

RADIOACTIVE/HAZARDOUS WASTE DRUM VENTING & HEADSPACE GAS CHARACTERIZATION MACHINE

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ABSTRACT

NFT Incorporated has designed, developed and deployed portable radioactive waste drum headspace gas sampling equipment at two U.S. Department of Energy (DOE) sites, successfully supporting TRU waste drum retrieval operations and drum characterization for disposal at the DOE Waste Isolation Pilot Plant (WIPP). The NFT Drum Venting System (DVS) has processed over 7,000 TRU waste drums at the Savannah River Site (SRS) and over 140 TRU waste drums at the Nevada Test Site (NTS). The equipment, systems, procedures, and involved personnel have successfully passed audit evaluation by DOE-CAO, and are approved to submit headspace gas characterization data. The DVS has successfully participated in three Performance Demonstration Program (PDP) cycles to date. The DVS design and operations were reviewed for safety considerations and successfully passed a DOE-SRS Operational Readiness Review (ORR).

The portable, skid mounted DVS can be flexibly deployed in an existing facility or set up in a temporary building to campaign waste stream retrieval and WIPP characterization operations. The DVS can provide sites with WIPP certified headspace gas characterization capability where none exists or can augment capacity at sites with current capability. The DVS performs two key tasks:

- ◆ Supports safe retrieval of unvented, legacy waste drums by eliminating the handling risks due to potential hydrogen deflagration (drum explosion). The DVS tests drum headspace gases, and if explosive levels of gases are present, evacuates the drum and backfills the headspace with non-flammable nitrogen gas. When re-analysis confirms that headspace gases are non-explosive, the DVS installs and approved High Efficiency Particulate Air (HEPA) grade filter vent. The installed HEPA filter vent prevents buildup of explosive gas, allowing safe handling and transportation in accordance with WIPP and Nuclear Regulatory Commission (NRC) requirements.
- ◆ Characterizes the headspace gases of vented or unvented TRU waste drums for disposal at the DOE WIPP site. The on-line, integrated gas chromatograph/mass spectrometer (GC/MS) analytical equipment and methods employed meet all criteria established by WIPP to analyze part-per-million levels of hydrogen, methane and 29 volatile organic compounds (VOCs).

Two key innovative design features include the modular, portable deployment capability, and the unique analytical system configuration. The self-contained, modular system provides engineered control of radiological and organic solvent emissions and is easily deployed in existing buildings with a simple electrical connection. The specially configured GC/MS analytical instrument provides an extended linear range of calibration, from the WIPP required quantitation limit up to the saturation concentration in air. This eliminates the need to dilute and re-analyze high concentration samples, providing "one-pass" analytical efficiency.

The DVS provides significant life cycle cost savings over existing, stationary systems. One advantage is the portable design; the cost of facilities to support retrieval or characterization is often negligible and limited to existing buildings. Additional savings are realized by the on-line integrated laboratory; the costs associated with sample collection, sample canister transportation to the remote laboratory, and canister cleaning for subsequent re-use are eliminated. The integrated system also has reduced numbers

of batch quality control samples required, increasing overall process throughput. Total per drum costs for drum venting and characterization are reduced by as much as 50%.

INTRODUCTION

This paper describes the design and test criteria required to fabricate, program, and test a radioactive waste drum venting and gas sampling system that meets all headspace gas sampling and analysis requirements of the U.S. Department of Energy (DOE) Waste Isolation Pilot Plant (WIPP). The NFT Headspace Sampling System (HSS) provides three primary functions:

- ◆ Punctures the drum lid and liner of packed radioactive waste drums to detect flammable levels of drum headspace gases. If flammable levels of gases are present in the drum, the HSS automatically evacuates and purges drums with dry, non-flammable nitrogen. This process removes drum explosion and flammability hazards, making drums safe to handle and transport.
- ◆ Safely installs a High Efficiency Particulate Air (HEPA) grade filter vent into an unvented drum of radioactive waste. The filter selectively retains radioactive particles while allowing gases to diffuse through the filter and out of the drum. The filter vent eliminates the buildup of flammable levels of hydrogen gas generated by the radiolytic decomposition of wastes such as paper and plastics.
- ◆ Collects a representative sample of drum headspace gas and automatically performs an analysis for hydrogen, methane, and Volatile Organic Compounds (VOCs) as required by the DOE WIPP Site. This analysis is performed by a specifically configured Gas Chromatograph/Mass Spectrometer (GC/MS). These data are used for two purposes: to ensure worker occupational exposure to VOCs are below action levels; and confirm correct Resource Conservation and Recovery Act (RCRA) waste code designation.

This paper describes or references descriptions of hardware, software, electronic control systems, and analytical systems. This specification describes software functional requirements, system design requirements, and validation test plans in full compliance with applicable software quality assurance criteria as required by the DOE-WIPP site. The NFT DVS system was awarded a U.S. Patent, "Methods of and Apparatus for Testing and Venting Drums of Radioactive Waste", No. 5,767,422, Serial No. 08/725,021.

