

ISOPAR[®] L RELEASE FROM SALTSTONE CURED AT 55 °C

C. J. Berry, M. B. Bronikowski, A. D. Cozzi, R. E. Eibling, A. R. Marinik, and
J. R. Zamecnik

May 2006

Process Science and Engineering
Savannah River National Laboratory
Aiken, SC 29808

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SAVANNAH RIVER NATIONAL LABORATORY

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REVIEWS AND APPROVALS

AUTHOR(S):

C.J. Berry, Environmental Biotechnology Date

M.G. Bronikowski, Actinide Technology Date

A.D. Cozzi, Process Science & Engineering Date

R.E. Eibling, Process Science & Engineering Date

J.R. Zamecnik, Process Science & Engineering Date

TECHNICAL REVIEWERS:

J.M. Duffey, Actinide Technology Date

APPROVERS

R. E. Edwards, Manager, Process Science and Engineering Date

D. A. Crowley, Manager, Stabilization Science Research Date

J. E. Occhipinti, Manager, Waste Solidification Engineering Date

EXECUTIVE SUMMARY

The decontaminated salt solution waste stream from the Modular Caustic Side Solvent Extraction Unit and the Salt Waste Processing Facility is anticipated to contain entrained extraction solvent. The decontaminated salt solution is scheduled to be processed through Tank 50 into the Saltstone Production Facility. This study, among others, has been undertaken because the solvent concentration in the decontaminated salt solution may cause flammability issues within the Saltstone Disposal Facility that may need to be addressed prior to operation.

Previous work at the Savannah River National Laboratory determined the release of Isopar[®] L from saltstone prepared with a simulated DSS with Isopar[®] L concentrations ranging from 50 to 200 $\mu\text{g/g}$ in the salt fraction and with test temperatures ranging from ambient to 95 °C. The results from the curing of the saltstone showed that the Isopar[®] L release data can be treated as a percentage of initial concentration in the concentration range studied. The majority of the Isopar[®] L that was released over the test duration was released in the first few days. The release of Isopar[®] L begins immediately and the rate of release decreases over time. At higher temperatures the immediate release is larger than at lower temperatures.

In this study, saltstone was prepared using a simulated decontaminated salt solution containing Isopar[®] L concentrations of 50 $\mu\text{L/L}$ (30 $\mu\text{g/g}$) and 100 $\mu\text{L/L}$ (61 $\mu\text{g/g}$) and cured at 55 °C. The headspace of each sample was purged and the Isopar[®] L was trapped on a coconut shell carbon tube. The amount of Isopar[®] L captured was determined using NIOSH Method 1501.

The percentage of Isopar[®] L released after 20 days was 1.4 – 3.7% for saltstone containing 50 $\mu\text{L/L}$ concentration and 2.1 – 4.3% for saltstone containing 100 $\mu\text{L/L}$ concentration. Given the measurement uncertainties in this work there is no clearly discernible relationship between percentage release and initial Isopar[®] L concentration.

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LIST OF ACRONYMS

CSSX	Caustic Side Solvent Extraction
DSS	Decontaminated Salt Solution
GC	Gas Chromatograph
GGBFS	Ground Granulated Blast Furnace Slag
LFL	Lower Flammability Limit
MCU	Modular CSSX Unit
m/e	Mass to Charge Ratio
MS	Mass Spectrometer
NFPA	National Fire Protection Association
NIOSH	National Institute for Occupational Safety and Health
ppm	Parts per million (volume basis)
SDF	Saltstone Disposal Facility
SPF	Saltstone Production Facility
SWPF	Salt Waste Processing Facility

1.0 INTRODUCTION AND BACKGROUND

The Decontaminated Salt Solution (DSS) waste stream from the Modular Caustic Side Solvent Extraction (CSSX) Unit (MCU) and the Salt Waste Processing Facility (SWPF) is anticipated to contain entrained extraction solvent. The decontaminated salt solution is scheduled to be processed through Tank 50 into the Saltstone Production Facility (SPF). The solvent concentration in the MCU DSS exit stream has caused flammability concerns in the Saltstone Disposal Facility (SDF).¹ Because of these concerns, the release of the volatile solvent component Isopar[®] L from saltstone has been studied to assess possible flammability issues in the saltstone vault.

The design basis carryover of solvent in the DSS stream is 50 $\mu\text{g/g}$ (35 $\mu\text{g/g}$ Isopar[®] L). The maximum expected Isopar[®] L concentration in the DSS sent to saltstone under normal process conditions was determined through small scale testing to be 88 $\mu\text{g/g}$.² The flammable material concentration is required by NFPA 69 to be below 25% of the lower flammability limit (LFL) of the flammable mixture in the vapor space of the vault if no safety interlocks are installed. If all of the Isopar[®] L is released instantaneously into the vault vapor space when curing grout, the allowable Isopar[®] L concentration in the DSS from the MCU sent to saltstone has been calculated to be less than 1 $\mu\text{g/g}$.³ Displacement breathing of the saltstone vault coupled with instantaneous Isopar[®] L release would allow the concentration of the Isopar[®] L in the DSS to be 4.2 $\mu\text{g/g}$ based upon the NFPA requirement.

