDD	ND (8)	ND (8)
1,2,3,4,6,7,8-HpCDD	113	107
OCDD	445	314
2,3,7,8-TCDF	12.5	18.6
1,2,3,7,8 (+ 1,2,3,4,8)-PeCDF	9.9	12.0
2,3,4,7,9-PeCDF	13.9	18.1
1,2,3,4,7,8 (+ 1,2,3,4,7,9)-HxCDF	18.7	23.7
1,2,3,6,7,8-HxCDF	10.7	15.8
2,3,4,6,7,8-HxCDF	3.3	8.7
1,2,3,7,8,9-HxCDF	ND (2)	ND (2)
1,2,3,4,6,7,8-HpCDF	13.2	29.4
1,2,3,4,7,8,9-HpCDF	ND (3)	ND (3)
OCDF	ND (10)	ND (10)
Total TCDD	182	325
Total PeCDD	175	281
Total HxCDD	86.7	81.7
Total HpCDD	221	217
Total TCDF	333	419
Total PeCDF	146	179
Total HxCDF	65.9	122
Total HpCDF	13.2	29.4

ND = Not detected, with quantitation limit given in parentheses

* Sum of two extractions of each sample

Data from Reference 8

TABLE 14

COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF FLY ASH
(with and without HCl pretreatment for PFE)

Analyte	Soxhlet (n=1) with HCl (µg/kg)†	PFE (n=2)* with HCl (µg/kg)†	PFE (n=2)* w/o HCl (µg/kg)‡
2,3,7,8-TCDD	0.32	0.36	0.28
1,2,3,7,8-PeCDD	1.6	2.1	1.7
1,2,3,4,7,8-HxCDD	1.2	1.4	1.2
1,2,3,6,7,8-HxCDD	2.4	2.7	2.4
1,2,3,7,9,9-HxCDD	2.4	2.3	2.2
1,2,3,4,6,7,8-HpCDD	8.2	9.6	8.1
OCDD	11.4	12.8	10.6
2,3,7,8-TCDF	3.7	4.3	3.4
1,2,3,7,8 (+ 1,2,3,4,8)-PeCDF	4.2	4.6	3.9
2,3,4,7,9-PeCDF	5.6	6.6	5.8
1,2,3,4,7,8 (+ 1,2,3,4,7,9)-HxCDF	7.8	8.7	5.4
1,2,3,6,7,8-HxCDF	7.2	8.5	5.3
2,3,4,6,7,8-HxCDF	6.6	7.2	4.5
1,2,3,7,8,9-HxCDF	0.43	0.56	0.30
1,2,3,4,6,7,8-HpCDF	18.0	17.6	16.8
1,2,3,4,7,8,9-HpCDF	2.3	2.4	2.0
OCDF	13.5	15.8	13.9
Total TCDD	12.0	12.4	10.5
Total PeCDD	16.6	20.5	16.2
Total HxCDD	38.2	42.4	36.7
Total HpCDD	15.0	19.8	16.0
Total TCDF	60.5	67.5	56.1
Total PeCDF	83.5	87.3	77.4
Total HxCDF	65.2	73.5	46.1
Total HpCDF	28.1	32.2	26.5

† Fly ash was pretreated with HCl, followed by a water rinse, and extracted with toluene.

‡ These samples received no HCl pretreatment, and were extracted with a mixture of toluene and acetic acid.

* Sum of two extractions of each sample
Data from Reference 8

TABLE 15

COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF SOIL (EC-2)

