FINAL REPORT

DESTRUCTION TECHNOLOGY DEMONSTRATION FOR ORGANICS IN TRANSURANIC WASTE

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ABSTRACT

General Atomics (GA) has recently completed a Phase I program for the development of a two-step alternative to incineration for the destruction of organics in transuranic wastes at the Savannah River Site. This process is known as thermal desorption-supercritical water oxidation, or TD-SCWO. The GA TD process uses heat to volatilize and transport organics from the waste material for subsequent treatment by SCWO. SCWO oxidizes organics in a steam medium at elevated temperatures and pressures in a manner that achieves excellent destruction efficiencies and compliance with all environmental requirements without the need for complex pollution-abatement equipment. This application of TD-SCWO is focused on a full-scale batch process for 55-gallon drums of mixed transuranic waste at the Savannah River Site.

The Phase I reduced-scale test results show that the process operates as intended on surrogate waste matrices chosen to be representative of Savannah River Site transuranic mixed wastes. It provides a high degree of hydrogen removal and full containment of the radionuclide surrogate, with minimal requirements for pre-treatment and post-treatment. Other test objectives were to verify that the process produces no dioxins or furans, and meets all applicable regulatory criteria for retention of toxic metals, particulate, and criteria pollutants, while meeting WIPP/WAC and TRUPACT-II requirements. Thermal desorption of surrogate SRS mixed wastes at 500 psi and 1000°F met all tested requirements for WIPP/WAC and TRUPACT-II. SCWO of the desorbed surrogate organic materials at 500 psi and 1500°F also appears to meet all requirements for a nonincineration alternative, although >99.99% DRE for chlorinated solvents has not yet been demonstrated.

TABLE OF CONTENTS

1. EXECUTIVE SUMMARY	1
2. INTRODUCTION.....	4
3. TEST OBJECTIVES	5
4. DESCRIPTION OF EQUIPMENT AND SET-UP	5
5. TESTING.....	14
5.1. Thermal Desorber Tests	14
5.2. Thermal Desorber-SCWO Integration and Optimization Tests.....	14
5.3. Demonstration Tests	16
5.4. Analytical Results.....	25
5.5. Success Criteria.....	27
6. CONCLUSION	30
7. FULL-SCALE TD-SCWO MODULE	32
8. REFERENCES.....	33
APPENDIX A. Sample TD-SCWO Run Plan	36
APPENDIX B. AirKinetics Report on Gas Effluent Analyses	42

LIST OF FIGURES

Figure 1. Process Flow Diagram (PFD) for GA TD-SCWO pilot plant.....	6
Figure 2. Pilot plant thermal desorber. Dimensions in inches.....	8
Figure 3. Pilot plant SCWO reactor. Dimensions in inches.....	9
Figure 4. Pilot plant TD canister and 55-gal drum.	10
Figure 5. Pilot plant thermal desorber.	11
Figure 6. Pilot plant SCWO reactor skid.....	11
Figure 7. Pilot plant SCWO reactor.....	12
Figure 8. PFD for TD-only tests.....	15
Figure 9. Plots for Run 8.2, recipe #2.....	17
Figure 10. Plots for Run 10.1, recipe #1.....	19
Figure 11. Plots for Run 11.0, recipe #3.....	20
Figure 12. Plots for Run 12.1, recipe #1.....	21
Figure 13. Ingredients for recipe #2.	22
Figure 14. Residue from a run with recipe #2.	23
Figure 15. Comparison of materials entering and leaving the SCWO unit.....	24
Figure 16. Process flow diagram for a full- scale TD-SCWO module.....	34
Figure 17. Architectural rendering of a full- scale TD-SCWO facility.....	35

LIST OF TABLES

Table ES-1. TD-SCWO Success Criteria.....	2
Table 1. Surrogate Recipes	13
Table 2. Parameters for TD-Only Tests	14
Table 3. Ultimate Analysis Results for TD Solids.....	25
Table 4. Cerium Analysis Results for TD Solids and Condensate	26
Table 5. Analytical Results for SCWO Liquid Effluent	26
Table 6. Analytical Results for SCWO Gas Effluent	27
Table 7. Success Criteria.....	28
Table 8. Determination of Hydrogenous Organic.....	29
Table 9. WIPP/WAC and TRUPACT II Requirements	30
Table 10. Comparison of TD-SCWO Pilot Plant and Full-Scale Module	32

Acronyms and Abbreviations

As	Arsenic
Be	Beryllium
Btu	British thermal unit
C	Carbon
CCl ₄	Carbon tetrachloride
C ₂ Cl ₄	Perchloroethylene or tetrachloroethylene
Cd	Cadmium
Ce	Cerium
CeO ₂	Cerium oxide
CO	Carbon monoxide
CO ₂	Carbon dioxide
Cr	Chromium
CW	Cooling water
DI	Deionized water
DRE	Destruction and removal efficiency
dscf	Dry standard cubic feet
dscm	Dry standard cubic meter
F	Flowmeter
ft	Feet
g	Grams
GA	General Atomics
gal	Gallons
GC/MS	Gas chromatography/mass spectrometry
GLS	Gas-liquid separator
H	Hydrogen
HC	Hydrocarbons
HCl	Hydrochloric acid
HEPA	High-efficiency particulate
Hg	Mercury
H ₂ O	Water
Hr	Hours
H ₂ SO ₄	Sulfuric acid
ID	Inner diameter
in	Inches
IPA	Isopropyl alcohol
kg	Kilogram
L	Liters
lb	Pounds
LCC	Life cycle cost
LV	Level instrument
MACT	Maximum achievable control technology
mg	Milligrams
min	Minute
ml	Milliliters
MM	Million
N	Nitrogen element
N ₂	Nitrogen gas

NaCl	Sodium chloride
ng	Nanograms
N ₂ O	Nitrous oxide
NO _x	Nitrogen oxides
O	Oxygen element
O ₂	Oxygen gas
OD	Outer diameter
oz	Ounce
P	Pressure
PAS	Pollution-abatement system
Pb	Lead
PCB	Polychlorinated biphenyl
PCDD	Polychlorinated dibenzodioxin
PCDF	Polychlorinated dibenzofuran
PFD	Process flow diagram
ppbvd	Parts per billion by volume dry
ppm	Parts per million
psi	Pounds per square inch
psia	Pounds per square inch absolute
psig	Pounds per square inch gauge
Pu	Plutonium
PVC	Polyvinyl chloride
RCRA	Resource Conservation and Recovery Act
S	Sulfur
Sb	Antimony
scfm	Standard cubic feet per minute
SCWO	Supercritical water oxidation
SO _x	Sulfur oxides
SRS	Savannah River Site
T	Temperature
TCLP	Toxicity Characteristic Leaching Procedure
TD	Thermal desorption
TEQ	Toxic equivalent
TOC	Total organic carbon
TRUPACT	TRansUranic PACkage Transporter
VOC	Volatile organic compound
WAC	Waste Acceptance Criteria
WIPP	Waste Isolation Pilot Plant
wt	Weight
τ	Residence time
µg	Microgram

1. EXECUTIVE SUMMARY

The United States Department of Energy's Savannah River Site (SRS), near Aiken, South Carolina, stores high activity plutonium-238-contaminated transuranic waste. This waste may not be shippable to the Waste Isolation Pilot Plant (WIPP) in TRUPACT-II containers due to excessive generation of hydrogen gas as a result of radiolysis of organic constituents in the waste matrix. Nonincineration means are sought by DOE for treatment of this waste to destroy the organics, eliminating the source of hydrogen, which would allow the waste to meet the TRUPACT-II hydrogen-concentration limit and the WIPP waste acceptance criteria (WAC). Under DOE Solicitation No. PRDA DE-RA26-01NT40981, GA was awarded a Phase I contract to perform surrogate mixed waste cold demonstration tests for a technology known as thermal desorption-supercritical water oxidation (TD-SCWO) for reducing the organic content to acceptable levels. GA combines two well-known and effective treatment processes, thermal desorption (TD) and supercritical water oxidation (SCWO), to achieve enhancements not possible with either technology alone, or with other potentially competitive technologies. GA is focusing on a full-scale batch process for 55-gallon drums of mixed transuranic waste at the Savannah River Site.

TD is well established as a treatment process for removal of organics from difficult solid matrices such as soils and mixed wastes. Heat is used to volatilize, gasify and transport organics from the inorganic residue for subsequent treatment. SCWO is a well-developed non-incineration process for destruction of organics in pumpable forms such as liquids and slurries of finely divided particles. SCWO oxidizes organics in a steam medium at elevated temperatures and pressures in a manner that achieves excellent destruction efficiencies and compliance with all environmental requirements without the need for complex pollution-abatement equipment. While typically operated at a pressure above the critical pressure for water (>3206 psia), GA has found that robust reaction and destruction efficiencies persist to pressures well below 1000 psi (Ref. 1, 2). In the Phase I work both TD and SCWO processes were operated at the relatively low pressure of 500 psi.

The primary objective of the Phase I Program was to demonstrate the ability of TD-SCWO to remove hydrogen from surrogate hydrogenous waste streams by nonincineration means. To qualify as a suitable technology, the proposed system would also have to show near-absolute containment of surrogate radionuclides and require minimal pre-treatment and post-treatment. Other test objectives were to verify that the process produces no dioxins or furans, and meets all applicable regulatory criteria for retention of toxic metals, particulate, and criteria pollutants, while meeting WIPP/WAC and TRUPACT-II requirements.

The Phase I testing utilized surrogate waste matrices chosen to be representative of Savannah River Site transuranic mixed wastes. Three different recipes comprising various mixtures of wood, paper, plastic, metal, leaded gloves, clay adsorbent and HEPA filters (primarily fiberglass) were used. Cerium oxide was added in a molar quantity to mimic plutonium-238 at a level of 5.7 grams per 55-gal drum, the maximum target value for TRUPACT-II containers. Carbon tetrachloride (CCl_4) and perchloroethylene (C_2Cl_4) spikes were added in amounts of 1 wt%. For each test the chosen feed mixture was packed into a 1.7-gal canister lined with thin PVC sheet to simulate a lined drum. The packed canister was then inserted in the thermal desorber unit to be processed by TD-SCWO.

The Phase I test results show that the GA TD-SCWO process operates as intended. Results are summarized in Table ES-1. All TD success criteria were demonstrated. The TD step provided a high degree of hydrogen removal and full containment of the cerium radionuclide

surrogate. The requirement for less than 5% hydrogenous organic in the residue was bettered by factors ranging from 2.5 to 33. With proper SCWO reactor temperature, all SCWO success criteria were also believed to be met, although the requirement of >99.99% destruction and removal efficiency (DRE) for chlorinated solvents has not yet been demonstrated.

Table ES-1. TD-SCWO Success Criteria

Requirement	Success Criteria	Status
Nonincineration	Peak process temperature measured via thermocouple to be <1600°F (871°C)	Demonstrated, see Figures 9-12
Hydrogen Removal	Total hydrogenous organic in residue <5 wt %	Demonstrated, see Table 8
Destruction and Removal Efficiency	Spiked constituents (CCl ₄ and C ₂ Cl ₄) measured in gaseous effluent to verify compliance with >99.99% destruction and removal	Demonstrated ~ 99.9% DRE
CO	CO level in gaseous effluent verified to be <100 ppm	Demonstrated, see Figures 10-12
Particulate Emissions	Particulate level in gaseous effluent verified to be <0.015 grains/dscf	Demonstrated, see Table 6
Mercury Removal	Hg level in gaseous effluent verified to be <45 µg/dscm	Demonstrated, see Table 6 (no Hg added to feed)
Cadmium and Lead Removal	Cd + Pb level in gaseous effluent verified to be <0.024 mg/dscm	Demonstrated, see Table 6
Antimony, Arsenic, Beryllium, and Chromium Removal	Sb + As + Be + Cr level in gaseous effluent verified to be <0.097 mg/dscm	Demonstrated, see Table 6
Radioactive Particle Containment	Measure CeO ₂ levels in gaseous and liquid effluent to determine degree of containment	Demonstrated, see Tables 4-6
Minimum Pretreatment	Minimum pretreatment requirements relative to other technologies	Demonstrated
Minimum Posttreatment	Minimum posttreatment gas cleaning process relative to other technologies	Demonstrated
Dioxin/Furan Emissions	Total dioxins and furans in gaseous effluent verified to be less than regulated levels of 0.2 ng/dscm (TEQ) at 7% O ₂	Demonstrated, see Table 6
WIPP/WAC	Meets requirements	Demonstrated, see Table 9
TRUPACT-II	Meets requirements	Demonstrated, see Table 9

Key conclusions from the Phase I testing are as follows:

- Thermal desorption of surrogate SRS mixed wastes at 500 psi and 1000°F meets all tested requirements for WIPP/WAC and TRUPACT-II.
- SCWO of the desorbed surrogate organic materials at 500 psi and 1500°F appears to meet all requirements for a nonincineration alternative. DRE for chlorinated solvents was slightly less than required during the lower temperature SCWO tests, so this temperature should be maintained to insure destruction.
- TD-SCWO technology is ready to proceed to Phase III, the full-scale module cold demonstration. (As per the PRDA, Phase II is not applicable to destruction technologies such as TD-SCWO.)

In summary, GA has demonstrated what may be a revolutionary new method for destruction of organics in mixed wastes that exist across the DOE complex. This new thermal treatment process is ideally suited to treating 55-gal drums of mixed waste without removing the materials from the drum. The process can also be adapted for continuous feed of bulk mixed wastes such as soils.

A Phase III Demonstration Program is needed to bring this new technology to full-scale, and to ready it for deployment at DOE sites with problematic mixed wastes.

2. INTRODUCTION

The United States Department of Energy's Savannah River Site (SRS), near Aiken, South Carolina, stores high activity plutonium-238-contaminated transuranic waste. This waste may not be shippable to the Waste Isolation Pilot Plant (WIPP) in TRUPACT-II containers due to excessive generation of hydrogen gas as a result of radiolysis of organic constituents in the waste matrix. Nonincineration means are sought by DOE for treatment of this waste to destroy the organics, eliminating the source of hydrogen, which would allow the waste to meet the TRUPACT-II hydrogen-concentration limit and the WIPP waste acceptance criteria (WAC). Under DOE Solicitation No. PRDA DE-RA26-01NT40981, GA was awarded a Phase I contract to perform surrogate mixed waste tests to demonstrate the feasibility of a technology known as thermal desorption-supercritical water oxidation (TD-SCWO) for reducing the organic content to acceptable levels.

General Atomics (GA) is developing a two-step alternative to incineration for the destruction of organics in transuranic wastes at the Savannah River Site (SRS). GA combines two well-known and effective treatment processes, thermal desorption (TD) and supercritical water oxidation (SCWO), to achieve enhancements not possible with either technology alone, or with other potentially competitive technologies. GA is focusing on a batch process for 55-gallon drums of mixed transuranic waste at the Savannah River Site.

TD is well established as a treatment process for removal of organics from difficult solid matrices such as soils and mixed wastes. Heat is used to volatilize, gasify and transport organics from the inorganic residue for subsequent treatment. SCWO is a well-developed non-incineration process for destruction of organics in pumpable forms such as liquids and slurries of finely divided particles. SCWO oxidizes organics in a steam medium at elevated temperatures and pressures in a manner that achieves excellent destruction efficiencies and compliance with all environmental requirements without the need for complex pollution-abatement equipment. While typically operated at a pressure above the critical pressure for water (>3206 psia), GA has found that robust reaction and destruction efficiencies persist to pressures well below 1000 psi (Ref. 1, 2). GA combines the TD and SCWO processes at a relatively low pressure of 500 psi to achieve removal and destruction of organics from solid matrices with minimum pretreatment and posttreatment requirements.

