VITRIFICATION OF M-AREA MIXED (HAZARDOUS AND RADIOACTIVE) F006 WASTES: I. SLUDGE AND SUPERNATE CHARACTERIZATION (U)

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EXECUTIVE SUMMARY

Solidification of Resource Conservation and Recovery Act (RCRA) "listed" mixed (hazardous and radioactive) wastes that were generated in the Savannah River Site's (SRS) M-Area facility were recently vitrified into borosilicate glass. The initial concept of solidification into glass began in 1987 when the sludge and supernates reported in this study were first analyzed at the Savannah River Technology Center (SRTC). A year later, the SRTC Professional Planning Committee (PPC) recommended that high nitrate containing (low-level) wastes, such as those existing in M-Area, be considered for vitrification. The investigation into vitrification of low-level high nitrate containing wastes was considered timely because of the potential for large waste volume reduction compared to alternative solidification into cement. Glass formulations were proposed by SRTC in the 1990-1994 timeframe and were used as the basis for privatization of a vitrification Vendor Treatment Facility (VTF).

The M-Area operations at the Savannah River Site (SRS) in Aiken, South Carolina, produced reactor components for nuclear weapons materials for the U.S. Department of Energy. The resulting waste was stored in nine tanks. The total volume in storage was initially ~1,200,000 gallons of which ~1/3 was a gelatinous hydroxide sludge. The supernate in the tanks was treated in a wastewater treatment facility and the resulting effluent released to a National Pollution Discharge Elimination System (NPDES) permitted outfall stream on the SRS site. There was approximately 425,000 gallons of waste sludge, which contained high concentrations of aluminum, sodium, uranium, and nitrate. The sludge was mixed with 275,000 gallons of spent SiO_2 rich filter aids. During the ensuing years of wastewater treatment of the supernate the type of filter aid varied from diatomaceous earth to perlite and/or perflo. All the filter aids were SiO_2 enriched and were of high surface area.

Vitrification into borosilicate glass was chosen as the preferred stabilization option because it reduced the volume of sludge by ~85% and reduced the final disposal volume by 96% compared to alternative stabilization technologies. The large volume reduction will allow for large associated savings in disposal and/or long term storage costs once the vitrified waste form is delisted.

The techniques used to chemically analyze the waste sludge, supernate, spent filter aid, and a "crust" that formed on the settled sludge solids are documented in this study. The analytic methods used to identify the phases present in the waste sludge are also detailed. All supernate chemical analyses were assessed for accuracy and consistency by performing mass balance calculations on the species in solution. All sludge chemical analyses were assessed for accuracy and consistency by performing mass balance calculations on the species identified by x-ray diffraction.

Zeolite-A was identified in the sludges when no zeolite had ever been used in the M-Area processes. Experiments were performed in this study to demonstrate that the Zeolite-A formed in-situ in the tanks at ambient temperature from reaction of the silica containing filter aids and the aluminate rich waste in a high caustic environment.

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INTRODUCTION

Technologies are being developed by the US Department of Energy's (DOE) Nuclear Facility sites to convert low-level and mixed (hazardous and radioactive) wastes to a solid stabilized waste form for permanent disposal. One of the alternative technologies is vitrification into a borosilicate glass waste form. The Environmental Protection Agency (EPA) has declared vitrification the Best Demonstrated Available Technology (BDAT) for high-level radioactive mixed waste[1] and produced a Handbook of Vitrification Technologies for Treatment of Hazardous and Radioactive Waste.[2] The DOE Office of Technology Development (OTD) has taken the position that mixed waste needs to be stabilized to the highest level reasonably possible to ensure that the resulting waste forms will meet both current and future regulatory specifications. Stabilization of low level and hazardous wastes in glass are in accord with the 1988 Savannah River Technology Center (SRTC), then the Savannah River Laboratory (SRL), Professional Planning Committee (PPC) recommendation[3] that high nitrate containing (low-level) wastes be incorporated into a low temperature glass (via a sol-gel technology). The investigation into this new technology was considered timely because of the potential for large waste volume reduction compared to solidification into cement.

Vitrification produces durable waste forms at volume reductions up to 97%.[4] Large reductions in volume minimize long-term storage costs, making vitrification a cost effective technology on a life cycle basis.[5] The US DOE Savannah River Site (SRS), which is operated by Westinghouse Savannah River Company (WSRC), investigated vitrification for disposal of various low-level and mixed wastes.[4,6] The first /mixed wastes vitrified in laboratory studies at SRS were the M-Area nickel plating line wastes reported in this study.

The M-Area operations at the SRS in Aiken, South Carolina, produced reactor components for nuclear weapons materials for the U.S. Department of Energy. The mixed waste resulting from these operations was a listed Resource Conservation and Recovery Act (RCRA) F006 waste[‡] which was stored in the M-Area Process Waste Interim Treatment/Storage Facility (PWIT/SF). The total volume in storage was initially ~1,200,000 gallons of which ~33% was a gelatinous hydroxide sludge containing high concentrations of aluminum, sodium, uranium, and nitrate plus the RCRA metal of concern, nickel. It had been demonstrated[7,8] that a volume reduction of 65-70% of the M-Area stored waste could be achieved by wastewater treatment of the supernate, with the resulting effluent released via a permitted National Pollution Discharge Elimination

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[‡] a wastewater treatment sludge from electroplating operations

System (NPDES) outfall. About 275,000 gallons of spent SiO_2 rich filter aids (perlite/perflo) were generated from the supernate treatment.

In January l989 South Carolina Department of Health and Environmental Control (SCDHEC) approved a construction permit for the DETF Supernate Transfer Facility. In May 1990 the DETF received a permit to operate the DETF. This allowed the DETF to be used to treat the supernate from the M-Area Interim Treatment/Storage Facility (IT/SF) tanks. There were six 35,000 gallon tanks (Tanks 1-6) and three 500,000 gallon tanks (Tanks 7, 8, 10). Tank 10 was filled with the spent filter aid. Treatment of the supernate from Tank 8 was completed in November 1992. Treatment of the supernate from Tank 7 was completed in October 1993. Treatment of the supernate in Tanks 1 through Tank 6 was completed in June 1994. The sludge from Tanks 1-8 and spent filter aid from Tank 10 were mixed together prior to final stabilization (Figure 1).

Comparisons of the waste volume reduction achieved by vitrification compared to alternative stabilization strategies in cement (Table I and Figure 1) shows that vitrification of M-Area composite waste (Option 3) reduces the stabilized volume by 96% relative to the base case cement stabilization. Volume reductions on the order of ~86% can be achieved by mixing portland cement with the homogenized waste, followed by pressure filtration and rinsing. This low volume cementation process has been designated FIST for FIltration and STabilization (Option 2).[7,8]

BACKGROUND

The Liquid Effluent Treatment Facility (LETF) was constructed in 1985 to process M-Area chemical wastes due to the federally mandated closure of the M-Area seepage basin. The LETF consisted of three facilities: the Dilute Effluent Treatment Facility (DETF), the Chemical Transfer Facility (CTF), and the Interim Treatment/Storage Facility (IT/SF). The DETF processed dilute wastewaters from the M-Area processes and released the clarified liquid to Tim's Branch.[9] The DETF was a conventional water treatment facility which used precipitation, flocculation, and filtration to remove dissolved metals. These solids were transferred through the Chemical Transfer Facility (CTF) to the IT/SF.

In May 1990 South Carolina Department of Health and Environmental Control (SCDHEC) approved a permit to operate the DETF-IST Supernate Transfer Facility.[9] The permit allowed the DETF to be used to treat the supernate from the M-Area Interim Treatment/Storage Facility (IT/SF) tanks. When the DETF-IST Supernate Transfer Facility concept for treatment of M-Area's supernate and subsequent discharge to Tim's branch was initiated, M-Area's waste solidification strategy was altered from solidification of sludge plus supernate to solidification of only the sludge and spent filter aid. Supernate treatment was completed in 1994. The current tank inventory of sludge in gallons is shown in Table II. Note that minor residual supernate volumes are associated with the sludge residues in each tank (Table II).
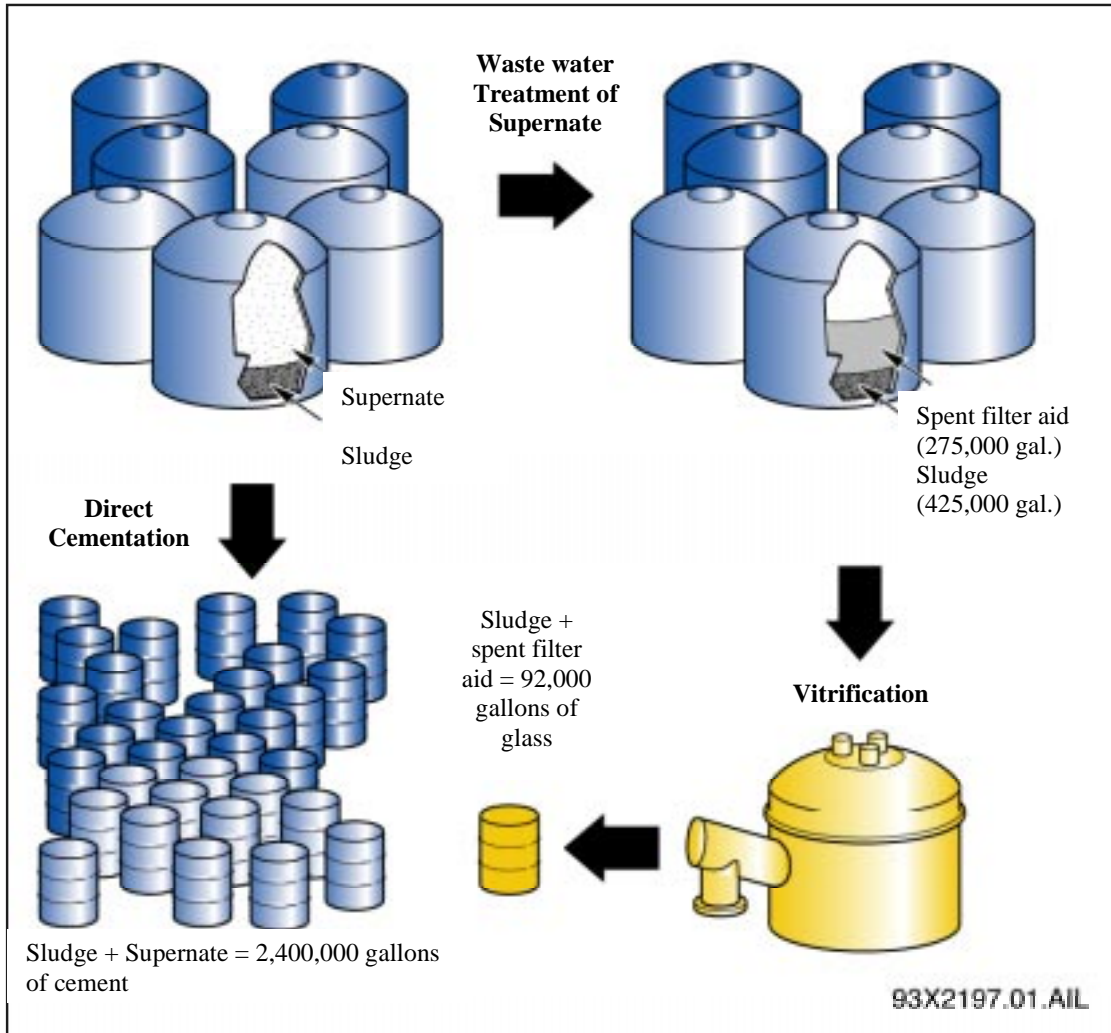
Figure 1. Schematic of the M-Area mixed waste solidification process. Large volume reductions can be achieved by combining wastewater treatment with vitrification instead of stabilization of sludge plus supernate in cement, e.g. 92,000 gallons of glass equals ~1673 drums (55-gallon size) while 2,400,000 gallons of cement makes 43,636 drums. This is a ratio of 1 drum of glass to every 26 drums of cement.

Table I. Comparative Volume Reductions for M-Area Waste Stabilization.

Stabilization Options	Volume Reduction*
Base Case (CEMENT WITH SUPERNATE) 1,200,000 gallons of M-Area supernate + sludge = 2,400,000 gallons cement	0%
Option 1 (CEMENT WITHOUT SUPERNATE) 700,000 gallons sludge + spent filter aid + cement = 1,400,000 gallons cement	42%
Option 2 (LOW VOLUME CEMENT USING FIST) 700,000 gallons wastewater treated sludge/spent filter aid + cement = 325,000 gallons cement	86%
Option 3 (VITRIFICATION) 700,000 gallons wastewater treated sludge + spent filter aid = 92,000 gallons glass	96%

* Relative to original 2,400,000 gallons grout projected if the sludge is not wastewater treated

The M-Area sludge and spent filter aid waste was solidified by vitrification in a Vendor Treatment Facility (VTF) in M-Area in 1996-1999.[10] A delisting petition for the vitrified waste has been submitted to EPA and is still under consideration. This would allow the vitrified waste to be disposed of in the SRS burial ground. If the vitrified waste is not delisted then the waste form will remain a RCRA listed waste and disposal in a permitted RCRA mixed waste repository will be necessary.

Samples of M-Area sludge and supernate were obtained in September 1986 from Tank 6 and 8 were being agitated at that time. Sludge samples from Tanks 1 through 6, and Tank 8 were obtained in January of 1988 after the agitatiors had been shut off for ~2 years. Analyses of these tank samples were previously documented.[11] Sludge samples from Tank 7 and filter aid samples from Tank 10 were obtained in March of 1992. Additional filter aid from Tank 10 was obtained in November of 1992. The sludge, supernate, and filter aid characterization documented in this study was completed so that durable and processable glass compositions could be formulated[12] and a waste treatability study[13] performed at SRTC to support the vitrification in a Vendor Treatment Facility (VTF).

