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Test Specification: TSP-W375-01-00027
Test Plan: TP-RPP-WTP-061
Test Exceptions: None
R&T Focus Area: Pretreatment
Test Scoping Statement(s): B-86

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Completeness of Testing

This report describes the results of work and testing specified by Test Specification TSP-W375-01-00027 (Johnson 2001) and Test Plan TP-RPP-WTP-061 (Burgeson 2001b). The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

Gordon Beeman, Manager
WTP R&T Support Project

Date

History Sheet

Rev	Date	Reason for revision	Revised by
0	August 2002	New Document	BK McNamara
1	September 2003	Update Table 3. 2, 3.3, 3.4, 4.2, 4.5; flagged 99Tc as SB=standards bias	BK McNamara
		Added Appendix F: ICP-MS Data Report Impacted by Standards Bias	
		Table of Contents updated to include Appendix F	

Summary of Testing

Objectives

Battelle – Pacific Northwest Division (PNWD), is conducting physical property testing of waste from underground storage tank number 241-AN-102 (AN-102) for Bechtel National, Inc. (BNI). The River Protection Project Waste Treatment Plant (RPP-WTP) will receive low-activity waste feed and high-level waste feed for processing. These wastes will be stored at the waste treatment plant in large receipt tanks while awaiting processing. The pretreatment equipment is housed in ventilated processing cells. Ventilation is provided for control of temperature and radiological contamination (Gehrke 2000a, 2000b). The design temperature range for the process cells is 15º to 45ºC. Evaluating the effects of temperature on the solubility of solids present in LAW solutions is important to ensuring the radionuclide removal requirements can be met. If the solids present in LAW solids are solubilized at the operating temperature in the RPP-WTP equipment, this could lead to an increase in the concentrations of strontium-90 (^{90}Sr) and TRU elements, which could affect the process for separating strontium and TRU elements. This report summarizes testing performed in accordance with Test Specification TSP-W375-01-00027 and Test Plan TP-RPP-WTP-061 as part of Scoping Statement B-86. A limited set of experiments were undertaken with waste slurry from Tank 241-AN-102 (AN-102) to assess the effects of changing temperature on the stored tank waste feeds. A single test with caustic added to the tank waste slurry was also performed to understand changes in component distribution resulting from the roughly 2.6 vol% caustic addition (148,302 kg of 50 wt% sodium hydroxide) to Tank AN-102 in September 2001 (CHG 2001). Test Specification TSP-W375-01-00027 (Johnson 2001) and Test Plan TP-RPP-WTP-061 (Burgeson 2001) requested that the data collected under the temperature experiment be compared with characterization data at 25°C, as described in C*hemical Analysis and Physical Properties Testing of 241-AN-102 Tank Waste-Supernatant and Centrifuged Solids* (Urie et al. 2002). The Test Specification also required that a statistical evaluation of the combined experimental data be performed to qualify the results.

The objectives of the test were:

- determine the solubility at 15º and 50ºC for the solids present in AN-102 tank waste.
- determine the solids solubility versus concentration of NaOH at 25ºC for the solids present in AN-102 tank waste.
- perform a statistical evaluation of the solids and liquids composition data, incorporating the recent solids and liquids characterization data by Urie et al. (2002).

Conduct of Testing

The effect of temperature on the composition of AN-102 tank waste was assessed using new experimental data collected at 15° and 50°C. These data are compared to the in-depth characterization data acquired at 25°C by Urie et al. (2002) for the as-received AN-102 supernatant.

The effect of caustic addition on the solubility of AN-102 solids was assessed using the supernatant data from Burgeson (2001) and Urie et al. (2002). The intent of the caustic addition experiment was to measure the effect of hydroxide on the distribution of tank components between supernatant and solid. Both the solid and supernatant compositions were analyzed after adding 0.05 g of 19M NaOH per mL tank waste slurry. The data were compared to the unaltered AN-102 supernatant and solids data, taken at 25°C, by Urie et al. (2002).

Results and Performance Against Objectives

Temperature and Solubility

The supernatant characterization data from Urie et al. (2002) at 25°C and newly acquired 15° and 50°C experimental data are compared in this report. A statistical evaluation of these data revealed many instances where the analytical data from Urie et al. did not correlate well with the 15° and 50°C experimental results reported here. However, for a large number of supernatant analytes, it is clear there is little change with temperature. In fact, for most cases, the data appear to be centered about a mean rather than indicative of a clear trend with change in temperature. From this comparison, only two analytes (nitrate and phosphate) exhibited concentration changes exceeding 20%.

An alternative approach was used to assess whether small changes in composition might be made more definitive. This approach used a regression analysis that considered only the 15° and 50°C experimental data. Although a small uncertainty in a data point at either temperature extreme will tend to emphasize the magnitude of response, these data were obtained at the same time and under the same process conditions. Table S.1 lists analytes that had good sample reproducibility, whose reported concentrations were well above stated minimum detection limits, and that demonstrated changes in concentration greater than 15% between 15° and 50°C.

Table S.1. Supernate Analytes that Displayed Statistically Significant Concentration Variations over 15° to 50°C Temperature Range

Analyte	Statistically Relevant Temperature Effect?	Relative Percent Change in Concentration[(a)]
Mn	Yes	-26
NO_3^-	Yes	21
[b]P	Yes	60
^{241}Am	Yes	17

(a) A negative sign indicates the concentration at 50°C was less than the concentration at 15°C.
(b) P is the total phosphorous concentration

Both statistical evaluations indicated that most analyte concentrations did not change with temperature. For some analytes, such as boron and silicon, nothing can be said with regard to temperature, due to experimental artifacts or measurement uncertainty. These analytes are flagged and their uncertainties discussed.

The change in concentration with temperature in the subset of analytes in Table S.1 is discussed. The conclusion is that the reported magnitude of change in concentration of Mn, NO_3^-, P, and ^{241}Am should be regarded with some caution due to the limited number of temperatures (two end points) used in the analysis. For instance, the increase in supernatant americium concentration was not followed by other trivalent analytes whose chemistries are similar: lanthanum, europium, or neodymium. Because of the relevance of americium-241 as a transuranic, the change in concentration was accepted here as real. The manganese, nitrate, and total phosphorus concentrations also appeared to change with temperature.

Distribution of Tank Waste Components Due to Caustic Addition

The effect of caustic addition on the solubility of AN-102 solids was assessed using the supernatant data from Burgeson (2001) and Urie et al. (2002). Caustic addition (0.2M to 0.83M hydroxide) to AN-102 slurries caused little change in the supernatant composition. The level of uncertainty encountered was below 15% for most analytes. For some of the analytes the magnitude of actual change was masked by uncertainties arising from sample process conditions and uncertainties in the analytical measurements.

A mass recovery was calculated for the supernatants for both data sets. Table S.2 shows the results of the calculation. The summation of cation and anionic concentrations yielded reasonable agreement with the measured dissolved solids in the supernatant for both the Urie et al. and Burgeson supernatant data.

Table S.2. Mass and Charge Balance of AN-102 Supernates and Solids

	Cations µg/mL	**Anions µg/mL**	**Total µg/mL**	**Wt% Solids**	**Mass Balance Ratio**[(a)]	**Charge Balance Ratio**[(b)]
25°C Supernatant (Urie)	186,000	397,000	583,000	53.5[(c)]	1.09	1.05
Average 15° + 50°C (Burgeson) Supernatant	200,000	359,000	559,000	52.1[(c)]	1.07	0.97
Caustic 25°C (Burgeson) Supernatant	210,000	358,000	568,000	51.4 [(c)]	1.11	1.15

(a) The mass recovery for the solids was calculated as a ratio (cations + anions)/ (dried solids).
(b) The charge balance ratio was calculated as cation equivalents/anion equivalents.
(c) Based on the total dissolved solids in the supernatant (cations + anions).

The wet centrifuged solids analyte concentrations from the added caustic experiment were in general higher than those collected by Urie et al. at 25°C. All inductively coupled plasma atomic emission spectrometry (ICP/AES) and radiochemical analytes are reported significantly higher, while the anion data are consistent with the wet centrifuged solids data from Burgeson. Because there were no observable differences in the caustic supernatant relative to the Urie et al. supernatant data, a systematic difference, perhaps due to sample management, seems likely. The disparity thus compromised the intended comparison of the solids data.

Differences in the two data sets likely arose from variations in sample process conditions between the Urie et al. and Burgeson solids prior to the analytical measurement, and from lack of enough experimental testing to evaluate this variation. The reported solids data therefore could be used for further evaluation of changes with respect to caustic addition. It is important to note that the extensive compositing of AN-102 slurry by Urie et al. and the good accuracy presented from numerous replicate samplings outweigh the accuracy in the data presented herein.

Quality Assurance Requirements

PNWD implemented the RPP-WTP quality requirements in a quality assurance project plan (QAPjP) as approved by the RPP-WTP quality assurance (QA) organization. The tests reported in Sections 3.1 through 4.7 and all analytical data collection were conducted in accordance with PNWD's quality assurance project plan, CHG-QAPjP, Rev.0, which invoked PNWD's Standards Based Management System (SBMS), compliant with DOE Order 414.1A Quality Assurance and 10 CFR 830, Energy/Nuclear Safety Management, Subpart A - Quality Assurance Requirements. Due to a change in the contract QA requirements after September 2001, a reanalysis of the hydroxide concentrations, reported in Sections 3.1 through 4.7 was conducted in accordance with PNWD's quality assurance project plan, RPP-WTP-QAPjP, Rev.0, which invoked NQA-1-1989 Part I, Basic and Supplementary Requirements, and NQA-2a-1990, Subpart 2.7. These quality requirements were implemented through PNWD's Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual (WTPSP). The quality of the data gathered during the earlier experiments was not impacted by the change in requirements.

PNWD addressed verification activities by conducting an Independent Technical Review of the final data report in accordance with procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and the reported work satisfied the Test Plan objectives. The review procedure is part of PNWD's WTPSP Manual.

Issues

None.

Terms and Abbreviations

AEA	alpha energy analysis
ASR	analytical service request
BBI	best basis inventory
BNI	Bechtel National, Inc.
DOE	United States Department of Energy
DQO	data quality objective
EQL	estimated quantitation limit
GEA	gamma energy analysis
HDPE	high-density polyethylene
HLRF	High Level Radiation Facility
HLW	high-level waste
IC	ion chromatography
ICP-AES	inductively coupled plasma-atomic emission spectrometry
ICP-MS	inductively coupled plasma-mass spectrometry
IX	ion exchange
KPA	kinetic phosphorescence
LAW	low-activity waste
LCS	laboratory control standard
MDL	method detection limit
MRQ	minimum reportable quantity
MS	matrix spike
MSD	matrix spike duplicate
%D	percent difference
PNWD	Battelle-Pacific Northwest Division
QA	quality assurance
QC	quality control
RPD	relative percent difference
RPL	Radiochemical Processing Laboratory
ORP	Office of River Protection
SAL	Shielded Analytical Laboratory
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic
UDS	undissolved solids
VOL%	volume percent
WCS	wet centrifuged solids
WTP	Waste Treatment Plant
Wt%	weight percent

Units

°C	degrees Centigrade
g	gram
G	gravity equivalent
g/mL	gram per milliliter
μg/g - μg/mL	microgram per gram / microgram per milliliter
μCi/g - μCi/mL	microcurie per gram / microcurie per milliliter
mL	milliliter

Definitions

Settled solids—the solids layer that separates from the bulk slurry after three days of gravity settling. These results may be reported as vol% or wt %. The wt% values may be on a wet basis (mass of settled solids contains the interstitial liquid) or a dry basis (dried at 105°C to a constant mass).

Centrifuged solids—the solids layer that separates from the bulk slurry after one hour of centrifugation at 1000 G's. These results may be reported as vol% or wt%. The wt% values may be on a wet basis (mass of settled solids contains the interstitial liquid) or a dry basis (dried at 105°C to a constant mass).

Dissolved solids—soluble solids. The solids remaining after complete drying of a filtered liquid at 105°C. Typically reported as wt %. During drying, most mass loss is due to water, but other volatile components (e.g., organics) may also be lost.

Undissolved Solids—solids excluding all interstitial liquid. This can be thought of as the solids left if all the supernatant and associated dissolved solids could be drained from the bulk slurry. The undissolved solids will generally include some materials that can be washed or dissolved during pretreatment.

Total Solids—solids remaining after drying to a stable mass at 105°C; includes dissolved and undissolved solids.

Contents

Figures

Tables

1.0 Introduction

Bechtel National, Inc. (BNI) is under contract with the U.S. Department of Energy (DOE) Office of River Protection (ORP) to design and construct facilities for the treatment of waste stored in the single- and double-shell tanks at the Hanford Site near Richland Washington (DOE 2000). The waste treatment plant (WTP) will receive tank waste feed transferred from the Hanford tank farms according to two designations: low-activity waste (LAW) feed and high-level waste (HLW) feed. The low-activity waste feed is tank waste supernatant that may contain up to 2 wt% entrained solids upon delivery from the tank farms. The HLW feed will contain solids and associated supernatant. The wastes will be characterized prior to formal acceptance of the delivered tank waste feed to ensure the feeds meet the envelope of specifications identified in Bechtel's contract. Both the LAW and HLW feeds will be processed and then vitrified after receipt. The LAW feed will be pretreated to remove cesium, technetium, strontium, transuranic (TRU) compounds, and entrained solids prior to vitrification. The HLW feed will be processed to separate solids and liquids; the solids will be washed with dilute caustic and then blended with LAW entrained solids and pretreatment solutions containing cesium, technetium, strontium, and TRU compounds prior to vitrification (Carter and Ashley 2000).

The LAW and HLW feeds will be stored at the WTP in large receipt tanks to await processing. The LAW pretreatment equipment is housed in ventilated processing cells. Ventilation is provided for control of temperature and radiological contamination (Gehrke 2000a, 2000b). The design temperature range for the process cells is 15º to 45ºC. At temperatures exceeding 25ºC, dissolution of key components may be enhanced and may exceed process removal requirements. At sufficiently cooler temperatures, the concentrations of various salts may be lowered, causing undesirable precipitation of solids. Therefore, temperature and solubility effects on the stored tank waste feed may impact the pretreatment path or require implementation of more restrictive measures for thermal stabilization of LAW process equipment. An assessment of the effect of temperature change on the unprocessed tank waste feed composition is needed to ensure the pretreatment processing steps will not be adversely affected (Johnson 2001).

Tank 241-AN-102 (AN-102) contains supernatant and saltcake solids. In March 2001, solids measurements indicated a substantial increase in the saltcake volume since the tank was core-sampled in 1990. This increase in saltcake solids[(a)] is the result of caustic depletion, the degradation of organic complexants, waste cooling, and liquid evaporation. In September 2001, caustic solution was added to the tank for corrosion control (CHG 2001).

This report describes the results of a limited set of tests conducted by PNWD to assess the impact of changes in temperature and caustic concentration on the solubility of the solids present in Tank AN-102 waste. Changes in tank waste composition might impact envelope limits or result in precipitation and clogging of lines during transport or pretreatment. The Test Specification for this work requested PNWD to evaluate the distribution of tank components present in the AN-102 waste sample between the solids and liquids at two temperatures, 15° and 50°C (Johnson 2001). The Test Specification further directed that these tests assess the composition of the LAW supernatant and HLW solids at 25°C for one elevated caustic concentration. This test would simulate the 2.6 vol% hydroxide added to AN-102 in September 2001 (CHG 2001).

The work was conducted according to Test Plan TP-RPP-WTP-061 Rev. 0, *Determining Solubility of AN-102 Solids versus Temperature and Concentration*, which is attached as Appendix E. The results of these tests were to be compared with 25°C data recently acquired on the composited AN-102 solids and liquids by Urie et al. (2002). This would then provide a third temperature for the evaluation and a single point of comparison for the caustic evaluation. The objectives of the test were to:

- determine the solubility at 15º and 50ºC for the solids present in AN-102 tank waste.
- determine the solids solubility versus concentration of NaOH at 25ºC for the solids present in AN-102 tank waste.
- perform a statistical evaluation of the solids and liquids composition data, incorporating the recent solids and liquids characterization data by Urie et al. (2002).

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[a] The solids in AN-102 are referred to in the Hanlon Tank Waste Summary Reports (Hanlon 2000) and in Best Basis Inventory descriptions as salt cake (see, for instance, CHG 2001).

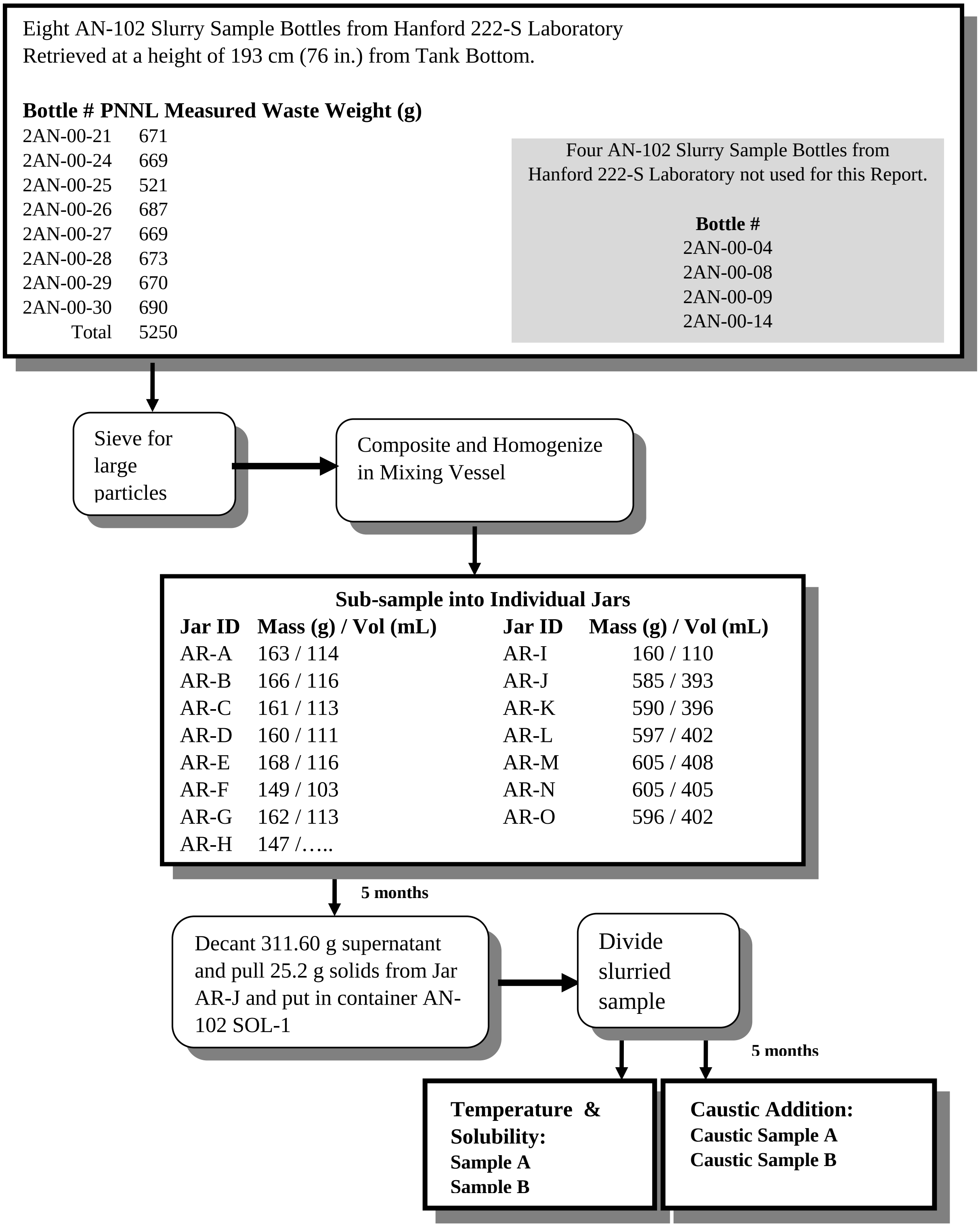
2.0 Sample Description

Tank farm operations grab-sampled the sludge layer of Tank AN-102 in August 2000 and retrieved 30 samples from Riser 22 at six different tank waste heights. Each sample, which consisted of wet solids plus supernatant incidental to obtaining the wet solids, was placed in a 500-mL bottle. The material collected from this sampling event was intended for characterization and process testing. Material with high solids content was required for the process testing; therefore, eight bottles representing solids and liquids collected 190 cm (74.8 in.) from the bottom of the tank were selected for compositing and homogenization. Each of the eight samples contained a settled layer of light brown solids with a dark brownish/black standing liquid. The solids content was approximately 50% by volume for each bottle.

The samples were received, homogenized, and subsampled in the High-Level Radiation Facility (HLRF). After verification of the homogenization, 15 subsamples, representing the entire composite sample, were collected. The samples were identified as AN-102-AR-A through AN-102-AR-O. Samples AN-102-AR-A, B, C, H and I were used for the physical, inorganic, radiochemical, and selected organic analyses reported in Urie et al. (2002). These data were the source of the room temperature (nominally 25°C) data in this report.

The experimental testing described within this report was performed using a sample identified in Figure 2.1 as AN-102-SOL-1, which was prepared on April 23, 2001, about five months after the initial compositing/homogenization sequence by Urie et al. (2002). The sample was reconstructed from the homogenized material using 25.2 g of settled solids to 311.6 g of supernatant. The density of this slurried material (1.432 g/mL) compared reasonably well with that reported by Urie et al. (1.406 g/mL). The percent total solids, determined by drying the suspension to constant weight, was approximately 1% lower (57.4%) than reported by Urie et al. (58.8%). The characterization data from Urie et al. are based on high wt% solids samples. Specifically, the jar identified in the flow diagram as AN-102 AR-J contained about 67.7 wt% settled solids. AN-102 SOL-1 was prepared as described above from the volumes of settled solids and supernatant in this jar. While the target settled solids weight was about 7.4 vol%, AN-102 SOL-1 was found to contain approximately 11(±3) vol% centrifuged solids. The target solid to liquid ratio was the calculated AN-102 static tank ratio of solids to liquid as reported by Hanlon (2000). The details of the sample collection are reported in Test Instruction TI-RPP-WTP-074 Rev. 1, *Energetics and Gas Generation Tests: AN-102 Solids Washing* (Bryan 2001).

The sample slurry density was determined in duplicate using a 5-mL volumetric flask. The sample was thoroughly mixed with a mechanical stirrer in the Shielded Analytical Laboratory (SAL) cells and transferred to a tared volumetric flask. The average density of the slurried material was determined to be 1.43 g/mL. Approximately 50 mL of the material was then transferred to four tared 125-mL, high-density polyethylene (HDPE) bottles. The mass and volume of each sample are listed in Table 2.1.



Eight AN-102 Slurry Sample Bottles from Hanford 222-S Laboratory Retrieved at a height of 193 cm (76 in.) from Tank Bottom.

Bottle #	PNNL Measured Waste Weight (g)
2AN-00-21	671
2AN-00-24	669
2AN-00-25	521
2AN-00-26	687
2AN-00-27	669
2AN-00-28	673
2AN-00-29	670
2AN-00-30	690
Total	5250

Four AN-102 Slurry Sample Bottles from Hanford 222-S Laboratory not used for this Report.

Bottle #
2AN-00-04
2AN-00-08
2AN-00-09
2AN-00-14

Sub-sample into Individual Jars

Jar ID	Mass (g) / Vol (mL)	Jar ID	Mass (g) / Vol (mL)
AR-A	163 / 114	AR-I	160 / 110
AR-B	166 / 116	AR-J	585 / 393
AR-C	161 / 113	AR-K	590 / 396
AR-D	160 / 111	AR-L	597 / 402
AR-E	168 / 116	AR-M	605 / 408
AR-F	149 / 103	AR-N	605 / 405
AR-G	162 / 113	AR-O	596 / 402
AR-H	147 /…..		

Decant 311.60 g supernatant and pull 25.2 g solids from Jar AR-J and put in container AN-102 SOL-1

Figure 2.1. Flow Diagram Connecting Receipt, Compositing/Homogenization of the Samples to the Construction of Sample AN-102-SOL-1 for the Temperature and Caustic Solubility Evaluation

Table 2.1. Measured Mass and Calculated Volume of AN-102 Slurry

AN-102 Sample ID	Slurry Mass, g	Slurry Volume, mL
Solubility Sample A	78.95	55.13
Solubility Sample B	78.61	54.89
Caustic Addition Sample A	79.01	55.16
Caustic Addition Sample B	79.26	55.35

3.0 Component Distribution Versus Temperature

3.1 Experimental: Temperature Control

The experimental apparatus consisted of a heating and cooling block, an orbital shaker, a recirculating bath, and a temperature controller. The orbital shaker table was a Lab-Line Instruments benchtop shaker (Model #3520) purchased from J-Kem Scientific, Inc. The top plate of the shaker table was modified at the factory on request. Nine wells were drilled into a 2-inch (10.5 x10.5 inch) aluminum block for sample placement. Each well was large enough to accommodate a 125-mL bottle. Channels ¾-inch in diameter were drilled into the side of the block to allow for passage of liquid coolant. The nine sample holes were centered in the aluminum block, and the remaining block depth was reduced to approximately ½ inch to reduce the weight of the block. This modification was necessary to ensure that the heating block could be transferred into the Shielded Analytical Laboratory (SAL) hot cells and then assembled.

The 15°C solubility test required a closed-loop chiller arrangement. The chiller reservoir was filled with a 50/50 mix of propylene glycol and water. The chiller was placed outside the hot cell and the chiller coolant was routed to the shaker table with ¾-inch reinforced tubing. The chiller unit was placed in a secondary containment basin to guard against the possibility of leaks of potentially contaminated coolant fluid.

The temperature controller used was a J-Kem Scientific Model 270 (J-Kem Electronics, Inc., St. Louis, MO). The controller consists of two separate circuits; one is a temperature control circuit and the other serves as an over-temperature device, which shuts down the system if a preset temperature is exceeded. Two K-type thermocouples (model CASS-116G-12-DUAL, Omega Engineering, Stamford, CT) were used to provide inputs to the temperature controller and over-temperature circuits. Both the J-Kem Model 270 temperature controller and the dual thermocouple were calibrated before use. A second, external record of the temperature was kept with a hand-held thermocouple thermometer and data logger (Cole Palmer, Digisense 91100-50), which also used calibrated K-type thermocouples. Using the data logger, the temperature was recorded every 30 minutes during the experimental runs. The collected data verified that both samples were stable (±0.5°C) during the 24-hour heating and cooling periods.

The heating and cooling system was tested using 50 mL of water in 125-mL high-density polyethylene (HDPE) bottles. The heating block was set to the target temperature (15° or 50°C), and the chiller was set to 13°C (for the 15°C test only). The water was permitted to equilibrate for approximately 30 minutes. The temperature of the water control sample was monitored continuously. Once the target temperature was obtained in the water control sample, the temperature of each tank waste slurry solution was measured.

3.2 Sample Preparation and Handling

The test specification, TSP-W375-01-00027 (Johnson 2001), stipulated that two temperatures, 15°C and 50°C, be used for the experimental determination of solids solubility versus temperature. In-depth characterization data were already available for the AN-102 supernatant and solids at approximately 25°C. The data collected under this experimental testing would be compared with that compiled by Urie et al. (2002) at 25°C. Duplicate samples of AN-102 waste slurry (see Table 2.1 for sample masses) were initially cooled to 15°C and held at that temperature for 24±1 hours. After sampling the supernatant, the duplicate slurry samples were heated to 50°C, held at temperature for 24±1 hours, and the supernatant sampled for analytical characterization.

For the 15°C solubility testing, the chiller was set to 13°C and the heating block to 15°C. The water control solution read 15.3°C and the two solubility slurry samples 15.1°C. The sample temperature was monitored every 15 minutes over two hours to verify that the samples were equilibrated. The two samples were shaken for 24 hours and 50 minutes. During this time, the temperature was recorded every hour. A review of the recorded temperature verified that it varied no more than ±0.3°C during the 24-hour period. The shaker table was turned off, and, with continued cooling, the solids were allowed to settle for about one hour. During this time, a 10-mL syringe equipped with a 0.45-µm nylon syringe filter was equilibrated at 15°C to maintain the solution temperature during filtering and avoid solubilization of suspended particles due to a temperature increase. The supernatant was sampled and filtered as quickly as possible to keep it at 15°C. However, the supernatant did not pass through the filter easily—it took approximately 20 minutes to filter the sample. Once filtered, the resulting supernatant samples, 15°C Sample A and 15°C Sample B, were aliquotted for analytical characterization.

After the 10-mL aliquot had been removed from both samples at 15°C, the tank waste slurries were brought up to 50°C. For the 50°C solubility test, the chiller was turned off and the heating block set at 50°C. The water control solution was 50.1°C, and the two solubility samples read 50.3 and 50.5°C. The sample temperature was monitored every 15 minutes over two hours to verify that the samples were equilibrated. The two samples were shaken for 24 hours and 50 minutes. During this time, the temperature was recorded every hour. A review of the recorded temperature verified that the temperature varied no more than ±0.3°C during the 24-hour period. The shaker table was turned off, and, with continued heating, the solids were allowed to settle for about one hour. During this time, a 10-mL syringe equipped with a 0.45-µm nylon syringe filter was equilibrated at 50°C. The intent was to maintain the solution temperature during filtering to avoid precipitation of particles due to a temperature decrease. The supernatant was sampled and filtered into 20 mL glass vials as quickly as possible to keep the supernatant at 50°C. Filtering of the supernatant took approximately 10 minutes. The filtered supernatant was collected into a glass vial that was equilibrated at 50°C. Once filtered, the two supernatant samples, 50°C Sample A and 50°C Sample B, were immediately aliquotted for analytical characterization. A concern arose that these samples might precipitate solids prior to analysis, and the samples were diluted as indicated in Table 3.1. The samples run at 15°C were not diluted in this way.

Table 3.1. Dilution of AN-102 Samples

Sample ID	Volume of Sample (mL)	Volume Deionized Water (mL)
AN-102-50°C A	0.100	9.992
AN-102-50°C B	0.100	10.016
AN-102-50°C B Duplicate	0.100	10.012

Each of the supernatant samples was analyzed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES), inductively coupled plasma-mass spectrometry (ICP-MS), and ion chromatography (IC). The samples were also analyzed for total organic carbon (TOC) and total inorganic carbon (TIC) content, weight percent total solids, total hydroxide, and radiochemical determination of ^{90}Sr, $^{239/240}$Pu, ^{241}Am, ^{155}Eu, ^{154}Eu, ^{242}Cm, $^{243/244}$Cm, and ^{137}Cs.

For Sample A at 15°C, duplicate TIC and TOC analyses were run. For Sample A at 50°C no duplicate analyses were run. For Sample B at 15°C, the anions Cl^-, F^-, NO_3^-, NO_2^-, SO_4^{2-}, PO_4^{3-}, OH^-, and the radiochemical data were run in duplicate; and for Sample B at 50°C, duplicate analyses were run for the entire analyte set.

Following the temperature and solubility experiments, the supernatant was analyzed for the target analytes defined in the Test Specification (Johnson 2001) (see Appendix D). The analytical processing of the supernatant is detailed in Figure 3.1. Analytical service request (ASR) number 6164 provided instructions to the laboratory to successfully complete the analytical and Quality Control (QC) requirements defined in the Test Specification.

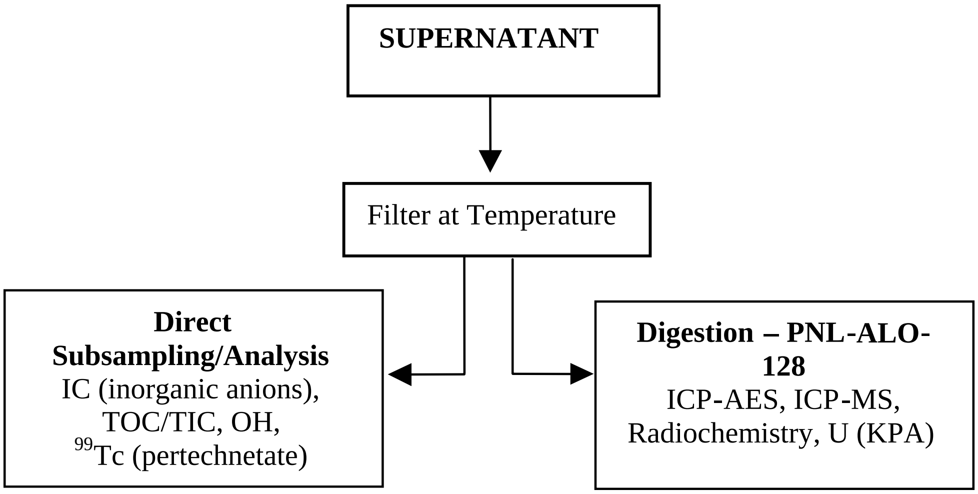


Figure 3.1. Flow Diagram for Analytical Processing of Supernatant Samples

3.3 Results: Component Distribution Versus Temperature

The average concentration of various supernatant waste components at 15°, 25°, and 50°C are reported in Tables 3.2 through 3.4. Results are reported in µg/mL or µCi/mL, as appropriate. The 25°C data are from the characterization of the homogenized AN-102 tank waste solids and supernatant and are reported in greater detail in *Chemical Analysis and Physical Property Testing of 241-AN-102 Tank Waste—Supernatant and Centrifuged Solids* (Urie et al. 2002). Analysis of the solids composition was not requested in the Test Specification (Johnson 2001) for the solubility versus temperature experiments.

