Technical Memorandum

To:	Wayne Praskins, EPA		
From:	Robert Lindfors, P.E., Gilbane Federal		
Date:	02 March 2015		
Subject:	Bench Tests of Secondary Smelter Waste Halaco Engineering Company Superfund Site Oxnard (Ventura County), California		
Contract/TO:	EP-S9-08-03/00052	**Gilbane DCN:**	07163.0053.0026

1.0 INTRODUCTION

Gilbane Federal (Gilbane) prepared this technical memorandum (TM) to describe activities and results related to bench tests conducted on samples of secondary smelter waste collected at the Halaco Engineering Company Superfund Site, Oxnard, Ventura County, California. The tests were conducted as part of the feasibility study (FS) being conducted by EPA to assess technologies that may be appropriate for remediating the site.

This TM is organized as follows: Section 1.0 – Introduction; Section 2.0 – Background; Section 3.0 – Purpose; Section 4.0 –Methods; Section 5.0 – Results; Section 6.0 – Discussion of Results; Section 7.0 – Summary and Conclusions; Section 8.0 – References; Section 9.0 – List of Tables; and Section 10.0 – List of Attachments.

2.0 BACKGROUND

A secondary smelter (scrap metal smelter) operated at the Halaco site from 1965 through 2004, generating a large amount of process waste material. Waste material remains at the site, nearly all of it in a pile estimated to be 700,000 cubic yards (CY) in volume. The pile, known as the Waste Management Unit (WMU), is about 15 acres in area and extends vertically from about 5 feet below grade to a height of about 35 feet. The WMU is adjacent to coastal wetlands near Ormond Beach (Pacific Ocean) in the city of Oxnard, Ventura County, California.

High concentrations of metals including arsenic, barium, chromium, copper, lead, nickel, and zinc have been reported in samples of the WMU waste. High concentrations (up to about 5 percent) of chloride also have been reported, likely related to the use of magnesium chloride, potassium chloride, and sodium chloride as fluxing salts in Halaco's smelting process. EPA is evaluating options for handling the waste, including (1) leaving it in place; or (2) beneficially reusing the material off site, possibly as a feedstock component for kilns that produce Portland cement.

EPA has consulted for several years with Diversified Minerals, Inc. (DMI), Oxnard, California, regarding the reuse option at cement plants. DMI has advised EPA that the alumina content in the WMU material makes it desirable for reuse at cement kilns as a feedstock component. However, the kiln operator identified as the best candidate to accept the material would like to see the maximum chloride content in the waste reduced to no more than 0.5 percent by weight.

Reportedly, the high operating temperatures in rotary- and shaft-type cement kilns (about 1,500 degrees Celsius [ºC]) tend to liberate any chlorine present, whether it is present in the feedstock or in the fuel source, and whether it is in its elemental (chlorine) form or its ionic (chloride) form. The presence of chlorine causes problems both in cement production (including formation of so-called "kiln rings" and pre-heater deposits, both of which can clog the kiln components) and in concrete applications, where high chlorine in cement can increase corrosion of steel reinforcing bars. Another concern regarding chlorine in kilns is the tightening of restrictions being implemented by the regulatory community on air emissions, including emissions of hydrochloric acid, from cement plants.

3.0 PURPOSE

Because the targeted chloride concentration (0.5 percent) is about five times less than the average chloride concentrations detected in samples of the WMU material, EPA desired to investigate methods for reducing the chloride in the waste. The bench tests described herein were intended to evaluate several approaches to removing or reducing chloride from the process waste pile. If a feasible approach was identified, the next step would be to conduct a pilot test using the material as a feedstock at an operating cement kiln. The bench test experiments were designed to identify processes that could be implemented effectively as full-scale operations at the Halaco site.