BACKGROUND

Legacy Radioactive Wastes

Nuclear weapons production programs have generated a variety of radioactive wastes. Since the early 1970's, wastes generated in DOE nuclear weapons operations were primarily packaged in unvented 55-gallon steel drums. This drummed waste was stored with the intention of future retrieval and final disposal. The wastes are now designated for permanent disposal at the DOE-WIPP facility near Carlsbad, New Mexico. These "legacy" wastes present handling and transportation safety concerns due to the potential presence of combustible gases inside the drum headspace. Combustible hydrogen and methane gases can be produced by the exposure of hydrogenous waste materials such as paper or plastics to radiation. This process, called radiolytic decomposition, provides a gas generation source within the drum. In addition to the generation of hydrogen and methane gas, flammable concentrations of VOC vapors may be present, introduced as part of the original waste mixture. Waste drums containing such explosive

gases are a safety risk during handling or transportation; the DOE complex has documented several accidents involving exploding waste drums¹. Since 1987, the DOE has required HEPA filter venting for all TRU wastes containers to allow combustible gases to diffuse to the atmosphere while fully containing radioactive materials.

In addition to the combustible gas concerns, most of these legacy wastes were packaged prior to the U.S. Government passing hazardous waste management legislation. The RCRA of 1982 imposed “cradle to grave” control over the generation, storage, and disposal of hazardous wastes. Wastes must be properly tested in accordance with applicable requirements of this Act prior to lawful disposal. Additional waste testing requirements are imposed by the WIPP facility Waste Acceptance Criteria (WAC) and Quality Assurance Program Plan (QAPP). The applicable regulations require 100% TRU waste drum headspace gas sampling and analysis for a prescribed list of compounds. This headspace gas characterization is performed for three primary reasons: a) to confirm proper RCRA waste code characterization of wastes, b) to assess and control worker exposure to hazardous vapors below the OSHA Threshold Limit Value (TLV), and c) to ensure drum gases are not flammable or explosive. Many of these DOE legacy wastes require further characterization of contents before shipment or disposal will be permitted.

NFT Drum Vent and Headspace Sampling System

NFT has designed and built a patented system that meets the demand for DOE radioactive waste drum venting and headspace gas sampling. The NFT Headspace Sampling System (HSS) is designed to automatically collect and analyze representative drum headspace gas samples, and if necessary, to safely and remotely eliminate safety concerns due to the presence of flammable or explosive headspace gases. The HSS uses process control software and an operator workstation touch screen interface to provide command and control of the drum venting and headspace sample and analysis process. A brief overview of this process is as follows:

- ◆ The drum is manually loaded into a specially designed cabinet. The cabinet is designed to withstand a maximum credible deflagration event. Computer control then raises the drum to contact the seal housing. The drum is lifted using a pneumatic lift; the lift assembly is mounted on load cells in order to measure and control upward force of the drum lid onto the seal-housing gasket.
- ◆ The powerhead assembly is mounted above the drum cabinet in a glovebox. This assembly, with a filter vent or plug installed into the powerhead socket, is lowered into the seal housing. The socket passes through two seals creating a sealed chamber on the drum lid surface.
- ◆ A sensor feedback loop maintains upward drum force against the seal-housing gasket during processing. A test of seal integrity is performed to verify a sealed chamber has been formed on the drum lid surface for each sampling event.
- ◆ The NFT self boring, self tapping filter vent assembly, mounted in the socket, bores a hole into the drum lid and pauses at a prescribed sample depth. The hollow stem filter or plug contains a flow path from inside the drum liner to the seal housing sample manifold.
- ◆ A sample manifold, consisting of ¼” stainless steel tubing, valves, and vacuum pumps is operated to withdraw a representative sample of headspace gas from the drum and deliver it to a Gas Chromatograph/Mass Spectrometer analytical instrument. The analytical instrument determines concentrations of hydrogen, methane and a minimum of 29 VOCs in the headspace gas sample in accordance with DOE-CAO data Quality Assurance Objectives.

- ◆ A sample is also drawn and routed to a combustible gas analyzer that provides real-time assessment of headspace gas flammability.
- ◆ If gases or vapors are present above the Lower Explosive Limit (LEL), the HSS will evacuate the explosive gases and purge the drum headspace with non-flammable nitrogen gas. Reanalysis is then performed to verify drum gases are below the LEL before proceeding.
- ◆ Metal chips generated during the boring operation are gently blown away from filter vent sealing surfaces by a puff of air.
- ◆ The filter vent or plug is then threaded and seated into the drum lid. Parameters controlled for this operation include motion sequence control to synchronize filter vent rotation and translation to the thread pitch.
- ◆ After radiometric survey and release of the drum to established unconditional release criteria, the drum is unloaded from the deflagration cabinet.

APPLICABLE CODES AND STANDARDS

External Requirements

ANSI/ASME NQA-1-1989, "Quality Assurance Program Requirements for Nuclear Facilities."

American Glovebox Society, "Guidelines for Gloveboxes," AGS-G001-1994, open front box (hood).

All pressure containing welds in structures that provide contamination shall be in accordance with the requirements of ASME Section IX, and welders shall be certified to ASME Section IX with copies of their certification provided to NFT prior to performance of welds.