Prior testing of Isopar[®] L release from curing saltstone at 75 and 95 °C demonstrated unacceptable concentrations of Isopar[®] L in the vapor space for these temperatures.⁴ Therefore, SRNL was asked to determine the Isopar[®] L release from curing saltstone at 55 °C.⁵ The release was determined from saltstone prepared with a simulated DSS solution containing Isopar[®] L concentrations of 50 $\mu\text{L/L}$ (30 $\mu\text{g/g}$) and 100 $\mu\text{L/L}$ (61 $\mu\text{g/g}$). Isopar[®] L was used without other solvent components to provide bounding release rates.

2.0 EXPERIMENTAL APPROACH

Simulated DSS was prepared as an “average salt solution” with minimal component omission.⁶ Cesium chloride was not added to the DSS per the customer request.⁵ The organic components were also removed from the recipe to preclude interactions with the Isopar[®] L. Table 2-1 is the batch sheet for one liter of DSS simulant.

Saltstone was prepared using premix materials (cement, slag, fly ash) obtained from the SPF and simulated DSS. The water-to-premix ratio (w/p) is mass of evaporable water from the waste (at ~110 °C) to the combined mass of the cement, slag, and fly ash. The premix composition, water-to-premix ratio and amounts of the materials used are listed in Table 2-2.

Table 2-1. Composition of One Liter of Simulated DSS.

Component	Concentration (M)	Material	Mass (g)
		H ₂ O	708.32
AlO ₂ ⁻	0.28	Al(NO ₃) ₃ •9H ₂ O	105.04
OH ⁻	2.06	NaOH (50 wt %)	254.96
NO ₃ ⁻	2.03	NaNO ₃	99.85
NO ₂ ⁻	0.5	NaNO ₂	34.50
K ⁺	0.015	KNO ₃	1.52
CO ₃ ²⁻	0.15	Na ₂ CO ₃ •H ₂ O	18.60
SO ₄ ²⁻	0.14	Na ₂ SO ₄	19.89
Cl ⁻	0.024	NaCl	1.40
F ⁻	0.028	NaF	1.18
PO ₄ ³⁻	0.007	Na ₂ HPO ₄ •7H ₂ O	1.88
C ₂ O ₄ ²⁻	0.02	Na ₂ C ₂ O ₄	2.68
SiO ₃ ²⁻	0.03	Na ₂ SiO ₃ •9H ₂ O	8.53
MoO ₄ ²⁻	0.00007	Na ₂ MoO ₄ •2H ₂ O	0.02
NH ₃	0.001	NH ₄ NO ₃	0.08
Cu	2.2E-5	CuSO ₄ •5H ₂ O	0.0057
Cr	1.4E-3	Na ₂ CrO ₄	0.2336
Zn	1.2E-4	Zn(NO ₃) ₂ •6H ₂ O	0.0364
Pb	1.1E-5	Pb(NO ₃) ₂	0.0034
Fe	2.6E-5	Fe(NO ₃) ₃ •9H ₂ O	0.0104
Sn	2.0E-5	SnCl ₂ •2H ₂ O	0.0060
Hg	2.5E-7	Hg(NO ₃) ₂ •H ₂ O	0.0001
		Total	1258.75
Density	1.27 g/mL		
Wt% solids	28.8%		

Table 2-2. Saltstone Premix and Slurry Compositions.

Premix	Premix (wt %)	Water/Premix	Saltstone (wt %)
Class F Fly Ash (FA)	45	0.60	24.4
GGBFS ^a (Slag)	45		24.4
Cement	10		5.4
Salt Solution	—		45.8

^aGround granulated blast furnace slag

The cement, fly ash, and slag were weighed and added to a glass vessel with a Teflon[®] lid. The proper amount of salt solution (124 grams) was added to the same mixing vessel. Table 2-3 is the data sheet used to prepare each of the vessels. The vessel was closed and vigorously agitated by hand to prepare the saltstone slurry. Next, a volumetric aliquot of Isopar L was added to the mixture (10 μ L for the 100 μ L/L concentration and 5 μ L for the 50 μ L/L concentration). The vessel was again sealed and further vigorously agitated by hand to ensure complete mixing. Finally, the saltstone slurry was poured into the test vessel and transported to the test oven, Figure 2-1. This was repeated for each test sample, which included triplicate test samples with 50 μ L/L (30 μ g/g) of Isopar[®] L in salt solution, triplicate test sample with 100 μ L/L (61 μ g/g) Isopar[®] L in salt solution, and a single blank test sample with no Isopar[®] L added.

Table 2-3. Data Sheet used to Prepare each of the Saltstone Mixes.

Saltstone Mix Data Sheet			
MIX # 0060		Date: 11/1/2005	
Material	%	WT%	Grams
Waste Solution: <u>MCU- isopar</u> Wt% Solids # <u>28.8</u> Grams Water <u>88.29</u>		45.76	124.00
Admixture: _____			
Admixture: _____			
Admixture: _____			
Premix		54.24	147.00
Cement (% of Premix)	10	5.42	14.70
Slag (% of Premix)	45	24.41	66.15
Fly Ash (% of Premix)	45	24.41	66.15
Total	100	100	271.00
Water to Premix Ratio	0.60		
Calculations: Salt density 1.27 Isopar density 0.78 55 °C Isopar only 100 ppm 9.8 μ L Isopar 50 ppm 4.9 μ L Isopar			



Figure 2-1. Saltstone slurry curing at 55 °C.