The integrated TD-SCWO process overcomes two fundamental problems, one related to TD and the other to SCWO. Traditionally, TD requires an afterburner and a complex pollution-abatement system (PAS) to destroy the desorbed organics, thus violating the non-incineration objective and compromising permitting and public acceptance. For the SRS application, the spread of Pu-238 contamination throughout the afterburner and PAS further compromises traditional TD technology. Traditionally, SCWO feed materials must be liquids or finely ground solids that can be slurried and pumped to high pressure. With TD-SCWO, however, TD desorbs organics from any form of solids and delivers the gas-phase organics to SCWO, thus resolving the feed pretreatment requirements of SCWO. In turn, SCWO resolves the afterburner/PAS requirements of TD by destroying the organics at SCWO conditions and capturing particulates, acids, and any other pollutants in the liquid effluent, and avoiding dioxin formation by rapid quench at the reactor outlet.

Integration of TD and SCWO subsystems is accomplished by operating at pressures of about 500 psig, retaining the advantages of SCWO and containing bulk solids in the pressurized TD. For the SRS application, a 55-gallon drum batch system is envisioned to minimize pretreatment

requirements and the potential spread of Pu-238 contamination. The proposed work was to be performed in phases as follows:

- Phase I - Proof of Principle Cold Demonstration
- Phase II - Proof of Principle Actual Waste Demonstration (decontamination technologies only, not applicable to TD-SCWO)
- Phase III - 1/5th or Larger Scale Cold Demonstration
- Phase IV - 1/5th or Larger Quantification and Actual Waste Treatment

This report covers the experimental work undertaken for the first phase of the overall development program, referred to as the Proof of Principle Cold Demonstration. Test results show that the process operates as intended. It provides a high degree of hydrogen removal and full containment of the radionuclide surrogate. All TD success criteria were demonstrated. All SCWO success criteria were also demonstrated with the exception of chlorinated solvent destruction and removal efficiency (DRE), which was slightly less than the target DRE during lower temperature tests. Maintaining somewhat higher SCWO temperatures will insure meeting the DRE success criterion. The process is ready to proceed to Phase III, the full-scale module cold demonstration. The facility at SRS may be comprised of as many as 5 full-scale modules, hence this demonstration has previously been referred to as "1/5th scale".

3. TEST OBJECTIVES

The primary objective of the Phase I Program was to demonstrate the ability of TD-SCWO to remove hydrogen from surrogate hydrogenous waste streams by nonincineration means. To qualify as a suitable technology, the proposed system would also have to show near-absolute containment of surrogate radionuclides and require minimal pre-treatment and post-treatment. Other test objectives were to verify that the process produces no dioxins or furans, and meets all applicable regulatory criteria for retention of toxic metals, particulate, and criteria pollutants, while meeting WIPP, WAC, and TRUPACT-II requirements. The Phase I testing utilized surrogate waste matrices chosen to be representative of Savannah River Site transuranic mixed wastes.

4. DESCRIPTION OF EQUIPMENT AND SET-UP

A process flow diagram (PFD) for the integrated TD-SCWO pilot plant at GA is shown in Fig. 1. To simulate an open 55-gal drum, a polyvinyl chloride (PVC)-lined steel canister without a top is packed with waste surrogate. The canister is placed within the desorber from the top and the desorber top head is then installed. A novel feature of this testing is the use of 500 psi as the SCWO operating pressure, which results in a much lower density reaction medium in comparison to the conventional SCWO operating pressure of 3400 psi.

To start the SCWO system, process water is supplied at 500 psi by the high pressure water pump, mixed with air supplied by a compressor, and passed through the heater. In the heater the process water and air are heated and vaporized by impedance heating, in which low voltage electrical current is passed through the walls of the piping. The resulting steam/air mixture heats the reactor to a temperature of about 700°F, at which point flow of auxiliary fuel (ethanol or diesel fuel) is begun. Reaction is initiated, and the heat of reaction brings the reactor temperature up to the nominal operating temperature of 1200-1500°F. Flow of cold, high pressure dilution water is now begun while the SCWO system is maintained at full operating

Figure 1. Process Flow Diagram (PFD) for GA TD-SCWO pilot plant.

conditions with auxiliary fuel awaiting the introduction of waste vapors from the desorber section.

Once the SCWO system has been started, the desorber wall heaters are energized. The pressure within the desorber is controlled at 500 psi by the back pressure of the SCWO system, and helps to access any containers within the canister to expose organics to desorption. As the desorber heats up, volatilized waste constituents pass through a heated line to the top of the SCWO reactor and are oxidized as they combine with excess oxidant in the reactor and then flow towards the reactor bottom. The flows of fuel and dilution water to the SCWO reactor are adjusted to compensate for the heating caused by oxidation of the TD vapors while air flow is kept constant.

In the SCWO reactor, the main constituents of the TD waste vapor are converted to CO_2 and H_2O in the oxidizing steam environment. Organically-bound nitrogen, e.g. from wood, is converted to a combination of N_2 and N_2O , while sulfur is converted to H_2SO_4 . HCl desorbed from the TD unit remains as HCl. Following mixing and oxidation the process stream flows to the SCWO reactor bottom where it mixes with a cold (and optionally alkaline) quench stream. Acid gases are converted to dissolved acids or salts and mercury, if present, is solubilized as mercuric chloride.

As shown in Fig. 1, the SCWO reactor incorporates a removable liner. The liner is fabricated from a suitable corrosion-resistant material, in this case titanium which has high resistance to chloride. The liner is relatively cheap and is easily removable to allow for the use of different materials, or for replacement should corrosion of the liner occur. In addition to isolating the reactor pressure vessel from corrosive species, the liner is purged with steam or nitrogen and has metallic insulation on its outer surface that shields the pressure vessel from the high temperatures in the reaction zone.

The quenched stream, with gases and dissolved salts, now exits the reactor. It is cooled to near ambient temperature by a series of annular heat exchangers with cooling water flowing through the annulus. The cooled process effluent is reduced in pressure to 250 psi and then flows to a gas-liquid separator from which the liquid and gas streams are depressurized separately. Following pressure letdown, the liquid effluent is monitored for pH. Liquid samples are collected as required from the line feeding the collection tank or from the collection tank itself.

The depressurized gas product is continuously analyzed on-line for CO , CO_2 , and O_2 content. Discrete gas samples can also be collected for later analysis. The gaseous effluent can be vented through a HEPA filter and a sulfur-impregnated carbon filter to capture any particles or mercury that may be present.

After desorption is complete, nominally a 7-hr period, the desorber wall heaters are turned off. The SCWO unit is shut down by discontinuing fuel flow. The reaction extinguishes and the heater is shut off. Cold water and air continue to flow to cool the reactor below 200°F , at which point all flows are stopped and the system is depressurized. Any desired inspections of the TD-SCWO system are now carried out. In particular, the desorber head is opened and the canister with residual solids is removed for inspection.

The key equipment items in the TD-SCWO system are the thermal desorber and the SCWO reactor. Diagrams of these vessels are shown in Figs. 2 and 3, respectively. A canister is

Figure 2. Pilot plant thermal desorber. Dimensions in inches.

Figure 3. Pilot plant SCWO reactor. Dimensions in inches.

shown within the desorber vessel in Figure 2. The general purpose thermal desorber has a design limit of 1150°F at 4000 psi. The general purpose SCWO reactor has a design limit of 1200°F at 4000 psi. Both vessels are operated at a reduced pressure of 500 psi for the SRS mixed waste application.

The canister to hold the waste charge in the pilot plant thermal desorber is fabricated of mild steel, with a length of about 34 in. (to mimic the 55-gal drums that would be used in the full-scale plant), an OD of 4 in. (maximum possible to fit within the desorber) and a wall thickness of 0.032 in. (minimum workable thickness). The bottom end of the canister is closed by welding on a disc of 0.032 in. steel. Thin PVC sheet of 0.012-in. thickness was used to line the canister to mimic the drum lining. The final volume within the lined canister is about 1.7 gallons, or about 3% of the size of an actual drum. Multiple canisters were made and canisters were sometimes reused.

Figure 4 shows the size of a canister as compared to a 55-gallon drum. The full height used for the canister was chosen to provide a worst case condition in terms of desorption path length.

Figure 4. Pilot plant TD canister and 55-gal drum.

Figures 5 through 7 are photographs of the thermal desorber, the SCWO reactor skid, and the SCWO reactor. Figures 5 and 7 correspond to the drawings of Figures 2 and 4, respectively. The proprietary appendix contains additional information regarding the equipment setup and operation.

Figure 5. Pilot plant thermal desorber.

Figure 6. Pilot plant SCWO reactor skid.

Figure 7. Pilot plant SCWO reactor.

Table 1 lists the surrogate recipes used in the testing. The recipes are based on typical drum compositions suggested by the Savannah River Site in December 2001. Cerium oxide has been added in a molar quantity to mimic plutonium-238 at a level of 5.7 grams per 55-gal drum, the maximum target value for TRUPACT-II containers. Carbon tetrachloride (CCl_4) and perchloroethylene (C_2Cl_4) spikes have been added in amounts of 1 wt%. Recipes do not include the weight of the drum/canister or PVC lining. The PVC lining was simulated by placing 34 grams of thin PVC sheet within the canister ID.

As shown in the table, recipe #1 contained about 600 g of volatile material, recipe #2 about 1000 g of volatile material, and recipe #3 about 1500 g of volatile material. Recipe #1 has the highest percentage of chlorine, about 10 wt%. Recipe #2 contains only about 5 wt% chlorine, but due to its greater mass has twice as much chlorine as Recipe #1 in absolute terms. Recipe #3 has only 1.7 wt% chlorine.

Table 1. Surrogate Recipes

Surrogate #1: Average Composition	wt%	kg per 55-gal drum	g per 1.7-gal canister	Notes
Iron base metal alloys	10.1	2.47	77.5	Use steel and stainless steel hardware
Other metals/Alloys	0.2	0.06	1.9	Use brass fittings
Oil Dry adsorbent	7.1	1.74	54.6	Purchase
Paper/Wood	38.8	9.5	298.2	Use available scrap
Lead lined gloves	0.9	0.21	6.6	Some supplied by SRS
PVC, polyethylene	40.9	10	313.9	Use household bags
CeO ₂	0.017		0.129	Purchase
CCl ₄	1.0		7.5	Purchase
C ₂ Cl ₄	1.0		7.5	Purchase
Total	100.0	24.0	767.8	
Approximate volatile content			600	
Surrogate #2: High metal and Lead				
Lead lined gloves	22.0	24.2	760.3	Some supplied by SRS
Other gloves (latex)	9.8	10.8	338.0	Purchase
PVC/Poly bags	4.5	4.9	154.9	Use household bags
Metal pieces (fittings)	59.4	65.5	2055.2	Use stainless steel fittings
Paint cans (empty but closed)	2.5	2.7	85.5	Use 4 oz. paint cans
CeO ₂	0.002		0.1	Purchase
CCl ₄	1.0		33.9	Purchase
C ₂ Cl ₄	1.0		33.9	Purchase
Total	100.0	108.1	3461.8	
Approximate volatile content			1000	
Surrogate #3: High Plastic and Adsorbent				
Organics (isopropanol) on wipes	11.0	8.6	271.2	Moisten wipes with isopropanol
Oil Dry adsorbent	26.7	21.0	660.5	Purchase
Aerosol cans with cleaner	5.2	4.1	128.7	Use isopropanol in 4 oz. paint cans
Glass (substitute for mercury thermometer)	5.8	4.6	144.7	Use available scrap
Polypropylene	47.8	37.7	1183.8	Purchase
HEPA Material	1.5	1.2	37.5	Purchase
CeO ₂	0.005		0.129	Purchase
CCl ₄	1.0		24.3	Purchase
C ₂ Cl ₄	1.0		24.3	Purchase
Total	100.0	77.3	2475.0	
Approximate volatile content			1500	

5. TESTING

5.1. Thermal Desorber Tests

The initial test series involved only the thermal desorber. A total of 36 such tests were carried out using the different surrogates and testing the effect of various parameters. Table 2 presents the parameters that were explored. The primary evaluation methods for these runs were observation of the varying rate of gas evolution, visual examination of the desorber residue and any tar or scale formation in the desorber exit line, and analysis of the canister residue for hydrogen. To determine gas evolution rate, the exit line from the desorber was passed through a coil in a water bath, through a liquid knockout trap, and then through a back-pressure regulator and a gas flowmeter. This setup provided information on the time course of volatilization and facilitated the setting of desorption parameters. Figure 8 shows the process flow diagram for the TD-only tests.

Table 2. Parameters for TD-Only Tests

Parameter	Range	Result
Pressure	500-1000 psig	Negligible difference between 500 and 1000 psi – use 500 psi
Temperature	600-1150°F	932°F (500°C) is sufficient for low residual hydrogen
Desorption time (heaters on to heaters off)	1-8 hr	Low residual hydrogen can be achieved in 2 hours – one hour heating up to 932°F and 1 hour holding at 932°F
Steam flow	0-8 lb/hr	Steam does little to reduce residual hydrogen and exacerbates corrosion by wet hydrochloric acid. Both 316 stainless steel and Alloy C-276 are subject to such corrosion.
Air flow	0-1.4 scfm	Introduction of air to the TD unit causes combustion and hot spots. Avoidance of hot spots requires such a low flow rate as to be impractical.

5.2. Thermal Desorber-SCWO Integration and Optimization Tests

Following the TD-only tests the range of favorable desorber parameters was used to set conditions for the integrated TD-SCWO system. The TD heatup time was increased from about 2 hours to 4-7 hours to properly match the SCWO reactor capacity. The longer heatup times are also closer to those expected for the full-scale application. The SCWO system was operated at essentially the same pressure as the desorber, 500 psi. During the integration and optimization tests a SCWO operating temperature of about 1300°F was found to be satisfactory, but this was eventually raised to 1540°F to reduce the CO spikes and insure good destruction efficiencies. The SCWO reactor residence time was 7-10 seconds. It was found that the SCWO effluent pH stayed above 2, and there was no need to use an alkaline quench.

Figure 8. PFD for TD-only tests.

5.3. Demonstration Tests

Following the TD-SCWO integration and optimization tests the final series of demonstration tests was undertaken. Appendix A provides a sample Run Plan for one of the TD-SCWO demonstration runs to illustrate system flows and procedures. Note that each successive run was performed at a slightly higher SCWO reactor temperature to reduce the CO spikes and improve destruction efficiencies during periods of high volatiles desorption.

Surrogate Recipe #2, 1300°F SCWO Temperature

Figure 9 shows key data for Run 8.2, which utilized recipe #2. The upper plot shows the thermal desorber heater set point temperature, which was controlled by thermocouples mounted on the outside wall of the desorber pressure vessel. There are actually three zones of “clam shell” heaters along the vessel and three separate heater controllers. For this test the heater set points were all entered manually, as indicated by the series of steps in the set point curve. Also shown in the upper plot is the TD internal temperature as measured by a thermocouple positioned just above the canister. In this case about 3 hours were required for the TD internal temperature to reach ~932°F (500°C), and this temperature was then held for about 1 hour.

The lower plot in Figure 9 shows corresponding key data for the SCWO unit. The maximum reactor temperature was about 1300°F (700°C). The SCWO gas effluent shows two distinct episodes of oxygen drop and carbon dioxide rise, signifying peaks in the volatile material coming over from the TD unit. During these separate desorption intervals the SCWO reactor temperature dropped briefly to relatively low levels in the range of 900°F (500°C). The higher rate of volatiles would normally give a higher SCWO reactor temperature but the system operator was instructed to increase the flow rate of cold diluting water to the SCWO reactor to compensate for increasing temperatures. Close control of reactor temperature was difficult during high rates of desorption due to the rapidly changing rate of volatiles desorption. During the second episode of high volatiles in Figure 9 the TD set point was reduced to slow the rate of desorption.

The TD-SCWO integrated system performed well and the liquid effluent remained clean.

Figure 9. Plots for Run 8.2, recipe #2.