Gilbane subcontracted with Wyoming Analytical Laboratories (WAL), Golden, Colorado to conduct bench tests with the objective of assessing various approaches to reduce the chlorides present in the waste material. Bench tests conducted at WAL included:

- Mixing the waste with clean water and varying the water-to-waste ratio
- Mixing the waste with clean water and varying the mixing time
- Mixing the waste with clean hot water and varying the mixing time
- Mixing the waste with clean water and detergent and varying the mixing time
- Grinding the waste into a fine powder, mixing with hot water, and varying the mixing time
- Mixing the waste with acid solution

WAL used its X-ray fluorescence (XRF) instrument (Bruker AXS model S4 Explorer) to provide rapid, non-destructive test results for total chlorine (tCl) in both solid- and aqueous-phase fractions. Results were reported as percent by dry weight relative to the initial solid sample. A limitation of the XRF analysis is that the instrument reports tCl, i.e., there is no differentiation between the elemental chlorine atoms possibly held in a mineral matrix and the chloride ions possibly present in the dissolved phase or absorbed on the surfaces of solid particles. However, as previously described, all chlorine in the waste would be liberated in the high kiln temperatures, so the XRF tests reporting tCl are appropriate.

3.1 Sample Collection

EPA coordinated with CH2M Hill to collect the samples from the WMU on 04 October 2012. Samples of process waste were collected from four of 12 WMU locations where EPA had collected samples as part of its remedial investigation (RI; EPA, 2011). The four locations were selected by EPA to represent the range of chloride concentrations reported during the RI. Samples were collected from the upper 4 feet of the WMU waste pile. The samples were assigned the

following names: TXIWMU1, TXIWMU4, TXIWMU9, and TXIWMU12. Attachment 1 includes a figure depicting sample locations on the waste pile along with a table with the associated horizontal coordinates.

The samples were collected using a 4-inch-diameter hand auger, homogenized, and transferred to sample containers per CH2M Hill's standard operating procedures (SOPs) for sample collection and homogenization (also included in Attachment 1). Sample containers were 8-ounce (oz), certified-clean glass jars with Teflon lids. The total sample volume collected at each location was approximately 32 oz. The samples were shipped under chain-of-custody (COC) protocol to WAL overnight, unpreserved, and at ambient temperature. A copy of the COC form is included in Attachment 1.

4.0 METHODS

The bench tests conducted for this study are presented below. The methods are presented as laboratory procedures where appropriate. Where mixtures, ratios, and percent-solutions are indicated, the proportions are by weight.

I. Calibration of the XRF instrument

A. The XRF instrument was used to analyze several certified reference materials (CRMs) containing metal oxides, per WAL's SOP for XRF analysis of cement materials (see Attachment 2). Calibration was performed to render values of accuracy and precision to meet requirements listed in ASTM C114 - *Standard Test Methods for Chemical Analysis of Hydraulic Cement*.

II. Initial characterization of waste samples

A. An initial characterization of each sample of process waste was conducted.

1. Each of four separate samples of process waste was homogenized using a spatula.
2. Each sample was weighed, oven-dried for 12-18 hours at 105ºC, and then re-weighed to derive a value of moisture content (MC). MC was analyzed in accordance with ASTM C311 - *Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete*.
3. Each dried sample was tested for metals using the XRF.
4. Each dried sample was tested for tCl using XRF. Units were derived as percent by dry weight.

III. Chloride removal while varying water-to-waste ratios

A. For three waste samples (sample TXIWMU4 was omitted due to low initial tCl content – see Section 5.1), mixtures with different water-to-waste ratios were prepared for analysis using deionized (DI) water.

1. For each sample, a mixture of 1 part water to 1 part waste was prepared. The mixture was agitated on a shaker table for 15 minutes. The mixture then was

allowed to gravity separate for 30 minutes. The mixture then was decanted, producing a slurry fraction and a supernatant fraction.

2. Each slurry fraction was weighed, oven-dried for 12-18 hours at 105ºC, and tested for MC and for tCl using the procedure described in Step II above, to derive a residual tCl value in percent dry weight.

