

***Final Report For Organics  
Partitioning Resulting From  
Operation Of An INTEC Double  
Needle Sampler***

*September 2003*



*Idaho National Engineering and Environmental Laboratory  
Bechtel BWXT Idaho, LLC*

**SAIC SCIENCE TECHNOLOGY  
APPLICATIONS RESEARCH CENTER**

**FINAL REPORT FOR ORGANICS PARTITIONING  
RESULTING FROM OPERATION OF AN  
INTEC DOUBLE NEEDLE SAMPLER**

Revision 1  
September 24, 2002

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## Acronym List

cc	cubic centimeter
EE	End Effector
g	gram
GC/MS	Gas Chromatograph/Mass Spectrometer
IDEQ	State of Idaho Department of Environmental Quality
INEEL	Idaho National Engineering and Environmental Laboratory
INTEC	Idaho Nuclear Technology and Engineering Center
l	liter
LDUA	Light Duty Utility Arm
M	Molar
mg	milligram
ml	milliliter
N	Normal
ng	nanogram
NWCF	New Waste Calcining Facility
PEWE	Process Equipment Waste Evaporator
psig	pounds per square inch gage
ppb	part per billion
ppm	part per million
RCRA	Resource Conservation and Recovery Act
STAR	Science Technology Applications Research (Center)
TCA	1,1,1-Trichloroethane
TFF	Tank Farm Facility
TOC	total organic carbon
μl	microliter
VOC	volatile organic constituents

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## 1.0 BACKGROUND

The double needle sampler testing is a continuation of previous test series that investigated the fate of organic species in the Process Equipment Waste Evaporator (PEWE) system at the Idaho Nuclear Technology and Engineering Center (INTEC). This test series was designed to investigate the effects of operation of the double needle sampling systems on volatile organic constituents in an acidic feed matrix.

The PEWE, located in CPP-604, reduces the volume of mixed waste sent to the Tank Farm Facility at the INTEC. The PEWE system consists of feed tanks for accumulation of waste solutions, two evaporator trains for processing the waste solution, a bottoms collection vessel, and condensate collection vessels. The waste solutions are transferred from the feed tanks to the evaporator vessel, where the solution is heated and circulated through the evaporator. The overhead vapors generated in the evaporator are condensed and transferred to the PEW condensate collection tanks. The condensate is accumulated in the condensate collection tanks for further processing at the Liquid Effluent Treatment and Disposal Facility, CPP-1618. The remaining solution is circulated through the evaporator until procedurally designated limits are met, at which time the solution is transferred to the bottoms collection vessel. The bottoms are eventually transferred to the Tank Farm Facility for storage.

Although extensive process information is available regarding wastes processed in the PEWE, there is less information regarding hazardous organic constituents. Hazardous organic species have been discharged to the PEWE system as a result of past operating practices. Studies were conducted to determine the fate of the organic compounds in the INTEC Tank Farm,<sup>1</sup> but these studies did not address the effects of the evaporation process on the fate of the organics. SAIC has conducted four bench-scale evaporator and two Light Duty Utility Arm tests to evaluate the fate of spiked organics in a high and low molarity nitric acid simulated feeds. The results of these tests indicated that INTEC sample collection and handling procedures do not significantly effect the concentration of volatile organic or semi-volatile organic constituents in the waste stream.<sup>2</sup>

### 1.1. Previous Organic Partitioning Tests

In 1998 a series of organic partitioning tests were initiated at SAIC's Science Technology Applications Research (STAR) Center located in Idaho Falls, Idaho. These tests were designed to support the Idaho National Engineering and Environmental Laboratory (INEEL) INTEC permitting, regulatory, and closure decision makers. Primary test objectives included:

- Quantify organic partitioning that results from STAR Center bench-scale evaporator operation,
- Quantify organic losses that result from operation of the Light Duty Utility Arm (LDUA) sampler end effector, and
- Determine organic losses that may be attributed to INTEC sampling procedures.

## 1.2. Conclusions from Previous Organic Partitioning Tests

Test series conclusions were based on quantitative data analysis as well as qualitative observations. Results provided a representation of the fate of organics in the PEWE system and LDUA sample end effector.

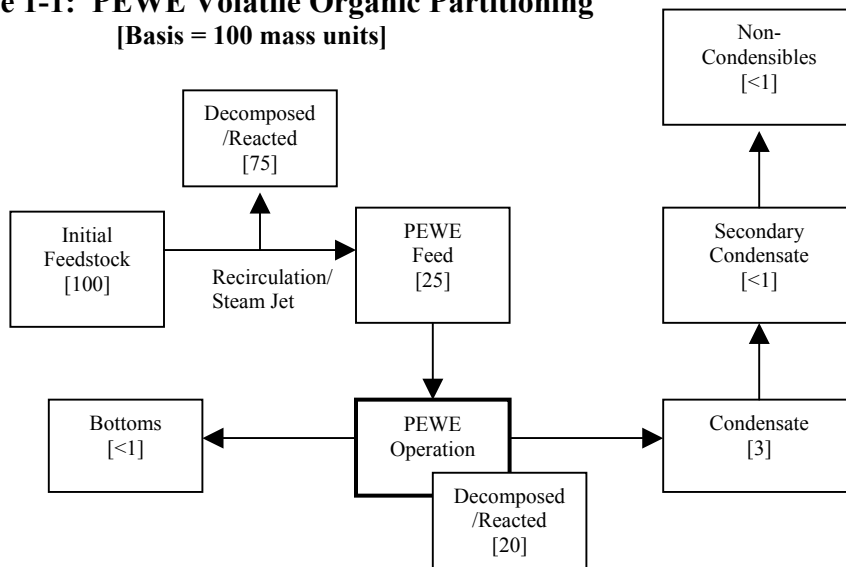
### 1.2.1. Bench-Scale Evaporator Conclusions

Analytical data revealed that up to 75% of the volatile organics and 40% of the semi-volatile organics spiked to the PEWE feedstock were destroyed, decomposed, reacted, or volatilized prior to evaporator processing. This percentage may be dependent upon the physical properties (e.g. vapor pressure) of each compound.<sup>2</sup>

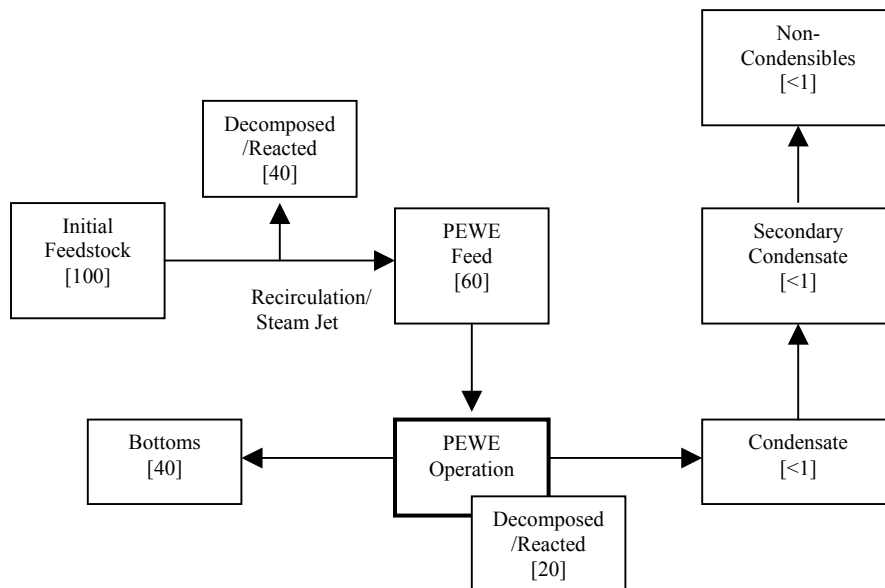
Bench-scale PEWE bottoms were virtually free of volatile organic compounds. Given that the bench-scale feedstock was spiked with 3-4 orders of magnitude higher volatile organic constituent (VOC) concentrations than found at INTEC, it is reasonable to conclude that detectable VOC concentrations will not be present in the TFF and were not introduced to the calciner. It is likely, however, that semi-volatile compounds processed through the PEWE will be present in the TFF and calciner feed.

Analytical data did not conclusively indicate that INTEC sample collection and handling procedures effect VOC or SVOC concentrations. Figures 1-1 and 1-2 below present simplified partitioning mass balances, from bench-scale evaporator operation, of volatile and semi-volatile organics.<sup>2</sup>

**Figure 1-1: PEWE Volatile Organic Partitioning**  
[Basis = 100 mass units]



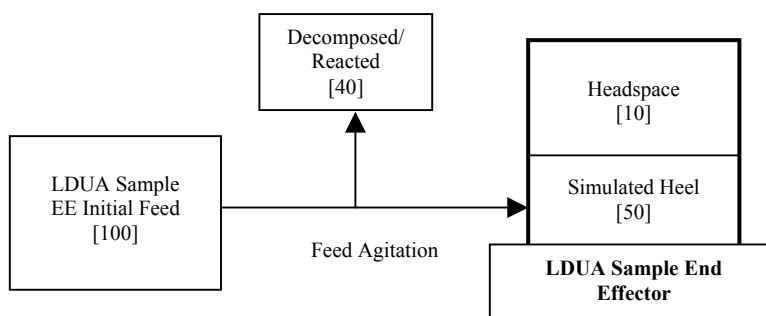
**Figure 1-2: PEWE Semi-Volatile Organic Partitioning**  
 [Basis = 100 mass units]



### 1.2.2. LDUA End Effector Conclusions

Analytical data indicated that LDUA sample EE operating vacuums effect volatilization of organic species in simulated tank heel residue. Each operating vacuum yielded consistent data resulting in a good material balance about the end effector. The data indicated that the higher the operating vacuum, the greater the resulting volatile organic volatilization. Based on LDUA testing, it was recommended that the end effector be operated at the lowest operating vacuum allowed by the heel matrix. Figure 1-3 below presents a simplified volatile organic balance about the LDUA sample EE.<sup>2</sup>

**Figure 1-3: LDUA Sample EE Volatile Organic Partitioning**  
 [Basis = 100 mass units]



An independent evaluation of INTEC analytical procedures (Analytical Chemistry Methods) compared to SW-846 specified methods was conducted to identify variances, should they exist, between the analytical protocols. Identified variances were satisfactorily addressed by the INTEC Remote Analytical Laboratory thereby establishing analytical equivalency.



## 2.0 TEST OBJECTIVES

In February 2001, the results of previous organic partitioning testing were presented to the State of Idaho Department of Environmental Quality (IDEQ). At this time, the IDEQ requested that additional testing be performed to statistically quantify potential losses of volatile organic species that may be attributed to the double needle sampling systems currently utilized at INTEC. As such, the double needle sampler testing was designed to determine the effects of operation of the double needle sampling systems on volatile organic species that have potentially been introduced into the PEWE system.

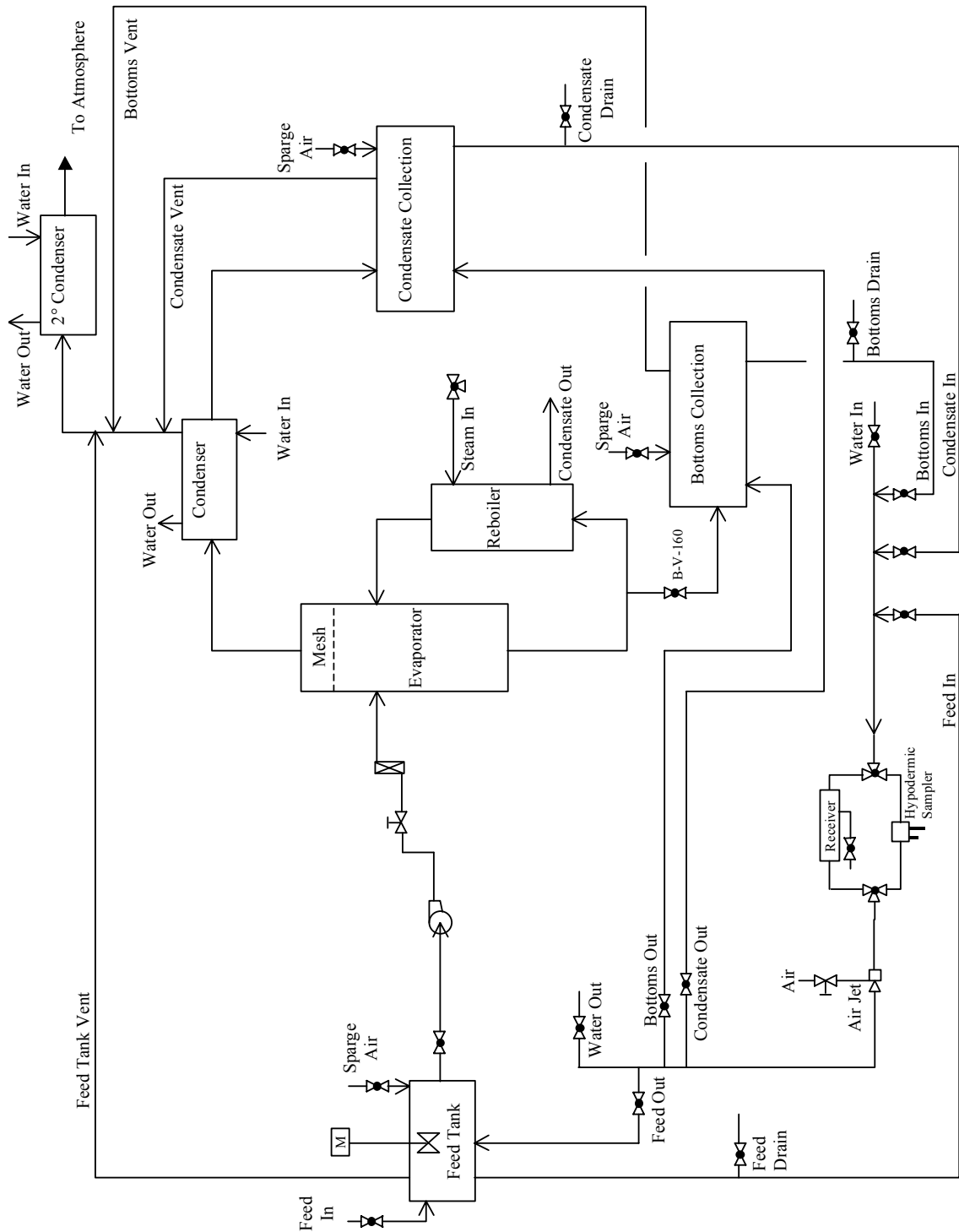
## 3.0 BENCH-SCALE PROCESS EQUIPMENT

The bench-scale PEWE system was been designed and constructed at the SAIC Science and Technology Applications Research (STAR) Center to model typical PEWE system operations. The bench-scale system consists of a feed tank, evaporator vessel with an attached thermosiphon reboiler, bottoms collection tank, condensate collection tank, and tank sampling station.