NFT Incorporated HSS Design Documents

Specification DS-9075-1 "*Design and Fabrication of the Drum Containment Cabinet.*"

Specification DS-9075-2 "*Design and Fabrication of the Glovebox Assembly.*"

Engineering Design File, Project A004-000, including the following:

DVS-III-01, Headspace Sample Line Heat Trace Calculations

DVS-III-02, WIPP Certified Sample Plug or Filter Vent Length Determination

DVS-III-03, Vacuum System Analysis

DVS-III-04, Vacuum System Design Review

DVS-III-05, Filter Design to Meet SAR Requirement

DVS-III-06, Sample Manifold Volume Calculation

DVS-III-07, TRU Waste Drum Annular Space Volume

DVS-III-08, Mechanical Drawings

DVS-III-09, Process Design Specification

DVS-III-010, Software System Description

DVS-III-011, Specification DS-9075-1, "*Design and Fabrication of the Drum Containment Cabinet*"

DVS-III-012, Specification DS-9075-2, "*Design and Fabrication of the Glovebox Assembly*"

DVS-III-013, "*DVS Explosion Vent & Deflagration Shield Analysis*"

VALIDATION TEST REQUIREMENTS

In addition to the design and process requirements stated in sections 3 and 5 of this document, the integrated system must demonstrate achievement of the following requirements:

1. The DVS must pierce the drum lid and liner, allow the insertion of the filter through the lid, and threading of the filter assembly into the drum lid.
2. The seal between the filter housing and the drum lid provides the contamination control boundary. Ensure that the seal is leak tight to HEPA grade.
3. a) The system vacuum shall remove over 1.5 times the total volume of the sample manifold piping and sample collection chamber, not to exceed 10 liters in total volume. b) The GC vacuum shall remove over 1.5 times the volume of the leg of piping from the distribution block to the instrument. c) The drum seal housing shall demonstrate a leak rate less than 155 torr per minute measured over 15 seconds. d) The sample manifold leak rate shall be less than 4.5 torr/second, measured over 15 seconds. e) The sample manifold and vacuum system shall demonstrate an ultimate pressure of 100 mtorr or less.
4. Demonstrate the ability of the GC/MS analytical instrument to meet DOE-CAO requirements related to calibration, continued calibration verification (CCV)/field reference standard (FRS), and blank analyses. This test will satisfy the precision and accuracy requirements for initial method performance sample acceptance criteria and QC sample acceptance criteria identified in NFT-DVSIII-001, Determination of Volatile Organic Compounds (VOCs) in Waste Container Headspace by the Gas Chromatography/Mass Spectrometry & Flame Ionization Detector, Rev. 0. The test is performed with the GC/MS integrated into the Drum Headspace Gas Sampling and Analysis System and controlled by the automated process controller software.
5. Demonstrate the ability to automatically evacuate and purge the headspace gases in a drum with an "inert" gas, e.g., dry nitrogen. A drum containing a non-explosive level (for safety) of flammable gases will be evacuated by pumping down the drum to an acceptable negative pressure relative to the outside environment, as determined by the vent/purge performance capability study. The LEL meter will be used for recording pre and post-purge % LEL values. A successful purge will be initiated by observation of correct chamber/drum evacuation and purge pressures and the reduction in LEL values.

GENERAL SYSTEM DESCRIPTION

Analytical Systems

The HSS has two drum headspace gas analysis systems: a Gas Chromatograph/Mass Spectrometer (GC/MS) specially fitted with auxiliary detectors, and an explosive gas meter. Both systems are piped into the sampling manifold.

Gas Chromatograph Mass Spectrometer

The GC/MS is specially configured to perform the following functions: a) analysis of TRU waste drum headspace gases according to all data Quality Assurance Objectives (QAOs) as specified by the DOE-WIPP Quality Assurance Program Plan (QAPP) for hydrogen, methane and 29 Volatile Organic Compounds (VOCs); and b) perform analyses for these compounds over an extremely wide range of concentrations. The latter function is designed to effectively quantitate drum headspace gases containing high concentrations of gases or vapors on a single pass through the HSS. NFT has identified a "one-pass" headspace gas analysis as a key design objective in order to provide cost-effective analysis of all expected TRU waste drum headspace gas conditions. The one-pass analysis eliminates the delays associated with dilution and re-analysis of high concentration samples, reduces costs to our clients, and makes drum processing more predictable when drums with high headspace gas concentrations of contaminants are encountered. This system provides a linear range of calibration for VOCs from the minimum detection limit defined by the DOE-QAOs up to and exceeding the maximum saturation concentration. Likewise, the linear calibrated range for hydrogen and methane is from minimum detection to well over 50% by volume. The GC/MS uses standard capillary column chromatography techniques coupled with auxiliary detectors. The extended range of calibration is achieved using the following key features:

- ◆ An **Ion Trap Mass Spectrometry Detector (MS)** has a sensitivity of an order of magnitude below that required by DOE minimum detection limit standards. The high sensitivity eliminates the need to pre-concentrate the gas sample as is typically done for quadrupole mass spectrometers. The ion trap MS is used to identify all VOCs by comparing and matching obtained spectra to a NIST standard spectral library. The MSD is always used to identify VOCs, and is selectively used to quantify VOCs when concentration ranges are in the detector linear range of calibration.
- ◆ A **Flame Ionization Detector** piped in parallel with the MSD. The FID is used to quantitate results of VOCs above the MS linear range of calibration, or 1200 ppmv. This type of detector has a linear range of quantitation for VOCs from 500 ppmv up to 100% concentration by volume.
- ◆ Special **Commercially Developed Software** that automatically selects the MS or FID result for quantitation based on the concentration of VOCs. Whenever a compound exceeds the linear range of calibration for the MS, the FID result is used.