Analyte	Soxhlet Results (n=10)		PFE Results (n=2)	
	ng/kg	% RSD	ng/kg	% RSD
2,3,7,8-TCDD	270	9.1	270	0.0
1,2,3,7,8-PeCDD	24	12	22	3.3
1,2,3,4,7,8-HxCDD	23	8.3	24	3.0
1,2,3,6,7,8-HxCDD	83	3.6	87	0.8
1,2,3,7,9,9-HxCDD	60	6.2	57	7.4
1,2,3,4,6,7,8-HpCDD	720	6.7	720	1.0
OCDD	4000	6.2	4200	0.0
2,3,7,8-TCDF *	100	7.3	82	2.6
1,2,3,7,8 (+ 1,2,3,4,8)-PeCDF	39	14	36	3.9
2,3,4,7,9-PeCDF	62	5.5	60	0.0
1,2,3,4,7,8 (+ 1,2,3,4,7,9)-HxCDF	740	5.3	690	0.0
1,2,3,6,7,8-HxCDF	120	6.2	120	0.0
2,3,4,6,7,8-HxCDF	45	9.0	60	1.2
1,2,3,7,8,9-HxCDF	4.9	31	5.3	15
1,2,3,4,6,7,8-HpCDF	2600	6.7	2500	0.0
1,2,3,4,7,8,9-HpCDF	160	5.5	160	0.0
OCDF	7800	8.3	7000	3.1
Total TCDD	430	9.7	370	1.9
Total PeCDD	300	3.7	280	7.7
Total HxCDD	720	5.8	690	2.0
Total HpCDD	1300	7.0	1300	0.0
Total TCDF	620	12	380	19
Total PeCDF	820	9.4	710	7.0
Total HxCDF	1900	5.7	1900	0.0
Total HpCDF	3800	8.2	3900	3.6

* Single-column analysis only, may include contributions from other isomers that may co-elute.
Data from Reference 8

TABLE 16

COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF SEDIMENT (HS-2)

Analyte	Soxhlet Results (n=10)		PFE Results (n=2)	
	ng/kg	% RSD	ng/kg	% RSD
2,3,7,8-TCDD	ND (1)	--	ND (1)	--
1,2,3,7,8-PeCDD	1.6	4.6	ND (1)	--
1,2,3,4,7,8-HxCDD	4.5	4.8	5.2	11
1,2,3,6,7,8-HxCDD	19	4.3	21	0.0
1,2,3,7,9,9-HxCDD	24	4.3	28	2.6
1,2,3,4,6,7,8-HpCDD	1200	8.1	1300	0.0
OCDD	6500	4.2	7100	0.0
2,3,7,8-TCDF *	8.5	11	6.6	5.4
1,2,3,7,8 (+ 1,2,3,4,8)-PeCDF	1.9	17	2.0	0.0
2,3,4,7,9-PeCDF	3.7	7.9	3.7	3.8
1,2,3,4,7,8 (+ 1,2,3,4,7,9)-HxCDF	17	7.3	17	4.3
1,2,3,6,7,8-HxCDF	3.7	5.6	4.0	5.4
2,3,4,6,7,8-HxCDF	3.7	18	4.4	3.2
1,2,3,7,8,9-HxCDF	ND (1)	--	ND (1)	--
1,2,3,4,6,7,8-HpCDF	91	1.6	96	3.7
1,2,3,4,7,8,9-HpCDF	5.2	6.7	5.3	6.7
OCDF	300	3.8	280	2.6
Total TCDD	3.9	14	2.5	34
Total PeCDD	17	7.8	10	10
Total HxCDD	510	5.6	570	1.3
Total HpCDD	4700	8.3	5100	11
Total TCDF	39	11	24	3.0
Total PeCDF	33	13	28	0.0
Total HxCDF	89	3.2	87	12
Total HpCDF	293	3.3	310	0.0

* Single-column analysis only, may include contributions from other isomers that may co-elute.

. ND = Not detected, with quantitation limit given in parentheses

. Data from Reference 8

TABLE 17

COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF
CONTAMINATED SEDIMENTS

Analyte	Hamilton Harbor		Parrots Bay	
	Soxhlet	PFE	Soxhlet	PFE
2,3,7,8-TCDD	3.7	3.1	19	19
1,2,3,7,8-PeCDD	5.1	5.4	8.3	6.0
1,2,3,4,7,8-HxCDD	6.4	7.2	8.6	6.7
1,2,3,6,7,8-HxCDD	27	26	26	17
1,2,3,7,9,9-HxCDD	20	28	24	18
1,2,3,4,6,7,8-HpCDD	460	430	280	250
OCDD	3100	3100	1900	1600
2,3,7,8-TCDF *	61	44	80	48
1,2,3,7,8 (+ 1,2,3,4,8)-PeCDF	14	14	ND (20)	9.8
2,3,4,7,9-PeCDF	26	25	22	14
1,2,3,4,7,8 (+ 1,2,3,4,7,9)-HxCDF	27	37	79	59
1,2,3,6,7,8-HxCDF	17	16	ND (20)	15
2,3,4,6,7,8-HxCDF	14	14	21	11
1,2,3,7,8,9-HxCDF	ND (2)	1.6	4.9	ND (1)
1,2,3,4,6,7,8-HpCDF	130	130	270	220
1,2,3,4,7,8,9-HpCDF	14	13	17	12
OCDF	270	210	510	370
Total TCDD	50	14	39	48
Total PeCDD	63	15	87	66
Total HxCDD	220	180	230	200
Total HpCDD	850	810	580	530
Total TCDF	370	130	400	270
Total PeCDF	290	110	180	170
Total HxCDF	240	160	230	230
Total HpCDF	350	290	400	360