Surrogate Recipe #1, 1400°F SCWO Temperature

Figure 10 shows key data for Run 10.1, which utilized recipe #1. This was the run during which SCWO gas effluent sampling was carried out by AirKinetics, Inc., and also the run for which the Run Plan is given in Appendix A. As compared to Run 8.2, a slower ramp of TD temperature was used - about 5.5 hours to 1000°F, followed by a 1-hr hold time at 1000°F. The maximum reactor temperature was held at nearly 1400°F (750°C). The lower plot in Figure 10 shows only a single protracted episode of high volatiles desorption as the TD temperature increases from about 660°F (350°C) to 930°F (500°C). The slower rate of change of volatiles desorption makes control of the SCWO reactor easier, and operation is smooth with temperatures above 1100°F (600°C) the entire time.

During the high volatiles desorption period, the CO level is seen to rise notably in Figure 10, signifying a short period of incomplete oxidation. When the results of the AirKinetics gas sampling analysis were received, the measured levels for carbon tetrachloride (CCl₄) and perchloroethylene (C₂Cl₄) were higher than expected, and likely due to inadequate time at temperature. Slightly higher SCWO temperatures are therefore needed to insure complete destruction of chlorinated compounds.

Surrogate Recipe #3, 1470°F SCWO Temperature

Figure 11 shows key data for Run 11.0, which utilized recipe #3. The ramp of TD temperature was essentially the same as that used during Run 10.1. In addition to using the recipe with the highest volatile content, this run utilized a maximum SCWO reactor temperature of about 1470°F (800°C). The higher reactor temperature had a significant effect on lowering gas effluent CO levels, with an average of 12 ppm over the course of the desorption.

Surrogate Recipe #1, 1540°F SCWO Temperature

Figure 12 shows key data for Run 12.1, which utilized recipe #1. This was the final run of the Phase I program, and was carried out for the site visit from DOE representatives. The ramp of TD temperature was essentially the same as that used during Runs 10.1 and 11.0. This run utilized a maximum SCWO reactor temperature of about 1540°F (840°C), and gas effluent CO was now near zero for the entire desorption period.

Figure 10. Plots for Run 10.1, recipe #1.

Figure 11. Plots for Run 11.0, recipe #3.

Figure 12. Plots for Run 12.1, recipe #1.

Figure 13 shows the appearance of the materials used to make up a single canister charge for recipe #2.

Figure 13. Ingredients for recipe #2.

Figure 14 shows the appearance of the recipe #2 materials after desorption. As shown in the photo, closed cans were typically deformed and had their lids disrupted. Residue from leaded gloves appeared as a charred version of the original material. Lead was originally present in the gloves as a red-orange layer signifying red lead oxide. Red lead oxide decomposes to metal at 500°C, however there were no indications of any molten lead metal. In addition, the residues contained low residual moisture. This is important because even inorganic hydrogen, e.g. in the form of water, can contribute to hydrogen generation in the actual radioactive waste.

Figure 15 provides a comparison of the desorbed material entering the SCWO unit with the liquid leaving the SCWO unit. This is an indication of the very clean effluents that are produced by the SCWO process. It should be noted that the condensate materials shown in Figure 15 are not normally observed during the TD-SCWO process, as they are generated in the TD unit and proceed directly to the SCWO unit. The condensate photos shown are of material collected from a TD-only test. As shown in the figure, some of the TD condensate is thick and tarry at room temperature.

Figure 14. Residue from a run with recipe #2.

Tarry TD Condensate

Liquid TD Condensate

SCWO Effluent

Figure 15. Comparison of materials entering and leaving the SCWO unit.

5.4. Analytical Results

Ultimate analysis of TD solid residues was carried out by Hazen Research, Inc., Golden, CO. The main purpose of this analysis was to determine residual hydrogen in the residue. Ultimate analysis was carried out on many TD-only runs as well most of the TD-SCWO runs. The results for the TD-SCWO testing are given in Table 3. As was shown in Figure 14, it was still possible in many cases to identify which residues had come from which starting materials. Since homogenization of entire batches would have been very difficult, several different samples were usually analyzed to determine the range of hydrogen content. As seen in the table, residual hydrogen ranged from 0.35% to 2.90%. In Table 3, the entry "Paper/IPA" indicates paper towels soaked with isopropyl alcohol. The entry "Typical" indicates miscellaneous ash that could not be identified with a particular starting material. Also, oxygen content was determined by difference. The can crystals found in Run 7.0 are of unknown composition, but contain high moisture content that may have been picked up from the atmosphere.

Table 3. Ultimate Analysis Results for TD Solids

TD-SCWO Run	Feed Recipe	Sample	Wt%						
			Moisture	Ash	S	C	H	N	O
5.0	#3	HEPA	4.97	20.50	0.09	72.40	1.34	1.63	<0.01
		Paper/IPA	10.99	9.46	0.11	78.13	0.84	0.16	0.31
		Typical	2.45	90.58	0.03	5.29	0.46	0.02	1.17
6.0	#1	Wood	5.13	1.79	0.02	92.26	1.44	0.23	<0.01
		PVC	3.91	7.91	0.05	87.07	1.94	0.11	<0.01
		Paper	6.05	7.87	0.10	79.81	1.69	0.20	4.28
		Typical	4.83	12.70	0.04	79.63	1.85	0.06	0.89
7.0	#2	Bottom	0.94	24.89	0.47	37.40	0.80	0.03	35.47
		PVC	2.68	6.97	0.10	80.18	1.52	0.12	8.43
		Can crystals	13.87	24.89	0.19	40.50	0.79	0.01	19.75
		Polypropylene	3.40	14.05	0.22	80.65	1.42	0.11	0.15
8.2	#2	Typical	0.58	27.54	0.35	15.92	0.44	<0.01	55.17
		Leaded glove	0.45	24.56	0.42	11.68	0.35	<0.01	62.54
		PVC	1.22	16.23	0.23	37.76	0.86	0.02	43.68
9.3	#3	Typical	0.72	91.12	0.06	5.23	1.71	<0.01	1.16
		HEPA	2.82	81.47	0.21	6.37	1.09	<0.01	8.04
		Paper/IPA	1.19	10.75	0.10	85.24	2.90	0.08	<0.01
10.1	#1	Paper	3.60	5.73	0.03	86.87	2.03	0.26	1.48
		PVC	3.08	8.50	0.03	86.33	1.97	0.14	<0.01
		Typical	2.18	79.82	0.06	15.27	0.55	0.04	2.08
		Wood	2.35	4.35	<0.01	88.42	2.42	0.25	2.21
11.0	#3	Paper/IPA	5.57	6.49	0.05	82.56	1.86	0.35	3.12
		HEPA	6.20	74.99	0.05	11.49	0.36	0.15	6.76
		PVC	2.69	21.75	0.03	75.17	1.75	0.11	<0.01
		Typical	1.35	85.62	0.04	11.20	0.79	0.04	0.95
		Wood	0.64	4.39	0.05	91.51	2.62	0.21	0.58
12.0	#1	Paper	5.07	5.05	0.03	83.19	1.76	0.32	4.58
		PVC	2.62	8.18	0.05	86.89	2.12	0.16	<0.01
		Typical	1.16	80.32	0.05	16.15	0.78	0.01	1.53

The ultimate analysis shows that residual moisture content varies from less than 1% to a high of nearly 14%. It should be noted that samples were not sealed and some residual moisture may have been picked up from the atmosphere.

Cerium was measured in solid residue and TD condensate in two TD-only runs. These are shown in Table 4. Cerium oxide was originally distributed throughout the canister charge. Subsequent to desorption, cerium was only detected in the residual solids at the bottom of the canister. No cerium was found in the small deposits located on the upper canister walls or vessel walls. Most importantly, no cerium was found in the TD condensate, indicating that no cerium would make its way to the SCWO unit. There was no attempt to account for all the cerium that was originally added to the canister as this would have involved very lengthy sample collection and analysis procedures due to the heterogeneity of the canister residue. While plutonium will not necessarily behave identically to the cerium surrogate, the cerium results nevertheless provide a positive indication for retention of plutonium in the waste drums.

Table 4. Cerium Analysis Results for TD Solids and Condensate

TD Run:	#17	#18
Feed recipe	#3	#1
Ce in solids from inside bottom of canister	10 mg/kg	Not analyzed
Ce in solids from inside top of canister	<16 mg/kg*	Not analyzed
Ce in solids from canister OD	<2.1 mg/kg	No solid present
Ce in solids from vessel wall above canister	<1.8 mg/kg	<9.3 mg/kg
Ce in condensate liquid (TD only)	<0.20 mg/L	<0.20 mg/L

*< indicates nondetectable

The SCWO liquid effluent was analyzed to confirm the immobility of cerium, to check for lead carryover, and to assess organic destruction. These analyses, carried out by Alpha Analytical Labs in Westborough, MA, are shown in Table 5. The "Sample" column shows at what time a particular grab sample was taken. Each sample was analyzed for only one analyte. As expected from the TD condensate results, cerium was nondetectable in the SCWO effluent. Lead was detected at a low level, well below the TCLP limit of 0.75 ppm. Total organic carbon (TOC) results were comparable to those observed with conventional SCWO at 3400 psi, i.e., in the range of 1 ppm.

Table 5. Analytical Results for SCWO Liquid Effluent

TD-SCWO Run	Feed Recipe	Sample	Ce, mg/L*	Pb, mg/L	TOC, mg/L
8.2	#2	12:15			1.2
		12:30		0.035	
		12:45	<0.10		
10.1	#1	17:00			1.3
		17:15	<0.10		
11.0	#3	19:30			1.3
		19:45	<0.10		
12.0	#1	15:15			1.0
		15:30	<0.10		

*< indicates nondetectable

As previously mentioned, the SCWO gas effluent was analyzed for key target species during Run 10.1. Samples were collected and analyzed by AirKinetics, Inc. of Huntington Beach, CA. Table 6 provides a summary of these results. The gas analyses will be further discussed below in the context of the test success criteria.

Table 6. Analytical Results for SCWO Gas Effluent

Analyte	Sampling Time	Units	Results
Particulates	12:50-17:48	mg/dscm	0.105 (= 5×10^{-5} grains/dscf)
Total PCDDs	11:47-17:47	ng/dscm	0.00489 (No 2,3,7,8 isomers, TEQ = 0)*
Total PCDFs			0.0192 (No 2,3,7,8 isomers, TEQ = 0)
Total PCDDs and PCDFs			0.0240 (No 2,3,7,8 isomers, TEQ = 0)
Antimony	12:50-17:48	µg/dscm	0.0131
Arsenic			ND (<0.565)
Beryllium			ND (<0.283)
Cadmium			0.0216
Chromium			ND (<0.565)
Cerium			ND (<1.40)
Lead			0.148
Mercury			0.158
Carbon tetrachloride	12:50-13:50	ppbvd	60
Perchloroethylene			68

*Ref. 3

5.5. Success Criteria

Success of the demonstration tests was measured by (1) attainment of hydrogen reduction goals, (2) meeting environmental criteria for processed waste and effluents, and (3) equipment operability at the specified conditions. Specific success criteria for the Phase I Cold Demonstration tests are defined in Table 7, along with the status based on results from the Phase I testing.

The TD-SCWO process met all success criteria with the exception of demonstrating >99.99% destruction and removal efficiency of carbon tetrachloride and perchloroethylene. Assuming the sample of Table 6 captured the bulk of the desorption peak of these compounds, as intended, a DRE of about 99.9% for each is calculated. This result is attributed to a low SCWO temperature during the gas sampling test (Run 10.1). A number of studies have shown that SCWO can attain >99.99% destruction of highly refractory chlorinated organics such as PCBs (Ref. 4) and hexachlorobenzene (unpublished GA/Modar data). Carbon monoxide is one of the more

Table 7. Success Criteria

Requirement	Success Criteria	Status
Nonincineration	Peak process temperature measured via thermocouple to be <1600°F (871°C)	Demonstrated, see Figures 9-12
Hydrogen Removal	Total hydrogenous organic in residue <5 wt %	Demonstrated, see Table 8
Destruction and Removal Efficiency	Spiked constituents (CCl ₄ and C ₂ Cl ₄) measured in gaseous effluent to verify compliance with >99.99% destruction and removal	Demonstrated ~ 99.9% DRE
CO	CO level in gaseous effluent verified to be <100 ppm	Demonstrated, see Figures 10-12
Particulate Emissions	Particulate level in gaseous effluent verified to be <0.015 grains/dscf	Demonstrated, see Table 6
Mercury Removal	Hg level in gaseous effluent verified to be <45 µg/dscm	Demonstrated, see Table 6 (no Hg added to feed)
Cadmium and Lead Removal	Cd + Pb level in gaseous effluent verified to be <0.024 mg/dscm	Demonstrated, see Table 6
Antimony, Arsenic, Beryllium, and Chromium Removal	Sb + As + Be + Cr level in gaseous effluent verified to be <0.097 mg/dscm	Demonstrated, see Table 6
Radioactive Particle Containment	Measure CeO ₂ levels in gaseous and liquid effluent to determine degree of containment	Demonstrated, see Tables 4-6
Minimum Pretreatment	Minimum pretreatment requirements relative to other technologies	Demonstrated
Minimum Posttreatment	Minimum posttreatment gas cleaning process relative to other technologies	Demonstrated
Dioxin/Furan Emissions	Total dioxins and furans in gaseous effluent verified to be less than regulated levels of 0.2 ng/dscm (TEQ) at 7% O ₂	Demonstrated, see Table 6
WIPP/WAC	Meets requirements	Demonstrated, see Table 9
TRUPACT-II	Meets requirements	Demonstrated, see Table 9

refractory compounds with regard to oxidation, and is a good general indicator of the completeness of reaction. When the SCWO reactor temperature is sufficiently high, CO levels will normally be less than a few ppm. As seen in Figure 10, this was not the case during Run 10.1, when the AirKinetics sampling was carried out and the maximum reactor temperature was only about 1400°F (750°C). In contrast, for Run 12.1 (Figure 12) with a maximum SCWO reactor temperature of about 1540°F (840°C), CO is near zero as expected for good SCWO operation. Based on prior SCWO data as well as the low CO level, it is likely that the >99.99% DRE criterion for the chlorinated solvents was met in Run 12.1. However, the Phase I program plan had allowance for only a single gas sampling event and gas analyses were not carried out for Run 12.1. Additional analyses for chlorinated hydrocarbons will be carried out in Phase III to demonstrate satisfactory DREs.

As seen in Table 7, the requirement for hydrogen removal was framed in terms of “hydrogenous organic”. This criterion is believed to address the shipping requirement that a container headspace should not comprise more than 5% of flammable gases including hydrogen. However, different organic constituents can contain widely differing hydrogen contents. Furthermore, gaseous hydrogen can be produced from inorganic sources (including water) as well as organic sources. As an indicator of the effectiveness of TD-SCWO for hydrogen removal, calculations were carried out to show that the hydrogen in the TD residues was equivalent to a polyethylene content of less than 5 wt%. These calculations are shown in Table 8 for one run with each recipe. From the measured mass of a residue, the mass of nonvolatile inorganics (e.g. metal parts, fiberglass from HEPA filters, oil-dry) was subtracted. This gave a residual quantity of char as shown in line 8 of the table. To be conservative, this char was assigned the maximum of the several hydrogen contents that had been determined by ultimate analysis (see Table 3), resulting in a residual hydrogen content as shown in line 10 of the table. This residual hydrogen was converted to a percent of the original mass (line 11) and finally expressed in terms of polyethylene (CH_2) equivalent (line 12). On this basis the hydrogen reduction meets the stipulated success criterion of less than 5% hydrogenous organic. As another measure of the treatment effectiveness, line 13 shows the factor by which hydrogen level has been reduced by the TD process. Finally, line 14 gives the factor by which the residual hydrogenous organic level bettered the required level of 5% (5% divided by line 12).