Testing of the double needle sampler system utilized the feed tank, sample station, and vessel off-gas systems only. An image of the STAR Center bench-scale evaporator system is presented as Figure 3-1 and a flow diagram of the bench-scale system is shown in Figure 3.2. The only modification to the bench-scale evaporator system for double needle sampler testing was the addition of a mechanical mixer to the feed tank



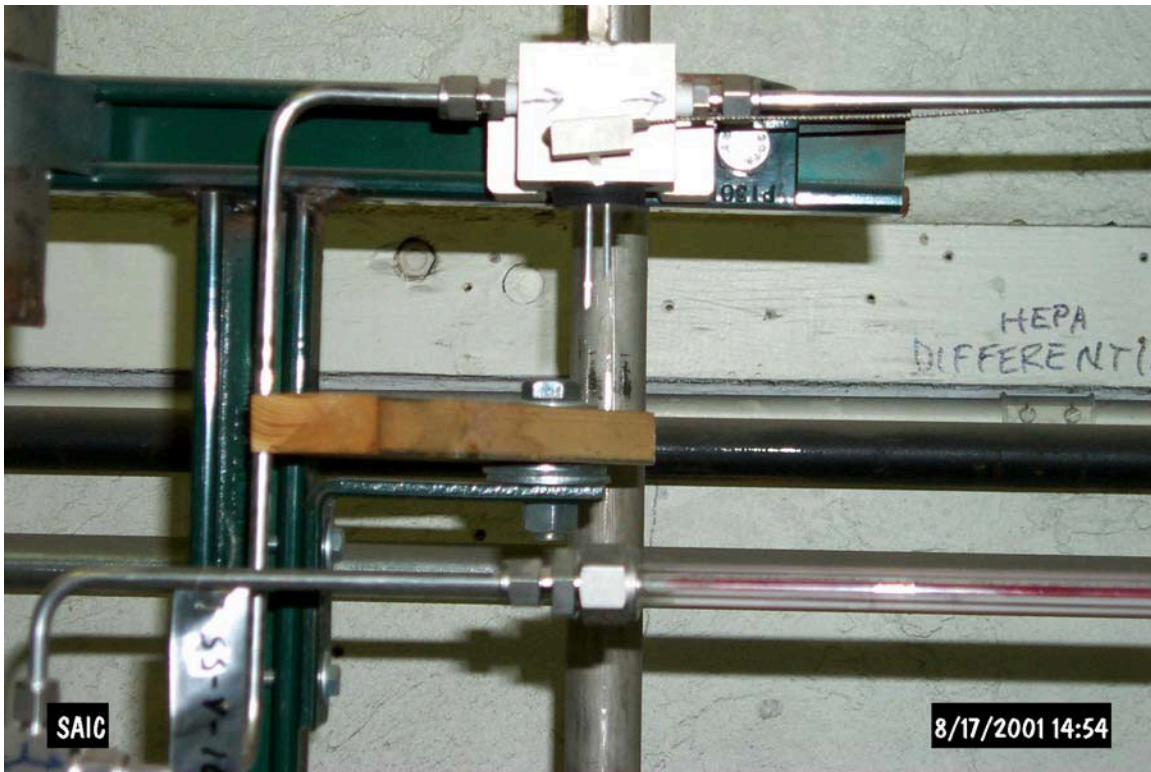
**Figure 3-1: STAR Center Bench-Scale Evaporator System**



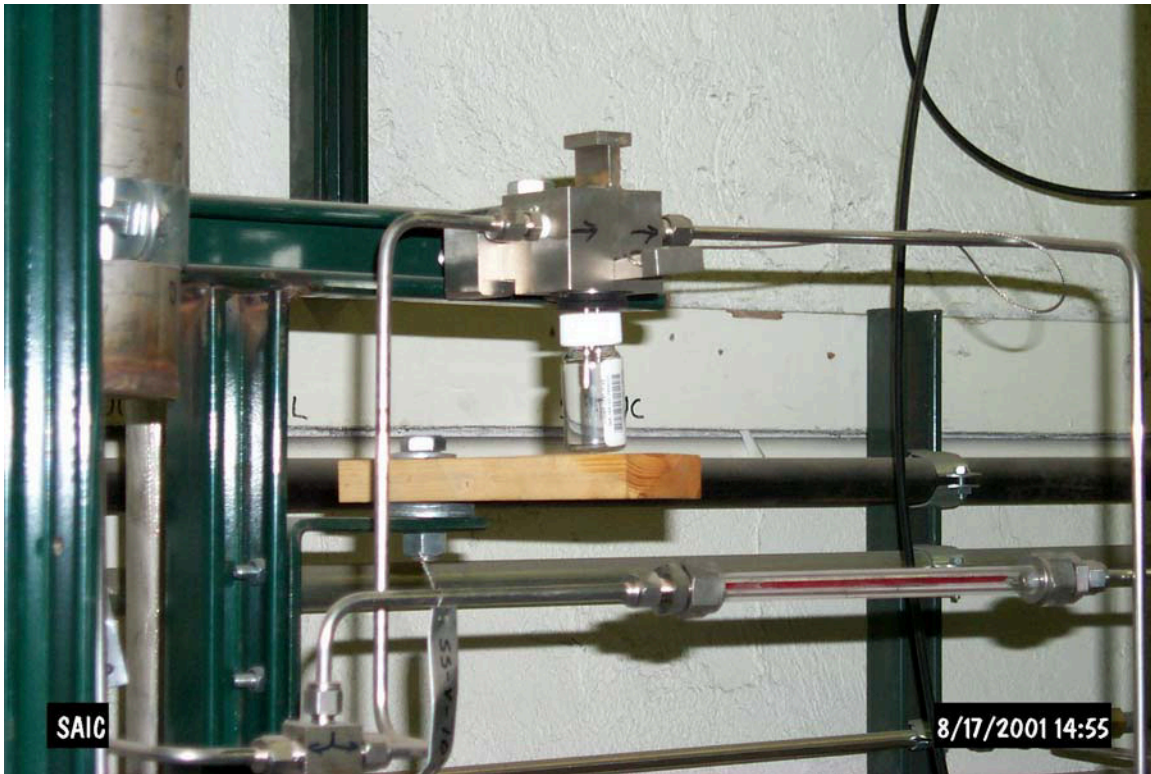
**Figure 3-2: Bench-Scale Evaporator System Diagram**

A sampling station and associated piping was assembled as part of the bench-scale system for the previous test series. The sample station was designed to model existing sample stations at INTEC. The double needle sample assembly was supplied by INTEC and is identical to the sample assemblies used at INTEC.

The sampling station can sample solutions from the feed tank, bottoms tank, and condensate tank. The sample station consists of a double hypodermic needle sampler and a larger volume sample receiver. An air jet is used to initiate sample flow through the sample station and recirculate the sample back to the respective tank. The hypodermic needle sampler consists of a double hypodermic needle sample assembly to which a sample bottle, fitted with a neoprene or Teflon diaphragm, can be attached. When the air jet is actuated, the sample flows into the sample bottle through one needle (the long needle) and exits through the other (the short needle), filling the sample bottle. This sampling method results in headspace in the top of the sample bottle. The double needle sampler was used exclusively throughout this testing. Figure 3-3 and 3-4 are images of the double needle sampling system.



**Figure 3-3 Double Needle Sampler**



**Figure 3-4 Double Needle Sampler with Sample Bottle**

## **4.0 DOUBLE NEEDLE SAMPLER TEST SUMMARY**

### **4.1. Feed Preparations**

Previous organic partitioning testing at the SAIC STAR Center was used to determine organic species spiking concentrations. Bench-scale evaporator tests in September, 1998, (Test 1 and 2) spiked target organic constituents to a concentration of 1,000 mg/l in the feed, which is 3-4 orders of magnitude higher than concentrations typical of INTEC waste streams. Data analysis revealed that this concentration could be lowered while still maintaining acceptable detection limits for the target organic constituents.

Additional organic partitioning tests in both the bench-scale evaporator system and with the LDUA end effector (from October 1998 to March 2000) utilized target organic constituents that were spiked to a concentration of 100 mg/l in the feed. These concentrations were still an order of magnitude higher than typical INTEC waste streams, but were required to help ensure detection of the target organics by the analytical laboratory.

The concentration of organic and inorganic constituents in the feed solution for the double needle sampler testing was similar to previous organic partitioning tests. Operations and sampling procedures were provided in a test plan entitled *Organics Partitioning Resulting from Operation of an INTEC Double Needle Sampler*<sup>3</sup> which is

provided as Attachment 1. Double needle sampler testing was conducted the week of July 9, 2001. All key operational and sampling procedures/parameters as well as significant testing observations were documented in a bound logbook. Copies of the logbook entries and datasheets are provided as Attachment 2.

Simulated feed materials were generated at the STAR Center to model known acid waste feed solutions processed in the PEWE system at INTEC. Organics were spiked into 44 liters of nitrated acid feed to concentrations of 100 mg/l. Organic constituents were added in greater concentrations than are typically found in INTEC waste streams. This helps ensure that the analytical methods applied to the sample solutions are capable of detecting the organic constituents present in the solutions. Table 4.1 shows the chemical species that are present in the simulated waste stream, the desired concentrations of the chemical, and the quantity of chemical required to obtain the desired concentration. The target volatile organics for this testing were carbon disulfide, toluene, and 1,1,1-trichloroethane.

**Table 4-1: Double Needle Sampler Feed Solutions (for 44 Liters of feed)**

Species	Chemical Formula	MW (g/mole)	Acid Feed		
			Desired Conc.	Units	Required Quantity (g)
Acid		63.01	1.2	N	3.34 L
Nitrate					
Aluminum	Al(NO <sub>3</sub> ) <sub>3</sub> Solution	2.2 Molar	5.4	g/l	4.00 L
Calcium	Ca(NO <sub>3</sub> ) <sub>2</sub> *4H <sub>2</sub> O	236.15	0.166	g/l	43.0
Iron	Fe(NO <sub>3</sub> ) <sub>3</sub> *9H <sub>2</sub> O	404.02	0.008	g/l	2.55
Sodium/Potassium	NaNO <sub>3</sub>	85.01	3.954	g/l	643.0
Carbon Disulfide	CS <sub>2</sub>	76.14	100	mg/l	3.48 ml
Toluene	C <sub>6</sub> H <sub>5</sub> CH <sub>3</sub>	92.14	100	mg/l	5.08 ml
1,1,1-Trichloroethane	CH <sub>3</sub> CCl <sub>3</sub>	133.40	100	mg/l	3.29 ml

The organic species present in the waste stream were spiked directly to the feed tank after negative pressure to the system had been established. The feed solution was mechanically mixed to help ensure the feed homogeneity prior to performance of the double needle sampler testing.

#### 4.2. Test Methodology and Sample Collection

After completion of feed preparation activities, the feed solution was mechanically mixed for 4 1/2 hours. The sampling rationale for the double needle sampler testing was to collect a total of twenty feed solution samples for comparison of volatile organic constituent concentrations. Ten samples were collected using the double needle sample system and ten samples were collected from the feed tank drain.

For comparison purposes, one sample was collected using the double needle sampling system and a second sample was immediately collected from the feed tank drain (spigot sample). This procedure was repeated until the full twenty samples were collected. Table 4-2 lists the samples identification number, sample location, analysis

type, and required sample volume for the double needle sampler testing. The samples collection methods for the double needle sampler system and the feed tank are discussed in the following sections.

**Table 4-2: Double Needle Sampling System – Sampling Requirements**

Sample ID No.	Description	Analytes	Volume
Feed Tank Sample			
05-FT-01	Direct Feed Tank Sample	Target	120 ml
05-FT-02	Direct Feed Tank Sample	Target	120 ml
05-FT-03	Direct Feed Tank Sample	Target	120 ml
05-FT-04	Direct Feed Tank Sample	Target	120 ml
05-FT-05	Direct Feed Tank Sample	Target	120 ml
05-FT-06	Direct Feed Tank Sample	Target	120 ml
05-FT-07	Direct Feed Tank Sample	Target	120 ml
05-FT-08	Direct Feed Tank Sample	Target	120 ml
05-FT-09	Direct Feed Tank Sample	Target	120 ml
05-FT-10	Direct Feed Tank Sample	Target	120 ml
Double Needle Samples			
05-DN-11	Double Needle Sample	Target	120 ml
05-DN-12	Double Needle Sample	Target	120 ml
05-DN-13	Double Needle Sample	Target	120 ml
05-DN-14	Double Needle Sample	Target	120 ml
05-DN-15	Double Needle Sample	Target	120 ml
05-DN-16	Double Needle Sample	Target	120 ml
05-DN-17	Double Needle Sample	Target	120 ml
05-DN-18	Double Needle Sample	Target	120 ml
05-DN-19	Double Needle Sample	Target	120 ml
05-DN-20	Double Needle Sample	Target	120 ml
Field Blank Sample			
05-QC-21	Distilled Water Field Blank	TOC	80 ml
05-QC-22	Distilled Water Trip Blank	TOC	80 mL
05-QC-23	Cross Contamination Sample	TOC	80 ml

#### 4.2.1. Sample Collection Using the Double Needle Sampler

The double needle sample station was designed and fabricated to model existing PEWE sampling capabilities. The double needle sampler and air jet were supplied by INTEC. Operation of the sampling station was similar to the approved INTEC liquid sampling procedures.

To begin sampling, the sample bottle was connected to the sample station by piercing the diaphragm with a double-hypodermic needle sampling assembly. Once connected, the sample stream was circulated through the sample bottle with the use of an air jet and air lift. The sample stream flowed into the bottle through one needle and exited through the other, filling the bottle. The sample stream was recirculated for 10 minutes on the first sample bottle. The double needle sampler was then isolated by

closing the inlet and outlet isolation valves, the sample bottle was removed from the double needle sampler, a new sample bottle was connected to the sample station, and the sample stream was recirculated through the second bottle for 5 minutes. This procedure was repeated until four sample bottles were filled using the double needle sampler.