Chemical Analysis and Physical Property Testing of 241-AN-102 Tank Waste—Supernatant and Centrifuged Solids

The data in the analytical results tables, Tables 3.2 and 3.3, include Data Flag columns using the following codes:

- U—Undetected. The analyte was analyzed for but not detected, e.g., no measurable instrument response or response was less than the method detection limit (MDL).
- J – Estimated Value. The reported value is estimated because: (1) the reported concentration is above the MDL but below the estimated quantitation level (EQL); (2) an associated quality control sample exceeded the acceptance criteria; (3) for radiochemical data the reported result has a propagated error of >10% for results > 10x the MDL.
- B – Analyte found in the associated laboratory preparation blank exceeded the acceptance criteria. The found concentration was either equal or more than the EQL, or higher than 5% of the measured concentration in the sample.

The MDL used in this report is estimated and includes all processing factors such as sample quantities used and dilutions resulting from digestion processing. For the inorganic anions by IC, the MDL is defined as the lowest calibration standard adjusted for the method processing factors.

For ICP-MS and ICP-AES, the MDL is based on instrument background signal using reagent water or dilute acid for a sample and adjusting for the processing factors. The TOC and TIC methods have established MDLs as stated in SW-846 (EPA 1986). For radiochemical methods, the minimum detectable activity (MDA) is calculated per the QA Plan and is based on the background counting statistics.

3.4 Data Limitations for the Supernatant Analytes at 15°

The reproducibility in the analyte concentrations reported in Table 3.2 is typically reported to the third significant figure. The data as presented have been converted from the reported data in µg/g to µg/mL by the supernatant density of 1.437 g/mL at 15°C. The raw data are reported from the analytical station with error flags to identify analyte values that may be suspect. The flags are identical at both temperatures for each analyte. Each flag is considered below.

- U – Undetected.

1) Ba, total U, and fluoride were not detected in the supernatant above the MDL/MDA and therefore, were flagged with a U.

- J – Estimated Value.

1) Fe, La, Cu, Mn, Mo, Nd, Si, and Sr were detected above the MDL but below the EQL. The reported values are estimated (J).

- X – Estimated Value – QC Criteria not met.

1) The blank spike recovery for K was 78%, outside the requirement of 80%-120%. The values reported for K in the samples could be biased low, and are therefore flagged as estimated value (X).

Table 3.2. Temperature Dependence Experiment—Concentrations in Supernatants at 15°C

Analyte	MDL / MDA (µg/mL)	AN-102 15°C Sample A (µg/mL)	Data Flag (a)	AN-102 15°C Sample A Duplicate (µg/mL)(b)	AN-102 15°C Sample B (µg/mL)	Data Flag	AN-102 15°C Sample B Duplicate (µg/mL)	Data Flag	Sample A and B Mean(c) (µg/mL)	RPD(d)
Al	5.3	11,900	--	--	11,800	--	--	--	11,850	NA
Ba	0.9	0.9	U	--	0.9	U	--	--	NA	NA(e)
Ca	22	482	--	--	476	--	--	--	479	NA
Cd	1.3	64	--	--	63	--	--	--	64	NA
Cr	1.8	194	--	--	193	--	--	--	194	NA
Fe	2.2	30	J	--	30	J	--	--	30	NA
K	178	2,300	X	--	2,300	X	--	--	2,300	NA
La	4.5	16	J	--	16	J	--	--	16	NA
Na	13	196,000	--	--	197,000	--	--	--	196,500	NA
Ni	2.7	436	--	--	430	--	--	--	433	NA
P	8.9	1,230	--	--	1,240	--	--	--	1,240	NA
Pb	8.9	178		--	178	--	--	--	125	NA
U	180	180	U	--	180	U	--	--	180	NA
B	4.5	103	--	--	130	--	--	--	120	NA
Cu	2.2	19	J	--	19	J	--	--	19	NA
Mn	4.5	13	J	--	12	J	--	--	13	NA
Mo	4.5	53	J	--	53	J	--	--	53	NA
Nd	8.9	36	J	--	36	J	--	--	36	NA
Si	44	187	J	--	187	J	--	--	187	NA
Sr	1.3	2	J	--	2	J	--	--	2	NA
Zr	4.5	9	--	--	8	--	--	--	9	NA
TIC	65	12,400	--	11,800	12,000	--	--	--	12,070	3.3
TOC	170	25,500	--	25,700	27,100	--	--	--	26,100	6.1
Cl^{-}(g)	630	4,300		4,000	6,810	--	--	--	5,040	45
F^{-} g)	630	630	U	630	630	U	--	--	NA	NA
NO_2^{-} g)	1,500	89,200	--	84,100	87,500	--	--	--	86,930	1.9
NO_3^{-} g)	3,800	193,000	--	199,000	199,000	--	--	--	197,000	3.1
PO_4^{3-} g)	1,300	5,580	--	5,260	6,390	--	--	--	5,740	14
SO_4^{2-} g)	1,300	14,200	--	13,600	14,400	--	--	--	14,070	1.4

Table 3.2. Con't

Analyte	MDL / MDA (µg/mL)	AN-102 15°C Sample A (µg/mL)	Data Flag [a]	AN-102 15°C Sample A Duplicate (µg/mL)[b]	AN-102 15°C Sample B (µg/mL)	Data Flag	AN-102 15°C Sample B Duplicate (µg/mL)	Data Flag	Sample A and B Mean[c] (µg/mL)	RPD[d]
Hydroxide	170	4800	--	4300	3900	--	4400	--	4,350	21
Wt% Dissolved Solids	--	51.6	--	--	51.5	--	--	--	51.6	0.2
Density g/mL	--	1.437	--	--	1.438	--	--	--	1.438	0.1
		(µCi/mL)			**(µCi/mL)**		**(µCi/mL)**		**(µCi/mL)**	
^{90}Sr	7E-01	6.1E+01	--	--	6.3E+01	--	5.7E+01	--	6.0E+01	5.5
^{99}Tc[SB]	6E-06	1.7E-01	--	--	1.7E-01	--	--	--	1.7E-01	NA
^{137}Cs	5E-02	3.9E+02	--	--	3.8E+02	--	--	--	3.8E+02	NA
$^{239/240}$Pu	2E-04	5.6E-03	--	--	5.7E-03	--	5.5E-03	--	5.6E-03	1.8
^{241}Am	2E-01	1.2E-01	--	--	1.2E-01	--	1.16E-01	--	1.2E-01	2.2
^{60}Co	2E-03	8.7E-02	--	--	8.5E-02	--	--	--	8.6E-02	NA
^{154}Eu	9E-03	2.2E-01	--	--	2.2E-01	--	--	--	2.2E-01	NA
^{155}Eu	1E-01	1.2E-01	--	--	1.1E-01	--	--	--	1.2E-01	NA
^{238}Pu [f]	3E-04	1.4E-03	--	--	1.2E-03	--	1.6E-03	--	1.4E-03	14
^{242}Cm[f]	2E-04	4.4E-04	--	--	5.3E-04	--	4.4E-04	--	4.7E-04	6.4
$^{243/244}$Cm[f]	2E-04	5.1E-03	--	--	5.1E-03	--	4.8E-03	--	5.0E-03	4.0

(a) In the Data Flag column a "- -" indicates there was no data flag or that the data was good.

(b) Analytes in the Sample Columns marked "- -" were not measured in duplicate.

(c)The Mean values were calculated for Sample A and B and duplicate values.

(d) The RPD values were calculated for Sample A and Sample B only where a duplicate was run.

(e) NA indicates that a Mean or RPD calculation was not required because the analyte concentration was below detection limits, or because no duplicate was run.

(f) The mean difference test calculated that uncertainties in the duplicate measurements fell in the 95% confidence level

(g) F^-, Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, and SO_4^{2-} results report only results above the EQL; therefore, the EQL is presented in this column. For all other analytes, the MDL is presented.

(SB) Standards Bias, see Appendix F.

3.5 Data Limitations for the Supernatant Analytes at 50°C

The reproducibility in the analyte concentrations reported in Table 3.3 is typically reported to the third significant figure. The data as presented have been converted from the reported data in µg/g to µg/mL by the supernatant density of 1.44 g/mL at 50°C. The raw data are reported from the analytical station with error flags to identify analyte values that may be suspect. The flags are identical at both temperatures for each analyte. Each flag is considered below.

- U – Undetected.
    1) Ba, total U, and fluoride were not detected in the supernate above the MDL/MDA and therefore, were flagged with a U.
- B – Blank Contamination.

1) Blank contamination was observed for Fe. The associated preparation blank 01-1624-B had a concentration of 1.36 µg/ml, exceeding 5% of the measured values. Reported values in the sample could be biased high.

2) Blank contamination was observed for B due to the leaching of the borosilicate glass sample vials. The concentration of the associated preparation blank was 50.7 µg/ml, exceeding 5% of the measured values. Reported values in the sample could be biased high.

3) Blank contamination was observed for Si due to the leaching of the borosilicate glass sample vials. The concentration of the associated preparation blank was107 µg/ml, exceeding 5% of the measured values. Reported values in the sample could be biased high.

- J – Estimated Value.

1) Cr, Cu, Mn, Mo, Nd, and Sr were detected above the MDL but below the EQL. The reported values are estimated (J).

2) The duplicate analysis for samples AN102-50A and AN102-50B for hydroxide resulted in an RPD of 34% and 19%, respectively; exceeding the QC criteria of 15%. The associated reported results are estimated (J).

3) The phosphate data at 50°C should be discarded. Examination of the phosphate data indicated that a 100X dilution of the sample resulted in detection of a greater quantity phosphate than a 10X dilution. The result may be explained if insoluble phosphate was present prior to dilution or if phosphate was tied up as colloidal material, which, in either case, solubilized upon dilution.

- X – Estimated Value – QC Criteria not met.

1) The blank spike recovery for K was 78%, and was outside the requirement of 80-120%. The values reported for K in the samples could be biased low, and are therefore flagged as estimated value (X).

Table 3.3. Temperature Dependence Experiment—Concentrations in Supernatants at 50°C

Analyte	MDL / MDA (µg/mL)	AN-102 50°C Sample A (µg/mL)	Data Flag(a)	AN-102 50°C Sample A Dup (µg/mL)(b)	Data Flag	AN-102 50°C Sample B (µg/mL)	Data Flag	AN-102 50°C Sample B Dup (µg/mL)	Data Flag	Sample A and B Mean, (µg/mL) (c)	RPD(d)
Al	5.5	11,900	--	--		11,900	--	11,700	--	11,800	1.7
Ba	0.9	0.9	U	--		0.9	U	0.9	U	NA	NA(e)
Ca	23	512	--	--		509	--	479	--	494	6.1
Cd	1.4	63	--	--		62	--	62	--	62	0
Cr	1.8	192	J	--		189	J	186	J	188	1.6
Fe	2.3	29	B	--		26	B	26	JB	26	0.0
K	180	2,300	X	--		2,300	X	2,200	X	2,250	4.4
La	4.6	16	J	--		16	J	16	J	16	0
Na	14	198,000	--	--		199,500	--	201,000	--	200,300	0.7
Ni	2.8	429	--	--		426	--	421	--	424	1.2
P	9.2	2,020	--	--		2,000	--	2000	--	2,000	0
Pb	9.2	174	--	--		175	--	172	--	174	1.7

Table 3.3. Con't

U	185	185	U	--		185	U	185		NA	NA
B	4.6	223	B	--		233	B	233	JB	233	0
Cu	2.3	19	J	--		17	J	17	J	17	0
Mn	4.6	9	J	--		9	J	9	J	9	0
Mo	4.6	53	J	--		53	J	52	J	53	1.9
Nd	9.2	35	J	--		33	J	33	J	33	0
Si	46	800	B	--		1,000	B	900	JB	950	10
Sr	1.4	2	J	--		2	J	2	--	2	0
Zr	4.6	9	--	--		9	--	9	--	9	0
TIC	65	12,000	--	--		11,800	--	--	--	NA	NA
TOC	170	27,200	--	--		26,600	--	--	--	NA	NA
Cl^{-}(g)	130	4,300	--	--		4,030	--	4,500	--	4,265	11
F^{-}(g)	130	130	U	--		130	U	130	U	NA	NA
NO_2^{-}(g)	2,500	94,000	--	--		92,800	--	97,900	--	95,350	5.3
NO_3^{-}(g)	2,500	235,000	--	--		235,000	--	248,000	--	241,500	5.4
PO_4^{3-}(g)	250	700	J	--		1,200	J	800	J	1,000	40
SO_4^{2-}(g)	250	14,900	--	--		14,800	--	15,700	--	15,250	5.9
Hydroxide	170	3800	J	5400		3600	J	4300	--	3,950	18
Wt% Dissolved Solids	--	53.0(f)	--	--		52.3	--	--	--	53(f)	1.3
Density g/mL	--	1.449	--	--		1.442	--	--	--	1.445(f)	0.5
		(µCi/mL)				**(µCi/mL)**		**(µCi/mL)**			
^{90}Sr	7E-01	5.7E+01	--	--		6.2E+01	--	5.9E+01	--	5.9E+01	5.0
^{99}Tc(SB)	6E-06	1.7E-01	--	--		1.6E-01	--	1.6E-01	--	1.6E-01	0
^{137}Cs	5E-02	3.6E+02	--	--		3.5E+02	--	3.6E+02	--	3.6E+02	2.8
$^{239/240}$Pu	2E-04	5.5E-03	--	--		5.5E-03	--	5.3E-03	--	5.4E-03	3.7
^{241}Am	1E-01	1.33E-01	--	--		1.3E-01	--	1.30E-01	--	1.3E-01	0
$^{243/244}$Cm	2E-03	6.9E-03	--	--		5.7E-03	--	5.8E-03	--	6.1E-03	1.7
^{242}Cm(f)	9E-03	6.0E-04	--	--		5.7E-04	--	5.1E-04	--	5.6E-04	11
^{238}Pu	1E-01	1.7E-03	--	--		1.5E-03	--	1.2E-03	--	1.5E-03	22
^{60}Co	3E-04	8.3E-02	--	--		7.8E-02	--	8.1E-02	--	8.1E-02	3.8
^{154}Eu	2E-04	2.2E-01	--	--		2.1E-01	--	2.1E-01	--	2.1E-01	0
^{155}Eu	2E-04	9.2E-02	--	--		8.5E-02	--	9.1E-02	--	8.9E-02	6.8

(a) In the Data Flag column a "- -" indicates there was no data flag or that the data was good.
(b) Analytes in the Sample Columns marked "- -" were not measured in duplicate.
(c) The Mean values were calculated for Sample A and B and duplicate values.
(d) The RPD values were calculated for the Sample B and its duplicate only.
(e) NA indicates that a Mean or RPD calculation was not required because the analyte concentration was below detection limits, or because no duplicate was run.
(f) The mean difference test calculated that uncertainties in the duplicate measurements fell in the 95% confidence level.
(g) F^-, Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, and SO_4^{2-} results report only results above the EQL; therefore, the EQL is presented in this column. For all other analytes, the MDL is presented.
(SB) Standards Bias, see Appendix F.

3.6 Data Limitations for Relevant Supernatant Analytes as Stated by Urie et al. (2002)

Table 3.4 is a subsection of data that has been transposed directly from Urie et al (2002), Table 8.4. No changes were made in data flagging or other data reporting. This section has been provided to provide a complete picture of the analytical results for the measurements taken at 25°C.

Again, for most analytes, the precision in duplicate analyte concentrations reported in Table 3.4 is reasonable. The raw data are reported from the analytical station with error flags to identify analyte values that may be suspect. Each flag was discussed by Urie and is considered below.

1) Several analytes were flagged with a J. As the code definitions suggest, the error in these analytes exceed 15%. Iron was additionally found in the analytical blank at a concentration that was comparable to the measured sample value.
2) Several analytes are flagged with a U. As listed in the code definitions, these analytes were not detectable in the supernatant.
3) The reported fluoride results represent the summation of fluoride, acetate, and formate concentrations because these were not resolvable on the inorganic anion analysis IC system. Thus the F may be biased high. Fluoride, reported with a U data flag, was below the detection limit. The fluoride results should be considered the upper bound concentration.
4) A QC violation was reported for the supernatant sodium concentration. The RPD required for Na was <3.5%; the supernatant RPD value obtained for Na was 4.3%. The serial dilution for this analyte was not measured
5) A QC violation was reported for the potassium supernatant concentration.
6) The pertechnetate QC (process duplicate and MS) failed, so this analysis was compromised.
7) The IC phosphate results for the supernatant were reported as less than the MDL (i.e., 5,000 μg/mL based on the dilution required at the IC). This result is consistent with the ICP-AES phosphorous results (average 1,800 μg/mL); i.e., the ICP-AES result on a phosphate basis is about 5600 μg/mL).
8) A QC deficiency is reported for hydroxide.
9) The analytical value for ^{155}Eu was flagged where the detection limit was only slightly above the MRQ value of 9.E-2 μCi/ml. Silicon and boron concentrations are cited with multiple flags, likely due to leaching of the borosilicate glass sample vials. This leaching also occurred in the process blanks to some extent.
10) The LCS and matrix spike recoveries were 96 to 107% for both Pu and Am. RPD and RSD values were generally quite good except for $^{243/244}$Cm where the process blank appears to have been contaminated in the hot cell. However, the RPD value for this value was only 7%.

Table 3.4. Solubility Temperature Dependence Experiment—Concentrations in Supernatant at 25°C(a)

Analyte	MDL (μg/mL)	AN-102 25°C Sample A (μg/mL)	Data Flag(b)	AN-102 25°C Sample A Dup. (μg/mL)	Data Flag	Mean (μg/mL)	RPD
Al	8.2	12,100	--	12,400	--	12,250	2.4
Ba	1.4	1.4	U	1.4	U	NA	NA(d)
Ca	34	465	--	513	--	489	9.8
Cd	2.0	61	--	62	--	62	1.8
Cr	2.7	216	--	213	--	215	1.4
Fe	3.4	38	JB	37	JB	37	0.8
K	270	2,000	JX	1,960	JX	1,980	2.0
La	6.8	160	J	15	J	16	6.5
Na	41	180,000	X(c)	188,000	X(c)	184,000	4.4
Ni	4.1	419	--	413	--	416	1.4
P	14	1,800	--	1,830	--	1,820	1.6
Pb	14	188	J	183	J	186	2.7
U	270	270	U	270	U	NA	NA
B	6.8	78	B	82	B	80	5.0
Cu	3.4	23	J	24	J	24	4.3
Mn	6.8	17	J	16	--	17	6.1
Mo	6.8	53	J	52	J	53	1.9
Nd	14	33	J	30	--	32	9.5
Si	68	290	JBX	180	JBX	235	47
Sr	2.0	2.0	J	2.0	--	2.0	0
Zr	6.8	8.4	J	7.9	J	8	6.0
TIC	140	10,900	--	11,000	--	10,900	0.9
TOC	140	29,400	--	29,100	--	29,250	1.0
Cl^-	2,500	4,780	--	4,860	--	4,820	1.7
F^-	2,500	2,500	U	2,500	U	NA	NA
NO_2^-	5,000	85,900	--	84,800	--	85,400	1.3
NO_3^-	5,000	223,000	--	219,000	--	221,000	1.8
PO_4^{3-}	5,000	5,000	U	5,000	U	NA	NA
SO_4^{2-}	5,000	16,800	--	16,900	--	16,90	0.6
Hydroxide	170	4300	X	NM		NA	NA
Wt% Dissolved Solids	--	53.5	--	52.6	--	53.1	2.1
Density, g/mL	--	1.436	--	1.438	--	1.437	0.139
	(μCi/mL)	(μCi/mL)		(μCi/mL)		(μCi/mL)	
^{90}Sr	7E-01	5.58E+01	--	5.86E+01	--	5.72E+01	5
^{99}Tc(SB)	6E-06	0.148(e)	--	--	--	NA	NA
^{137}Cs	5E-02	3.66E+02	--	3.71E+02	--	3.69E+02	1.4
^{241}Am	2E-04	1.49E-01	--	1.52E-01	--	1.51E-01	2
$^{239/240}$Pu	2E-04	6.00E-03	--	5.80E-03	--	5.90E-03	3.4
^{60}Co	2E-03	8.58E-02	--	8.39E-02	--	8.49E-02	2.2
^{154}Eu	9E-03	2.30E-01	--	2.31E-01	--	2.31E-01	0.4
^{155}Eu	1E-01	1.0E-01	U	1.0E-01	U	NA	NA

Table 3.4. Con't

Analyte	MDL (µg/mL)	AN-102 25°C Sample A (µg/mL)	Data Flag[(b)]	AN-102 25°C Sample A Dup. (µg/mL)	Data Flag	Mean (µg/mL)	RPD
^{238}Pu	3E-04	1.66E-03	--	1.63E-03	--	1.65E-03	1.8
^{242}Cm	2E-04	6.5E-04	--	6.0E-04	--	6.3E-04	7.8
$^{243/244}$Cm	2E-04	6.5E-03	B	6.9E-03	B	6.7E-03	6

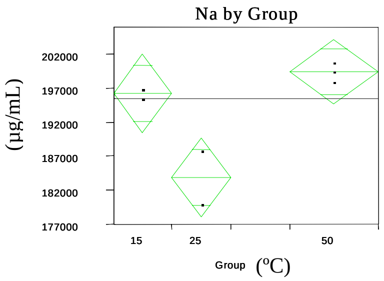
(a) Data from Urie et al. 2002.
(b) In the Data Flag column a "--" indicates there was no data flag for that analyte
(c) The RPD required for Na was <3.5%; the supernatant RPD value obtained for Na was 4.3%. The serial dilution for this analyte was not measured.
(d) NA indicates that a Mean or RPD calculation was not required because the analyte concentration was below detection limits, or because no duplicate was run.
(e) ^{99}Tc measured by ICP-MS
(SB) Standards Bias, see Appendix F.

3.7 Discussion: Component Distribution Versus Temperature

The Test Specification for this work requested PNWD to evaluate the solubility of the solids present in the AN-102 sample at two temperatures, 15°C and 50°C (Johnson 2001). These results were to be compared with results obtained by Urie et al. (2002) at 25°C. Our initial evaluation of these data revealed many instances where the analytical data from Urie et al. did not correlate well with the 15° and 50°C experimental results reported here. Representative samples of these comparisons are plotted (µg/mL versus temperature) in Figure 3.2 for aluminum, sodium, potassium, nitrite, TOC, TIC, sulfate, and manganese. Figure 3.3 plots the nitrate, total phosphorous, and phosphate concentrations, for which the greatest changes with temperature were observed. For a large number of supernatant analytes, listed in Tables 3.2 through 3.4, it is clear there is little change with temperature. In fact, for most cases, and as can be seen in Figure 3.2, the data appear to be centered on a mean rather than indicative of a clear trend with change in temperature. The exceptions are nitrate, phosphate, and phosphorous. The nitrate concentration increases with temperature about 21%. The phosphate/phosphorus pair display opposite trends with temperature.

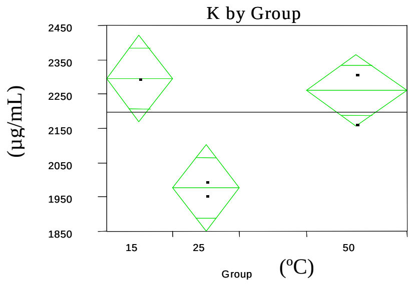
The variability in the phosphate replicate data, the B flag (noting blank contamination), the greater sensitivity of the ICP-AES for total phosphorus measurement, and the good reproducibility of the phosphorus data indicate that the total phosphorus data rather than phosphate analyses are the appropriate metric to gain insight as to phosphorus distribution as a function of temperature.

The range in the total phosphorous concentration with temperature is, about 61%. The increase may be in part due to coincidental error in the measurement. However, the total phosphorus and nitrate values are not flagged, and the sign of the concentration change is in agreement with our conventional understanding of nitrate and phosphate solubility. Little is known about the total phosphorus distribution in the tank solids. Species containing phosphates, pyrophosphates, double phosphate salts (Mason and Ashcroft 1989), and organic phosphorus (Urie et al. 2002) may all be present. Double phosphate salts are known and have been characterized in Hanford tank solids (Herting 2000). The observed changes in the total phosphorus concentration may be subsidized by dissolution of such salts with increased temperature. The change in concentration of the nitrate and total phosphorus concentrations are addressed further below.


Na by Group

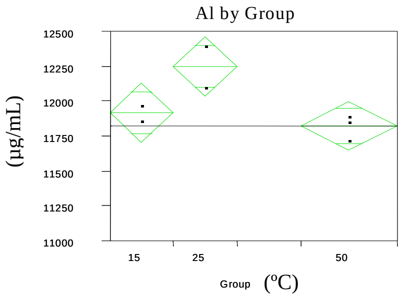
Means for Oneway Anova

Temp	Mean	Std Error
15	196280	2315.6
25	184000	2315.6
50	199500	1890.6


K by Group

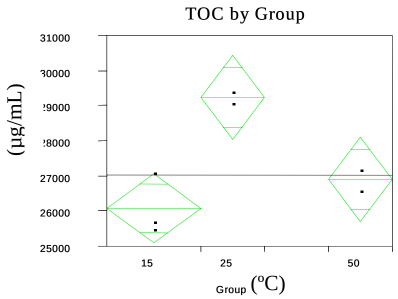
Means for Oneway Anova

Temp	Mean	Std Error
15	2301.00	50.041
25	1980.0	50.041
50	2264.67	40.858


Al by Group

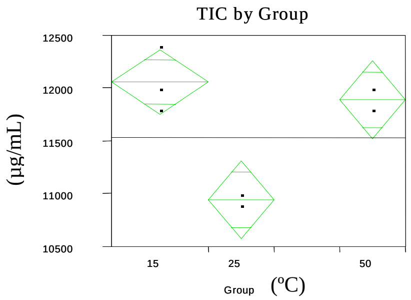
Means for Oneway Anova

Temp	Mean	Std Error
15	11920.0	85.430
25	12250.0	85.430
50	11825.3	69.753


TOC by Group

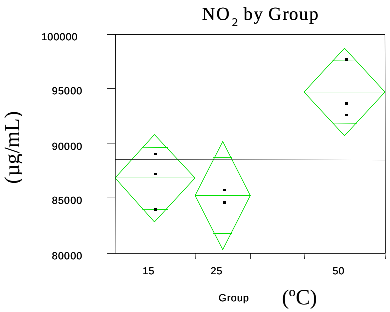
Means for Oneway Anova

Temp	Mean	Std Error
15	26100.0	386.87
25	29250.0	473.81
50	26900.0	473.81


TIC by Group

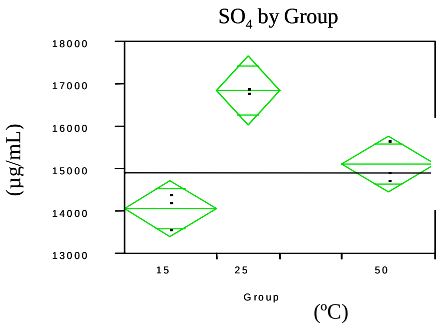
Means for Oneway Anova

Temp	Mean	Std Error
15	12066.7	120.19
25	10950.0	147.20
50	11900.0	147.20


NO_2 by Group

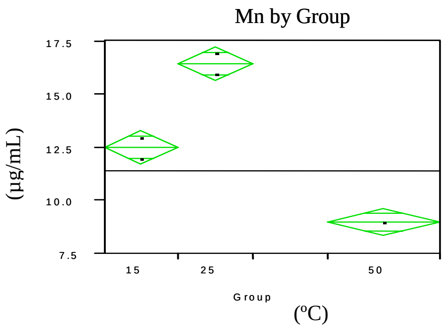
Means for Oneway Anova

Temp	Mean	Std Error
15	86938.7	1658.6
25	85350.0	2031.3
50	94810.0	1658.6


SO_4 by Group

Means for Oneway Anova

Temp	Mean	Std Error
15	14073.3	278.97
25	16850.0	341.67
50	15117.3	278.97


Mn by Group

Means for Oneway Anova

Temp	Mean	Std Error
15	12.5000	0.31623
25	16.5000	0.31623
50	9.0000	0.25820

Figure 3.2. Relevant Analyte Concentrations[a] at 15°, 25°, and 50°C [a]

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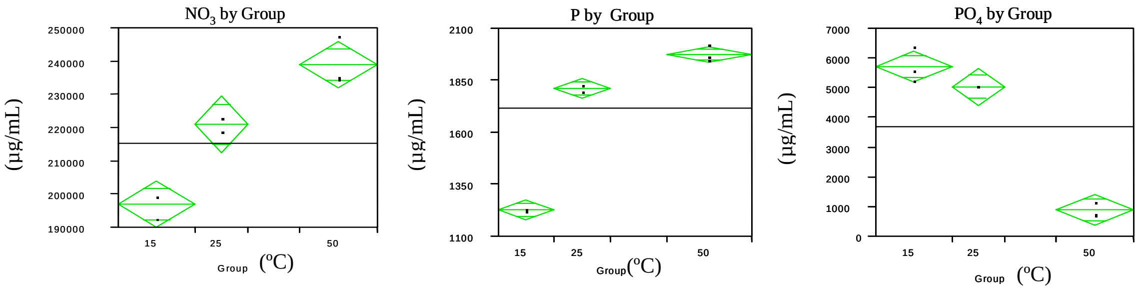
[a] The vertical span of the diamond indicates the 95% confidence interval, and the center horizontal line within the diamond indicates the group mean. Horizontal overlap marks drawn above and below the mean provide a visual means to determine whether groups with equal sample size are statistically significant. If the y-axis projection of two diamonds intersects within the overlap marks then the data are not statistically different at the 95% confidence level.



Means for Oneway Anova

Temp	Mean	Std Error
15	197160	2871.9
25	221000	3517.4
50	239255	2871.9

Means for Oneway Anova

Temp	Mean	Std Error
15	1232.00	18.479
25	1815.00	18.479
50	1980.67	15.088

Means for Oneway Anova

Temp	Mean	Std Error
15	5743.67	218.20
25	5000.00	267.24
50	885.33	218.20

Figure 3.3. Supernatant Analyte Concentrations that Changed Significantly Between 15°C and 50°C[a]

The random appearance of the juxtaposed data in Figure 3.2 likely arose from sample handling and, to a lesser extent, accuracy in the analytical measurement. Differences in sample process conditions probably obscure legitimate changes in supernatant composition with temperature. These conditions include:

1. Processing of supernatant samples, detailed by Urie et al., was accomplished by centrifuging followed by decanting the supernatant. In this report, the maintenance of a constant temperature during liquid sampling was important so that liquid samples were pulled at temperature from the settled sample and filtered. The filtering step used in the temperature study, however, did not go well. The difficulty in filtering might be due in part to colloidal material plugging the filter. The filtering step may have played a critical role with regard to accuracy. At 15°C, soluble components would remain soluble (as the syringe slowly heated) over the duration of the filtering. At 50°C, however, soluble analytes might precipitate in the syringe or on the filter as the solution temperature dropped over the period of filtering.
2. Analytical measurements that lose information due to filtration of precipitates, which formed over time in analytical samples.
3. Evaporative sample loss under variable temperature and ventilation conditions in the hot cell.
4. Normal analytical uncertainty arising from measurement of complex mixtures.

The horizontal span of the diamonds is proportional to the number of measurements made at that temperature. Here the numbers of measurements are generally only 2 or 3, so the diamond widths are not dramatically different. If one group instead had 10 measurements and another 100, the diamond for the latter would be 10 times as wide. Further explanation of the plots is stated at the beginning of Appendix A.

3.8 An Alternative Approach to Temperature Versus Solubility

Due to the scattered look of the combined Urie/Burgeson data, an alternative analysis was undertaken based on observed changes the 15° to 50°C data only. It is important to note that the extensive compositing of AN102 slurry by Urie et al. and the good accuracy presented from numerous replicate samplings likely outweigh the accuracy in the data presented herein. However, the samples herein were prepared and (batch) analyzed at the same time and resulted in analytical data that are perhaps less susceptible to variation caused by sample processing and handling. This analysis then uses only two temperatures to formulate conclusions; an approach that clearly has the potential to prejudice analyte concentrations with variation in temperature.

This evaluation identified 12 analytes whose concentration change over the temperature range of 15° to 50°C was statistically significant. Table 3.5 reports the identified analytes and provides the percent concentration change observed for each analyte. The analysis used the measured reproducibility of an analyte at each temperature to calculate a probability (p-value) that the observed variation with temperature was significant. Table 3.5 lists p-values from this analysis in the third column. For a p-value of less than 0.05, the concentration change for that analyte is statistically significant. The final column considers details such as the flagging of the data. This final column quite regularly disagrees with the statistical test due to arguments stated in the ensuing discussion.

Table 3.5. Analytes that Displayed Statistically Significant Concentration Variations over the Temperature Range 15° to 50°C

Analyte	Relative % Change in Concentration[a]	p-value[b]	Statistically Relevant Temperature Effect?	Relevant[c] Temperature Effect?
B	95	0.0019	Yes	No
Mn	-26	0.0026	Yes	Yes
P	61	0.0001	Yes	Yes
NO_2^-	9	0.0220	Yes	No
NO_3^-	21	0.0009	Yes	Yes
SO_4^{2-}	7	0.0495	Yes	No
^{241}Am	17	<0.0001	Yes	No
^{137}Cs	-7	0.0177	Yes	No
$^{243/244}$Cm	24	0.0472	Yes	No
^{60}Co	-6	0.0494	Yes	No
^{155}Eu	-22	0.0075	Yes	No

(a) A negative sign indicates the concentration at 50°C was less than that at 15°C.
(b) A statistical value of less than p=0.050 is identified as significant.
(c) Based on known analytical behaviors, chemistries and other factors.