Table 8. Determination of Hydrogenous Organic

	TD-SCWO Run:	Run 12.0	Run 8.2	Run 9.3
	Feed Recipe	#1	#2	#3
1	Initial mass in recipe, g	802	3496	2509
2	Nonvolatiles in recipe, g	136	2331	856
3	Organics in recipe, g	666	1165	1653
4	Hydrogen in recipe, g	50	61	203
5	Allowable organic after treatment (5%), g	40.1	175	125
6	Allowable H after treatment (as CH_2), g	5.7	25	17.9
7	Total residue, g	245	2900	875
8	Char residue, g	109	569	19
9	Measured max H% in char residue	2.12	0.86	2.9
10	Residual H calculated, g	2.3	4.89	0.54
11	Final %H based on initial mass	0.3	0.14	0.02
12	Final % CH_2 equivalent (to be <5%)	2.0	0.98	0.15
13	H reduction factor	21	12	374
14	H reduction factor below 5%	2.5	5.1	33

Table 7 calls for the TD residue to meet WIPP/WAC and TRUPACT-II requirements. Table 9 provides a detail of these requirements along with the status based on results from the Phase I testing. There do not appear to be any concerns in meeting all of these requirements.

Table 9. WIPP/WAC and TRUPACT II Requirements

Waste Type	Waste Acceptance Criteria	TD-SCWO Phase I Results
Free liquids	Less than 1 volume % of external container. Less than 1 inch in bottom of internal containers.	No free liquids observed in Phase I residues.
Pyrophoric materials	<1% radionuclide pyrophorics. No nonradioactive pyrophorics	No pyrophoric materials observed in Phase I residues. (None in feed.)
Sealed containers	No sealed containers greater than 4 liters	Containers crushed and/or lids separated in Phase I testing.
Hazardous waste	Limited to RCRA hazardous waste codes that ensure no ignitable, corrosive, or reactive characteristics	Phase I residues are relatively inert inorganics and char.
Explosives, corrosives, and compressed gases	No explosives, corrosives, or compressed gases	No explosives, corrosives, or compressed gases observed in Phase I residues. (No explosives or compressed gases in feed.)
Headspace gas VOC concentrations	Every container will be headspace gas sampled	No residual VOCs observed in Phase I residues.
PCB concentration	<50 ppm	Not tested in Phase I. High temperature in TD will ensure that no PCBs remain.
Test category waste	Steady-state hydrogen gas generation rate is less than or equal to the rate specified in the TRUPACT-II Safety Analysis	Phase I shows total hydrogenous organic in residue <5 wt%. Typically <<5 wt%.
Flammable VOCs	<500 ppm total in the headspace of any payload container	No flammable VOCs observed in Phase I residues.

6. CONCLUSION

The findings of the Phase I testing are as follows:

- Thermal desorption of surrogate SRS mixed wastes at 500 psi and 1000°F meets all tested requirements for WIPP/WAC and TRUPACT-II.
- SCWO of the desorbed surrogate organic materials at 500 psi and 1500°F appears to meet all requirements for a nonincineration alternative. DRE for chlorinated solvents was slightly less than required during the lower temperature SCWO tests, so this temperature should be maintained to insure destruction.
- Air oxidant is preferred over pure oxygen for SCWO to allow the use of a corrosion-resistant titanium lining.
- TD-SCWO technology is ready to proceed to Phase III, the full-scale module cold demonstration.

Based on the results obtained during Phase I, TD-SCWO appears to represent a promising alternative to incineration of mixed radioactive wastes. Potential benefits for the application of TD-SCWO technology are discussed below.

Low Life Cycle Costs. TD-SCWO will require less gas treatment technology than any known competitive technology due to the internal scrubbing achieved by SCWO. At most, a chiller, activated carbon and HEPA filters will be required to capture trace mercury and other trace contaminants. TD-SCWO is extremely compact, requiring a smaller footprint than any known competitive technology due to the higher throughput achieved at elevated pressure and smaller gas cleaning system. Reduced carryover of Pu-238 from the batch TD to SCWO may enable the SCWO system to be contact-maintained - a potentially significant advantage over competing technologies. For nonradioactive applications, GA has performed cost analyses that have generally compared favorably with incineration for the same waste feed.

Low Health and Safety Risks to the Worker and the Public. TD-SCWO is a sealed, pressurized system with the pressurized components enclosed by secondary containment and vented through HEPA filters and activated carbon. In the event of process upsets, relief valves open to discharge the system contents to a dump tank for reprocessing at a later date. All maintenance operations are performed on the depressurized system. Effluents are monitored continuously to ensure that they meet all regulatory standards. Leading indicators of process performance are also tracked by the process control computer system to provide ongoing assurance of operation within specification.

Low Risks for Detrimental Impact to the Environment. TD-SCWO is an inherently clean process that eliminates the conditions that lead to de novo synthesis of dioxins or furans. TD-SCWO will minimize emissions of the metals and radionuclides that are currently in the drummed wastes by retaining most of these wastes in the TD, and capturing the remainder in the SCWO liquid effluent.

Low Quantity of Waste Materials Requiring Disposal. TD-SCWO will densify the wastes allowing repackaging into fewer drums for transport to WIPP. Solid effluents from SCWO (obtained by filtering and drying the SCWO liquid effluent) will generally be lower in volume than for any other technology, because TD-SCWO mineralizes all organic feeds to CO₂, water, and mineral salts. This will maximize the volume reduction and minimize the resultant residues to be disposed.

Low Amount of Secondary Waste Generated. Because TD-SCWO does not produce dioxins or furans, no activated carbon is required to meet the stringent new Clean Air Maximum Achievable Control Technology (MACT) standards for dioxin and furan emissions. Mercury emissions will be collected on activated carbon filters (sulfur impregnated to fix the mercury in an insoluble form) and will be disposed of as a stabilized hazardous waste. TD-SCWO is expected to generate less secondary waste than competitive technologies. With the exception of mercury (mercury was not tested), metals including radionuclides and other particulates will be retained within the TD-SCWO system and will not add loading to the backup HEPA filters, thus reducing the frequency of replacement of these filters. Solids are discharged from the batch TD along with the other drum residues, and are filtered out of the SCWO liquid effluents for subsequent drying and disposal. Salts crystallized from SCWO liquid effluent (primarily NaCl from PVC processing) will be recombined with the main process solids discharge and repackaged for transport to WIPP. Here, again, TD-SCWO should produce less secondary waste than other competitive technologies because of the simpler gas cleaning process.

Low Hazard Level and Category of Waste. TD-SCWO will efficiently destroy all organic constituents in the waste. Metals (except mercury which was not tested) and radionuclides will be retained with the bulk desorbed waste and will be repackaged for disposal at WIPP. Mercury

will be retained on sulfur-impregnated activated carbon for off-site disposal. Remaining metals may prevent the residue from meeting the Land Disposal Ban, as measured via Toxic Characteristic Leaching Procedure (TCLP) testing, and further stabilization may be required. Most importantly, TD-SCWO will not generate any dioxins or furans, which is less likely for competitive technologies which do not eliminate the conditions that lead to de novo synthesis of dioxins and furans.

Ability to Meet Regulatory Requirements. TD-SCWO inherently meets all regulatory criteria under the MACT rules. This includes destruction of organic compounds, retention of metals within the process, and all emissions within regulatory standards, including particulates, HCl, SO_x, NO_x, and CO, and again, dioxins and furans. Competitive technologies are less likely to meet all regulatory requirements without extensive additional pollution abatement equipment. RCRA permits have been obtained for three of GA's SCWO systems in record time, attesting to the degree to which the technology meets regulatory requirements.

7. FULL-SCALE TD-SCWO MODULE

Phase III involves the construction and cold-testing of a full-scale TD-SCWO module for the targeted SRS drummed wastes. The full-scale TD-SCWO module is sized to treat a 55-gal drum, including overpacked drums. Table 10 provides a comparison of some of the key design features of the existing pilot plant with the full-scale scale module, which represents a scale-up of 32x. Both systems use a single thermal desorber and cold-wall operation of the SCWO reactor. As shown in the table, the desorption heating cycle time for the full-scale scale module should be about the same as for the Phase 1 tests as both the quantity of organic and the SCWO reactor are scaled up by a factor of 32.

For additional capacity, multiple 55-gallon TD modules can be added feeding a single SCWO system. The TD units would cycle between loading, operating, and unloading, alternately feeding the continuously operating SCWO unit. If necessary, active cooling can be used to decrease the turnaround time for the TD units. In this manner, with an 8-hr heatup/desorption time, a single full-scale SCWO unit can process 3 drums per day. For even greater capacity, multiple TD-SCWO modules may be installed.

Table 10. Comparison of TD-SCWO Pilot Plant and Full-Scale Module

Design Parameter	Pilot-Scale (existing)	Full-Scale Module
Batch TD waste volume	1.7 gallons	55 gallons
TD heating cycle time	7 hours	8-10 hours
Batch TD dimensions	4" ID x 5 ft tall	26" ID x 4 ft tall
SCWO reactor dimensions	4" ID x 5 ft tall	15" ID x 12 ft tall
SCWO heat release	44,000 Btu/hr	1.4 MMBtu/hr
SCWO air demand	40 lb/hr	1300 lb/hr
System pressure	500 psig	500 psig
System temperature	1000–1600°F	1000–1600°F

Figure 16 shows a process flow diagram applicable to a full-scale TD-SCWO module. Figure 17 shows an architectural rendering of a full-scale TD-SCWO module facility. Drums will be accessed for TD by remote removal of a bung plug or the entire drum lid. The full-scale TD-SCWO module control system will incorporate automated SCWO reactor temperature control to enhance ease of operation. If the low carryover of radionuclides suggested by the Phase I

testing holds true, the SCWO system will be amenable to contact maintenance and operating costs will be significantly reduced.

8. REFERENCES

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Figure 16. Process flow diagram for a full- scale TD-SCWO module.

Figure 17. Architectural rendering of a full- scale TD-SCWO facility.

APPENDIX A. Sample TD-SCWO Run Plan

Run Number:	TDSCWO-10.1
Date:	09/19/02
Project:	TD SCWO – Thermal Desorption-Supercritical Water Oxidation
Run Description:	Recipe #1
Approved:	
Charge #	30117300

1. Objectives

- Volatilize organics in the thermal desorber (TD) and oxidize in SCWO unit at 500 psi.
- No quench flow to minimize CO spikes.
- Gas sampling to be carried out by AirKinetics.

2. Test Conditions

Feed and feed concentration	Hot gases from desorber
Feed flowrate est.	30 g organic/min peak + 65 g/min of TD nitrogen purge
Feed temperature	<500°C
Process water flowrate	130 g/min through Ohmic heater from P-300
Dilution water flowrate	As required to maintain reactor temp (650-700°C). From P-100 to nozzle core.
Fuel	Diesel
Fuel flowrate	~ 5 to 10 g/min, adjust to achieve reactor at 650°C with minimum flow from P-100.
Vessel reactor temperature	650°C (TE-652)
Air flowrate	300 g/min
Reactor pressure	500 psig
Reactor τ	~7-8 seconds at peak desorber flow
Purge fluid	Water
Purge flowrate	20 g/min
Quench flowrate	Off
Quenched temperature	<500°C
Effluent pH	Should be > 2.0 based on prior runs
Hours at Steady State	TD at ~500°C for 1 hour

3. Feed Recipe (Desorber canister charge)

Surrogate #1: Average Composition	wt%	g per canister	Notes
Iron base metal alloys	10.09	77.5	Use steel and stainless steel hardware
Other metals/alloys	0.25	1.9	Use brass fittings
Oil Dry (kitty litter)	7.11	54.6	Purchase
Paper/Wood	38.84	298.2	Use scrap
Lead lined gloves, gloves, gaskets	0.86	6.6	Use lead-lined gloves supplied by SRS
PVC, polyethylene	40.88	313.9	Use household bags, etc.
CeO ₂	0.02	0.129	Purchase
CCl ₄	0.98	7.5	Purchase
C ₂ Cl ₄	0.98	7.5	Purchase
Total	100.0	767.7	

The canister should be lined with 34 grams of PVC to simulate the drum liner before loading the surrogate.

4. Configuration and Setup

1. See attached PFD.
2. Calibrate low range CO meter at 100 ppm. We are required to demonstrate CO<100 ppm.
3. Clean the ½" x 0.065-in. C-276 line connecting the TD and SCWO units. The orifice has been removed since run 8.1.
4. Vessel reactor internals are removable C-276 insulated liner with titanium Grade 2, 3-in Sch. 10 pipe (3.5 in. OD x 3.28 in. ID).
5. The nozzle core tube will carry the hot (but cooling down) gases from the TD, diesel fuel and DI water (P-100). The core tube will be ¼ inch x 0.065 wall Alloy C-276 tubing.
6. The nozzle annulus tube will carry pre-heated air and water. The annulus tube will be a standard Dugway nozzle, ½" x 0.065 wall Alloy C-276 tubing, with the orifice block drilled through to 0.277 inches to allow the core tube to end flush with the annulus.
7. The optional gas and liquid effluent filters are not installed for this run.
8. All heaters on the TD unit and the jumper heating tape are operational.

5. Procedure

1. Startup
 - a. Pressurize reactor with water and air and heat to $\geq 270^{\circ}\text{C}$ using air and water through the preheater. Activate heating tape on overhead line from TD to SCWO.
 - b. Start P-100 process water at minimum reliable flow.
 - c. Start diesel fuel at ~ 3 g/min and adjust as required to attain reactor temperature of 650°C . Ramp fuel to target reactor temperature while adjusting pre-heater outlet temperature to $\sim 550^{\circ}\text{C}$ to 600°C . In view of the low operating pressure, make sure system is operating cleanly with low CO.
2. Transition to feed and operate
 - a. Use the following ramp rates for the TD heaters:
 1. $25^{\circ}\text{C}/\text{min}$ to 250°C .
 2. $3^{\circ}\text{C}/\text{min}$ to 370°C .
 3. $0.5^{\circ}\text{C}/\text{min}$ to 450°C .
 4. $1^{\circ}\text{C}/\text{min}$ to 480°C .
 5. $10^{\circ}\text{C}/\text{min}$ to 550°C . When setpoint is reached, raise setpoint to 580°C .
When setpoint is reached, raise setpoint to 600°C .
 - b. Set P-100 as necessary to maintain 650°C in SCWO reactor.
 - c. Run until TD has been at $\sim 500^{\circ}\text{C}$ for 1 hour.
3. Shut down
 - a. Turn off TD heaters.
 - b. Turn off ohmic heater.
 - c. Turn off fuel, then air.
 - d. Shut down all SCWO system flows. Allow GLS to drain to depressurize SCWO system.
 - e. Disconnect the TD and SCWO systems.

6. Contingencies

1. Watch for feed line plugging. As the TD vapors cool from mixing with P-100 water, tars will condense in the feed line.

2. Hydrochloric acid corrosion of the nozzle and downstream components, especially the steel GLS, may be a problem. Quench may be brought on if the effluent pH falls below 2.

7. Sample Schedule

Sample #	Description	Frequency	Quantity	Analyses
MMDDYY HH:MM – Effluent	Liquid effluent	15 min	250 ml	TOC, cerium
TD-SCWO1-Sx (x indicates sample number)	TD residues	End of run		Ultimate, cerium
AirKinetics sampling	Gas Effluent	Start when TD temperature is 240°C		See AirKinetics quote

Only selected samples will be analyzed, others will be saved for archival purposes.

8. Post-Run Inspection

1. Inspect TD canister and take usual weights and samples. If run was successful, send usual solid samples to Hazen.
2. Inspect nozzle and TD-SCWO connecting line.
3. Take digital photographs of significant items.
4. Complete post run inspection sheet prior to TD-SCWO restart.