- * Single-column analysis only, may include contributions from other isomers that may co-elute.
. ND = Not detected, with quantitation limit given in parentheses
. Data from Reference 8

FIGURE 1

GENERAL STRUCTURES OF DIBENZO-*p*-DIOXIN (TOP) AND DIBENZOFURAN (BOTTOM)

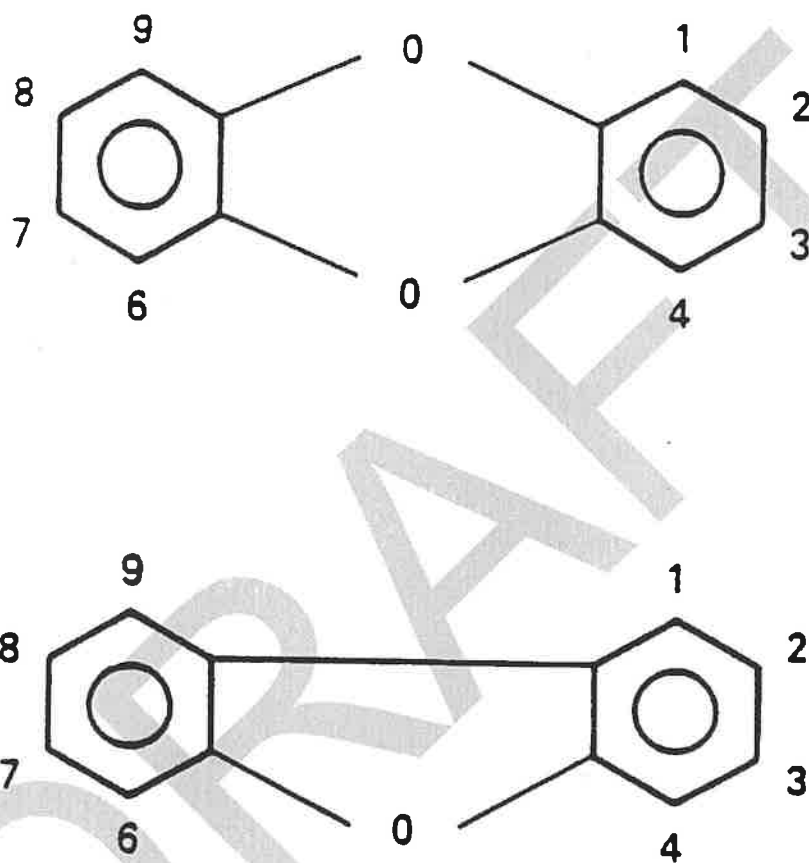
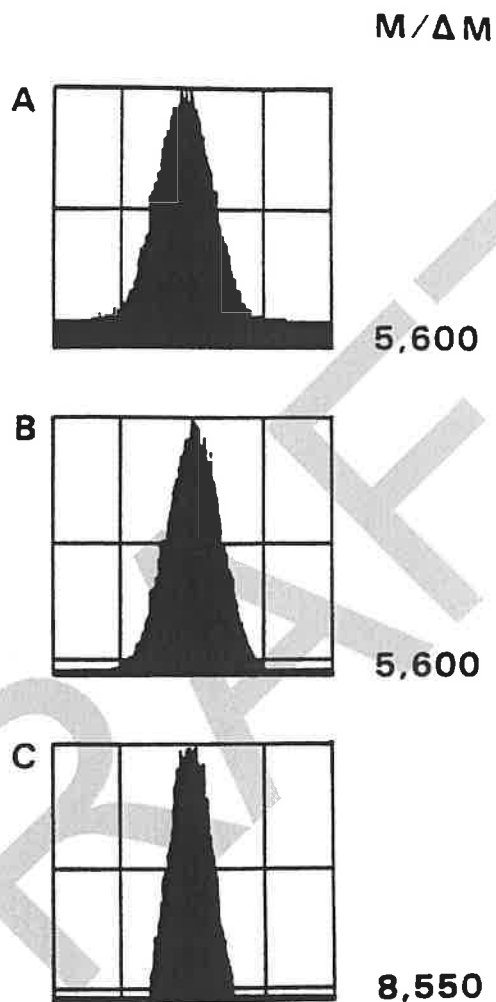