The observed shifts in the boron (and silicon) concentrations were due to leaching from the borosilicate glass vials during acid digestion of the samples and are often observed with these types of measurements.

The analytical blanks were also found to contain significant concentrations of boron and silicon, as referred to in the Data Limitations section. The boron concentration data cannot be used to infer any result with regard to change in temperature. The changes indicated for nitrite, sulfate, cobalt-60, and cesium-137 concentrations were below 10%, and whether these changes are real is hidden in the reported uncertainty of the measurement.

The statistical analysis reported that the europium-155 concentration decreased by 22%, the americium-241 concentration increased by 17%, and the curium-243/244 concentration increased by 24% between 15° and 50°C. Changes in the europium-155 concentration were not reflected in the europium-154 concentration and possibly correlate to counting errors associated with the presence of cesium-137. Moreover, the europium-155 concentrations are B-flagged, indicating that the analytical blank europium-155 concentration may contribute to the measured value. The apparent increase in the curium-243/244 concentrations at 15° and 50°C are flagged for low matrix spike recoveries. This suggests the presence of an interferent, and the apparent increase in the curium-243/244 concentrations should be regarded with suspicion. Barring a sampling error in the 15°C data, the increase for americium-241 appears to be real. The magnitudes of these changes for americium can be challenged on a chemical basis. The lanthanum, neodymium, and europium-154 concentrations are fairly constant between 15° and 50°C. It is difficult to perceive why americium would be different chemically from these other, chemically similar trivalent species. However, the solubilities of these elements are different. The solubility of ^{241}Am(III) in 5$\underline{M}$ hydroxide is about $1.6 \times 10^{-6}\underline{M}$. At the concentration indicated, about $10^{-13}\underline{M}$, it is reasonable that some americium could dissolve at higher temperature. The trivalence of americium in the tank solids is assumed, but Am(IV,V,and VI) hydroxo compounds are stable in alkali media. Higher valence oxides may have been formed in the acid processing of waste, and the hydroxides formed by addition of caustic. Little is known of the solubilities of these compounds (Peretrukhin 1996).

The nature of changes in manganese, phosphorous, and nitrate concentrations in the supernatants is better discussed by considering the solids data. First, a correction is required for the contribution to the measured concentration of any component due to the component's presence in the interstitial liquid. The correction is applied to the total solids to correct for analytes present in the interstitial liquids and has been outlined by Urie et al. (2002). The final column in Table 3.6 lists the calculated percentages of analyte in the Urie et al. undissolved solids. The correction confirmed that the percentage of chloride, fluoride, nitrite, and nitrate are near zero in the AN-102 wet undissolved solids. The correction implies that a significant increase in the analyte's solution concentration as a function of temperature is unlikely. On the other hand, the correction as applied to the phosphate and total phosphorus data, suggest that phosphate is present in the undissolved solids.

Table 3.6. Interstitial Correction of Anion Concentrations in the Solids

Analyte	WCS[(a)] Urie et al. (µg/mL)	Applied Correction Urie et al. (µg/mL)	Percent Solids Urie et al.
Cl^-	3330	550	10
F^-	3800	<DL	<DL
NO_2^-	55100	5,818	6
NO_3^-	133000	5,020	2
PO_4^{3-}	8175	550	51
P	1400	358	16
SO_4^{2-}	16500	6,730	29
(a) Wet centrifuged solids.			

The full, corrected wet centrifuged solids data set is reported along with the corrected undissolved solids data from Urie et al. in Appendix C. The total phosphorus values are included in Table 3.6 and are used in further discussion because they have more credibility than the reported phosphate data.

The statistical analysis indicates that total phosphorus concentration increased 61% between 15° and 50°C. The interstitial liquid correction to the Urie et al. solids data suggests that between 16% (as total phosphorus, P) to 51% (as phosphate) of the phosphate was in the wet centrifuged solids. If 100% solubilization of this total phosphorus (380 µg/mL) remaining in the undissolved solids were to dissolve, a change in the Urie et al. supernate concentration of roughly 17% would result. Because of the general disagreement in the total phosphorus/phosphate data, the magnitude of change cannot be ascertained. The observed 61% change may be excessive, but the total phosphorus concentration (phosphate) appears to be a valid function of temperature. Less soluble double salts, such as $Na_7F(PO_4)_2 \bullet 19H_2O$ or Na_3FSO_4 (Herting 2000), may be present in the undissolved solids and aid the concentration changes with temperature. Dissolution of these salts would also increase the sodium concentration. However, the calculated sodium value from this dissolution at 202,000 µg/mL[(a)] and the measured value at 200,000 µg/mL (50°C data) are too similar to conclude that this mechanism for solubilization of the phosphate is valid.

The change in the supernatant nitrate concentration can be considered in similar fashion. The reported 21% increase, or 42,000 µg[(b)] nitrate, would arise from dissolution of nitrate in the solids. The interstitial liquid correction to the solids, shown in Table 3.6, provides for a near-zero percent concentration of nitrate in the Urie and Burgeson undissolved solids; therefore, the magnitude of this reported change can be considered questionable. The change in nitrate concentration would additionally be accompanied by dissolution of sodium and would increase the sodium concentration from 196,000 to 211,000 µg/mL in the supernatant. This is less than a 10% change and therefore is likely below the certainty in the nitrate analyses. The measured sodium concentration in the supernatants between 15° and 50°C is fairly constant. The presence of a less soluble form of nitrate, the double sodium sulfate salt $Na_3(NO_3)(SO_4)$ (Herting

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[a] A 61% increase is converted to the phosphate basis with a resulting equivalency of 6,100 µg/mL PO_4^{3-}.
6100 µg/mL x [7 mol Na /2 mol PO_4^{3-}] x [22.989 g/mol Na/94.9 g/mol PO_4^{3-}] = 5,200 µg/mL increase in sodium. Therefore we would expect to measure an increase of only 197,000 + 5,200 = 202,000 µg/mL sodium in the 50°C supernatant.

[b] 239000 (50°C) – 197000(15°C) = 42000 µg/mL NO_3^-.

2000), in the undissolved solids might also add to the reported 21% increase in nitrate concentration. Again, the sodium concentration would also increase to approximately 250,000 μg/mL, and this is not observed.

The statistical analysis in Table 3.5 reports a 26% decrease in the supernatant manganese concentration. The manganese concentrations at 15°C (1.3 μg/mL) and 50°C (1.8 μg/mL) are J-flagged, indicating uncertainties exceeding 15% in the reported concentrations. The change in manganese concentration exceeds this uncertainty, and the replicate analyses have good reproducibility. A species responsible for Mn(IV) solubility in the supernatant needs to be invoked to account for the presence of manganese in the supernatant. Complexed Mn(III) or mixed Mn(IV)/Mn(III) species are reported to be preferentially reduced and solubilized by organic complexants (Levason and McAuliffe 1972).

3.9 Conclusion: Component Distribution Versus Temperature

Evaluation of the averaged experimental temperature solubility data indicates that the supernatant concentrations are, for the most part, constant with respect to changes in temperature. An alternative (statistical) analysis of analyte concentrations, based on the temperature extremes, i.e., using the 15° and 50°C data only, resulted in a similar conclusion as the initial evaluation of the combined Urie/Burgeson data set. For some analytes, such as boron and silicon, no estimate can be made with respect to temperature due to experimental artifacts. Table 3.7 lists analytes that had good sample reproducibility, whose reported concentrations were well above stated minimum detection limits, and that demonstrated changes in concentration greater than 15% between 15° and 50°C, and additionally, whose variation with temperature was shown to be statistically significant.

Table 3.7. Supernate Analytes that Displayed Statistically Significant Concentration Variations over 15° to 50°C Temperature Range

Analyte	Statistically Relevant Temperature Effect?	Relative Percent Change in Concentration[a]
Mn	Yes	-26
NO_3^-	Yes	21
P	Yes	60
^{241}Am	Yes	17

(a) A negative sign indicates the concentration at 50°C was less than the concentration at 15°C.

The reported magnitude of the variation in concentration with temperature for Mn, NO_3, PO_4, and ^{241}Am should be regarded with some caution, because the result here is based on only two temperatures. The increase in supernatant americium concentration is not followed by other trivalent analytes whose chemistries are similar; lanthanum, europium, or neodymium, however, the solubilities of these are different and the measured concentration of ^{241}Am is several orders of magnitude below its solubility limit. The manganese, nitrate, and phosphorus concentrations likewise appear to vary with temperature, and these changes are consistent with conventional solubility increase with temperature.

4.0 Experimental: Variation in Component Distribution with Caustic Addition

4.1 Introduction

The intent of this experiment was to examine the effect of caustic addition on the solubility of the solids by analyzing the solids and supernatant of the tank waste slurry after the addition of 3.4 vol% of 19M NaOH per mL of tank waste slurry. The data collected under this experimental testing would be compared to the available AN-102 solid and liquid data (Urie et al. 2002) to understand the effect of caustic addition on the tank waste slurry.

4.2 Experimental

Approximately 50 mL of AN-102 tank waste slurry was transferred to two 125-mL HDPE bottles as described in Section 2. A targeted mass of 1.87 g of 19M NaOH solution was added to each sample bottle. Table 4.1 provides the mass of tank waste slurry and the added sodium hydroxide solution. After addition of sodium hydroxide, the sample bottles were capped and shaken at ambient hot cell temperature (27°C) for 13 days and 5 hours. At the end of this period the slurries were allowed to settle overnight, and then the volume of settled solids was measured. The samples were centrifuged at ~1000g for one hour, and the supernatant was separated from the settled solids to perform volume and mass measurements necessary to determine the weight percent centrifuged solids. After centrifugation, two solid samples, Caustic Solid Sample A and Caustic Solid Sample B, were submitted for analytical characterization. Two supernatant samples, Caustic Sample A and Caustic Sample B, were also submitted for analytical characterization. These samples were not prefiltered.

The settled solids and supernatant samples were acid digested according to procedure PNL-ALO-128, *HNO_3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater*, in the SAL hot cells. Aliquots of the digested subsamples were analyzed by ICP-MS, ICP-AES, total U by kinetic phosphorescence analysis (KPA), and the following radiochemical analyses: total alpha, gamma emitters by GEA, $^{239/240}$Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, $^{243/244}$Cm, and ^{90}Sr.

The supernatant and wet-centrifuged solids were analyzed for the target analytes defined in the Test Specification (Johnson 2001). The analytical processing of the caustic supernatant and wet centrifuged solids are detailed in Figure 4.1. ASR number 6164 provided instructions to the laboratory to successfully complete the analytical and QC requirements defined in the Test Specification.

Table 4.1. Composition of the AN-102 Sample for Caustic Addition Experiments

Sample ID	AN-102 Slurry Mass, g (mL)	19M NaOH Addition g (mL)
Caustic Addition Sample A	79.0118 (54.9)	2.8059 (1.92)
Caustic Addition Sample B	79.2583 (55.0)	2.8081 (1.93)

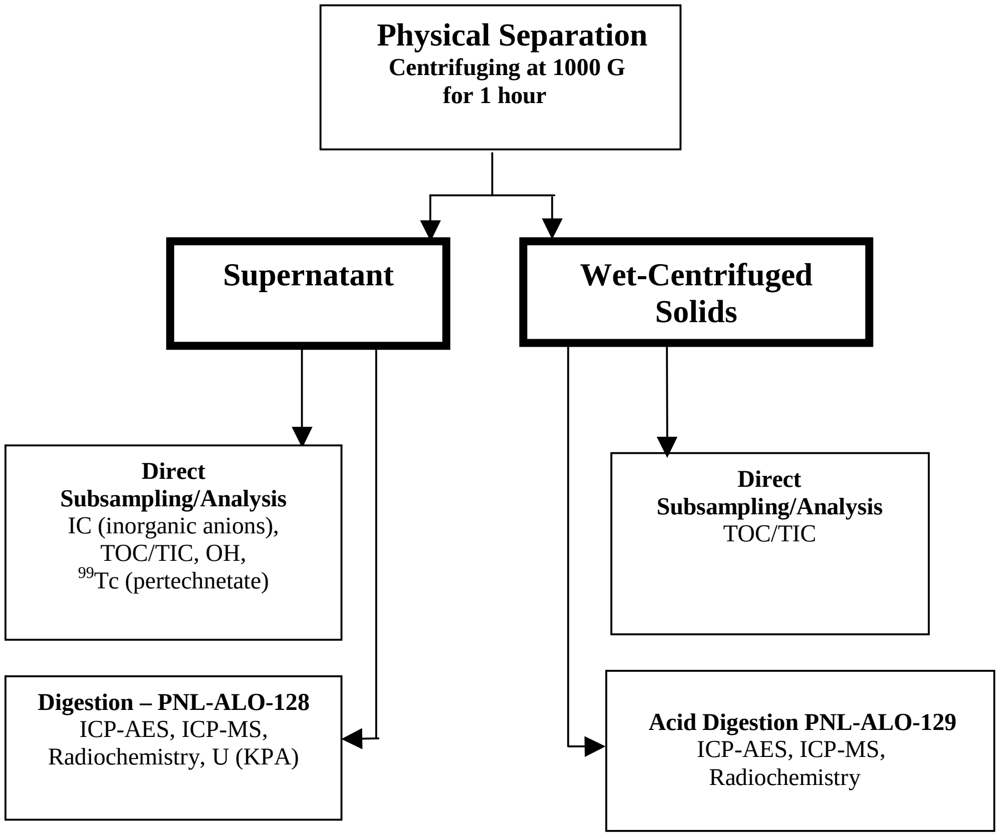

Figure 4.1. Flow Diagram for Analytical Processing of Supernatant and Wet Centrifuged Solids

4.3 Results: Variation in Supernatant Component Distribution with Caustic Addition

The intent of the caustic addition experiment was to measure the effect of hydroxide on the distribution of tank components between supernatant and solid. Both the solid and supernatant compositions were analyzed after adding 0.05 g of 19M NaOH per mL tank waste slurry. This addition increased the hydroxide concentration by about 0.63 meq hydroxide per mL tank waste slurry. The in-depth characterization data from Urie et al. (2002) were already available for the unaltered AN-102 supernatant and solids and are included in this report unchanged to present the reviewer a complete picture of the analytical results. The results for the treated supernatant are reported in µg/mL or µCi/mL, as appropriate, in Table 4.2.

4.4 Data Limitations for Caustic Supernatant Analytes

For most analyte concentrations reported in Table 4.2, the precision is in the third significant figure. The data as presented have been converted from the reported data in µg/g to ug/mL by the supernatant density of 1.437 g/mL. Also, the addition of caustic to samples A and B diluted analyte concentrations by

3.3%. The data as presented have been corrected by this amount. The data are reported from the analytical station with error flags to identify analyte values that may be suspect. Each flag is considered below.

- U – Undetected.
  1) Ba, total U, and fluoride were not detected in the supernate above the MDL/MDA and therefore, were flagged with a U.
- B – Blank Contamination.
  1) Blank contamination was observed for Fe. The associated preparation blank 01-1624-B had a concentration of 1.36 µg/ml, exceeding 5% of the measured values. Reported values in the sample could be biased high.
  2) Blank contamination was observed for Si due to the leaching of the borosilicate glass sample vials. The concentration of the associated preparation blank was 107 µg/ml, exceeding 5% of the measured values. Reported values in the sample could be biased high.
  3) Blank contamination was observed for B due to the leaching of the borosilicate glass sample vials. The concentration of the associated preparation blank was50.3 µg/ml, exceeding 5% of the measured values. Reported values in the sample could be biased high.
- J – Estimated Value.
  1) Ca, La, Pb , Cu, Mo, and Nd, were detected above the MDL but below the EQL. The reported values are estimated (J).
- X – Estimated Value – QC Criteria not met.
  1) The blank spike recovery for K was 78%, outside the requirement of 80-120%. The values reported for K in the samples could be biased low, and are therefore flagged as estimated value (X).

Table 4.2. Supernatant Analyte Concentrations after Caustic Addition

Analyte	**MDL / MDA (µg/mL)**	**Caustic Sample A, (µg/mL)**	**Data Flag**[a]	**Caustic Sample B, (µg/mL)**	**Data Flag**	**Mean (µg/mL)**	**RPD**[c]
Al	10	11,600	--	11,734	--	11,670	1.2
Ba	1.7	1.7	U	1.7	U	NA	NA[b]
Ca	43	459	J	459	J	459	0
Cd	2.6	61	--	60	--	61	1.6
Cr	3.4	191	--	191	--	191	0
Fe	4.3	27	B	25	B	26	7.7
K	340	2,370	X	2,220	X	2,295	6.5
La	8.6	16	J	15	J	16	6.4
Na	26	204,000	--	210,000	--	207,000	2.9
Ni	5.2	419	--	421	--	420	0.48
P	17	1,780	--	1,780	--	1,780	0
Pb	17	178	J	163	J	171	8.8
U	340	340	U	340	U	NA	NA
B	8.6	103	B	103	B	103	0
Cu	4.3	17	J	16	J	17	6.1

Table 4.2. Con't

Analyte	MDL / MDA (μg/mL)	Caustic Sample A, (μg/mL)	Data Flag(a)	Caustic Sample B, (μg/mL)	Data Flag	Mean (μg/mL)	RPD(c)
Mo	8.6	54	J	54	J	54	0
Nd	17	37	J	31	J	34	18
Si	86	142	B	--	--	NA(b)	NA(b)
TIC	65	11,000	--	10,900	--	10,950	0.91
TOC	170	26,800	--	25,800	--	26,300	3.8
$Cl^{-(g)}$	630	4,000	--	3,700	--	3,850	7.8
$F^{-(g)}$	630	630	U	630	U	NA	NA
$NO_2^{-(g)}$	1,300	88,000	--	82,700	--	85,350	6.2
$NO_3^{-(g)}$	3,800	202,000	--	200,000	--	201,000	1.0
$PO_4^{3-(g)}$	1,300	6,200	--	5,900	--	6,050	4.9
$SO_4^{2-(g)}$	1,300	14,000	--	13,000	--	13,500	7.4
Hydroxide	170	13000	--	12500	--	12,750	3.9
Wt% Dissolved Solids		50.6	--	51.3	--	51	1.4
Density g/mL		1.430	--	1.434	--	1.432	0.28
	(μCi/mL)	(μCi/mL)		(μCi/mL)		(μCi/mL)	
^{90}Sr	7E-01	5.7E+01	--	5.8E+01	--	5.7E+01	1.7
^{99}Tc(SB)	6E-06	1.7E-01	--	1.7E-01	--	1.7E-01	0
^{137}Cs	5E-02	3.4E+02	--	3.5E+02	--	3.4E+02	2.9
$^{239/240}$Pu	2E-04	5.1E-03	--	5.5E-03	--	5.3E-03	7.6
^{241}Am (d)	2E-01	1.3E-01	--	1.5E-01	--	1.3E-01	14
^{60}Co	2E-03	7.8E-02	--	7.9E-02	--	7.9E-02	1.3
^{154}Eu	9E-03	2.0E-01	--	2.1E-01	--	2.0E01	4.9
^{155}Eu(e)	1E-01	8.2E-02	--	1.1E-01	--	9.2E-02	29
^{238}Pu	3E-04	1.2E-03	--	1.4E-03	--	1.3E-03	15
^{242}Cm(f)	2E-04	4.2E-04	--	6.9E-04	--	5.6E-04	49
$^{243/244}$Cm(f)	2E-04	4.3 E-3	--	6.5 E-3	--	5.0 E-3	41

(a) In the Data Flag column a "- -" indicates there was no data flag or that the data was good.
(b) NA indicates that a Mean or RPD calculation was not required because the analyte concentration was below detection limits, or because no duplicate was run.
(c) Mean and RPD values were calculated here between Samples A and B, as no duplicates were run.
(d) RPD value was higher than 15% for ^{241}Am but the data passed the mean difference test.
(e) RPD value was higher than 15% for ^{155}Eu but the data passed the mean difference test.
(f) RPD value was higher than 15% for ^{242}Cm and $^{243/244}$Cm, but the data passed the mean difference test.
(g) F^-, Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, and SO_4^{2-} results report only results above the EQL; therefore, the EQL is presented in this column. For all other analytes, the MDL is presented.
(SB) Standards Bias, see Appendix F.

4.5 Variation in Solids Component Distribution With and Without Caustic Addition

The results for the solids after addition of caustic are reported in µg/g or µCi/g, as appropriate, in Tables 4.3 and 4.4.

4.6 Data Limitations for Wet Centrifuged Solid Analytes after Caustic Addition (Table 4.3)

The data are reported from the analytical station with error flags to identify analyte values that may be suspect. Each flag is considered below.

- U – Undetected.

  1) Total Bi, U, As, Be, Sb, Se, and fluoride were not detected in the supernate above the MDL/MDA and therefore, were flagged with a U.

- B – Blank Contamination.

  1) Blank contamination was observed for B due to the leaching of the borosilicate glass sample vials. The concentration of the associated preparation blank was 800 µg/g, exceeding 5% of the measured values. Reported values in the sample could be biased high.

- J – Estimated Value.

  1) Ba, Pb, Sr, Ca, Zr, and Sr was detected above the MDL but below the EQL. The reported values are estimated (J).

  2) The matrix spike for Na was 131%, outside the requirement of 75-125%. Subsequent serial dilutions were within the required 10%. Therefore, the reported values for Na were qualified as estimated (J).

  3) The matrix spike for ^{241}Am was 67%, Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for any inaccuracy of the method on the matrix. The reported results reflect this correction. The reported values for ^{241}Am were qualified as estimated (J).

- X – Estimated Value – QC Criteria not met.

  1) The blank spike recovery for K was 78%, outside the requirement of 80-120%. The values reported for K in the samples could be biased low, and are therefore flagged as estimated value (X).

Table 4.3. Analytical Results for the Wet Centrifuged Solids after Caustic Addition

Analyte	MDL / MDA (µg/mL)	Caustic Solid Sample A (µg/g)	Data Flag [a]	Caustic Solid Sample B (µg/g)	Data Flag	Caustic Solid Sample B Dup. (µg/g)	Data Flag	Mean[f] (µg/g)	RPD[e]
Al	27	37,100	--	37,900	--	38,100	--	37,700	2.1
Ba	4.5	36	J	36	J	36	J	36	0
Bi	45	45	U	45	U	45	U	45	NA[d]
Ca	112	590	--	560	J	560	J	570	5.2
Cd	6.7	66	--	66	--	66	--	66	0
Cr	9.0	2,200	--	2,290	--	2,310	--	2,270	4.0
Fe	11	1,450	--	1,540	--	1,540	--	1,510	6.0
Mn	23	392	--	411	--	415	--	406	4.7
Na	67	280,000	J	283,000	J	285,000	J	282,670	1.1
Ni	14	450	--	460	--	460	--	460	2.2
P	45	2,140	--	2,120	--	2,150	--	2,140	0.94
Pb	45	347	--	350	J	360	J	352	0.86
Si	225	1,130	B	1,100	B	1,100	B	1,110	2.7
Sr	6.7	10	J	10	J	10	--	10	0
U	900	106	U	116	U	-- [b]	--	NA	NA
As	112	112	U	112	U	--	--	NA	NA
Be	4.5	4.5	U	4.5	U	--	--	NA	NA
K	900	2,100	X	2,100	X	--	--	2,100	0
Sb	225	225	U	225	U	--	--	NA	NA
Se	112	112	U	112	U	--	--	NA	NA
Zr	23	68	J	69	J	70	J	69	1.5
TIC	190	11,400	--	12,000	--	--	--	11,700	5.1
TOC	510	28,000	--	24,300	--	--	--	26,150	14
Cl^-	120	2,390	--	2,320	--	2,360	--	2,360	3.0
F^-[h]	120	120	U	61	U	120	U	NA	NA
NO_2^-[h]	2,400	52,400	--	53,600	--	50,000	--	52,000	2.3
NO_3^-[h]	2,400	124,000	--	136,000	--	117,000	--	125,700	9.2
PO_4^{3-}[h]	240	3,800	--	4,400	--	3,300	--	3,830	15
SO_4^{2-}[h]	240	10,800	--	10,900	--	10,900	--	10,870	0.92
Hydroxide	170	NM[c]	--	NM	--	NM	--	NA	NA
Wt% total solids	--	57.5	--	57.7	--	--	--	58	0.35
Wt% centrifuged solids	--	7.4	--	7.2	--	--	--	7	2.7
	(uCi/g)	**(uCi/g)**		**(uCi/g)**		**(uCi/g)**		**(uCi/g)**	
^{137}Cs	5E-02	3.6E+02	--	3.6E+02	--	3.6E+02	--	3.6E+02	0
$^{239/240}$Pu	2E-04	7.1E-02	--	6.1E-02	--	6.4E-02	--	6.5E-02	15
^{241}Am	1E-01	5.9E-01	J	5.9E-01	J	5.1E-01	J	5.9E-01	0
^{60}Co	3E-04	9.2E-02	--	9.2E-02	--	9.3E-02	--	9.2E-02	0
^{154}Eu	2E-04	6.7E-01	--	6.7E-01	--	6.4E-01	--	6.7E-01	0
^{155}Eu[g]	2E-04	3.1E-01	--	2.5E-01	--	3.4E-01	--	3.0E-01	21
^{238}Pu[g]	1E-01	7.0E-02	--	4.0E-02	--	4.0E-02	--	5.0E-02	54

Table 4.3. Con't

^{242}Cm	2E-03	1.7E-03	--	2.0E-03	--	1.7E-03	--	1.8E-03	16
$^{243/244}$Cm$^{(g)}$	2E-03	6.0 E-02	--	4.0E-02	--	3.0 E-02	--	4.6E-02	40

(a) In the Data Flag column a "--" space indicates there was no data flag or that the data was good. Analytes marked "--" were not measured in duplicate.
(b) Analytes marked "--" were not measured in duplicate.
(c) Hydroxide was not measured in the solids.
(d) NA indicates that a Mean or RPD calculation was not required because the analyte concentration was below detection limits, or because no duplicate was run.
(e) RPD values were calculated for Sample B and its duplicate only.
(f) The mean value was calculated for Sample A and B, and the Sample B duplicate.
(g) RPD value was higher than 15% for ^{155}Eu, ^{238}Pu, and $^{243/244}$Cm but the data passed the mean difference test.
(h) F$^-$, Cl$^-$, NO$_2^-$, NO$_3^-$, PO$_4^{3-}$, and SO$_4^{2-}$ results report only results above the EQL; therefore, the EQL is presented in this column. For all other analytes, the MDL is presented.

4.7 Data Limitations for Wet Centrifuged Solid Analytes as Reported in Table 4.4 by Urie et al. (2002)

Table 4.4 is a subsection of data that has been transposed directly from Urie et al (2002), Table 8.6. No changes were made in data flagging or other data reporting. This section has been provided to provide a complete picture of the analytical results for the measurements taken at 25°C. The data flags are discussed below.

- U – Undetected.
  1) Ag, Bi, Be, Sb, Se, and ^{242}Cm are reported below the detection limit.
- B – Blank Contamination.
  1. The chloride concentration in the solids is flagged with a B. Chloride was found in the analytical blank at concentrations comparable to the measured value in the sample.
  2. Pb and Si were also reported with blank contamination
- X - QC violation
  1. A QC violation is reported for the sodium concentration. The RPD required for Na was <3.5%; the supernatant RPD value obtained for Na was 4.3%. The serial dilution for this analyte was not measured.
  2. A QC violation is reported for the potassium supernatant concentration.

While there were no data flags for the IC phosphate or ICP-AES total phosphorus concentrations, the data were disparate and Urie provided this explanation.

> The IC phosphate results on a leach of the wet centrifuged solids averages 8,200 μg/g, compared with the ICP-AES result of 4,300 μg/g (or 1,400 μg/g phosphorus). The acid digestion preparation (for ICP-AES) should provide a better dissolution of the phosphate than the water leach (for IC). The two most likely explanations for the discrepancy: 1) the IC results are biased high due to the presence of co-eluting organic anions or 2) the ICP-AES results are biased low due to precipitation of phosphate (e.g., as zirconium phosphate).

Table 4.4. Results from Urie et al. for Analytes in the Wet Centrifuged Solids Without Added Caustic

Analyte	MDL / MDA (μg/mL)	Solid Sample A (μg/g)	Data Flag $^{(a)}$	Solid Sample A Duplicate (μg/g)	Data Flag	Mean (μg/g)	RPD
Ag	2.1	2.1	UX	2.5	UX	NA	NA $^{(c)}$
Al	5.1	28,000	--	29,000	--	28500	3.5
Ba	0.8	27	--	28	--	27.5	3.6
Bi	10	9	U	10	U	NA	NA
Ca	21	434	--	407	--	421	6.4
Cd	1.3	37	--	39	--	38	5.3
Cr	1.7	1,800	--	1,850	--	1825	2.7
Fe	2.1	1,150	--	1,180	--	1160	2.6
Mn	4.2	250	--	260	--	255	3.9
Na	63	160,000	X	173,000	X	166000	7.8
Ni	2.5	254	--	261	--	258	2.7
P	8.5	1,390	J	1,420	B	1405	2.1
Pb	8.5	245	B	244	J	245	0.4
Si	4.2	190	JB	210	JB	200	10
Sr	1.3	6.1	JB	6.2	J	6.15	1.6
U	170	170	U	170	U	NA	NA
As	21	5.8	JB	7.2	J	6.50	22
Be	0.32	1.3	J	0.98	J	1.14	28
K	170	1,100	JX	1,100	JX	1100	0
Sb	42	42	U	50	U	NA	NA
Se	21	21	U	25	U	NA	NA
Zr	4.2	46	--	47	--	47	2.2
TIC	560	17,100	--	17,600	--	17,400	2.9
TOC	300	25,900	--	25,300	--	25600	2.3
$Cl^{-(d)}$	250	3,520	B	3,140	--	3300	11
$F^{-(d)}$	250	3,900	--	3,700	--	3800	5.3
$NO_2^{-(d)}$	500	57,400	--	52,700	--	55000	8.5
$NO_3^{-(d)}$	500	137,000	--	128,000	--	13300	6.8
$PO_4^{3-(d)}$	500	8,970	--	7,380	--	8170	19
$SO_4^{2-(d)}$	500	17,500	--	15,400	--	16500	13
Wt% total solids		58.5	--	59.3	--	58.9	1.4
Wt% centrifuged solids		(b)		(b)		NA	NA
	(μCi/g)	**(μCi/g)**		**(μCi/g)**		**(μCi/g)**	
^{137}Cs	4E-02	2.16E+02	--	2.16E+02	--	216	0
$^{239/240}$Pu	2E-03	4.16E-02	--	4.17E-02	--	4.17E-02	0.2
^{241}Am	2E-03	4.44E-01	--	4.38E-01	--	4.41E-01	1.4
^{60}Co	3E-03	5.76E-02	--	5.66E-02	--	5.71E-02	1.8
^{154}Eu	9E-03	5.09E-01	--	5.15E-01	--	5.12E-01	1.2
^{155}Eu	2E-01	3.14E-01	--	3.26E-01	--	3.20E-01	3.8
^{238}Pu	2E-03	1.09E-02	--	1.29E-02	--	1.19E-02	17
^{242}Cm	4E-06	4E-06	U	4E-06	U	NA	NA
$^{243/244}$Cm	2E-03	1.81E-02	B	1.62E-02	BJ	1.72E-02	11

Table 4.4. Con't

(a) In the Data Flag column a "--" indicates there was no data flag or that the data was good. Analytes marked NM were not measured

(b) The wt% centrifuged solids was measured on five bottles of homogenized as-received material, but only the average is reported here. This value cannot be compared with the centrifuged solids of the soluble slurry material because the slurry was designed to be approximately 7 vol% settled solids.

(c) NA indicates that the RPD calculation is not required because the analyte concentration was below detection limits.

(d) F^-, Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, and SO_4^{2-} results report only results above the EQL; therefore, the EQL is presented in this column. For all other analytes, the MDL is presented.

4.8 Discussion: Caustic Supernatant and Solids Composition

4.8.1 Supernatant Composition after Caustic Addition

Supernatant analyte concentrations resulting from the addition of caustic are compared with native supernate data in Table 4.5. Several analytes were flagged as not detected and were removed for the comparison.

Chloride, iron, copper, phosphate, sulfate, $^{243/244}$Cm, and ^{238}Pu exhibit changes greater than 15%. The better part of these analytes are flagged and this is discussed below. In Table 4.5, the direction of change is marked with a negative sign if the caustic addition diminished the analyte concentration relative to the native supernatant. The reported decrease in chloride and SO_4^{2-} are likely artifacts of sample handling or of the anion measurement. Conventionally, the solubilities of these anions should increase with dilution. The copper and iron concentrations are flagged in both the Urie and Burgeson data sets as estimates and cannot be used in this evaluation. The iron values in particular are suspect because iron was found in the analytical blank. The phosphate data from Urie et al. are marked as undetected analyte and cannot be compared here. The reported $^{243/244}$Cm and ^{238}Pu concentration changes are not followed by change in the ^{242}Cm and $^{239/240}$Pu concentrations. One of the curium values is flagged with a blank contamination, possibly skewing this number high.

The relative changes listed in the final column of Table 4.5 may be real in some cases, but clearly for most of the unflagged analytes changes, real or not, are hidden in uncertainty in the measurement (below 15%).