Table II. M-Area Tank Inventories as of April, 1994

IT/SF Tank #	Sludge Volume[tt] (Gallons)	Supernate* Volume[tt] (Gallons)	Total Volume[tt] (Gallons)
1	4,780	60	4,840
2	4,380	0	4,380
3	9,860	1,570	11,430
4	16,600	940	17,540
5	6,350	850	7,200
6	4,630	210	4,840
7	150,550	34,750	185,300
8	160,750	~35,000	~195,000
10[†]	~270,100	~20,000	~290,000
TOTAL	~625,000	~95,000	~720,000

*   residual supernate that cannot be removed from the sludge

†   spent filter aid, 220,100 gallons have been generated to date and an additional ~50,000 are to   be generated

††   Volumes measured December 31, 1993, Specification No., X-SPP-M-00001, Attachment 9.1, Revision 4
      Statement of Work for AA07211N (May 31, 1994)

EXPERIMENTAL

Sludge-Supernate Analyses

Samples of agitated sludge-supernate from IT/SF Tanks 6 and 8 were received in 1986. The samples were agitated and immediately dissolved in HNO_3. This cold acid dissolution was carried out at a pH of 0.45 to insure that any solids component was dissolved and analyzed. The acidified samples were analyzed by inductively coupled plasma (ICP) spectroscopy for metal cations and by Davis-Gray titration with potassium dichromate for uranium. The preferred analytic technique for uranium concentrations greater than 1 g/L is the Davis-Gray titration. For samples with <0.1 g/L, a laser induced emission spectroscopy technique such as Laser Syntrex or Chemcheck is the preferred technique. For concentrations between 0.1 and 1 g/L both techniques have inherent difficulties. In solutions low in redox containing elements, the Davis-Gray titration can be used at uranium concentrations <1 g/L.

Alkali concentrations in the sludge-supernate were analyzed by ICP and atomic adsorption (AA) spectroscopy. Anions, such as PO_4, which are only partially soluble in water, were measured as phosphorous (P) by ICP analysis on the acidified sludge-supernate samples. In addition phosphorous was measured in the unacidified supernate-slurry as water soluble phosphate (PO_4) by Ion Chromatography (IC). The remaining anions were assumed to be water soluble and were also analyzed by IC on unacidified supernate. Chloride and fluoride were measured by Ion Selective Electrode (ISE) due to interference from the water peak during analysis of Cl by IC. The CO_3 of the samples was determined by a precipitation methodology followed by dissolution in 0.1M HCl, and titration with 0.1M NaOH. The specific gravity of the sludge-supernate and the pH of the supernate were also determined.

Sludge Analyses (Tanks 1-6 and Tank 8)

Sludge samples from IT/SF Tanks 1-6 and Tank 8 were obtained in January l988. The supernate was decanted off each sample. The wt% solids were obtained in duplicate from samples dried at 105°C. Additional samples were dried overnight at 90°C to obtain x-ray diffraction (XRD) analyses of the crystalline species. Drying at 90°C evaporates adsorbed water but does not evaporate water of crystallization of hydrated species. In cases where precursor, poorly crystallized phases were observed in the XRD spectra, the samples were sequentially heated at 300°C and 600°C to crystallize the poorly formed phase(s) and aid in phase identification and mass balance calculations.

The sludge was dried at ~300°C to remove both residual hydroxide species and waters of hydration. Dried sludge compositions were determined in duplicate in March, 1988 and in duplicate in November 1988 because the Ni and K analyses had been omitted from the March analyses.   Moreover, the HCl/HF dissolution had been used to determine Si by AA in March and this number was biased higher than the ICP analysis for Si. This bias was attributed to the HF dissolving Si from the glass bead in the nebulizer of the AA. Silica was redetermined by AA from the Na_2O_2 dissolution in November l988.

Different ICP instruments were used for the March l988 and November l988 analyses. The detection limits for each of these instruments is listed in Table III . The major elements (K, Na, Ca, Mg, Si, Al, and Fe) are accurate to within ±10% at concentrations >1 ppm. Within a factor of two of the detection limits listed (Table III), the precision of any element is only ±100%.

The dried sludge was completely analyzed by the following techniques:

- Dissolution by Na_2O_2 with an HCl uptake
  - ICP for Al, Ca, Fe, Mg, Mn, Li, Si, Cr, B, Sr, Ti, P, Ba, Pb, Mo, Zn, Cu (March and November l988)
  - AA for Si (November 1988)

- Dissolution by HCl/HF bomb
  - ICP for Na, Ni, Zr (November l988)
  - AA for Ni due to Ni interferences on ICP in the presence of U (November, 1988)
  - AA for Na (March and November 1988)
  - AA for K (November l988)
  - AA for Si (March 1988)

- Uranium by Davis-Gray titration (March 1988)

- Dissolution by Na_2O_2 with a H_2O uptake
  - IC for SO_4, NO_3, PO_4 (March 1988)
  - ISE for Cl and F (March 1988)

Sludge Crust (Tank 8)

A layer of white crystalline material was found to have formed on the surface of the sludge in Tank 8. This layer separated the sludge from the supernate and was thought to have precipitated from the supernate. The same crust was identified in most of the M-Area tanks but only one sample was analyzed. The crust varied from 9.51 to 12.7 mm (3/8 to ½ inch). A small slab of crust from Tank 8 was received for analysis in early 1989. The sample was chemically analyzed in triplicate using the same techniques as used for the sludge analyses for Tank 8. The sample phases were also identified by XRD and the sample was examined by Scanning Electron Microscopy (SEM).

Sludge and Spent Filter Aid Analyses (Tank 7 and Tank 10)

Sludge samples from IT/SF Tank 7 and Tank 10 were obtained in March 1992. A second larger sample of Tank 10 was obtained in November 1992 that was more representative of the contents of Tank 10.   For each tank the wt% solids were obtained in duplicate from samples dried at 105°C. Tank 7 and 10 samples were dried overnight at 90°C to obtain x-ray diffraction (XRD) analyses of the crystalline species without driving off any hydroxide species. In cases where broad poorly crystallized diffraction peaks were observed in the XRD spectra, the samples were heat treated at 600°C to crystallize the poorly formed phase(s) and aid in phase identification.

The chemical analyses of the sludge compositions were determined in duplicate. The sludge was dried at ~300°C to remove residual hydroxide species and completely analyzed for the remaining cations and ions by the following techniques:

- Dissolution by Na_2O_2 with an HCl uptake
  - ICP for Al, Ca, Fe, Mg, Mn, Li, Si, Cr, B, Sr, Ti, P, Ba, Pb, Mo, Zn, Cu
  - AA for Si

- Dissolution by HCl/HF bomb
  - ICP for Na, Ni, Zr
  - AA for Ni, Na, K, Si

- Uranium by ChemCheck

- Dissolution by Na_2O_2 with a H_2O uptake
  - IC for SO_4, NO_3, PO_4
  - ISE for Cl and F

Virgin Filter Aid Analyses

Perflo, perlite, and diatomaceous earth are high silica containing additives used in M-Area processing. Samples of these additives, which are oxide based, were analyzed only for cations in November 1988. Additional perlite and perflo were analyzed in February, 1993. The following techniques were used:

- H_2O content by drying at 600°C overnight

- Dissolution by Na_2O_2 with an HCl uptake
  - ICP for Al, Ca, Fe, Mg, Mn, Li, Si, Cr, B, Sr, Ti, P, Ba, Pb, Mo, Zn, Cu

- Dissolution by HCl/HF bomb
  - ICP for Na, Ni, Zr
  - AA for Ni, Na, K

XRD analyses were also performed on perflo and diatomaceous earth both before and after chemical treatment with NaOH in order to determine if chemical alteration of these additives was occurring due to the high pH environment in the IT/SF storage tanks. The filter aids were analyzed by XRD before chemical treatment. The filter aids were then treated with 6M NaOH solution for 29 hours, filtered, dried at 80°C overnight, dried an additional 1.5 hours at 200°C, and the XRD analysis repeated.

QUALITY ASSURANCE

All analyses were conducted at Savannah River Technology Center (SRTC) in accordance with DOE/RW-0214 and ASME NQA-1 based quality assurance program in use at the time the work was performed. The analytic results are recorded in laboratory notebooks DPSTN-4475, DPSTN-4725, and DPSTN-4695. The data and calculations from this report were used in the Vendor Design Specification Number X-SPP-M-0001, Revision 5. For this reason all of the calculations were verified by Glenn Diener and documented in calculation package Q-CLC-M-94012 on May 31, 1994 and accepted by John B. Pickett on June 11, 1994.

Table III. Detection Limits of Analytic Instrumentation

Element	December 1986- October 1988 ppm (Ref. 14)	November 1988- present ppm (Ref. 15)	Technique
Al	0.09	0.025	ICP
Ca	0.03	0.0001	ICP
Cu	0.02	0.003	ICP
Fe	0.01	0.004	ICP
K	0.005	0.005	AA
Mg	0.003	0.0001	ICP
Na	0.06	0.025	ICP
P	0.04	0.03	ICP
Pb	0.07	0.05	ICP
Ru	0.02	0.025	ICP
Si	0.03	0.02	ICP
Ti	0.002	0.003	ICP
Zr	0.002	0.004	ICP

RESULTS AND DISCUSSION

Chemical Analysis of the Sludge-Supernate

Samples of agitated sludge-supernate slurry from IT/SF Tanks 6 and 8 were dissolved in HNO_3 as described previously and analyzed. The pH of the slurry varied from 11.90-12.25 (Table IV). The specific gravity was ~1.10 g/mL while the wt% solids varied from 10.22 to 11.05. The sludge-supernate slurry contains high concentrations of sodium, nitrate, phosphate and aluminum (Table IV).

The cation analyses by ICP are given in Table IV. Sodium analyzed by ICP and AA gave comparable results (Table IV). Uranium analyses were determined by Davis-Gray titration even though the concentrations were <1 g/L because significant quantities of other redox species were not present in the slurry (Table IV). The uranium concentrations were low enough not to cause spectral interferences. This allowed Ni, Pb, and Zn to be determined by ICP analysis.

Acid soluble PO_4, was analyzed as P by ICP on the acidified sludge-supernate samples. Water soluble PO_4 was measured by IC and is lower than the acid soluble PO_4 values listed in Table IV. This implies that water soluble PO_4 is a major constituent of the supernate and that a PO_4 containing solid is a major constituent of the sludge portion of the slurry. The remaining water soluble anions were analyzed by IC while the CO_3 of the samples was analyzed by a precipitation-titration technique.

From the analyses in Table IV, the major elements in the slurry are Na, Al, NO_3, and PO_4. At pH values >11 the stable Al species is AlO_2^- and at pH values >12 the major P species is PO_4.[16] The major aqueous species in the supernate-sludge at pH values of ~12 are $NaNO_3$, $NaAlO_2$, and Na_3PO_4. A molar anion-cation mass balance based on this speciation is shown at the bottom of Table IV. The mass balance shows excellent agreement and consistency in the analyses performed.

Chemical Analysis of Sludge and Spent Filter Aids

The chemical analysis of the M-Area sludge (Tanks 1 through 6 and 8) was initially determined in duplicate in March 1988 and a second set of duplicate analyses performed in November 1988. Chemical analysis of sludge from Tank 7 and spent filter aid from Tank 10 was performed in May 1992. The Tank 10 chemical analysis was repeated on a more representative sample in July 1993. The data is summarized in Table V and the replicate analyses for each tank are given in Appendix I. Different ICP units were used for the March l988 and November l988 analyses and this accounts for some of the variation in detection limits and some of the large standard deviations (Appendix I).

Table IV. **Analyses of M-Area Waste Slurry (Sludge + Supernate)**

Constituent (Analysis Method)	IT/SF-Tank 6 (gm/L)	IT/SF Tank 8 Replicate A (gm/L)	IT/SF Tank 8 Replicate B (gm/L)
CATIONS AND ANIONS			
Uranium (D-G titration)	0.596	0.090	0.070
Sodium (ICP)	36.327	36.054	34.616
Sodium (AA)	31.253	37.228	36.308
Aluminum (ICP)	3.57	3.28	2.23
Calcium (ICP)	0.045	0.011	0.010
Iron (ICP)	0.048	0.005	0.006
Magnesium (ICP)	0.004	0.0004	0.0004
Nickel (ICP)	0.193	0.056	0.066
Silicon (ICP)	0.287	0.014	0.010
Potassium (AA)	0.035	0.020	0.020
Chromium (ICP)	0.009	0.004	0.004
Zirconium (ICP)	0.005	0.003	0.003
Titanium (ICP)	0.004	0.002	0.002
Lead (ICP)	0.14	0.061	0.07
Phosphorous (ICP)	3.02	2.19	2.07
Phosphorous (IC)	2.06	1.64	1.25
Nitrate (IC)	59.13	76.32	75.95
Chloride (ISE)	0.22	0.22	0.30
Sulfate (IC)	0.21	0.21	0.72
Carbonate (titration)	5.37	3.24	3.36
PROPERTIES			
pH	12.25	11.92	11.90
Sp Gravity (g/mL)	1.08	1.09	1.12
% Solids	10.35	10.22	11.05
CATION/ANION MASS BALANCE (moles/L)			
$\sum Na^+$	1.6	1.56	1.50
$\sum NO_3^=, PO_4^{-3}, AlO_2^-$	1.37	1.56	1.55

All wt% sludge solids were measured at 105°C. All the sludge samples for analysis were dried at 300°C prior to dissolution. Sodium was analyzed by AA in all the HCl/HF dissolutions and by ICP only in the HCl/HF dissolutions performed after November 1988. The agreement between the various analyses for Na is excellent (Table V and Appendix I). Nickel was determined by both ICP and AA in all analyses after November l988. Nickel by ICP is difficult due to interferences in the presence of uranium and AA is the preferred technique for analysis of Ni in the presence of uranium. To ensure that Ni determined by AA did not exhibit an enhancement in the presence of uranium (Table V and Appendix I), the uranium was precipitated out of one series of samples and the Ni analyses repeated. The Ni analyses determined by AA in the presence and absence of uranium were the same. Zinc and lead analyses by ICP can also be affected by the presence of uranium but no alternate technique is available for their determination.

Silicon was analyzed by ICP and by AA in all the analyses. In the March 1988 analyses the silicon concentration had been measured from the HCl/HF dissolution. This number was biased higher than the ICP analyses for Si or the AA analyses for Si from the Na_2O_2 dissolution (analyses after November, l988). This bias was attributed to the HF dissolving Si from the glass bead in the nebulizer of the AA. The results for the average silicon concentration determined by AA is therefore, only the average of the duplicate November analyses. Correspondingly, the AA results for Si in Appendix I are shown as italics and are not included in the average dry wt% sludge analyses. Although ICP is the preferred technique for analysis of Si, the ICP results for Si have higher standard deviations (Appendix I) than the AA results. The November 1988 AA results for Si are considered more accurate and used preferentially in this study.

Phosphorous was measured as P by ICP for sludges dissolved in Na_2O_2 with an HCl uptake. Phosphorous was measured as PO_4 by ICP for sludges dissolved in Na_2O_2 with an H_2O uptake. The observed differences in wt % P shown in Table IV and Appendix I is attributed to differences in the acid soluble and water soluble concentrations of P. Note that the acid soluble P is always greater than the water soluble P.

Tank 10 spent filter aid wt% solids were determined at 105°C. Additional sample was dried at 300°C and the same dissolution procedures that were used on the dried sludge were used on the dried spent filter aid. Both the May 1992 and the July 1993 analyses are given in Table V. The details are given in Appendix I.