Hydrogen concentrations are quantified using a Thermal Conductivity Detector (TCD), a single detector providing wide range response from below the required MDL up to 100%.

This special analytical system configuration provides cost-effective analyses and reduces drum processing throughput rates. Each detector is applied in a manner that employs its unique technical efficacy. The mass spectrometer is state of the art detection technology that very effectively identifies minute quantities of organic compounds. This detector is used to identify all VOCs, and quantify them in the lower concentration ranges. The FID's key feature is wide range linear response; it is used to quantify previously identified VOCs at high concentrations in the headspace gas. The HSS analytical system is calibrated according to approved DOE/EPA methods prescribed for the mass spectrometer, flame ionization, and thermal conductivity detectors employed. The MS linear range of calibration overlaps that of the FID in the range from 500 to 1200 ppmv. For a detailed system description and calibration method see the NFT analysis procedure referenced in section 3.0.

Explosive Gas Meter

Piped into the sample collection manifold is an explosive gas meter that provides real-time flammability assessment of drum headspace gases. This instrument reads in percent of Lower Explosive Limit (%LEL), drum gases are automatically evacuated to the HSS exhaust stack and backfilled with a purge of non-flammable nitrogen gas. This is performed after headspace sample collection for the GC/MS analysis. This feature helps ensure worker safety during subsequent drum handling operations.

Automated Drum Penetration and Sample Collection

The HSS automatically penetrates the drum using a specially designed, self-drilling, self-tapping hollow core filter vent. The filter or plug is installed into a socket inside the glovebox powerhead prior to drum HSS operations. A Programmable Logic Controller (PCL) commands and controls the process that penetrates the lid, collects a representative headspace gas sample for GC/MS analysis, evacuates and purges flammable drum headspace gases if necessary, and then installs and seals the filter vent onto the drum lid. The drum penetration cabinet is designed and proof tested to 12 times the maximum credible accident pressure (see reference).

Automated drum penetration and representative sample collection is supported by the following subsystems and components.

The Filter Vent or Plug

The hollow stem filter vent provides a free flow path from inside the TRU waste drum 90-mil liner and the annular space between liner and drum. The free flow path is provided under two conditions: a) when the filter vent is at sample collection depth, and b) when the vent or plug is fully inserted and sealed on the drum lid surface. The filter vent is a HEPA grade filter designed to meet minimum WIPP hydrogen diffusion requirements ($1.9\text{E-}6$ mole/mole fraction/second). The self-drilling, self-tapping filter vent houses the HEPA-grade, activated carbon, filter element. The filter is manufactured to rigorous NQA-1 Quality Assurance standards and individually tested and certified to HEPA filter specifications.

Sample Collection Manifold

The sample manifold connects the seal housing within the glovebox to the GC/MS and LEL analytical systems. The housing creates a sealed chamber on the drum lid surface when the drum is lifted to the housing seal gasket and when the powerhead socket is lowered through two seals. The sample manifold is composed of ¼" stainless steel tubing, and is specially welded, cleaned and leak tested in accordance with good vacuum system design and fabrication practices. High-vacuum electric solenoid valves and vacuum pumps operate according to a prescribed and tested process controlled by the PLC to deliver a representative sample to the analytical instruments.

To prevent cross-contamination between samples, the manifold is heated continuously to above 50°C, purged between sample collection runs with high purity nitrogen gas, and evacuated. This process ensures that any residual gases or compounds are flushed from the system between each sample collection event. A schematic diagram of the sample manifold is shown in Attachment 1.

Powerhead Assembly – The Linear Drive Actuator

Inside the glovebox, above the housing that creates a sealed chamber on the drum lid surface, is the linear drive actuator. This mechanism operates the powerhead assembly and socket that holds the filter vent. It is powered by a stepper motor that can be controlled to provide very small and precise vertical translation. The linear drive is microprocessor controlled, and communicates with the process PCL ladder logic program using a selection of programmable motion sequences. Translation distance and velocity is controlled for each motion sequence. The linear drive translation is synchronized with the nutrunner rotation during the thread-to-seat operation to match the filter vent or plug thread pitch.

Powerhead Assembly – The Nut Runner

The Rototool nutrunner (NR) applies rotation and torque to the powerhead socket and filter vent assembly during bore and thread-to-seat operations. The nutrunner is microprocessor controlled. Settings for each motion sequence are programmed into the controller. The HSS PLC ladder logic program invokes the proper sequence at the correct time. The NR rotation speed is synchronized with the LD translation velocity during the thread-to-seat operation to ensure correct seating of the filter vent or plug.

Contamination Control – Isolation of Drum Contents

During all stages of HSS, the interior of the drum is isolated from the inside of the deflagration cabinet and the glovebox by a sealed chamber. This seal is created when the drum is lifted to the seal-housing gasket and when the powerhead socket is lowered past two seals. The seal integrity on the drum lid surface is tested prior to each sampling event and serves two purposes: it ensures that headspace gases are representative of the drum contents and not contaminated by infiltration of air; and it provides the first level of radioactive particle containment, if required.