As shown in Table 4-2, the analytical laboratory required 120-ml of sample volume for performance of the volatile organic analysis. Since sampling with the double needle sampler results in head space being present in the sample vials, four 40-ml sample vials were required for each sample series using the double needle sampling system. Sample collection and analysis of volatile organic species requires that no head space be present in the sample containers. Therefore, the fourth sample vial collected was used to fill the first three sample vials, the punctured septa were replaced, and the resultant sample volume was 120-ml.

Previous studies at INTEC have analyzed and recommended operating conditions for jet transfer systems. The preferred minimum sample circulation rate is 100 ml/min and a rate of 200 – 300 ml/min is considered a good circulation rate.<sup>4</sup> The flow rate of the sample stream through the double needle sampler was measured at the conclusion of the double needle sampler testing. The flow rate was measured by placing a 1 liter graduated cylinder on the sample return line and recording the volume of liquid that was transferred to the cylinder in 1 minute. The sample stream flow rate was determined to be 270 ml/min and would be considered a good recirculation rate.

The double needle sample system was operated with an initial sample recirculation time of 10 minutes and three subsequent recirculation times of 5 minutes each. The 10 minute recirculation would result in 2700 ml (2.7 l) of sample stream recirculating through the double needle sampler, which was approximately 6 percent of the total feed volume. Each subsequent 5 minute recirculation would result in 1350 ml (1.35 l) of sample stream recirculating through the sample system, which was approximately 3 percent of the total feed volume.

Overall, 6750 ml (2700 ml + 3\*1350 ml) of sample stream was recirculated through the double needle sampler to obtain the 120-ml of sample volume required for analysis. This equates to approximately 15 percent of the total feed volume being recirculated for each sample series obtained using the double needle sampler.

#### 4.2.2. Sample Collection from the Feed Tank Drain

The feed tank samples were collected directly into 40-ml amber glass vials with Teflon lined septum. Three amber glass vials were required for each sample to obtain the desired 120-ml of total sample volume. The procedure for collecting direct feed tank samples is listed below:

1. Place a container below the feed tank drain valve .
2. Open the feed tank drain valve and allow feed solution to flush the drain line, then close the feed tank drain valve

3. Place an empty sample container below the feed tank drain valve.
4. Open the feed tank drain valve and allow the solution to gravity flow into sample container.
5. When the sample container is full, close the drain valve.
6. Put the lid on the sample container, verify zero headspace, and place the sample container in the required shipping device for shipment to the analytical laboratory.

### **4.3. Analytical Results**

All samples were analyzed by Hauser Laboratories located in Boulder, CO. Liquid organic samples were analyzed with a gas chromatograph/mass spectrometer (GC/MS) in accordance with SW 846-8260 guidelines. Liquid sample calibration curves for each analyte ranged from 50 ng to 400 ng. The chain of custody forms and the analytical package received from Hauser is included in Attachment 3. Hauser Laboratories conducted the analyses for the previous organic partitioning studies and is familiar with the intricacies of performing volatile organic analyses in an acidic matrix. Although every effort was made to minimize data uncertainty, the following factors potentially contributed to error.

1. Feed homogeneity: It was thought that with adequate mechanical feed mixing (5 hours), the spiked organics would be thoroughly dispersed/homogenized within the feed mixture. During collection of the initial feed tank sample, organic globules were visible in the sample bottle. The organic globules were also observed during previous bench-scale evaporator tests that utilized a recirculation pump for mixing. The initial feed tank sample was returned to the feed tank and mechanical mixing was reinitiated. In addition, a small amount of sparge air was introduced to the feed tank to promote mixing.
2. Chemical Reaction: Organic partitioning testing resulting from typical INTEC operations has been conducted since 1998. This testing has resulted in both qualitative and quantitative determinations for the partitioning of organics in INTEC systems. However, the rates of reaction and mechanisms between nitric acid and volatile organic constituents have not been fully quantified for the various INTEC operating scenarios.



## 5.0 DATA INTERPRETATION

### 5.1. Double Needle Sampler Test Data

The primary objective of the double needle sampler testing was to determine the effects of operation of INTEC double needle sampling systems on volatile organic species that were spiked in known concentrations to a typical PEWE feed solution. The target organic constituents (carbon disulfide, 1,1,1-trichloroethane, and toluene) were spiked to a concentration of 100 mg/l in the feed solution, which is an order of magnitude greater than concentrations typically anticipated for INTEC waste streams. This spiking concentration helps ensure detection of the target organic constituents by the analytical laboratory.

The feed was then mechanically mixed for approximately 4 1/2 hours to homogenize the acid matrix and the organics. After mixing, an initial feed tank sample was obtained using the procedure described in Section 4.2.2. The feed tank drain line was flushed into a new, clean container. Organic globules were present in the solution drained from the feed tank which indicated in a non-homogenous feed mixture. The samples were added back to the feed tank and the feed tank solution was allowed to mix for an additional 30 minutes. A small amount of air sparge was also applied to the feed tank to assist with mixing.

Sampling activities were then reinitiated with the double needle sampling system. Four 40-ml sample bottles were required for each data series collected using the double needle sampler. A feed tank sample series was collected immediately following the double needle sampler operation. Three 40-ml sample bottles were required for each direct feed tank sample.

Table 5-1 lists the analytical results from the double needle sampler testing. The results have been arranged in the order the samples were collected to assist with the comparison of the double needle sampler and the direct feed tank samples.

**Table 5-1. Analysis of Double Needle Sampler versus Direct Feed Tank Samples for Volatile Organic Constituents.**

Sample ID	Sample Location	Time of Day	CS <sub>2</sub> (ng/uL)	1,1,1-TCA (ng/uL)	Toluene (ng/uL)
05-FT-01A	Feed Tank	9:45	3.2	3.69	3.74
05-DN-11A	Double Needle	9:20 - 09:45	9.92	10.52	10.32
05-DN-11A (dup)	Double Needle	9:20 - 09:45	9.6	10.16	9.96
05-FT-02A	Feed Tank	10:11	0.74	0.82	0.68
05-DN-12A	Double Needle	09:51 - 10:16	1.31	1.87	1.93
05-FT-02B	Feed Tank	10:11	0.49	0.6	0.64
05-DN-12B	Double Needle	09:51 - 10:16	0.74	1.12	1.26
05-FT-02B (dup)	Feed Tank	10:11	0.5	0.63	0.65
05-FT-03A	Feed Tank	10:37	0.48	0.39	0.17
05-DN-13A	Double Needle	10:12 - 10:37	0.2	0.31	0.37
05-FT-04A	Feed Tank	11:03	0.23	0.18	0.05
05-DN-14A	Double Needle	10:38 - 11:03	0.05	0.07	0.1
05-FT-05A	Feed Tank	11:30	0.14	0.1	bloq
05-DN-15A	Double Needle	11:05 - 11:30	bloq	bloq	bloq
05-FT-06A	Feed Tank	11:56	0.12	0.11	nd
05-DN-16A	Double Needle	11:31 - 11:56	nd	nd	nd
05-FT-07A	Feed Tank	12:22	0.08	0.07	nd
05-DN-17A	Double Needle	11:57 - 12:22	nd	nd	nd
05-FT-08A	Feed Tank	12:48	nd	nd	nd
05-DN-18A	Double Needle	12:23 - 12:48	nd	nd	nd
05-FT-09A	Feed Tank	13:14	0.06	0.08	nd
05-DN-19A	Double Needle	12:49 - 13:14	nd	nd	nd
05-FT-10A	Feed Tank	13:40	0.09	0.09	nd
05-DN-20A	Double Needle	13:15 - 13:40	nd	nd	nd
dup = duplicate nd = not detected (detection limit = 0.025 ng/ul) bloq = below limit of quantitation (0.050 ng/ul) 1,1,1-TCA = 1,1,1-trichloroethane					

The most obvious trend in the volatile organic data was the consistent decrease in the concentration of the organic constituents with each sample series until most of the organic species were below the analytical detection limit. The detection limit for the organic constituents was 0.050 ng/μl or 50 parts per billion. This would suggest that the nitric acid solution was reacting/decomposing the spiked volatile organic constituents and/or the organic constituents were being stripped from solution by the air jet, air sparge, air lift. Both of these mechanisms most likely played a role in decreasing the volatile organic concentrations in the feed solution.

It should be noted that the spiked concentration of the volatile organics (100 mg/L) was significantly greater than the concentrations that would be typical of INTEC waste streams. The higher concentrations were required to ensure that detectable quantities of volatile organics would be present for analysis. It appears that some air

stripping of volatile organic constituents may occur during operation of the double needle samplers, however, the air stripping occurs after sample collection and should not significantly effect the sample validity. The effectiveness of air stripping techniques is dependent on a number of variables (such as volatile organic concentration, operating temperatures, Henry's Law constants, and flow rates) and the higher spiking concentrations in the feed solution may have contributed to observed air stripping effects.<sup>6</sup>

Figures 5-1, 5-2, and 5-3 graphically show the reduction in concentration of the target volatile organics for both spigot and double needle samples. It can be seen that all three of the organic constituents follow the same general curve for reduction in concentration. Comparison of the analytical results relative to the time of day the samples were obtained results in a good correlation between the double needle and spigot samples. The mean time for the double needle sample period (25 minutes each) was utilized to compare the sample results. Attachment 4 contains photos of the bench-scale evaporator system at the SAIC STAR Center.



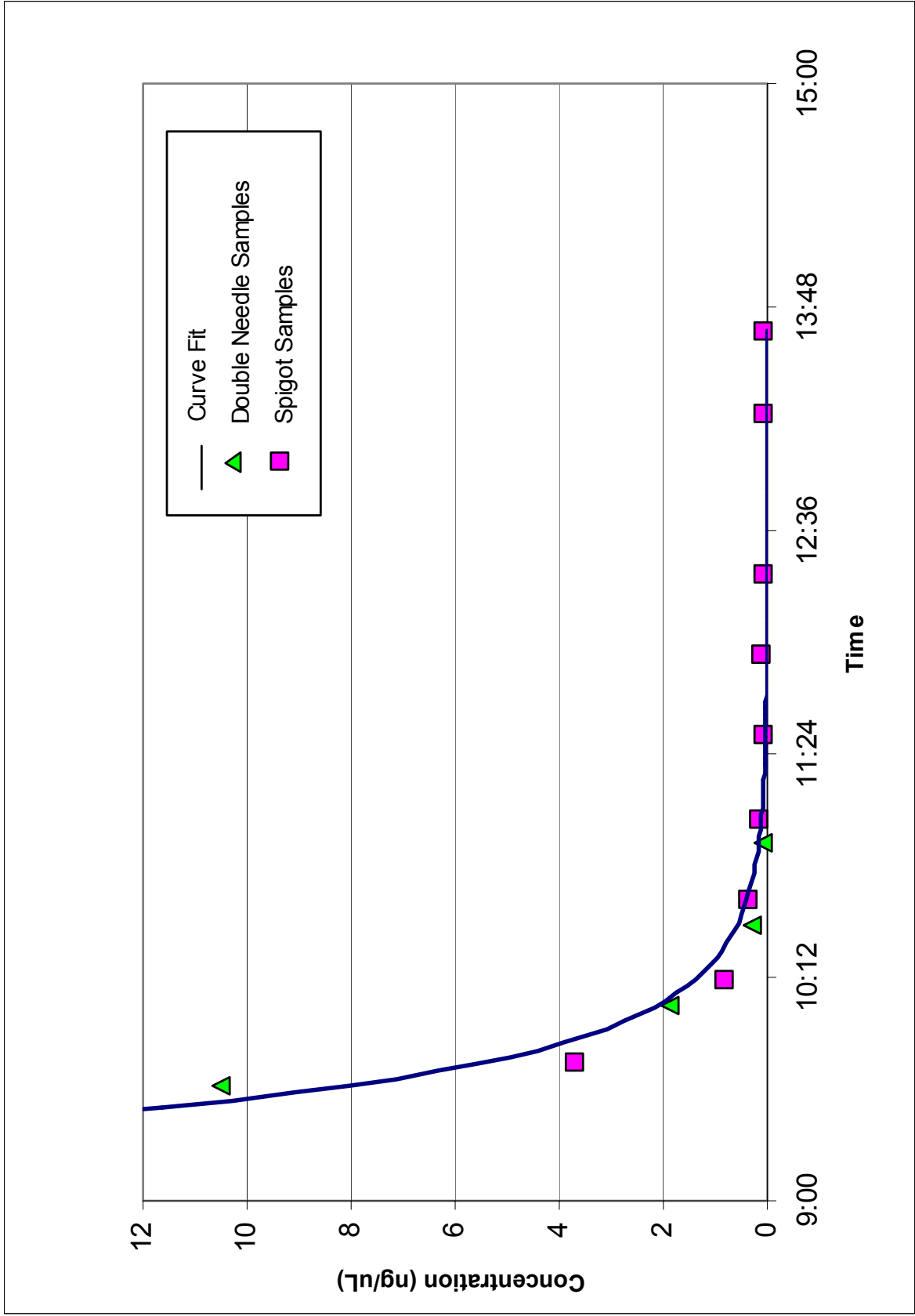


Figure 5-2: Comparison of Time Relative Sample Analysis for 1,1,1-Trichloroethane Data.<sup>7</sup>