GA Pilot Plant Run Plan

APPENDIX B. AirKinetics Report on Gas Effluent Analyses

ENGINEERING TEST REPORT

SCRUBBER EFFLUENT

Source Location:

**General Atomics
3550 General Atomics Ct
San Diego, California 92121-1122**

Test Date: September 19, 2002**Issue Date: October 23, 2002****Revision: 0****Prepared for:**

**General Atomics
18 Wachusett View Drive
Westborough, Massachusetts 01581**

Prepared by:

**AirKinetics, Inc.
5932 Bolsa Avenue, Suite 105
Huntington Beach, California 92649
714-373-0998
714-895-1915 (Fax)
AKI No.: 11303**

Prepared By:

Victor Zankich
Report Writer

Reviewed By:

Shawn Graham
Project Manager

EMISSIONS CHARACTERIZATION
AND TESTING SERVICES

TABLE OF CONTENTS

	<u>Page</u>
1.0 SUMMARY	
1.1 Source Information.....	1-1
1.2 Testing Firm Information.....	1-1
1.3 Test Information.....	1-1
1.4 Test Personnel.....	1-2
2.0 TEST RESULTS AND DATA PRESENTATION	2-1
3.0 INTRODUCTION.....	3-1

APPENDICES**A. RESULTS TABULATION**

1.0 Unit No. 1	
a. Particulate.....	A-1
b. Dioxin/Furans.....	A-5
c. Metals.....	A-9

B. EXAMPLE CALCULATIONS

1.0 Particulate	B-1
2.0 Dioxin/Furans.....	B-3
3.0 Metals	B-5

C. ANALYTICAL DATA

1.0 Unit No. 1	
a. Particulate.....	C-1
b. Dioxin/Furans.....	C-4
c. Metals.....	C-13
d. CCl ₄ and Cl ₄	C-23

Engineering Test Report
General Atomics
San Diego, California

Page: 1 - 1
Revision: 0
Date: October 23, 2002

1.0 SUMMARY

1.1 Source Information

Facility Name and Address: General Atomics
3550 General Atomics Ct
San Diego, California 92121-1122

Source Tested: Scrubber Effluent Stack

Facility Contact: Glenn Hong, General Atomics

Phone Number: 508-870-0109

1.2 Testing Firm Information

Firm Name and Address: AirKinetics, Inc.
5932 Bolsa Avenue, Suite 105
Huntington Beach, CA 92649

Firm Contact: Shawn Graham

Phone Number: 714-373-0998, Ext. 27

Subcontractors: Air Toxics Ltd., Fulsom, CA
Alta Analytical Laboratory Inc, El Dorado Hills, CA

1.3 Test Information

Test Requested By: General Atomics

Firm Contact: Glenn Hong

Phone Number: 508-870-0109

Test Objective: To determine compliance with permitted emission limits

Test Methods:	EPA 5	Particulate
	EPA 23	Dioxin/Furans
	EPA 29	Metals
	EPA TO15	CCl ₄ and Cl ₄

Test Dates: September 19, 2002

1.4 Test Personnel

Test Coordinators: Glenn Hong, General Atomics,

AirKinetics Test Personnel: Shawn Graham, Project Manager
Jose Orellana, Sampling Team Leader

Engineering Test Report
General Atomics
San Diego, California

Page: 2 - 1
Revision: 0
Date: October 23, 2002

2.0 TEST RESULTS AND DATA PRESENTATION

The results of the testing are summarized in Tables 2-1. Results tabulations are presented in Appendix A. Example calculations are given in Appendix B. Analytical data are provided in Appendix C.

TABLE 2-1
SCRUBBER EFFLUENT TEST RESULTS

Sampling Method	Run Range	Parameter	Units	Result
EPA 5	1250-1748	Particulate	mg/DSCM	0.105
EPA 23	1147-1747	Total PCDDs	ng/DSCM	0.00489
		Total PCDFs	ng/DSCM	0.0192
		Total PCDDs and PCDFs	ng/DSCM	0.0240
EPA 29	1250-1748	Antimony	ug/DSCM	0.0131
		Arsenic	ug/DSCM	ND (<0.565)
		Beryllium	ug/DSCM	ND (<0.283)
		Cadmium	ug/DSCM	0.0216
		Chromium	ug/DSCM	ND (<0.565)
		Cerium	ug/DSCM	ND (<1.40)
		Lead	ug/DSCM	0.148
		Mercury	ug/DSCM	0.158
EPA TO15	1250-1350	Carbon Tetrachloride	ppbvd	60
		C ₂ Cl ₄ Tetrachloride	ppbvd	68

ND – Not Detected

Engineering Test Report
General Atomics
San Diego, California

Page: 3 - 1
Revision: 0
Date: October 23, 2002

3.0 INTRODUCTION

On September 19, 2002 AirKinetics, Inc. conducted source emissions testing for General Atomics at General Atomics in San Diego, California. The testing was conducted on Scrubber Effluent. The objective of the test program was to determine compliance with permitted emission limits. The methods used during this test program were EPA Method 5 for particulate, EPA Method 23 for dioxin/furans, EPA Method 29 for Metals and EPA Method TO15 for CCl_4 and Cl_4 .

All sampling was conducted at a single point and constant rate. The sampling equipment for particulate, metals and dioxin/furans was leak checked before and after each test run with no leaks found. The evacuated canister used to determine CCl_4 and Cl_4 concentration presented in this report has been corrected for the determined leak.

APPENDIX A
RESULTS TABULATION
a. PARTICULATE

ISOKINETIC SAMPLING TRAIN RESULTS - METHOD: **M5**

Client Name	GENERAL ATOMICS		Operator		JO&SCG
Plant Name	GENERAL ATOMICS		Project #		11303
Sampling Location	SCRUBBER EFFLUENT		Standard Temperature, °F		68
Run Number		M5-1			
Run Date		09/19/02			
Run Start Time	hh:mm	1250			
Run Stop Time	hh:mm	1748			
Meter Calibration Factor	Y	0.9940			
Sample Volume	ft ³	211.582			
Total Sampling Time	min	298			
Average Meter Temperature	°F	91.4			
Barometric Pressure	in Hg	29.9			
Average Delta H	in H ₂ O	1.50			
Absolute Meter Pressure	in Hg	30.0			
Total Water Volume Collected	mL	0.0			
Volume of Water vapor @ STP	SCF	0.000			
Volume Metered @ STP	DSCF	201.933			
Calculated Stack Moisture	% H ₂ O	0.0			

A 2

ISOKINETIC SAMPLING TRAIN RESULTS - METHOD: M5

Total Particulate	mg	0.6			
Concentration, grams/DSCF	gms/DSCF	2.97E-06			
Concentration, Gr/DSCF	gr/DSCF	4.59E-05			
Concentration mg/DSCM	mg/DSCM	1.05E-01			

A 3

MOISTURE ANALYTICAL RESULTS - METHOD

EPA 5

Plant Name General AtomicsJob No. 11303City/State San Diego, CASampling Location Scrubber Effluent

Run Number

M5-1

Sampling Date

9/19/02

Analysis Date

9/20/02

Analyst

SG

Reagent DI H2O

Final Weight, g

157.5

Tared Weight, g

200.0

Water Catch, g

42.5

Reagent

Final Weight, g

Tared Weight, g

Water Catch, g

Reagent 4% KMnO₄/10% H₂SO₄

Final Weight, g

ml used, g*

Empty Tared Weight, g

Water Catch, g

*Weight of Reagent calculated by multiplying ml of KMnO₄ by density of 1.1 g/ml

CONDENSED WATER, g

42.5

Silica Gel

Final Weight, g

233.8

Tared Weight, g

200.0

ADSORBED WATER, g

33.8

TOTAL WATER COLLECTED, g

83.7

Balance No. Lab-1☐ Triple Beam☒ Electronic

Balance located in stable, draft-free area?

☒ Yes☐ No

Comments

Reviewer

AirKinetics, Inc.

APPENDIX A
RESULTS TABULATION
b. DIOXIN/FURANS

ISOKINETIC SAMPLING TRAIN RESULTS - METHOD: **EPA 23**

Client Name	GENERAL ATOMICS	Operator		JO&SCG
Plant Name	GENERAL ATOMICS	Project #		11303
Sampling Location	SCRUBBER EFFLUENT	Standard Temperature, °F		58.5

Run Number		M23-1			
Run Date		09/19/02			
Run Start Time	hh:mm	1147			
Run Stop Time	hh:mm	1747			
Meter Calibration Factor	Y	1.0013			
Sample Volume	ft ³	259.652			
Total Sampling Time	min	360			
Average Meter Temperature	°F	80.5			
Barometric Pressure	in Hg	29.9			
Average Delta H	in H ₂ O	1.10			
Absolute Meter Pressure	in Hg	30.0			
Total Water Volume Collected	mL	0.0			
Volume of Water vapor @ STP	SCF	0.000			
Volume Metered @ STP	DSCF	254.402			
Calculated Stack Moisture	% H ₂ O	0.0			

A 6

Plant Name:	GENERAL ATOMICS	
Sampling Location:	SCRUBBER EFFLUENT	
Run Number:	M23-1	
Run Date:	09/19/02	
Parameter	Catch Weight	Concentrations
	(pg)	(ng/DSCM)
PCDDs		
2,3,7,8-TCDD	0	0.00E+00
Other TCDD	35.2	4.89E-03
Total TCDD	0	0.00E+00
1,2,3,7,8-PeCDD	0	0.00E+00
Other PeCDD	0	0.00E+00
Total PeCDD	0	0.00E+00
1,2,3,4,7,8-HxCDD	0	0.00E+00
1,2,3,6,7,8-HxCDD	0	0.00E+00
1,2,3,7,8,9-HxCDD	0	0.00E+00
Other HxCDD	0	0.00E+00
Total HxCDD	0	0.00E+00
1,2,3,4,6,7,8-HpCDD	0	0.00E+00
Other HpCDD	0	0.00E+00
Total-HpCDD	0	0.00E+00
OCDD	0	0.00E+00
TOTAL PCDDs	35	4.89E-03
PCDFs		
2,3,7,8-TCDF	0	0.00E+00
Other TCDF	138	1.92E-02
1,2,3,7,8-PeCDF	0	0.00E+00
2,3,4,7,8-PeCDF	0	0.00E+00
Other PeCDF	0	0.00E+00
1,2,3,4,7,8-HxCDF	0	0.00E+00
1,2,3,6,7,8-HxCDF	0	0.00E+00
2,3,4,6,7,8-HxCDF	0	0.00E+00
1,2,3,7,8,9-HxCDF	0	0.00E+00
Other HxCDF	0	0.00E+00
1,2,3,4,6,7,8-HpCDF	0	0.00E+00
1,2,3,4,7,8,9-HpCDF	0	0.00E+00
Other HpCDF	0	0.00E+00
OCDF	0	0.00E+00
TOTAL PCDFs	138	1.92E-02
TOTAL PCDDs and PCDFs	173	2.40E-02

MOISTURE ANALYTICAL RESULTS - METHOD

EPA 23

Plant Name General AtomicsJob No. 11303City/State San Diego, CASampling Location Scrubber Effluent

Run Number

M23-1

Sampling Date

9/19/02

Analysis Date

9/20/02

Analyst

SG

Reagent HPLC GRADE WATER

Final Weight, g

157.0

Tared Weight, g

200.0

Water Catch, g

43.0

Reagent XAD

Final Weight, g

304.5

Tared Weight, g

304.4

Water Catch, g

0.1

Reagent

Final Weight, g

ml used, g*

Empty Tared Weight, g

Water Catch, g

*Weight of Reagent calculated by multiplying ml of KMnO_4 by density of 1.1 g/ml

CONDENSED WATER, g

42.9

Silica Gel

Final Weight, g

242.3

Tared Weight, g

200.0

ADSORBED WATER, g

42.3

TOTAL WATER COLLECTED, g

90.6

Balance No. Lab-1☐ Triple Beam☒ Electronic

Balance located in stable, draft-free area?

☒ Yes☐ No

Comments

Reviewer

AirKinetics, Inc.

APPENDIX A
RESULTS TABULATION
c. METALS

ISOKINETIC SAMPLING TRAIN RESULTS - METHOD: **EPA 29**

Client Name	GENERAL ATOMICS	Operator		SCG
Plant Name	GENERAL ATOMICS	Project #		11303
Sampling Location	SCRUBBER EFFLUENT	Standard Temperature, °F		68
Run Number		M29-1		
Run Date		09/19/02		
Run Start Time	hh:mm	1250		
Run Stop Time	hh:mm	1748		
Meter Calibration Factor	Y	1.0328		
Sample Volume	ft ³	199.752		
Total Sampling Time	min	298		
Average Meter Temperature	°F	81.1		
Barometric Pressure	in Hg	29.9		
Average Delta H	in H ₂ O	1.50		
Absolute Meter Pressure	in Hg	30.0		
Total Water Volume Collected	mL	0.0		
Volume of Water vapor @ STP	SCF	0.000		
Volume Metered @ STP	DSCF	201.855		
Calculated Stack Moisture	% H ₂ O	0.0		

A 10

ISOKINETIC SAMPLING TRAIN RESULTS - METHOD: EPA 29

Concentration and Emission Rate Data Summary					
Run Number		M29-1			
Antimony	ug	0.075			
Concentration ug/DSCM	ug/DSCM	1.31E-02			
Arsenic	ug	< 3.230			
Concentration ug/DSCM	ug/DSCM	< 5.65E-01			
Beryllium	ug	< 1.615			
Concentration ug/DSCM	ug/DSCM	< 2.83E-01			
Cadmium	ug	0.124			
Concentration ug/DSCM	ug/DSCM	2.16E-02			
Chromium	ug	< 3.230			
Concentration ug/DSCM	ug/DSCM	< 5.65E-01			
Cerium	ug	< 7.980			
Concentration ug/DSCM	ug/DSCM	< 1.40E+00			
Lead	ug	0.846			
Concentration ug/DSCM	ug/DSCM	1.48E-01			
Mercury	ug	< 0.904			
Concentration ug/DSCM	ug/DSCM	< 1.58E-01			

A 11

Plant Name:	GENERAL ATOMICS	
Sampling Location:	SCRUBBER EFFLUENT	
Run Number:	M23-1	
Run Date:	09/19/02	
Parameter	Catch Weight	Concentrations
	(pg)	(ng/DSCM)
PCDDs		
2,3,7,8-TCDD	4.48	6.22E-04
Other TCDD	30.72	4.26E-03
1,2,3,7,8-PeCDD	3.44	4.77E-04
Other PeCDD	0	0.00E+00
1,2,3,4,7,8-HxCDD	5.45	7.56E-04
1,2,3,6,7,8-HxCDD	5.17	7.18E-04
1,2,3,7,8,9-HxCDD	5.21	7.23E-04
Other HxCDD	-10.55	-1.46E-03
1,2,3,4,6,7,8-HpCDD	5.81	8.06E-04
Other HpCDD	0	0.00E+00
OCDD	12.8	1.78E-03
TOTAL PCDDs	63	8.68E-03
PCDFs		
2,3,7,8-TCDF	3.35	4.65E-04
Other TCDF	134.65	1.87E-02
1,2,3,7,8-PeCDF	3.56	4.94E-04
2,3,4,7,8-PeCDF	3.32	4.61E-04
Other PeCDF	-3.44	-4.77E-04
1,2,3,4,7,8-HxCDF	2.57	3.57E-04
1,2,3,6,7,8-HxCDF	2.47	3.43E-04
2,3,4,6,7,8-HxCDF	2.89	4.01E-04
1,2,3,7,8,9-HxCDF	3.31	4.59E-04
Other HxCDF	-8.47	-1.18E-03
1,2,3,4,6,7,8-HpCDF	2.61	3.62E-04
1,2,3,4,7,8,9-HpCDF	3.22	4.47E-04
Other HpCDF	-2.95	-4.09E-04
OCDF	9.28	1.29E-03
TOTAL PCDFs	156	2.17E-02
TOTAL PCDDs and PCDFs	219	3.04E-02

A 12

MOISTURE ANALYTICAL RESULTS - METHOD

EPA 29

Plant Name General Atomics Job No. 11303
 City/State San Diego, CA Sampling Location Scrubber Effluent

Run Number	M29-1		
Sampling Date	9/19/02		
Analysis Date	9/20/02		
Analyst	SG		

Reagent 5% HNO₃/10% H₂O₂

Final Weight, g	240.0		
Tared Weight, g	271.8		
Water Catch, g	31.8		

Reagent EMPTY IMPINGER

Final Weight, g	30.9		
Tared Weight, g	30.9		
Water Catch, g			

Reagent 4% KMnO₄/10% H₂SO₄

Final Weight, g	595.6		
200 ml used, g*	220.0	220.0	220.0
Empty Tared Weight, g	380.6		
Water Catch, g	5.0	220.0	220.0

*Weight of Reagent calculated by multiplying ml of KMnO₄ by density of 1.1 g/ml

CONDENSED WATER, g	36.8	220.0	220.0
--------------------	------	-------	-------

Silica Gel

Final Weight, g	231.6		
Tared Weight, g	200.0		
ADSORBED WATER, g	31.6		

TOTAL WATER COLLECTED, g	5.2	220.0	220.0
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Balance No. Lab-1☐ Triple Beam☒ Electronic

Balance located in stable, draft-free area?