Contamination Control – The High Efficiency Particulate Air (HEPA) Air Handling Train

During HSS processing, the drum and its contents are isolated from the deflagration cabinet and glovebox interior. The cabinet and glovebox are kept under negative pressure (airflow) conditions to ensure control of any radioactive particles that may escape from the drum during processing if an off-normal event occurs. The air handling train is a two-stage HEPA filtration system that is certified and leak tested. The air train blower motor is operated at all times during processing. Two motor speeds are employed, one

for normal operations when all cabinet access doors are closed, and a high speed used to ensure proper airflow when doors are opened for drum placement, removal, or monitoring.

PROCESS OVERVIEW

1. **Operational Readiness:** The daily operational readiness checklist is completed. All instruments must be functional, fully calibrated, with acceptable readiness status documented. Prior to staging for processing, drums are surveyed for surface contamination and container integrity. Acceptable drums are loaded onto the system drum dolly. The drum number and other relevant information is recorded on the process log sheet.
2. **System Startup:** The operator initiates system startup from the touch screen interface. The system pump, compressor, and air handling train blower starts and reaches operating levels. The operator verifies proper glovebox and cabinet negative pressure. The operator closed all doors, inserts the filter vent in the glovebox power head socket, and initiates drum vent and headspace gas sample collection and analysis via the touchscreen interface. Safety interlock design will not allow drum processing until the large and small access doors are closed, and a drum is in the processing chamber. This system readiness information is provided by proximity sensors and load cell signals relayed to the PLC processor; when all conditions for operation are acceptable, the “weigh drum” touch screen is displayed, enabling drum processing.
3. **Weigh Drum:** Valve 10 opens providing compressed air to the drum lift air bladder. Valve 10 remains open until the photoelectric eye in the drum cabinet indicates that the drum is lifted to proper height for weighing. The drum lift pauses for 5 seconds, allowing the dual load cells to weigh the drum and display weight on the operator touchscreen. The drum then lifts to contact the seal-housing gasket. Drum lift continues until the selected upward seal force set point is reached. The drum lid to seal housing gasket seal force is monitored and Valve 10 is re-opened if seal force drops below the set point.
4. **Lower Powerhead:** The powerhead assembly, with the filter installed into the socket, lowers into the seal housing. The filter socket passes through the two dual lip oil seals, and continues until the filter drill point contacts the drum lid surface. This creates a sealed chamber on the drum lid surface. The lowering motion proceeds until the filter vent tip contacts the drum lid surface. This contact is identified by the powerhead load cell, which stops downward translation when a touch point force of 20 pounds is detected. If the operator inadvertently forgets to install a filter vent into the powerhead socket, no touch point force will occur. This condition causes the powerhead to return to home position, and a “no filter in socket” touchscreen to appear. The screen directs the operator to install the filter vent. When “continue” is pressed at this touch screen, the powerhead again lowers to touch point.
5. **Filter Insertion Chamber Seal Test:** Sample line valves 4, 5, and 6 are opened linking the running system vacuum to the manifold. Simultaneously, valve 2 is opened introducing high purity nitrogen gas into the seal housing chamber and sample line. The chamber and sample line is purged for 15 seconds. Valve 2 is then closed to isolate the nitrogen source and the sample manifold is pumped down to 300 torr (less than $\frac{1}{2}$ atmospheric pressure). Valve 6 is then closed isolating the system vacuum pump from the manifold. A pressure transducer measures the seal housing chamber initial and final pressures; the PLC program then calculates the leak rate based on a 15-second interval. An acceptable seal test is performed if the leak rate is less than 115 torr per minute.

6. **Process Quality Control:** At this point in the process, the operator is presented with various options, including: calibrate GC/MS (Field Reference Standard/Continuing Calibration Verification), calibrate LEL meter, PDP Sample, Blank Sample, and manifold leak test. If any of these steps are desired, they are performed now when the drum is up, the powerhead is lowered, and a successful seal test is complete. These process steps diverge from the basic operation and follow descriptions presented in the following sections. If process quality control samples were previously run in the specified interval, and a routine sample collection, analysis, and drum filter vent installation is desired, the operator depresses the touchscreen button “continue”.
7. **Bore to Sample Depth:** The powerhead assembly, consisting of the linear drive actuator and nutrunner tool, provides vertical filter socket translation and rotation. The powerhead bores through the drum lid and lowers the filter to sample depth. At sample depth a flow path is created from inside the drum 90-mil plastic drum liner and annular space, through the hollow, fluted filter vent stem, and into the seal housing inlet port of the sample manifold. The bore stroke is a specially designed, non-sparking, low-heat process with very low feed and speed. While the bore is in process, valve 4 is closed, isolating the sample line from the seal housing chamber, and valve 6 is opened to evacuate the sample line to 100 mtorr.
8. **Sample Collection:** Valves 4, 5 and 6 are opened, exposing the seal housing sample chamber to the running system vacuum pump. The vacuum pump is operated for 15 seconds. Two and one-half times the sample manifold the seal housing volume is withdrawn, in order to remove any non-representative gases from the seal housing and manifold. Valve 6 is then closed, isolating the system vacuum from the sample manifold. The instrument isolation valve 7 or 8 is opened and the GC (instrument) vacuum pump is operated for a minimum of eleven seconds to withdrawn two and one-half times the volume of the instrument leg piping. When the sample manifold pressure is at ambient, a start signal is sent to the ready GC/MS instrument, or by default to GC/MS number one. The GS/MS instrument captures a 120 μ l sample of headspace gases and begins a 40-minute analysis cycle. The correct instrument isolation valve 7 or 8 is actuated by means of an instrument ready signal sent to the PLC processor. The total volume of drum headspace gases withdrawn is less than one liter. All manifold valves are closed after the sample is collected.
9. **Explosive Gas Analysis, Purge and Evacuate:** During the sample collection step above, drum headspace exhaust gases are sent to real-time explosive gas analyzer downstream from the vacuum pump. The manifold valves are closed, and a 40-second pause enables the LEL meter to analyze the drum gases and return a signal to the PLC processor in units of percent of LEL. If drum headspace gases exceed the %LEL setpoint limit (usually 95%), then drum evacuate and purge is automatically initiated. Evacuate is conducted by opening manifold valves 4, 5, and 6, exposing the seal housing chamber to the vacuum pump. Evacuation proceeds until the set point duration is achieved. When evacuation is complete, manifold valves are closed and the nitrogen cylinder valve 2 is opened to introduce non-flammable gas into the drum. The flow path is through the seal-housing chamber and through the hollow, fluted filter vent core and into the drum. Once nitrogen purge is complete, a sample of gas is again withdrawn from the drum for %LEL analysis. Manifold valves 4, 5, and 6 are opened for 15 seconds to expose the drum interior to the system vacuum pump. The process is repeated until the drum headspace gases are below the %LEL set point.