Each test vessel was placed into the oven and connected to a passive sampling system using Teflon lined tubing, Figure 2-2. After each vessel was connected, the oven was heated to 55 °C. Test vessels were sealed and vented only through the passive sampling system maintained at ambient laboratory temperatures. The sampling system contained a two-section activated carbon sampling tube, Figure 2-3. Each tube contained 150 mg of activated coconut shell carbon (CSC) in two sections (100 mg front bed/ 50 mg back bed). The method utilized for quantifying the adsorbed Isopar[®] L is based upon the adapted NIOSH method (1501) used in previous testing.⁷

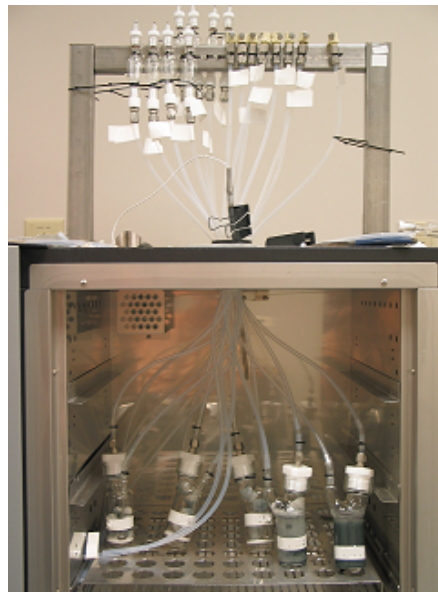


Figure 2-2. Vessels plumbed to passive sampling system.

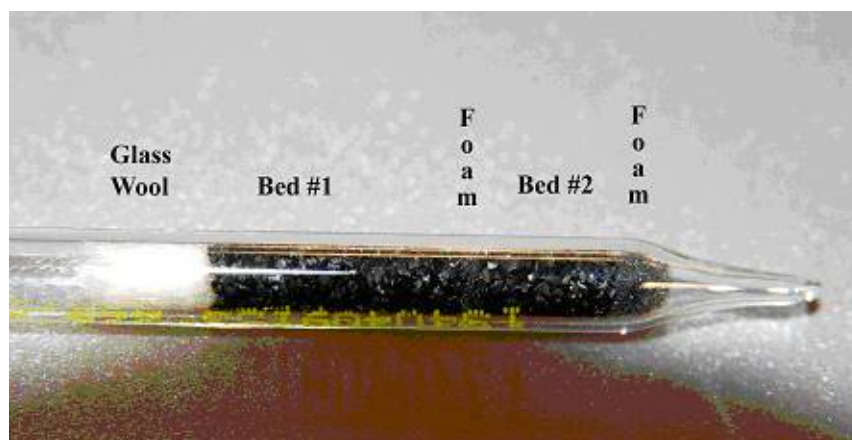


Figure 2-3. Coconut shell carbon sampling tube.

To determine the amount of Isopar[®] L released from a saltstone sample during a particular time period, the headspace of the sample vessel was purged with air for approximately 5 to 10 minutes at a flow rate of about 100 mL/min. The purpose of the air purge was to sweep the contents of the headspace through the CSC sampling tube prior to removing the sampling tube from the sample vessel. The front carbon bed (closest bed to the incoming airflow) of the sampling tube is designed to adsorb volatile organic compounds (VOCs) that pass through the tube. The back bed (that closest to the outgoing vent end) is designed to adsorb VOCs not retained on the front bed due to saturation of the front bed or due to volume breakthrough caused by excessive airflow.

After the air purge was complete, the CSC tube was removed and replaced with a fresh sampling tube. The CSC tube removed from the sample vessel was opened, and the front and back CSC beds were removed from the tube and each bed was placed into a separate 2-mL sample vial.

The CSC was transferred into the appropriate sample vials and the organic compounds were eluted from the CSC using 1 mL of carbon disulfide (CS₂). After the addition of CS₂ eluent, the sealed sample vials were gently agitated and allowed a minimum of 30 minutes to ensure complete elution of the CSC prior to analysis. Samples were analyzed by gas chromatography coupled with mass spectrometric detection (GC-MS).

To determine the recovery efficiency of the CSC tubes, four CSC tubes were spiked with approximately 2 μ L Isopar[®] L. The CSC tubes were desorbed and analyzed. An average of $92.4 \pm 4.3\%$ of the Isopar[®] L was recovered from the tubes. Analytical data will be reported assuming 100% recovery of the Isopar[®] L. It is also assumed that 100% of the Isopar[®] L released from the saltstone during testing was recovered on the CSC tubes. This assumption was based on the reported recoveries of Isopar[®] L of greater than 90% from samples held at 95 °C.⁴

Isopar[®] L is a complex mixture of organic compounds composed of multiple branched aliphatic (i.e., Isoparaffinic) hydrocarbons all sharing a distillation fraction. Given this complexity, analysis of Isopar[®] L with GC-MS required significant method development to account for summation of the multiple peak signals that result from the varying of hydrocarbons that make up Isopar[®] L. The mass spectrum for one of the compounds in Isopar[®] L is displayed in Figure 2-4. The mass spectra of all of the compounds are characterized by having significant signals at mass-to-charge ratio (m/e) of 57, 71 and 85 with the 57 signal generally the most significant. The GS-MS was operated in the selective ion monitoring mode

using the m/e 57 signal to generate the chromatogram. Isopar[®] L has a “fingerprint” chromatogram as shown in Figure 2-5, in that the chromatogram always consists of peaks of similar shape and area between two specific retention times. To quantify the results of Isopar[®] L analysis with a mass selective detector, two basic methods could be employed. Either the summation of all integrated peak areas is used, or the peak area of a single peak is used to represent the total material. In method development for these analyses the summation method proved far more reliable. Therefore, the results are reported based upon the summation of all integrated peaks between consistent retention times.