FIGURE 2

PEAK PROFILE DISPLAYS DEMONSTRATING THE EFFECT OF THE DETECTOR ZERO
ON THE MEASURED RESOLVING POWER



In this example, the true resolving power is 5,600.

- A) The zero was set too high; no effect is observed upon the measurement of the resolving power.
- B) The zero was adjusted properly.
- C) The zero was set too low; this results in overestimating the actual resolving power because the peak-to-peak noise cannot be measured accurately.

FIGURE 3
TYPICAL 12-Hr ANALYSIS SEQUENCE OF EVENTS.

Analytical Procedure

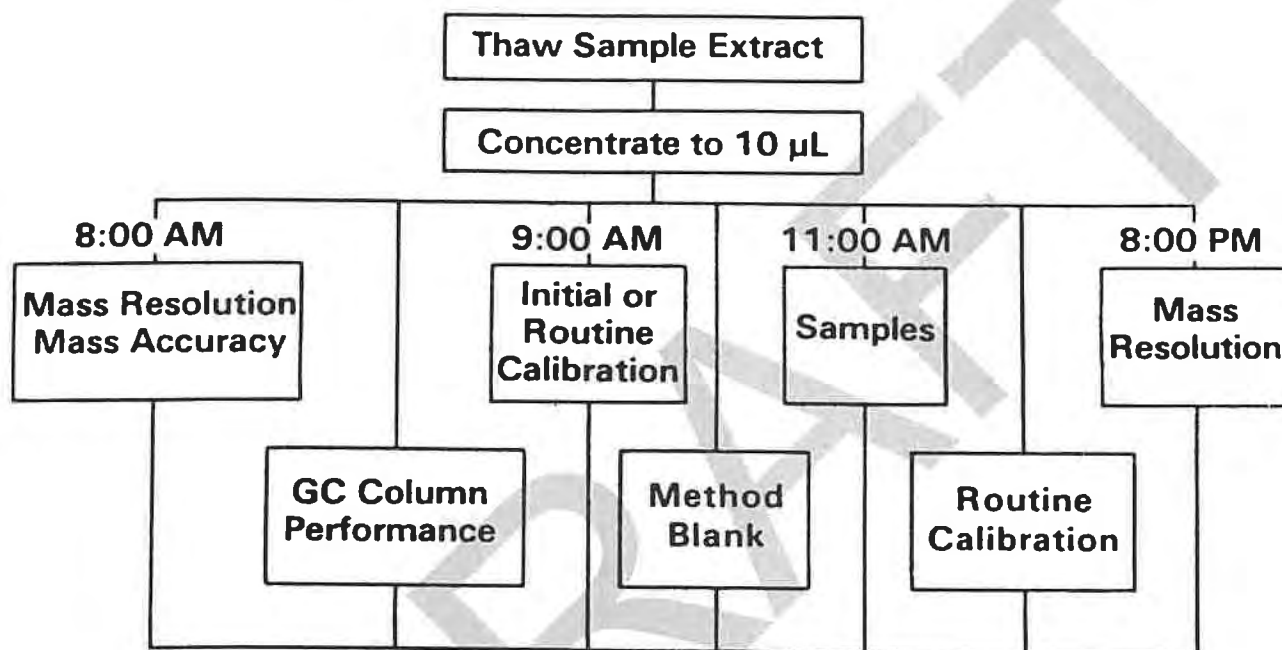


FIGURE 4

SELECTED ION CURRENT PROFILE FOR M/Z 322 (TCDDS) PRODUCED BY MS ANALYSIS
OF THE GC PERFORMANCE CHECK SOLUTION ON A 60 M DB-5 FUSED-SILICA
CAPILLARY COLUMN UNDER THE CONDITIONS LISTED IN SEC. 11.6.