10. Chip Blow: Metal drum lid cuttings are generated during the boring process and form a small conical pile around the filter vent shaft on the top of the drum lid. To remove these metal chips from the filter gasket to drum lid sealing surface, a small puff of air is directed at the chips to blow them out of the way. This is done by opening valve 1, directing the oil less air compressor supply, regulated at 10 psig, to the chip pile for 5 seconds.
11. Install Filter: The powerhead assembly installs the drum filter vent, correctly compressing the neoprene gasket to provide a HEPA grade seal at the drum lid surface. The stroke is accomplished by coordinated signals to the nutrunner tool and linear drive actuator that matches rotation and translation to the filter thread pitch. When this stroke is complete, valve 11 is opened to release air from the drum lift bladder and lower the drum.
12. Survey and Remove Drum: The touch screen interface prompts the operator to open the small access door to survey the drum lid and drum cabinet for radioactive contamination. If the survey results show no contamination present, the operator presses continue on the touch screen to process another drum or exit to shut the operation down. If exit is selected, valve 13 is opened for 5 seconds to relieve vacuum at the system vacuum inlet; this allows subsequent ease of pump restart. If the radiometric survey indicates presence of contamination, appropriate authorities are notified and de-contamination procedures are initiated. Proximity sensors on the large and small access doors provide positive air handling train blower motor control. Three blower speeds are used. One speed ensures cabinet and glovebox negative pressure is at least .3" H₂O. The second speed ensures that airflow through the small access door is 150 to 200 linear feet per minute. The third speed ensures that airflow through large access door is at least 100 linear feet per minute at the drum head height. The PCL processor varies the blower motor speed based on the proximity switch signals from the large and small access doors. This control system ensures automatic and proper airflow and negative pressure control during all processing conditions. During processing, the interior of the drum and all radioactive or chemical hazards contained in it are isolated from the cabinet and glovebox by the seal-housing chamber.

Quality Control Sample and Analysis Process

1. Quality Control Samples: A specific suite of Quality Control (QC) samples is required by the DOE-CAO, and closely follows standard EPA analysis practice for environmental samples. QA sample analysis must meet specific, quantitative criteria prior to sample and analysis of TRU waste drum headspace gases. The required frequency is no less than one suite of samples per batch of 20 drums. This QC protocol documents that the sample collection and analytical system is operating properly, and meets all applicable Quality Assurance Objective (QAO) criteria. Copies of the QC sample analysis reports are included in each data package submittal for a batch of drums. For the NFT system, the suite of QC samples are: a) the Field Reference Standard/Continuing Calibration Verification (FRS/CCV), b) the FRS/CCV duplicate, and c) the blank. The QC sample process and sequence is described below.
2. Standard Gas: A bottle of gas containing known amounts of hydrogen, methane, and at least six DOE-CAO listed Critical Target Compounds (CTCs) is piped into the pressure leg of the manifold and controlled by valve no. 3. This cylinder of gas is certified to known accuracy to standards traceable to the National Institute of Standards and Technology (NIST). The flow path of all QC standards is exactly the same as a routine drum headspace gas sample, except that rather than coming from the interior of the drum it is piped into the sealed sample chamber in direct contact with the drum lid surface.