3. Each supernatant was tested for aqueous tCl using XRF. Back calculations were performed to convert the result to percent dry weight of the initial waste sample (see Section 5.2 for the calculation approach).

4. Steps III.A.1 through III.A.3 were repeated using the following water-to-waste ratios: 2:1, 3:1, 10:1, and 20:1.

IV. Separation of supernatants

A. To evaluate whether gravity separation of the mixtures was possible, the supernatant fractions generated from each batch in Step III.A.1 (except for the 20:1 ratios) were allowed to settle. Turbidity readings were taken at 0, 15, 30, 45, and 60 minutes. Turbidity was analyzed using procedures consistent with *Method 2130-B, Nephelometric Method*, from *Standard Methods for the Examination of Water and Wastewater (17th edition)*, a mutual publication from the American Public Health Association (APHA), American Water Works Association (AWWA), and the Water Environment Federation (WEF). Results were reported in nephelometric turbidity units (NTUs).

V. Chloride removal while varying mixing times at select temperatures

A. Selection of waste sample

1. Sample TXIMU9 was selected for the mixing time experiments because it had the highest initial tCl content, and the 3:1 water-to-waste mixture showed promising tCl removal (see Section 5.2).

B. Assessment of mixing times at ambient temperature (20ºC)

1. A mixture was prepared using a 3:1 water-to-waste ratio (DI water). The mixture was stirred continuously at ambient temperature. Aliquots were drawn from the mixture at 1 hour, 2 hours, and 3 hours and filtered. Each filtrate was analyzed for aqueous tCl.

2. To assess the residual tCl in the waste (solid) material after 3 hours of mixing, a portion of the mixture was decanted and the slurry was weighed, oven-dried for 12-18 hours at 105ºC, and analyzed for MC and tCl.

3. To assess whether filtering would further decrease the residual tCl in the waste material, the other portion of the mixture was filtered using a 200-mesh sieve. The filter cake was oven-dried for 12-18 hours at 105ºC and analyzed for MC and tCl.

C. Assessment of mixing times at 35ºC

1. DI water was heated to 35ºC. A waste mixture was prepared using a 3:1 water-to-waste ratio. The mixture was stirred continuously while the temperature was held at 35ºC. Aliquots were drawn from the mixture after 1 hour, 2 hours, and 3 hours, and filtered. Each filtrate was analyzed for aqueous tCl.
2. At the 3-hour interval, a portion of the mixture was decanted and the slurry was weighed, oven-dried for 12-18 hours at 105ºC, and analyzed for MC and tCl.
3. The remaining mixture was filtered using a 200-mesh sieve. The filter cake was oven-dried for 12-18 hours at 105ºC and analyzed for MC and tCl.

D. Assessment of mixing times at 70ºC

1. DI water was heated to 70ºC. A waste mixture was prepared using a 3:1 water-to-waste ratio. The mixture was stirred continuously while the temperature was held at 70ºC. Aliquots were drawn from the mixture after 1 hour, 2 hours, and 3 hours and filtered. Each filtrate was analyzed for aqueous tCl.
2. At the 3-hour interval, a portion of the mixture was decanted and the slurry was weighed, oven-dried for 12-18 hours at 105ºC, and analyzed for MC and tCl.
3. The remaining mixture was filtered using a 200-mesh sieve. The filter cake was oven-dried for 12-18 hours at 105ºC and analyzed for MC and tCl.

VI. Chloride removal using detergent solution

A. To assess whether mixing with a detergent solution would result in lowering the residual tCl in the waste material, an aqueous solution of detergent was prepared by adding Triton X detergent into DI water (ambient temperature) at 0.1 percent.

B. A mixture was prepared using a 3:1 detergent solution-to-waste ratio. The mixture was stirred continuously at ambient temperature. Aliquots were drawn from the mixture after 1 hour, 2 hours, and 3 hours and filtered. Each filtrate was analyzed for aqueous tCl.