☒ Yes☐ No

Comments

Reviewer

AirKinetics, Inc.

APPENDIX B
EXAMPLE CALCULATIONS
1.0 PARTICULATE

EXAMPLE CALCULATIONS, RUN M5-1**ABSOLUTE PRESSURE, INCHES OF MERCURY**

$$\begin{aligned} P_s &= P_{bar} + P_g/13.6 \\ &= 29.90 + 0.00/13.6 \\ &= 29.90 \end{aligned}$$

VOLUME OF WATER VAPOR, STANDARD CUBIC FEET

$$\begin{aligned} V_{wstd} &= 0.002667 * [(T_{std} + 460) / P_{std}] * V_{lc} \\ &= 0.002667 * [(68 + 460) / 29.92] * 0.0 \\ &= 0.000 \end{aligned}$$

SAMPLED VOLUME OF SOURCE GAS, DRY STANDARD CUBIC FEET

$$\begin{aligned} V_{mstd} &= [(T_{std} + 460)/P_{std}] * Y * V_m * (P_{bar} + \Delta H/13.6) / (460 + t_m) \\ &= [(68 + 460)/29.92] * 0.9940 * 211.582 * (29.90 + 1.500/13.6) / (460 + 91) \\ &= 201.933 \end{aligned}$$

MOISTURE CONTENT, PERCENT BY VOLUME

$$\begin{aligned} \%H_2O &= V_{wstd} / (V_{wstd} + V_{mstd}) \\ &= 0.000 / (0.000 + 201.933) \\ &= 0.00 \end{aligned}$$

FILTERABLE PARTICULATE CONCENTRATION, GRAINS PER DRY STANDARD CUBIC FOOT

$$\begin{aligned} gr/DSCF &= (Catch/Conversion) * 7,000 / 453.592 / V_{mstd} \\ &= (0.60/1000) * 7,000 / 453.592 / 201.933 \\ &= 0.00005 \end{aligned}$$

APPENDIX B
EXAMPLE CALCULATIONS
2.0 DIOXIN/FURANS

EXAMPLE CALCULATIONS, RUN M23-1**ABSOLUTE PRESSURE, INCHES OF MERCURY**

$$\begin{aligned} P_s &= P_{\text{bar}} + P_g/13.6 \\ &= 29.90 + 0.00/13.6 \\ &= 29.9 \end{aligned}$$

VOLUME OF WATER VAPOR, STANDARD CUBIC FEET

$$\begin{aligned} V_{\text{wstd}} &= 0.002667 * [(T_{\text{std}} + 460) / P_{\text{std}}] * V_{\text{lc}} \\ &= 0.002667 * [(68 + 460) / 29.92 * 0.0 \\ &= 0.000 \end{aligned}$$

SAMPLED VOLUME OF SOURCE GAS, DRY STANDARD CUBIC FEET

$$\begin{aligned} V_{\text{mstd}} &= [(T_{\text{std}} + 460)/P_{\text{std}}] * Y * V_m * (P_{\text{bar}} + \Delta H/13.6) / (460 + t_m) \\ &= [(68 + 460)/ 29.92] * 1.0013 * 259.652 * (29.90 + 1.100/13.6) / (460 + 80) \\ &= 254.402 \end{aligned}$$

MOISTURE CONTENT, PERCENT BY VOLUME

$$\begin{aligned} \%H_2O &= V_{\text{wstd}} / (V_{\text{wstd}} + V_{\text{mstd}}) \\ &= 0.000 / (0.000 + 254.402) \\ &= 0.0 \end{aligned}$$

TOTAL PCDDs CONCENTRATION, NANOGRAMS PER DRY STANDARD CUBIC METER

$$\begin{aligned} \text{ng/DSCM} &= (\text{Catch/Conversion}) * 1,000,000,000 / (V_{\text{mstd}} * 0.02832) \\ &= (63/1000000000000) * 1,000,000,000 / (254.402 * 0.02832) \\ &= 0.009 \end{aligned}$$

APPENDIX B

EXAMPLE CALCULATIONS

3.0 METALS

EXAMPLE CALCULATIONS, RUN M29-1**VOLUME OF WATER VAPOR, STANDARD CUBIC FEET**

$$\begin{aligned} V_{wstd} &= 0.002667 * [(T_{std} + 460) / P_{std}] * V_{lc} \\ &= 0.002667 * [(68 + 460) / 29.92] * 0.0 \\ &= 0.000 \end{aligned}$$

SAMPLED VOLUME OF SOURCE GAS, DRY STANDARD CUBIC FEET

$$\begin{aligned} V_{mstd} &= [(T_{std} + 460) / P_{std}] * Y * V_m * (P_{bar} + \Delta H / 13.6) / (460 + t_m) \\ &= [(68 + 460) / 29.92] * 1.0328 * 199.752 * (29.90 + 1.500 / 13.6) / (460 + 81) \\ &= 201.855 \end{aligned}$$

MOISTURE CONTENT, PERCENT BY VOLUME

$$\begin{aligned} \%H_2O &= V_{wstd} / (V_{wstd} + V_{mstd}) \\ &= 0.000 / (0.000 + 201.855) \\ &= 0.00 \end{aligned}$$

CADMIUM CONCENTRATION, MICROGRAMS PER DRY STANDARD CUBIC METER

$$\begin{aligned} \mu g/DSCM &= (Catch/Conversion) * 1,000,000 / (V_{mstd} * 0.02832) \\ &= (0.12 / 1,000,000) * 1,000,000 / (201.855 * 0.02832) \\ &= 0.022 \end{aligned}$$

APPENDIX C
ANALYTICAL DATA
a. PARTICULATE

PARTICULATE SAMPLING LABORATORY RESULTS (Version 11.16.99)

Plant Name: General Atomics

AKI Ref#: 11303

Sampling Location Scrubber Effluent

Filename: Newpart1.xls

Date Received: September 20, 2002

File Pathway: H:\JOBS\11JOBS\300JOBS\11303\NEWPART1.XLS

Run Number

M5-1

Sample ID/Container #

init.

date

F / 3964

date

F /

date

F /

GM 09/27/02 @ 3.5545

GM

09/26/02

3.5549

0.0000

0.0000

Baggie Tare Wt., g.

3.0751

0.0000

0.0000

Filter Tare Wt., g.

B319

0.4793

0.0000

0.0000

FILTER SAMPLE WT., g.

0.0001

0.0000

0.0000

Sample ID/Container #

init.

date

R / 3967

date

R /

date

R /

GM 09/27/02 @ 3.5333

GM

09/26/02

3.5333

0.0000

0.0000

Tare Wt., g.

(75 ml)

3.5327

(ml)

0.0000

(ml)

0.0000

RINSE SAMPLE WT., g.

0.0006

0.0000

0.0000

Filter Catch, mg.

0.1

0.0

0.0

Rinse Catch, mg.

0.6

0.0

0.0

Rinse Blank Residue, mg.

0.1

0.0

0.0

Net Rinse Catch, mg.

0.4

0.0

0.0

FILTERABLE PARTICULATE, mg.

0.6

0.0

0.0

		Visual Analysis of Filters:				
Blank Beaker #	B / 3962	Run Number:	Color:	Texture:	F.Matter:	R.Comparison:
Final wt., mg.	3.5272	M5-1	Clean	None	None	N/A
Tare wt., mg.	3.5270					
Residue, mg.	0.2					
Volume, ml	100					
Density, mg/ml	785.0					
Conc., mg/mg	2.5E-06 <--					
Upper Limit, mg/	1.0E-05					
		Visual Analysis of Rinses:				
Balance Spanned?	September 26, 2002	Run Number:	Color:	Texture:	F.Matter:	R.Comparison:
Specific Comments about Samples:		M5-1	Black	Carbon Like	None	N/A

REAGENT BLANK LABORATORY RESULTS (Version 11.16.99)

Plant Name: General Atomics

AKI Ref#: 11303

Filename: Newpart1.xls

Sampling Location N/A

Date Received: September 20, 2002

File Pathway: H:\JOBS\11JOBS\300JOBS\11303\NEWPART1.XLS

Run Number

Acetone

Sample ID/Container #

init.

date

B / 3962

GM

09/27/02

3.5273

GM

09/26/02

@ 3.5272

Tare Wt., g.

(100 ml)

3.5270

SAMPLE WT., g.

0.0002

Date of full balance span

September 26, 2002

Notes and comments:

APPENDIX C

ANALYTICAL DATA

b. DIOXIN/FURANS

Method Blank

EPA METHOD 23

Matrix: MMS	QC Batch No.: 3379	Lab Sample: 0-MB001							
Sample Size: Sample	Date Extracted: 2-Oct-02	Date Analyzed DB-5: 7-Oct-02							
		Date Analyzed DB-225: NA							
Analyte	Conc. (pg/sample)	DL ^a	EMPC ^b	MDL ^c	Qualifiers	Labeled Standard	%R	LCL-UCL ^d	Qualifiers
2,3,7,8-TCDD	ND	1.62		3.38		IS 13C-2,3,7,8-TCDD	87.3	40 - 130	
1,2,3,7,8-PeCDD	ND	2.50		24.8		13C-1,2,3,7,8-PeCDD	74.4	40 - 130	
1,2,3,4,7,8-HxCDD	ND	1.66		24.2		13C-1,2,3,6,7,8-HxCDD	81.7	40 - 130	
1,2,3,6,7,8-HxCDD	ND	3.47		25.9		13C-1,2,3,4,6,7,8-HpCDD	72.5	25 - 130	
1,2,3,7,8,9-HxCDD	ND	3.50		21.7		13C-OCDD	49.6	25 - 130	
1,2,3,4,6,7,8-HpCDD	ND	3.16		11.3		13C-2,3,7,8-TCDF	83.4	40 - 130	
OCDD	ND	8.76		39.4		13C-1,2,3,7,8-PeCDF	73.3	40 - 130	
2,3,7,8-TCDF	ND	1.85		3.28		13C-1,2,3,6,7,8-HxCDF	94.8	40 - 130	
1,2,3,7,8-PeCDF	ND	3.37		11.0		13C-1,2,3,4,6,7,8-HpCDF	87.3	25 - 130	
2,3,4,7,8-PeCDF	ND	3.15		5.85		13C-OCDF	63.7	25 - 130	
1,2,3,4,7,8-HxCDF	ND	1.43		15.5		PS 37C-2,3,7,8-TCDD	98.4	70 - 130	
1,2,3,6,7,8-HxCDF	ND	1.09		10.5		13C-2,3,4,7,8-PeCDF	100	70 - 130	
2,3,4,6,7,8-HxCDF	ND	1.27		16.1		13C-1,2,3,4,7,8-HxCDF	96.7	70 - 130	
1,2,3,7,8,9-HxCDF	ND	1.46		28.8		13C-1,2,3,4,7,8-HxCDF	98.0	70 - 130	
1,2,3,4,6,7,8-HpCDF	ND	1.74		8.91		13C-1,2,3,4,7,8,9-HpCDF	95.2	70 - 130	
1,2,3,4,7,8,9-HpCDF	ND	2.15		19.9		AS 13C-1,2,3,7,8,9-HxCDF	97.6	40 - 130	
OCDF	ND	7.31		39.7					
Totals						Toxic Equivalent Quotient (TEQ) Data ^e			
Total TCDD	ND	1.62				TEQ (Min-Max): 0 - 6.72			
Total PeCDD	ND	2.50							
Total HxCDD	ND	3.54				a. Sample specific estimated detection limit.			
Total HpCDD	ND	2.16				b. Estimated maximum possible concentration.			
Total TCDF	ND	1.85				c. Method detection limit.			
Total PeCDF	ND	3.25				d. Lower control limit - upper control limit			
Total HxCDF	ND	1.22				e. TEQ based on International Toxic Equivalent Factors (ITEF-1989).			
Total HpCDF	ND	1.92							

Analyst: JMH

Approved By: Martha M. Maier

08-Oct-2002 13:34

OPR Results			EPA METHOD 23			
Matrix:	MM5	QC Batch No.:	3379	Lab Sample:	0-OPR001	
Sample Size:	Sample	Date Extracted:	2-Oct-02	Date Analyzed DB-5:	7-Oct-02	
				Date Analyzed DB-225:	NA	
Analyte	Spike Conc.	Conc. (ng/mL)	OPR Limits	Labeled Standard	%R	LCL-UCL
2,3,7,8-TCDD	2.50	2.58	1.75 - 3.25	IS 13C-2,3,7,8-TCDD	83.5	40 - 130
1,2,3,7,8-PeCDD	12.5	13.7	8.75 - 16.25	13C-1,2,3,7,8-PeCDD	72.7	40 - 130
1,2,3,4,7,8-HxCDD	12.5	14.2	8.75 - 16.25	13C-1,2,3,6,7,8-HxCDD	74.0	40 - 130
1,2,3,6,7,8-HxCDD	12.5	15.0	8.75 - 16.25	13C-1,2,3,4,6,7,8-HpCDD	68.6	25 - 130
1,2,3,7,8,9-HxCDD	12.5	14.9	8.75 - 16.25	13C-OCDD	45.1	25 - 130
1,2,3,4,6,7,8-HpCDD	12.5	14.5	8.75 - 16.25	13C-2,3,7,8-TCDF	80.7	40 - 130
OCDD	25.0	29.8	17.5 - 32.5	13C-1,2,3,7,8-PeCDF	75.4	40 - 130
2,3,7,8-TCDF	2.50	2.69	1.75 - 3.25	13C-1,2,3,6,7,8-HxCDF	86.2	40 - 130
1,2,3,7,8-PeCDF	12.5	14.1	8.75 - 16.25	13C-1,2,3,4,6,7,8-HpCDF	82.5	25 - 130
2,3,4,7,8-PeCDF	12.5	14.3	8.75 - 16.25	13C-OCDF	60.9	25 - 130
1,2,3,4,7,8-HxCDF	12.5	14.4	8.75 - 16.25	AS 13C-1,2,3,7,8,9-HxCDF	95.6	40 - 130
1,2,3,6,7,8-HxCDF	12.5	15.3	8.75 - 16.25			
2,3,4,6,7,8-HxCDF	12.5	14.9	8.75 - 16.25			
1,2,3,7,8,9-HxCDF	12.5	14.5	8.75 - 16.25			
1,2,3,4,6,7,8-HpCDF	12.5	13.7	8.75 - 16.25			
1,2,3,4,7,8,9-HpCDF	12.5	13.0	8.75 - 16.25			
OCDF	25.0	27.8	17.5 - 32.5			