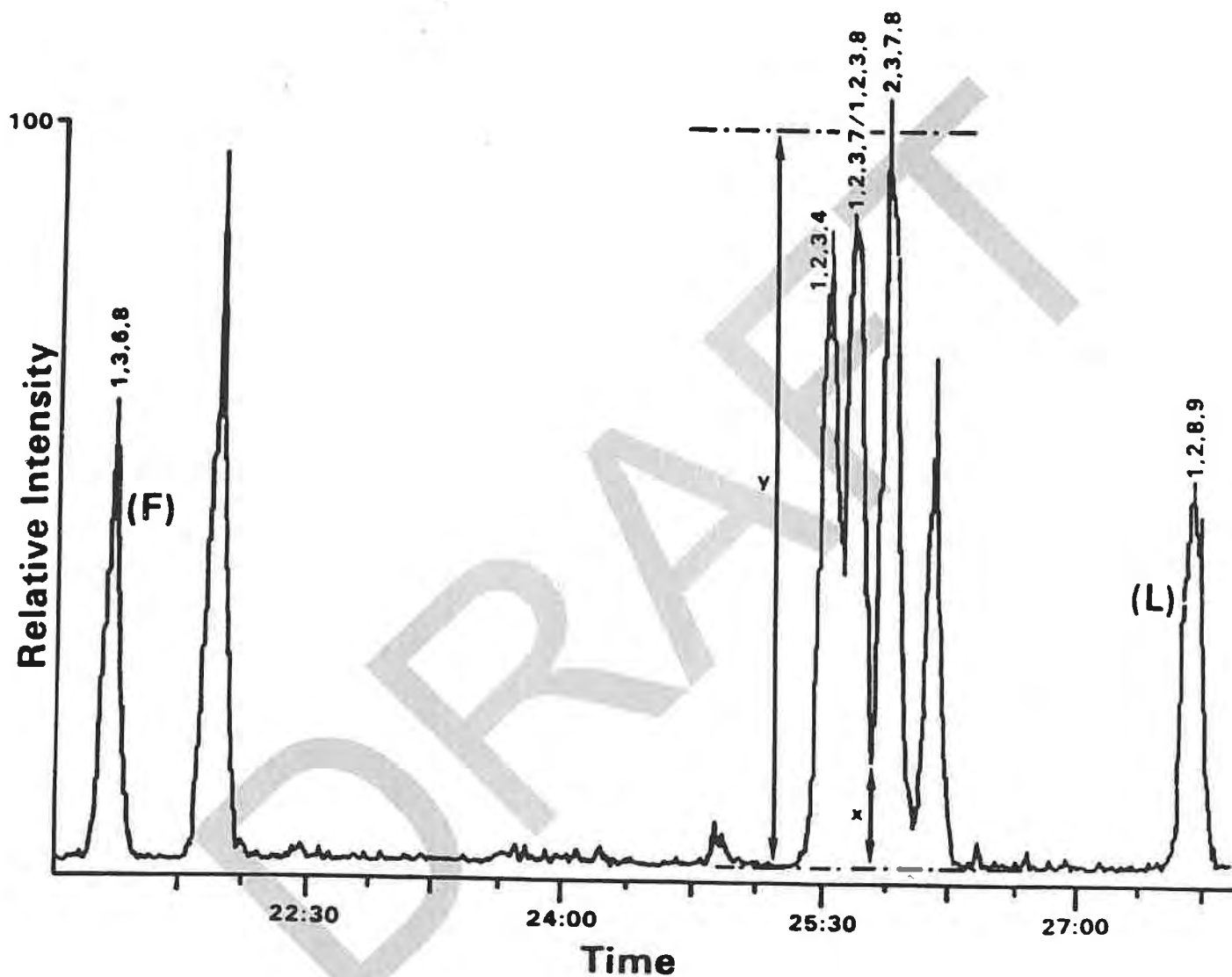