C. At the 3-hour interval, a portion of the mixture was decanted and the slurry fraction was weighed, oven-dried for 12-18 hours at 105ºC, and analyzed for MC and tCl.

D. The remaining mixture was filtered using a 200-mesh sieve. The filter cake was oven-dried and analyzed for MC and tCl.

VII. Chloride removal from ground (pulverized) samples

A. To assess whether grinding the material could improve chloride removal, samples TXIWMU1, TXIWMU9, and TXIWMU12 were analyzed as follows.

1. Each sample was pulverized using a ring-and-puck mill so that all material would pass the 200-mesh sieve.
2. For each sample 70ºC DI water was added to form a mixture of 20 parts water to 1 part waste.
3. Each mixture was stirred continuously while a temperature of 70ºC was maintained.

4. Aliquots were drawn from each mixture after 3 hours, 4 hours, and 72 hours, and filtered. Each filtrate was analyzed for aqueous tCl.
5. After 72 hours, each mixture was filtered using a 200-mesh sieve. The filter cake was oven-dried for 12-18 hours at 105ºC and analyzed for MC and tCl.

VIII. Chloride removal using nitric acid solution

A. DI water was heated to 70ºC. A waste mixture was prepared using a 5:1 water-to-waste ratio using sample TXIWMU9. The mixture was stirred continuously for 24 hours while the temperature was held at 70ºC.

B. The mixture then was allowed to gravity separate for 30 minutes. The mixture then was decanted to form a supernatant fraction and a slurry fraction. The supernatant fraction was allowed to settle, and an aqueous-phase tCl measurement was recorded. The slurry fraction was filtered and washed with DI water. This material was oven-dried for 12-18 hours at 105ºC.

C. A solution of 1-percent nitric acid was prepared using DI water. A mixture was then prepared using 3 parts acid solution to 1 part dried washed material. The mixture was stirred for 1 hour.

D. The mixture then was allowed to gravity separate for 30 minutes, and then was decanted to form a supernatant fraction and a slurry fraction. The slurry fraction was weighed, oven-dried for 12-18 hours at 105ºC, and analyzed for tCl.

E. To assess the types and relative amounts of cations and anions remaining in the supernatant fraction (which would possibly require treatment and disposal during full-scale waste processing), the supernatants from Steps VII.B and VII.D were each allowed to evaporate. The XRF was used to derive the composition of the solid material that remained in each supernatant sample after evaporation.

5.0 RESULTS

5.1 Initial Characterization

Visual inspection indicated that samples TXIWMU1, TXIWMU9, and TXIWMU12 appeared as grey silty sands. Sample TXIWMU4 appeared as a brown silty sand. Table 1 shows the mineral results for all samples as reported by the XRF machine. For purposes of this analysis, all metals were reported as oxides.

Except for TXIWMU4, the composition of metals in the samples featured high amounts of aluminum oxide (greater than 35 percent) and magnesium oxide (about 16 percent or greater). Silica oxide was present in all samples, and exceeded 50 percent in sample TXIWMU4.

Table 2 presents the results of initial characterization for MC and tCl. MC values ranged from 15.7 percent to 27.1 percent. Values of tCl for samples TXIWMU1, TXIWMU9, and TXIWMU12 were approximately 3 percent. The tCl value for TXIWMU4 was 0.17 percent. Because samples TXIWMU1, TXIWMU9, and TXIWMU12 appear to be more representative of the bulk of the waste material, they were retained as the samples of interest for subsequent experiments, and sample TXIWMU4 was dropped from consideration.