Uranium was analyzed by David-Gray titration for the sludges in Tanks 1-6 and 8. Uranium was analyzed by laser emission spectroscopy (Chemcheck) for Tanks 7 and 10 (Table V).

Chemical Analysis of Sludge Crust

The sludge crust from Tank 8 was analyzed in triplicate using the same procedures used for the sludge analyses from Tank 8. The results are reported in Table VI which shows that the crust is primarily Al enriched. Note that U was not analyzed by dissolution and ICP but U was identified as a component of the crust by the Energy Dispersive Spectroscopy (EDS) during the Scanning Electron Microscopy (SEM) analysis. The U was primarily attached to the sludge side of the crust. X-ray analysis indicated that $Al(OH)_3$ was the primary crystalline phase and this is

confirmed by the mass balance calculations in Table VI. The mass balance calculations do not sum to 100±5 wt% since all of the elements and the amount of moisture in the sample were not determined before chemical analysis.

Chemical Analysis of Virgin Filter Aid

The virgin filter aids used in wastewater treatment of the M-Area supernate were primarily perflo and diatomaceous earth. These two filter aids were chemically analyzed in March, 1988. Two other filter aids used subsequently, perlite 2000X and Renissance perlite (200 SP), were analyzed in February 1993. The diatomaceous earth and perflo filter aids were dried at 600°C overnight to determine their water content. The higher surface area perlites were more problematic, e.g. a constant weight for dissolution and subsequent chemical analysis was difficult to obtain. Hence, the perlites were fused at 1150°C for 4 hours before chemical analysis. Therefore, the perlite wt% moisture represents this higher temperature. The same dissolution procedures that were used on the M-Area sludge were used on the perflo, perlite and diatomaceous earth. Anions were not analyzed because these materials are oxide based and oxygen is the only anion present in these solids. A good analysis of an oxide bearing material should sum to 100±5 wt% on an oxide basis.[17]

Table VII compares the analyses of perflo, diatomaceous earth, and perlite in terms of oxide wt%. The elemental analyses and the conversions to oxide wt% are given in Appendix II. The oxide wt% sums in **Table VII** are within 100±5 wt% for all of these species. Since these species do not contain uranium and have not been in contact with M-Area wastes, the presence of Zn, Pb, and Ni is considered significant because some of the trace elements observed in the sludge analyses are originating from the processing additives. The diatomaceous earth was the least pure of all the filter aids used in processing.

IDENTIFICATION OF PHASE CONSTITUENTS IN M-AREA SLUDGE AND CRUST

X-ray diffraction was performed on sludge samples that had been dried at 90°C for 12 hours. The crystalline sludge species in the eight tanks included varying amounts of $NaNO_3$, $Al(OH)_3$ (both gibbsite and bayerite polymorphs), $Na_{12}Al_{12}Si_{12}O_{48} \bullet 27H_2O$ or the Ca substituted form $Ca_6Al_{12}Si_{12}O_{48} \bullet 30H_2O$ (Linde Zeolite-A), and poorly crystallized $Na_6U_7O_{24}$ (partially converted in some tanks to $Na_2U_2O_7$), and $AlPO_4$. The Tank 10 spent filter aid was primarily amorphous but contained $NaNO_3$, Zeolite-A, and a trace of $AlPO_4$. The relative amounts of each phases in each tank was estimated by the relative peak heights of each species in the x-ray diffraction pattern. The results of the peak height analysis are given in

Table VIII. Four designations are used, major for species present in the greatest quantity, trace for species present in the smallest quantities, present for species present in average concentration, and none for species not identified by XRD.

The identification of the sodium uranate species was difficult because it was poorly crystallized, e.g. the x-ray diffraction reflections for this phase were very broad instead of being sharply defined (see Figure 2). Positive identification of the poorly crystallized uranium phase was made by sequential heat treatment to higher and higher temperatures. After each heat treatment the dried sludge was analyzed by XRD again. Sludge from Tank 1 was heat treated at 300°C for 3 hours. The XRD indicated that the uranium phase was still poorly crystallized (
), e.g. the uranate diffraction peaks are still broad and poorly defined. A subsequent heat treatment at 600°C overnight was performed which crystallized the $Na_6U_7O_{24}$ to $Na_2U_2O_7$ (
). This transformation has been reported previously in the literature[18] and was used to confirm the presence of the $Na_2U_2O_7$ precursor phase, $Na_6U_7O_{24}$. Similar results were obtained on Tank 7 samples heat treated to 600°C overnight. Note that the $NaNO_3$ "disappears" from the higher temperature spectra shown in Figure 3 as the nitrate in the $NaNO_3$ vaporizes at these temperatures.

Table V. Summary of M-Area Sludge Analyses and Mass Balance Calculations

Element (dry wt%)	Tank 1 (11/88)	Tank 2 (11/88)	Tank 3 (11/88)	Tank 4 (11/88)	Tank 5 (11/88)
CATIONS AND ANIONS					
Al (ICP)	11.145	7.130	6.383	3.043	9.778
Ca (ICP)	0.325	0.310	0.304	0.205	0.327
Fe (ICP)	0.495	0.260	0.655	0.215	0.495
Mg (ICP)	0.154	0.076	0.069	0.070	0.137
Mn (ICP)	0.539	1.315	1.025	0.734	0.392
Na (ICP)	8.830	12.430	15.425	21.555	10.515
Na (AA)	8.960	14.363	16.713	25.435	10.718
Li (ICP)	<0.002	<0.002	<0.002	<0.002	<0.002
Ni (ICP	0.695	0.390	0.275	0.055	0.555
Ni (AA)	0.876	0.556	0.385	0.082	0.758
Si (ICP)	9.613	2.408	3.675	1.631	10.790
Si (AA*)	16.140	3.140	4.105	3.323	14.030
Cr (ICP)	0.015	0.013	0.010	0.015	0.015
B (ICP)	0.060	0.075	0.025	0.015	0.015
U (D-G)	5.246	4.786	3.229	1.281	3.858
Sr (ICP)	0.004	0.004	0.005	0.002	0.003
Zr (ICP)	0.032	0.010	0.010	0.080	0.010
Ti (ICP)	0.040	0.014	0.015	0.017	0.033
K (AA*)	0.122	0.033	0.054	0.021	0.104
P (ICP)***	1.548	1.465	1.953	1.298	1.270
P (Calc IC)**	0.774	0.975	1.291	0.376	<0.848
Ba (ICP)	0.008	0.017	0.030	0.032	0.016
Pb (ICP)	0.161	0.213	0.157	0.136	0.124
Mo (ICP)	0.011	0.010	0.010	0.010	0.010
Zn (ICP)	0.960	2.155	1.720	1.294	0.945
Cu (ICP)	0.018	0.016	0.014	0.020	0.009
F (ISE)	<0.02	<0.265	<0.02	<0.02	<0.02
Cl (ISE)	0.113	0.189	0.142	0.220	0.843
SO_4 (IC)	<0.50	<0.50	<0.50	<0.50	<1.31
NO_3 (IC)	14.275	34.660	50.580	59.500	12.000
PO_4 (IC)**	2.989	2.375	3.960	1.152	<2.6
PROPERTIES					
% SOLIDS (105°C)	35.37	38.98	9.24	22.34	32.49
MOLAR MASS BALANCE					
$Al(OH)_3$	27.06	15.72	12.35	4.73	23.80
$NaNO_3$	19.57	47.51	69.33	81.56	16.45
Filter Aid	36.31	5.79	8.52	7.03	20.04
$Na_6U_7O_{24}$	6.89	6.29	4.24	1.68	5.07
Ca Zeolite-A	2.99	2.85	2.79	1.88	3.00
$AlPO_4$	4.75	4.49	5.99	3.98	3.89
SUM	97.55	82.64	103.22	101.86	81.25

*AA Silicon from Na_2O_2 dissolution; all anions from Na_2O_2 fusion with H_2O uptake (see text)
water soluble phosphate only, * acid soluble phosphate, N/A = Not Analyzed

Table V. Summary of M-Area Sludge Analyses and Mass Balance Calculations

Element (dry wt%)	Tank 6 (11/88)	Tank 7 (5/92)	Tank 8 Average (11/88)	Tank 10 (5/92)	Tank 10 (7/93)
CATIONS AND ANIONS					
Al (ICP)	10.013	6.392	10.734	8.896	9.642
Ca (ICP)	0.497	0.420	0.307	0.351	0.425
Fe (ICP)	0.740	0.529	0.402	0.870	1.046
Mg (ICP)	0.188	0.125	0.122	0.122	0.122
Mn (ICP)	1.535	0.005	0.422	0.035	0.049
Na (ICP)	9.670	12.292	7.010	7.196	5.132
Na (AA)	10.228	13.430	7.056	7.433	5.233
Li (ICP)	<0.002	<0.001	<0.002	<0.001	<0.001
Ni (ICP	0.425	1.871	0.728	0.064	0.081
Ni (AA)	0.601	1.985	0.990	<0.06	<0.06
Si (ICP)	12.310	15.869	11.301	24.785	25.144
Si (AA*)	18.000	17.985	14.715	27.445	24.845
Cr (ICP)	0.020	<0.004	0.017	<0.004	<0.004
B (ICP)	0.010	0.037	0.010	<0.004	<0.004
U (D-G, chemcheck†)	4.080	3.005†	5.133	0.045†	0.222†
Sr (ICP)	0.006	N/A	0.006	N/A	0.002
Zr (ICP)	0.010	<0.002	0.010	<0.002	<0.002
Ti (ICP)	0.038	0.024	0.029	0.027	0.062
K (AA*)	0.107	0.580	0.121	2.605	2.053
P (ICP)***	1.135	1.137	1.896	1.031	0.959
P (Calc IC)**	0.244	1.963	0.973	1.035	0.997
Ba (ICP)	0.047	N/A	0.053	N/A	0.016
Pb (ICP)	0.245	0.156	0.137	0.035	0.032
Mo (ICP)	0.015	<0.004	0.009	<0.004	0.005
Zn (ICP)	2.875	0.176	0.794	0.172	0.464
Cu (ICP)	0.026	0.086	0.022	0.003	0.065
F (ISE)	<0.02	0.050	N/A	0.060	1.189
Cl (ISE)	0.060	0.675	0.059	0.310	0.134
SO_4 (IC)	<0.5	0.237	<0.5	<0.2	0.332
NO_3 (IC)	12.870	15.135	16.376	7.935	6.202
PO_4 (IC)**	0.749	6.019	2.984	3.175	3.057
PROPERTIES					
% SOLIDS (105°C)	37.12	30.85	38.23	17.56	19.97
MOLAR MASS BALANCE					
$Al(OH)_3$	24.15	13.98	25.06	23.12	23.81
$NaNO_3$	17.64	20.75	22.45	27.48	8.50
Filter Aid	40.05	40.27	33.07	52.95	53.62
$Na_6U_7O_{24}$	5.36	3.95	6.75	0.06	0.29
Ca Zeolite-A	4.57	3.86	2.82	1.30	3.91
$AlPO_4$	3.48	3.49	5.82	3.16	2.94
SUM	95.26	86.29	95.96	108.065	95.58

Table VI. Summary of M-Area Crust Analyses from Tank 8

Element (dry wt%)	Replicate #1 ADS#200027146	Replicate #2 ADS#200027147	Replicate #3 ADS#200027148	Average
CATIONS AND ANIONS				
Al (ICP)	27.553	23.568	24.330	25.150
Ca (ICP)	BDL	0.015	BDL	0.005
Fe (ICP)	0.223	0.048	0.056	0.109
Mg (ICP)	0.006	0.007	0.011	0.008
Mn (ICP)	0.004	BDL	0.006	0.003
Na (ICP)	1.490	1.640	1.200	1.443
Na (AA)	1.340	1.060	1.310	1.237
Li (ICP)	0.006	BDL	BDL	0.002
Ni (ICP	0.022	0.038	0.058	0.039
Ni (AA)	0.071	0.106	0.124	0.100
Si (ICP)	0.858	0.969	1.268	1.032
Si (AA*)	1.610	1.400	1.560	1.523
Cr (ICP)	BDL	BDL	BDL	BDL
B (ICP)	0.086	0.042	0.040	0.056
U (D-G)	N/A	N/A	N/A	N/A
Sr (ICP)	BDL	BDL	BDL	BDL
Zr (ICP)	BDL	BDL	BDL	BDL
Ti (ICP)	BDL	BDL	BDL	BDL
K (AA*)	0.004	<0.001	0.014	0.009
P (ICP)***	0.184	0.160	0.234	0.193
P (Calc IC)**	0.774	1.002	1.171	0.982
Ba (ICP)	BDL	BDL	BDL	BDL
Pb (ICP)	BDL	BDL	BDL	BDL
Mo (ICP)	0.009	BDL	0.007	BDL
Zn (ICP)	0.145	0.149	0.139	0.144
Cu (ICP)	BDL	BDL	0.004	BDL
F (ISE)	0.116	0.495	0.105	0.239
Cl (ISE)	1.520	0.977	0.413	0.970
SO_4 (IC)	N/A	N/A	N/A	N/A
NO_3 (IC)	3.716	6.062	8.496	6.091
PO_4 (IC)**	2.374	3.073	3.590	3.012
PROPERTIES				
% SOLIDS (105°C)	N/A	N/A	N/A	N/A
MOLAR MASS BALANCE				
$Al(OH)_3$	79.19	67.67	69.75	72.21
$NaNO_3$	5.09	8.31	11.65	8.35
Filter Aid	3.73	3.24	3.61	3.53
$Na_6U_7O_{24}$	N/A	N/A	N/A	N/A
Ca-zeolite	BDL	0.14	BDL	0.05
$AlPO_4$	0.56	0.49	0.72	0.59
SUM	88.578	79.85	85.73	84.72

N/A = Not Analyzed; BDL = Below Detection Limit

Table VII. Summary of M-Area Filter Aid Analyses

Oxide (dry wt%)	Diatomaceous Earth (DE) ‡	Super-Cel Perflo‡	Perlite* 2000X	Perlite* 200SP
Al_2O_3	16.08	12.36	12.48	12.93
CaO	0.91	0.92	0.70	0.61
Fe_2O_3	0.92	1.67	0.88	1.02
MgO	0.13	0.12	0.11	0.30
MnO_2	0.90	0.59	0.10	0.08
Na_2O	3.28	3.55	4.48	3.42
NiO	0.31	BDL	BDL	0.08
SiO_2	67.32	72.95	74.75	75.43
B_2O_3	BDL	BDL	BDL	BDL
ZrO_2	0.01	0.01	0.01	0.01
TiO_2	0.07	0.07	0.06	0.10
K_2O	4.46	4.82	5.42	5.05
P_2O_5	0.44	0.09	0.03	0.03
BaO	0.03	0.04	0.04	0.02
PbO	0.19	0.10	BDL	BDL
ZnO	1.12	0.67	0.19	0.19
CuO	0.03	0.03	BDL	BDL
SUM	96.20	97.99	99.24	99.28
MOISTURE CONTENT BEFORE DRYING/FUSING				
H_2O	7.37	0.20	0.04	0.13