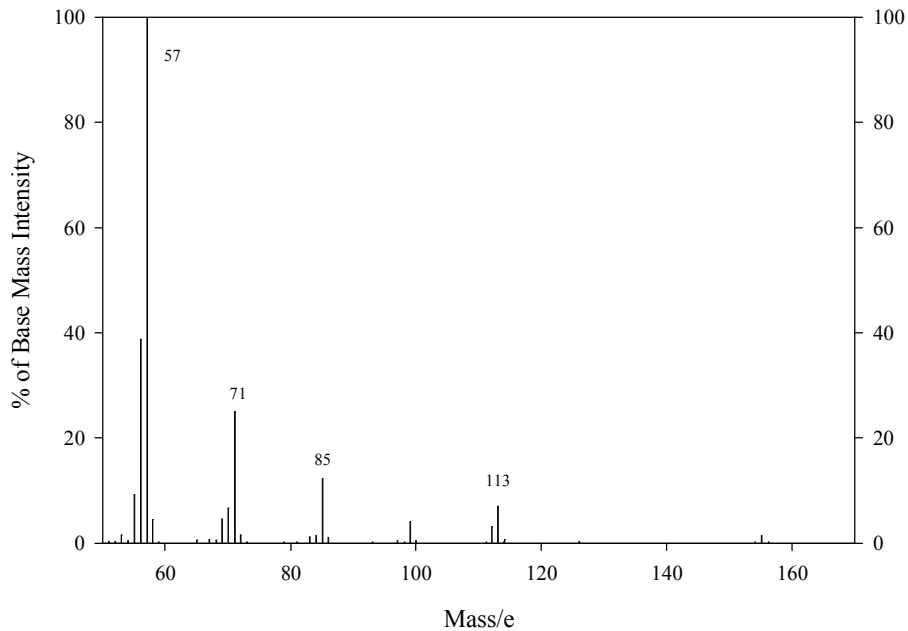


Figure 2-4. Mass spectrum of Isopar[®] L component

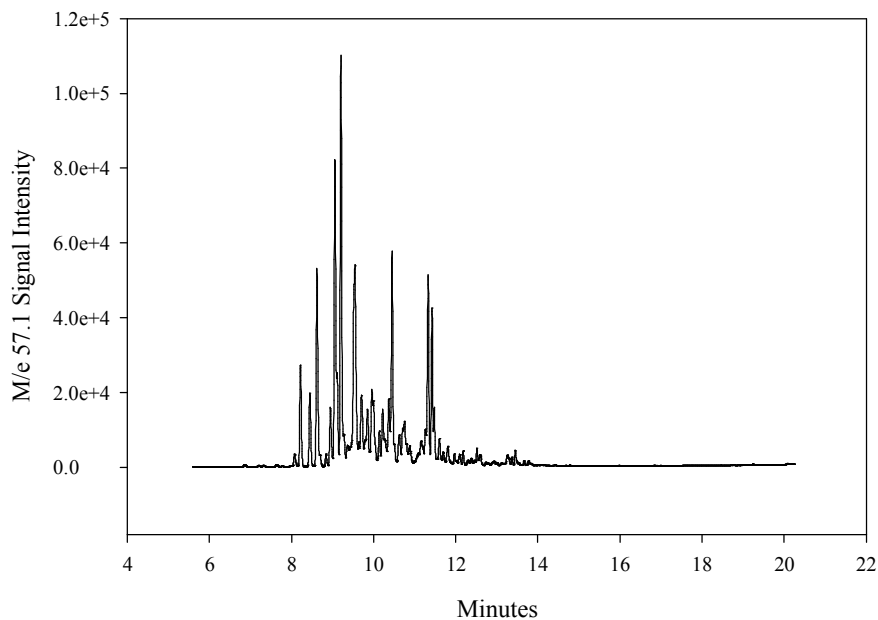


Figure 2-5. GC-MS chromatogram of Isopar[®] L using selective ion monitoring mode.

Different GC-MS systems were used to analyze Isopar samples. All systems used similar method parameters for analysis to keep consistent retention times. All samples were analyzed using an Agilent 5973 GC-MS equipped with an Agilent 7683 automatic liquid autosampler. The details of the analytical method are listed below in Table 2-4.

Table 2-4. Isopar[®] L GC-MS Analytical Method

Capillary Column	5% Phenyl, 95% Dimethyl Silicones (DB-5MS)
Column Length	30 or 60 meters
Column Inner Diameter	0.25 or 0.32 mm
Film Thickness	0.25 or 1.0 μ m
Carrier Gas	He, constant flow
Carrier Flow Rate	1 - 1.3 mL/minute
Injection Method	Split 1:10
Injector Temperature	300 °C
Initial Oven Temperature	100 °C (hold for 4 minutes)
Temperature Ramp	8.0 degrees/minute to 230 °C
GC Run Time	20.25 minutes
GC-MS Interface Temperature	280 °C
Detector	Quadrupole Mass Spectrometer
Ionization	Electron Impact
GC-MS Mode	Selective Ion Monitoring
Ion Monitored	m/e 57
Autosampler Rinse Agent	Methanol

Standards were prepared by adding a known mass of Isopar[®] L to a Class A volumetric flask and diluting to the mark with CS₂. The analytical sequence of the samples analyzed consisted of the following pattern: External Standards of 1, 10, 100, and 545 μ g/mL, all samples; and then external standards of 1, 10, 100, and 1000 μ g/mL. The calibration curve for these analytical sequences was based upon the initial standards and checked by the last standard set. However, the analytical sequence utilized for the last four sample sets consisted of the following pattern: External standards of 1, 10, 100, and 1000 μ g/mL, four samples, a second standard set (1, 10, 100, and 1000), four samples, third standard set, etc. The use of this alternate analytical sequence provided the ability to re-establish the calibration curve after every four samples to account for drift inherent in the analytical method. Figure 2-6 shows two examples of the calibration drift among standards during an analytical cycle. The response area of the standards decreases with each additional cycle.