3. Field Reference Standard (FRS)/Continuing Calibration Verification (CCV): The FRS/CCV sample collection and analysis is initiated by the operator at the touchscreen interface. The calibration process sequence is available for the operator to actuate at the touch screen only after the drum is up against the seal-housing gasket, the powerhead is lowered, and the seal housing leak check is successfully complete. This standard acts as both a CCV sample and a FRS sample. Acceptable analysis criteria include accurate analysis of all compounds to within $\pm 30\%$ of the certified concentration. When the operator selects "GC/MS calibration" from the touch screen, valve no. 2 opens along with the sample manifold valves 4, 5, and 6 passing high-purity nitrogen gas into the sample chamber and manifold piping. After a 15 second period, valves 2 and 4 are closed, and the sample manifold evacuated to 100 mtorr. These steps are conducted to purge and clean the sample collection chamber and manifold piping. When manifold cleaning is complete, valve no. 3 opens, passing the NIST certified gas into the sample chamber and sample manifold piping. Simultaneously, the sample manifold valves nos. 4, 5 and 6 are actuated, opening a flow path through the manifold, to the operating system vacuum pump, and out to the exhaust plenum. Valve 3 is open for a 15-second duration; then it and valve 6 are closed isolating the sample manifold from the calibration gas and vacuum pump. The GC vacuum pump is then started and operated for a minimum 11-second duration to remove non-representative gases from the GC/MS instrument internal piping. When the minimum duration GC vacuum operation is achieved, the PLC process controller waits for the vacuum transducer to indicate sample manifold pressure is at ambient. Then, a start signal is sent to the GC/MS instrument to capture a 120 μ l sample and begin the 40-minute analysis process. The GC/MS analysis process is controlled by a personal computer using Varian Chromatography Systems commercial software. These process control steps consistently deliver a known quantity of standard gas to the instrument inlet, eliminating variation introduced by varying instrument inlet pressures.
4. Duplicate Sample: A duplicate FRS/CCV sample is analyzed just after the initial FRS/CCV in order to determine if sampling and analysis process is precise (relative percent difference of the first and second sample) to within $\pm 25\%$. Step no. 5 described above is repeated to perform the duplicate sample collection and analysis.
5. Blank Sample: When the CCV/FRS sample and duplicate are successfully completed, a blank sample is collected and analyzed. The blank sample is high purity nitrogen, piped into the pressure side of the seal housing, and follows the same path as the calibrations and headspace gas samples. The operator selects "blank" from the touch screen to initiate this process. The blank analysis steps are the same as that for the FRS/CCV and duplicate above, except that valve no. 2 (to the nitrogen tank) is operated instead of valve no. 3. The blank sample is acceptable if compounds are present at less than three times the Program Required Quantitation Limit (PRQL).

Sample Manifold Pumpdown and Leak Test

1. Requirement: the DOE-CAO requires that drum headspace gas sample collection manifolds demonstrate evacuation to 100 mtorr, with a leak rate of no more than 4.5 mtorr per second. This is to ensure that the sample collection process is essentially leak-free, collecting and delivering a sample of representative drum headspace gases to the analytical instrument.

2. Sample Manifold Pumpdown and Leak Test: The target pressure is entered into the PCL program by means of a password protected touch screen entry, and is set at 100 mtorr. The leak rate criteria of no more than 4.5 mtorr per second is hard-coded into the PLC processor software. The operator initiates the test by pushing the “manifold leak test” touch screen button. The PCL processor controls the following operations: the manifold isolation valve no. 4 is closed, and valves 5 and 6 are opened exposing the sample manifold to the operating system vacuum pump. Valve no. 6 (system vacuum isolation valve) is closed when the target pressure of 100 mtorr is reached. At this point the calibrated vacuum gage records an initial pressure at time equals zero seconds, and the final pressure at time equals fifteen seconds.
3. Test Disposition: The PLC processor then calculates the leak rate and compares the result to the acceptance criteria of no more than 4.5 mtorr per second. If the leak rate passes, the “manifold leak test PASSED” touch screen is displayed. If the leak rate fails; the “manifold leak test FAILED” screen appears, prompting the operator and/or supervision to take corrective action prior to proceeding.

Performance Demonstration Sample Analysis

1. Requirement: The DOE-CAO requires semi-annual analysis of a blind sample that test the laboratories' ability to provide data of known quality in terms of precision and accuracy. The blind sample is provided in a 6-liter Summa stainless-steel canister, and contains concentrations of hydrogen, methane, and VOCs unknown to the laboratory. The results of the analysis are submitted to the DOE-CAO for evaluation and scoring. All laboratories working for the DOE-CAO TRU waste program must successfully complete the PDP sample prior to conducting characterization work.
2. The PDP sample analysis is automated through the PLC processor. A touch screen button “PDP sample” initiates the process. When the initial PDP sample button is pressed, a touch screen display directs the operator to install the PDP summa canister at the PDP tee inside the instrument cabinet. Once the canister is installed, the operator presses the touchscreen ready button. This opens the PDP isolation valve no. 9 and valve 6, exposing the “PDP leg” of the sample manifold to the operating system vacuum pump. When manifold vacuum is 100 mtorr or less, valve no. 9 is closed and the touch screen display directs the operator to open the manual PDP summa canister valve. When this is complete, the operator pushes the “sample” touch screen button to initiate the sample process. The sample process then opens valve 9 while the GC vacuum pump operates until manifold pressure reaches ambient. When the sample manifold vacuum gage indicates ambient pressure, the GC vacuum is stopped and the GC/MS start signal is sent simultaneously. The instrument captures a 120 μ l aliquot of PDP gas and performs a 40-minute analysis cycle. This cycle is repeated as often as necessary to complete the PDP blind sample analysis.