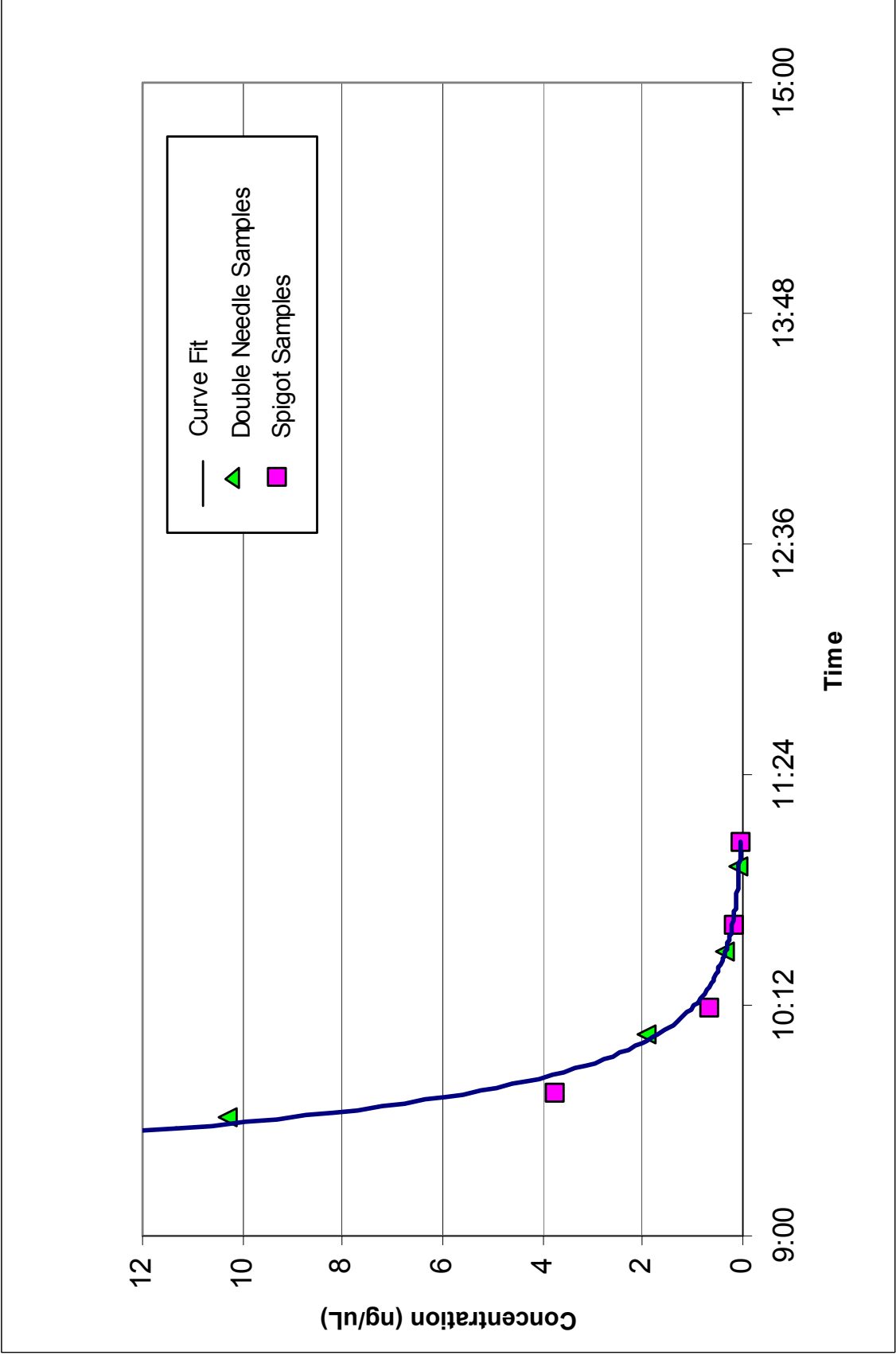


Figure 5-3: Comparison of Time Relative Sample Analysis for Toluene Data<sup>7</sup>

## **5.2. Comparison with Previous Organic Partitioning Tests**

Previous bench-scale evaporator tests concluded that up to 75 percent of the volatile organic species spiked to the feedstocks were destroyed, decomposed, reacted, or volatilized prior to evaporator processing. The bench-scale evaporator feeds were steam jetted and air sparged prior to being sampled and fed to the evaporator.

For the LDUA test series, the concentration of the organic constituents in the feed solution was 100 mg/l. The analytical data from Test 2 with the LDUA end effector yielded volatile organic constituent concentrations of 0.2 to 1 mg/l, which is a 99 percent reduction in the volatile organic constituents concentrations. The LDUA Test 2 feed stock was mechanically mixed for 5 hours prior to sampling with the end effector.

The double needle sampler testing resulted in a reduction of approximately 90 percent of the volatile organic constituents after five hours of mechanical mixing (including 20 minutes of air sparge) and almost complete reduction by the end of the test. This is consistent with the conclusions documented from previous organic partitioning tests (bench-scale evaporator and LDUA EE operation) and indicates that the acidic nature of the solution plays a key role in the reduction of VOC concentrations in typical INTEC waste streams.

## **5.3. Comparison with INTEC Sampling**

The double needle sampler test was designed to be a bench-scale operation (44 liter tank volume) for ease of sample collection and to minimize hazardous waste generation. The proportions utilized in the double needle sampler test are quite different from those present in the INTEC waste processing system.

The blend and hold tanks (VES-NCC-101, -102, and -103) typically hold 3,000 to 4,000 gallons. The sparge volume and the volume of air supplied to the airlift may be comparable and may similarly affect the solution transported to the sampler. The PEW tanks do receive the sample return from the double needle sampling system and could be affected by the air-jet, but this effect would be small due to the large volume of the tanks (the evaporator feed sediment tank, VES-WL-132 and the evaporator feed collection tank, VES-WL-133 have 4,700 gallon and 19,000 gallon capacities, respectively).<sup>9</sup>

Analysis of INTEC tank farm facility solutions for volatile organic constituents have been consistently less than the minimum quantification level of 10 ng/ml.<sup>5</sup> Based on the results of the organic partitioning test series conducted at the SAIC STAR Center, it is reasonable to infer that any volatile organic constituents introduced to the INTEC waste system have been destroyed, decomposed, reacted, or volatilized as a result of typical plant conditions and reactions with the nitric acid waste matrix.



## 6.0 CONCLUSIONS

Test series conclusions are based on quantitative data analysis as well as qualitative observations. The results provide representation of the fate of volatile organic constituents and the validity of samples that have been collected using INTEC double needle samplers.

Direct statistical comparison of the double needle and spigot samples is difficult due to decomposition, reaction, and/or volatilization of the target organic constituents. However, a good correlation between the double needle and spigot samples is observed by graphical representation of the analytical data relative to the time of day the samples were collected. This data indicates that samples collected utilizing a double needle sampler are comparable to spigot samples.

After 4 1/2 hours of mixing with a mechanical mixer, organic globules were still visible in a sample. Then after air sparging and mechanical mixing for 30 minutes, the first samples were taken and a sequence of sampling every few minutes was initiated. More than 90% of the organics were destroyed by the first sample and roughly 60-80% of the remaining quantities were destroyed in each 25-minute period through the remainder of the test.

Analytical data, compiled for previous organic partitioning studies (tests 1 through 4 in the bench scale evaporator and LDUA end effector testing), indicated that volatile organic constituents would be destroyed, decomposed, reacted, or volatilized as a result of typical INTEC plant conditions. The bench-scale evaporator utilized steam recirculation and air sparging to mix the tank contents, while the LDUA tests employed a mechanical mixer. A reduction of up to 99 percent of the volatile organic constituents concentration was observed in these tests. The rapid reduction of volatile organics concentrations, as evidenced in bench-scale evaporator, LDUA EE, and double needle sampler testing, suggests that nitric acid digestion plays a key role in the reduction of volatile organic concentrations in typical INTEC waste streams.

Based on the reduction of volatile organic constituents and previous sampling of INTEC tank farm facility solutions, it is reasonable to infer that volatile organic constituents introduced to the INTEC waste system are most likely destroyed, decomposed, reacted, or volatilized as a result of typical plant conditions and/or reactions with the nitric acid waste matrix and will be below quantification limits. The double needle and spigot samples both yielded comparative results. The double needle sampler may contribute to some air stripping of organics, but is negligible compared to the rapid degradation of the volatile organic compounds in nitric acid waste solutions. It is unlikely that trace volatile organic concentrations could be detected, in INTEC waste streams, utilizing any sampling method.

## 7.0 REFERENCES

1. Radian Corporation, *Results of a Laboratory Study to Assess the Fate of Organic Compounds in Synthetic ICPP Tank Farm Waste*, July 17, 1995.
2. SAIC, *PEWE and LDUA Bench-Scale Test Series, Organic Partitioning Comprehensive Final Report*, July 23, 2000.
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6. SAIC, *INEEL High Level Waste and Facilities Disposition Environmental Impact Statement, ICPP Delisting Study*, April, 1998.
7. Millet, C.B., letter to M.B. Heiser, "Organic Analysis with Double Needle Samplers", August 16, 2002.

**ATTACHMENT 1**

**Double Needle Sampling System Test Plan**

**Double Needle Sampling System**

**Test Plan**

**Organics Partitioning Resulting from  
Operation of an INTEC Double Needle Sampler**

**Test 5, Double Needle Sampler Statistical Testing**

**SAIC STAR Center**

**June 25, 2001**

Prepared for:  
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Contract No. K00-564414, TO 10

**Double Needle Sampling System**

**Test Plan**

**Organics Partitioning Resulting from  
Operation of an  
INTEC Double Needle Sampler**

**Test 5, Double Needle Sampler Statistical Testing**

**SAIC STAR Center**

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# Test 5, Double Needle Sampler Statistical Testing

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## 1.0 BACKGROUND

The Process Equipment Waste Evaporator (PEWE), located in CPP-604, reduces the volume of mixed waste sent to the Tank Farm Facility at the Idaho Nuclear Technology and Engineering Center (INTEC). The PEWE system consists of feed tanks for accumulation of waste solutions, two evaporator trains for processing the waste solution, a bottoms collection vessel, and condensate collection vessels. The waste solutions are transferred from the feed tanks to the evaporator vessel, where the solution is heated and circulated through the evaporator. The overhead vapors generated in the evaporator are condensed and transferred to the PEW condensate collection tanks. The condensate is accumulated in the condensate collection tanks for further processing at the Liquid Effluent Treatment and Disposal (LET&D) Facility, CPP-1618. The remaining solution is circulated through the evaporator until procedurally designated limits are met, at which time the solution is transferred to the bottoms collection vessel. The bottoms are eventually transferred to the Tank Farm for storage.

Hazardous organic species have been discharged to the PEWE system as a result of past operating practices. Studies have been conducted to determine the fate of the organic compounds in the INTEC Tank Farm,<sup>1</sup> but these studies did not address the effects of the evaporation process on the fate of the organics. SAIC has conducted four bench-scale studies to evaluate the fate of spiked organics in a high and low molarity nitric acid simulated feeds. These results of these tests indicated that INTEC sample collection and handling procedures do not significantly effect the concentration of volatile organic or semi-volatile organic constituents in the waste stream.<sup>2</sup> This test (Test 5) is designed to validate the sample collection (double needle sampler systems) and handling procedures currently employed by INTEC operations.

## 2.0 TEST OBJECTIVES

The purpose of Test 5 is to determine the effects of operation of the double needle sampling systems on volatile organic species that have been introduced into the PEWE system. Data will be evaluated to quantify the fate of organic constituents due to sampling system operation. Results will be incorporated into a final report designed to support INTEC RCRA compliance activities. This will be accomplished as follows:

- 1) A mechanical mixer will be added to the PEWE feed tank. The mechanical mixer will ensure a homogenous feed solution for sampling operations.
- 2) Samples will be collected and analyzed for spiked organic-constituents. This will involve collecting feed samples directly from the feed tank and also using the double needle sampling system. Samples will then be analyzed for spiked organic constituents as well as total organic carbon.
- 3) Evaluate the effects of the double needle sampling system on spiked organic constituents. This is accomplished by collecting approximately ten direct samples and ten double needle samples, in a planned test series, for comparison.

- 4) Verify conclusions drawn from previous bench-scale tests. Fill in double needle sampling system data gaps necessary to support organic partitioning hypotheses.

### **3.0 BENCH-SCALE SYSTEM DESIGN**

A bench-scale PEWE system has been designed and constructed at the SAIC Science and Technology Applications Research (STAR) Center to model typical PEWE system operations. The bench-scale system consists of a feed tank, evaporator vessel with an attached thermosiphon reboiler, bottoms collection tank, condensate collection tank, and tank sampling station. A flow diagram of the bench-scale system is shown in Figure 3.1. The only modification to the bench-scale evaporator system for this test was the addition of a mechanical mixer to the feed tank.

A sampling station and associated piping has been assembled as part of the bench-scale system. The sampling station can sample solutions from the feed tank, bottoms tank, and condensate tank. The sample station consists of a double hypodermic needle sampler and a larger volume sample receiver. An air jet is used to initiate sample flow through the sample station and recirculate the sample back to the respective tank. The hypodermic needle sampler consists of a double hypodermic needle sample assembly to which a sample bottle, fitted with a neoprene or Teflon diaphragm, can be attached. When the air jet is actuated, the sample flows into the sample bottle through one needle and exits through the other, filling the sample bottle. This sampling method will result in headspace in the top of the sample bottle.

The sample receiver is a section of stainless steel pipe with an inlet and an outlet for the sample solution. When the air jet is actuated the sample flows into the bottom of the sample receiver and exits through the top, filling the receiver. The sample is then drained from the receiver into sample bottles by opening the drain valve located at the bottom of the receiver.





## 4.0 SYSTEM OPERATIONS

The following subsections provide the Test 5 operations details. These include feed preparation, sampler system operation, sample collection, and system cleaning procedures. Additional subsections address health and safety and waste management.

### 4.1 Feed Preparation

Simulated feed materials will be generated at the STAR Center to model known acid waste feed solutions processed in the PEWE system at INTEC. Organic constituents will be spiked in the feed material to determine the partitioning of these species as a result of double needle sampling system operation. Organic constituents are added in greater concentrations than are typically found in INTEC waste streams. This helps ensure that the analytical methods applied to the sample solutions are capable of detecting the organic constituents present in the solutions. Table 4.1-1 shows the chemical species that are present in the simulated waste stream, the desired concentrations of the chemical, and the quantity of chemical required to obtain the desired concentration.

**Table 4.1-1: Test 5 Feed Solutions (for 44 Liters of feed)**

Species	Chemical Formula	MW (g/mole)	Acid Feed		
			Desired Conc.	Units	Required Quantity (g)
Acid		63.01	1.2	N	3.34 L
Nitrate					
Aluminum	Al(NO <sub>3</sub> ) <sub>3</sub> Solution	2.2 Molar	5.4	g/l	4.00 L
Calcium	Ca(NO <sub>3</sub> ) <sub>2</sub> *4H <sub>2</sub> O	236.15	0.166	g/l	43.0
Iron	Fe(NO <sub>3</sub> ) <sub>3</sub> *9H <sub>2</sub> O	404.02	0.008	g/l	2.55
Sodium/Potassium	NaNO <sub>3</sub>	85.01	3.954	g/l	643.0
Carbon Disulfide	CS <sub>2</sub>	76.14	100	mg/l	3.48 ml
Toluene	C <sub>6</sub> H <sub>5</sub> CH <sub>3</sub>	92.14	100	mg/l	5.08 ml
1,1,1-Trichloroethane	CH <sub>3</sub> CCl <sub>3</sub>	133.40	100	mg/l	3.29 ml

The organic species present in the waste stream will be spiked directly to the feed tank after negative pressure to the evaporator vessels has been established. The feed solution will be mechanically mixed to help ensure the feed is homogenous prior performance of the double needle sampler test series.