*The high surface area of these filter aids made it difficult to get an accurate weight for the dissolution and subsequent chemical analysis. In these cases the filter aid was fused at 1150°C for 4 hours, cooled, broken out of the crucible it was fused in, and then weighed for dissolution and analysis
BDL = Below Detection Limit
‡ Dried at 600°C
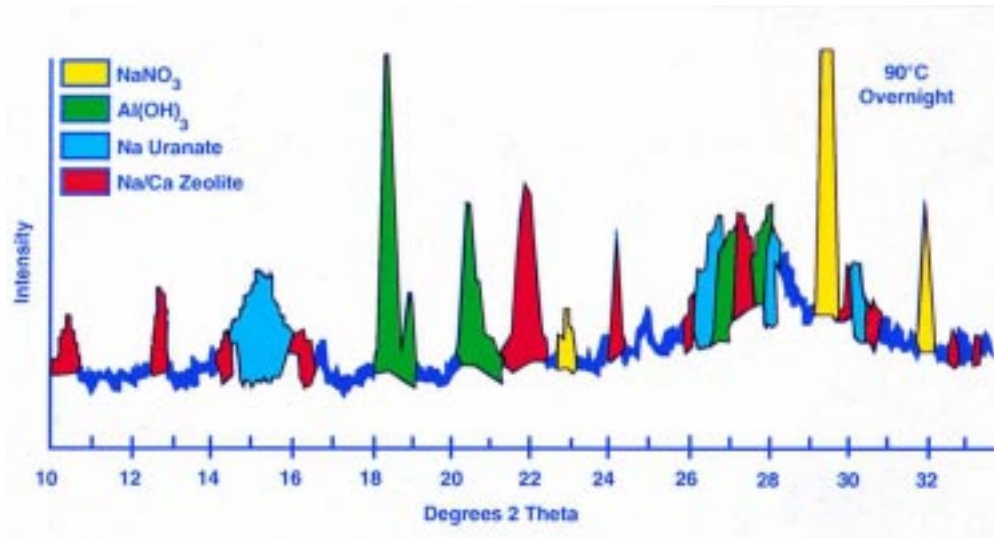
Figure 2. X-ray diffraction pattern of the M-Area sludge dried at 90º overnight. Note how broad the x-ray diffraction peaks are for the sodium uranate phase compared to the other crystalline species present.
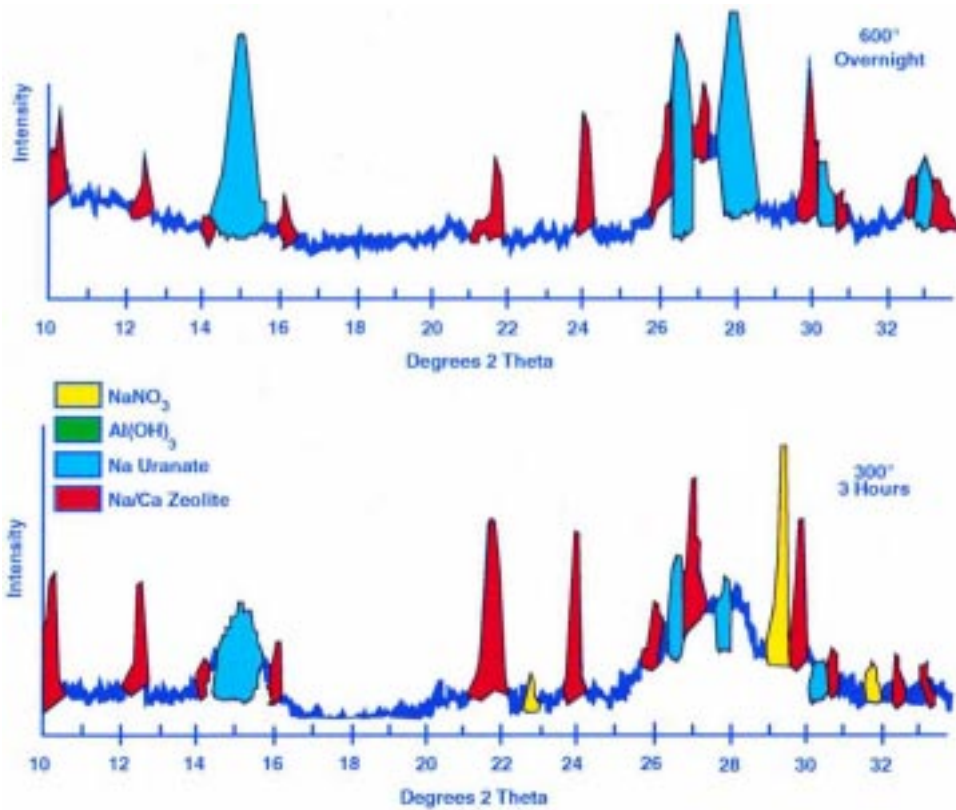
Figure 3. X-ray diffraction pattern of the M-Area sludge heat treated at 300ºC and 600ºC.

Table VIII. X-Ray Diffraction Phase Identification and Relative Concentrations of the Species in M-Area Sludge and Spent Filter Aid

Tank ID	NaNO$_3$[*]	Al(OH)$_3$[**]	Na$_6$U$_7$O$_{24}$ and/or Na$_2$U$_2$O$_7$[***]	Zeolite-A[****]	AlPO$_4$	pH
1	Major	Present	Major	Major	None	12.3[†]
2	Major	Trace	Major	None	None	12.9[†]
3	Major	None	Trace	None	Trace	10.5[†]
4	Major	None	Trace	None	None	9.6[†]
5	Major	Present	Major	Present	None	12.8[†]
6	Major	Major	Present	None	Trace	12.4[†] 12.3[††] 11.2[††††]
7	Major	Present	Present	Trace	Trace	11.9[†††]
8 (P42)[‡]	Major	Present	Present	Major	None	11.9[††]
8 (P43)[‡]	Major	Present	Present	Major	None	12.0[††] 11.9[†††]
10	Major	None	None	Trace	Trace	8 to 10[†††††]

‡   Tank 8 was sampled from two different riser locations.
*   JCPDS #7-271
**   JCPDS #29-41
***   JCPDS #20-1161 which is a deleted pattern which is closely related Na$_2$U$_2$O$_7$ (#26-973 or #26-974)
****   JCPDS #11-589
†   Enwright Laboratories, Greenville, SC, First Phase Analytical Report for 300-M Area Sampling Effort: AX 0720919 (October 29, 1987)
††   pH of sludge-supernate slurry (this study Table IV)
†††   Enwright Laboratories, Greenville, SC, Final Report for M-Area Sludge and Supernatant Analyses: AX 0720919 (February 3, 1988); measured on Ports 16 and 17 not 42 and 43
††††   WSRC 320-M Laboratory analysis
†††††   WSRC 320-M Laboratory analysis; pH has varied from 8 to 10 over a period of 4 years depending on chemistry of the tank additions

The Na$_6$U$_7$O$_{24}$ phase has previously reported to form when uranium is precipitated from uranyl nitrate solution when excess sodium hydroxide is added.[19] It is the primary uranate species which is stable in the presence of excess alkali.[19] Since the M-Area waste tanks normally stabilize at a pH of 11-12 (due to excess NaOH used during the aluminum dissolution process), the identification of this sodium uranate is consistent with the chemical environment of the IT/SF tanks, e.g. the tanks with the lowest pH (Tanks 3 and 4) have only trace amounts of Na$_6$U$_7$O$_{24}$ in the x-ray spectra (Table VIII). Conversely, the tanks with the highest U concentrations and

highest pH (Tanks 1,5,8) have the highest concentrations of $Na_6U_7O_{24}$.

In addition, the tanks (Tanks 3 and 4) with the lowest Al concentration have no $Al(OH)_3$ in the XRD spectra. Conversely, the tanks (Tanks 1, 5, 8) with the highest Al contain the most $Al(OH)_3$ in the XRD spectra. The tanks with the highest pH values which also have significant concentrations of $Al(OH)_3$ (Tanks 1 and 5) are the tanks containing the most Zeolite-A. The quantitative phase analysis of the tanks is, therefore, consistent with the individual tank chemistries and environments (Table VIII).

The primary crystalline phase of the M-Area crust was $NaNO_3$ and $Al(OH)_3$ (primarily the gibbsite polymorph) with trace amounts of $Na_2U_2O_7$ and Zeolite-A. This was confirmed by SEM-EDS analysis which showed Al to be the major component of the crust, followed by Si, U and Na. This is also in agreement with the chemical analyses given in Table VI.

IDENTIFICATION OF IN-TANK ZEOLITE FORMATION

The zeolite identified in the M-Area sludge was identified as either a calcium or a sodium containing Zeolite-A with the nominal formulas $Ca_6Al_{12}Si_{12}O_{48} \cdot 30H_2O$ and $Na_{12}Al_{12}Si_{12}O_{48} \cdot 27H_2O$. However, zeolite was not used in M-Area processing nor had any zeolite been added to the tanks. This finding indicated that the zeolite formed from reaction of various components in the tank. In order to confirm this hypothesis, samples of virgin perflo and diatomaceous earth were analyzed by x-ray diffraction and shown to be amorphous (see Figure 4). Samples of perflo and diatomaceous earth were treated with 6M NaOH solution at room temperature for 29 hours. The solution pH was 13.73. The solution was filtered off and the solids dried at 80°C for ~ 17 hours. The solids were still wet, and were dried for an additional 1.5 hours at 200°C. The dried solids were identified by x-ray analysis to be Na_2SiO_3 (JCPDS #16-818) and $Na_3Si_3Al_3O_{12} \cdot NaCl$ (sodalite - JCPDS #20-495), a zeolite formed from Zeolite-A by substitution of NaCl or other sodium species such as NaOH into the Zeolite-A lattice structure. In order to ensure that crystallization of the sodalite was due to the treatment with NaOH and not the drying, untreated filter aids were dried at 200°C for 1.5 hours. The untreated filter aids remained x-ray amorphous confirming that the Zeolite phases formed by reaction of the silica rich filter aids with NaOH.

In tank formation of Zeolite-A by chemically catalyzed crystallization of perflo and diamateceous earth due to the high caustic content and the high pH of the M-Area supernate is consistent with the known formation of zeolites. For example, Linde B_1 zeolite is fabricated via a patented process which treats aluminosilicate gel ("Alfloc") in concentrated NaOH at temperatures of 60-110°C.[20]

Additional evidence for in-tank, chemically catalyzed crystallization of Zeolite-A from the aluminosilicate rich portion of the sludge was achieved by dissolving M-Area sludge in 6M HNO_3 and analyzing the undissolved residue. The acid resistant residue in the M-Area sludge is pure SiO_2 (crystalline cristobolite and traces of quartz). Because cristobolite and quartz are crystalline species and acid resistant, they are the breakdown products of a crystalline silicate not of the amorphous perflo. This indicates that the cristobolite is the breakdown product of the Zeolite-A and not the original filter aid.
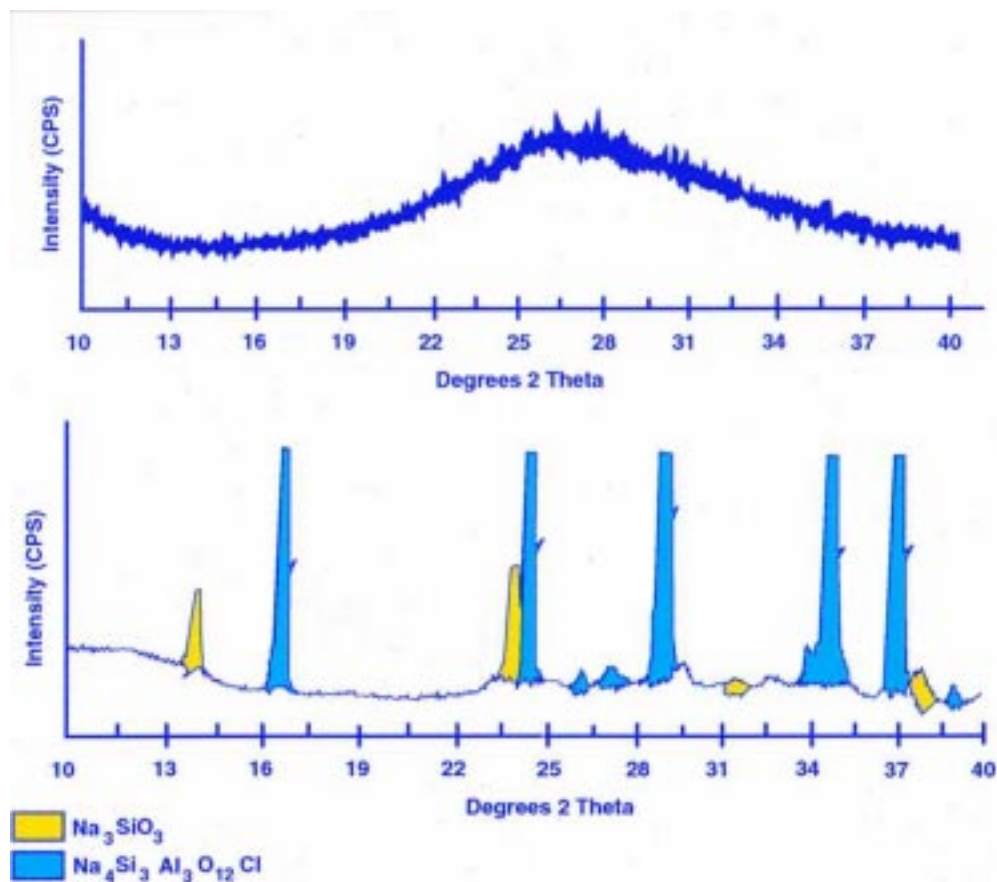
Figure 4. X-ray analysis of untreated (virgin) perflo and diatomaceous earth indicated that these materials were amorphous (top) while treatment with 6M NaOH for 29 hours at room temperature crystallized Na_2SiO_3 and sodalite (a mineral related in structure to Zeolite-A).