Table 4.5. Comparison of the Averaged Supernatant Results from the Caustic Addition Experiment and the AN-102 Supernatant Characterization by Urie et al.(a)

Analyte	Urie Average 25°C	Data Flag(b)	Caustic Supernatant 25°C	Data Flag	RPD(d)
Al	12,250	--	11,700	--	-4.5
Ca	490	--	460	J	-6.1
Cd	62	--	61	--	-1.3
Cr	215	--	191.1	--	-11
Fe	38	JB	27	B	-31
K	1980	JB	2290	X	-16
La	15.5	--	15.6	J	-0.6
Na	184000	--	207000	--	+10
Ni	416	--	420	--	+1
P	1850	--	1780	--	-1.9
Pb	190	--	170	--	-8.1
Cu	24	J	17	J	-27
Mo	53	J	54	J	+2
Nd	32	J	34	J	+8
TIC	10950	--	11,300	--	+3.2
TOC	29250	--	27200	--	-7.0
Cl^-	4820	--	4010	--	-17
NO_2^-	85350	--	88300	--	+3.5
NO_3^-	221000	--	208,000	--	+5.9
PO_4^{3-}	5000	U	6280	--	NA(b)
SO_4^{2-}	16850	--	14,300	--	-15
	(μCi/mL)		**(μCi/mL)**		
^{99}Tc(SB)	1.5E-01	--	1.7E-01	--	+14
^{90}Sr	5.7E+01	--	5.8E+01	--	+2
$^{239/240}$Pu	5.9E-03	--	5.5E-03	--	-7
^{241}Am	1.5E-01	--	1.4E-01	--	-7
^{137}Cs	3.7E+02	--	3.5E+02	--	-5
$^{243/244}$Cm	6.7E-03	B	5.4E-03	--	-19
^{238}Pu	1.6E-03	--	1.3E-03	--	-19
^{60}Co	8.5E-02	--	7.9E-02	--	-7
^{154}Eu	2.3E-01	--	2.1E-01	--	-9
^{155}Eu	1.0E-01	--	9.5E-02	--	-5

(a) Analytes flagged with a "U" for all duplicate measurements have been omitted.

(b) In the Data Flag column a "--" indicates there was no data flag or that the data was good.

(c) NA indicates that an RPD calculation is not required.

(d) RPD values are calculated for comparison to overall measurement uncertainties in Tables 3.4 and 4.2.

(SB) Standards Bias, see Appendix F.

4.9 Solids: Variation in Component Distribution with Caustic Addition

The changes in concentration of the supernatant analytes are fairly constant with respect to both temperature and the caustic addition. A comparison of the solids data, on the other hand, indicated rather large changes in concentration. An attempt to resolve the differences in the two data sets is warranted by the data in Table 4.6. The following sections probe the solids data for consistency. This includes balances of sodium and caustic compositions and mass recovery and charge balance calculations for supernatant analytes. A mass recovery for the solids is not calculated because contributions from non-ionic species and waters of hydration cannot be accounted for accurately with the data presented here. Table 4.6 lists the averaged solids data from Urie et al. and the caustic addition experiment in columns three and four, respectively. Column five compares the two data sets, and column six provides a flag that indicates a high value (+) or low value (-). The Burgeson analytes are high (+), in every case, for all unflagged ICP-AES and radiochemical measurements. The result suggests systematic differences in the two data sets. The ICP-AES and radiochemical analytes in the solids characterization originated from the same sample digest. The anion measurements came from a separate sample digestion. This same procedure was used for both the Urie and Burgeson data sets.

Table 4.6. Comparison of Undissolved Solids from Urie et al. and the Caustic Solids Analytes

Analyte	Method	Averaged Caustic Solids (Burgeson) (μg/g)	Averaged Solids (Urie) (μg/g)	RPD Between Urie Solids and Caustic Solids	Indicator[c]
Al	ICP-AES	37700	28500	30	+
Ba	ICP-AES	36	27.5	30	+
Bi	ICP-AES	<37[a]	<9[a]	NA	+
Ca	ICP-MS	571	420	36	+
Cd	ICP-AES	66	38	74	+
Cr	ICP-MS	2300	1800	24	+
Fe	ICP-AES	1500	1200	30	+
Mn	ICP-AES	400	260	58	+
Na	ICP-MS	283,000	167,000	70	+
Ni	ICP-AES	450	260	76	+
P	ICP-AES	2100	1400	52	+
Pb	ICP-MS	350	240	44	+
U	ICP-AES	111	65	70	+
As	ICP-MS	<3	7	-57[a]	-
Be	ICP-AES	<0.8	<1	-32[a]	-
K	ICP-AES	2050	1100	87[a]	+
Sb	ICP-MS	<0.046	0.16	-71[a]	-
Se	ICP-AES	<4.2	68	-94	-
Si	ICP-AES	1100	200	450	+
Sr	ICP-AES	10	6	60	+
Zr	ICP-AES	69	47	48	+
TIC	IC	11700	17400	-32	-

Table 4.6. Con't

Analyte	Method	Averaged Caustic Solids (Burgeson) (μg/g)	Averaged Solids (Urie) (μg/g)	RPD Between Urie Solids and Caustic Solids	Indicator[(c)]
TOC	IC	25300	25600	-1	-
Cl^-	IC	2360	3140	-29	-
F^-	IC	< 100[(a)]	3800	-97	-
NO_2^-	IC	52000	55000	-5.5	-
NO_3^-	IC	126,000	133,000	-5.0	-
PO_4^{3-}	IC	3800	8200	-53	-
SO_4^{2-}	IC	10,800	16,500	-34	-
		(μCi/g)	**(μCi/g)**	**(μCi/g)**	
^{238}Pu	GEA	5.0E-02	1.2E-02	320	+
$^{239/240}Pu$	GEA	6.5E-02	4.2E-02	57	+
^{241}Am	GEA	5.6E-01	4.4E-01	28	+
$^{243/244}Cm$	GEA	4.6E-02	1.7E-02	170	+
^{60}Co	GEA	9.0E-02	5.7E-02	62	+
^{137}Cs	GEA	3.6E+02	2.2E+02	67	+
^{154}Eu	GEA	6.6E-01	5.1E-01	29[(b)]	+
^{155}Eu	GEA	3.0E-01	3.2E-01	-6.0[(b)]	+

(a) These analytes were flagged as not detected (U) in the solids.

(b) Note that the magnitude and direction of the change for ^{155}Eu and ^{154}Eu are different. The large RPD value could be due to uncertainty in the J-flagged ^{155}Eu value, which might be associated with the high ^{137}Cs background.

(c) Column six provides a flag that indicates a high value (+) or low value (-).

4.9.1 Hydroxide Balance for the Supernatant

The addition of caustic to the Burgeson slurry brought the hydroxide concentration from about 0.24M to about 0.85M. The former value was calculated (4125±700 μg/mL) based on averaged (15° and 50°C) temperature/solubility data from Burgeson[a]. The second value (0.883M) was calculated from this averaged value, in the mol hydroxide basis, plus the known hydroxide added (0.643M). Conversion yields 15×10^3 μg /mL, which reasonably agrees with the measured value (Burgeson) of 13×10^3 μg /mL, given that the presence of AlO_2^- and CO_3^{2-} might obscure the measurement. The agreement between the calculated hydroxide and measured hydroxide concentrations is a first indicator that the caustic was not measurably decreased by reaction and precipitation.

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[a] The average was calculated, omitting the Sample A duplicate, which had an anomalously high value.

4.9.2 Sodium Balance for the Supernatant

Based on average measurements of the Burgeson temperature/solubility data, 198,000 ±2000 μg/mL (8.6 ± 0.3M), the caustic (0.643M) addition increased the sodium as calculated to 9.24±0.2M or 213,000 μg/mL. This value reasonably matches the measured (caustic data) sodium value of 207,000 ± 4200 μg/mL. The close agreement indicates that the sodium was wholly present in the supernatant after caustic addition. The agreement of both the sodium and hydroxide argues for negligible impact on the solids composition due to the caustic addition.

4.9.3 Mass Recoveries of the Supernatant Components

Sodium and nitrate are the predominant species found in the AN-102 supernatants. A number of assumptions commonly used in such calculations were followed. Aluminum was assumed to be present as the aluminate anion. The TIC analyses provided the carbonate data, and the oxalate data were directly measured (distinct from TOC). The mass recovery for the supernatant analytes was calculated by ratio of the sums of the cationic and anionic species to the dissolved solids weight (dried to constant weight at 105°C). The ratio should evaluate as a number near 1.0 if the measured analyte concentrations are correct and the solids are in fact dry (see Equation 1).

$$\text{Mass Recovery} = [\Sigma \text{ total analyte concentration}]/[\text{wt solids } (\mu g)]$$
$$= [Na^+ + K^+ + AlO_2^- + CO_3^{-2} + C_2O_4^{-2} + Cl^- + F^- + OH^- + NO_3^- + NO_2^- + PO_4^{-3} + SO_4^{-2}]/[\text{wt solids}] \quad (1)$$

Example calculation: The total analyte concentration in the supernatant (Urie et al. 2002) was 583,000 μg/mL. The dissolved solids were reported by Urie as 53.5% (535,000 μg g/mL). The ratio yields 1.09. The mass recoveries for the supernatants are listed in Table 4.7.

Table 4.7. Mass and Charge Balance Results for AN-102 Supernatants and Solids

	Cations	**Anions**	**Total**	**Wt% Solids**	**Mass Balance Ratio**[(a)]	**Charge Balance Ratio**[(b)]
25°C Supernatant (Urie)	186,000	397000	583,000	53.5[(c)]	1.09	1.04
Average 15° + 50°C (Burgeson) Supernatant	200,000	359,000	559,000	52.1[(c)]	1.07	1.16
Caustic 25°C (Burgeson) Supernatant	210000	358,000	568,000	51.4[(c)]	1.11	1.15

(a) The mass balance for the solids was calculated as a ratio (cations + anions)/(dried solids).
(b) The charge balance ratio was calculated as cation equivalents/anion equivalents.
(c) Total dissolved solids in the supernate (Σcations + anions).

4.9.4 Charge Balance of the Supernatant Components

The laboratory AN-102 supernatant cation and anion data were converted to equivalents by assuming the anionic or cationic form; their ratio was calculated to demonstrate charge balance. Equation 2 itemizes the analytes used in the charge balance. The ratio for the balanced charge should be 1.0. The results of the charge balance calculations are reported in Table 4.7.

Total cations (μeq/g) = $[Na^+]/23.0 + [K^+]/39.1$
Total anions (μeq/g) = $[AlO_2^-]/59.0 + 2*[CO_3^{-2}]/60.0 + 2*[C_2H_4O_2^{2-}]/88.0 + [Cl^-]/35.5 + [F^-]/19.0 + [OH^-]/17.0 + [NO_3^-]/62.0 + [NO_2^-]/46.0 + 3*[PO_4^{-3}]/94.9+ 2*[SO_4^{-2}]/96.0.$ (2)

Total cations (μeq /g)/total anions (μeq/g) = 1.0.

The supernatant data from Burgeson at 15° and 50°C were averaged and the mass recovery ratio calculated as 1.07. The Urie et al. supernatant data recovery was 1.09 and caustic-added supernatant mass balance similarly calculated to 1.11. The supernatant ratios are skewed high. Changes in the sodium and/or anion concentrations near 10% would be required to account for this result; this is not inconsistent with the accuracy apparent in the data set. Moreover, a number of uncharacterized organic materials are present as anions (Urie et al. 2002) and are not accounted for in these calculations.

The supernatant data from Burgeson at 15° and 50°C were averaged and the charge balance ratio calculated as 1.16. The caustic-added supernatant charge balance similarly calculated to 1.15 using the dissolved solids from the caustic addition experiment. Because the sodium balance discussed above seems appropriate for the supernatant data, it may be that the nitrate and/or the combined anion measurements skew the ratios to higher ratio value. Overall, the mass recoveries and charge balances yield reasonably consistent results for each supernatant composition.

From these calculations, the supernatant data for the Burgeson and Urie data sets appear to be self-consistent. This consistency strongly argues for some systemic difference between the solids data sets. The reported solids data therefore could not be used for further evaluation of changes with respect to caustic addition.

A plausible explanation for the difference is that the mass of processed wet centrifuged solids for analysis was much larger for the AN-102 characterization data reported by Urie et al. (2000). The total mass of samples sent by Burgeson to analytical workstations was less than 1 g and in some cases less than 0.5 g. This raises the question that these wet centrifuged solids suffered evaporative loss to a greater degree than the Urie solids. The loss would have occurred under the high ventilation condition and temperature (≈28°C) in the hot cell or through other handling. This weight loss would then translate to the observed higher value for the Burgeson data set. Alternatively, incomplete drying of the larger amount of solids used by Urie et al. might result in a larger value of the wt% wet centrifuged solids. An alternate source of inconsistency might arise from non-uniformity in the solids that were sampled.

4.10 Conclusions

A limited assessment of the effects of temperature and caustic addition on the composition of AN-102 tank waste was carried out. Guidance provided in the Test Specification (Johnson 2001) directed that a statistical evaluation be used to compare these data with tank characterization data compiled at 25°C (Urie et al. 2002). However, compatibility of the data sets was brought into question. Both the Urie samples and the samples reported herein used the same analytical preparation and instrumental methods. However, general processing of samples was different.

Our initial evaluation of the combined supernatant data led to the conclusion that the two samples should be viewed as statistically different. Further consideration of the low accuracy between the combined data sets points to the conclusion that sample preparation, that is, sample size and processing in general led to reporting of uncertainties that may exceed 15% for any given analyte.

A second evaluation, using only the relative changes between 15° and 50°C, again were consistent with a lack of response in concentration with change in temperature. For some analytes, such as born and silicon, nothing could be said with regard to temperature, due to experimental artifacts or measurement uncertainty. These analytes are flagged and their uncertainties were discussed. Nitrate, phosphorous, americium, and manganese demonstrated changes in concentration that were greater than 15% between 15° and 50°C, had good sample reproducibility, and had reported concentrations that were well above stated minimum detection limits. Because only two temperatures were used, the magnitude of the reported changes should not be regarded as established.

Caustic addition of between 0.2M and 0.83M hydroxide to AN-102 slurries likewise caused little change in the supernatant composition. The level of uncertainty encountered was below 15% for most analytes. For some of the analytes the magnitude of actual change was masked by uncertainties arising from sample process conditions and uncertainties in the analytical measurements.

Comparison of the Urie and Burgeson wet centrifuged solids data indicated the presence of systematic differences, especially in the ICP-AES data. Sampling errors related to evaporative losses from the Burgeson solids (small quantity) or incomplete drying of the Urie wet centrifuged solids (large quantity) may have given rise to this apparent error. Further evaluation of caustic induced variations in the solids between the Urie and Burgeson data was compromised by the systematic discrepancies. The reported solids data therefore could not be used for further evaluation of changes with respect to caustic addition. It is important to note that the extensive compositing of AN102 slurry by Urie et al. and the good accuracy presented from numerous replicate samplings likely outweigh the accuracy in the data presented herein.

4.11 Recommendations

The impact of reported changes in the supernatant nitrate or manganese concentration with temperature is arguably negligible with regard to pretreatment activities. An increase in the phosphorus concentration was observed with increase in temperature. The reverse behavior might be expected on cooling. For large volumes of tank waste that are stored at low temperatures and over significant periods of time, swings in the phosphate concentration may result in precipitation of phosphate, leading to clogging. Because americium is transuranic, an increase in concentration, with increase in temperature might impact envelope limit specifications.

The results as presented here cannot be considered more than a scoping study. In order to carefully verify analyte concentration changes, significant revision of the method would improve the results;

- The system should be evaluated over four or five temperatures in duplicate
- The temperature extremes should further perturb the system: 5°C to 70°C.
- Each temperature should be equilibrated for a longer duration, i.e., one week.
- Evaluation should be done with temperature increase followed by temperature decrease.
- Phase separation should be done in a temperature-equilibrated centrifuge.
- Subsamples should not be filtered.
- Sub samples should be diluted in the appropriate media into small (low void space) vials.
- Digestion should be done on the batch samples and analytical data performed as a single batch.

5.0 References

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Appendix A

Statistical Evaluation of Temperature on Analyte Solubility Between 25° and 50°C

Appendix A: Statistical Evaluation of Temperature on Analyte Solubility Between 25° and 50°C

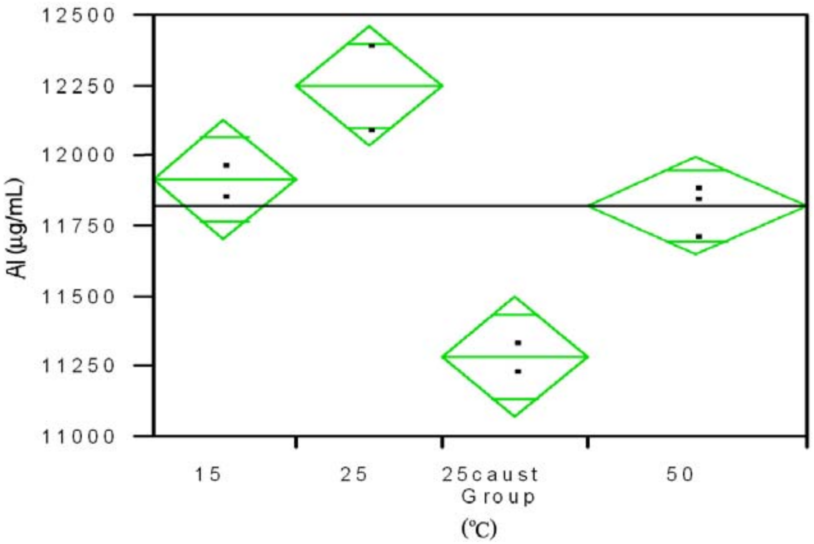
The analysis below includes the caustic supernatant data taken at 25°C for an alternative analysis. The vertical span of the diamond indicates the 95% confidence interval, and the center horizontal line within the diamond indicates the group mean. Horizontal overlap marks drawn above and below the mean provide a visual means to determine whether groups with equal sample size are statistically significant. If the y-axis projection of two diamonds intersects within the overlap marks then the data are not statistically different at the 95% confidence level.

The horizontal span of the diamonds is proportional to the number of measurements made at that temperature. Here the numbers of measurements are generally only 2 or 3, so the diamond widths are not dramatically different. If one group instead had 10 measurements and another 100, the diamond for the latter would be 10 times as wide. Note that the "within group" standard deviation is often assumed equal for all groups in this type of application, and that is the case here. Then since the "standard error of the mean" for the group is this standard deviation divided by the number of measurements in the group, the wider diamonds would have the least vertical spread as well. Again with only 2 or 3 measurements this does not have dramatic effect. With the 10 and 100 case, the group with 10 measurements would have a diamond over three times as tall (square root of 10 compared to square root of 100). This indicates the estimate of the mean is not as precise with the smaller number of measurements.

The vertical tic marks on the horizontal axis simply align with the ends of the diamonds above. A gap was left between the 25 and 50 diamonds to better illustrate the trend between the 15, 25, and 50 temperatures associated with the three groups. Relative to the experimental data, the error in the temperature is very small.

A

Al by Group



Oneway Anova

Summary of Fit

RSquare	0.928489
RSquare Adj	0.885583
Root Mean Square Error	120.8165
Mean of Response	11822.33
Observations (or Sum Wt)	9

Analysis of Variance

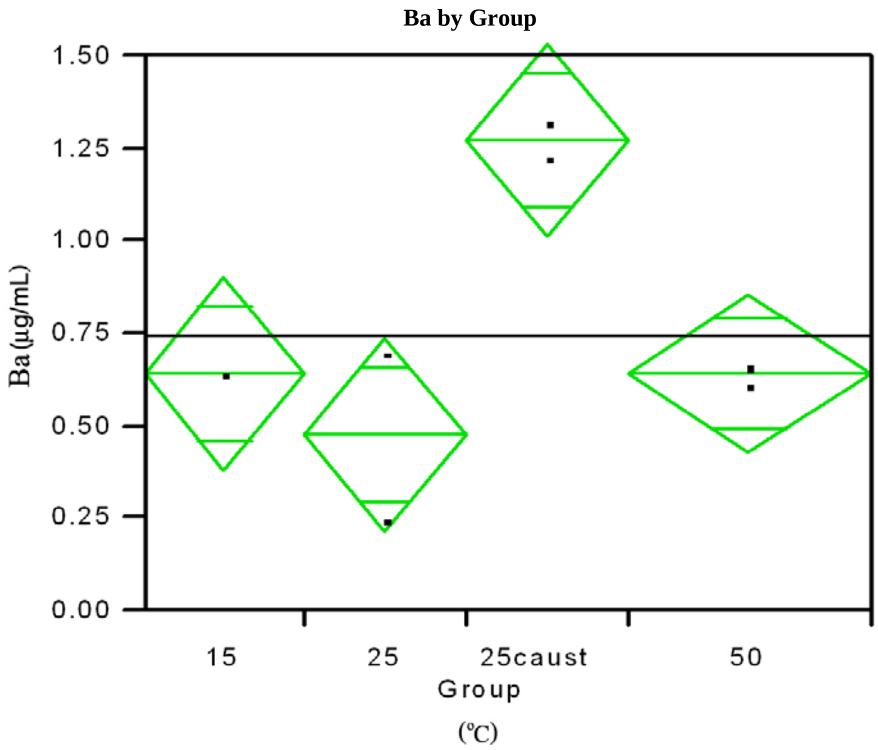
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	947604.8	315868	21.6398
Error	5	72983.2	14597	Prob>F
C Total	8	1020588.0	127574	0.0027

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	11920.5	85.430
25	2	12250.0	85.430
25caust	2	11292.0	85.430
50	3	11825.3	69.753

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		351.959	106.8501	3.29	0.0216
Caustic		-606.041	106.8501	-5.67	0.0024



Oneway Anova

Summary of Fit

RSquare	0.87602
RSquare Adj	0.801632
Root Mean Square Error	0.146538
Mean of Response	0.746667
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.75863333	0.252878	11.7764
Error	5	0.10736667	0.021473	Prob>F
C Total	8	0.86600000	0.108250	0.0105

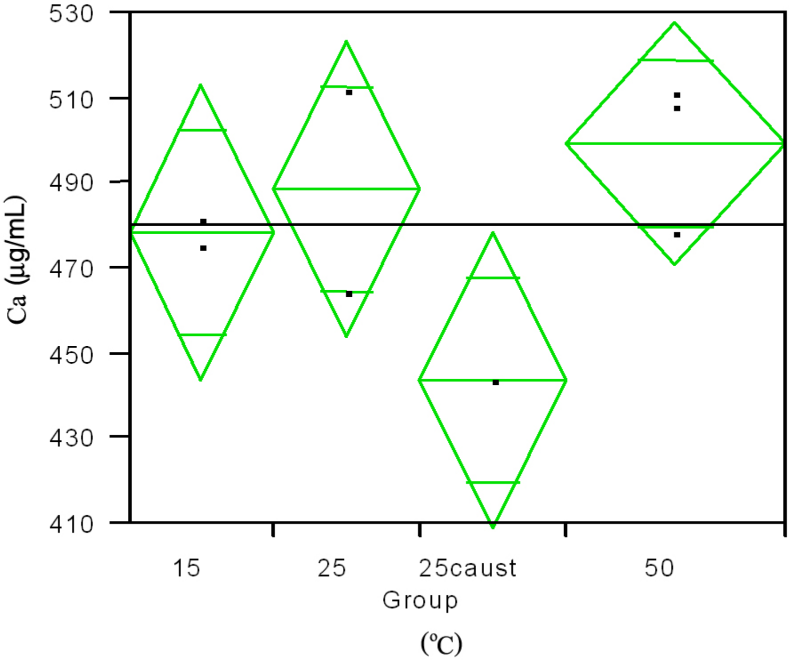
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	0.64000	0.10362
25	2	0.47500	0.10362
25 caustic	2	1.27500	0.10362
50	3	0.64667	0.08460

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-0.16716	0.129601	-1.29	0.2535
Caustic		0.632836	0.129601	4.88	0.0045

Ca by Group



Oneway Anova

Summary of Fit

RSquare	0.682914
RSquare Adj	0.492662
Root Mean Square Error	19.16246
Mean of Response	480.4444
Observations (or Sum Wt)	9

Analysis of Variance

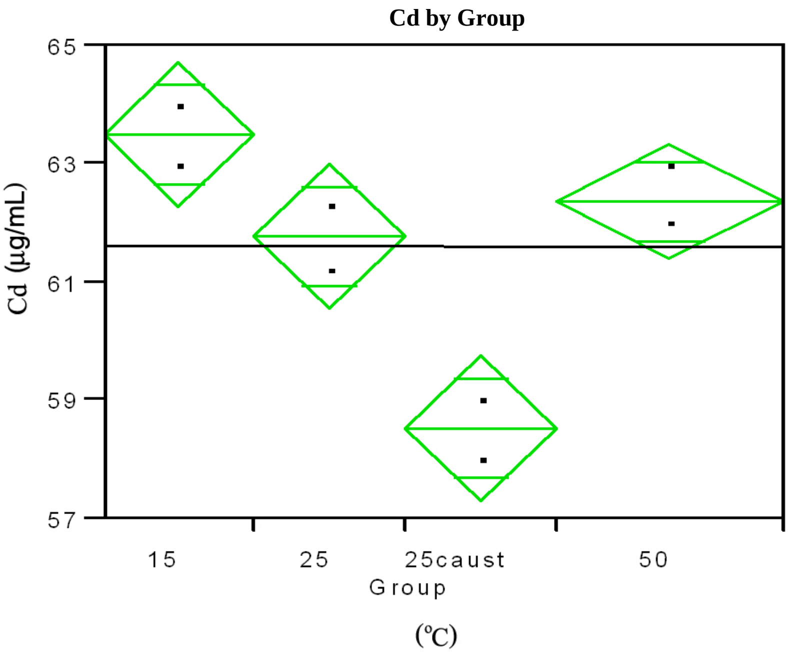
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	3954.2222	1318.07	3.5895
Error	5	1836.0000	367.20	Prob>F
C Total	8	5790.2222	723.78	0.1013

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	479.000	13.550
25	2	489.000	13.550
25caust	2	444.000	13.550
50	3	500.000	11.063

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		3.8024	16.94741	0.22	0.8314
Caustic		-41.1976	16.94741	-2.43	0.0593



Oneway Anova

Summary of Fit

RSquare	0.925198
RSquare Adj	0.880316
Root Mean Square Error	0.674043
Mean of Response	61.61111
Observations (or Sum Wt)	9

Analysis of Variance

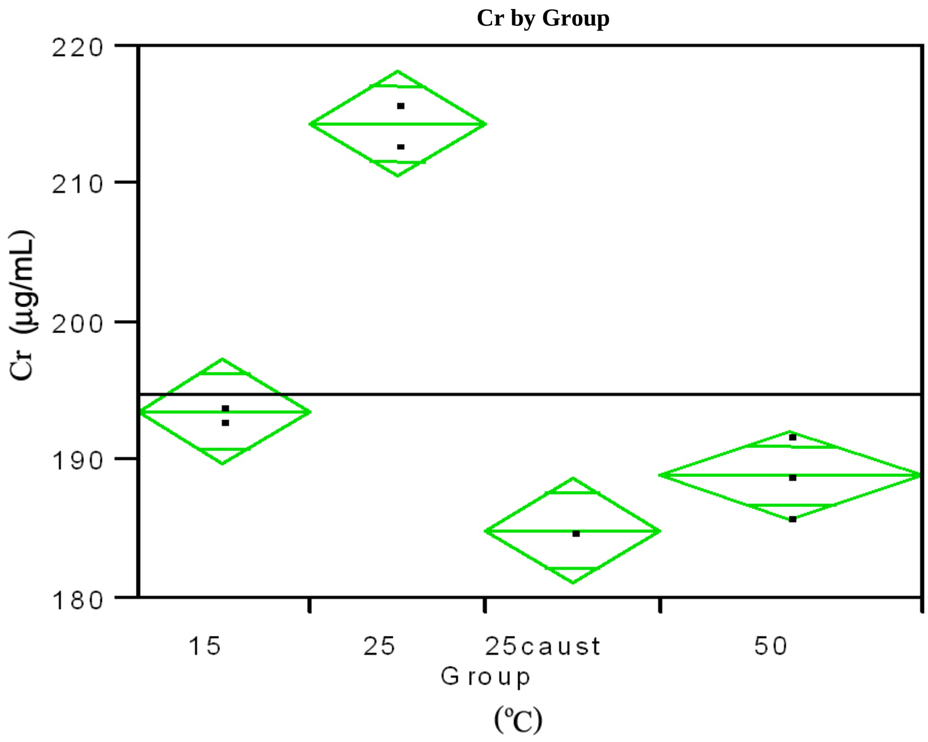
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	28.097222	9.36574	20.6142
Error	5	2.271667	0.45433	Prob>F
C Total	8	30.368889	3.79611	0.0030

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	63.5000	0.47662
25	2	61.7500	0.47662
25caust	2	58.5000	0.47662
50	3	62.3333	0.38916

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-1.44172	0.596124	-2.42	0.0602
Caustic		-4.69172	0.596124	-7.87	0.0005


Cr by Group

Oneway Anova

Summary of Fit

RSquare	0.979006
RSquare Adj	0.96641
Root Mean Square Error	2.144761
Mean of Response	194.7778
Observations (or Sum Wt)	9

Analysis of Variance

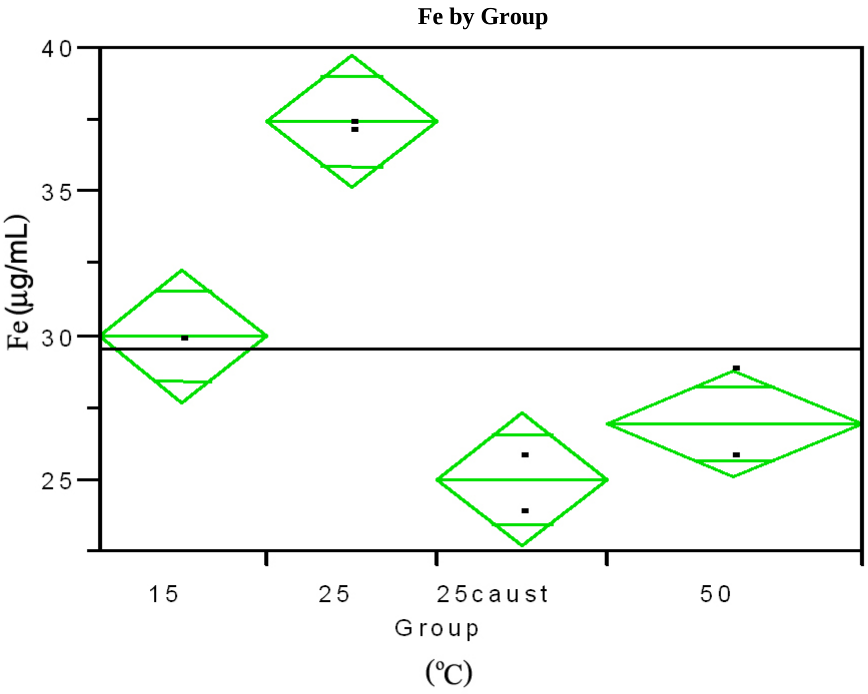
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1072.5556	357.519	77.7214
Error	5	23.0000	4.600	Prob>F
C Total	8	1095.5556	136.944	0.0001

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	193.500	1.5166
25	2	214.500	1.5166
25caust	2	185.000	1.5166
50	3	189.000	1.2383

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		22.2096	1.896862	11.71	0.0001
Caustic		-7.2904	1.896862	-3.84	0.0121



Oneway Anova

Summary of Fit

RSquare	0.95857
RSquare Adj	0.933712
Root Mean Square Error	1.268464
Mean of Response	29.54444
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	186.13722	62.0457	38.5617
Error	5	8.04500	1.6090	Prob>F
C Total	8	194.18222	24.2728	0.0007

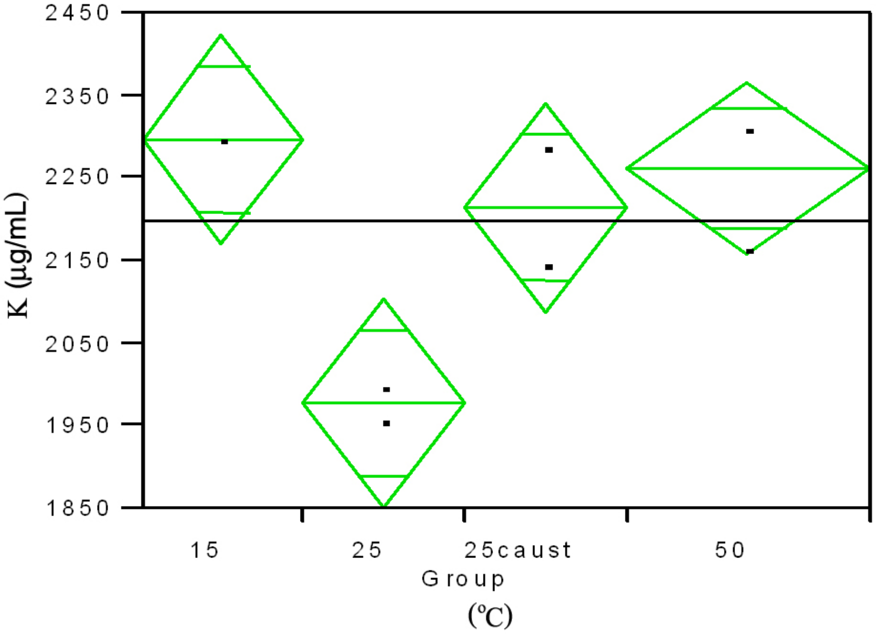
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	30.0000	0.89694
25	2	37.4500	0.89694
25caust	2	25.0000	0.89694
50	3	27.0000	0.73235

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		8.296	1.121833	7.40	0.0007
Caustic		-4.154	1.121833	-3.70	0.0140