### 4.2 Start-up Instructions

#### 4.2.1 Pre-Start

- 4.2.1.1 Turn on the exhaust blower and adjust the bleed air supply valve (OG-V-127) to provide vacuum in the evaporator system. The evaporator should be operated at slightly negative conditions (~3 inches WC).

- 4.2.1.2 Verify the sample station equipment is properly configured for operation. Verify all vessel drain, sample supply, sample return, and air sparge valves are closed. The peristaltic pump and isolation ball valve (F-V-121) provide isolation of feed solution to the evaporator and must be turned off and closed, respectively..
  - 4.2.1.3 Turn on the STAR Center cooling tower to initiate cooling water flow to the primary and secondary condensers.
  - 4.2.1.4 Verify that feed preparations activities have been completed, and that the feed tank contains the required volume of feed for the test.
- 4.2.2 Feed Preparation
- 4.2.2.1 Add the pre-measured organic species to the feed vessel through the organic addition valve (F-V- 125) located on the top of the feed vessel.
  - 4.2.2.2 Turn on the mechanical mixing device and allow the feed solution to mix for approximately 5 hours.

### **4.3 Sample Collection Instructions**

Samples will be collected to determine organic partitioning associated with double needle sample system operations and perform material balances on the constituents present in the simulated feeds. Table 4.3-1 shows a matrix of the required samples, the sample location, and volume of sample that will be collected during testing. Special instructions for sampling activities will be included in the PEWE Bench-Scale Testing Logbook.

#### 4.3.1 Sample Collection from Tank Drains

Samples will be collected from tank drains for comparison to double needle sampling system samples. This will enable direct comparison of the effects of the double needle sampler on the removal of organics.

- 4.3.1.1 Place a container below the feed tank drain valve (F-V-124).
- 4.3.1.2 Open the feed tank drain valve (F-V-124) and allow feed solution to flush the drain line, then close the feed tank drain vale (F-V-124).
- 4.3.1.3 Place an empty sample container below the feed tank drain valve.
- 4.3.1.4 Open the feed tank drain valve (F-V-124) and allow the solution to gravity flow into sample container.
- 4.3.1.5 When the sample container is full, close the drain valve.

- 4.3.1.6 Put the lid on the sample container, verify zero headspace, and place the sample container in the required shipping device for shipment to the analytical laboratory.

**Table 4.3-1: Double Needle Sampler System Sampling Requirements**

Sample ID No.	Description	Analytes	Volume
Feed Tank Sample			
05-FT-01	Direct Feed Tank Sample	Target	120 ml
05-FT-02	Direct Feed Tank Sample	Target	120 ml
05-FT-03	Direct Feed Tank Sample	Target	120 ml
05-FT-04	Direct Feed Tank Sample	Target	120 ml
05-FT-05	Direct Feed Tank Sample	Target	120 ml
05-FT-06	Direct Feed Tank Sample	Target	120 ml
05-FT-07	Direct Feed Tank Sample	Target	120 ml
05-FT-08	Direct Feed Tank Sample	Target	120 ml
05-FT-09	Direct Feed Tank Sample	Target	120 ml
05-FT-10	Direct Feed Tank Sample	Target	120 ml
Double Needle Samples			
05-DN-11	Double Needle Sample	Target	120 ml
05-DN-12	Double Needle Sample	Target	120 ml
05-DN-13	Double Needle Sample	Target	120 ml
05-DN-14	Double Needle Sample	Target	120 ml
05-DN-15	Double Needle Sample	Target	120 ml
05-DN-16	Double Needle Sample	Target	120 ml
05-DN-17	Double Needle Sample	Target	120 ml
05-DN-18	Double Needle Sample	Target	120 ml
05-DN-19	Double Needle Sample	Target	120 ml
05-DN-20	Double Needle Sample	Target	120 ml
Field Blank Sample			
05-QC-21	Distilled Water Field Blank	TOC	80 ml
05-QC-22	Distilled Water Trip Blank	TOC	80 mL
05-QC-23	Cross Contamination Sample	TOC	80 ml

#### 4.3.2 Sample Station Sample Collection

A sample station has been designed and fabricated to model existing PEWE sampling capabilities. Operation of the sampling station will be similar to PEWE sampling procedures. The sample station is equipped with a double hypodermic needle sampler and a larger sample receiver. An air jet is used to draw the sample from the respective tank through the sample station. The feed, bottoms, and condensate tanks can be sampled using either the hypodermic needle sampler or the sample receiver.

Sampling will occur after the feed tank has been thoroughly mixed using a mechanical mixer. The feed tank and the hypodermic needle sampler will be used exclusively for this test series. Table 4.3-1 shows the required number of samples and the sample volume.

- 4.3.2.1 Verify the sample receiver drain (SS-V-108) and vent valves (SS-V-107) are closed.
- 4.3.2.2 Attach a sample bottle to the hypodermic sample assembly. Align the long hypodermic needle next to the sample bottle cap's edge with the short hypodermic needle as near the center of the septum as possible.
- 4.3.2.3 Push the sample bottle up (with hand positioned on the bottom half of the sample bottle) until it is firmly seated against the needle block gasket.
- 4.3.2.4 Position the sample station three-way valves (SS-V-109 and SS-V-110) to route sample solution through the hypodermic sampler.
- 4.3.2.5 Open the sample supply and sample return isolation valves from the tank to be sampled as shown in the table below.

**Note: Feed tank valves (SS-V-112A and SS-V-112B) will be used exclusively for this test series.**

**Table 4.3-2: Sample Station Valve Line-up**

Open Supply Valve	Tank To Be Sampled	Open Return Valve
SS-V-112A	Feed Tank	SS-V-112B
SS-V-113A	Condensate Tank	SS-V-113B
SS-V-111A	Bottoms Tank	SS-V-111B

- 4.3.2.6 Adjust the air flow to the sample station airlift to about 0.5 scfh.
- 4.3.2.7 Initiate airflow to the sample station air jet to start circulating solution through the sample station by throttling valve AIR-V-106. Record the air supply pressure and air flow rate to the air jet. The air flow through the jet should be between 5 and 20 scfh.
- 4.3.2.8 Allow the sample to recirculate through the sample station for 10 minutes. If multiple samples are required from the same tank, circulate solution for each subsequent sample through the sample station for 5 minutes.
- 4.3.2.9 After the sample has circulated for the specified period of time, shut off the air supply to the air jet by closing valve AIR-V-106.
- 4.3.2.10 Shut off the air supply to the sample station air lift.

4.3.2.11 Shut the respective sample station supply and return valves as shown in Table 4.3-2.

4.3.2.12 Disconnect the sample bottle from the sampling apparatus and install a new sample bottle (if required).

*Note: When removing the sample bottle from the hypodermic sampler, lower the sample bottle so that both needles are in the headspace above the liquid. Wait 30 – 60 seconds (until the needles stop dripping). Quickly pull the sample bottle straight down and away from the needles.*

4.3.2.13 If more sample volume is required, repeat steps 4.3.2.1 through 4.3.2.9 as necessary.

4.3.2.14 Upon completing sampling activities for each tank, drain the sample lines and sample station equipment into a collection vessel.

4.3.2.15 Open the water supply (SS-V-114A) and return (SS-V-114B) isolation valves to the sample station and thoroughly rinse the sample station lines. Collect the water in a collection vessel.

4.3.2.16 Close the water supply (SS-V-114A) and return (SS-V-114B) isolation valves to the sample station and drain the remaining water from the sample station.

4.3.2.17 Transfer the solution collected from the sampling operations into the VOA sample vials to be supplied to the analytical laboratory and verify that zero headspace is present in the sample vials.

#### **4.4 System Cleaning**

The feed tank and sampling system will require cleaning prior to Test 5 to prevent potential cross contamination of trace concentration of organic residues from previous testing. Water, dilute nitric acid, and/or steam will be employed to clean the evaporator system. The system will be cleaned after all sampling activities associated with the operation have been completed.

4.4.1 Verify that all tanks have been completely drained of test solutions.

4.4.2 Supply steam to the evaporator vessel, to steam clean the evaporator system, by opening the steam supply valve to the evaporator. Turn on the steam generator and allow steam to circulate through the evaporator and associated vessels for approximately 30 minutes.

4.4.3 Charge the feed tank and evaporator vessel with water or dilute nitric acid solution.

- 4.4.4 Heat and circulate the solution through the evaporator by following the evaporator startup instructions.
- 4.4.5 After cleaning the evaporator, drain the rinse solution remaining in the evaporator vessel to the bottoms collection vessel.
- 4.4.6 Circulate the rinsate through the sample lines and the sample station as described in the sample collection instructions.
- 4.4.7 Drain all solutions from the evaporator and associated vessels. A sample of the solution will be obtained to verify that no cross contamination of organics has occurred.

#### **4.5 Health and Safety Requirements**

Hazardous chemicals will be used during bench-scale system tests and will include nitric acid and organic species. Personnel protective equipment will be provided to mitigate potential exposure to these substances.

When handling acidic solutions, personnel shall wear neoprene gloves and a face shield. In addition, Draeger sample tubes will be used to monitor the presence of nitric acid vapors in the operating area.

When handling dry chemicals, all measurement of chemicals shall be conducted in a ventilated area. The dry chemicals will then be added to the liquid feed makeup vessel. Gloves shall be worn during all dry chemical handling operations.

When handling organic species, perform all liquid measurements in a ventilated area. The organic species will then be added to the liquid feed makeup vessel. Gloves shall be worn during all organic species handling operations. Once the feed solution has been added to the feed tank, maintain the evaporator system at slightly negative conditions to prevent organic vapors from entering the operating area. Additionally, Draeger sample tubes will be used to monitor the for the presence of organic vapors in the operating area.

The evaporator system will contain surfaces that are maintained at elevated temperatures. Heated surfaces shall be insulated to protect personnel during evaporator operation. Signs will be posted to notify personnel of potentially hot surfaces.

#### **4.6 Waste Management**

The double needle sampler test series will require generation of simulated INTEC waste streams. The total volume of simulated waste solutions and rinse waters will not exceed 50 gallons. Simulated solutions from double needle sampler testing will remain in the feed for use during a Paint Filter Leach Test (PFLT) of the PEWE system off-gas. The

PFLT will be performed after completion of the double needle sampler test series. Waste management and disposal is detailed in the test plan for the PFLT.

## 5.0 SAMPLE AND ANALYSIS RATIONALE

The primary objective of the double needle sampler test series is to quantify organic partitioning which results from INTEC sample collection and handling procedures. As such, the sampling system operation and sampling strategy has been designed to simulate INTEC operations. Prior to previous tests, it was postulated that organic partitioning results from the following operational/sampling activities:

- air sparging of evaporator feed, condensate and bottoms tanks,
- steam jet transfer of liquid waste to PEWE feed tanks,
- air jet transfer and hypodermic needle sample collection,
- sample transfer from sample collection bottles to no-headspace organic vials, and
- >4° C temporary storage following sample collection.

These tests (Tests 1 through 4) demonstrated that the effects of air sparging and simulated steam jetting on spiked organic concentrations was significantly greater than the effects of air jetted sample collection and sample handling. After air sparging and steam jetting of feed solutions, approximately 75 percent of the spiked VOCs were reacted, decomposed, or volatilized and were no longer present in the feed solution.

This test has been designed to verify the effects of operation of the double needle sampling system only. As such, air sparge and steam jet operations will not be conducted.

### 5.1 Data Quality Objectives

The US Environmental Protection Agency (EPA) developed the Data Quality Objectives (DQOs) process as a system-based tool to support decision making which often follows characterization activities. Although the double needle sampler testing does not affect remedial or corrective actions, elements of the DQO process are helpful in clearly defining and bounding characterization, ultimately resulting in defensible data. The following subsections address relevant DQO issues.

#### 5.1.1 Problem Statement

Quantification of volatile organic losses due to operation of double needle sampling systems is unachievable at INTEC due to high radioactivity and sampling/analysis logistics. PEWE bottoms are transferred to the tank farm, and PEWE condensate serves as feed to the LET&D. These systems are subject to regulatory scrutiny; bench-scale sampling and analysis will contribute to the INTEC characterization database to be used for permitting and to demonstrate permit compliance.



### 5.1.2 Data Users

Double needle sampling system data will be used to support permitting activities conducted by BBWI and DOE-ID for the State of Idaho. Data may also be used by INTEC personnel to better understand the fate of organics due to double needle sampler operations.

### 5.1.3 Study Boundaries and Decisions

All reasonable efforts have been made to simulate INTEC operations and sampling protocols during sampler system testing. Target analytes (Table 5.2-1 below) were determined by evaluating PEWE feed data and selecting representative spike constituents. Spiking concentrations were bounded by analytical quantitation limits. Nitric acid and inorganic feedstock molarity was determined by evaluating and simulating PEWE feed.

Data generated by this test is anticipated to verify/validate that current INTEC sampling methods (i.e. double needle sampler operation) do not result in significant losses of volatile organic species. This data will be used to supplement INTEC process knowledge and ongoing RCRA sampling and analysis in generating a characterization database which will be used for operational permitting and to demonstrate permit compliance.

## 5.2 Target Analytes

Table 5.2-1 below lists test target analytes. Target analytes were chosen to best represent expected PEWE feed constituents with regard to vapor pressures. TOC analysis will be used for trip blanks and detection of potential cross contamination.

**Table 5.2-1: Target Analytes**

<b>Volatile Organics</b>	<b>Other Analysis</b>
1,1,1-trichloroethane	Total Organic Carbon (TOC)
toluene	
carbon disulfide	

## 5.3 Organic Spiking Concentrations

Whereas spiking concentrations of inorganics is predicated upon simulating INTEC liquid waste feed molarities and specific gravity, organic spiking concentrations are dependent upon test objectives and analytical detection limits. Based on previous tests, detection limits are expected to vary between 1 and 10 mg/l. To help ensure detection in all samples, organic spiking concentrations will be 10 times expected detection, or 100

mg/l. The volume of organics to be added to a 44 liter feedstock is provided in table 5.3-1 below.