5.2 Water-to-Waste Ratios

As described in Section 4.0, samples TXIWMU1, TXIWMU9, and TXIWMU12 were each subjected to water-to-waste ratios of 1:1, 2:1, 3:1, 10:1, and 20:1, with a mixing time of 15 minutes. The following observations were made during these experiments:

- After settling and decanting the waste/water mixtures into slurry and supernatant fractions, the slurry fractions still had substantial moisture, referred to as "extract liquid".
- When the slurry was dried, the tCl in the moisture/porewater redeposited onto the solid fraction.

The following formula was used to calculate the amount of redeposited tCl.

$$Redeposited\ tCl = \frac{MC}{100 - MC} \times \frac{Aqueous\ tCl}{ER}$$

Where: *ER = extract ratio = dilution factor*
MC = moisture content of the slurry
Aqueous tCl = total chlorine (percent) measured in the supernatant

Note: *The aqueous tCl concentration in the extract solution was assumed to be equal to the measured tCl value in the supernatant.*

Results are presented in Table 3. The "residual tCl" measurement reflects the tCl that remained in the solid fraction after decanting, plus the "redeposited tCl" from the extract solution. As expected, increases in the water ratio resulted in increases in tCl removed from the sample. However, the residual tCl for the 20:1 mixtures was 1.82 percent (averaged for the three samples), well in excess of the desired threshold value of 0.5 percent. The results of the various water-to-waste experiments indicated that significant increases in tCl removal would not be attained by dilution ratios higher than 20:1.

5.3 Settling Times in Supernatant

All supernatants produced by the decanting process in the water-to-waste experiments appeared cloudy, indicating the presence of suspended solids. For water-to-waste ratios other than the 2:1 ratio, only slight decreases in turbidity values were noticed during the 1-hour duration of the tests (see Table 4). For the 2:1 ratios of samples TXIWMU1, TXIWMU9, and TXIWMU12 , the NTU values after 1 hour declined by factors of 45 percent, 56 percent, and 64 percent, respectively.

5.4 Temperature Experiments on Sample TXIWMU9

Sample TXIWMU9 was selected for temperature-variation experiments because of its high initial tCl content (>3 percent; see Table 2). A water-to-waste ratio of 3:1 was selected as the mix for the temperature experiments, because that ratio was somewhat effective in removing tCl (see Table 3), and could be reasonably implementable in a full-scale application. Table 5 indicates the test results. As indicated, mixing at higher temperatures had little or no effect on elevating the tCl values in the aqueous fraction, and, correspondingly, no effect on reducing the residual tCl in the solid portion of the waste. Residual tCl of the solid (dried slurry) portion of the sample after mixing for 3 hours at 70ºC was 1.94 percent dry weight.

5.5 Detergent Effects

Mixing sample TXIWMU9 with detergent (1 percent Triton X) solution at a ratio of 3 parts solution to 1 part waste (at ambient temperature) showed virtually no effect on aqueous tCl values compared to the values seen in the non-detergent mixture. Overall residual tCl on the solid (dried slurry) portion of the sample after mixing for 3 hours at ambient temperature was 2.08 percent dry weight (see Table 5).

5.6 Tests on Ground (Pulverized) Samples

After 1 hour of mixing at 70ºC, the values of residual tCl of mixtures formed with pulverized samples (in weight percentages) were 1.13, 0.91, and 0.96 for samples TXIWMU1, TXIWMU9, and TXIWMU12, respectively. Increasing the mixing times to 3, 4, and 72 hours had no appreciable effect on tCl values (see Table 6).

5.7 Tests of Acidification on Sample TXIWMU9

The acidification test was carried out on the slurry generated by first water-washing a portion of sample TXIWMU9 for 24 hours at 70ºC, using a water-to-waste ratio of 5:1. The dried solid material generated from the water-only step was used to prepare a mixture of 3:1 water-to-waste using 1-percent nitric acid. After 1 hour of stirring, the tCl value of the resulting dried slurry was 0.64 percent.