K by Group



Oneway Anova

Summary of Fit

RSquare	0.839011
RSquare Adj	0.742418
Root Mean Square Error	70.76887
Mean of Response	2199.444
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	130505.06	43501.7	8.6860
Error	5	25041.17	5008.2	Prob>F
C Total	8	155546.22	19443.3	0.0199

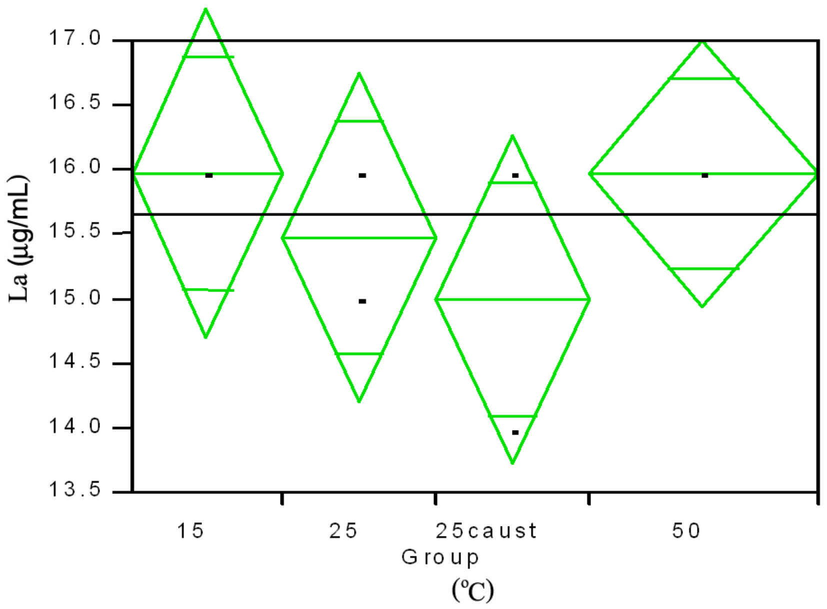
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	2301.00	50.041
25	2	1980.00	50.041
25caust	2	2219.50	50.041
50	3	2264.67	40.858

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-311.53	62.58792	-4.98	0.0042
Caustic		-72.03	62.58792	-1.15	0.3018

La by Group



Oneway Anova

Summary of Fit

RSquare	0.375
RSquare Adj	0
Root Mean Square Error	0.707107
Mean of Response	15.66667
Observations (or Sum Wt)	9

Analysis of Variance

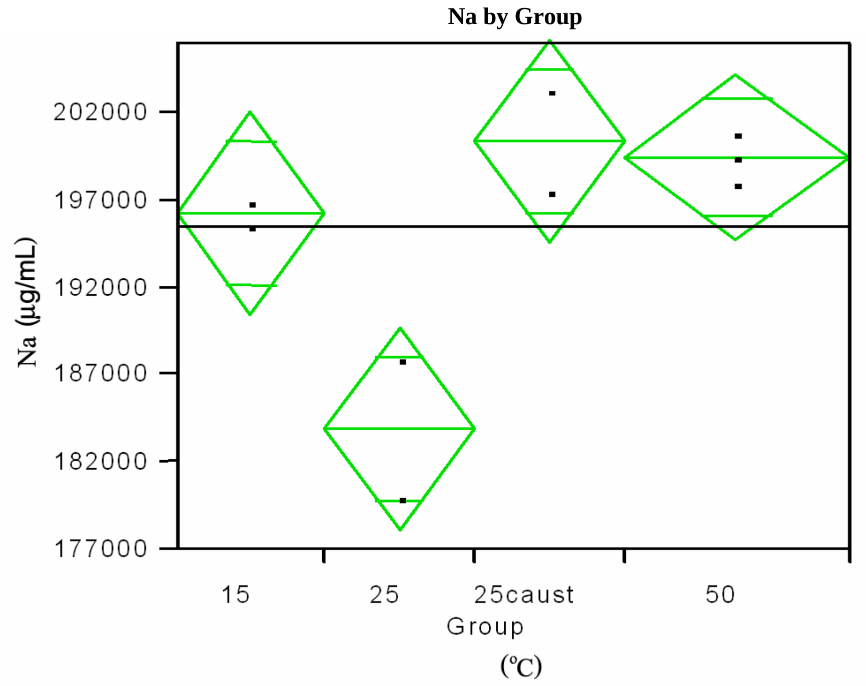
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1.5000000	0.500000	1.0000
Error	5	2.5000000	0.500000	Prob>F
C Total	8	4.0000000	0.500000	0.4649

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	16.0000	0.50000
25	2	15.5000	0.50000
25caust	2	15.0000	0.50000
50	3	16.0000	0.40825

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-0.5064	0.625367	-0.81	0.4549
Caustic		-1.0064	0.625367	-1.61	0.1685



Oneway Anova

Summary of Fit

RSquare	0.871473
RSquare Adj	0.794358
Root Mean Square Error	3274.686
Mean of Response	195560.8
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	363555627	1.2119e8	11.3008
Error	5	53617855	10723571	Prob>F
C Total	8	417173482	52146685	0.0115

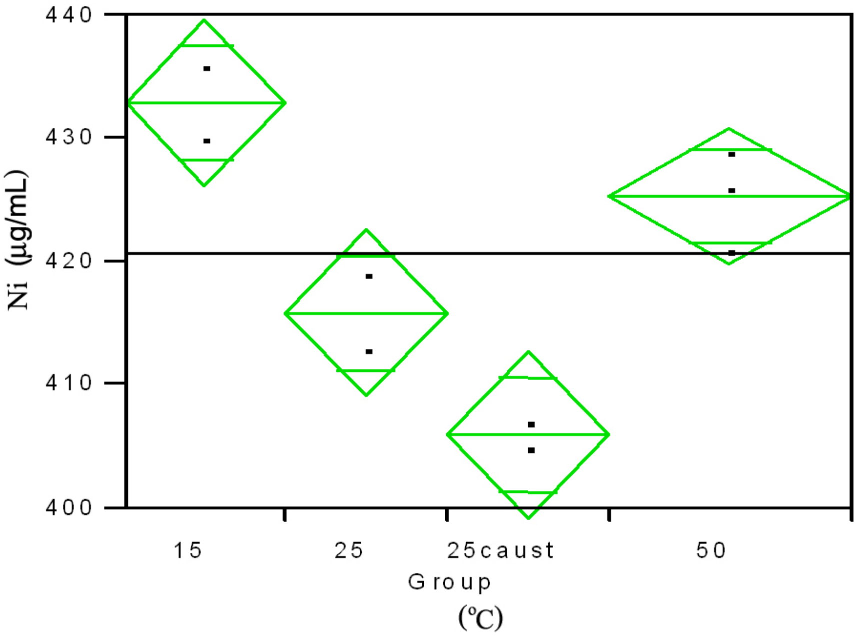
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	196280	2315.6
25	2	184000	2315.6
25caust	2	200494	2315.6
50	3	199500	1890.6

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-13279.4	2896.193	-4.59	0.0059
Caustic		3214.568	2896.193	1.11	0.3175

Ni by Group



Oneway Anova

Summary of Fit

RSquare	0.922684
RSquare Adj	0.876295
Root Mean Square Error	3.759433
Mean of Response	420.6667
Observations (or Sum Wt)	9

Analysis of Variance

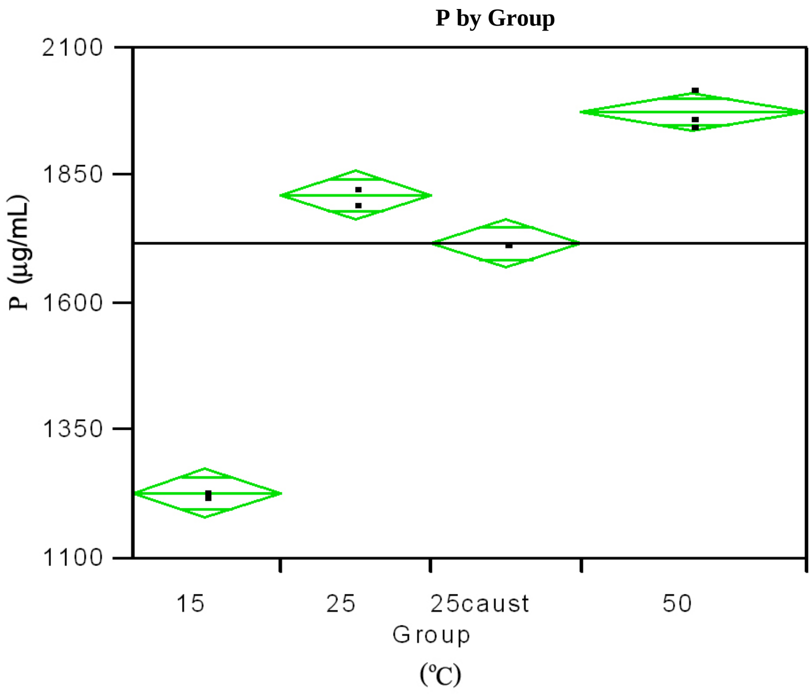
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	843.33333	281.111	19.8899
Error	5	70.66667	14.133	Prob>F
C Total	8	914.00000	114.250	0.0033

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	433.000	2.6583
25	2	416.000	2.6583
25caust	2	406.000	2.6583
50	3	425.333	2.1705

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-14.9804	3.324824	-4.51	0.0064
Caustic		-24.9804	3.324824	-7.51	0.0007



Oneway Anova

Summary of Fit

RSquare	0.995133
RSquare Adj	0.992213
Root Mean Square Error	26.13299
Mean of Response	1719.333
Observations (or Sum Wt)	9

Analysis of Variance

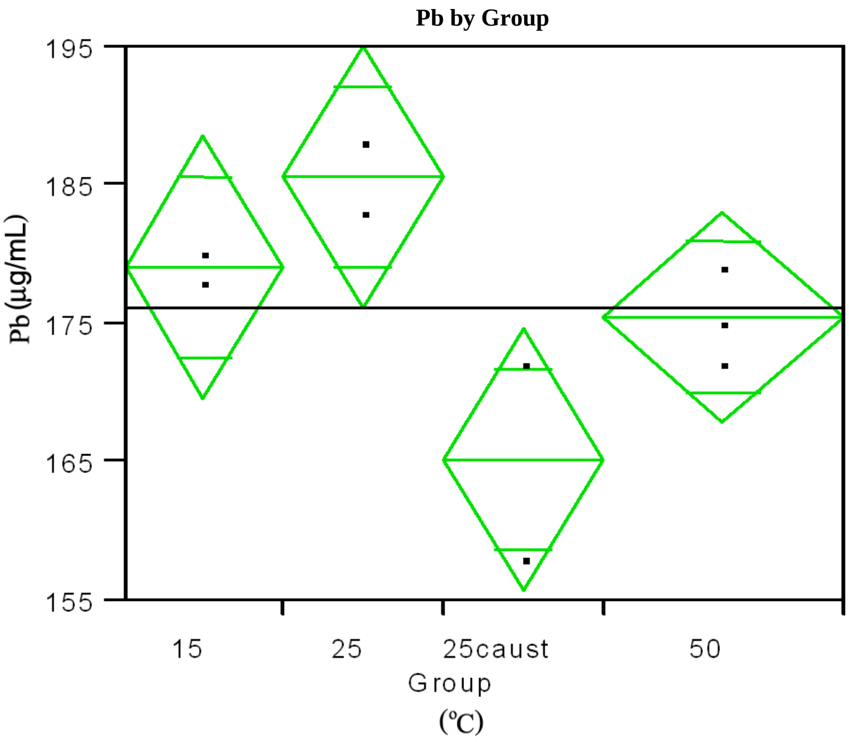
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	698177.33	232726	340.7738
Error	5	3414.67	683	Prob>F
C Total	8	701592.00	87699	<.0001

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	1232.00	18.479
25	2	1815.00	18.479
25caust	2	1719.00	18.479
50	3	1980.67	15.088

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		368.3876	23.1123	15.94	0.0000
Caustic		272.3876	23.1123	11.79	0.0001



Oneway Anova

Summary of Fit

RSquare	0.763052
RSquare Adj	0.620883
Root Mean Square Error	5.237684
Mean of Response	176.1111
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	441.72222	147.241	5.3672
Error	5	137.16667	27.433	Prob>F
C Total	8	578.88889	72.361	0.0507

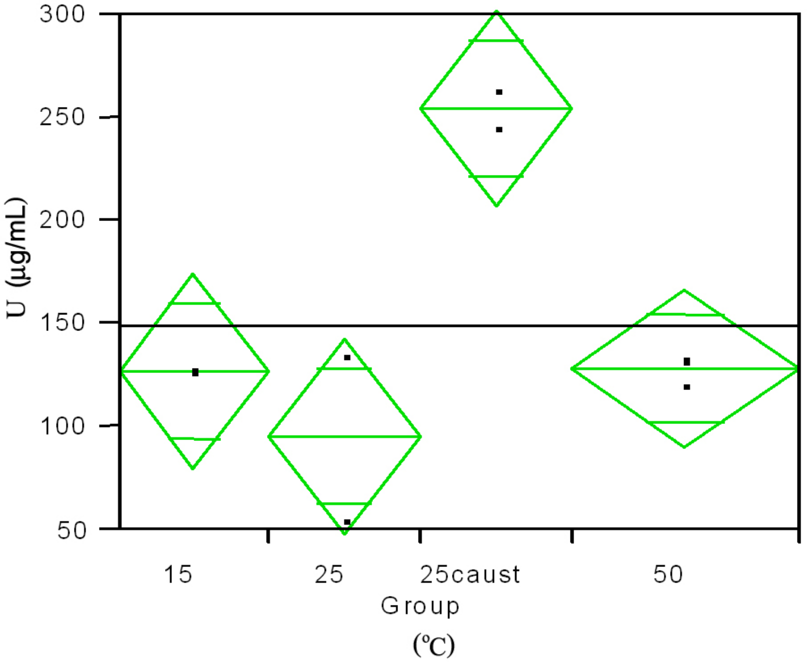
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	179.000	3.7036
25	2	185.500	3.7036
25caust	2	165.000	3.7036
50	3	175.333	3.0240

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		7.477162	4.632216	1.61	0.1674
Caustic		-13.0228	4.632216	-2.81	0.0375

U by Group



Oneway Anova

Summary of Fit

RSquare	0.898256
RSquare Adj	0.837209
Root Mean Square Error	26.27229
Mean of Response	149
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	30468.833	10156.3	14.7143
Error	5	3451.167	690.2	Prob>F
C Total	8	33920.000	4240.0	0.0065

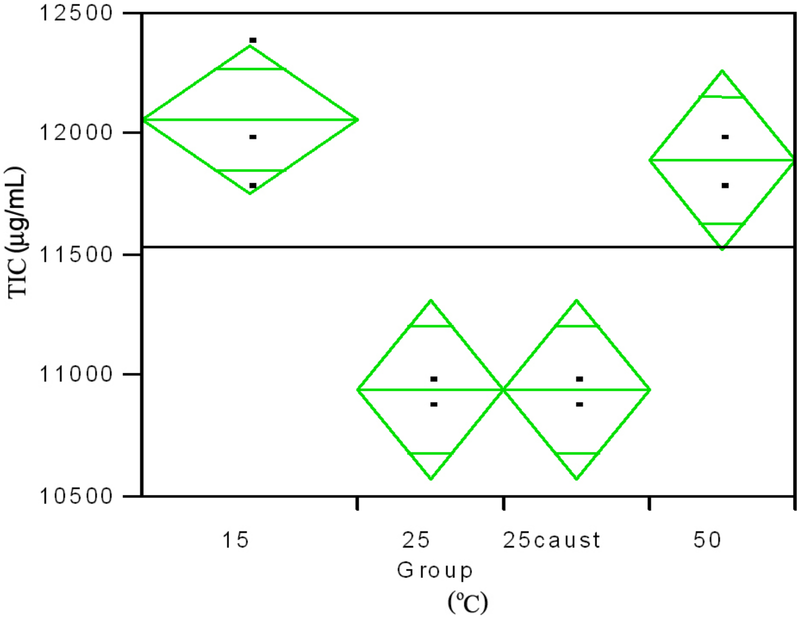
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	127.500	18.577
25	2	95.000	18.577
25caust	2	255.000	18.577
50	3	128.667	15.168

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-32.8848	23.23487	-1.42	0.2161
Caustic		127.1152	23.23487	5.47	0.0028

TIC by Group



Oneway Anova

Summary of Fit

RSquare	0.919753
RSquare Adj	0.871605
Root Mean Square Error	208.1666
Mean of Response	11533.33
Observations (or Sum Wt)	9

Analysis of Variance

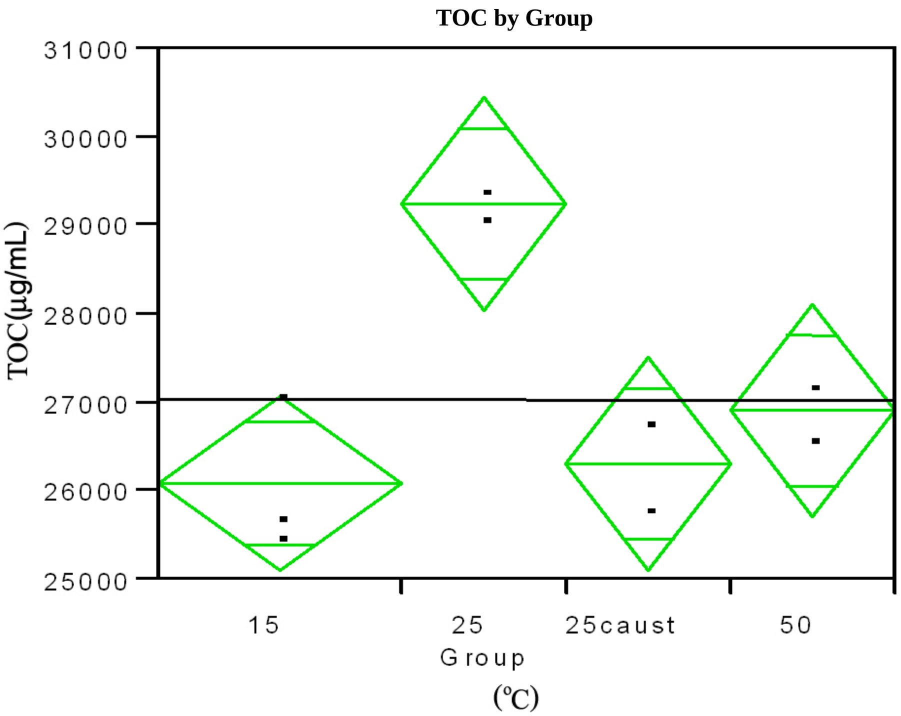
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	2483333.3	827778	19.1026
Error	5	216666.7	43333	Prob>F
C Total	8	2700000.0	337500	0.0036

Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	12066.7	120.19
25	2	10950.0	147.20
25caust	2	10950.0	147.20
50	2	11900.0	147.20

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-1073.85	175.5125	-6.12	0.0017
Caustic		-1073.85	175.5125	-6.12	0.0017



Oneway Anova

Summary of Fit

RSquare	0.857871
RSquare Adj	0.772594
Root Mean Square Error	670.0746
Mean of Response	27022.22
Observations (or Sum Wt)	9

Analysis of Variance

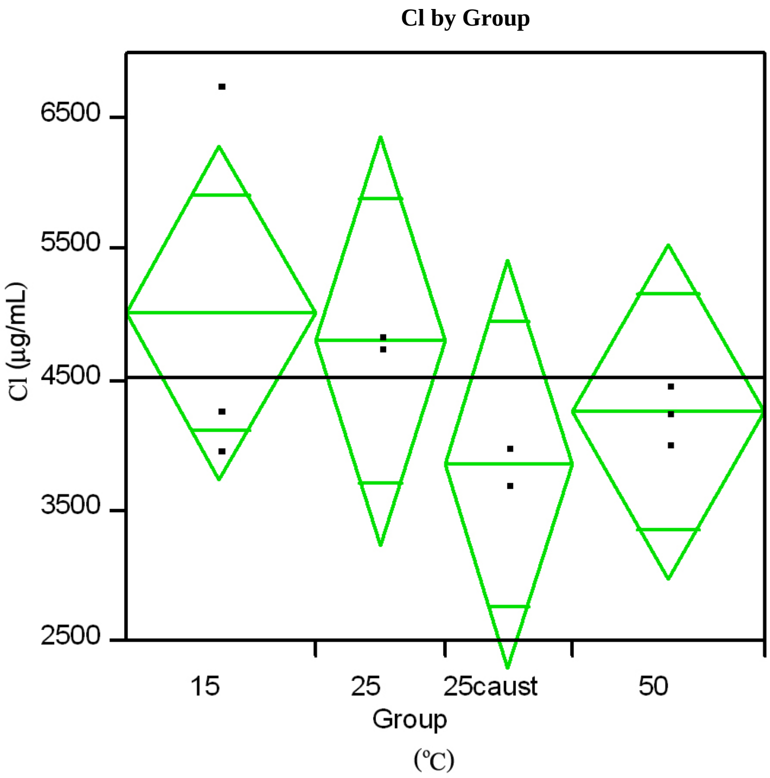
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	13550556	4516852	10.0598
Error	5	2245000	449000	Prob>F
C Total	8	15795556	1974444	0.0147

Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	26100.0	386.87
25	2	29250.0	473.81
25caust	2	26300.0	473.81
50	2	26900.0	473.81

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		2910.76	564.943	5.15	0.0036
Caustic		-39.24	564.943	-0.07	0.9473



Oneway Anova

Summary of Fit

RSquare	0.287568
RSquare Adj	-0.06865
Root Mean Square Error	906.7526
Mean of Response	4528.7
Observations (or Sum Wt)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1991252.8	663751	0.8073
Error	6	4933201.3	822200	Prob>F
C Total	9	6924454.1	769384	0.5342

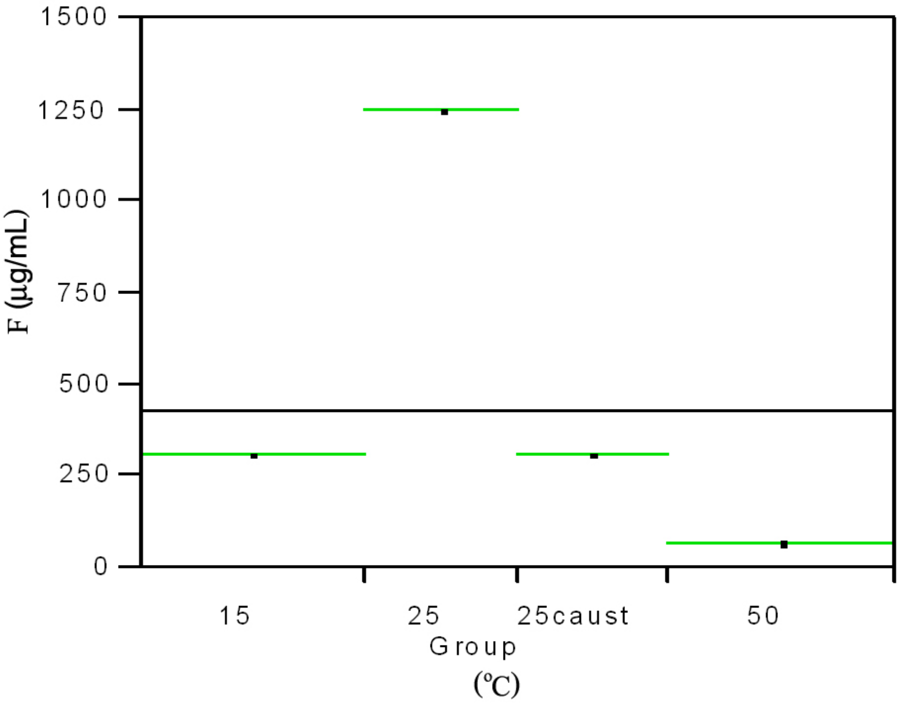
Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	5032.67	523.51
25	2	4820.00	641.17
25caust	2	3876.00	641.17
50	3	4265.67	523.51

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		4.678932	757.1225	0.01	0.9953
Caustic		-939.321	757.1225	-1.24	0.2610

F by Group



Oneway Anova

Summary of Fit

RSquare	0.999994
RSquare Adj	0.999991
Root Mean Square Error	1.333333
Mean of Response	427.65
Observations (or Sum Wt)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1799701.4	599900	337444
Error	6	10.7	2	Prob>F
C Total	9	1799712.0	199968	<.0001

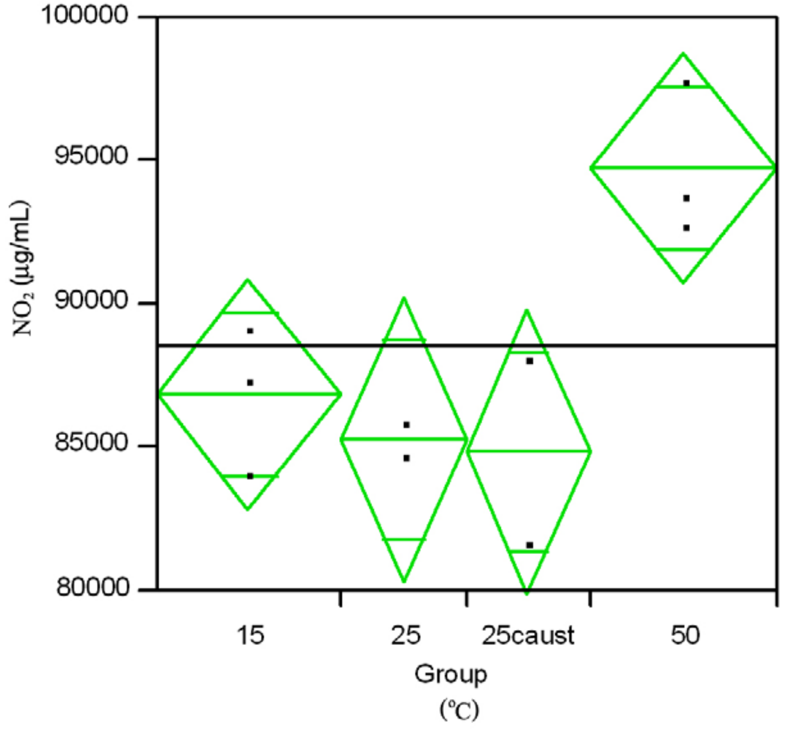
Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	312.50	0.76980
25	2	1250.00	0.94281
25caust	2	312.50	0.94281
50	3	71.33	0.76980

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		1006.35	1.113314	903.92	0.0000
Caustic		68.84962	1.113314	61.84	0.0000

NO$_2$ by Group



Oneway Anova

Summary of Fit

RSquare	0.776357
RSquare Adj	0.664536
Root Mean Square Error	2872.702
Mean of Response	88582.6
Observations (or Sum Wt)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	171885558	57295186	6.9428
Error	6	49514515	8252419	Prob>F
C Total	9	221400072	24600008	0.0223

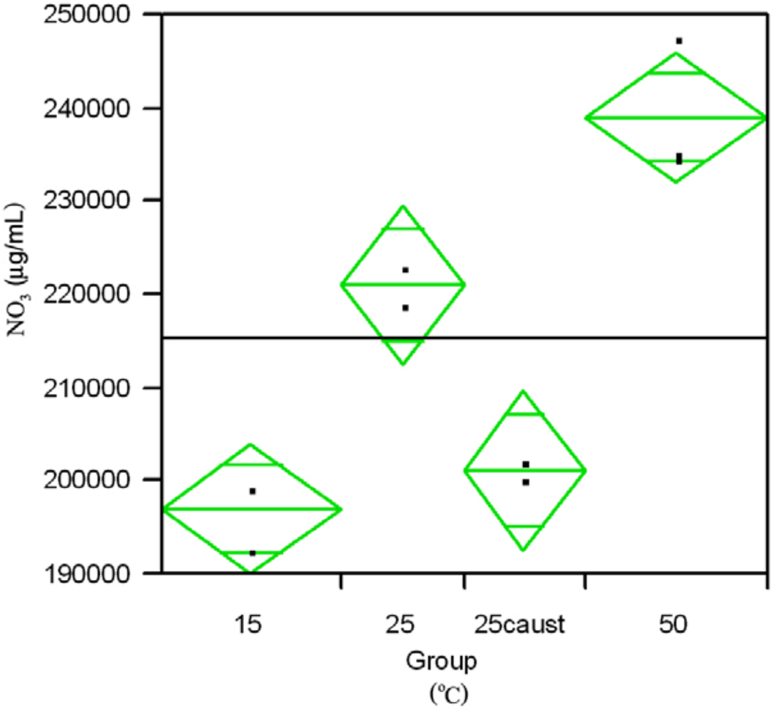
Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	86938.7	1658.6
25	2	85350.0	2031.3
25caust	2	84940.0	2031.3
50	3	94810.0	1658.6

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-3874.67	2398.675	-1.62	0.1574
Caustic		-4284.67	2398.675	-1.79	0.1243

NO_3 by Group



Oneway Anova

Summary of Fit

RSquare	0.955301
RSquare Adj	0.932951
Root Mean Square Error	4974.289
Mean of Response	215358.9
Observations (or Sum Wt)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	3172858976	1.0576e9	42.7432
Error	6	148461331	24743555	Prob>F
C Total	9	3321320307	3.6904e8	0.0002

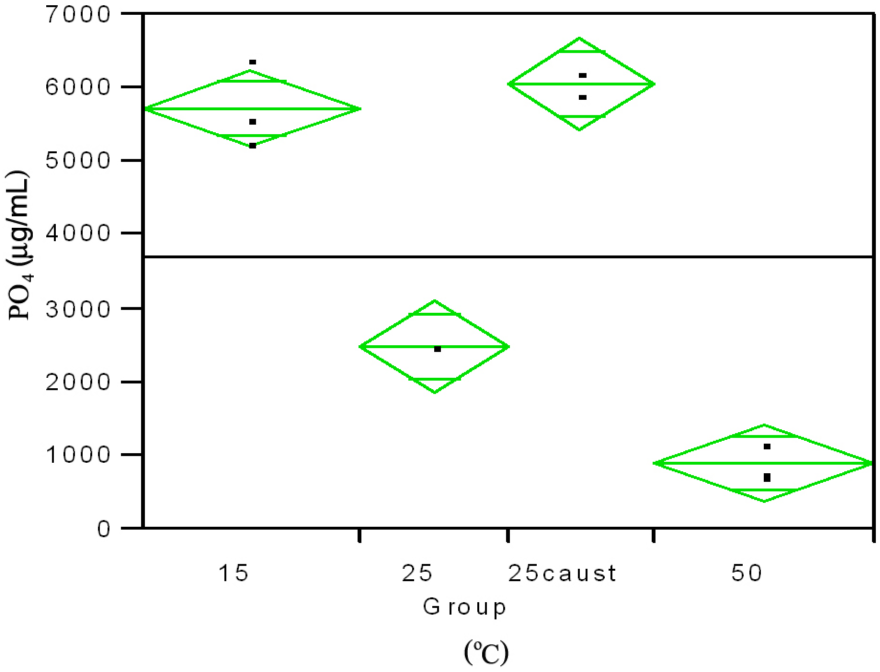
Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	197160	2871.9
25	2	221000	3517.4
25caust	2	201173	3517.4
50	3	239255	2871.9

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		11721.97	4153.493	2.82	0.0303
Caustic		-8105.03	4153.493	-1.95	0.0989

PO$_4$ by Group



Oneway Anova

Summary of Fit

RSquare	0.98329
RSquare Adj	0.974934
Root Mean Square Error	377.9377
Mean of Response	3703.1
Observations (or Sum Ws)t10	

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	50429438	16809813	117.6854
Error	6	857021	142836.9	Prob>F
C Total	9	51286459	5698495	<.0001

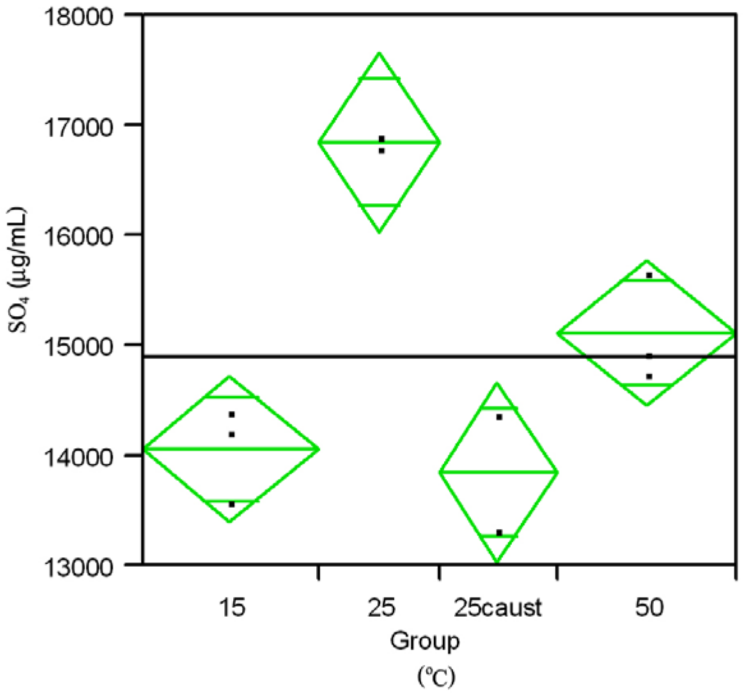
Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	5743.67	218.20
25	2	2500.00	267.24
25caust	2	6072.00	267.24
50	3	885.33	218.20