Analyst: JMH

Approved By:

Martha M. Maier 08-Oct-2002 13:34

Sample ID: M23-1		EPA METHOD 23			
Client Data		Sample Data		Laboratory Data	
Name:	AirKinetics, Inc.	Matrix:	MM5	Lab Sample:	22875-001
Project:		Sample Size:	Sample	QC Batch No.:	3379
Date Collected:	19-Sep-02			Date Analyzed DB-5:	7-Oct-02
Time Collected:	NA			Date Received:	25-Sep-02
				Date Extended:	2-Oct-02
				Date Analyzed DB-225:	NA
Analyte	Conc. (pg/sample)	DL ^a	EMPC ^b	MDL ^c	Qualifiers
2,3,7,8-TCDD	ND	4.48		3.38	IS 13C-2,3,7,8-TCDD 87.4 40 - 130
1,2,3,7,8-PeCDD	ND	3.44		24.8	13C-1,2,3,7,8-PeCDD 75.4 40 - 130
1,2,3,4,7,8-HxCDD	ND	5.45		24.2	13C-1,2,3,6,7,8-HxCDD 80.1 40 - 130
1,2,3,6,7,8-HxCDD	ND	5.17		25.9	13C-1,2,3,4,6,7,8-HpCDD 70.5 25 - 130
1,2,3,7,8,9-HxCDD	ND	5.21		21.7	13C-OCDD 50.7 25 - 130
1,2,3,4,6,7,8-HpCDD	ND	5.81		11.3	13C-2,3,7,8-TCDF 80.6 40 - 130
OCDD	ND	12.8		39.4	13C-1,2,3,7,8-PeCDF 79.2 40 - 130
2,3,7,8-TCDF	ND	3.35		3.28	13C-1,2,3,6,7,8-HxCDF 95.5 40 - 130
1,2,3,7,8-PeCDF	ND	3.56		11.0	13C-1,2,3,4,6,7,8-HpCDF 88.5 25 - 130
2,3,4,7,8-PeCDF	ND	3.32		5.85	13C-OCDF 65.0 25 - 130
1,2,3,4,7,8-HxCDF	ND	2.57		15.5	PS 13C-2,3,7,8-TCDD 100 70 - 130
1,2,3,6,7,8-HxCDF	ND	2.47		10.5	13C-2,3,4,7,8-PeCDF 100 70 - 130
2,3,4,6,7,8-HxCDF	ND	2.89		16.1	13C-1,2,3,4,7,8-HxCDD 101 70 - 130
1,2,3,7,8,9-HxCDF	ND	3.31		28.8	13C-1,2,3,4,7,8-HxCDF 101 70 - 130
1,2,3,4,6,7,8-HpCDF	ND	2.61		8.91	13C-1,2,3,4,7,8,9-HpCDF 92.7 70 - 130
1,2,3,4,7,8,9-HpCDF	ND	3.22		19.9	AS 13C-1,2,3,7,8,9-HxCDF 93.4 40 - 130
OCDF	ND	9.28		39.7	
Toxic Equivalent Quotient (TEQ) Data ^e					
TEQ (Min-Max): 0 - 11.7					
Total TCDD	35.2				a. Sample specific estimated detection limit.
Total PeCDD	ND	3.44			b. Estimated maximum possible concentration.
Total HxCDD	ND	5.28			c. Method detection limit.
Total HpCDD	ND	5.81			d. Lower control limit - upper control limit.
Total TCDF	138		152		e. TEQ based on International Toxic Equivalent Factors (TEF-1989).
Total PeCDF	ND	3.44			
Total HxCDF	ND	2.77			
Total HpCDF	ND	2.88			

Analyst: JMH

Approved By:

Martha M. Maier 08-Oct-2002 13:34

Project 22875

C 7

APPENDIX

DATA QUALIFIERS & ABBREVIATIONS

A	The amount detected is below the Lower Calibration Limit of the instrument.
B	This compound was also detected in the method blank.
D	The amount reported is the maximum possible concentration due to possible chlorinated diphenylether interference.
E	The amount detected is above the Upper Calibration Limit of the instrument.
H	The signal-to-noise ratio is greater than 10:1.
I	Chemical Interference
*	See Cover Letter
Conc.	Concentration
DL	Sample-specific estimated detection limit
EMPC	Estimated Maximum Possible Concentration
NA	Not applicable
ND	Not Detected
TEQ	Toxic Equivalency

CURRENT CERTIFICATIONS

NELAP — (Primary AA: California, Certificate No. 02102CA)

Department of the Navy

U.S. Army Corps of Engineers

U.S. EPA Region 5

Commonwealth of Kentucky — (Certificate No. 90063)

Bureau of Reclamation — Mid-Pacific Region — (MP-470, Res-1.10)

Commonwealth of Kentucky — (Certificate No. 90063)

Commonwealth of Virginia — (Certificate No. 00013)

State of Alaska, Department of Environmental Conservation — (Certificate No. OS-00197)

State of Arkansas, Department of Health — (Approval granted through CA certification)

State of Arkansas, Department of Environmental Quality

State of California — (Certificate No. 1640)

State of Connecticut — (Certificate No. PH-0182)

State of Florida — (Certificate No. 87456)

State of Louisiana, Department of Health and Hospitals — (Certificate No. LA000014)

State of Louisiana, Department of Environmental Quality

State of Mississippi — (Approval granted through CA certification)

State of Nevada — (Certificate No. CA413)

State of New Jersey — (Certificate No. CA003)

State of New York, Department of Health — (Certificate No. 11411)

State of North Carolina — (Certification No. 06700)

State of North Dakota, Department of Health — (Certificate No. R-078)

State of Oregon — (Certificate No. CA413)

State of Pennsylvania — (Certificate No. 68-490)

State of South Carolina — (Certificate No. 87002001)

State of Tennessee — (Certificate No. 02996)

State of Texas — (Certificate No. TX247-1000A)

State of Utah — (Certificate No. E-201)

State of Washington — (Certification No. C091)

State of Wisconsin — (Certificate No. 998036160)

State of Wyoming — (USEPA Region 8 Ref: 8TMS-Q)

C 10

22875

0.6°C

AirKinetics, Inc.

EMISSIONS CHARACTERIZATION AND TESTING SERVICES

REQUEST FOR ANALYSIS

PURCHASE ORDER No.: 1844 - GM

JOB NAME: General Atomics

LABORATORY: Alta Analytical

JOB No.: 11303

DATE SAMPLES
WERE TRANSMITTED: 9-25-02

EXPECTED DATE
OF RESULTS: 10-18-02

SAMPLE MATRIX: Acetone, Methylene Chloride and Toluene

TYPE OF ANALYSIS REQUIRED: Please analyze according to EPA Method 23 for Dioxin/Furans.

A full raw data package will be required.

[illegible]

* For Laboratory Comments (temp., labels, etc.)

Samples Relinquished by:

Date/Time: 9/25/02 1155

Transported by:

Date/Time: 9/25/02 1155

Transported to: Alta Analytical

5070 Robert J. Mathews Parkway

Eldorado Hills, CA 95762

Received by:

Date/Time: 9/25/0 11:53

STANDARD OPERATING PROCEDURE

Attachment 10.B.1

SAMPLE LOG-IN CHECKLIST

ALTA Project No.: 22875

1. Date Samples Arrived: <u>09/25/02</u> Initials: <u>[Signature]</u> Location:			
2. Time / Date logged in: <u>09/25/02</u> <u>13:45</u> Initials: <u>[Signature]</u> Location: <u>WR-2</u>			
3. Samples Arrived By: (circle) <u>FedEx</u> <u>UPS</u> <u>World Courier</u> Other: <u>HAND CARRY</u>			
4. Shipping Preservation: (circle) <u>Ice</u> <u>Blue Ice</u> / <u>Dry Ice</u> / <u>None</u> Temp °C <u>0.6°C</u>			
5. Shipping Container(s) Intact? If not, describe condition in comment section.			YES X
6. Shipping Container(s) Custody Seals Present? Intact? If not intact, describe condition in comment section.			NO X
7. Shipping Documentation Present? (circle) <u>Shipping Label</u> <u>Airbill</u> Tracking Number:			NA X
8. Sample Custody Seal(s) Present? No. of Seals _____ or Seal No. _____ Intact? If not intact, describe condition in comment section.			X
9. Sample Container Intact? If no, indicate sample condition in comment section.			X
10. Chain of Custody (COC) or other Sample Documentation Present?			X
11. COC/Documentation Acceptable? If no, complete COC Anomaly Form.			X
12. Shipping Container (circle): <u>ALTA</u> <u>Client</u> Retain or <u>Return</u> or Disposed			
13. Container(s) and/or Bottle(s) Requested?			X
14. Drinking Water Sample? (HRMS Only) If yes, Acceptable Preservation? Y or N Preservation Info From? (circle) <u>COC</u> or <u>Sample Container</u> or <u>None Noted</u>			X

Comments:

C 12

APPENDIX C
ANALYTICAL DATA
c. METALS

AIRKINETICS, INC.

Client Sample ID: M29-1

TOTAL Metals

Lot-Sample #...: G2I250284-001

Date Sampled...: 09/19/02

Date Received...: 09/25/02

Matrix.....: AIR

PARAMETER	RESULT	REPORTING LIMIT	UNITS	METHOD	PREPARATION- ANALYSIS DATE	WORK ORDER #
Prep Batch #...: 2270246						
Hg (KMnO4)	ND	0.10	ug	SW846 7470A	09/26-09/27/02	ESVTH1AA
Hg (FH)	0.012 B	0.030	ug	SW846 7470A	09/26-09/27/02	ESVTH1AL
Hg (HNO3/H2O2)	ND G	0.51	ug	SW846 7470A	09/26-09/27/02	ESVTH1AM
Hg (Empty)	ND G	0.19	ug	SW846 7470A	09/26-09/27/02	ESVTH1AN
Prep Batch #...: 2270294						
Hg (HCl)	0.14	0.10	ug	SW846 7470A	09/27/02	ESVTH1AC
Prep Batch #...: 2273457						
Arsenic	ND	3.4	ug	SW846 6020	09/26-10/14/02	ESVTH1AD
Beryllium	ND	1.7	ug	SW846 6020	09/26-10/14/02	ESVTH1AE
Cadmium	0.13 B	1.7	ug	SW846 6020	09/26-10/14/02	ESVTH1AF
Cerium	ND	8.4	ug	SW846 6020	09/26-10/14/02	ESVTH1AG
Chromium	ND	3.4	ug	SW846 6020	09/26-10/14/02	ESVTH1AH
Lead	0.89 B	1.7	ug	SW846 6020	09/26-10/14/02	ESVTH1AJ
Antimony	0.079 B	3.4	ug	SW846 6020	09/26-10/14/02	ESVTH1AK

NOTE(S):

B Estimated result. Result is less than RL.

G Elevated reporting limit. The reporting limit is elevated due to matrix interference.

AIRKINETICS, INC.

Client Sample ID: M29-RB

TOTAL Metals

Lot-Sample #....: G2I250284-002

Date Sampled....: 09/19/02

Date Received...: 09/25/02

Matrix.....: AIR

PARAMETER	RESULT	REPORTING LIMIT	UNITS	METHOD	PREPARATION- ANALYSIS DATE	WORK ORDER #
Prep Batch #....: 2270246						
Hg (KMnO4)	0.033 B	0.10	ug	SW846 7470A	09/26-09/27/02	E8VTVLAA
Hg (FB)	0.012 B	0.030	ug	SW846 7470A	09/26-09/27/02	E8VTVLAL
Hg (HNO3/H2O2)	ND G	0.60	ug	SW846 7470A	09/26-09/27/02	E8VTVLAM
Hg (Empty)	ND G	0.20	ug	SW846 7470A	09/26-09/27/02	E8VTVLAN
Prep Batch #....: 2270294						
Hg (HCl)	0.079 B	0.10	ug	SW846 7470A	09/27/02	E8VTVLAC
Prep Batch #....: 2273457						
Arsenic	ND	3.8	ug	SW846 6020	09/26-10/14/02	E8VTVLAD
Beryllium	ND	1.9	ug	SW846 6020	09/26-10/14/02	E8VTVLAE
Cadmium	0.14 B	1.9	ug	SW846 6020	09/26-10/14/02	E8VTVLAF
Cerium	ND	9.4	ug	SW846 6020	09/26-10/14/02	E8VTVLAG
Chromium	ND	3.8	ug	SW846 6020	09/26-10/14/02	E8VTVLAH
Lead	0.34 B	1.9	ug	SW846 6020	09/26-10/14/02	E8VTVLAJ
Antimony	0.094 B	3.8	ug	SW846 6020	09/26-10/14/02	E8VTVLAK

NOTE(S):

B Estimated result. Result is less than RL.

G Elevated reporting limit. The reporting limit is elevated due to matrix interference.

QC DATA ASSOCIATION SUMMARY

G2I250284

Sample Preparation and Analysis Control Numbers

<u>SAMPLE#</u>	<u>MATRIX</u>	<u>ANALYTICAL METHOD</u>	<u>LEACH BATCH #</u>	<u>PREP BATCH #</u>	<u>MS RUN#</u>
001	AIR	SW846 6020		2273457	2273246
	AIR	SW846 7470A		2270246	2270106
	AIR	SW846 7470A		2270294	
002	AIR	SW846 6020		2273457	2273246
	AIR	SW846 7470A		2270246	2270106
	AIR	SW846 7470A		2270294	

METHOD BLANK REPORT

TOTAL Metals

Client Lot #....: G2I250284

Matrix.....: AIR

PARAMETER	RESULT	REPORTING LIMIT	UNITS	METHOD	PREPARATION- ANALYSIS DATE	WORK ORDER #
MB Lot-Sample #: G2I270000-246 Prep Batch #....: 2270246						
Hg (Empty)	ND	0.20	ug	SW846 7470A	09/26-09/27/02	E81KJ1AE
Hg (FH)	ND	0.20	ug	SW846 7470A	09/26-09/27/02	E81KJ1AC
Hg (HNO3/H2O2)	ND	0.20	ug	SW846 7470A	09/26-09/27/02	E81KJ1AD
Hg (KMnO4)	ND	0.20	ug	SW846 7470A	09/26-09/27/02	E81KJ1AA
MB Lot-Sample #: G2I270000-294 Prep Batch #....: 2270294						
Hg (HCl)	ND	0.20	ug	SW846 7470A	09/27/02	E81VL1AA
MB Lot-Sample #: G2I300000-457 Prep Batch #....: 2273457						
Antimony	ND	0.66	ug	SW846 6020	09/26-10/14/02	E85E41AH
Arsenic	ND	0.66	ug	SW846 6020	09/26-10/14/02	E85E41AA
Beryllium	ND	0.33	ug	SW846 6020	09/26-10/14/02	E85E41AC
Cadmium	ND	0.33	ug	SW846 6020	09/26-10/14/02	E85E41AD
Cerium	ND	1.7	ug	SW846 6020	09/26-10/14/02	E85E41AE
Chromium	ND	0.66	ug	SW846 6020	09/26-10/14/02	E85E41AF
Lead	ND	0.33	ug	SW846 6020	09/26-10/14/02	E85E41AG