Analysis of the solid residue that remained after evaporating the supernatant of the water-only mixture indicated a mix of chloride salts of calcium, potassium, magnesium, and sodium. Analysis of the solid residue that remained after evaporating the supernatant of the acidified mixture indicated a mixture of nitrogen-containing salts with some chlorine. Table 8 lists the chemical species with corresponding weight percentages in the residue, as reported by WAL.

6.0 DISCUSSION OF RESULTS

The initial characterization of the WMU samples using XRF for metals indicated aluminum as the metal with the highest concentration and magnesium with the second highest (see Table 1) – this trend is consistent with the metals results reported for the RI. The initial characterization of three of four WMU samples for chlorine yielded tCl values around 3 percent (see Table 2), too high for the material to be used as a feedstock in cement plants. The fourth sample, TXIWMU4, differed from the other three samples (TXIWMU1, TXIWMU9, and TXIWMU12) in physical appearance, metals concentrations, and tCl content. This indicates that the WMU pile may be somewhat heterogeneous. Samples TXIWMU1, TXIWMU9, and TXIWMU12 are believed to be more representative of the waste pile.

Mixing the waste samples with water was somewhat effective in removing tCl, but the residual tCl (chlorine remaining in the solid waste material after mixing, separating, and drying), even for the 20:1 mixing ratio, yielded an average tCl of 1.82 percent, well above the kiln-desired threshold of 0.5 percent (see Table 3). The 20:1 water-to-waste ratio likely represents a feasible limit for any full-scale application for treating 700,000 CY of WMU material.

In assessing settling times of the waste/water mixes, NTU values did decrease somewhat for each of the three samples for all mix ratios tested after 1 hour (see Table 4). However, all supernatants remained turbid, likely indicating a significant amount of suspended solids. The presence of

suspended solids in the aqueous fraction presents a waste management issue in a full-scale site application, in that some type of active filtration or other separation technique would probably be required to achieve waste discharge requirements (WDRs) for suspended solids.

Results of tests using hot water in the waste mixture for sample TXIWMU9 indicated that temperatures had only a modest effect on removing tCl from the solid (slurry) fractions (see Table 5). The residual tCl value after mixing at 70ºC for 3 hours was 1.94 percent, still well above the kiln threshold value of 0.5 percent. The energy required to heat waste mixtures in a full-scale application likely would add significantly to the cost of treatment. Similarly, using detergent solution in the water/waste mixture indicated no improvement in removing chlorine from the waste sample. This result is not surprising, in that detergents generally have little or no effect on solubilizing metals.

Results of testing on three mixtures using pulverized samples at a 20:1 water-to-waste ratio (water at 70ºC) indicated that the average aqueous tCl concentrations reached as high as 1.98 percent after mixing for 3 hours. This value corresponds to an average estimated residual tCl in the waste of 0.89 percent, still above the kiln threshold (Table 6). Longer mixing times did not increase the aqueous tCl, and did not decrease the residual tCl.

Treating sample TCIWMU9 by mixing with hot (70ºC) water for 24 hours at a 5:1 water-to-waste ratio, settling, filtering, drying, then acidifying (mixing with 1-percent nitric acid at a ratio of 3:1 for 1 hour) was successful in removing chloride from the solid matrix down to 0.64 percent, the lowest value of any treatment attempted during this study, but still exceeding the kiln threshold of 0.5 percent (see Table 7). The necessity of heating the water could be re-evaluated if the acidification option appeared to be feasible.

The chemical compositions of the dissolved species in the supernatants remaining after the hot-water step and the acidification experiment were assessed by allowing the supernatants to evaporate, then using XRF on the residual solids. Analysis of the supernatant from the water-only step indicated the presence of chloride salts with the following cations: calcium, magnesium, potassium and sodium (Table 8). Analysis of the supernatant from the acidification experiment indicated the presence of nitrate salts, with magnesium being the prevalent cation. Though unreported by the XRF analysis, species containing arsenic, lead, and zinc likely were present in the residual solids, taking into consideration the origin of the process waste (i.e., secondary smelter operations), and the reported results from the RI (EPA, 2011). These species would be likely to present considerable challenges for handling the liquid wastes that would be generated during a full-scale site application.