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-1856.48	315.5694	-5.88	0.0011
Caustic		1715.518	315.5694	5.44	0.0016

SO_4 by Group



Oneway Anova

Summary of Fit

RSquare	0.894926
RSquare Adj	0.842389
Root Mean Square Error	483.1893
Mean of Response	14900.6
Observations (or Sum Wt)	10

Analysis of Variance

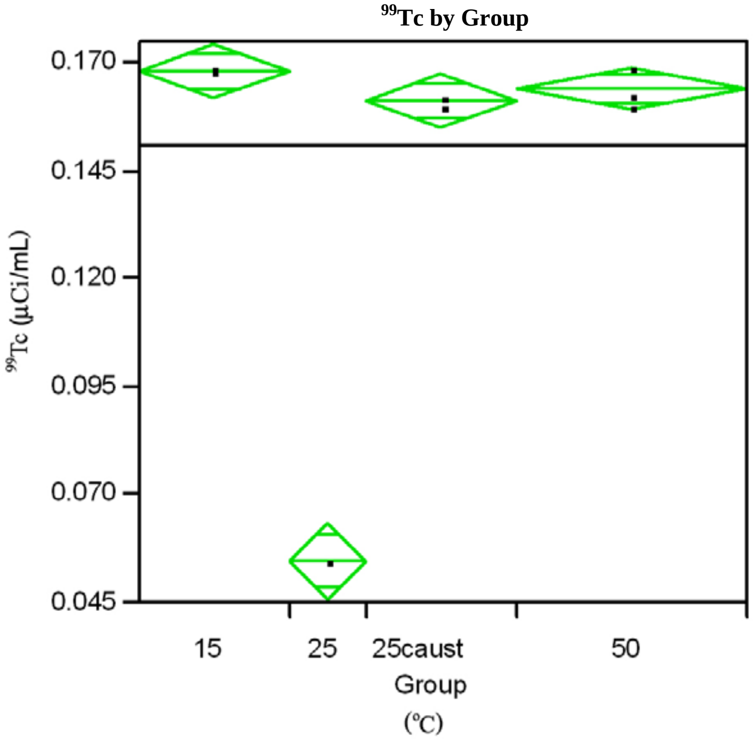
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	11931009	3977003	17.0342
Error	6	1400831	233472	Prob>F
C Total	9	13331840	1481316	0.0024

Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	14073.3	278.97
25	2	16850.0	341.67
25caust	2	13867.0	341.67
50	3	15117.3	278.97

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		2472.487	403.4591	6.13	0.0009
Caustic		-510.513	403.4591	-1.27	0.2527



Oneway Anova

Summary of Fit

RSquare	0.995832
RSquare Adj	0.992705
Root Mean Square Error	0.003335
Mean of Response	0.151688
Observations (or Sum Wt)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.01063097	0.003544	318.5309
Error	4	0.00004450	0.000011	Prob>F
C Total	7	0.01067547	0.001525	<.0001

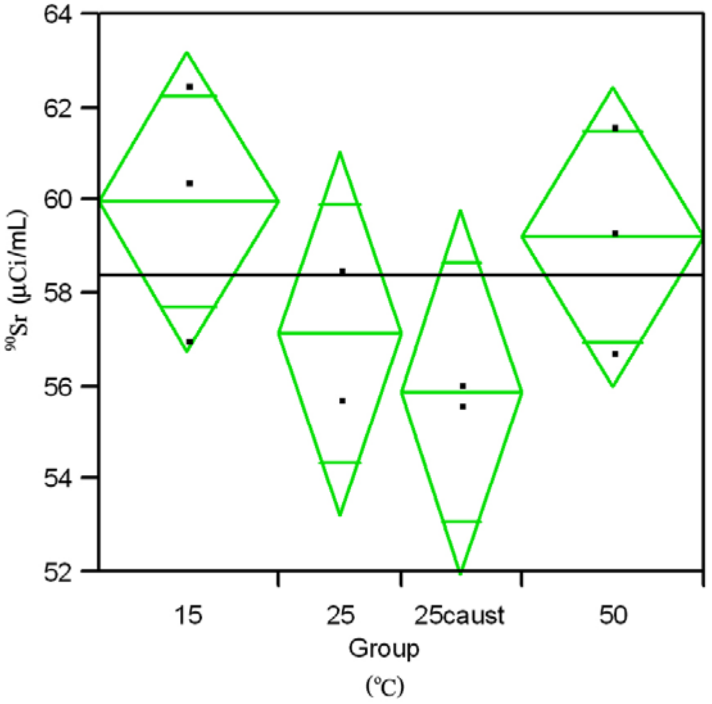
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	0.169500	0.00236
25	1	0.055500	0.00334
25caust	2	0.162000	0.00236
50	3	0.165000	0.00193

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-0.11278	0.003781	-29.82	0.0000
Caustic		-0.00628	0.002952	-2.13	0.1005

^{90}Sr by Group



Oneway Anova

Summary of Fit

RSquare	0.454028
RSquare Adj	0.181043
Root Mean Square Error	2.288619
Mean of Response	58.43
Observations (or Sum Wt)	10

Analysis of Variance

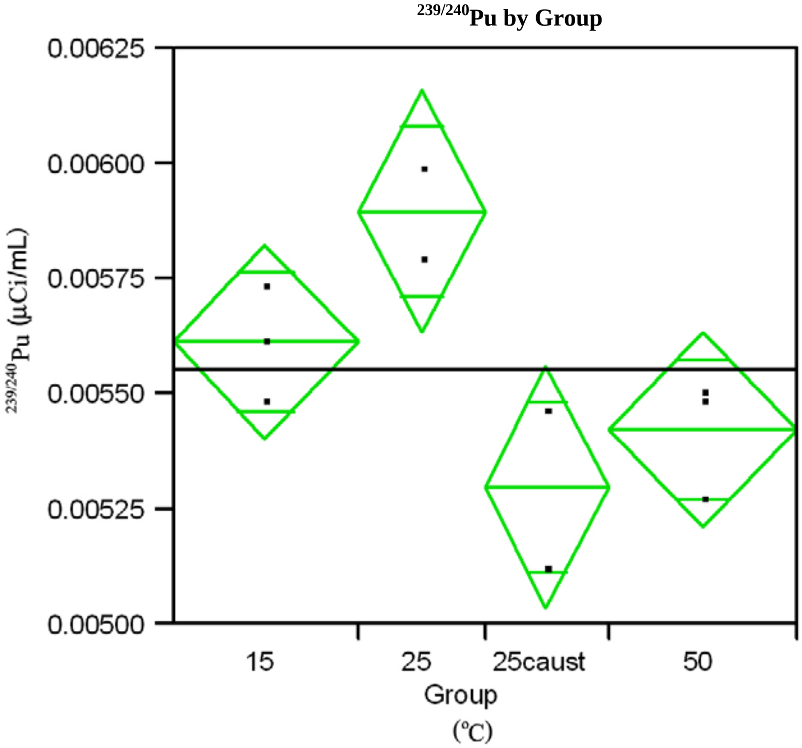
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	26.134333	8.71144	1.6632
Error	6	31.426667	5.23778	Prob>F
C Total	9	57.561000	6.39567	0.2725

Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	60.0667	1.3213
25	2	57.2000	1.6183
25caust	2	55.9000	1.6183
50	3	59.3000	1.3213

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-2.67145	1.91095	-1.40	0.2116
Caustic		-3.97145	1.91095	-2.08	0.0829



Oneway Anova

Summary of Fit

RSquare	0.751874
RSquare Adj	0.627812
Root Mean Square Error	0.000154
Mean of Response	0.005553
Observations (or Sum Wt)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00000043	1.43e-7	6.0604
Error	6	0.00000014	2.359e-8	Prob>F
C Total	9	0.00000057	6.338e-8	0.0301

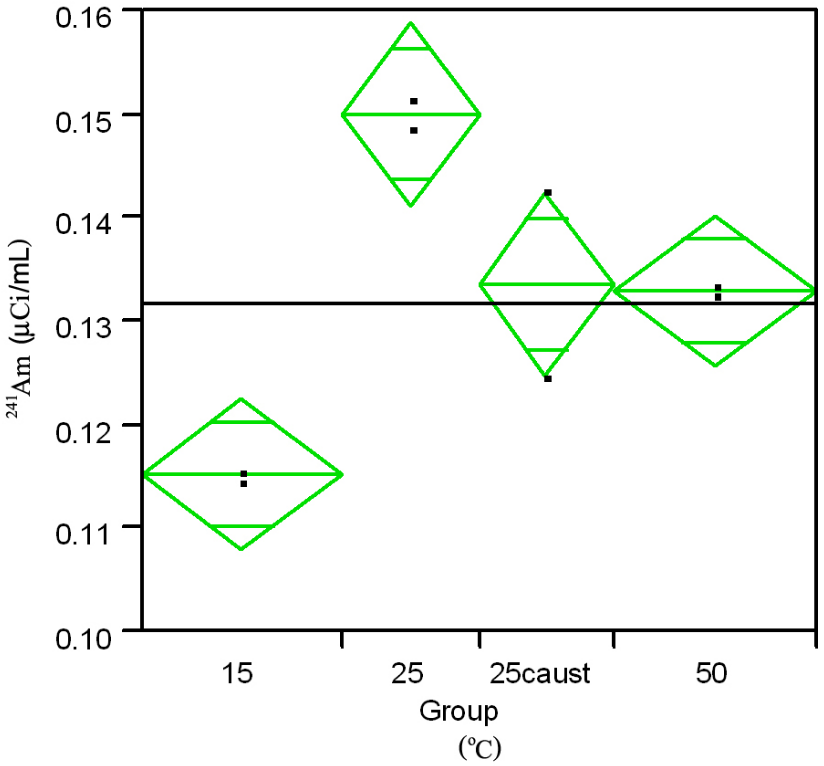
Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	0.005617	0.00009
25	2	0.005900	0.00011
25caust	2	0.005300	0.00011
50	3	0.005427	0.00009

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		0.000335	0.00013	2.58	0.0419
Caustic		-0.00026	0.00013	-2.04	0.0877

^{241}Am by Group



Oneway Anova

Summary of Fit

RSquare	0.899163
RSquare Adj	0.848744
Root Mean Square Error	0.005289
Mean of Response	0.1316
Observations (or Sum Wt)	10

Analysis of Variance

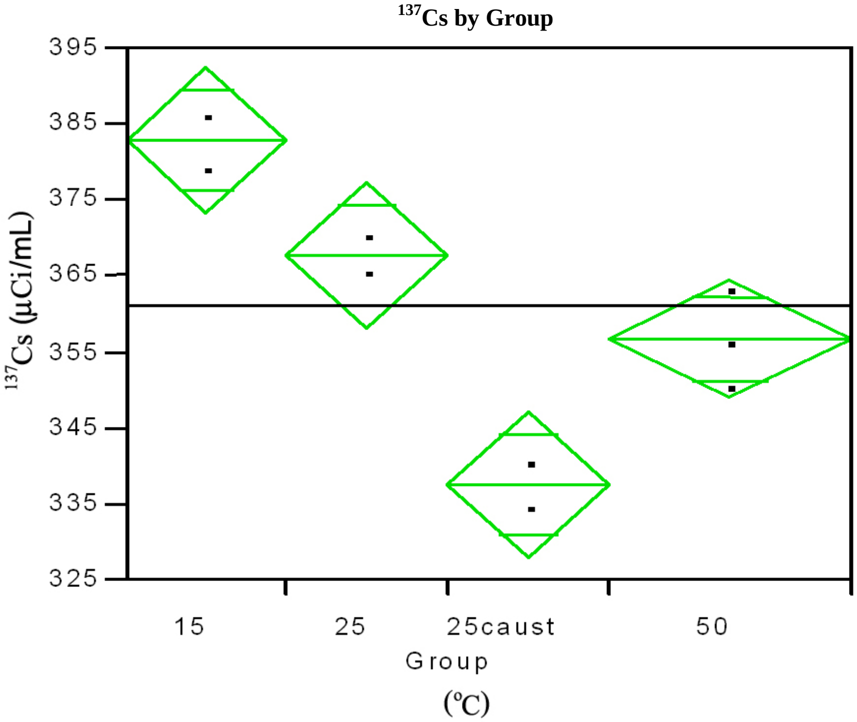
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00149657	0.000499	17.8340
Error	6	0.00016783	0.000028	Prob>F
C Total	9	0.00166440	0.000185	0.0022

Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	0.115667	0.00305
25	2	0.150500	0.00374
25caust	2	0.134000	0.00374
50	3	0.133333	0.00305

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		0.029734	0.004415	6.74	0.0005
Caustic		0.013234	0.004415	3.00	0.0241



Oneway Anova

Summary of Fit

RSquare	0.940869
RSquare Adj	0.905391
Root Mean Square Error	5.285199
Mean of Response	361.3333
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	2222.3333	740.778	26.5195
Error	5	139.6667	27.933	Prob>F
C Total	8	2362.0000	295.250	0.0017

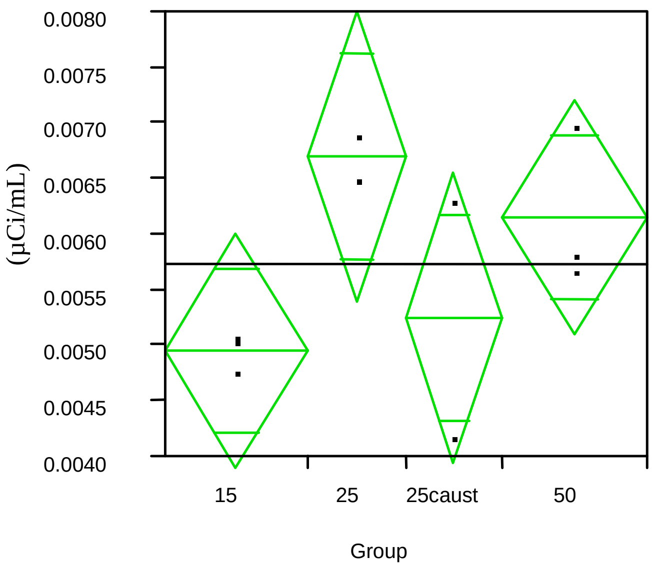
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	383.500	3.7372
25	2	368.500	3.7372
25caust	2	338.000	3.7372
50	3	357.333	3.0514

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-7.66964	4.674239	-1.64	0.1618
Caustic		-38.1696	4.674239	-8.17	0.0004

$^{243/244}$Cm by Group



(°C)

Oneway Anova

Summary of Fit

RSquare	0.577034
RSquare Adj	0.365551
Root Mean Square Error	0.000757
Mean of Response	0.005731
Observations (or Sum Wt)	10

Analysis of Variance

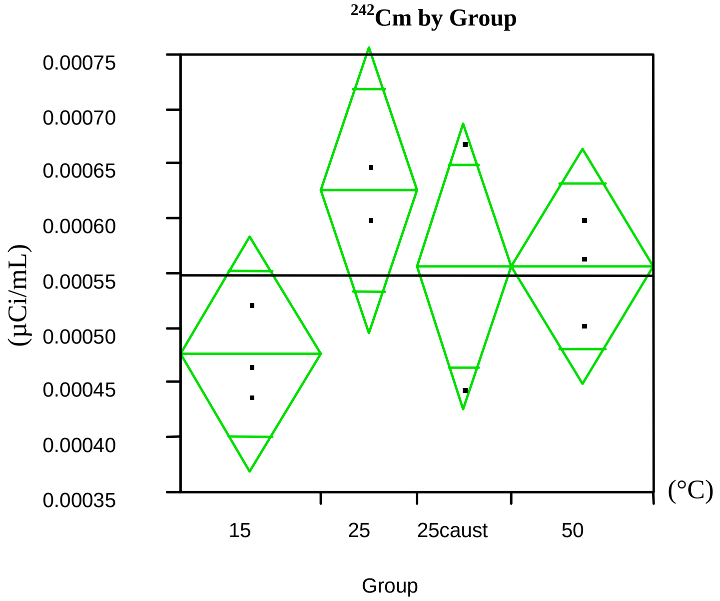
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00000469	0.000002	2.7285
Error	6	0.00000344	5.733e-7	Prob>F
C Total	9	0.00000813	9.037e-7	0.1365

Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	0.004967	0.00044
25	2	0.006710	0.00054
25caust	2	0.005250	0.00054
50	3	0.006163	0.00044

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		0.001399	0.000637	2.20	0.0706
Caustic		-6.1E-05	0.000637	-0.10	0.9268



Oneway Anova

Summary of Fit

RSquare	0.443691
RSquare Adj	0.165537
Root Mean Square Error	0.000077
Mean of Response	0.000549
Observations (or Sum Wt)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00000003	9.348e-9	1.5951
Error	6	0.00000004	5.861e-9	Prob>F
C Total	9	0.00000006	7.023e-9	0.2863

Means for Oneway Anova

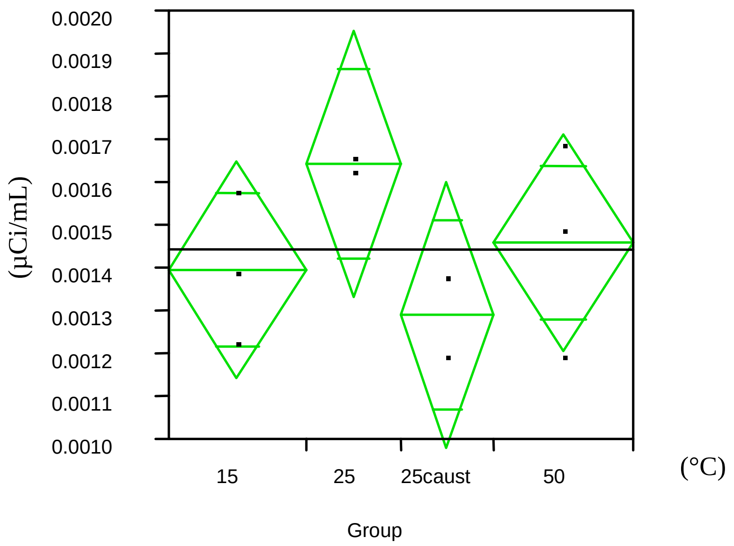
Level	Number	Mean	Std Error
15	3	0.000479	0.00004
25	2	0.000628	0.00005
25caust	2	0.000561	0.00005
50	3	0.000559	0.00004

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		0.000126	5.87E-05	2.15	0.0756
Caustic		5.89E-05	5.87E-05	1.00	0.3542

(°C)

^{238}Pu by Group



Oneway Anova

Summary of Fit

RSquare	0.403291
RSquare Adj	0.104936
Root Mean Square Error	0.000182
Mean of Response	0.001445
Observations (or Sum Wt)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00000013	4.493e-8	1.3517
Error	6	0.0000002	3.324e-8	Prob>F
C Total	9	0.00000033	3.714e-8	0.3437

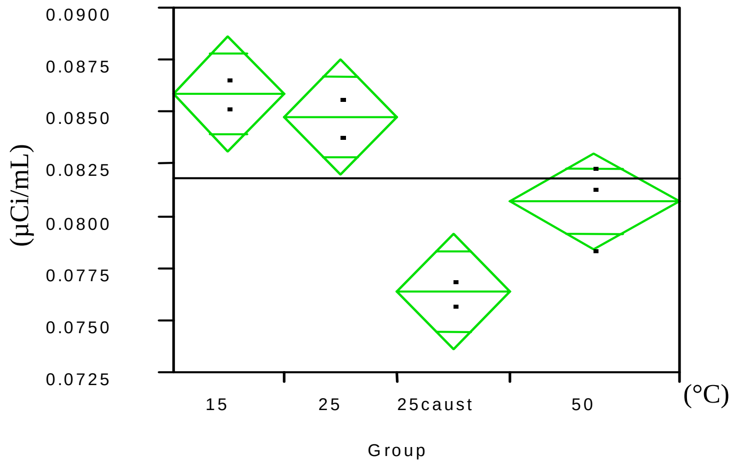
Means for Oneway Anova

Level	Number	Mean	Std Error
15	3	0.001400	0.00011
25	2	0.001645	0.00013
25caust	2	0.001290	0.00013
50	3	0.001460	0.00011

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		0.000227	0.000155	1.47	0.1932
Caustic		-0.00013	0.000155	-0.82	0.4418

^{60}Co by Group



Oneway Anova

Summary of Fit

RSquare	0.905303
RSquare Adj	0.848485
Root Mean Square Error	0.001552
Mean of Response	0.081878
Observations (or Sum Wt)	9

Analysis of Variance

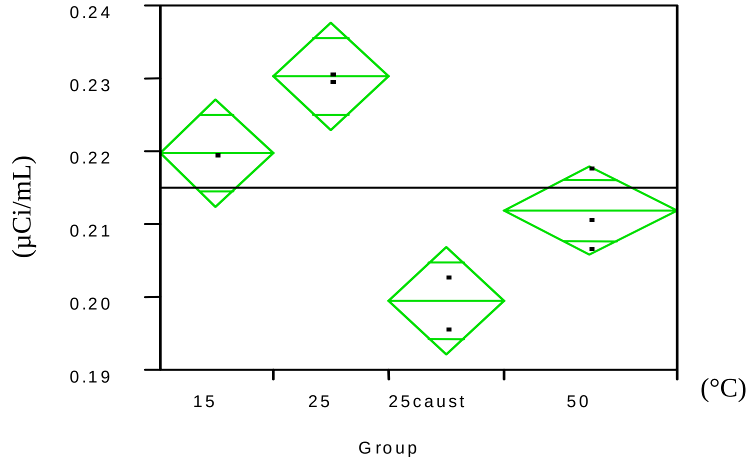
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00011515	0.000038	15.9334
Error	5	0.00001205	0.000002	Prob>F
C Total	8	0.00012720	0.000016	0.0054

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	0.086000	0.00110
25	2	0.084850	0.00110
25caust	2	0.076400	0.00110
50	3	0.080800	0.00090

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		0.000303	0.001376	0.22	0.8345
Caustic		-0.00815	0.001376	-5.92	0.0020



^{154}Eu by Group

Oneway Anova

Summary of Fit

RSquare	0.922659
RSquare Adj	0.876254
Root Mean Square Error	0.004171
Mean of Response	0.215111
Observations (or Sum Wt)	9

Analysis of Variance

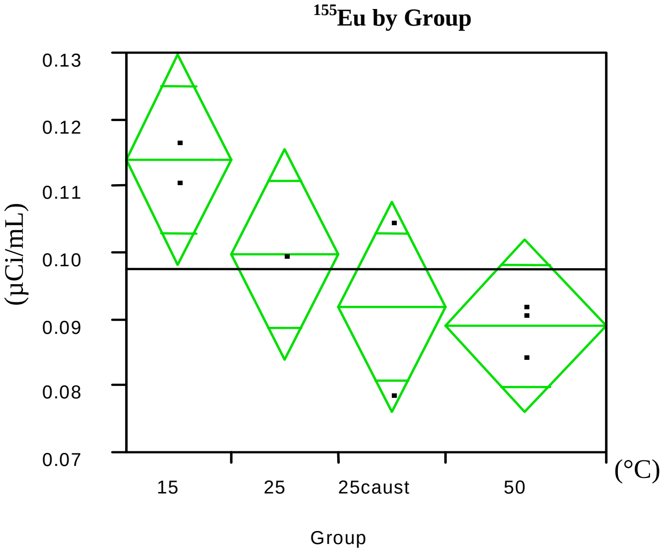
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00103789	0.000346	19.8829
Error	5	0.00008700	0.000017	Prob>F
C Total	8	0.00112489	0.000141	0.0033

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	0.220000	0.00295
25	2	0.230500	0.00295
25caust	2	0.199500	0.00295
50	3	0.212000	0.00241

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		0.0127	0.00369	3.44	0.0184
Caustic		-0.0183	0.00369	-4.96	0.0042



Oneway Anova

Summary of Fit

RSquare	0.678312
RSquare Adj	0.485299
Root Mean Square Error	0.008805
Mean of Response	0.097778
Observations (or Sum Wt)	9

Analysis of Variance

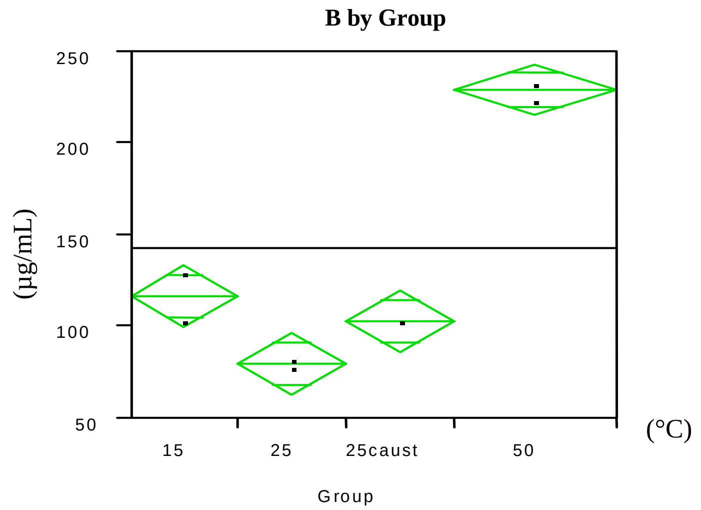
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00081743	0.000272	3.5143
Error	5	0.00038767	0.000078	Prob>F
C Total	8	0.00120510	0.000151	0.1048

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	0.114000	0.00623
25	2	0.100000	0.00623
25caust	2	0.092050	0.00623
50	3	0.089300	0.00508

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-0.00698	0.007792	-0.90	0.4113
Caustic		-0.01493	0.007792	-1.92	0.1135



Oneway Anova

Summary of Fit

RSquare	0.987634
RSquare Adj	0.980215
Root Mean Square Error	9.371944
Mean of Response	143.1111
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	35075.722	11691.9	133.1147
Error	5	439.167	87.8	Prob>F
C Total	8	35514.889	4439.4	<.0001

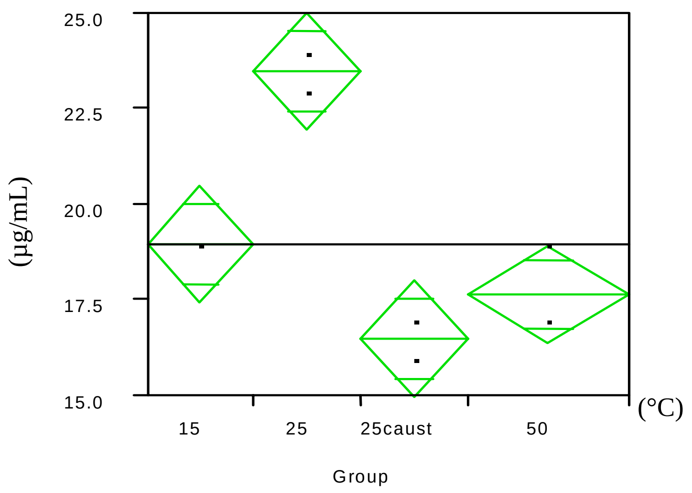
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	116.500	6.6270
25	2	80.000	6.6270
25caust	2	103.000	6.6270
50	3	229.667	5.4109

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-68.9124	8.288607	-8.31	0.0004
Caustic		-45.9124	8.288607	-5.54	0.0026

Cu by Group



Oneway Anova

Summary of Fit

RSquare	0.94086
RSquare Adj	0.905376
Root Mean Square Error	0.856349
Mean of Response	19
Observations (or Sum Wt)	9

Analysis of Variance

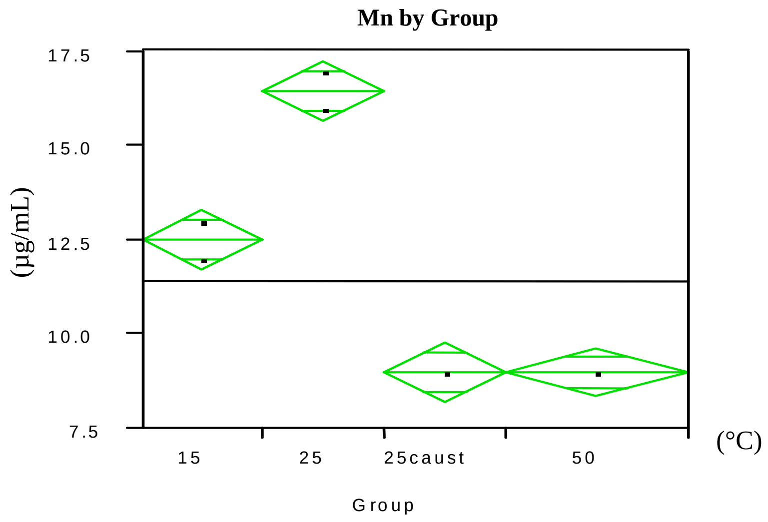
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	58.333333	19.4444	26.5152
Error	5	3.666667	0.7333	Prob>F
C Total	8	62.000000	7.7500	0.0017

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	19.0000	0.60553
25	2	23.5000	0.60553
25caust	2	16.5000	0.60553
50	3	17.6667	0.49441

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		4.873724	0.757356	6.44	0.0013
Caustic		-2.12628	0.757356	-2.81	0.0377



Oneway Anova

Summary of Fit

RSquare	0.988127
RSquare Adj	0.981003
Root Mean Square Error	0.447214
Mean of Response	11.44444
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	83.222222	27.7407	138.7037
Error	5	1.000000	0.2000	Prob>F
C Total	8	84.222222	10.5278	<.0001

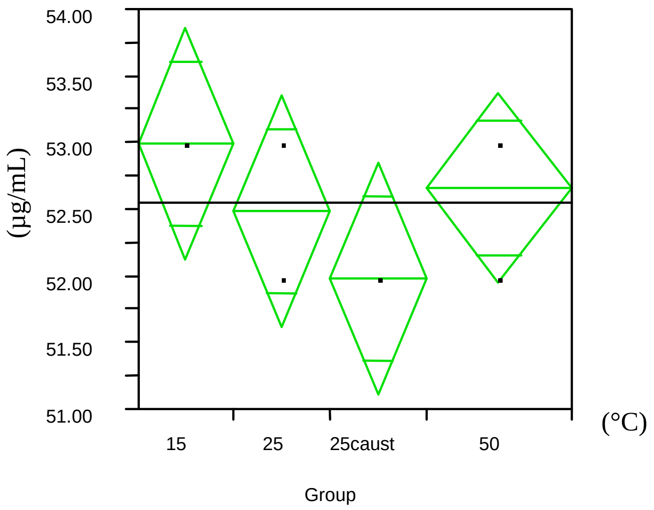
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	12.5000	0.31623
25	2	16.5000	0.31623
25caust	2	9.0000	0.31623
50	3	9.0000	0.25820

Std Error uses a pooled estimate of error variance

	Contrast	Std Err	t-dist	Pr>|t|
25 Degree	4.996	0.395519	12.63	0.0001
Caustic	-2.504	0.395519	-6.33	0.0014

Mo by Group



Oneway Anova

Summary of Fit

RSquare	0.475
RSquare Adj	0.16
Root Mean Square Error	0.483046
Mean of Response	52.55556
Observations (or Sum Wt)	9

Analysis of Variance

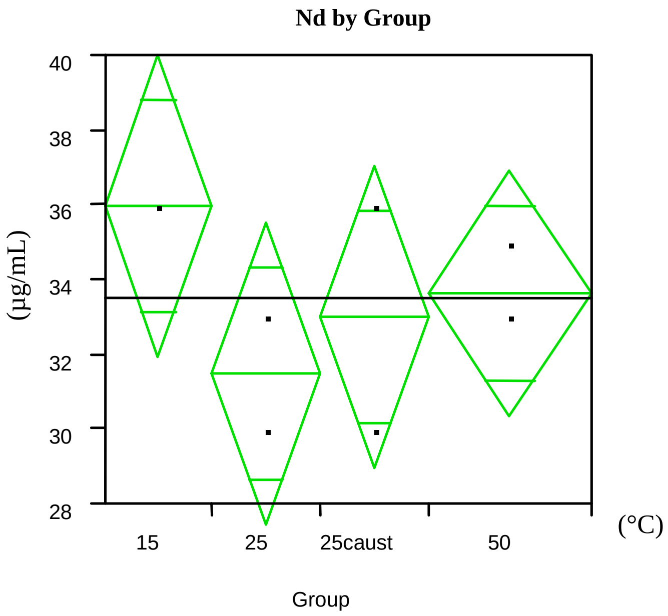
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1.0555556	0.351852	1.5079
Error	5	1.1666667	0.233333	Prob>F
C Total	8	2.2222222	0.277778	0.3204

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	53.0000	0.34157
25	2	52.5000	0.34157
25caust	2	52.0000	0.34157
50	3	52.6667	0.27889

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-0.42588	0.427213	-1.00	0.3646
Caustic		-0.92588	0.427213	-2.17	0.0824



Oneway Anova

Summary of Fit

RSquare	0.455529
RSquare Adj	0.128846
Root Mean Square Error	2.243509
Mean of Response	33.55556
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	21.055556	7.01852	1.3944
Error	5	25.166667	5.03333	Prob>F
C Total	8	46.222222	5.77778	0.3469

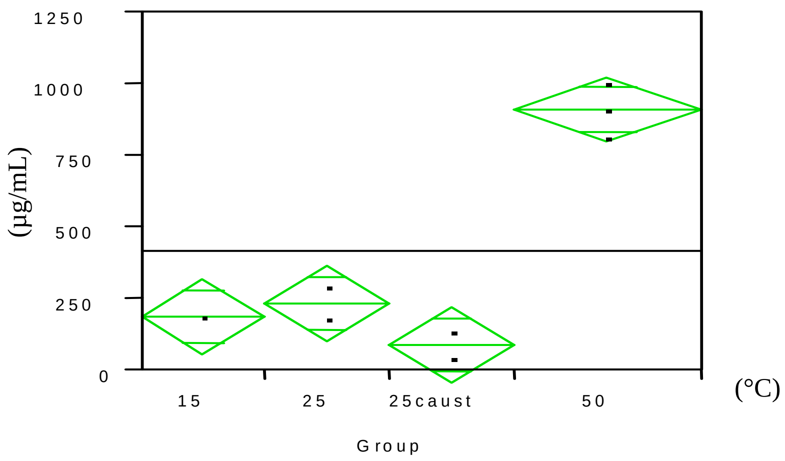
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	36.0000	1.5864
25	2	31.5000	1.5864
25caust	2	33.0000	1.5864
50	3	33.6667	1.2953

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-3.84708	1.984164	-1.94	0.1102
Caustic		-2.34708	1.984164	-1.18	0.2900

Si by Group



Oneway Anova

Summary of Fit

RSquare	0.974813
RSquare Adj	0.9597
Root Mean Square Error	75.67452
Mean of Response	416.8889
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1108173.7	369391	64.5041
Error	5	28633.2	5727	Prob>F
C Total	8	1136806.9	142101	0.0002

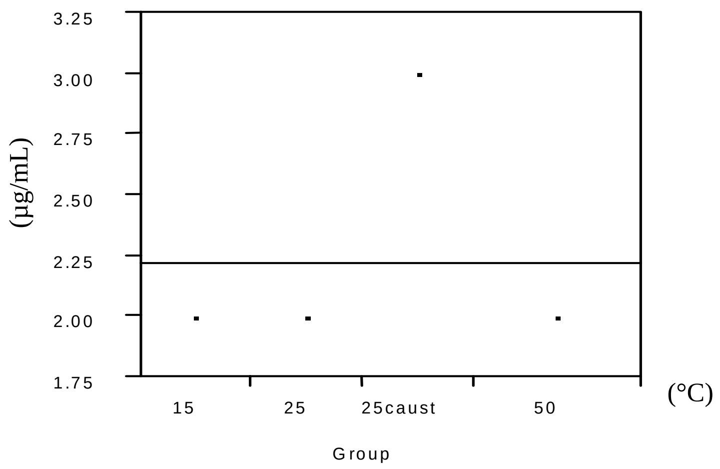
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	187.000	53.510
25	2	235.000	53.510
25caust	2	91.500	53.510
50	3	908.333	43.691

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-158.376	66.92674	-2.37	0.0642
Caustic		-301.876	66.92674	-4.51	0.0063

Sr by Group



Oneway Anova

Summary of Fit

RSquare	1
RSquare Adj	1
Root Mean Square Error	?
Mean of Response	2.222222
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1.5555556	0.518519	?
Error	5	-0.0000000	-1.1e-15	Prob>F
C Total	8	1.5555556	0.194444	?