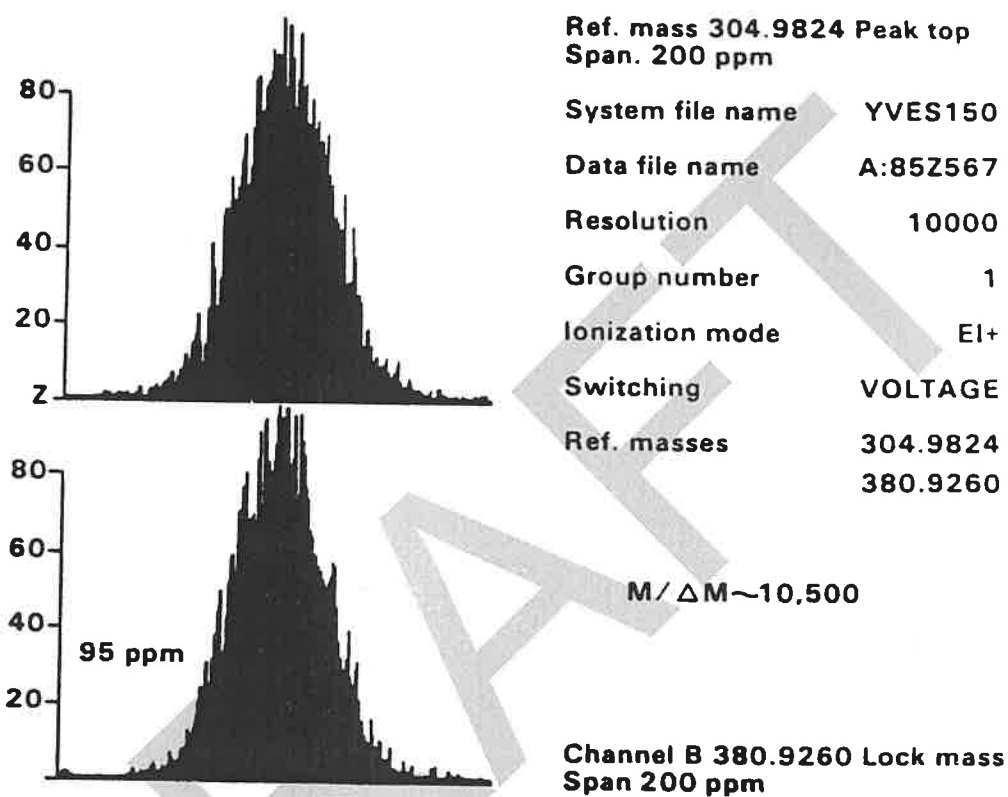