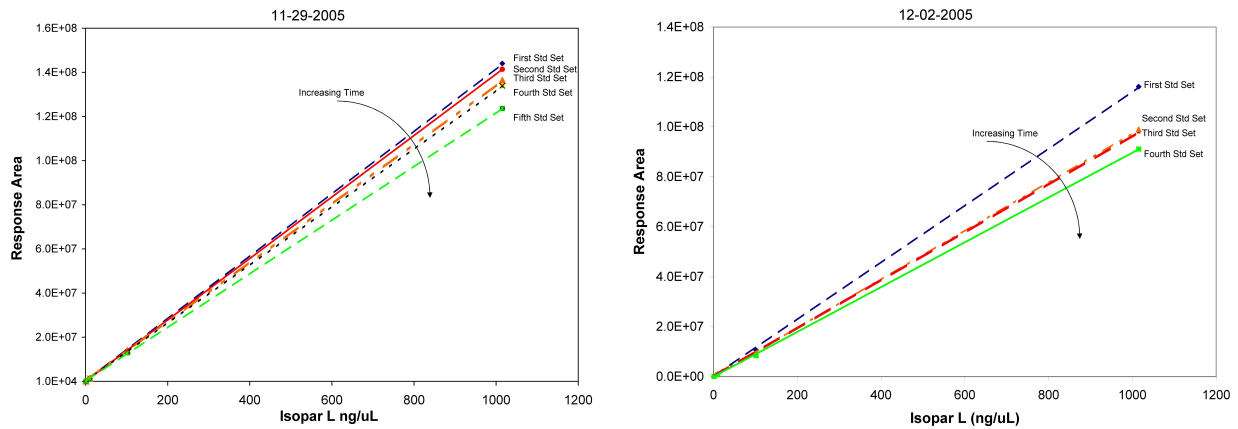


Figure 2-6. Calibration curves established during analysis.

In processing the results, these calibration curves were used to develop a drift curve based on the slopes of the calibration curves over time. This was determined by plotting the calibration curve slope for each set of standards as a function of time and fitting a curve to the slopes, providing a time based calibration curve. For example, given the five sets of standards from 11-29, Table 2-5, a calibration curve was calculated for each set. This was done by plotting the four standard response areas as a function of concentration and forcing the line through the origin to retrieve a calculated slope for that set of standards. The concentration of the standard (or sample) is then calculated by dividing the response area by the calculated slope. To correct for the drift in calibration, the calculated slopes are plotted as a function of time and a time dependent calibration curve is fitted, Figure 2-7. The fit equation is then used to calculate the time corrected concentration for the standards and samples. The drift seen in the curve relates the downward trend in sensitivity of the detector to the time a sample is processed. Therefore, the results are based upon a time adjusted calibration curve accounting for drift with each set of standards. The drift curve was established over three separate analytical sequences of varying length and found to be fairly consistent with respect to slope. However, the y-intercept of the linear trend line of each fell over time, which indicates an electron multiplier instability issue. The drift of the signal therefore does not detract from the validity of the data due to the standard calibration curves.

Table 2-5. Standard Data from GC-MS 11-29-2005.

Standard Concentration (µg/mL)	Response Area	Calculated Slope	Calculated Concentration (µg/mL)	Elapsed Time (min)	Time Corrected Concentration (µg/mL)
1015	144070587	141886	1015.4	0	1007.3
101.5	13843080	141886	97.6	24	97.2
10.15	1382924	141886	9.7	48	9.8
1.015	130954	141886	0.9	72	0.9
1015	141449940	139310	1015.4	168	1020.4
101.5	13632356	139310	97.9	191	98.8
10.15	1418784	139310	10.2	215	10.3
1.015	125382	139310	0.9	239	0.9
1015	136398129	134411	1014.8	335	1016.0
101.5	13936977	134411	103.7	358	104.3
10.15	1349220	134411	10.0	382	10.1
1.015	120679	134411	0.9	406	0.9
1015	133920273	131901	1015.3	478	1026.2
101.5	12980069	131901	98.4	502	99.9
10.15	1350124	131901	10.2	526	10.4
1.015	116193	131901	0.9	549	0.9
1015	123684834	121912	1014.5	764	1005.5
101.5	12933809	121912	106.1	788	105.7
10.15	1202455	121912	9.9	811	9.9
1.015	119148	121912	1.0	835	1.0