MASS BALANCE CALCULATIONS

The x-ray diffraction analysis indicated that the crystalline species present in the M-Area sludges are $NaNO_3$, $Na_6U_7O_{24}$, $Al(OH)_3$, $AlPO_4$, and Zeolite-A of the approximate formula $Ca_6Al_{12}Si_{12}O_{48} \cdot 30H_2O$ (with a molecular weight of 2210 grams) or $Na_{12}Al_{12}Si_{12}O_{48} \cdot 27H_2O$ (with a molecular weight of 2242 grams). The remainder of the sludge is amorphous perlite or perflo. A mass balance based on these 5 crystalline species and amorphous SiO_2 was calculated from the element wt% analyses given in Table V and Table VI for the M-Area sludges and crust, respectively.

Table IX shows a sample molar mass balance calculation for Tank-1 sludge. In the molar mass balance calculations all of the molar NO_3 was assumed to form $NaNO_3$. All of the molar U was assumed to form $Na_6U_7O_{24}$. All of the molar Ca was assumed to form Ca rich Zeolite-A. All of the acid soluble molar PO_4 determined by ICP was assumed to form $AlPO_4$. The molar Al concentration minus the molar amount of Al assigned to $AlPO_4$ minus the molar concentration of Al in the Zeolite-A was assumed to form $Al(OH)_3$. All of the Si determined by AA minus the amount of Si consumed in the Zeolite-A phase, was assigned to the filter aid as described below.

The average molecular formulas for the filter aid species were determined from the normalized oxide mole% analyses given in Appendix II. Based on the data in Appendix II the average molecular formulas for these filter aid species can be written as follows on an equal oxygen basis:

$$\text{perflo} = K_{0.08}Na_{0.08}Al_{0.165}Si_{0.83}O_2$$

$$\text{diatomaceous earth} = K_{0.08}Na_{0.08}Al_{0.23}Si_{0.79}O_2.$$
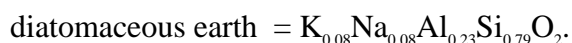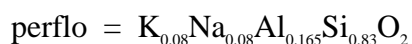
For perflo the average molecular weight is 64.53 and for diatomaceous earth the average molecular weight is 65.45. Since the relative amounts of each filter aid used in any one M-Area tank is unknown, an average molecular weight of 65 grams was used to calculate the contribution of the filter aids to the mass balance.

The resulting mass balance results are summarized in Table V and Table VI and Appendix I. The mass balance sums calculated from the chemical analyses in Table V sum to be between 95-105 mole% indicating that most of the chemical analyses are accurate. Deviations from mass balance of 100 ± 5 mole% appear to be due to incorrect speciation of Na. Excess Na over that assigned to $NaNO_3$ and $Na_6U_7O_{24}$ was calculated as moles/100grams (Appendix I). This probably should have been assigned to the Zeolite-A phase since this is a mixed Na-Ca Zeolite-A but the mass balance calculations assumed a pure Ca bearing Zeolite-A. The Na may also be partially tied up in minor phases such as NaCl and sodium aluminate phases which did not show up during x-ray diffraction analysis due to their low volume fraction. Interestingly, the tanks with excess Na in the mass balance (Tanks 1, 5 and 6) have relatively high pH values, while the tanks such as 3 and 4 which do not have excess Na have lower pH values.

Table IX. Example of Mass Balance Calculation for M-Area Sludge (Tank 1)

Row ID	Column A Element(wt%)		Column B Mass Balance Calculations		
3	Al (ICP)	11.145			
4	Ca (ICP)		0.325		
5	Fe (ICP)	0.495			
6	Mg (ICP)	0.154			
7	Mn (ICP)	0.539			
8	Na (ICP)	8.83			
9	Na (AA)	8.96	$Al(OH)_3$	=	((B3/26.98)-(B22/30.97))-((B4/40.08)*2)*78
10	Li (ICP)	<0.002	$NaNO_3$	=	B40*84.995
11	Ni (ICP)	0.695	**Filter Aid**	=	((B14/28.08-(B4/40.08)/6))*65
12	Ni (AA)	0.876	$Na_6U_7O_{24}$ =	=	((B17/238.029)/7)*2188.14
13	Si (ICP)	9.613	**Ca-Zeolite-A**	=	(((B4/40.08)/6))*(2210)
14	Si (AA*)	16.14	$AlPO_4$	=	(B22/30.97)*94.97
15	Cr (ICP)	0.015			
16	B (ICP)	0.06	**SUM**	=	SUM(B44:B49)
17	U (D-G)	5.246			
18	Sr (ICP)	0.004			
19	Zr (ICP)	0.032	**Excess moles**		
20	Ti (ICP)	0.0395	**of Na over**		
21	K (AA)	0.1215	$NaNO_3$ +	=	(B39-B40-B41)-
22	P (ICP)	1.5475	$Na_6U_7O_{24}$		(((B17/238.03)/7)*6)
23	P (Calc IC)	0.975			
24	Ba (ICP)	0.008			
25	Pb (ICP)	0.161			
26	Mo (ICP)	0.0105			
27	Zn (ICP)	0.9595			
28	Cu (ICP)	0.018			
29					
30	F (ISE)	<0.02			
31	Cl (ISE)	0.1125			
32	SO^4 (IC)	<0.50			
33	NO_3 (IC)	14.275			
34	PO_4(IC)**	2.9885			
35					
36	% SOLIDS (105°C)		35.37		
37	% SOLIDS (90°C)		34.43		
38					
39	Moles of Na		=B8/22.9898		
40	Moles of NO_3		=B33/62.0049		
41	Moles of Cl		=B31/35.453		

The mass balance summaries (Table V) agree with the concentrations of the phases predicted from the x-ray diffraction peak height analysis (Table VIII): Tanks 3 and 4 with low pH have less $Al(OH)_3$ and only trace amount of $Na_6U_7O_{24}$ while Tanks 1, 2, 5, 6, and 8 contain the most $Al(OH)_3$ and $Na_6U_7O_{24}$. All of the tanks have $NaNO_3$ as the major species. Tank 10 has the largest fraction of filter aid and the lowest concentrations of $Na_6U_7O_{24}$.

AVERAGE M-AREA WASTE COMPOSITION

M-Area sludge and spent filter aid was homogenized to form macrobatches of melter feed for the Vendor Treatment Facility (VTF). Using the measured tank volumes (Table II), the analyzed tank chemistry (Table V and Table VII), the analyzed wt% solids (Table V), and the measured specific gravity of the wastes (Table X), an element contribution per tank can be calculated as follows:

Dry element wt%/tank (Kgs) = Tank Volume(gal.) x (3.75 L/gal.) x Specific Gravity(Kg/L) x (wt% solids)(dry element wt%)

In a similar manner, the sludge-supernate composition of Tank 8 in Table IV can be used as an approximation of the supernate associated with the sludge in each tank (Table II and Table X), e.g. 95,000 gallons, the dry element wt% contribution from the associated supernate can be calculated. The dry element wt% can then be summed over all the tanks for the sludge and supernate contributions. This can then be converted from element wt% to oxide wt% (Table XI) to determine what glass forming additives must be added. Note that Table XI does not include ~3700 gallons of slightly enriched (1.1%) U^{235} Mark 15 filter cake waste and ~1200 gallons of excess Watts Plating Line solution which was blended with the tank waste for final stabilization in the M-Area Vendor Treatment Facility (VTF).

CONCLUSIONS

M-Area sludge, supernate, crust, and filter aids were characterized. The sludge plus supernate is composed primarily of soluble sodium nitrate, aluminate and phosphate species. The chemical analyses, XRD analyses, and mass balance calculations demonstrated that $NaNO_3$, $Na_6U_7O_{24}$, $Al(OH)_3$, $AlPO_4$, a Ca-Na bearing Zeolite-A, and amorphous filter aids used in M-Area processing are the main constituents. The $Al(OH)_3$ was the main constituent of the crust and the $NaNO_3$ was the main component of the sludge. A minimum of three dissolutions and up to five different analytic methodologies were used to achieve each sludge/spent filter aid analysis. Mass balance calculations were used to verify the accuracy and consistency of the multiple chemical analyses. Many of the trace elements in the sludge are originating from the filter aids added during processing. The Zeolite-A forms in-situ in the tanks due to reaction of the silica rich filter aids with the aluminate bearing wastes in a high caustic environment.

The high concentrations of glass-forming species in the M-Area waste, e.g. Na, Al, Si and P, indicate that high waste loadings can be achieved during vitrification of this waste compared to solidification in cement. In addition, nitrate (NO_3) species volatilize during vitrification, mitigating nitrate leaching concerns normally associated with cementitious waste forms.

Table X. Measured wt% Solids and Specific Gravity of M-Area Tank Wastes

TANK #	Sludge Volume (Gallons)	Wt% Solids at 105ºC†	Specific Gravity (Kg/L)
1	4,780	35	1.18*
2	4,380	39	1.35*
3	9,860	9	1.05*
4	16,600	22	1.15*
5	6,350	32	1.08*
6	4,630	37	1.23*
7	150,550	31	1.21**
8	160,750	38	1.24*
10	~270,100	17	1.10**
Associated Supernate	~95,000	10	1.1***

†measured in this study at 105ºC (see Table V)
*Enwright Laboratories, Greenville, SC, Final Report for M-Area Sludge and Supernate Analyses: AX 0720919 (February 3, 1988)
**Estimated
*** Measured in this study (see Table IV Tank 8 average as an approximation)

Table XI. M-Area Calculated Composite Sludge/Residual Supernate Composition.[†]

Oxide	Oxide Wt%	Species	Molar Mass Balance
Al_2O_3	20.27	$Al(OH)_3$	23.18
CaO	0.59	$NaNO_3$	17.88
Fe_2O_3	0.96	Filter Aid	47.06
MgO	0.24	$Na_6U_7O_{24}$	3.48
MnO	0.37	Ca Zeolite-A	3.15
Na_2O	17.74	$AlPO_4$	4.32
NiO	1.52	SUM	99.06
SiO_2	47.48		
Cr_2O_3	0.01		
B_2O_3	0.06		
UO_2	4.21		
TiO_2	0.05		
K_2O	1.28		
P_2O_5	4.19		
BaO	0.03		
PbO	0.15		
MoO_3	0.01		
ZnO	0.77		
CuO	0.05		
SO_4	0.07		
NO_3	19.10		
SUMS	102.97		
WT%	23.8		

† Calculated total inventory. Composition does not include ~3700 gallons of slightly enriched (1.1%) U^{235} Mark 15 filter cake waste and ~1200 gallons of excess Watts Plating Line solution were homogenized with the tank waste for final stabilization in the M-Area Vendor Treatment Facility (VTF).

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APPENDIX I M-AREA SLUDGE ANALYSES (ELEMENT WT%)

Tank 1	TK-1-1	TK-1-2	TK-1-1	TK-1-2	TANK 1
ANALYSIS #	111468	111469	21295	21296	AVERAGE
ANALYSIS DATE	3/1/88	3/1/88	11/1/88	11/1/88	
RECORDED	DPSTN 4475	DPSTN 4475	DPSTN 4475	DPSTN 4475	
Al (ICP)	11.300	11.000	10.850	11.430	11.145
Ca (ICP)	0.228	0.192	0.530	0.350	0.325
Fe (ICP)	<0.004	<0.004	0.500	0.490	0.495
Mg (ICP)	0.143	0.133	0.190	0.150	0.154
Mn (ICP)	0.238	0.317	1.100	0.500	0.539
Na (ICP)			8.810	8.850	8.830
Na (AA)	10.010	9.140	8.590	8.100	8.960
Li (ICP)	<0.002	<0.002	<0.002	<0.002	<0.002
Ni (ICP)			0.680	0.710	0.695
Ni (AA)			0.884	0.868	0.876
Si (ICP)	6.870	7.190	11.990	12.400	9.613
Si (AA*)	*19.760*	*21.960*	16.700	15.580	16.140
Cr (ICP)	0.009	0.010	0.020	0.020	0.015
B (ICP)	<0.002	<0.002	0.090	0.030	0.060
U (D-G)	5.180	5.311			5.246
Sr (ICP)	0.002	0.001	0.010	<0.001	0.004
Zr (ICP)	0.055	0.054	0.010	0.010	0.032
Ti (ICP)	0.038	0.040	0.040	0.040	0.040
K (AA*)			0.124	0.119	0.122
P (ICP)*	2.200	1.910	1.090	0.990	1.548
P (Calc IC)**	1.040	0.909			0.975
Ba (ICP)	0.008	0.007	<0.001	<0.001	0.008
Pb (ICP)	0.048	0.086	0.320	0.190	0.161
Mo (ICP)	0.012	0.010	0.010	0.010	0.011
Zn (ICP)	0.618	0.580	1.760	0.880	0.960
Cu (ICP)	0.013	0.009	0.020	0.030	0.018
F (ISE)	<0.02	<0.02			<0.02
Cl (ISE)	0.113	0.112			0.113
SO4 (IC)	<0.09	<0.50			<0.50
NO3 (IC)	13.280	15.270			14.275
PO4(IC)**	3.190	2.787			2.989
% SOLIDS (105°C)	35.370				35.370
% SOLIDS (90°C)	34.430				34.430
Moles of Na	0.435	0.398	0.374	0.352	0.390
Moles of NO3	0.214	0.246			0.230
Moles of Cl	0.003	0.003			0.003

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution
  all anions from Na2O2 fusion with H2O uptake (see text)

**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE

Al(OH)3	27.06
NaNO3	19.57
Filter Aid	36.31
Na6U7O24	6.89
Ca-Zeolite-A	2.99
AlPO4	4.75
SUM	97.55
EXCESS moles Na over NaNO3 and Na-U =	0.141