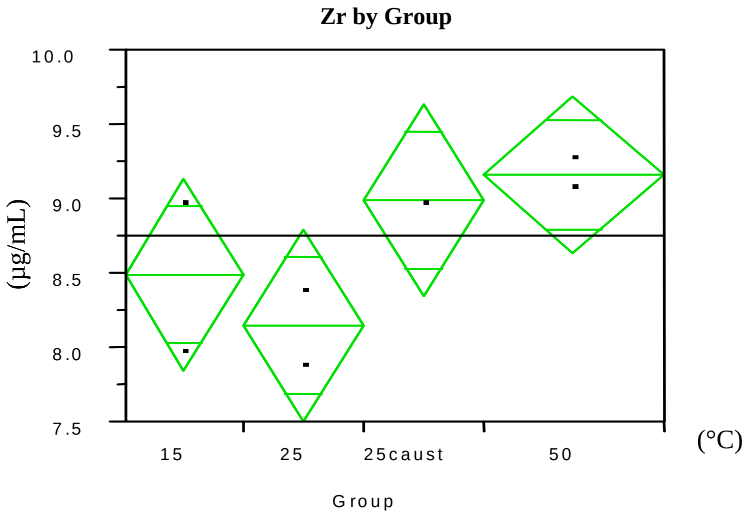
Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	2.00000	?
25	2	2.00000	?
25caust	2	3.00000	?
50	3	2.00000	?

Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-0.0008	?[a]	?	?
Caustic		0.9992	?	?	?

(a) The standard error is reported as zero, so the t-distribution and probability calculations are undefined.



Oneway Anova

Summary of Fit

RSquare	0.695799
RSquare Adj	0.513278
Root Mean Square Error	0.361017
Mean of Response	8.755556
Observations (or Sum Wt)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1.4905556	0.496852	3.8122
Error	5	0.6516667	0.130333	Prob>F
C Total	8	2.1422222	0.267778	0.0919

Means for Oneway Anova

Level	Number	Mean	Std Error
15	2	8.50000	0.25528
25	2	8.15000	0.25528
25 caustic	2	9.00000	0.25528
50	3	9.16667	0.20843

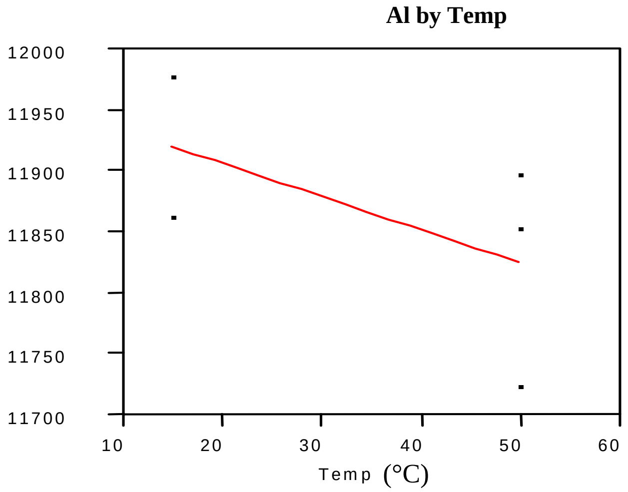
Std Error uses a pooled estimate of error variance

		Contrast	Std Err	t-dist	Pr>|t|
25 Degree		-0.54407	0.319287	-1.70	0.1491
Caustic		0.305932	0.319287	0.96	0.3820

Appendix B

Statistical Evaluation of Temperature on Analyte Solubility at 15° and 50°C

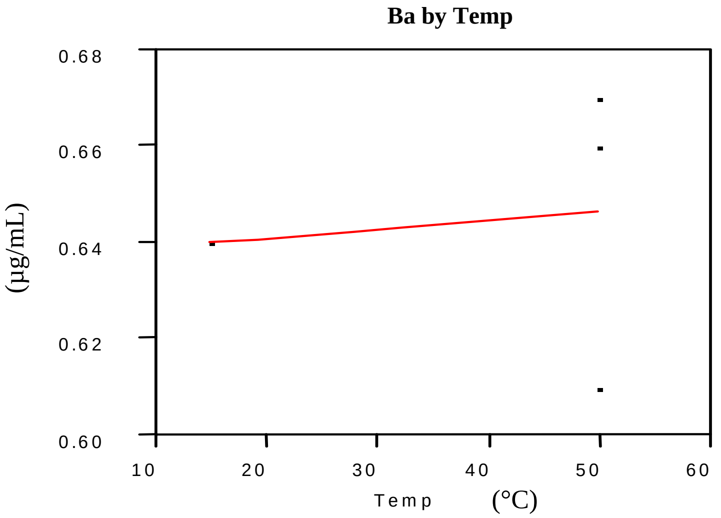
Appendix B: Statistical Evaluation of Temperature on Analyte Solubility at 15° and 50°C



RSquare	0.321053
Root Mean Square Error	87.52746
Mean of Response	11863.4
Observations (or Sum Wt)	5

Parameter Estimates

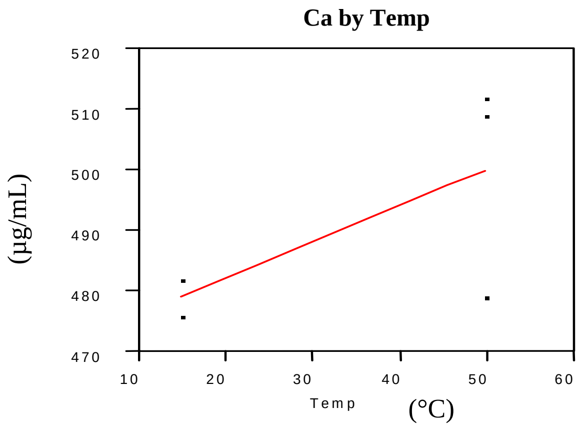
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	11961.286	91.02993	131.40	<.0001
Temp	-2.719048	2.282893	-1.19	0.3193



RSquare	0.025157
Root Mean Square Error	0.026247
Mean of Response	0.644
Observations (or Sum Wt)	5

Parameter Estimates

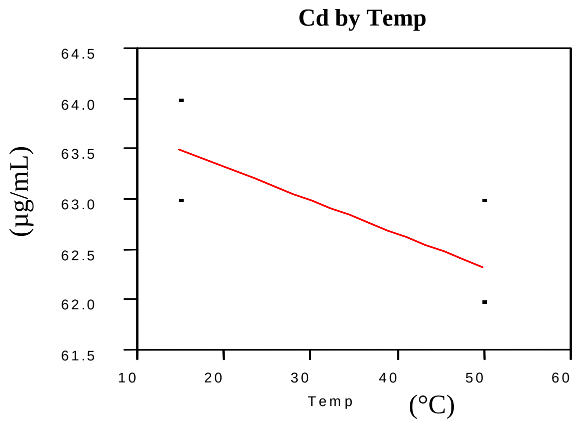
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.6371429	0.027297	23.34	0.0002
Temp	0.0001905	0.000685	0.28	0.7989



RSquare	0.436202
Root Mean Square Error	15.09967
Mean of Response	491.6
Observations (or Sum Wt)	5

Parameter Estimates

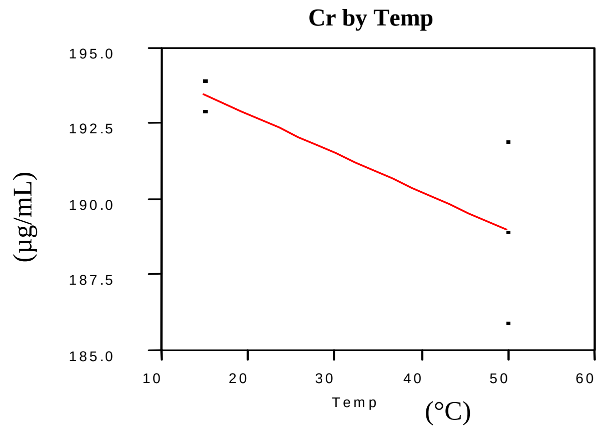
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	470	15.70389	29.93	<.0001
Temp	0.6	0.39383	1.52	0.2250



RSquare	0.583333
Root Mean Square Error	0.62361
Mean of Response	62.8
Observations (or Sum Wt)	5

Parameter Estimates

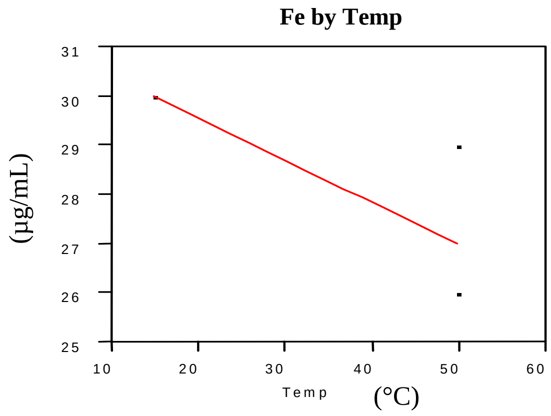
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	64	0.648564	98.68	<.0001
Temp	-0.033333	0.016265	-2.05	0.1328


Cr by Temp

RSquare	0.567757
Root Mean Square Error	2.483277
Mean of Response	190.8
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	195.42857	2.582647	75.67	<.0001
Temp	-0.128571	0.064769	-1.99	0.1414

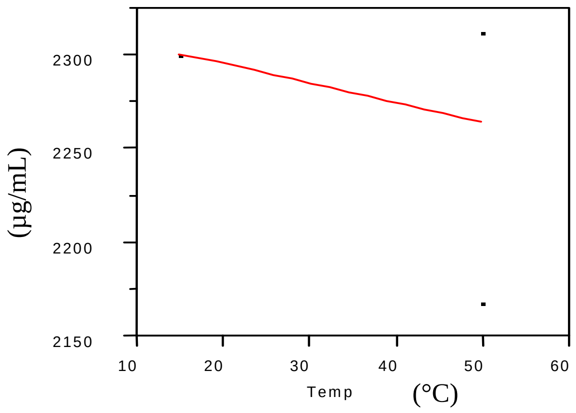

Fe by Temp

RSquare	0.642857
Root Mean Square Error	1.414214
Mean of Response	28.2
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	31.285714	1.470804	21.27	0.0002
Temp	-0.085714	0.036886	-2.32	0.1027

K by Temp

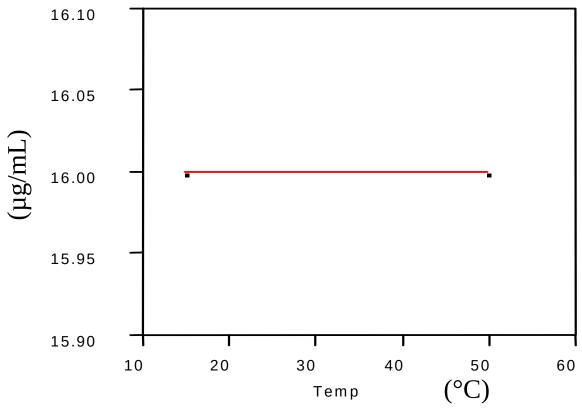


RSquare	0.101542
Root Mean Square Error	68.35366
Mean of Response	2279.2
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	2316.5714	71.08887	32.59	<.0001
Temp	-1.038095	1.782802	-0.58	0.6012

La by Temp

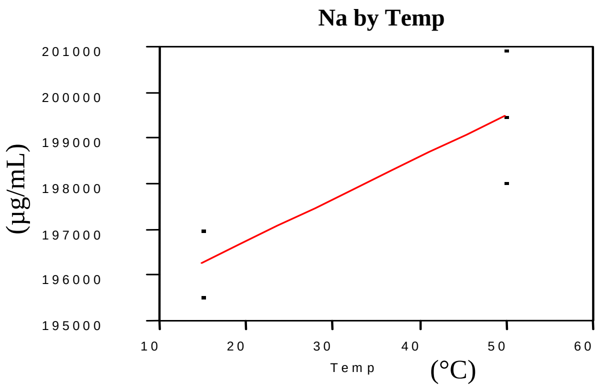


RSquare	?
Root Mean Square Error	0
Mean of Response	16
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	16	0	?	?[a]
Temp	0	0	?	?

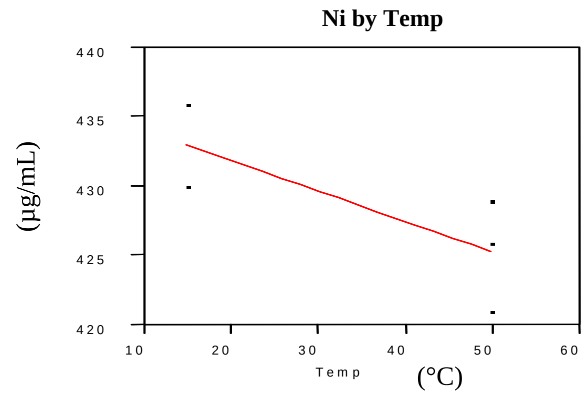
(a) There is no estimate of variability or the estimate of variability is zero. The t ratio is a ratio then divided by zero. The out is the question mark. For La, the estimate of variability is zero.



RSquare	0.704693
Root Mean Square Error	1318.188
Mean of Response	198211.8
Observations (or Sum Wt)	5

Parameter Estimates

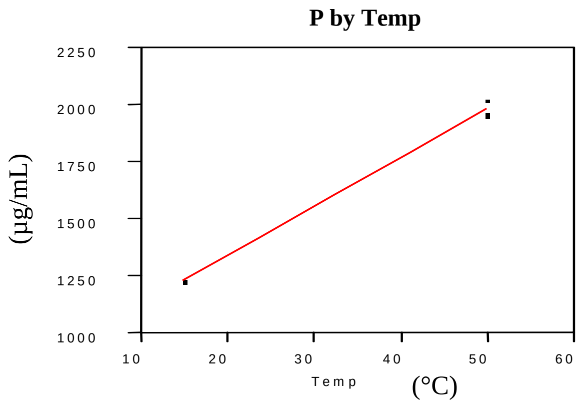
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	194900.14	1370.937	142.17	<.0001
Temp	91.990476	34.38103	2.68	0.0753



RSquare	0.581958
Root Mean Square Error	4.109609
Mean of Response	428.4
Observations (or Sum Wt)	5

Parameter Estimates

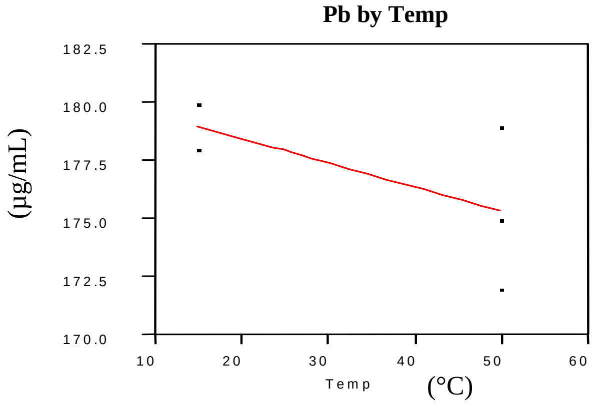
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	436.28571	4.274058	102.08	<.0001
Temp	-0.219048	0.107187	-2.04	0.1336



RSquare	0.995612
Root Mean Square Error	31.436
Mean of Response	1681.2
Observations (or Sum Wt)	5

Parameter Estimates

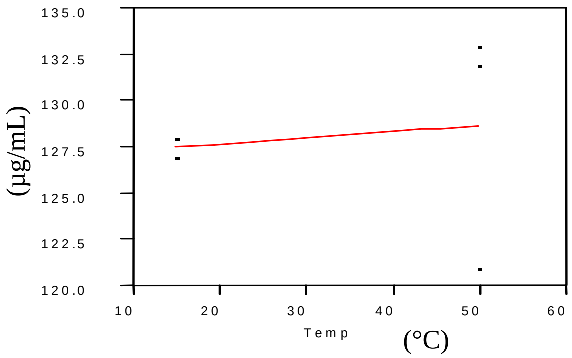
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	911.14286	32.69394	27.87	0.0001
Temp	21.390476	0.819915	26.09	0.0001



RSquare	0.376947
Root Mean Square Error	2.981424
Mean of Response	176.8
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	180.57143	3.100728	58.24	<.0001
Temp	-0.104762	0.077762	-1.35	0.2706

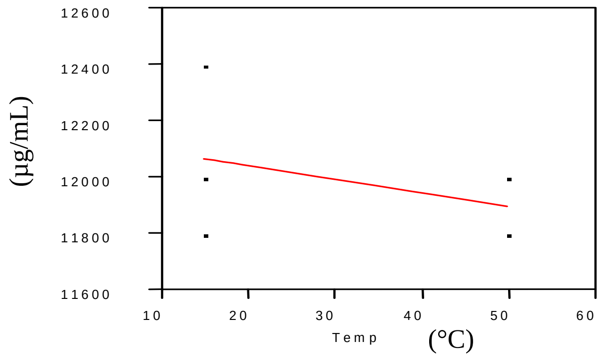


U by Temp

RSquare	0.017988
Root Mean Square Error	5.451809
Mean of Response	128.2
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	127	5.669967	22.40	0.0002
Temp	0.0333333	0.142194	0.23	0.8297

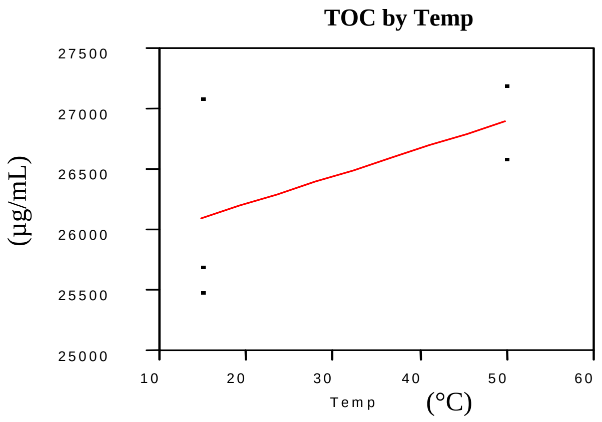


TIC by Temp

RSquare	0.138889
Root Mean Square Error	262.4669
Mean of Response	12000
Observations (or Sum Wt)	5

Parameter Estimates

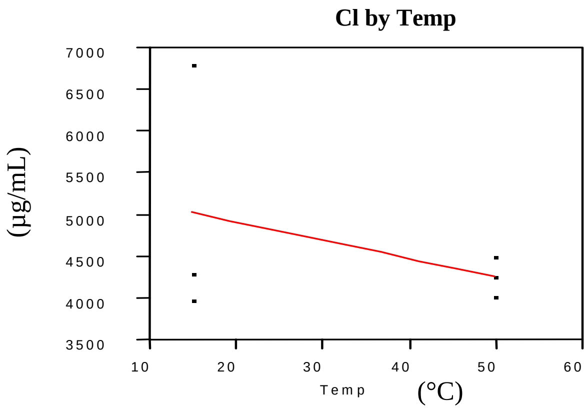
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	12138.095	230.629	52.63	<.0001
Temp	-4.761905	6.845669	-0.70	0.5367



RSquare	0.311183
Root Mean Square Error	752.7727
Mean of Response	26420
Observations (or Sum Wt)	5

Parameter Estimates

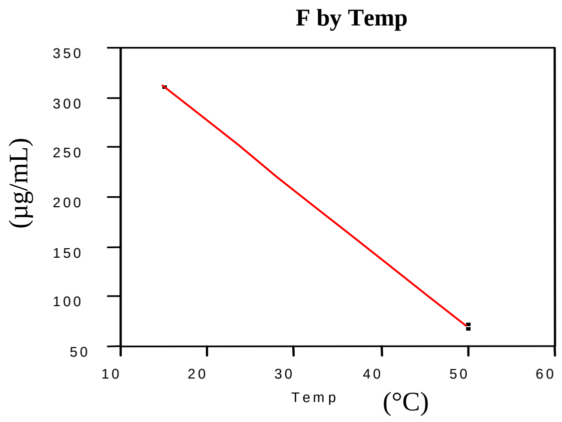
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	25757.143	661.4593	38.94	<.0001
Temp	22.857143	19.63384	1.16	0.3285



RSquare	0.152879
Root Mean Square Error	1105.63
Mean of Response	4649.167
Observations (or Sum Wt)	6

Parameter Estimates

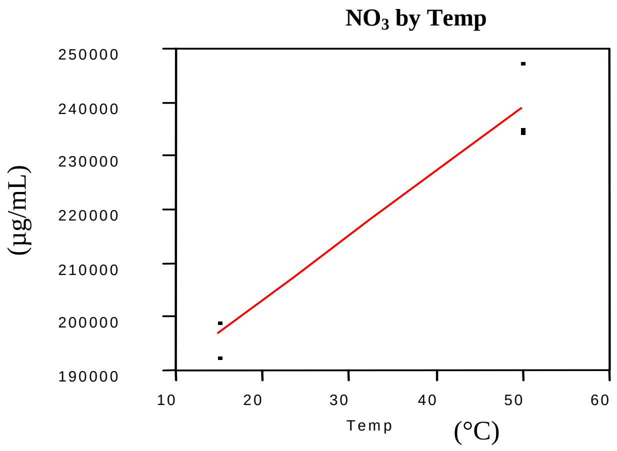
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	5361.381	952.0604	5.63	0.0049
Temp	-21.91429	25.79267	-0.85	0.4434



RSquare	0.999878
Root Mean Square Error	1.632993
Mean of Response	191.9167
Observations (or Sum Wt)	6

Parameter Estimates

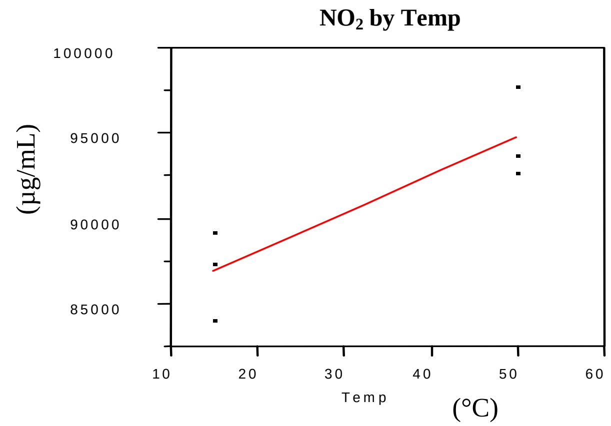
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	415.85714	1.406174	295.74	<.0001
Temp	-6.890476	0.038095	-180.9	<.0001



RSquare	0.950441
Root Mean Square Error	5886.36
Mean of Response	218207.3
Observations (or Sum Wt)	6

Parameter Estimates

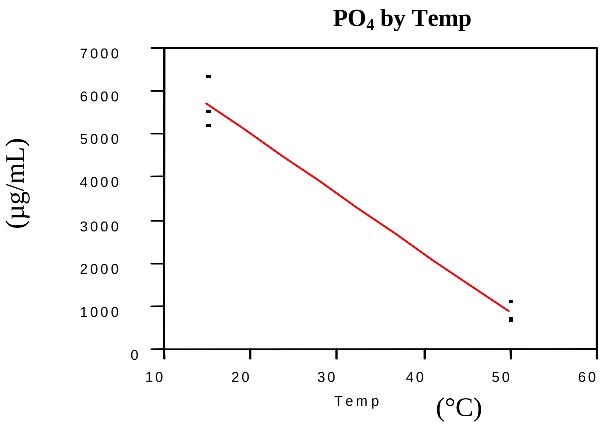
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	179118.81	5068.756	35.34	<.0001
Temp	1202.7238	137.3198	8.76	0.0009


NO_2 by Temp

RSquare	0.768033
Root Mean Square Error	2649.035
Mean of Response	90874.33
Observations (or Sum Wt)	6

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	83565.238	2281.089	36.63	<.0001
Temp	224.89524	61.79794	3.64	0.0220

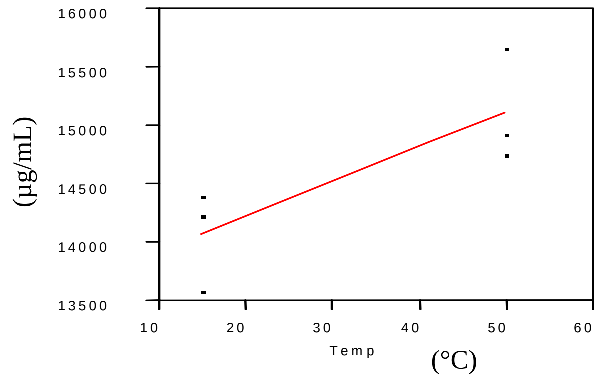

PO_4 by Temp

RSquare	0.977579
Root Mean Square Error	450.5611
Mean of Response	3314.5
Observations (or Sum Wt)	6

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	7825.8095	387.9791	20.17	<.0001
Temp	-138.8095	10.5109	-13.21	0.0002

SO_4 by Temp

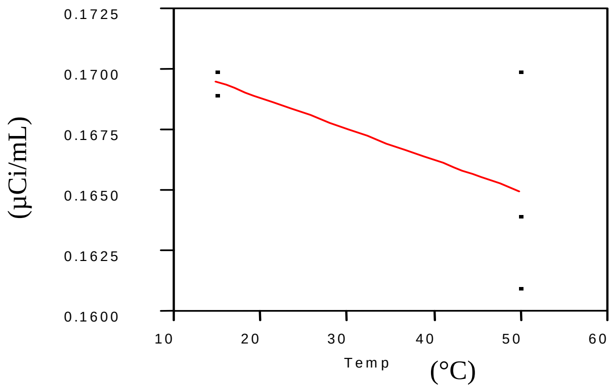


RSquare	0.659932
Root Mean Square Error	458.9334
Mean of Response	14595.33
Observations (or Sum Wt)	6

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	13625.905	395.1884	34.48	<.0001
Temp	29.828571	10.70621	2.79	0.0495

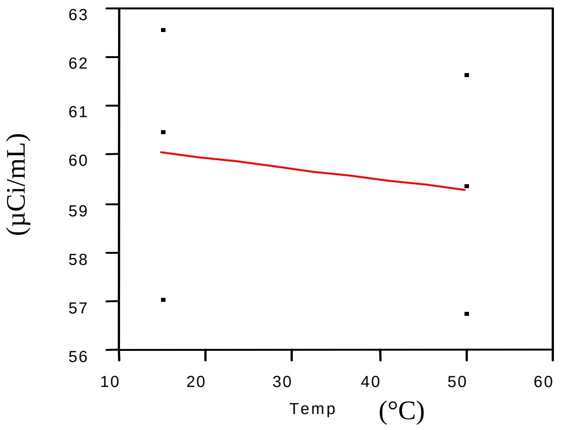
^{99}Tc by Temp



RSquare	0.363772
Root Mean Square Error	0.003764
Mean of Response	0.1668
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1714286	0.003914	43.79	<.0001
Temp	-0.000129	0.000098	-1.31	0.2816

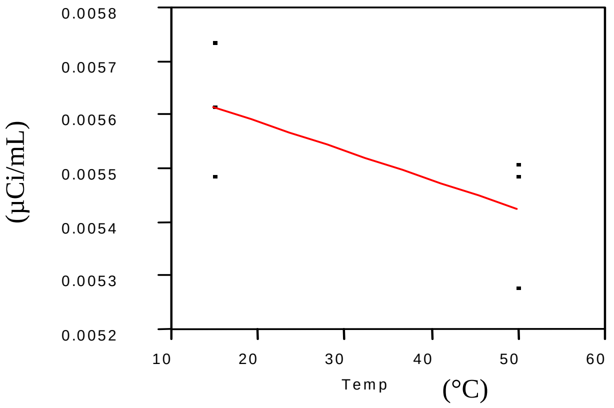


^{90}Sr by Temp

RSquare	0.031145
Root Mean Square Error	2.618524
Mean of Response	59.68333
Observations (or Sum Wt)	6

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	60.395238	2.254816	26.78	<.0001
Temp	-0.021905	0.061086	-0.36	0.7380

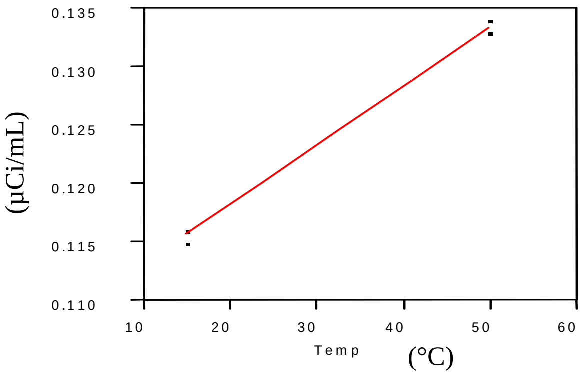


$^{239/240}$Pu by Temp

RSquare	0.459352
Root Mean Square Error	0.000126
Mean of Response	0.005522
Observations (or Sum Wt)	6

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0056981	0.000109	52.42	<.0001
Temp	-0.000005	0.000003	-1.84	0.1390

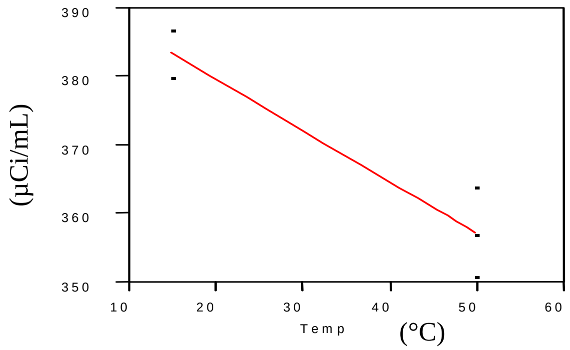


^{241}Am by Temp

RSquare	0.99716
Root Mean Square Error	0.000577
Mean of Response	0.1245
Observations (or Sum Wt)	6

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1080952	0.000497	217.43	<.0001
Temp	0.0005048	0.000013	37.48	<.0001



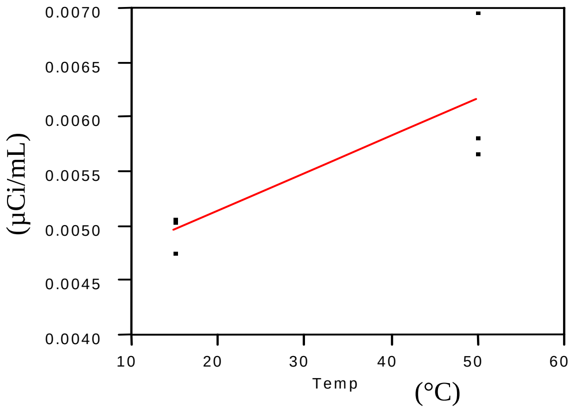
^{137}Cs by Temp

RSquare	0.882717
Root Mean Square Error	6.03232
Mean of Response	367.8
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	394.71429	6.273708	62.92	<.0001
Temp	-0.747619	0.157335	-4.75	0.0177