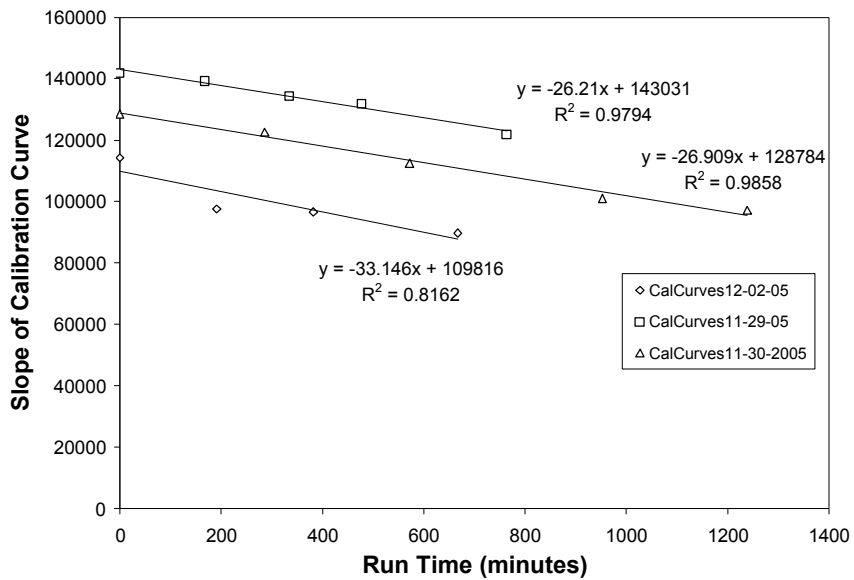


Figure 2-7. Time dependent calibration curves used to correct for signal drift.

3.0 RESULTS

Samples were collected over a 28-day period. Prior testing exhibited a tendency for the Isopar release rate to be greatest in the first 24 hours with a significant decrease in release rate after three days.⁴ Therefore, samples were pulled after approximately three hours and again at approximately twenty-four hours after preparing the saltstone. Following the initial two sampling times, all sampling times were at approximately seven-day intervals. Every vessel was sampled at each specified sampling time, allowing for triplicate analysis of each test concentration.

The release of Isopar[®] L for the 50 $\mu\text{L/L}$ (30 $\mu\text{g/g}$) concentration tests averaged 0.38 and 0.01 $\mu\text{g/g}$ salt solution at 3 and 24 hours, respectively. Release of Isopar[®] L for the 100 $\mu\text{L/L}$ (61 $\mu\text{g/g}$) concentration test averaged 0.90 and 0.39 $\mu\text{g/g}$ salt solution at 3 and 24 hours, respectively. As seen in Figure 3-1, the cumulative release did continue in an upward trend throughout the testing, however, the amount of release per sample time nearly leveled off. Shaded areas show the uncertainty in the measured values expressed as one standard deviation when release is plotted as a percentage of the added Isopar[®] L, Figure 3-2. The release rate, in this case, can be defined as the tangential slope of the curves in Figure 3-1 at a given time, and is shown in Figure 3-3.

After the first 24 hours of curing, release rates were significantly reduced (see Figure 3-3). This is in part due to the changing nature of the saltstone. As prepared, saltstone is a fresh slurry with a nominal effective viscosity of 60 centipoise. During this time, the Isopar[®] L can move easily through the fresh slurry and evaporate from the surface. As the hydration of the cementitious materials progresses, the slurry transforms from a self-leveling fluid to self-supporting structure. The development of a crystalline structure provides resistance to the transport of the Isopar[®] L, effectively reducing its availability at the surface for evaporation.

The release rate of Isopar[®] L from saltstone is related to the temperature (curing) of the saltstone. Moreover, the temperature of a system containing Isopar[®] L drives the vapor pressure of Isopar[®] L such that the temperature rise due to the heat of hydration would be the primary influence of the release rate of Isopar[®] L in the curing saltstone, Figure 3-4. The release from the ambient samples in Reference 4 exceeds those from samples at 55 °C. This is attributed to the improved precision in the analytical method between the two studies and the small difference in vapor pressure (release) of Isopar[®] L that is observed between ambient (25 °C) and 55 °C, 0.7, 4.4 torr, respectively. The curve fit is forced through the origin (zero release for sample with zero vapor pressure) and allows for the estimation of release of Isopar[®] L from saltstone over a range of temperatures. It should be noted that the release curve resembles a curve that would be expected for evaporation of a liquid (vapor pressure), indicating that the primary driving force for Isopar[®] L from saltstone is vapor pressure (temperature).