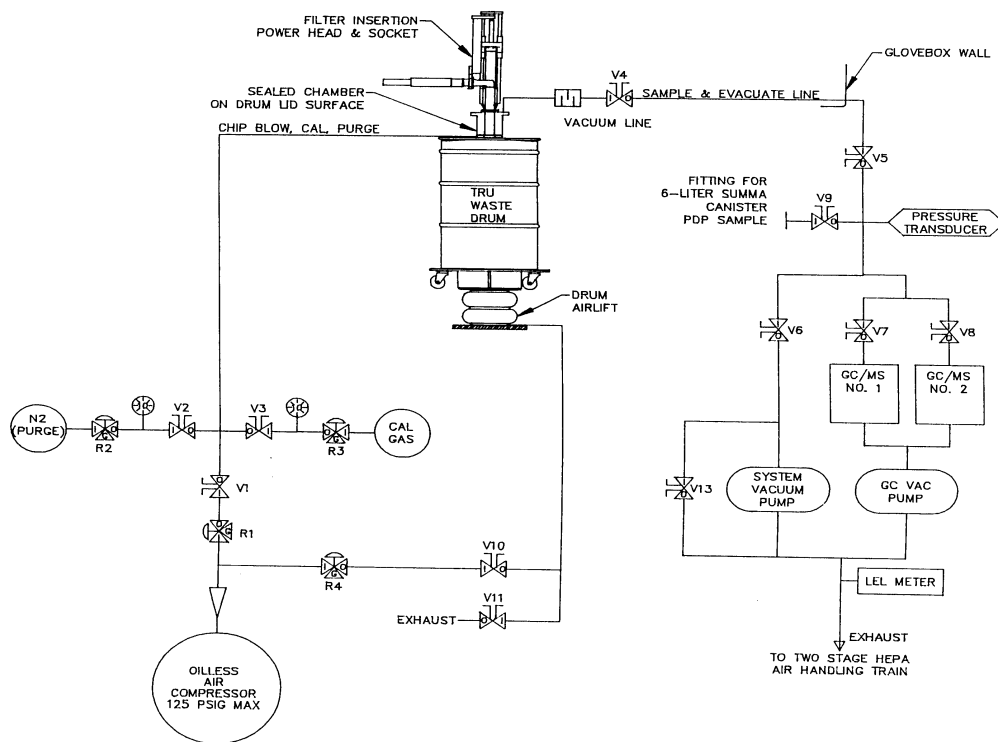
Summa Canister Sample Collection

1. **Summa Canister Installation:** If required, the NFT HSGS&A system can collect summa canisters of drum headspace gases. A summa canister is connected to the PDP tee inside the instrument cabinet; the operator initiates a summa canister collection by depressing the touchscreen button “summa sample”. This option appears after the drum is raised, passed seal test, and the filter vent is at sample depth in the drum lid. When the touch screen button is depressed, manifold valves 5 and 6 are opened until the sample manifold pressure is less than 100 mtorr.
2. **Sample Collection:** When “collect sample” is selected from the touch screen, valve 4 is opened, drawing drum headspace gases out of the drum, through the system vacuum and out to two-stage HEPA filtered exhaust for 15 seconds. Then valve 6 is closed, and the PDP (summa) isolation valve 9 opened. The evacuated summa canister draws drum headspace gas into it. The touch screen display then directs the operator to open the manual summa canister valve and monitor canister pressure at the canister pressure gage. (If the canister is not equipped with a pressure gage the vacuum gage readout at the operator console can be used.) When pressure is at ambient, the operator closed the manual summa canister valve. When this is complete, the operator presses “continue” on the touch screen, which closed all manifold valves, and directs the operator to remove the summa canister.

Operation Setpoint Parameters

1. **Password Protection:** Operation setpoint parameters are protected from inadvertent or unauthorized revision by a four-character password.
2. **Parameters Available for Change:** The touch screen allows revision of several process control parameters. The following parameters can be changed: a) upward seal force in pounds; b) seal test evacuation pressure in psig; c) drum evacuation duration in seconds; d) drum purge duration in seconds; e) manifold evacuation pressure in mtorr; f) LEL alarm set in percent. These operational parameters are typically not changed without consultation with the NFT Chief Engineer.
3. **Change Confirmation:** Once revised parameter settings are entered using the touchscreen numerical keypad, “enter” and “next” buttons are pressed. A new screen appears, requesting the operator to confirm that the entered value is correct. If the new parameter value is correct, the operator presses the confirmation button and proceeds through the menu.

DVS WIPP CERTIFIED SAMPLE MANIFOLD SCHEMATIC (PIPING OMITTED FOR STEPPER MOTOR PURGE, PNEUMATIC VALVE PILOT & GC/MS GASES)



DEVICE	PURPOSE
V1	BLOW CHIP
V2	PURGE DRUM
V3	CALIBRATION
V4	ISOLATE SAMPLE LINE
V5	ISOLATE PDP SAMPLE
V6	ISOLATE SYS VAC
V7	GC/MS #1
V8	GC/MS #2
V9	PDP SAMPLE VALVE
V10 (NOT SHOWN)	DRUM UP
V11 (NOT SHOWN)	DRUM DOWN
V12 (NOT SHOWN)	PURGE STEP MOTOR
V13	SYSVAC INLET BYPASS
SYSVAC	SYSTEM EVACUATION
GC/VAC	GC/MS SAMPLING
PT	MEASURE PRESSURE
LEL METER	MEASURE EXPLOSIVE GAS
PHOTOEYE	DRUM WEIGHT PAUSE
WEIGHT LOAD CELL	WEIGH DRUM
PH FORCE LOAD CELL	TOUGH POINT DETERMINATION

Figure. 1.

“Headspace Sampling System Sample Manifold Schematic”