**Table 5.3-1: Organic Volumes per 46 Liter Feedstock**

Spiked Organic	Volume
1,1,1-Trichloroethane	3.48 ml
Carbon disulfide	5.08 ml
Toluene	3.29 ml

#### **5.4 Quality Control**

Two quality control (QC) samples will be collected. The first quality control sample is a trip blank to monitor for organic cross contamination that may occur during sample shipping. The second QC sample will be collected following pre-cleaning to monitor for cross contamination.

The analytical laboratory maintains and adheres to a quality control manual that can be provided upon request.

### **6.0 REPORTING**

A detailed operations/sample logbook will be maintained. All operational observations and any deviations from procedure will be recorded. All pertinent sample collection information (date of sample, quantity of sample, observations, etc.) will be recorded. Following the receipt and evaluation of analytical data, a comprehensive final report will be generated which will include:

- operations and sampling description,
- data reduction spreadsheet,
- organic partitioning calculations,
- statistical analysis of sampling data,
- conclusions,
- additional study recommendations, if any,
- copy of logbook, and
- Attachment 1 data sheets.

## 7.0 REFERENCES

1. Radian Corporation, *Results of a Laboratory Study to Assess the Fate of Organic Compounds in Synthetic ICPP Tank Farm Waste*, July 17, 1995.
2. Science Applications International Corporation, *PEWE and LDUA Test Series, Organics Partitioning Comprehensive Final Report*, Revision 0, June 2000.

**Attachment A**

**Double Needle Sampler  
Data Sheets**



## **ATTACHMENT 2**

### **Double Needle Sampler – Logbook Entries and Data Sheets**

**Double Needle Sampler Data Sheets**  
Data to be Taken prior to and During Sampling

DATE: 09 July, 2001

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Time	System Pressure (In. WC)	Feed Tank Level (L)	Feed Tank Temp (°F)	Primary Cond. Exit Temp (°C)	Cooling Water Supply Temp (°F)	Primary Supply Temp (°F)	Cooling Water Exit Temp (°F)	Cooling Water Flow	Sampling Operations Readings		
									Sample ID	Air Jet Flow	Air Pressure
	E-PI-1	F-SG-101	E-TC-10	E-TC-9	E-TE-11	E-TE-10	E-TE-13	E-FI-13	AIR-FI-106	PSI	AIR-FI-116
0410	ADD	0706	77	76	OFF	75	OFF	OFF			
0436	0	44	77	77	NA	74	NA	NA			
0519	0	44	76	77	NA	74	NA	NA			
0819	-3"	44	75	73	71	73	71	10.0m			
0858	-3"	44	74	74	70	73	70	10.0m			
0900	-3"	43	74	71	74	74	76	10			
0929											
0930											
0933	-2"	43									
0935	-2"										
0938	+2"										
0940	-2"	43	71								
0945	-2"										
0951	-2"										
0954	+2"										
0956	-2"										
0958	+2"										
1001	-2"	42	69								
1006	-2"	42	69	68	74			10			
1009	+2"										
1011											

PEWE Bench-Scale Test 5: Double Needle Sampler Statistical Testing, June 25, 2001

**Double Needle Sampler Data Sheets**  
Data to be Taken prior to and During Sampling

PAGE 2/

DATE: July 9, 2001

Time	System Pressure (In. WC)	Feed Tank Level (L)	Feed Tank Temp (°F)	Primary Cond. Exit Temp. (°C)	Cooling Water Supply Temp. (°F)	Primary Cond. Supply Temp. (°F)	Cooling Water Exit Temp. (°F)	Sample ID	Sampling Operations Readings		Air Lift SCFM Flow
									AIR-FI-106 Flow	AIR Pressure	
1012	2"	42	68					05DN13A	5.5	90	20.2
1017	2"							"	5.5	94	20.2
1022	3"	42	67					05DN13B	5.5	100	20.2
1025	3"							"	5.5	105	20.2
1027	3"	42	67					05DN13C	5.5	105	20.2
1030	3"							"	5.5	95	20.2
1032	3"	42	67					05DN13X	5.5	95	20.2
1035	4"							"	5.5	80	20.2
1037	4"	42	67					05FT03	5.5	75	20.2
1038	3"	42	67					05DN14A	5.5	85	20.2
1040	3"	41	66					05DN14B	5.5	95	20.2
1050	3"							"	5.5	100	20.2
1053	3"	41	66					05DN14C	5.5	100	20.2
1058	3"	41	66					05DN14X	5.5	105	20.2
1103								05FT04			
1105	4"	41	66					05DN15A	5.5	82	20.2
1110	4"	41	65					"	5.5	85	20.2
1115	3"	41	65					05DN15B	5.5	90	20.2
1120	3"	40	65					05DN15C	5.5	100	20.2
1125	3"	40	65					05DN15X	5.5	100	20.2
1130	3"	40	65					05FT05	5.5	95	20.2

PEWE Bench-Scale Test 5: Double Needle Sampler Statistical Testing, June 25, 2001



**Double Needle Sampler Data Sheets**  
Data to be Taken prior to and During Sampling

DATE: 7/9/01

PAGE 3/

Time	System Pressure (In. WC)	Feed Tank Level (L)	Feed Tank Temp (°F)	Primary Cond. Exit Temp. (°C)	Cooling Water Supply Temp. (°F)	Primary Cond. Supply Temp. (°F)	Cooling Water Exit Temp. (°F)	Cooling Water Flow	Sampling Operations Readings			
									Sample ID	Air Jet Flow	Air Pressure	Air Lift SCFH Flow
	E-PI-1	F-SG-101	E-TC-10	E-TC-9	E-TE-11	E-TE-12	E-TE-13	E-FI-13	AIR-FI-106			AIR-FI-116
1131	3"	40	65						5.0	80		<0.2
1138	3"	40	65						5.5	85		<0.2
1141	3"	40	64						5.5	95		<0.2
1146	3"	40	64						5.5	100		<0.2
1151	3"	40	64						5.5	100		<0.2
1156	4"	40	64						5.5	85		<0.2
1157	4"	40	64						5.0	80		<0.2
1202	4"	40	64						5.0	85		<0.2
1207	3	40	64						5.5	95		<0.2
1212	3	40	64						5.5	100		<0.2
1217	2	40	64						5.5	100		<0.2
1222												
1223	3	39	64						5.5	85		<0.2
1228	3	39	64						5.0	80		
1232	3	39	64						5.5	85		
1238	3	39	64						5.5	90		<0.2
1240	3	39	64						5.5	95		<0.2
1243	3	39	64						5.5	95		<0.2
1248	3	39	63						5.5	100		<0.2
1249	3	39	63						5.5	100		<0.2
1254	3	39	63						5.5	80		<0.2
1259	3	39	63						5.5	85		<0.2
1304	3	39	63						5.5	80		<0.2



JULY 6 2001

1230 Added 10 L of water to the feed tank and initiated air jet recirculation through the double needle sampler. This will be the cross contamination sample. -TDT

1245 Beginning Feed makeup. Cleaned Feed makeup vessel. Chemical measured and added to feed are listed below. -TDT  
 Water:  $2\frac{1}{2}$  L<sup>TDT</sup>  
 Calcium Nitrate: 43.06 grams [ $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ]  
 Iron Nitrate: 2.64 grams [ $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ]  
 Sodium Nitrate: 623 grams [ $\text{NaNO}_3$ ]  
 NITRIC ACID: 3.35 Liters [ $\text{HNO}_3$  - 69%]  
 Aluminum Nitrate: 4.0 Liters [2.2M  $\text{Al}(\text{NO}_3)_3$  solution]

1315 Stopped air jet recirculation of the water in the feed tank. -TDT

1340 Obtained cross-contamination sample  
 REMOVE 05-OC-23A and -23B for TOC analysis and placed in laboratory fridge -TDT

1400 Adding Feed solution to the feed tank.  
 Added water to feed tank to generate 44 liters of feed.

NOT  
 9.48  
 8.93  
 9.50  
 7/09/2001

0410 Spiking the feed with organics.  
 Carbon disulphide: 3.5 ml  $\rightarrow$   
 Toluene: 5.1 ml  $\rightarrow$   
 1,1,1-TCA: 3.3 ml  $\rightarrow$

3  
 20X (20 mm DIA) 0420 Organics added to feed and mixed in @ 25 percent -TDT

VERTASE 0800 prepared field blank (05-OC-21A and -21B) and trip blank (05-OC-22A and -22B)  
 DI water samples -TDT

SAMPLES



 End of page

JULY 9, 2001

0820	TURNED ON COOLING water and Exhaust fan. System pressure is @ -3" WC. -TDT	JULY 9, 2001	1027	Switch
0900	Mixer off. Mix time = 4 hr 40 minutes. Began pulling direct sample 05-FT-01 series (A, B, 3C). -TDT		1032	Switch
0905	LOTS OF ORGANIC GLOBBLES WERE Apparent in the first direct samples. The samples were added back to the tank. Test Plan deviation - we turned on the air sparge. Rotameter setting is at 20. Pressure is 90 psig. Will sample again @ 9:30 - TDT Drill mixer ON 70		1037	Comple
0920	STARTED AIR JET Recirculation for Sample 05- <del>FT</del> <sup>DN</sup> -11 (A, B, 3C) We will pull the direct sample after the jetted sample. -TDT		1038	Comple
0930	Switched bottles on the Double Needle Sample -TDT		1048	Switch
0935	Switched bottles to 05-DN-11C on Needle Sample -TDT		1053	Switch
0940	Switched bottles to 05-DN-11X on Needle Sample -TDT		1058	Switch
0945	Completed sample set 05-DN-11 -TDT		1103	Comple
0946	Started recirculating sample set 05-DN-12A and completed pulling samples 05-FT-01A - 01B and 01C -TDT			05-DI
0956	Switched sample bottles to 05- <del>12</del> <sup>DN</sup> -12B on needle system - TDT		1104	TURN:
1001	Switched sample bottles to 05-DN-12C on sampler -TDT		1105	COMPL
1006	Switched sample bottles to 05-DN-12X on sampler -TDT			FT
1011	Completed sampling set 05-DN-12 -TDT			05-DN
1012	Completed putting feed tank samples 05-FT-02 and started needle samples 05- <del>FT</del> <sup>DN</sup> -03A -TDT		1115	Switch
1022	Switched sample bottles to 05-DN-13B on the sampler system -TDT		1120	Switch
			1125	Switch
			1130	Complete
				series
			1131	Compl
				series
				on the
			1141	Switch
			1146	Switch
			1151	Switch
			1156	Complete
				series
			1157	Comple
				series
				on the

JULY 9, 2001	
and Exhaust 2-3" WC. -TDT 5 minutes. 05-FT-01	1027 Switched sample bottles to 05-DN-13C on the sampling system -TDT
	1032 Switched sample bottles to 05-DN-13X on the sampling system -TDT
WERE	1037 Completed Double Needle Sample Set 05-DN-13 -TDT
samples.	1038 Completed Feed tank sample set 05-FT-03 and Started Double Needle Sampleset 05-DN-14A -TDT
nk to the	1048 Switched sample bottles to 05-DN-14B -TDT
- WC	1053 Switched sample bottles to 05-DN-14C -TDT
Rotameter	1058 Switched sample bottle to 05-DN-14X -TDT
is 90 psig.	1103 Completed sampling Double Needle Sample series 05-DN-14 -TDT
TDT Drill mixer on	1104 TURNED THE DRILL MIXER OFF -TDT
in	1105 COMPLETED SAMPLING Feed tank series <sup>FT</sup> <sup>TDT</sup> 05-DN-04 and initiated recirculation of 05-DN-15A -TDT
3C)	
ke after	
Needle Sample -TDT	1115 Switched sample bottles to 05-DN-15B -TDT
on Needle Sample -TDT	1120 Switched sample bottles to 05-DN-15C -TDT
Needle Sample -TDT	1125 Switched sample bottles to 05-DN-15X -TDT
-TDT	1130 Completed Sampling the Double Needle sampler series 05-DN-15 -TDT
05-DN-12A	
05-FT-01A	1131 Completed sampling the feed tank sample series 05-FT-05 and initiated recirculation on the Double Needle series 05-DN-16A -TDT
12B on needle	1141 Switched sample bottles to 05-DN-16B -TDT
-12C on sampler -TDT	1146 Switched sample bottles to 05-DN-16C -TDT
12X on sampler -TDT	1151 Switched Sample bottles to 05-DN-16X
2 -TDT	1156 Completed sampling the Double needle sampler series 05-DN-16 -TDT
ke	
samples	1157 Completed Sampling the feed tank sample series 05-FT-06 and initiated recirculation on the double needle sample 05-DN-17A -TDT
U-13B on	

JULY 9, 2001

1207	Switched Sample bottles to 05-DN-17 <sup>B</sup> -TDT	JULY 9, 2001	
1212	Switched Sample bottles to 05-DN-17C -TDT	1333	Measure
1217	Switched Sample bottles to 05-DN-17X -TDT		Needle
1222	Completed Sampling Double Needle sampler Series 05-DN-17 - TDT		Cylinder
1223	Completed Sampling <del>Series</del> <sup>TDT</sup> Feed tank series 05-FT-07 and began recirculating series 05-DN-18A -TDT	1400	Flow the s, Total :
1233	Switched Sample bottles to 05-DN-18B -TDT	1435	Prepar
1238	Switched Sample bottles to 05-DN-18C -TDT		Sampl.
1243	Switched Sample bottles to 05-DN-18X -TDT		with
1248	Completed Sampling Double Needle Sampler Series 05-DN-18 - TDT		around
1249	Completed Sampling Feed tank sample Series 05-FT-08 and began recirculating Double Needle Sample 05-DN-19A -TDT		
1259	Switched Sample bottles to 05-DN-19B -TDT		
1304	Switched Sample bottles to 05-DN-19C -TDT		
1309	Switched Sample bottles to 05-DN-19X -TDT		
1314	Completed Sampling Double Needle Sampler Series 05-DN-19 -TDT		
1315	Completed Sampling Feed tank Sample Series 05-FT-09 and began recirculating Double needle Sample 05-DN-20A -TDT		
1325	Switched sample bottles to 05-DN-20B -TDT		
1330	Switched sample bottles to 05-DN-20C -TDT		
1335	Switched sample bottles to 05-DN-20X -TDT		
1340	Completed Sampling Double Needle Sampler Series 05-DN-20 -TDT		
1341	Completed Sampling Feed tank Sample Series 05-FT-10 - TDT		