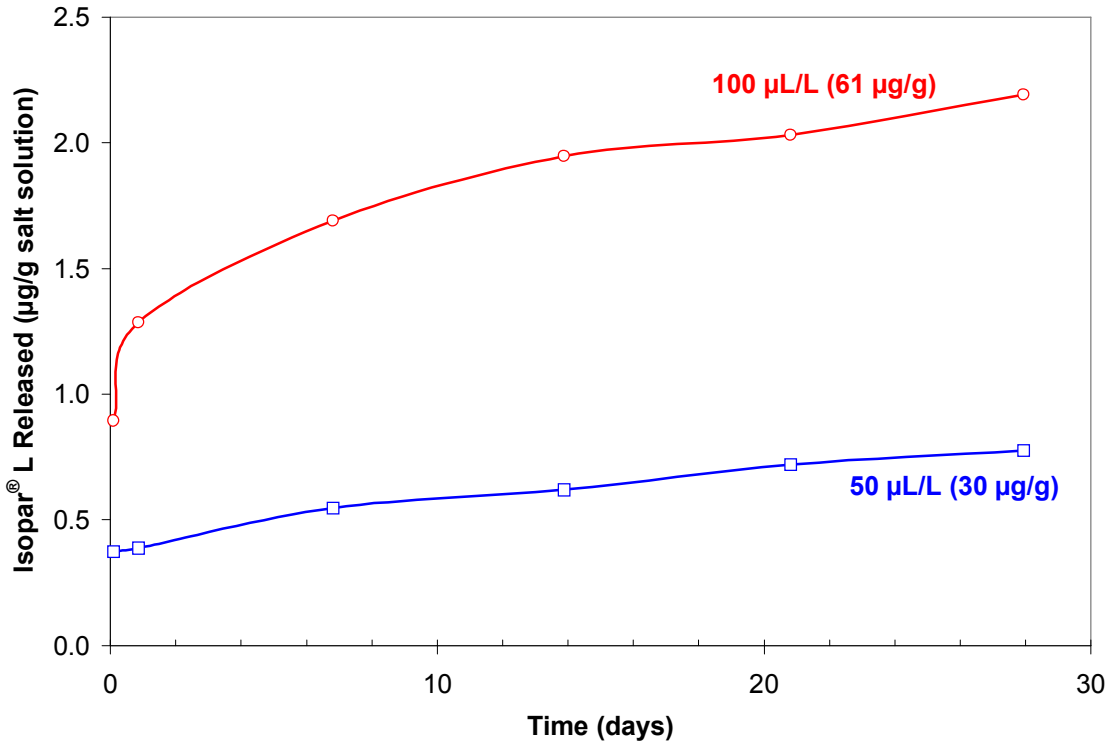


Figure 3-1. Cumulative release of Isopar[®] L from saltstone at 55 °C.

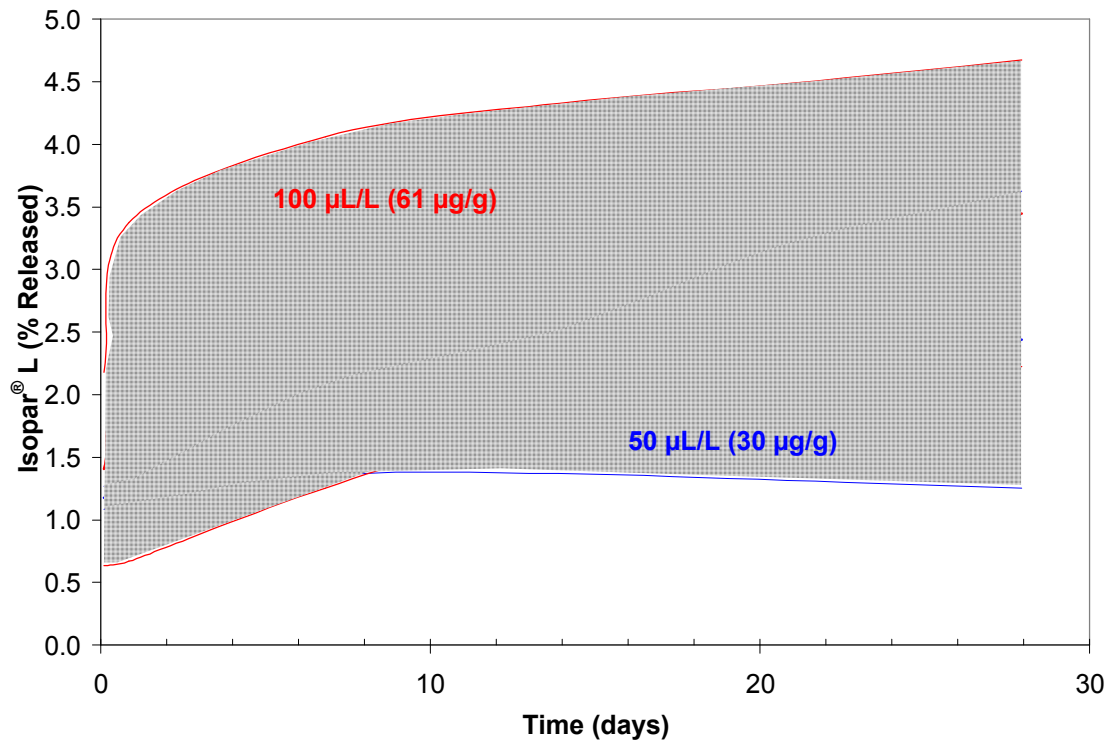


Figure 3-2. Cumulative release as a percentage of Isopar[®] L from saltstone at 55 °C.