Tank 2	TK-2-1	TK-2-2	TK-2-1	TK-2-2	TANK 2 AVERAGE	TANK 2 STANDARD DEVIATION
ANALYSIS #	111470	111471	21297	21298		
ANALYSIS DATE	3/1/88	3/1/88	11/1/88	11/1/88		
RECORDED	DPSTN 4475	DPSTN 4475	DPSTN 4475	DPSTN 4475		
Al (ICP)	8.010	7.490	6.710	6.310	7.130	0.764
Ca (ICP)	0.119	0.112	0.450	0.560	0.310	0.229
Fe (ICP)	<0.004	<0.004	0.270	0.250	0.260	0.014
Mg (ICP)	0.028	0.024	0.120	0.130	0.076	0.057
Mn (ICP)	<0.004	<0.004	1.300	1.330	1.315	0.021
Na (ICP)			12.470	12.390	12.430	0.057
Na (AA*)	14.780	15.360	13.780	13.530	14.363	0.857
Li (ICP)	<0.002	<0.002	<0.002	<0.002	<0.002	
Ni (ICP)			0.390	0.390	0.390	0.000
Ni (AA*)			0.566	0.546	0.556	0.014
Si (ICP)	2.540	2.430	2.290	2.370	2.408	0.105
Si (AA*)	*11.670*	*9.770*	3.040	3.240	3.140	0.141
Cr (ICP)	0.007	0.006	0.020	0.020	0.013	0.008
B (ICP)	<0.002	<0.002	0.080	0.070	0.075	0.007
U (D-G)	4.718	4.853			4.786	0.095
Sr (ICP)	0.002	0.002	0.001	0.010	0.004	0.004
Zr (ICP)	<0.002	<0.002	0.010	0.010	0.010	0.000
Ti (ICP)	0.009	0.008	0.020	0.020	0.014	0.007
K (AA*)			0.033	0.032	0.033	0.001
P (ICP)*	2.260	1.780	0.920	0.900	1.465	0.670
P (Calc IC)	0.789	0.760			0.774	0.020
Ba (ICP)	0.031	0.003	<0.001	<0.001	0.017	0.020
Pb (ICP)	0.059	0.074	0.350	0.370	0.213	0.170
Mo (ICP)	<0.004	<0.004	0.010	0.010	0.010	0.000
Zn (ICP)	<0.004	<0.004	2.130	2.180	2.155	0.035
Cu (ICP)	0.011	0.012	0.020	0.020	0.016	0.005
F (ISE)	<0.265	<0.265			<0.265	
Cl (ISE)	0.191	0.187			0.189	0.003
SO4 (IC)	<0.5	<0.5			<0.5	
NO3 (IC)	34.970	34.350			34.660	0.438
PO4(IC)**	2.418	2.332			2.375	0.061
% SOLIDS (105°C)	38.980				38.980	
% SOLIDS (90°C)	38.540				38.540	
Moles of Na	0.643	0.668	0.599	0.589	0.625	0.037
Moles of NO3	0.564	0.554			0.559	0.007
Moles of Cl	0.005	0.005			0.005	0.000

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution
 all anions from Na2O2 fusion with H2O uptake (see text)
**water soluble phosphate only; ***acid soluble phosphate

MOLAR MASS BALANCE

Al(OH)3	15.716
NaNO3	47.511
SiO2 as Filter Aid	5.789
Na6U7O24	6.285
Ca-zeolite	2.851
AlPO4	4.492
SUM	82.644
EXCESS moles Na over NaNO3 and Na-U =	0.049

Tank 3	TK-3-1	TK-3-2	TK-3-1	TK-3-2	TANK 3 AVERAGE	TANK 3 STANDARD
ANALYSIS #	111472	111473	21299	21300		DEVIATION
ANALYSIS DATE	3/1/88	3/1/88	11/1/88	11/1/88		
RECORDED	DPSTN 4475	DPSTN 4475	DPSTN 4475	DPSTN 4475		
Al (ICP)	6.600	5.240	4.050	9.640	6.383	2.409
Ca (ICP)	0.154	0.171	0.600	0.290	0.304	0.207
Fe (ICP)	<0.004	<0.004	0.310	1.000	0.655	0.488
Mg (ICP)	0.035	0.019	0.150	0.070	0.069	0.058
Mn (ICP)	<0.004	<0.004	1.510	0.540	1.025	0.686
Na (ICP)			15.370	15.480	15.425	0.078
Na (AA*)	16.700	15.260	16.910	17.980	16.713	1.119
Li (ICP)	<0.002	<0.002	<0.002	<0.002	<0.002	
Ni (ICP)			0.300	0.250	0.275	0.035
Ni (AA*)			0.423	0.346	0.385	0.054
Si (ICP)	5.060	3.640	3.280	2.720	3.675	0.998
Si (AA*)	*10.200*	*11.830*	4.550	3.660	4.105	0.629
Cr (ICP)	<0.004	<0.004	0.010	0.010	0.010	0.000
B (ICP)	<0.002	<0.002	0.040	0.010	0.025	0.021
U (D-G)	3.023	3.434			3.229	0.291
Sr (ICP)	0.002	0.002	0.010	<0.0005	0.005	0.005
Zr (ICP)	<0.002	<0.002	0.010	0.010	0.010	0.000
Ti (ICP)	0.005	<0.001	0.020	0.020	0.015	0.009
K (AA*)			0.057	0.051	0.054	0.004
P (ICP)*	2.320	2.170	1.280	2.040	1.953	0.463
P (Calc IC)	1.255	1.327			1.291	0.051
Ba (ICP)	0.031	0.029	<0.001	<0.001	0.030	0.001
Pb(ICP)	0.019	0.017	0.390	0.200	0.157	0.178
Mo (ICP)	<0.004	<0.004	0.010	0.010	0.010	0.000
Zn (ICP)	<0.004	<0.004	2.450	0.990	1.720	1.032
Cu (ICP)	0.009	0.007	0.010	0.030	0.014	0.011
F (ISE)	<0.02	<0.02			<0.02	
Cl (ISE)	0.142	0.143			0.142	0.001
SO4 (IC)	<0.5	<2.0			<0.5	
NO3 (IC)	49.500	51.660			50.580	1.527
PO4(IC)**	3.850	4.070			3.960	0.156
% SOLIDS (105°C)	9.240				9.240	
% SOLIDS (90°C)	6.800				6.800	
Moles of Na	0.726	0.664	0.736	0.782	0.727	0.049
Moles of NO3	0.798	0.833			0.816	0.025
Moles of Cl	0.004	0.004			0.004	0.000

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution

   all anions from Na2O2 fusion with H2O uptake (see text)

**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE SPECIATION

Al(OH)3	12.35
NaNO3	69.33
Filter Aid	8.52
Na6U7O24	4.24
Ca-Zeolite-A	2.79
AlPO4	5.99
SUM	103.222
EXCESS moles Na over	
NaNO3 and Na-U =	-0.100

Tank 4	TK-4-1	TK-4-2	TK-4-1	TK-4-2	TANK 4 AVERAGE	TANK 4 STANDARD
ANALYSIS #	111474	111475	21301	21302		DEVIATION
ANALYSIS DATE	3/1/88	3/1/88	11/1/88	11/1/88		
RECORDED	DPSTN 4475	DPSTN 4475	DPSTN 4475	DPSTN 4475		
Al (ICP)	1.730	1.200	0.990	8.250	3.043	3.486
Ca (ICP)	0.108	0.030	0.420	0.260	0.205	0.173
Fe (ICP)	<0.004	<0.004	0.040	0.390	0.215	0.247
Mg (ICP)	0.059	0.040	0.100	0.080	0.070	0.026
Mn (ICP)	0.682	0.465	1.320	0.470	0.734	0.403
Na (ICP)			21.110	22.000	21.555	0.629
Na (AA)	24.180	24.080	26.060	27.420	25.435	1.606
Li (ICP)	<0.002	<0.002	<0.002	<0.002	<0.002	
Ni (ICP)			0.080	0.030	0.055	0.035
Ni (AA)			0.114	0.050	0.082	0.045
Si (ICP)	1.070	0.742	0.560	4.150	1.631	1.693
Si (AA)	*2.560*	*2.350*	0.826	5.820	3.323	3.531
Cr (ICP)	<0.004	<0.004	0.010	0.020	0.015	0.007
B (ICP)	<0.002	<0.002	0.020	0.010	0.015	0.007
U (D-G)	1.321	1.240			1.281	0.057
Sr (ICP)	0.002	<0.001	<0.0005	<0.0005	0.002	
Zr (ICP)	0.138	0.093	0.010	<0.004	0.080	0.065
Ti (ICP)	0.023	0.015	0.010	0.020	0.017	0.006
K (AA)			0.023	0.018	0.021	0.004
P (ICP)*	1.210	1.100	0.270	2.610	1.298	0.970
P (Calc IC)	0.320	0.432			0.376	0.079
Ba (ICP)	0.033	0.031	<0.001	<0.001	0.032	0.001
Pb (ICP)	0.046	0.016	0.320	0.160	0.136	0.138
Mo (ICP)	0.013	0.007	0.010	0.010	0.010	0.002
Zn (ICP)	1.340	0.927	2.100	0.810	1.294	0.583
Cu (ICP)	<0.004	<0.004	0.010	0.030	0.020	0.014
F (ISE)	<0.02	<0.02			<0.02	
Cl (ISE)	0.171	0.269			0.220	0.069
SO4 (IC)	<0.5	<0.5			<0.5	
NO3 (IC)	50.000	69.000			59.500	13.435
PO4(IC)**	0.980	1.324			1.152	0.243
% SOLIDS (105°C)	22.340				22.340	
% SOLIDS (90°C)	19.870				19.870	
Moles of Na	1.052	1.047	1.134	1.193	1.106	0.070
Moles of NO3	0.806	1.113			0.960	0.217
Moles of Cl	0.005	0.008			0.006	0.002

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution
   all anions from Na2O2 fusion with H2O uptake (see text)
**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE SPECIATION

Al(OH)3	4.73
NaNO3	81.56
Filter Aid	7.03
Na6U7O24	1.68
Ca-Zeolite-A	1.88
AlPO4	3.98
SUM	100.862
EXCESS moles Na over	
NaNO3 and Na-U =	0.142

Tank 5	TK-5-1	TK-5-2	TK-5-2	TK-5-2	TANK 5	TANK 5
ANALYSIS #	111476	111477	21303	21304	AVERAGE	STANDARD
ANALYSIS DATE	3/1/88	3/1/88	11/1/88	11/1/88		DEVIATION
RECORDED	DPSTN 4475	DPSTN 4475	DPSTN 4475	DPSTN 4725		
Al (ICP)	9.540	9.600	10.000	9.970	9.778	0.241
Ca (ICP)	0.151	0.375	0.410	0.370	0.327	0.118
Fe (ICP)	<0.004	<0.005	0.510	0.480	0.495	0.021
Mg (ICP)	0.094	0.102	0.180	0.170	0.137	0.045
Mn (ICP)	0.027	<0.005	0.660	0.490	0.392	0.328
Na (ICP)			11.090	9.940	10.515	0.813
Na (AA)	10.940	10.600	11.580	9.750	10.718	0.762
Li (ICP)	<0.002	<0.002	<0.002	<0.002	<0.002	
Ni (ICP)			0.560	0.550	0.555	0.007
Ni (AA)			0.777	0.739	0.758	0.027
Si (ICP)	10.200	10.300	11.410	11.250	10.790	0.628
Si (AA)	*14.210*	*10.080*	13.990	14.070	14.030	0.057
Cr (ICP)	<0.004	<0.005	0.020	0.010	0.015	0.007
B (ICP)	<0.002	<0.002	0.020	0.010	0.015	0.007
U (D-G)	4.180	3.536			3.858	0.455
Sr (ICP)	0.002	0.004	<0.0005	<0.0005	0.003	0.001
Zr (ICP)	<0.002	<0.002	0.010	0.010	0.010	0.000
Ti (ICP)	0.024	0.029	0.040	0.040	0.033	0.008
K (AA)			0.110	0.097	0.104	0.009
P (ICP)*	1.570	2.200	0.670	0.640	1.270	0.755
P (Calc IC)	<0.848	N/A				
Ba (ICP)	0.029	0.036	0.000	0.000	0.016	0.019
Pb (ICP)	0.031	<0.014	0.190	0.150	0.124	0.083
Mo (ICP)	<0.004	<0.005	0.010	0.010	0.010	0.000
Zn (ICP)	<0.004	<0.005	1.070	0.820	0.945	0.177
Cu (ICP)	0.005	0.009	0.010	0.010	0.009	0.002
F (ISE)	<0.02	<0.02			<0.02	
Cl (ISE)	0.843	N/A			0.843	
SO4 (IC)	<1.31	N/A			<1.31	
NO3 (IC)	12.000	N/A			12.000	
PO4(IC)**	<2.6	N/A			<2.6	
% SOLIDS (105°C)	32.490				32.490	
% SOLIDS (90°C)	28.290				28.290	
Moles of Na	0.476	0.461	0.504	0.424	0.466	0.033
Moles of NO3	0.194	N/A			0.194	
Moles of Cl	0.024	N/A			0.024	

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution
   all anions from Na2O2 fusion with H2O uptake (see text)
**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE SPECIATION

Al(OH)3	23.798
NaNO3	16.449
SiO2 as Filter Aid	29.042
Na6U7O24	5.067
Ca-zeolite	3.001
AlPO4	3.894
SUM	81.251
EXCESS moles Na over	
NaNO3 and Na-U =	0.259

Tank 6	TK-6-1	TK-6-2	TK-6-1	TK-6-2	TANK 6 AVERAGE	TANK 6 STANDARD
ANALYSIS #	111478	111479	21305 & 6	21307		DEVIATION
ANALYSIS DATE	3/1/88	3/1/88	11/1/88	11/1/88		
RECORDED	DPSTN 4475	DPSTN 4475	DPSTN 4725	DPSTN 4725		
Al (ICP)	9.870	9.740	11.020	9.420	10.013	0.698
Ca (ICP)	0.203	0.195	1.180	0.410	0.497	0.466
Fe (ICP)	<0.004	<0.004	0.850	0.630	0.740	0.156
Mg (ICP)	0.102	0.101	0.350	0.200	0.188	0.117
Mn (ICP)	BDL	BDL	2.650	0.420	1.535	1.577
Na (ICP)			11.000	8.340	9.670	1.881
Na (AA)	9.480	9.180	11.980	10.270	10.228	1.256
Li (ICP)	<0.002	<0.002	<0.002	<0.002	<0.002	
Ni (ICP)			0.430	0.420	0.425	0.007
Ni (AA)			0.604	0.597	0.601	0.005
Si (ICP)	10.800	10.700	11.750	15.990	12.310	2.499
Si (AA)	*14.760*	*13.190*	17.800	18.200	18.000	0.283
Cr (ICP)	<0.004	<0.004	0.020	0.020	0.020	0.000
B (ICP)	<0.002	<0.002	<0.009	0.010	0.010	
U (D-G)	4.519	3.640			4.080	0.622
Sr (ICP)	0.004	0.004	0.010	<0.0005	0.006	0.003
Zr (ICP)	<0.002	<0.002	0.010	0.010	0.010	0.000
Ti (ICP)	0.026	0.026	0.050	0.050	0.038	0.014
K (AA)			0.108	0.106	0.107	0.001
P (ICP)***	1.680	1.520	0.650	0.690	1.135	0.541
P (Calc IC)**	0.233	0.255			0.244	0.016
Ba (ICP)	0.048	0.046	<0.001	<0.001	0.047	0.001
Pb (ICP)	0.048	0.021	0.760	0.150	0.245	0.348
Mo (ICP)	<0.004	<0.004	0.020	0.010	0.015	0.007
Zn (ICP)	<0.004	<0.004	5.000	0.750	2.875	3.005
Cu (ICP)	0.012	0.010	0.040	0.040	0.026	0.017
F (ISE)	<0.02	N/A			<0.02	
Cl (ISE)	0.060	0.060			0.060	0.000
SO4 (IC)	<0.5	<0.5			<0.5	
NO3 (IC)	12.650	13.090			12.870	0.311
PO4(IC)**	0.715	0.783			0.749	0.048
% SOLIDS (105°C)	37.120				37.120	
% SOLIDS (90°C)	36.980				36.980	
Moles of Na	0.412	0.399	0.521	0.447	0.445	0.055
Moles of NO3	0.204	0.211			0.208	0.005
Moles of Cl	0.002	0.002			0.002	0.000