DN-17<sup>B</sup> -TDT  
DN-17C -TDT  
DN-17X -TDT  
Sampler

take series  
ring series

-18B -TDT  
-18C -TDT  
-18X -TDT  
Sampler

sample  
recirculating

19A -TDT  
DN-19B -TDT  
DN-19C -TDT  
DN-19X -TDT  
Sampler

Sample  
recirculating  
-20A -TDT  
-20B -TDT  
-20C -TDT  
-20X -TDT

Sampler  
Sample

JULY 9, 2001

1335 Measured the flow through the Double Needle sampler by placing a graduated cylinder beneath the sample system drain. Flow was measured at 270 ml/min through the system - TDT

1400 Total Samples # = 66 40ml bottles = 2.64 L -TDT

1435 Prepared shipping package for Hausser Lab. Samples are contained in plastic bags with six blocks of blue ice and vermiculite around all the samples -TDT

*MBL*

END OF PAGE  
Today Home

## **ATTACHMENT 3**

### **Double Needle Sampler – Analytical Report**



- Client:** Science Applications International Corporation  
545 Shoup Avenue  
Idaho Falls, ID 83402  
Attn: Troy Thomson
- Objective:** The client requested the services of Hauser, Inc., to quantify specified volatile organic compounds (VOC's) contained in twenty acidic, aqueous samples. The client also requested total organic carbon analysis (TOC) on three samples. The samples submitted were aqueous solutions containing 1.2 molar nitric acid. Samples were received in sixty-six separate containers. The VOC samples were received in triplicate and the TOC samples were received in duplicate. All samples were contained in 40mL, amber, VOA vials.
- Method:** Total Organic Carbon  
Samples were analyzed using a Seivers® 800 Total Organic Carbon analyzer. The samples were analyzed in duplicate and results are listed in the Results section of this report.
- Gas Chromatography / Mass Spectrometry – Purge and Trap  
Samples were analyzed for three VOC compounds: carbon disulfide, 1,1,1-trichloroethane, and toluene. A four point calibration curve for each analyte was analyzed in the range of 50 to 400 ng. An internal standard mix was added to each sample to monitor the responses of three internal standard compounds. The following instrumentation was utilized:
- Purge and trap unit (P&T) connected to a gas chromatograph (GC) / mass spectrometer (MS) system (Hewlett Packard® 5890 GC / 5972 MSD)  
GC/MS Instrument ID #: 202  
Purge and Trap Unit: Tekmar® 3000  
Analytical Method: VOASAI.C.M  
Column type: DB-624, 60 m, 0.25 mm i.d., 1.4 µm film thickness  
A detailed printout of the GC/MS method parameters listed in VOASAI.C.M is included in the data package.
- Samples:** A complete listing of all samples submitted for analysis is included in the Chain of Custody form contained in this data package.

#### **P&T/GC/MS Calibration:**

A calibration curve was prepared by adding various amounts standard mix (2425-108-1) containing target analytes to a Luerlock® syringe containing 5mL of Nanopure DI. Internal standard (IS) mix (2425-107-1) was also added to each standard. Standards were analyzed in ascending order of concentration. The response ratios of target analytes to the internal standard compounds were plotted in a linear regression for each analyte. The correlation coefficients for the responses of each analyte in the calibration curve were all >0.990. This was deemed acceptable for purposes of quantitation within the 50 to 400 ng concentration range. For daily instrument calibration, a 250 ng check standard was analyzed. The recovery of the check standard compounds was required to be within 10% of the nominal 250 ng concentration for each VOC analyte. All check standards met these criteria on a daily basis. Following check standards a daily instrument blank was analyzed to monitor for any target analyte contamination of the P&T/GC/MS system.

This report applies only to the sample, or samples, investigated and is not necessarily indicative of the quality or condition of apparently identical or similar products. As a mutual protection to clients, the public and these Laboratories, this report is submitted and accepted for the exclusive use of the client to whom it is addressed and upon the condition that it is not to be used, in whole or in part, in any advertising or publicity matter without prior written authorization from Hauser Laboratories. This report may be copied only in its entirety.

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• Ph: (800) 241-2322 • Ph: (303) 581-0079 • FAX: (303) 581-0195



**TOC Analysis Results:**

The following table lists the results of duplicate analyses on the TOC samples. Sample results are in parts per billion (ppb as ng/mL). Sample 05-QC-23 was diluted 1:50 due to high sample concentration. The total inorganic carbon level was high in sample 05-QC-23 which may limit the accuracy of its' result.

Sample Identification	"A" Result (ppb)	"B" Result (ppb)	Average (ppb)
05-QC-21	262	153	208
05-QC-22	168	154	161
05-QC-23	3,290	2,750	3,020

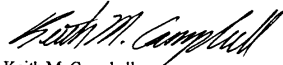
**P&T/GC/MS Sample Analysis Results:**

The qualitative and quantitative data gathered from P&T/GC/MS sample analysis are summarized on a spreadsheet included in this data package. All sample, sample duplicate, and check standard data is included on the attached spreadsheet. Quality control was ensured by performing a duplicate sample analysis once every ten samples. Additionally, a sample was analyzed as both a non-spiked sample and as a sample spiked with 100 ng of each analyte once every ten samples. The table below lists the amounts of each target analyte detected in the non-spiked and spiked samples and the percentage recovery of the spiked analytes. The limit of quantitation is 50 ng and the detection limit is 25 ng.

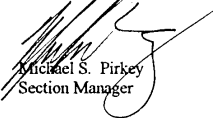
Sample ID	Sample Amount (µL)	Carbon disulfide (ng)	1,1,1-Trichloroethane (ng)	Toluene (ng)
05-FT-02A	100	73.6	82.1	67.9
05-FT-02A spike	100	170	187	163
% Recovery		96.4	105	95.1
05-FT-07A	1,000	75.2	68.7	Nd
05-FT-07A spike	1,000	142	150	102
% Recovery		66.8	81.3	102

nd = not detected

**REPORT PREPARED BY:**

  
Keith M. Campbell  
Chemist

**REPORT REVIEWED BY:**

  
Michael S. Pirkey  
Section Manager

Date: 7/11/01 - 7/17/01

VOC Target Analyte Results from Purge and Trap GC/MS Analysis

Sample ID	Sample Amount (uL)	Carbon disulfide (ng)	1,1,1-Trichloroethane (ng)	Toluene (ng)	Carbon disulfide (ng/uL)	1,1,1-Trichloroethane (ng/uL)	Toluene (ng/uL)
05-FT-01A	100	320	369	374	3.20	3.69	3.74
05-DN-11A	25	248	263	258	9.92	10.52	10.32
05-DN-11A dup	25	240	254	249	9.60	10.16	9.96
250ng check std.	na*	252	241	263	na	na	na
05-FT-02A	100	73.6	82.1	67.9	0.74	0.82	0.68
05-FT-02A spike	100	170	187	163	1.70	1.87	1.63
05-DN-12A	50	65.3	93.4	96.7	1.31	1.87	1.93
05-FT-03A	500	241	197	83.2	0.48	0.39	0.17
05-DN-13A	500	101	156	184	0.20	0.31	0.37
05-FT-04A	1000	226	176	52.3	0.23	0.18	0.05
05-DN-14A	1000	52.2	74.0	97.5	0.05	0.07	0.10
05-FT-05A	1000	137	104	<50	0.14	0.10	bloq**
05-DN-15A	1000	<50	<50	<50	bloq	bloq	bloq
05-FT-06A	1000	119	106	nd***	0.12	0.11	nd
250ng check std.	na	242	240	256	na	na	na
05-DN-16A	1000	nd	nd	nd	nd	nd	nd
05-DN-16A dup	1000	nd	nd	nd	nd	nd	nd
05-FT-07A	1000	75.2	68.7	nd	0.08	0.07	nd
05-FT-07A spike	1000	142	150	102	0.14	0.15	0.10
05-DN-17A	1000	nd	nd	nd	nd	nd	nd
05-FT-08A	1000	82.8	84.7	nd	nd	nd	nd
05-DN-18A	1000	nd	nd	nd	nd	nd	nd
05-FT-09A	1000	61.8	75.7	nd	0.06	0.08	nd
05-DN-19A	1000	nd	nd	nd	nd	nd	nd
05-FT-10A	1000	85.9	93.7	nd	0.09	0.09	nd
05-DN-20A	1000	nd	nd	nd	nd	nd	nd
05-FT-02B	200	97.3	120	127	0.49	0.60	0.64
05-FT-02B dup	200	100	126	130	0.50	0.63	0.65
05-DN-12B	200	148	223	251	0.74	1.12	1.26
250ng check std.	na	258	275	256	na	na	na

na\* = not applicable  
 bloq\*\* = below limit of quantitation (0.050 ng/uL)  
 nd\*\*\* = not detected  
 detection limit = 0.025 ng/uL

TOPLEVEL PARAMETERS

Method Information For: C:\HPCHEM\1\METHODS\VOASAIC.M

Method Sections To Run:

- ( ) Save Copy of Method With Data
- ( ) Pre-Run Cmd/Macro =
- (X) Data Acquisition
- (X) Data Analysis
- ( ) Post-Run Cmd/Macro =

Method Comments:

Purge and Trap GC/MS method for identification of unknowns: J&W 60m DB-624, 0.25mm i.d., 1.4um film.

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC  
Injection Source: Manual  
Injection Location: Rear  
Mass Spectrometer: Enabled

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	250 C
Detector A:	Off	50 C
Detector B:	On	250 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.25 minutes
Oven Max:	260 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Oven Program:

Initial Temperature:	40 C
Initial Time:	1.00 minutes

	Rate	Final	Final
Method: VOASAIC.M		Wed Jul 11 13:27:11 2001	

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TOPLEVEL PARAMETERS

Method Information For: C:\HPCHEM\1\METHODS\VOASAIC.M

Method Sections To Run:

- ( ) Save Copy of Method With Data
- ( ) Pre-Run Cmd/Macro =
- (X) Data Acquisition
- (X) Data Analysis
- ( ) Post-Run Cmd/Macro =

Method Comments:

Purge and Trap GC/MS method for identification of unknowns: J&W 60m DB-624, 0.25mm i.d., 1.4um film.

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC  
Injection Source: Manual  
Injection Location: Rear  
Mass Spectrometer: Enabled

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	250 C
Detector A:	Off	50 C
Detector B:	On	250 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.25 minutes
Oven Max:	260 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Oven Program:

Initial Temperature:	40 C
Initial Time:	1.00 minutes

	Rate	Final	Final
Method: VOASAIC.M		Wed Jul 11 13:27:11 2001	

Page: 1

Level	(C/minute)	Temperature (C)	Time (minutes)
1	10.0	250	8.00
2(A)	0.0	0	0.00
3(B)	0.0	0	0.00
Next Run Time:		30.00 minutes	

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

Constant Flow: Off  
 Constant Flow Pressure: 0.0 psi  
 Constant Flow Temperature: 50 C  
 Initial Pressure: 0.0 psi  
 Initial Time: 650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00
Total Program Time:		650.00 minutes	

Column Length: 30.00 m  
 Column Diameter: 0.530 mm  
 Gas: He  
 Vacuum Compensation: Off

Inlet B:

Constant Flow: Off  
 Constant Flow Pressure: 20.0 psi  
 Constant Flow Temperature: 50 C  
 Initial Pressure: 17.0 psi  
 Initial Time: 480.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00
Total Program Time:		480.00 minutes	

Column Length: 60.00 m  
 Column Diameter: 0.250 mm  
 Gas: He  
 Vacuum Compensation: On

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	Off	0.20	0.00	No
B	On	0.00	20.00	No

HP5890 Valve and Relay Information

Initial Setpoints:

5890 Valves:	Valve 1: Off	Valve 2: Off	Valve 3: Off	Valve 4: On
19405 Valves:	Valve 5: Off	Valve 6: Off	Valve 7: Off	Valve 8: Off
19405 Relays:	Relay 1: Off	Relay 2: Off	Relay 3: Off	Relay 4: Off

HP5890 Detector Information

Detector	Type	State
A	FID	Off
B	---	Off

HP5890 Signal Information

Not saving signal data.