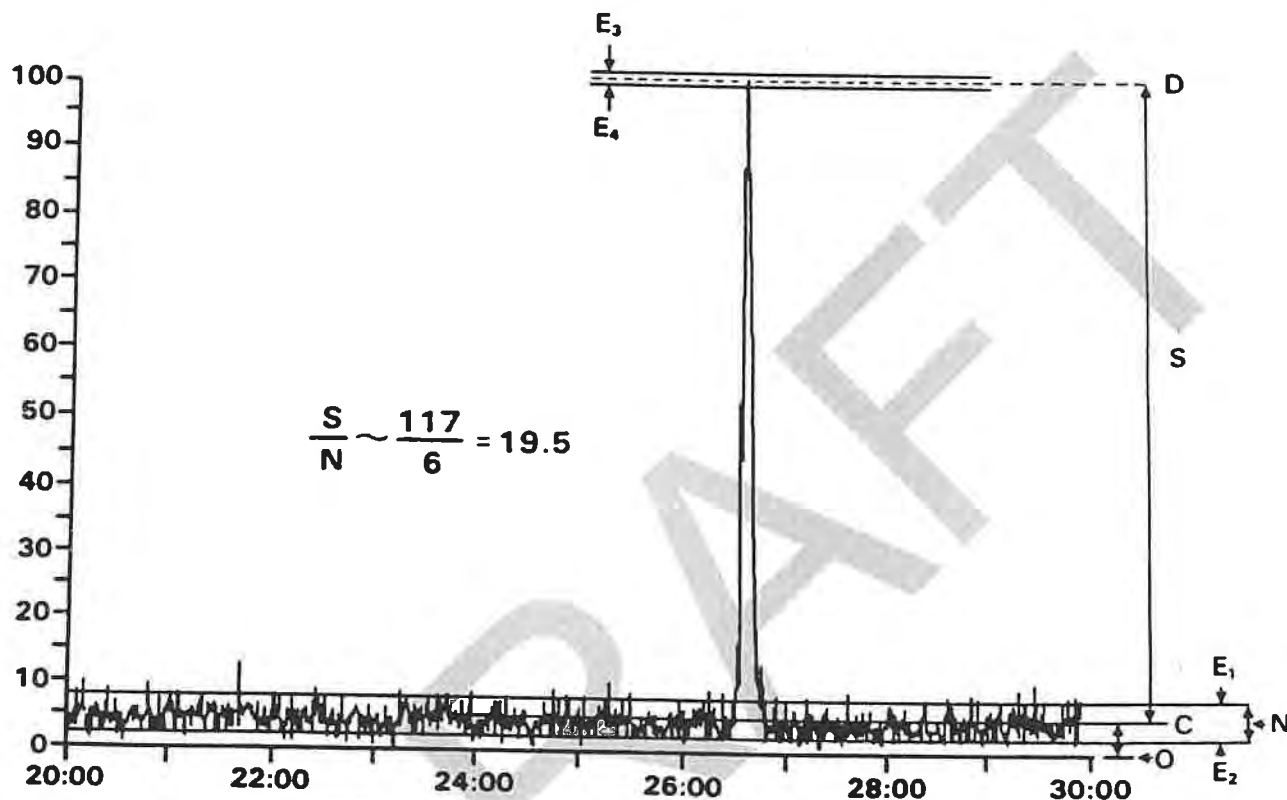
FIGURE 5

PEAK PROFILES REPRESENTING TWO PFK REFERENCE IONS AT M/Z 305 AND 381



The resolution of the high-mass signal is 95 ppm at 5 percent of the peak height; this corresponds to a resolving power $M/\Delta M$ of 10,500 (10 percent valley definition).

FIGURE 6
MANUAL DETERMINATION OF S/N.



The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E₁ and E₂, and between the apex average noise extremes, E₃ and E₄, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

APPENDIX A

PROCEDURES FOR THE COLLECTION, HANDLING, ANALYSIS, AND REPORTING OF WIPE TESTS PERFORMED WITHIN THE LABORATORY

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

A.1 Perform the wipe tests on surface areas of two inches by one foot with glass fiber paper saturated with distilled-in-glass acetone using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL of distilled-in-glass acetone. Place an equal number of unused wipers in 200 mL acetone and use this as a control. Add 100 μ L of the sample fortification solution (Sec. 7.8) to each jar containing used or unused wipers.

A.1.1 Close the jar containing the wipers and the acetone and extract for 20 min using a wrist action shaker. Transfer the extract into a K-D apparatus fitted with a concentration tube and a three-ball Snyder column. Add two PTFE or Carborundum™ boiling chips and concentrate the extract to an apparent volume of 1.0 mL on a steam bath. Rinse the Snyder column and the K-D assembly with two 1-mL portions of hexane into the concentrator tube, and concentrate its contents to near dryness with a gentle stream of nitrogen. Add 1.0 mL of hexane to the concentrator tube and swirl the solvent on the walls.

A.1.2 Prepare a neutral alumina column as described in Sec. 11.5.2.2 and follow the steps outlined in Secs. 11.5.2.3 through 11.5.2.5.

A.1.3 Add 10 μ L of the recovery standard solution as described in Sec. 11.5.3.6.

A.2 Concentrate the contents of the vial to a final volume of 10 μ L (either in a minivial or in a capillary tube). Inject 2 μ L of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as described in the analytical method in Sec. 11.8. Perform calculations according to Sec. 11.9.

A.3 Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 10 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 3 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is $10 \times 5 = 50$ pg/WTE and the positive response for the blank would be $3 \times 5 = 15$ pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

A.4 At a minimum, wipe tests should be performed when there is evidence of contamination in the method blanks.

A.5 An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed (use multiplication factors listed in footnote (a) from Table 1 for other congeners). This value corresponds to 2½ times the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high efficiency particulate absorbent (HEPA) filter and then wash with a detergent. A new set of

wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory after corrective action has been taken.

DRAFT