$^{243/244}$Cm by Temp

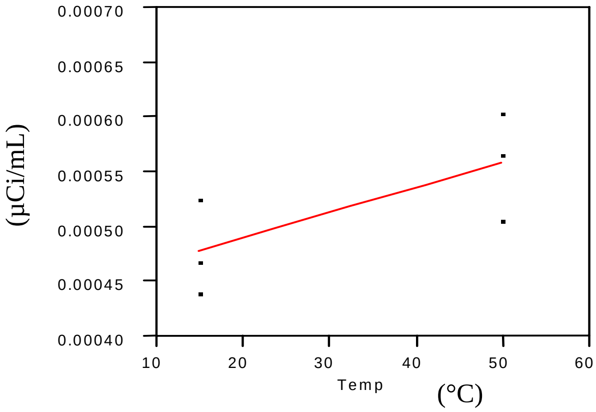


RSquare	0.667469
Root Mean Square Error	0.000517
Mean of Response	0.005565
Observations (or Sum Wt)	6

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0044538	0.000445	10.00	0.0006
Temp	0.0000342	0.000012	2.83	0.0472

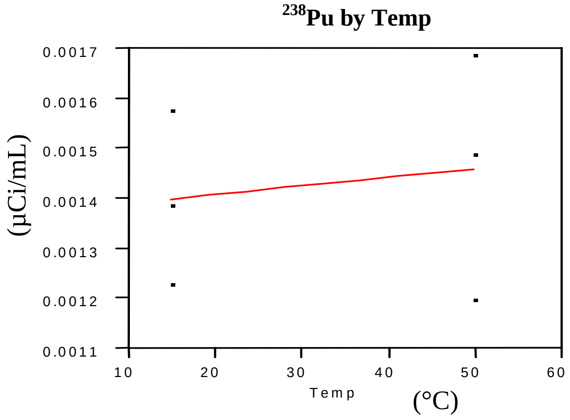
^{242}Cm by Temp



RSquare	0.528081
Root Mean Square Error	0.000047
Mean of Response	0.000519
Observations (or Sum Wt)	6

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0004442	0.00004	11.09	0.0004
Temp	0.0000023	0.000001	2.12	0.1018

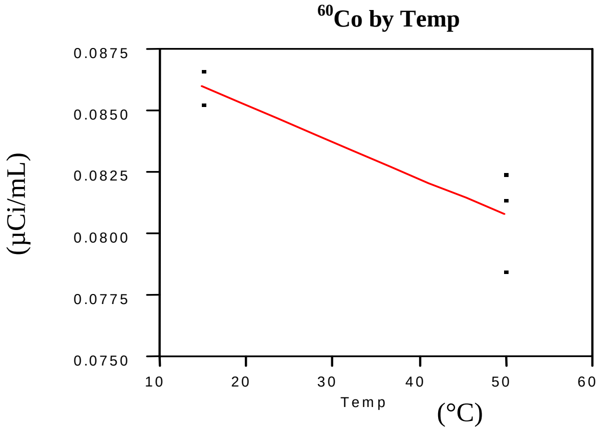


^{238}Pu by Temp

RSquare	0.028693
Root Mean Square Error	0.000214
Mean of Response	0.00143
Observations (or Sum Wt)	6

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0013743	0.000184	7.47	0.0017
Temp	0.0000017	0.000005	0.34	0.7483

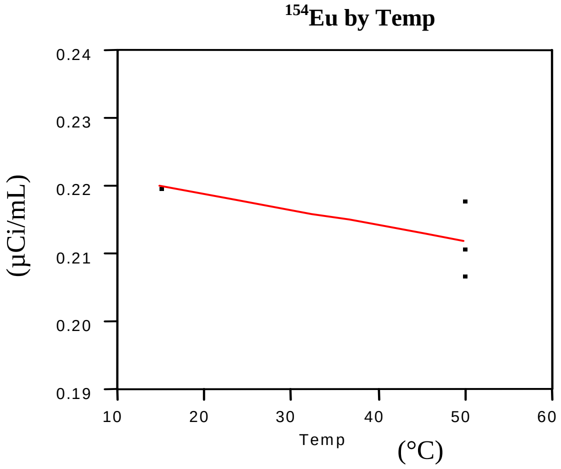


^{60}Co by Temp

RSquare	0.773161
Root Mean Square Error	0.001781
Mean of Response	0.08288
Observations (or Sum Wt)	5

Parameter Estimates

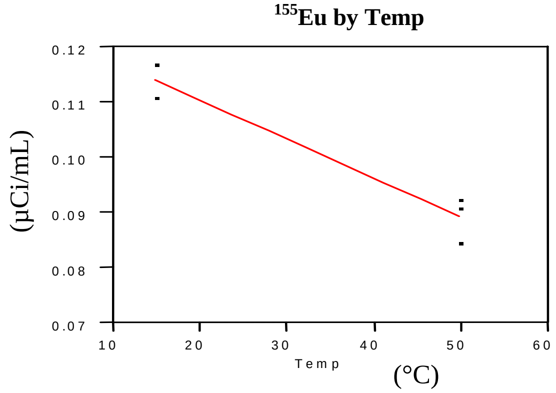
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0882286	0.001853	47.62	<.0001
Temp	-0.000149	0.000046	-3.20	0.0494



RSquare	0.553314
Root Mean Square Error	0.004546
Mean of Response	0.2152
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.2234286	0.004728	47.26	<.0001
Temp	-0.000229	0.000119	-1.93	0.1495

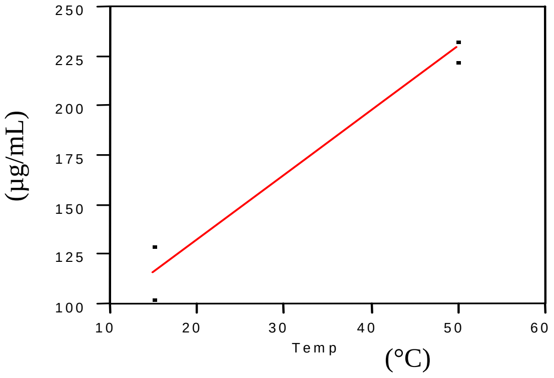


RSquare	0.933373
Root Mean Square Error	0.004174
Mean of Response	0.09918
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1245857	0.004341	28.70	<.0001
Temp	-0.000706	0.000109	-6.48	0.0075

B by Temp

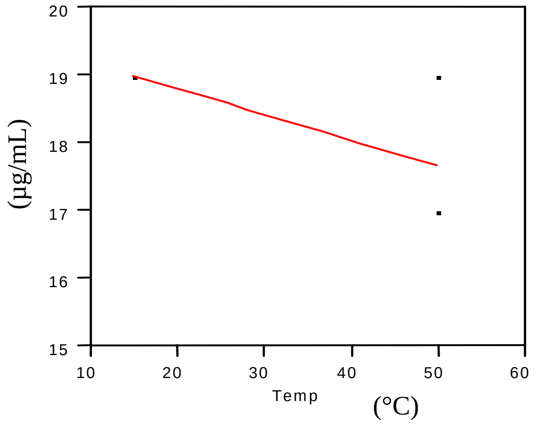


RSquare	0.97271
Root Mean Square Error	11.98842
Mean of Response	184.4
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	68	12.46815	5.45	0.0121
Temp	3.2333333	0.312682	10.34	0.0019

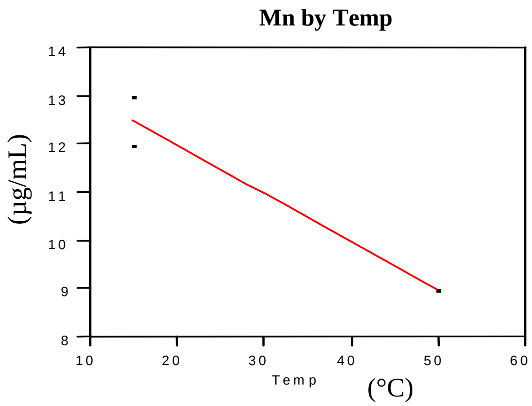
Cu by Temp



RSquare	0.444444
Root Mean Square Error	0.942809
Mean of Response	18.2
Observations (or Sum Wt)	5

Parameter Estimates

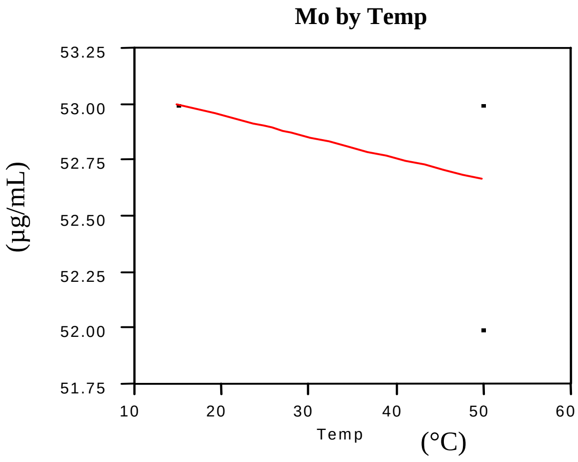
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	19.571429	0.980536	19.96	0.0003
Temp	-0.038095	0.02459	-1.55	0.2191



RSquare	0.967105
Root Mean Square Error	0.408248
Mean of Response	10.4
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	14	0.424585	32.97	<.0001
Temp	-0.1	0.010648	-9.39	0.0026

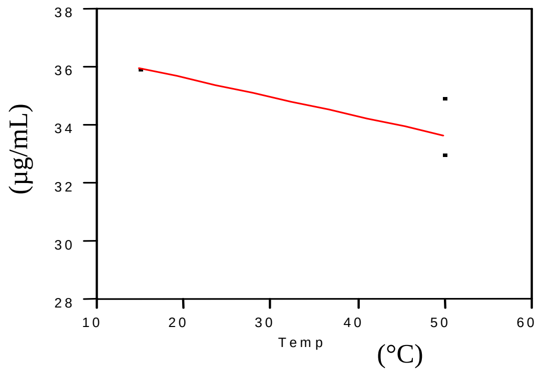


RSquare	0.166667
Root Mean Square Error	0.471405
Mean of Response	52.8
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	53.142857	0.490268	108.40	<.0001
Temp	-0.009524	0.012295	-0.77	0.4950

Nd by Temp

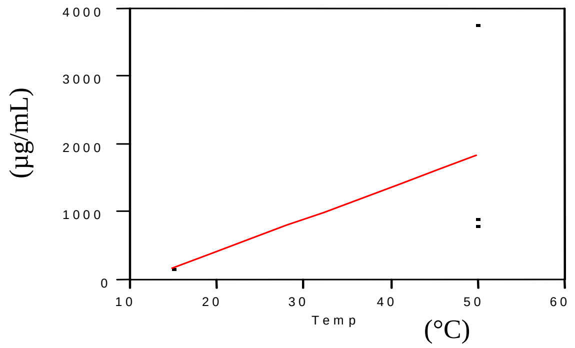


RSquare	0.710145
Root Mean Square Error	0.942809
Mean of Response	34.6
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	37	0.980536	37.73	<.0001
Temp	-0.066667	0.02459	-2.71	0.0731

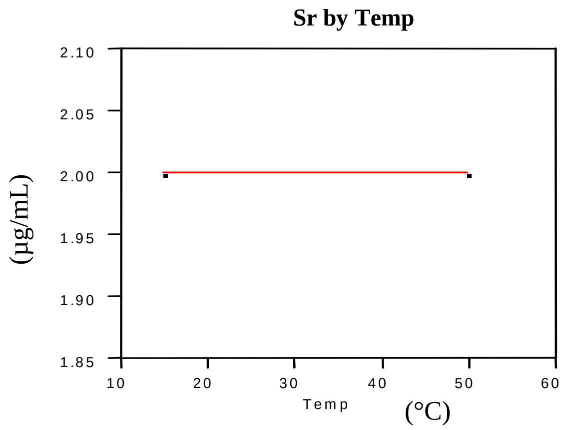
Si by Temp



RSquare	0.364088
Root Mean Square Error	1376.635
Mean of Response	1175.2
Observations (or Sum Wt)	5

Parameter Estimates

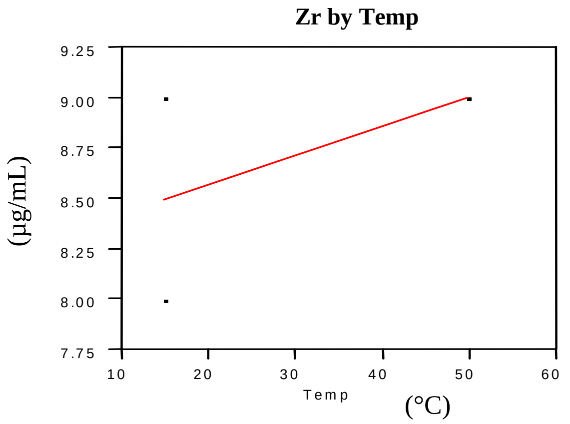
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-518.8571	1431.722	-0.36	0.7411
Temp	47.057143	35.90544	1.31	0.2813



RSquare	?
Root Mean Square Error	0
Mean of Response	2
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	2	0	?	?
Temp	0	0	?	?

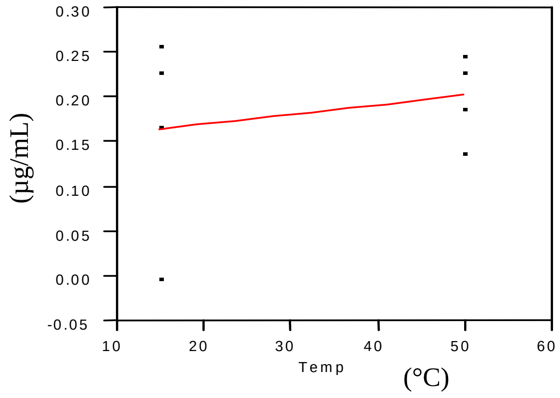


RSquare	0.375
Root Mean Square Error	0.408248
Mean of Response	8.8
Observations (or Sum Wt)	5

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	8.2857143	0.424585	19.51	0.0003
Temp	0.0142857	0.010648	1.34	0.2722

OH by Temp

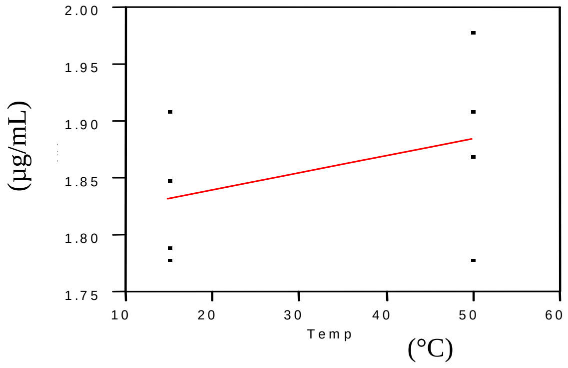


RSquare	0.055817
Root Mean Square Error	0.089046
Mean of Response	0.18375
Observations (or Sum Wt)	8

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1489286	0.066405	2.24	0.0661
Temp	0.0010714	0.001799	0.60	0.5732

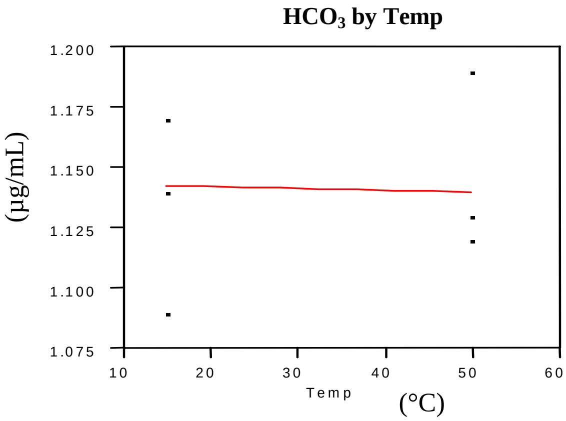
CO_3 by Temp



RSquare	0.147838
Root Mean Square Error	0.072772
Mean of Response	1.85875
Observations (or Sum Wt)	8

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.81	0.054269	33.35	<.0001
Temp	0.0015	0.00147	1.02	0.3470



RSquare	0.001626
Root Mean Square Error	0.035765
Mean of Response	1.14125
Observations (or Sum Wt)	8

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.1435714	0.026672	42.88	<.0001
Temp	-0.000071	0.000723	-0.10	0.9245

Appendix C

Interstitial Correction of Solids

Appendix C: Interstitial Correction of Solids

Table C.1. Data Tables of Interstitial Correction of Caustic Solids from Burgeson et al.

Analyte	AN-102 Caustic Solids WCS (μg/g)	AN-102 Caustic Solids UDS (μg/g)	Data Flag	Caustic Supernate (μg/mL)	Caustic Supernate (μg/g)	Solids (%)
Al	37700	30600	--	11600	8100	79
Ba	36	34	J	3	2	95
Bi	<37	--[a]	U	--	--	--
Ca	571	290	J	460	320	47
Cd	66	28	--	61	43	40
Cr	2270	2,150	--	191	133	94
Fe	1510	1500	--	27	19	99
Mn	406	--	--	--	--	--
Na	283000	158000	X	204000	143000	52
Ni	454	198	--	419	293	40
P	2100	1,050	--	1,800	1,200	46
Pb	352	244	J	178	124	66
Si	1110	1020	--	142	99	91
Sr	10	--	--	--	--	--
Zr	69	--	--	--	--	--
TIC	11700	4,770	--	11400	7950	38
Cl^-	2360	8,300	--	4160	2900	0
F^-	< 101	0	U	647	452	0
NO_2^-	52000	0	--	91220	63700	0
NO_3^-	126000	0	--	209134	146000	0
PO_4^{3-}	3800	0	--	6437	4500	0
SO_4^{2-}	10800	1730	--	14900	10400	14
As	2.80	- -		--	--	--
Be	0.77	--	U	--	--	--
K	2058	--	U	2400	1,660	27
Pu	0.067	608	--	--	--	--
Sb	0.046	--	J	--	--	--
Se	4.20	--	U	--	--	--
	μCi/g	**μCi/g**		**μCi/g**	**μCi/g**	
U	110	0	U	510	360	0
^{238}Pu	1.3E-03	4.9E-02	--	1.2E-03	9.E-04	98
$^{239/240}$Pu	4.6E-02	6.2E-02	--	5.3E-03	4.E-03	94
^{240}Cm	1.3E-03	1.0E-03	X	4.6E-04	3.E-04	76
$^{243/242}$Cm	4.6E-02	4.3E-02	JX	4.3E-03	3.E-03	93
^{60}Co	9.2E-02	4.4E-02	--	7.8E-02	5.E-02	45
^{137}Cs	3.6E+02	1.5E+02	--	3.5E+02	2.E+02	38
^{154}Eu	6.6E-01	5.4E-01	--	2.0E-01	1.E-01	79
^{155}Eu	3.0E-01	2.5E-01	J	8.2E-02	6.E-02	81
^{241}Am	5.5E-01	4.7E-01	X	1.3E-01	9.E-02	84

(a) Analytes marked with "- -" were not measured.

(b) In the Data Flag column a "--" indicates there was no data flag or that the data was good.

Table C.2. Data Tables of the Interstitial Correction of Caustic Solids from Urie. AN-102 Undissolved Solids (wet-weight and dry-weight basis) (µg/g)

Analyte	WCS				Supernatant[a]		UDS[b]	
	Measure Method	MDL µg/g	Average µg/g	Data Flag	Measure Method	Average µg/mL	Average[c] µg/g (wet)	Average µg/g (dry)
Ag	ICP-AES	2E+00	2E+00	UX	ICP-AES	0	<2E+00	<1E+01
Al	ICP-AES	5E+00	2.85E+04	--	ICP-AES	1.23E+04	2.14E+04	1.13E+05
B	ICP-AES	4E+00	9.35E+01	B	ICP-AES	8.00E+01	4.74E+01	2.51E+02
B	ICP-MS	2E+00	3.95E+01	BX	NM	NM	3.95E+01	2.09E+02
Ba	ICP-AES	8E-01	2.75E+01	--	ICP-AES	0	2.75E+01	1.46E+02
Be	ICP-AES	8E-01	9E-01	U	ICP-AES	0	<9E-01	<5E+00
Be	ICP-MS	3E-01	1.12E+00	J	NM	NM	1.12E+00	5.93E+00
Bi	ICP-AES	9E+00	9E+00	U	ICP-AES	0	<9E+00	<5E+01
Ca	ICP-AES	2E+01	4.21E+02	--	ICP-AES	4.89E+02	1.38E+02	7.33E+02
Cd	ICP-AES	1E+00	3.80E+01		ICP-AES	6.18E+01	2.38E+00	1.26E+01
Ce	ICP-AES	2E+01	1.85E+01	J	ICP-AES	0	1.85E+01	9.79E+01
Ce	ICP-MS	4E-01	2.30E+01	--	NM	NM	2.30E+01	1.22E+02
Cl	IC	3E+02	3.33E+03	B	IC	4.82E+03	5.50E+02	2.91E+03
Co	ICP-AES	4E+00	5E+00	U	ICP-AES	3.90E+00	<5E+00	<2E+01
Co	ICP-MS	8E-02	2.36E+00	--	NM	NM	2.36E+00	1.25E+01
Cr	ICP-AES	2E+00	1.83E+03	--	ICP-AES	2.15E+02	1.70E+03	9.00E+03
Cu	ICP-AES	2E+00	1.90E+01	J	ICP-AES	2.35E+01	5.44E+00	2.88E+01
Eu	ICP-AES	9E+00	9E+00	U	ICP-AES	0	<9E+00	<5E+01
F	IC	3E+02	3.80E+03	--	IC	0	3.80E+03	2.01E+04
Fe	ICP-AES	2E+00	1.17E+03	--	ICP-AES	3.75E+01	1.14E+03	6.05E+03
K	ICP-AES	2E+02	1.10E+03	JX	ICP-AES	1.98E+03	<2E+02[c]	<9E+02[c]
La	ICP-AES	4E+00	3.20E+01	J	ICP-AES	1.55E+01	2.31E+01	1.22E+02
Mn	ICP-AES	4E+00	2.58E+02	--	ICP-AES	1.65E+01	2.48E+02	1.31E+03
Mo	ICP-AES	4E+00	3.30E+01	J	ICP-AES	5.25E+01	2.72E+00	1.44E+01
Mo	ICP-MS	2E+00	3.30E+01	--	NM	NM	3.30E+01	1.75E+02
Na	ICP-AES	6E+01	1.67E+05	X	ICP-AES	1.84E+05	6.04E+04	3.19E+05
Nd	ICP-AES	9E+00	6.45E+01	J	ICP-AES	3.15E+01	4.63E+01	2.45E+02
Ni	ICP-AES	3E+00	2.58E+02	--	ICP-AES	4.16E+02	1.75E+01	9.28E+01
NO_2^-	IC	5E+02	5.51E+04	--	IC	8.54E+04	5.82E+03	3.08E+04
NO_3^-	IC	5E+02	1.33E+05	--	IC	2.21E+05	5.02E+03	2.66E+04
Oxalate	IC	5E+02	2.88E+04	X	IC	4.60E+02	2.85E+04	1.51E+05
P	ICP-AES	9E+00	1.41E+03	--	ICP-AES	1.82E+03	3.58E+02	1.89E+03
Pb	ICP-AES	9E+00	2.45E+02	B	ICP-AES	1.86E+02	1.37E+02	7.28E+02
PO_4^{3-}	IC	5E+02	8.18E+03	--	IC	0	8.18E+03	4.33E+04
Si	ICP-AES	4E+01	2.00E+02	JB	ICP-AES	2.35E+02	6.44E+01	3.41E+02
Sn	ICP-AES	1E+02	1E+02	U	ICP-AES	0	<1E+02	<7E+02
SO_4^{2-}	IC	5E+02	1.65E+04	--	IC	1.69E+04	6.73E+03	3.56E+04
Sr	ICP-AES	1E+00	6.15E+00	J	ICP-AES	2.30E+00	4.82E+00	2.55E+01
TIC-F[d]	TOC-TC	6E+02	1.63E+04	--	TOC-TC	2.31E+04	3.00E+03	1.59E+04
TIC-P[d]	TIC-TOC	7E+01	1.74E+04	--	TIC-TOC	1.10E+04	1.10E+04	5.84E+04
TOC-F[d]	TOC-TC	4E+02	1.97E+04	--	TOC-TC	1.81E+04	9.24E+03	4.89E+04

Table C.2. Con't

Analyte	WCS				Supernatant(a)		UDS(b)	
	Measure Method	**MDL µg/g**	**Average µg/g**	**Data Flag**	**Measure Method**	**Average µg/mL**	**Average(c) µg/g (wet)**	**Average µg/g (dry)**
TOC-P(d)	TIC-TOC	2E+02	2.56E+04	--	TIC-TOC	2.93E+04	8.73E+03	4.62E+04
U	ICP-AES	2E+02	2E+02	U	ICP-AES	0	<2E+02	<1E+03
U	ICP-MS	3E-01	6.55E+01	X	U(KPA)	1.19E+01	5.86E+01	3.10E+02
Zn	ICP-AES	4E+00	2.20E+01	J	ICP-AES	6.20E+00	2.20E+01	1.16E+02
Zr	ICP-AES	4E+00	4.65E+01	--	ICP-AES	8.15E+00	4.18E+01	2.21E+02
Organics								
Aroclor 1016/1242	GC/ECD	8.00E-03	8.00E-03	U	GC/ECD	0	<8E-03	<4E-02
Aroclor 1221	GC/ECD	8.00E-03	8.00E-03	U	GC/ECD	0	<8E-03	<4E-02
Aroclor 1232	GC/ECD	8.00E-03	8.00E-03	U	GC/ECD	0	<8E-03	<4E-02
Aroclor 1248	GC/ECD	8.00E-03	8.00E-03	U	GC/ECD	0	<8E-03	<4E-02
Aroclor 1254	GC/ECD	8.00E-03	1.70E-02	--	GC/ECD	0	1.70E-02	9.00E-02
Aroclor 1260	GC/ECD	8.00E-03	3.00E-02	--	GC/ECD	0	3.00E-02	1.59E-01
Total PCB	GC/ECD	4.80E-02	7.90E-02	--	GC/ECD	0	7.90E-02	4.18E-01

WCS = wet centrifuged solids; UDS = undissolved solids; NM = not measured.

(a) If the analyte is measured but not detected above the MDL, the supernatant analyte concentration is set to 0 (zero). If the analyte is not measured, the supernatant is assumed to have no contribution to the WCS results; the average field is left blank and the supernatant concentration is set to 0 (zero) when calculating the UDS concentration.

(b) If analyte is measured in the WCS but is not above the MDL the UDS results are set to <MDL of the WCS.

(c) When the calculated UDS results are <0 or =0, the UDS results are set to <MDL of the WCS.

(d) P = carbon from persulfate method; F = carbon from furnace method-TIC by difference (TIC=TC-TOC).

(e) In the Data Flag column a "--" indicates there was no data flag or that the data was good.

Table C.3. AN-102 Undissolved Solids (wet-weight and dry-weight basis) (µCi/g)

Analyte	WCS				Supernatant(a)		UDS(b)	
	Measure Method	**MDL/MDA µCi/g**	**Average µCi/g**	**Data Flag**	**Measure Method**	**Average µCi/mL**	**Average µCi/g WCS**	**Average µCi/g DS**
^{60}Co	GEA	3e-03	5.71e-02	--	GEA	8.49e-02	8.16e-03	4.32e-02
^{90}Sr	Rad	1e+00	1.44e+02	--	Rad	5.72e+01	1.11e+02	5.85e+02
^{99}Tc	ICP-MS	2e-03	9.90e-02	--	ICP-MS	1.48e-01	1.36e-02	7.21e-02
^{137}Cs	GEA	4e-02	2.16e+02	--	GEA	3.69e+02	3.44e+00	1.82e+01
^{152}Eu	GEA	1E-02	1E-02	U	NM	NM	<1E-02	<5E-02
^{154}Eu	GEA	9E-03	5.12E-01	--	NM	NM	5.12E-01	2.71E+00
^{155}Eu	GEA	2E-01	3.20E-01	--	GEA	0	3.20E-01	1.69E+00
^{233}U	ICP-MS	8E-06	5.60E-05	J	NM	NM	5.60E-05	2.96E-04
^{234}U	ICP-MS	3E-05	3E-05	U	NM	NM	<3E-05	<2E-04
^{235}U	ICP-MS	1E-08	9.87E-07	--	NM	NM	9.87E-07	5.22E-06
^{236}U	ICP-MS	2E-07	1.23E-06	J	NM	NM	1.23E-06	6.51E-06
^{238}U	ICP-MS	2E-07	2.19E-05		NM	NM	2.19E-05	1.16E-04
^{238}Pu	Rad	2E-03	1.19E-02	BJ	Rad	1.65E-03	1.10E-02	5.79E-02
^{239}Pu	ICP-MS	9E-04	5.56E-02	--	ICP-MS	6.47E-03	5.18E-02	2.74E-01
^{240}Pu	ICP-MS	6E-04	1.51E-02	--	ICP-MS	2.02E-03	1.39E-02	7.35E-02
$^{239/240}$Pu	Rad	2E-03	4.17E-02	--	Rad	5.90E-03	3.82E-02	2.02E-01
^{241}Pu	Rad	1E-01	1E-01	U	NM	NM	<1E-01	<5E-01
^{241}Am	GEA	2E-01	4.21E-01	J	GEA	1.65E-01	3.25E-01	1.72E+00
^{241}Am	Rad	2E-03	4.41E-01	--	Rad	1.51E-01	3.54E-01	1.87E+00
^{242}Cm	Rad	1E-03	2E-03	U	NM	NM	<2E-03	<8E-03
$^{243/244}$Cm	Rad	2E-03	1.72E-02	BJ	Rad	6.71E-03	1.33E-02	7.03E-02

WCS = wet centrifuged solids; UDS = undissolved solids; NM = not measured.

(a) If the analyte is measured but not detected above the MDL, the supernatant analyte concentration is set to 0 (zero). If the analyte is not measured, the supernatant is assumed to have no contribution to the WCS results; the average field is left blank and the supernatant concentration is set to 0 (zero) when calculating the UDS concentration.

(b) If analyte is measured in the WCS, but is not above the MDL, the UDS results is set to < MDL of the WCS.

(c) In the Data Flag column a “--“ indicates there was no data flag or that the data was good.

Appendix D

Test Specification for Determining Solubility Versus Temperature and Concentration for AN-102 Solids

Appendix D: Test Specification for Determining Solubility Versus Temperature and Concentration for AN-102 Solids

Appendix E

Test Plan for Determining Solubility of AN-102 Solids Versus Temperature and Concentration

Appendix E: Test Plan for Determining Solubility of AN-102 Solids Versus Temperature and Concentration

Appendix F

ICP-MS Data Report Impacted by Standards Bias

Appendix F: ICP-MS Data Report Impacted by Standards Bias

Project / WP#:	**42365/W57957**
ASR#:	6164
Client:	Ingrid Burgeson

Sample Identification:

RPL#	Client ID
01-1624	**AN102-15A**
01-1625	**AN102-15B**
01-1626	**AN102-50A**
01-1627	**AN102-50B**
01-1627D	**AN102-50B-Dup**
01-1628	**AN102-Caustic A**
01-1629	**AN102-Caustic B**

Data Impact Narrative:

A data bias for Tc-99 was found when standards were analyzed using standard radionuclide counting techniques. The bias was reported by Karl Pool, the Analytical Support Operations Lead in the 325 building (Radiochemical Processing Laboratory). The biased data for Tc-99 was used in analysis and a brief discussion of that impact follows.

In report WTP-RPT-052: “Evaluation of Temperature and Caustic Effects on Tank Waste Solubility for Hanford Tank 241-AN-102”, the relative changes in solids and supernatant composition with change in temperature and caustic addition were evaluated. Since the samples were “batch analyzed”, for all samples, the calibration was consistent for each analyte and the magnitude of the relative changes was not affected by the bias. For Tc-99, the data taken at 15°C and 50C°, for instance, was well above MDL and did not did not seem to suffer any internal consistency. Any changes in analyte concentration between 15°C and 50°C were negligible, as seen below in the table. Additionally, these data were compared to data obtained at 25°C by Urie et al (2002), also shown in the table below. With regard to the standards bias, it is important to note that the two sets of data were obtained about one year apart. The changes between the two data sets, per analyte, are within statistical error -or differences in the sample management, as discussed in WTP-RPT-052.

Action:

The data bias appears to have had no impact on the relative changes across the temperatures evaluated. However, the impacted data has been flagged in each data table in this revision of

WTP-RPT-052. rev.1 for indication purposes only and should only be used for estimation purposes, as the reported bias may have an impact on the absolute concentration of Tc-99.

	MDL / MDA (μg/mL)	**AN-102 15°C Sample A (μg/mL)**	**AN-102 15°C Sample A Duplicate (μg/mL)**	**AN-102 15°C Sample B (μg/mL)**
^{99}Tc	6E-06	1.7E-01	1.7E-01	--
	MDL / MDA (μg/mL)	**AN-102 50°C Sample A (μg/mL)**	**AN-102 50°C Sample A Duplicate (μg/mL)**	**AN-102 50°C Sample B (μg/mL)**
^{99}Tc	6E-06	1.7E-01	1.6E-01	1.6E-01
	MDL / MDA (μg/mL)	**AN-102 25°C Sample A (μg/mL)**	**AN-102 25°C Sample A Duplicate (μg/mL)**	
^{99}Tc	6E-06	1.5E-01		^{99}Tc

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