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
1	Testplot	0.053	5.000	0.00	1.00
2	Testplot	0.053	5.000	0.00	650.00

MS ACQUISITION PARAMETERS

General Information

-----  
Tune File : atune.u  
Acquisition Mode : Scan

MS Information

--  
Solvent Delay : 4.00 min  
EM Absolute : False  
EM Offset : 106  
Resulting EM Voltage : 2247.1

[Scan Parameters]

Low Mass : 35  
High Mass : 500

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Threshold : 150  
Sample # : 1 A/D Samples 2

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS  
-----

DATA ANALYSIS PARAMETERS  
-----

Method Name: C:\HPCHEM\1\METHODS\VOASAIC.M

Percent Report Settings  
-----

Sort By: Signal

Output Destination

Screen: No  
Printer: Yes  
File: No

Integration Events: AutoIntegrate

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings  
-----

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
nist98.L	0

Integration Events: voapurge.e

Report Type: Summary

Output Destination

Screen: No  
Printer: Yes  
File: No

Generate Report During Run Method: No

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Quantitative Report Settings

Report Type: Detailed

Output Destination

Screen: No  
Printer: Yes  
File: No

Generate Report During Run Method: Yes

Purge and Trap Analysis

Calibration Last Updated: Wed Jul 11 10:35:22 2001

Reference Window: 2.00 Minutes  
Non-Reference Window: 1.00 Minutes  
Correlation Window: 0.10 minutes  
Default Multiplier: 1.00  
Default Sample Concentration: 0.00

Compound Information

-----  
1) Bromochloromethane (ISTD TR)

Ret. Time 9.93 min., Extract & Integrate from 9.43 to 10.43 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 129.75			voasaic.e
Q1 127.75	73.80	20.0	voasaic.e
Q2 131.80	23.00	20.0	voasaic.e

Lvl ID	Conc (ng)	Response
1	250.000	305065
2	250.000	272546
3	250.000	300138
4	250.000	249603

Qualifier Peak Analysis ON ISTD conc: 250.000 ng  
Curve Fit: Linear

-----  
2) Carbon disulfide (TR)

Ret. Time 7.68 min., Extract & Integrate from 7.18 to 8.18 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 75.90			voasaic.e
Q1 43.95	18.10	20.0	voasaic.e
Q2 77.90	8.20	20.0	voasaic.e

Lvl ID	Conc (ng)	Response
--------	-----------	----------

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1	50.000	355811
2	100.000	693592
3	200.000	1502872
4	400.000	2659589

Qualifier Peak Analysis ON  
Curve Fit: Linear

3) 1,1,1-Trichloroethane ( TR)

Ret. Time 10.32 min., Extract & Integrate from 9.82 to 10.82 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 96.85			voasaic.e
Q1 98.85	66.20	20.0	voasaic.e
Q2 61.00	50.40	20.0	voasaic.e

Lvl ID	Conc (ng)	Response
1	50.000	167587
2	100.000	356325
3	200.000	763874
4	400.000	1396727

Qualifier Peak Analysis ON  
Curve Fit: Linear

4) Carbon tetrachloride ( TR)

Ret. Time 10.57 min., Extract & Integrate from 10.07 to 11.07 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 116.85			voasaic.e
Q1 118.75	93.90	20.0	voasaic.e
Q2 120.75	32.10	20.0	voasaic.e

Lvl ID	Conc (ng)	Response
1	50.000	144957
2	100.000	305283
3	200.000	649195
4	400.000	1214606

Qualifier Peak Analysis ON  
Curve Fit: Linear

5) 1,4-Difluorobenzene (ISTD TR)

Ret. Time 11.22 min., Extract & Integrate from 10.72 to 11.72 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 113.95			voasaic.e
Q1 63.00	28.10	20.0	voasaic.e
Q2 87.90	20.10	20.0	voasaic.e

Lvl ID	Conc (ng)	Response
1	250.000	1365437
2	250.000	1233069
3	250.000	1266519

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4 250.000 1138585

Qualifier Peak Analysis ON ISTD conc: 250.000 ng  
Curve Fit: Linear

6) Toluene (TR)

Ret. Time 13.50 min., Extract & Integrate from 13.00 to 14.00 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 91.00			voasaic.e
Q1 92.00	57.40	20.0	voasaic.e
Q2 65.00	15.10	20.0	voasaic.e

Lvl ID	Conc (ng)	Response
1	50.000	363135
2	100.000	708934
3	200.000	1499019
4	400.000	2664921

Qualifier Peak Analysis ON  
Curve Fit: Linear

7) Tetrachloroethene (TR)

Ret. Time 14.37 min., Extract & Integrate from 13.87 to 14.87 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 165.70			voasaic.e
Q1 163.70	83.90	20.0	voasaic.e
Q2 167.75	48.60	20.0	voasaic.e

Lvl ID	Conc (ng)	Response
1	50.000	59041
2	100.000	113686
3	200.000	237048
4	400.000	420546

Qualifier Peak Analysis ON  
Curve Fit: Linear

8) Chlorobenzene-d5 (ISTD)

Ret. Time 15.66 min., Extract & Integrate from 15.16 to 16.16 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 116.95			voasaic.e
Q1 82.00	65.00	20.0	voasaic.e
Q2 118.95	31.30	20.0	voasaic.e

Lvl ID	Conc (ng)	Response
1	250.000	1139095
2	250.000	1029566
3	250.000	1101464
4	250.000	966717

Qualifier Peak Analysis ON ISTD conc: 250.000 ng

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---

Curve Fit: Linear

---

END OF DATA ANALYSIS PARAMETERS

---

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July 9, 2001  
 Project Contact: Troy Thomson  
 (208)528-2145  
 (208)524-4750

HAUSER LABORATORY, UHIII

Test 5 -- Double Needle Sampler

Sample Matrix	Sample Identifier	no. of containers	Target VOC Analytes	TOC	Additional Comments
Aqueous pH-7	05-QC-21 (A,B)	2		X	
Aqueous pH-7	05-QC-22 (A,B)	2		X	
Aqueous pH-7	05-QC-23 (A,B)	2		X	
Aqueous pH-2.1	05-FT-01 (A,B,C)	3	X		ACID CONCENTRATION = ~1.2 M HNO <sub>3</sub>
Aqueous pH-2.1	05-DN-11 (A,B,C)	3	X		
Aqueous pH-2.1	05-FT-02 (A,B,C)	3	X		
Aqueous pH-2.1	05-DN-12 (A,B,C)	3	X		
Aqueous pH-2.1	05-FT-03 (A,B,C)	3	X		
Aqueous pH-2.1	05-FT-13 (A,B,C)	3	X		
Aqueous pH-2.1	05-FT-04 (A,B,C)	3	X		
Aqueous pH-2.1	05-DN-14 (A,B,C)	3	X		
Aqueous pH-2.1	05-FT-05 (A,B,C)	3	X		
Aqueous pH-2.1	05-DN-15 (A,B,C)	3	X		

7/11 →  
 7/12 →

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 Hauser Laboratory

Relinquished by: Troy Thomson  
 SAIC

Page 1 of 2

TARGET VOC Analytes: Carbon disulfide  
 1,1,1-Trichloroethane  
 Toluene

COPY

July 9, 2001  
 Project Contact: Troy Thomson  
 (208)528-2145  
 (208)524-4750

HAUSER LABORATORY # 01111

Test 5 -- Double Needle Sampler

Sample Matrix	Sample Identifier	no. of containers	Target VOC Analytes	TOC	Additional Comments	
Aqueous pH- <1	05-FT-06 (A,B,C)	3	X		ACID CONCENTRATION = ~1.2 M HNO <sub>3</sub>	
Aqueous pH- <1	05-DN-16 (A,B,C)	3	X			
Aqueous pH- <1	05-FT-07 (A,B,C)	3	X			
Aqueous pH- <1	05-DN-17 (A,B,C)	3	X			
Aqueous pH- <1	05-DN-08 (A,B,C)	3	X			
Aqueous pH- <1	05-DN-18 (A,B,C)	3	X			
Aqueous pH- <1	05-FT-09 (A,B,C)	3	X			
Aqueous pH- <1	05-DN-19 (A,B,C)	3	X			
Aqueous pH- <1	05-FT-10 (A,B,C)	3	X			
Aqueous pH- <1	05-DN-20 (A,B,C)	3	X			
Aqueous pH-						
Aqueous pH-						
Aqueous pH-						
Aqueous pH-						
TOTAL CONTAINERS = 66						

7/12  
 7/13  
 →

Received by:  
 Hauser Laboratory

Relinquished by: Troy Thomson  
 SAIC

Target Analytes = Carbon disulfide  
 1,1,1-Trichloroethane  
 Toluene

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## **ATTACHMENT 4**

### **Double Needle Sampler – Photos of Process Equipment**

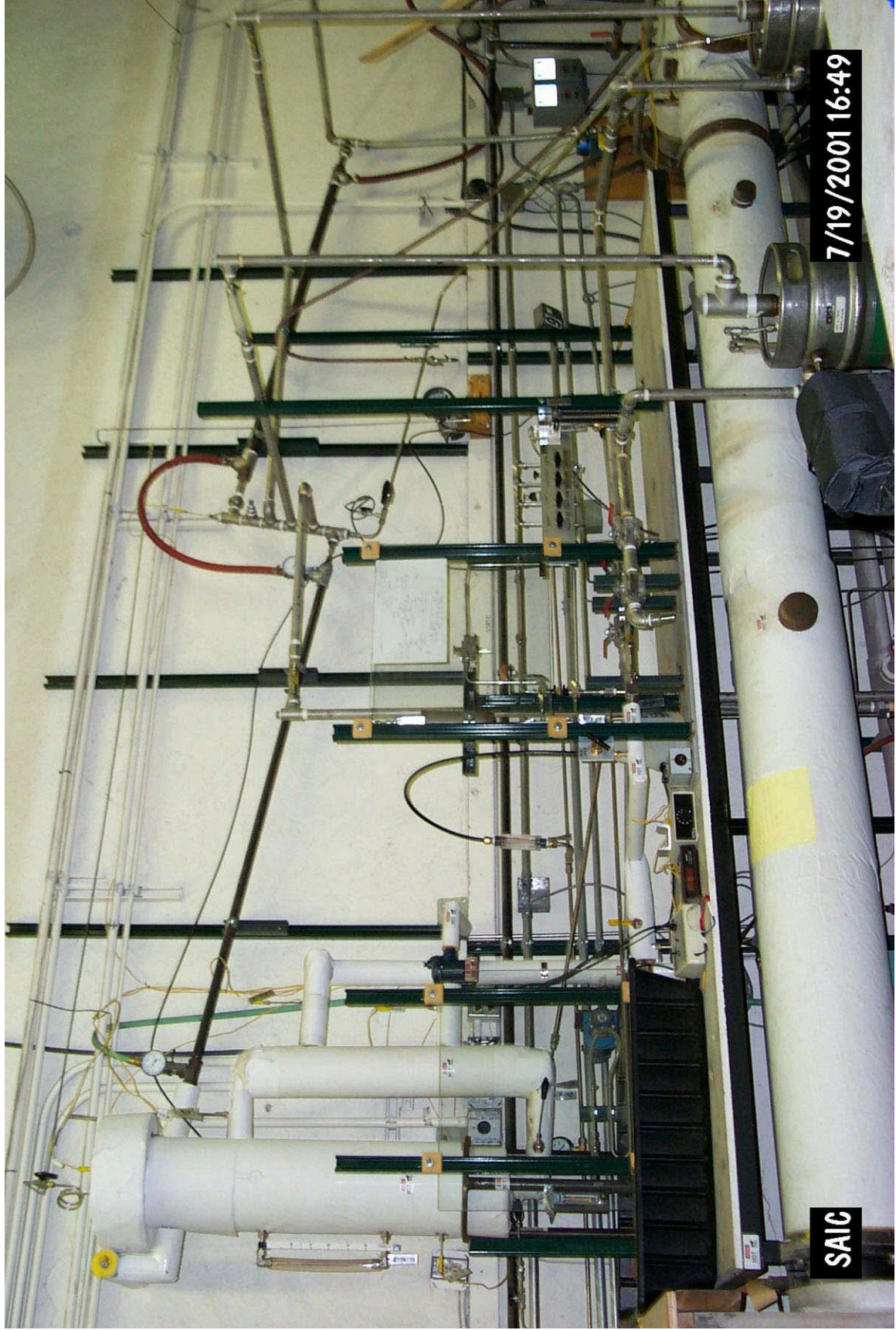


Figure D-1: Bench-scale evaporator system.



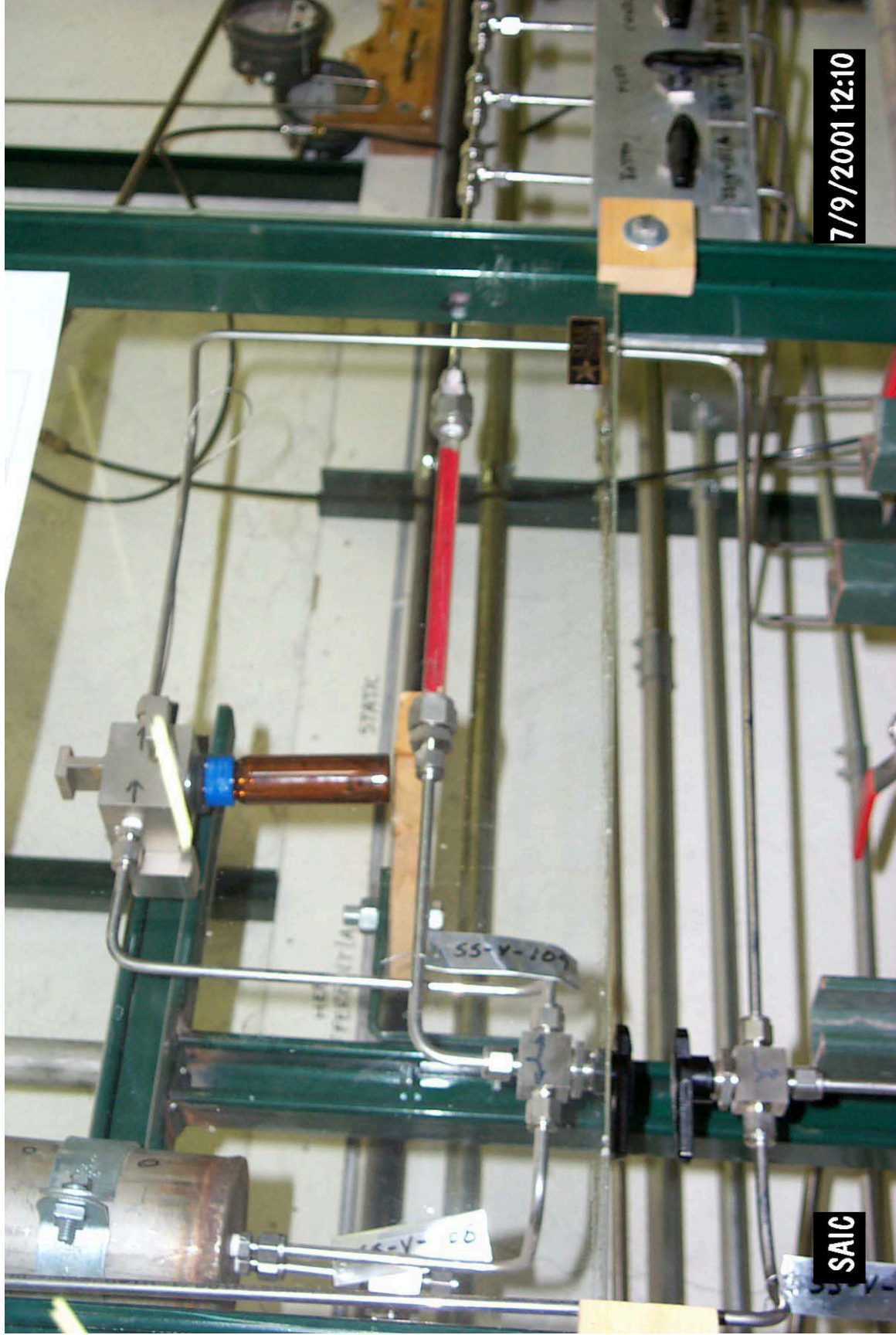


Figure D-2: Double Needle Sampler with 40-ml sample bottle.



Figure D-3: Double Needle Sampler Operations



Figure D-4: Double Needle Sample – Flow rate measurement.