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

LABORATORY CONTROL SAMPLE EVALUATION REPORT

TOTAL Metals

Lot-Sample #....: G2I250284

Matrix.....: AIR

PARAMETER	PERCENT RECOVERY	RECOVERY LIMITS	RPD	RPD LIMITS	METHOD	PREPARATION- ANALYSIS DATE	PREP- BATCH #
Hg (Empty)	103	(80 - 120)			SW846 7470A	09/26-09/27/02	2270246
	98	(80 - 120)	5.0	(0-20)	SW846 7470A	09/26-09/27/02	2270246
Hg (FH)	103	(80 - 120)			SW846 7470A	09/26-09/27/02	2270246
	98	(80 - 120)	5.0	(0-20)	SW846 7470A	09/26-09/27/02	2270246
Hg (HNO3/H2O)	103	(80 - 120)			SW846 7470A	09/26-09/27/02	2270246
	98	(80 - 120)	5.0	(0-20)	SW846 7470A	09/26-09/27/02	2270246
Hg (KMnO4)	103	(80 - 120)			SW846 7470A	09/26-09/27/02	2270246
	98	(80 - 120)	5.0	(0-20)	SW846 7470A	09/26-09/27/02	2270246
Hg (HCl)	108	(80 - 120)			SW846 7470A	09/26-09/27/02	2270246
	107	(80 - 120)	0.93	(0-20)	SW846 7470A	09/27/02	2270294
Antimony	99	(75 - 125)			SW846 6020	09/27/02	2270294
	96	(75 - 125)	3.0	(0-20)	SW846 6020	09/26-10/14/02	2273457
Arsenic	96	(75 - 125)			SW846 6020	09/26-10/14/02	2273457
	94	(75 - 125)	1.7	(0-20)	SW846 6020	09/26-10/14/02	2273457
Beryllium	91	(75 - 125)			SW846 6020	09/26-10/14/02	2273457
	87	(75 - 125)	4.5	(0-20)	SW846 6020	09/26-10/14/02	2273457
Cadmium	99	(75 - 125)			SW846 6020	09/26-10/14/02	2273457
	97	(75 - 125)	2.4	(0-20)	SW846 6020	09/26-10/14/02	2273457
Cerium	103	(75 - 125)			SW846 6020	09/26-10/14/02	2273457
	105	(75 - 125)	2.1	(0-20)	SW846 6020	09/26-10/14/02	2273457
Chromium	99	(75 - 125)			SW846 6020	09/26-10/14/02	2273457
	97	(75 - 125)	1.7	(0-20)	SW846 6020	09/26-10/14/02	2273457
Lead	101	(75 - 125)			SW846 6020	09/26-10/14/02	2273457
	98	(75 - 125)	3.2	(0-20)	SW846 6020	09/26-10/14/02	2273457

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

LABORATORY CONTROL SAMPLE DATA REPORT

TOTAL Metals

Lot-Sample #...: G2I250284

Matrix.....: AIR

PARAMETER	SPIKE AMOUNT	MEASURED AMOUNT	UNITS	PERCENT RECVRY	RPD	METHOD	PREPARATION- ANALYSIS DATE	PREP BATCH #
Hg (Empty)	1.00	1.03	ug	103		SW846 7470A	09/26-09/27/02	2270246
	1.00	0.980	ug	98	5.0	SW846 7470A	09/26-09/27/02	2270246
Hg (FH)	1.00	1.03	ug	103		SW846 7470A	09/26-09/27/02	2270246
	1.00	0.980	ug	98	5.0	SW846 7470A	09/26-09/27/02	2270246
Hg (HNO3/H2O)	1.00	1.03	ug	103		SW846 7470A	09/26-09/27/02	2270246
	1.00	0.980	ug	98	5.0	SW846 7470A	09/26-09/27/02	2270246
Hg (KMnO4)	1.00	1.03	ug	103		SW846 7470A	09/26-09/27/02	2270246
	1.00	0.980	ug	98	5.0	SW846 7470A	09/26-09/27/02	2270246
Hg (HCl)	1.00	1.08	ug	108		SW846 7470A	09/27/02	2270294
	1.00	1.07	ug	107	0.93	SW846 7470A	09/27/02	2270294
Antimony	16.5	16.3	ug	99		SW846 6020	09/26-10/14/02	2273457
	16.5	15.8	ug	96	3.0	SW846 6020	09/26-10/14/02	2273457
Arsenic	66.0	63.4	ug	96		SW846 6020	09/26-10/14/02	2273457
	66.0	62.3	ug	94	1.7	SW846 6020	09/26-10/14/02	2273457
Beryllium	66.0	59.9	ug	91		SW846 6020	09/26-10/14/02	2273457
	66.0	57.2	ug	87	4.5	SW846 6020	09/26-10/14/02	2273457
Cadmium	66.0	65.3	ug	99		SW846 6020	09/26-10/14/02	2273457
	66.0	63.8	ug	97	2.4	SW846 6020	09/26-10/14/02	2273457
Cerium	33.0	34.0	ug	103		SW846 6020	09/26-10/14/02	2273457
	33.0	34.8	ug	105	2.1	SW846 6020	09/26-10/14/02	2273457
Chromium	66.0	65.1	ug	99		SW846 6020	09/26-10/14/02	2273457
	66.0	64.0	ug	97	1.7	SW846 6020	09/26-10/14/02	2273457
Lead	66.0	67.0	ug	101		SW846 6020	09/26-10/14/02	2273457
	66.0	64.9	ug	98	3.2	SW846 6020	09/26-10/14/02	2273457

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

MATRIX SPIKE SAMPLE EVALUATION REPORT

TOTAL Metals

Client Lot #...: G2I250284

Matrix.....: AIR

Date Sampled...: 09/19/02

Date Received...: 09/25/02

<u>PARAMETER</u>	<u>PERCENT RECOVERY</u>	<u>RECOVERY LIMITS</u>	<u>RPD LIMITS</u>	<u>METHOD</u>	<u>PREPARATION- ANALYSIS DATE</u>	<u>WORK ORDER #</u>
MS Lot-Sample #: G2I250284-001 Prep Batch #...: 2270246						
Hg (HNO3/H2O 99 G		(75 - 125)		SW846 7470A	09/26-09/27/02	ESVTH1AP
95 G		(75 - 125) 4.4	(0-20)	SW846 7470A	09/26-09/27/02	ESVTH1AQ

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

G Elevated reporting limit. The reporting limit is elevated due to matrix interference.

MATRIX SPIKE SAMPLE DATA REPORT

TOTAL Metals

Client Lot #....: G2I250284

Matrix.....: AIR

Date Sampled....: 09/19/02

Date Received...: 09/25/02

PARAMETER	SAMPLE AMOUNT	SPIKE AMT	MEASRD AMOUNT	UNITS	PERCNT RECVRY	RPD	METHOD	PREPARATION- ANALYSIS DATE	WORK ORDER #
MS Lot-Sample #: G2I250284-001 Prep Batch #....: 2270246									
Hg (HNO3/H2O2)									
	ND	2.55	2.61 G	ug	99		SW846 7470A	09/26-09/27/02	E8VTH1AP
	ND	2.55	2.50 G	ug	95	4.4	SW846 7470A	09/26-09/27/02	E8VTH1AQ

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

G Elevated reporting limit. The reporting limit is elevated due to matrix interference.

SAMPLE DUPLICATE EVALUATION REPORT

Metals

Client Lot #....: G2I250284

Work Order #....: E8VTH-SMP

Matrix.....: AIR

E8VTH-DUP

Date Sampled....: 09/19/02

Date Received...: 09/25/02

PARAM RESULT	DUPLICATE RESULT	UNITS	RPD	RPD LIMIT	METHOD	PREPARATION- ANALYSIS DATE	PREP BATCH #
Arsenic							
ND	ND	ug	0	(0-20)	SD Lot-Sample #: G2I250284-001		
Beryllium							
ND	ND	ug	0	(0-20)	SW846 6020	09/26-10/14/02	2273457
Cadmium							
0.13 B	0.14 B	ug	3.5	(0-20)	SD Lot-Sample #: G2I250284-001		
Cerium							
ND	ND	ug	0	(0-20)	SW846 6020	09/26-10/14/02	2273457
Chromium							
ND	ND	ug	0	(0-20)	SD Lot-Sample #: G2I250284-001		
Lead							
0.89 B	0.91 B	ug	2.0	(0-20)	SW846 6020	09/26-10/14/02	2273457
Antimony							
0.079 B	0.086 B	ug	9.4	(0-20)	SD Lot-Sample #: G2I250284-001		
					SW846 6020	09/26-10/14/02	2273457

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

B Estimated result. Result is less than RL.

APPENDIX C
ANALYTICAL DATA

d. CCL₄ AND CL₄

EPA TO15 Results Correction

	Reported Concentration (ppmvd)	Corrected Concentration (ppmvd)
Carbon Tetrachloride	30	60
Tetrachloride	34	68

	(inches of Hg)
Barometric Pressure	29.9
Summa Canister Vacuum	
Initial	15
Final	0

Corrected Concentration = Reported Concentration * Barometric Pressure / (Initial Vacuum - Final Vacuum)

AIR TOXICS LTD.

AN ENVIRONMENTAL ANALYTICAL LABORATORY

WORK ORDER #: 0209522

Work Order Summary

CLIENT: Mr. Gary Mata
AirKinetics, Inc.
5932 Bolsa Avenue STE 105
Huntington Beach, CA 92649

BILL TO: Mr. Gary Mata
AirKinetics, Inc.
5932 Bolsa Avenue STE 105
Huntington Beach, CA 92649

PHONE: 714-373-0998

P.O. #

FAX: 714-895-1915

PROJECT # 11303 General Atomics

DATE RECEIVED: 9/25/02

CONTACT: Karen Burden

DATE COMPLETED: 10/8/02

FRACTION #**NAME****TEST****RECEIPT****VAC./PRES.**

01A

TO-15

Modified TO-15

0.0 "Hg

02A

Lab Blank

Modified TO-15

NA

03A

LCS

Modified TO-15

NA

CERTIFIED BY:

Laboratory Director

DATE: 10/09/02

Certification numbers: CA NELAP - 02110CA, NY NELAP - 11291, UT NELAP - 9166389892, LA NELAP/LELAP- AI 30763

Name of Accrediting Agency: NELAP/Florida Department of Health, Scope of Application: Clean Air Act,

Accreditation number: E87680, Effective date: 07/01/02, Expiration date: 06/30/03

Air Toxics Ltd. certifies that the test results contained in this report meet all requirements of the NELAC standards

This report shall not be reproduced, except in full, without the written approval of Air Toxics Ltd.

180 BLUE RAVINE ROAD, SUITE B FOLSOM, CA - 95630

(916) 985-1000, (800) 985-5955, FAX (916) 985-1020

LABORATORY NARRATIVE**Modified TO-15****AirKinetics, Inc.****Workorder# 0209522**

One 6 Liter Summa Canister sample was received on September 25, 2002. The laboratory performed analysis via modified EPA Method TO-15 using GC/MS in the full scan mode. The method involves concentrating to 0.5 liters of air. The concentrated aliquot is then flash vaporized and swept through a water management system to remove water vapor. Following dehumidification, the sample passes directly into the GC/MS for analysis. See the data sheets for the reporting limits for each compound.

During the five point calibration, two low-level standards are used. The low-level standard for TO-15 compounds is spiked at 0.5 ppbv and represents the reporting limit for these compounds. The low-level standard for the non-TO-15 compounds is spiked at 2.0 ppbv and represents the reporting limit for these compounds. The TO-15 compounds are present in both standards but are excluded from reporting in the 2.0 ppbv standard since a lower level is already included in the curve.

Method modifications taken to run these samples include:

<i>Requirement</i>	<i>TO-15</i>	<i>ATL Modifications</i>
Sampling/concentrator system	Nafion Drier	Multisorbent concentrator
Blank	Humid Air Blank	Humid Nitrogen Blank for standard analysis. Dry Nitrogen Blank for low level analysis.
BFB acceptance criteria	CLP protocol	SW-846 protocol
Concentration of IS spike	10 ppbv	25 ppbv for TO-14; 10 ppbv for low level
Dilutions for initial calibration	Dynamic dilutions or static using canisters	Syringe dilutions
ICAL %RSD acceptance criteria	30% or less, 40% or less for up to two compounds	30% or less for standard compounds, 40% or less for non-standard and polar compounds or = 30% pooled RSD of all compounds
IS recoveries	Within 40% of mean over ICAL for blanks, and w/in 40% of daily CCV for samples.	Within 40% of CCV recoveries for blank and samples.
IS RTs	± 0.33 min from most recent calibration (either ICAL or daily)	± 0.33 min of RT in daily CCV
Daily CCV	70 - 130%	Standard compounds: 70 - 130% for at least 90%; Non-standard and polar compounds: 60 - 140% for at least 80%
RF for quantitation	From daily CCV	From ICAL
MSD scan range	35 - 300 amu	35 - 350 amu

Receiving Notes

There were no receiving discrepancies.

Analytical Notes

There were no analytical discrepancies.

Definition of Data Qualifying Flags

Seven qualifiers may have been used on the data analysis sheets and indicates as follows:

B - Compound present in laboratory blank greater than reporting limit (background subtraction not performed).

J - Estimated value.

E - Exceeds instrument calibration range.

S - Saturated Peak.

Q - Exceeds quality control limits.

U - Compound analyzed for but not detected above the reporting limit.

N - The identification is based on presumptive evidence.

File extensions may have been used on the data analysis sheets and indicates as follows:

a-File was requantified

b-File was quantified by a second column and detector

r1-File was requantified for the purpose of reissue

AIR TOXICS LTD.

SAMPLE NAME: TO-15

ID#: 0209522-01A

MODIFIED EPA METHOD TO-15 GC/MS FULL SCAN

File Name:	0209522-01A	Date:	12/19/02
File Size:	2.68	Date:	12/19/02

Compound	Rot. Limit (ppbv)	Rpt. Limit (uG/m3)	Amount (ppbv)	Amount (uG/m3)
Carbon Tetrachloride	1.3	8.8	30	190
Tetrachloroethene	1.3	9.2	34	240

Container Type: 6 Liter Summa Canister

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	93	70-130
Toluene-d8	89	70-130
4-Bromofluorobenzene	82	70-130

AIR TOXICS LTD.

SAMPLE NAME: Lab Blank

ID#: 0209522-02A

MODIFIED EPA METHOD TO-15 GC/MS FULL SCAN

Sample ID: 0209522-02A	Date of Collection: 12/19/2002
Lab: 000	Date of Analysis: 12/19/2002

Compound	Rpt. Limit (ppbv)	Rpt. Limit (uG/m3)	Amount (ppbv)	Amount (uG/m3)
Carbon Tetrachloride	0.50	3.2	Not Detected	Not Detected
Tetrachloroethene	0.50	3.4	Not Detected	Not Detected

Container Type: NA - Not Applicable

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	95	70-130
Toluene-d8	91	70-130
4-Bromofluorobenzene	85	70-130

AIR TOXICS LTD.

SAMPLE NAME: LCS

ID#: 0209522-03A

MODIFIED EPA METHOD TO-15 GC/MS FULL SCAN

File Name	00203211	Date of Collection	NA
Dil Factor	100	Date of Analysis	10/2/02

Compound	Rpt. Limit (ppbv)	Rpt. Limit (uG/m3)	%Recovery
Carbon Tetrachloride	0.50	3.2	117
Tetrachloroethene	0.50	3.4	110

Container Type: NA - Not Applicable

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	105	70-130
Toluene-d8	90	70-130
4-Bromofluorobenzene	83	70-130

0209522

AirKinetics, Inc.

EMISSIONS CHARACTERIZATION AND TESTING SERVICES

REQUEST FOR ANALYSIS

PURCHASE ORDER No. **1846-GM**

YOUR NAME: General Atanols

LABORATORY: Air Force, Ltd.

JOB No. 11303

DATE SAMPLES
WERE TRANSMITTED: 9-25-02

EXPECTED DATE
OF RESULTS: 10-9-02

SAFETY DATA SHEET Stock Code:

TYPE OF ANALYSIS REQUIRED: Please analyze according to EPA Method TO-15 for COB and CCM only. A full raw data package will be required. Contact Karon Surden for further details.

[illegible]

* For Liberty: Contributions: \$200 - \$250

Sample Retention on file *[Signature]*

Exit Time: 2:30 PM 11-31

... ..

Transported to: Air Force Liaison

180 Blue River Road, Suite B

Folsom, CA 95630

Received by: _____

Date Time: 06/25/2012