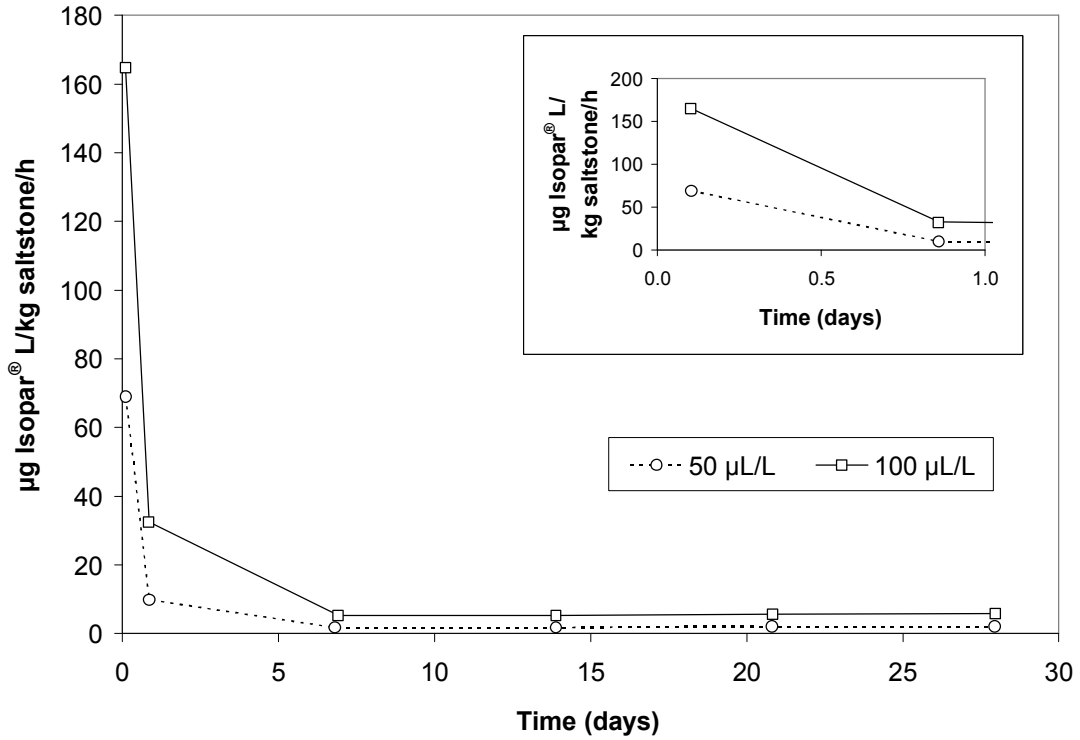


Figure 3-3. Release rate of Isopar[®] L from saltstone.

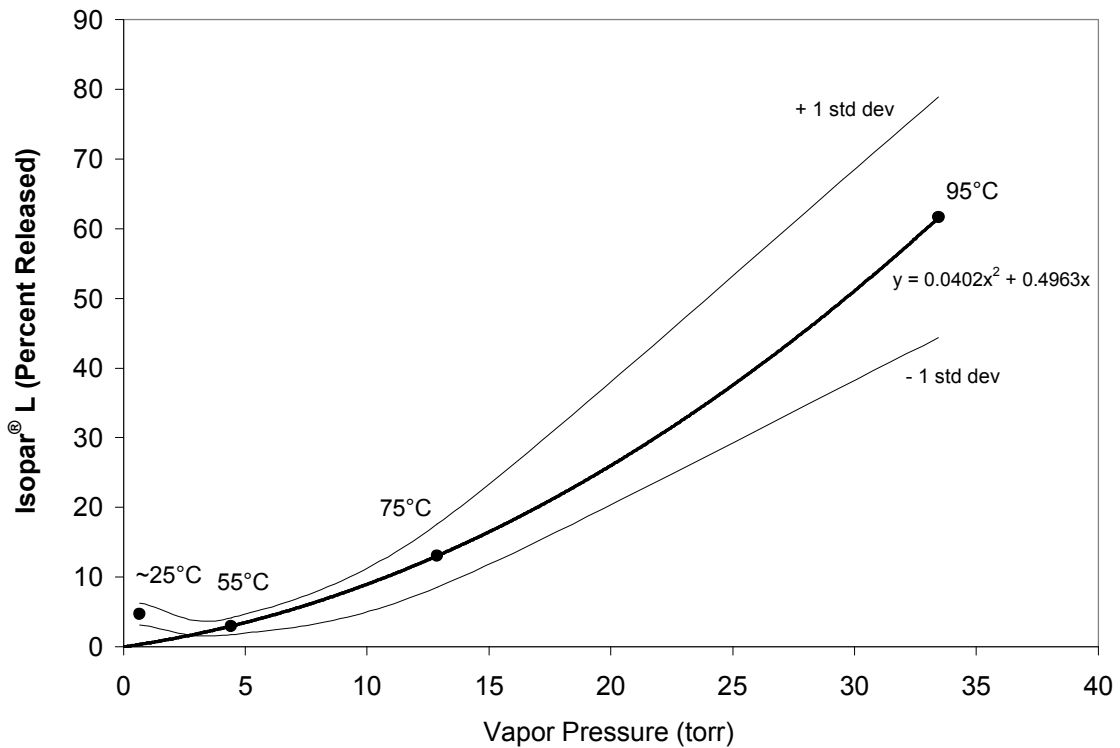


Figure 3-4. Percent of Isopar[®] L released as a function of vapor pressure.

4.0 CONCLUSIONS

The data generated at 55 °C fits the same trend as the previous testing.⁴ The temperature of the saltstone dictates the vapor pressure of the Isopar[®] L contained within the saltstone, which, in combination with the changing nature of the saltstone due to curing, is the ultimate driver for release of Isopar[®] L. Overlap of the ambient data from Reference 4 and the data collected in this study is attributed to both the increase in the analytical precision gained between the two studies and the small difference in the vapor pressure of Isopar[®] L between the two test temperatures. All of the release data collected in the two studies can be fit to a curve that allows an estimation of the amount of Isopar[®] L that would be released from saltstone over a range of given temperatures.

The percent released (the total of Isopar[®] L released as a percent of the total initially in the Saltstone matrix), is primarily dependent on the curing temperature. A curve fit forced through the origin (zero release for sample with zero vapor pressure) allows for the estimation of release of Isopar[®] L from saltstone over a range of temperatures. At 55 °C, the percent released was 1.4 – 3.7% for the 50 µL/L (30 µg/g) concentration and 2.1 – 4.3% for the 100 µL/L (61 µg/g) concentration. Given the measurement uncertainties in this work there is no clearly discernible relationship between percentage release and initial Isopar[®] L concentration.

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