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution
  all anions from Na2O2 fusion with H2O uptake (see text)
**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE SPECIATION

Al(OH)3	24.15
NaNO3	17.64
Filter Aid	40.05
Na6U7O24	5.36
Ca-Zeolite-A	4.57
AlPO4	3.48
SUM	95.26
EXCESS moles Na over	
NaNO3 and Na-U =	0.223

Tank 7	TK-7-1	TK-7-2		TANK 7 AVERAGE	TANK 7 STANDARD DEVIATION
ANALYSIS #	200076566	200076567			
ANALYSIS DATE	5/12/92	5/12/92			
RECORDED	DPSTN 4725	DPSTN 4725			
Al (ICP)	6.236	6.549		6.392	0.222
Ca (ICP)	0.576	0.264		0.420	0.220
Fe (ICP)	0.456	0.601		0.529	0.102
Mg (ICP)	0.136	0.115		0.125	0.014
Mn (ICP)	0.006	0.003		0.005	0.002
Na (ICP)	12.863	11.720		12.292	0.808
Na (AA)	13.820	13.040		13.430	0.552
Li (ICP)	<0.001	<0.001		<0.001	
Ni (ICP)	1.781	1.961		1.871	0.127
Ni (AA)	1.837	2.133		1.985	0.209
Si (ICP)	15.715	16.023		15.869	0.218
Si (AA)	*18.260*	*17.710*		17.985	0.389
Cr (ICP)	<0.004	<0.004		<0.004	
B (ICP)	0.073	<0.004		0.037	
U (Chemcheck)	2.880	3.130		3.005	0.177
Sr (ICP)	<0.000	<0.000		<0.000	
Zr (ICP)	<0.002	<0.002		<0.002	
Ti (ICP)	0.028	0.019		0.024	0.006
K (AA)	0.530	0.630		0.580	0.071
P (ICP)*	1.624	0.651		1.137	0.688
P (Calc IC)**	1.931	1.994		1.963	0.045
Ba (ICP)	<0.00	<0.00		0.000	
Pb (ICP)	<0.020	0.156		0.156	
Mo (ICP)	<0.004	<0.004		<0.004	
Zn (ICP)	0.171	0.181		0.176	0.007
Cu (ICP)	<0.001	0.172		0.172	
F (ISE)	0.050	0.050		0.050	0.000
Cl (ISE)	0.700	0.650		0.675	0.035
SO4 (IC)	0.271	0.204		0.237	0.047
NO3 (IC)	15.260	15.010		15.135	0.177
PO4(IC)**	5.922	6.116		6.019	0.137
% SOLIDS (105°C)	30.860	30.840		30.850	0.014
% SOLIDS (90°C)	N/A	N/A		N/A	N/A
Moles of Na	0.601	0.567		0.584	0.024
Moles of NO3	0.246	0.242		0.244	0.003
Moles of Cl	0.020	0.018		0.019	0.001

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution
  all anions from Na2O2 fusion with H2O uptake (see text)
**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE SPECIATION

Al(OH)3	13.98
NaNO3	20.75
Filter Aid	40.27
Na6U7O24	3.95
Ca-Zeolite-A	3.86
AlPO4	3.49
SUM	86.291
EXCESS moles Na over	
NaNO3 and Na-U =	0.329

Tank 8 (P42)	TK-8-P42-1	TK-8-P42-2	TK-8-P42-1	TK-8-P42-2	TANK 8-P42 AVERAGE	TANK 8-P42 STANDARD
ANALYSIS #	111480	111481	21308	21309		DEVIATION
ANALYSIS DATE	3/1/88	3/1/88	11/1/88	11/1/88		
RECORDED	DPSTN 4475	DPSTN 4475	DPSTN 4725	DPSTN 4725		
Al (ICP)	10.900	10.700	10.780	10.770	10.788	0.083
Ca (ICP)	0.183	0.183	0.340	0.560	0.317	0.178
Fe (ICP)	<0.004	<0.004	0.440	0.400	0.420	0.028
Mg (ICP)	0.096	0.087	0.130	0.160	0.118	0.033
Mn (ICP)	<0.004	<0.004	0.440	0.770	0.605	0.233
Na (ICP)			7.590	5.850	6.720	1.230
Na (AA)	6.810	6.730	8.220	7.250	7.253	0.684
Li (ICP)	<0.002	<0.002	<0.002	<0.002	<0.002	
Ni (ICP)			0.740	0.740	0.740	0.000
Ni (AA)			0.959	0.952	0.956	0.005
Si (ICP)	10.900	10.500	11.390	11.510	11.075	0.465
Si (AA)	*14.260*	*14.860*	14.060	15.030	14.545	0.686
Cr (ICP)	0.004	<0.004	0.020	0.020	0.015	0.009
B (ICP)	<0.002	<0.002	0.010	0.010	0.010	0.000
U (D-G)	6.840	6.023			6.432	0.578
Sr (ICP)	0.004	0.004	<0.0005	0.010	0.006	0.003
Zr (ICP)	<0.002	<0.002	0.010	0.010	0.010	0.000
Ti (ICP)	0.022	0.020	0.030	0.040	0.028	0.009
K (AA)			0.124	0.121	0.123	0.002
P (ICP)*	2.730	2.780	1.350	1.310	2.043	0.823
P (Calc IC)	0.964	1.023			0.993	0.042
Ba (ICP)	0.049	0.051	<0.001	<0.001	0.050	0.001
Pb (ICP)	<0.012	<0.012	0.150	0.220	0.185	0.049
Mo (ICP)	<0.004	<0.004	0.010	0.010	0.010	0.000
Zn (ICP)	<0.004	<0.004	0.770	1.280	1.025	0.361
Cu (ICP)	0.016	0.011	0.040	0.020	0.022	0.013
F (ISE)	N/A	N/A			N/A	
Cl (ISE)	0.059	0.060			0.059	0.001
SO4 (IC)	<0.5	<0.5			<0.5	
NO3 (IC)	15.810	16.750			16.280	0.665
PO4(IC)**	2.955	3.136			3.046	0.128
% SOLIDS (105°C)	34.480				34.480	
% SOLIDS (90°C)	31.650				31.650	
Moles of Na	0.296	0.293	0.358	0.315	0.315	0.030
Moles of NO3	0.255	0.270			0.263	0.011
Moles of Cl	0.002	0.002			0.002	0.000

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution

   all anions from Na2O2 fusion with H2O uptake (see text)

**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE SPECIATION

Al(OH)3	24.81
NaNO3	22.32
Filter Aid	32.64
Na6U7O24	8.45
Ca-Zeolite-A	2.91
AlPO4	6.26
SUM	97.39
EXCESS moles Na over	
NaNO3 and Na-U =	0.030

Tank 8 (P43)	TK-8-P43-1	TK-8-P43-2	TK-8-P43-1	TK-8-P43-2	TANK 8-P43 AVERAGE	TANK 8-P43 STANDARD
ANALYSIS #	111482	111483	21310	21311		DEVIATION
ANALYSIS DATE	3/1/88	3/1/88	11/1/88	11/1/88		
RECORDED	DPSTN 4475	DPSTN 4475	DPSTN 4725	DPSTN 4725		
Al (ICP)	10.700	9.500	11.310	11.210	10.680	0.831
Ca (ICP)	0.195	0.291	0.340	0.360	0.297	0.074
Fe (ICP)	0.229	<0.004	0.460	0.460	0.383	0.133
Mg (ICP)	0.122	0.099	0.140	0.140	0.125	0.019
Mn (ICP)	0.065	0.014	0.430	0.450	0.240	0.232
Na (ICP)			6.960	7.640	7.300	0.481
Na (AA)	7.530	3.650	9.130	7.130	6.860	2.308
Li (ICP)	<0.002	<0.002	<0.002	<0.002	<0.002	
Ni (ICP)			0.710	0.720	0.715	0.007
Ni (AA)			0.948	1.100	1.024	0.107
Si (ICP)	11.700	10.400	12.090	11.920	11.528	0.768
Si (AA)	*14.590*	*13.360*	15.140	14.630	14.885	0.361
Cr (ICP)	<0.004	<0.004	0.020	0.020	0.020	0.000
B (ICP)	<0.002	<0.002	0.010	0.010	0.010	0.000
U (D-G)	5.960	1.710			3.835	3.005
Sr (ICP)	0.004	0.006	<0.0005	<0.0005	0.005	0.001
Zr (ICP)	0.007	<0.002	0.010	0.010	0.009	0.002
Ti (ICP)	0.031	0.025	0.030	0.030	0.029	0.003
K (AA)			0.118	0.120	0.119	0.001
P (ICP)*	2.330	2.040	1.330	1.300	1.750	0.516
P (Calc IC)	0.955	0.950			0.953	0.004
Ba (ICP)	0.057	0.054	<0.001	<0.001	0.056	0.002
Pb (ICP)	0.042	0.026	0.140	0.150	0.090	0.065
Mo (ICP)	0.005	<0.004	0.010	0.010	0.008	0.003
Zn (ICP)	0.177	<0.002	0.730	0.780	0.562	0.335
Cu (ICP)	0.017	0.012	0.030	0.030	0.022	0.009
F (ISE)	N/A	N/A			N/A	
Cl (ISE)	0.062	0.057			0.059	0.004
SO4 (IC)	<1.0	<1.0			<1.0	
NO3 (IC)	17.450	15.495			16.473	1.382
PO4(IC)**	2.930	2.913			2.922	0.012
% SOLIDS (105°C)	41.990				41.990	
% SOLIDS (90°C)	33.120				33.120	
Moles of Na	0.328	0.159	0.397	0.310	0.298	0.100
Moles of NO3	0.281	0.250			0.266	0.022
Moles of Cl	0.002	0.002			0.002	0.000

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution

  all anions from Na2O2 fusion with H2O uptake (see text)

**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE SPECIATION

Al(OH)3	25.31
NaNO3	22.58
Filter Aid	33.49
Na6U7O24	5.04
Ca-Zeolite-A	2.72
AlPO4	5.37
SUM	94.52
EXCESS moles Na over	
NaNO3 and Na-U =	0.019

Tank 10	TK-10-1	TK-10-2		TANK 10 AVERAGE	TANK 10 STANDARD DEVIATION
ANALYSIS #	200076568	200076569			
ANALYSIS DATE	5/12/92	5/12/92			
RECORDED	DPSTN 4725	DPSTN 4725			
Al (ICP)	8.613	9.179		8.896	0.400
Ca (ICP)	0.322	0.379		0.351	0.040
Fe (ICP)	0.892	0.847		0.870	0.032
Mg (ICP)	0.105	0.138		0.122	0.023
Mn (ICP)	0.035	0.035		0.035	0.000
Na (ICP)	7.594	6.798		7.196	0.563
Na (AA)	7.860	7.006		7.433	0.604
Li (ICP)	<0.001	<0.001		<0.001	
Ni (ICP)	0.066	0.062		0.064	0.003
Ni (AA)	<0.06	<0.06		<0.06	
Si (ICP)	23.950	25.620		24.785	1.181
Si (AA)	*26.720*	*28.170*		27.445	1.025
Cr (ICP)	<0.004	<0.004		<0.004	
B (ICP)	<0.004	<0.004		<0.004	
U (Chemcheck)	0.040	0.050		0.045	0.007
Sr (ICP)	0.000	0.000		0.000	0.000
Zr (ICP)	<0.002	<0.002		<0.002	
Ti (ICP)	0.027	0.026		0.027	0.001
K (AA)	2.500	2.710		2.605	0.148
P (ICP)*	0.976	1.085		1.031	0.077
P (Calc IC)	0.995	1.076		1.035	0.058
Ba (ICP)	0.000	0.000		0.000	
Pb (ICP)	0.071	<0.02		0.071	
Mo (ICP)	<0.004	<0.004			
Zn (ICP)	0.169	0.175		0.172	0.004
Cu (ICP)	<0.001	0.005		0.005	
F (ISE)	0.060	0.060		0.060	0.000
Cl (ISE)	0.360	0.260		0.310	0.071
SO4 (IC)	<0.2	<0.2		<0.2	
NO3 (IC)	7.640	8.229		7.935	0.416
PO4(IC)**	3.050	3.300		3.175	0.177
% SOLIDS (105°C)	17.520	17.600		17.560	0.057
% SOLIDS (90°C)	N/A	N/A		N/A	
Moles of Na	0.342	0.305		0.323	0.026
Moles of NO3	0.123	0.133		0.128	0.007
Moles of Cl	0.010	0.007		0.009	0.002

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution
  all anions from Na2O2 fusion with H2O uptake (see text)
**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE SPECIATION

Al(OH)3	21.76
NaNO3	10.88
Filter Aid	62.39
Na6U7O24	0.06
Ca-Zeolite-A	3.22
AlPO4	3.16
SUM	101.469
EXCESS moles Na over	
NaNO3 and Na-U =	0.195

New Tank 10	NEW TK10-A	NEW TK10-B		NEW TANK 10 AVERAGE	TANK 10 STANDARD DEVIATION
ANALYSIS #	300011338	300011339			
ANALYSIS DATE	7/6/93	7/6/93			
RECORDED	DPSTN 4695	DPSTN 4695			
Al (ICP)	9.672	9.612		9.642	0.042
Ca (ICP)	0.403	0.447		0.425	0.031
Fe (ICP)	0.904	1.188		1.046	0.201
Mg (ICP)	0.120	0.124		0.122	0.003
Mn (ICP)	0.048	0.050		0.049	0.001
Na (ICP)	5.255	5.008		5.132	0.175
Na (AA)	5.198	5.268		5.233	0.049
Li (ICP)	0.004	0.002		<0.001	
Ni (ICP)	0.080	0.081		0.081	0.001
Ni (AA)	0.052	0.061		<0.06	
Si (ICP)	25.377	24.910		25.144	0.330
Si (AA)	*24.880*	*24.810*		24.845	0.049
Cr (ICP)	0.009	0.005		<0.004	
B (ICP)	<0.004	<0.004		<0.004	
U (Chemcheck)	0.295	0.148		0.222	0.104
Sr (ICP)	0.002	0.002		0.002	0.000
Zr (ICP)	0.011	0.016		<0.002	
Ti (ICP)	0.060	0.064		0.062	0.003
K (AA)	2.046	2.060		2.053	0.010
P (ICP)*	1.081	0.837		0.959	0.173
P (Calc IC)**	0.984	1.010		0.997	0.019
Ba (ICP)	0.017	0.015		0.016	0.001
Pb (ICP)	0.037	0.026		0.032	0.008
Mo (ICP)	0.006	0.004		0.005	0.001
Zn (ICP)	0.465	0.462		0.464	0.002
Cu (ICP)	0.095	0.035		0.065	0.042
F (ISE)	0.920	1.459		1.189	0.381
Cl (ISE)	0.121	0.147		0.134	0.019
SO4 (IC)	0.178	0.486		0.332	0.218
NO3 (IC)	6.082	6.322		6.202	0.170
PO4(IC)**	3.016	3.099		3.057	0.058
% SOLIDS (105°C)	19.960	19.980		19.970	0.014
% SOLIDS (90°C)	5.380	5.380		5.380	
Moles of Na	0.226	0.229		0.228	0.002
Moles of NO3	0.098	0.102		0.100	0.003
Moles of Cl	0.003	0.004		0.004	0.001

*AA Silicon values in italics from HCl/HF dissolution; others from Na2O2 dissolution
   all anions from Na2O2 fusion with H2O uptake (see text)
**water soluble phosphate only; ***acid soluble phosphate

MASS BALANCE SPECIATION

Al(OH)3	23.81
NaNO3	8.50
Filter Aid	56.13
Na6U7O24	0.29
Ca-Zeolite-A	3.91
AlPO4	2.94
SUM	95.58
EXCESS moles Na over NaNO3 and Na-U =	0.127

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APPENDIX II M-AREA VIRGIN FILTER AID ANALYSES

SAMPLE ID = DIATOMACEOUS EARTH (DPSTN=4725, ADS# 100021313-AFTER DRYING AT 600°C)

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%	NORM OXIDE WT%	M.W.	MOLE FRACTION	NORM OXIDE MOLE %
Al2O3	8.5100	1.8895	16.0796	15.5271	101.9600	0.1577	8.5292
CaO	0.6500	1.3992	0.9095	0.8782	56.0794	0.0162	0.8771
Fe2O3	0.6400	1.4297	0.9150	0.8836	159.6922	0.0057	0.3099
FeO		1.2865	0.0000	0.0000	71.8464	0.0000	0.0000
MgO	0.0800	1.6583	0.1327	0.1281	40.3114	0.0033	0.1780
MnO2	0.5700	1.5825	0.9020	0.8710	70.9374	0.0127	0.6877
Na2O	2.4300	1.3480	3.2756	3.1631	61.9790	0.0529	2.8583
Li2O		2.1525	0.0000	0.0000	29.8774	0.0000	0.0000
NiO	0.2400	1.2726	0.3054	0.2949	74.7094	0.0041	0.2211
SiO2	31.4700	2.1393	67.3238	65.0102	60.0848	1.1205	60.5987
Cr2O3		1.4616	0.0000	0.0000	151.9974	0.0000	0.0000
B2O3		3.2201	0.0000	0.0000	69.6204	0.0000	0.0000
UO2		1.1344	0.0000	0.0000	270.0388	0.0000	0.0000
ThO2		1.1379	0.0000	0.0000	264.0400	0.0000	0.0000
SrO		1.1826	0.0000	0.0000	103.6194	0.0000	0.0000
ZrO2	0.0100	1.3508	0.0135	0.0130	123.2188	0.0001	0.0059
TiO2	0.0400	1.6680	0.0667	0.0644	79.8988	0.0008	0.0452
K2O	3.7000	1.2046	4.4570	4.3039	94.2034	0.0473	2.5588
Cs2O		1.0602	0.0000	0.0000	281.8094	0.0000	0.0000
Sb2O3		1.1970	0.0000	0.0000	291.4982	0.0000	0.0000
P2O5	0.1900	2.2910	0.4353	0.4203	141.9370	0.0031	0.1659
Nd2O3		1.1660	0.0000	0.0000	336.4782	0.0000	0.0000
La2O3		1.1728	0.0000	0.0000	325.8100	0.0000	0.0000
Y2O3		1.2699	0.0000	0.0000	225.8082	0.0000	0.0000
BaO	0.0300	1.1165	0.0335	0.0323	153.3394	0.0002	0.0118
PbO	0.1800	1.0772	0.1939	0.1872	223.1900	0.0009	0.0470
CeO2		1.2284	0.0000	0.0000	172.1200	0.0000	0.0000
MoO3		1.5003	0.0000	0.0000	143.9382	0.0000	0.0000
ZnO	0.9000	1.2447	1.1202	1.0817	81.3800	0.0138	0.7445
CuO	0.0200	1.2518	0.0250	0.0242	79.5454	0.0003	0.0170
H2O	7.3700	1.0000	7.3700	7.1167	18.0000	0.4094	22.1439
SUMS			103.5589	100.0000		1.8490	100.0000

SAMPLE ID = PERFLO (DPSTN=4725, ADS# 100021312-AFTER DRYING AT 600°C)

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%	NORM OXIDE WT%	M.W.	MOLE FRACTION	NORM OXIDE MOLE %
Al2O3	6.5400	1.8895	12.3573	12.5865	101.9600	0.1212	8.0597
CaO	0.6600	1.3992	0.9235	0.9406	56.0794	0.0165	1.0951
Fe2O3	1.1700	1.4297	1.6727	1.7038	159.6922	0.0105	0.6966
FeO		1.2865	0.0000	0.0000	71.8464	0.0000	0.0000
MgO	0.0700	1.6583	0.1161	0.1182	40.3114	0.0029	0.1915
MnO2	0.3700	1.5825	0.5855	0.5964	70.9374	0.0083	0.5489
Na2O	2.6300	1.3480	3.5452	3.6110	61.9790	0.0572	3.8039
Li2O		2.1525	0.0000	0.0000	29.8774	0.0000	0.0000
NiO		1.2726	0.0000	0.0000	74.7094	0.0000	0.0000
SiO2	34.1000	2.1393	72.9501	74.3028	60.0848	1.2141	80.7398
Cr2O3		1.4616	0.0000	0.0000	151.9974	0.0000	0.0000
B2O3		3.2201	0.0000	0.0000	69.6204	0.0000	0.0000
UO2		1.1344	0.0000	0.0000	270.0388	0.0000	0.0000
ThO2		1.1379	0.0000	0.0000	264.0400	0.0000	0.0000
SrO		1.1826	0.0000	0.0000	103.6194	0.0000	0.0000
ZrO2	0.0100	1.3508	0.0135	0.0138	123.2188	0.0001	0.0073
TiO2	0.0400	1.6680	0.0667	0.0680	79.8988	0.0008	0.0555
K2O	4.0000	1.2046	4.8184	4.9077	94.2034	0.0511	3.4014
Cs2O		1.0602	0.0000	0.0000	281.8094	0.0000	0.0000
Sb2O3		1.1970	0.0000	0.0000	291.4982	0.0000	0.0000
P2O5	0.0400	2.2910	0.0916	0.0933	141.9370	0.0006	0.0429
Nd2O3		1.1660	0.0000	0.0000	336.4782	0.0000	0.0000
La2O3		1.1728	0.0000	0.0000	325.8100	0.0000	0.0000
Y2O3		1.2699	0.0000	0.0000	225.8082	0.0000	0.0000
BaO	0.0400	1.1165	0.0447	0.0455	153.3394	0.0003	0.0194
PbO	0.0900	1.0772	0.0969	0.0987	223.1900	0.0004	0.0289
CeO2		1.2284	0.0000	0.0000	172.1200	0.0000	0.0000
MoO3		1.5003	0.0000	0.0000	143.9382	0.0000	0.0000
ZnO	0.5400	1.2447	0.6721	0.6846	81.3800	0.0083	0.5492
CuO	0.0200	1.2518	0.0250	0.0255	79.5454	0.0003	0.0209
H2O	0.2000	1.0000	0.2000	0.2037	18.0000	0.0111	0.7389
SUMS			98.1796	100.0000		1.5037	100.0000

SAMPLE ID = PERLITE 2000X (DPSTN=4725, ADS# 200002283-AFTER MELTING AT 1150°C)

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%	NORM OXIDE WT%	M.W.	MOLE FRACTION	NORM OXIDE MOLE %
Al2O3	6.6040	1.8895	12.4783	12.5644	101.9600	0.1224	8.0277
CaO	0.5010	1.3992	0.7010	0.7058	56.0794	0.0125	0.8199
Fe203	0.6160	1.4297	0.8807	0.8868	159.6922	0.0055	0.3617
FeO		1.2865	0.0000	0.0000	71.8464	0.0000	0.0000
MgO	0.0630	1.6583	0.1045	0.1052	40.3114	0.0026	0.1700
MnO2	0.0650	1.5825	0.1029	0.1036	70.9374	0.0015	0.0951
Na2O	3.3230	1.3480	4.4794	4.5103	61.9790	0.0723	4.7407
Li2O		2.1525	0.0000	0.0000	29.8774	0.0000	0.0000
NiO	<0.005	1.2726	0.0000	0.0000	74.7094	0.0000	0.0000
SiO2	34.9410	2.1393	74.7493	75.2652	60.0848	1.2441	81.6034
Cr2O3		1.4616	0.0000	0.0000	151.9974	0.0000	0.0000
B203		3.2201	0.0000	0.0000	69.6204	0.0000	0.0000
UO2		1.1344	0.0000	0.0000	270.0388	0.0000	0.0000
ThO2		1.1379	0.0000	0.0000	264.0400	0.0000	0.0000
SrO		1.1826	0.0000	0.0000	103.6194	0.0000	0.0000
ZrO2	0.0080	1.3508	0.0108	0.0109	123.2188	0.0001	0.0058
TiO2	0.0370	1.6680	0.0617	0.0621	79.8988	0.0008	0.0507
K2O	4.5020	1.2046	5.4231	5.4605	94.2034	0.0576	3.7761
Cs2O		1.0602	0.0000	0.0000	281.8094	0.0000	0.0000
Sb2O3		1.1970	0.0000	0.0000	291.4982	0.0000	0.0000
P2O5	0.0130	2.2910	0.0298	0.0300	141.9370	0.0002	0.0138
Nd2O3		1.1660	0.0000	0.0000	336.4782	0.0000	0.0000
La2O3		1.1728	0.0000	0.0000	325.8100	0.0000	0.0000
Y2O3		1.2699	0.0000	0.0000	225.8082	0.0000	0.0000
BaO	0.0360	1.1165	0.0402	0.0405	153.3394	0.0003	0.0172
PbO	<0.019	1.0772	0.0000	0.0000	223.1900	0.0000	0.0000
CeO2		1.2284	0.0000	0.0000	172.1200	0.0000	0.0000
MoO3		1.5003	0.0000	0.0000	143.9382	0.0000	0.0000
ZnO	0.1510	1.2447	0.1879	0.1892	81.3800	0.0023	0.1515
CuO	0.0200	1.2518	0.0250	0.0252	79.5454	0.0003	0.0206
H2O	0.0400	1.0000	0.0400	0.0403	18.0000	0.0022	0.1458
SUMS			99.3146	100.0000		1.5245	100.0000

SAMPLE ID = PERLITE 200SP (DPSTN=4725, ADS# 300002284-AFTER MELTING AT 1150°C)

	ELEMENT WT%	GRAV FACTOR	OXIDE WT%	NORM OXIDE WT%	M.W.	MOLE FRACTION	NORM OXIDE MOLE %
Al2O3	6.8450	1.8895	12.9336	13.0212	101.9600	0.1269	8.3010
CaO	0.4360	1.3992	0.6101	0.6142	56.0794	0.0109	0.7119
Fe2O3	0.7100	1.4297	1.0151	1.0220	159.6922	0.0064	0.4160
FeO		1.2865	0.0000	0.0000	71.8464	0.0000	0.0000
MgO	0.1800	1.6583	0.2985	0.3005	40.3114	0.0074	0.4846
MnO2	0.0500	1.5825	0.0791	0.0797	70.9374	0.0011	0.0730
Na2O	2.5360	1.3480	3.4185	3.4417	61.9790	0.0552	3.6094
Li2O		2.1525	0.0000	0.0000	29.8774	0.0000	0.0000
NiO	<0.005	1.2726	0.0000	0.0000	74.7094	0.0000	0.0000
SiO2	35.2590	2.1393	75.4296	75.9400	60.0848	1.2554	82.1518
Cr2O3		1.4616	0.0000	0.0000	151.9974	0.0000	0.0000
B2O3		3.2201	0.0000	0.0000	69.6204	0.0000	0.0000
UO2		1.1344	0.0000	0.0000	270.0388	0.0000	0.0000
ThO2		1.1379	0.0000	0.0000	264.0400	0.0000	0.0000
SrO		1.1826	0.0000	0.0000	103.6194	0.0000	0.0000
ZrO2	0.0080	1.3508	0.0108	0.0109	123.2188	0.0001	0.0057
TiO2	0.0620	1.6680	0.1034	0.1041	79.8988	0.0013	0.0847
K2O	4.1956	1.2046	5.0540	5.0882	94.2034	0.0537	3.5108
Cs2O		1.0602	0.0000	0.0000	281.8094	0.0000	0.0000
Sb2O3		1.1970	0.0000	0.0000	291.4982	0.0000	0.0000
P2O5	0.0140	2.2910	0.0321	0.0323	141.9370	0.0002	0.0148
Nd2O3		1.1660	0.0000	0.0000	336.4782	0.0000	0.0000
La2O3		1.1728	0.0000	0.0000	325.8100	0.0000	0.0000
Y2O3		1.2699	0.0000	0.0000	225.8082	0.0000	0.0000
BaO	0.0180	1.1165	0.0201	0.0202	153.3394	0.0001	0.0086
PbO	<0.020	1.0772	0.0000	0.0000	223.1900	0.0000	0.0000
CeO2		1.2284	0.0000	0.0000	172.1200	0.0000	0.0000
MoO3		1.5003	0.0000	0.0000	143.9382	0.0000	0.0000
ZnO	0.1550	1.2447	0.1929	0.1942	81.3800	0.0024	0.1551
CuO		1.2518	0.0000	0.0000	79.5454	0.0000	0.0000
H2O	0.1300	1.0000	0.1300	0.1309	18.0000	0.0072	0.4726
SUMS			99.3278	100.0000		1.5281	100.0000