7.0 SUMMARY AND CONCLUSIONS

Bench tests were conducted to assess the possible reuse of WMU material as a source of alumina in the manufacture of Portland cement. Because the targeted chloride concentration of 0.5 percent is about five times less than the average chloride concentrations measured in samples of the WMU material, the bench tests evaluated several possible approaches to remove chloride.

Experiments were performed using WMU samples that contained tCl (total chloride concentration) at about 3 percent. These experiments included mixing with water, mixing with hot water, mixing with detergent, and mixing with hot water followed by acidification.

None of these experiments was successful in reducing tCl to the kiln-desired threshold of 0.5 percent. The treatment approach that achieved the lowest tCl consisted of mixing the waste with hot (70ºC) water at a ratio of 5:1 for 24 hours, followed by acidification (mixing with 1 percent nitric acid at a ratio of 3:1 for 1 hour). This approach reduced the tCl concentration to about 0.6 percent.

The experiment in which waste samples were pulverized then heated and washed achieved the next best result, reducing the tCl in the waste to about 0.9 percent. The experiments in which the waste samples were washed at ambient temperature without acidification or pulverization reduced tCl in the wastes to slightly less than 2 percent.

The logistics of heating, pulverizing, and/or acidifying the wastes in a full-scale application would be complex and cumbersome, generating significant waste streams that would also require treatment and handling. Based on the bench test results, even the most promising option for removing chloride – hot water washing followed by acidification – would likely rank low in effectiveness and in implementability, if formally evaluated as a Superfund remedy.

Because the targeted chloride level of 0.5 percent was not achieved, use of the waste in the manufacture of Portland cement would probably require blending the Halaco waste with other (low chlorine) feedstock materials to reduce the overall chlorine input into the kiln, or identifying a kiln less sensitive to chlorine. No other qualifying kilns have been identified in California, but efforts are underway to identify an out-of-state kiln less sensitive to chloride.

8.0 REFERENCES

United States Environmental Protection Agency (EPA), 2011. *Halaco Superfund Site: EPA testing results for the Halaco properties in Oxnard, California*. 29 August.

9.0 LIST OF TABLES

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2 Test Results for Initial Characterization – Moisture Content and Total Chlorine
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10.0 LIST OF ATTACHMENTS

1 Sampling Information
2 WAL's SOP for XRF Analysis (based on ASTM C114)

TABLES

Table 1 - Test Results for Initial Characterization - Metals
Bench Tests on Secondary Smelter Waste
Halaco Engineering Company Superfund Site
Oxnard, California

Al_2O_3	BaO	CaO	Fe_2O_3	K_2O	MgO	MnO_2	Na_2O	P_2O_5	SO_3	SiO_2	SrO	TiO_2	Sum
Sample TXIWMU1													
35.3	0.525	1.05	1.83	0.982	15.9	0.482	0.930	0.0517	0.388	17.8	0.0345	0.637	100
Sample TXIWMU4													
18.0	0.145	3.66	3.87	2.67	4.73	0.236	2.26	0.191	0.218	53.3	0.0455	0.600	100
Sample TXIWMU9													
35.9	0.622	1.14	1.43	0.822	19.4	0.683	0.837	0.0538	0.330	10.3	0.0307	0.637	100
Sample TXIWMU12													
37.2	0.782	1.05	1.00	0.662	20.3	0.583	0.768	0.0498	0.398	7.69	0.0285	0.754	100

Abbreviations

@ - at

ºC - Degrees Celsius

Notes

1. All results are in units of percent by dry weight.
2. The XRF instrument reports concentrations of total metals regardless of speciation. It is assumed that the metals are in the form of oxides.

Table 2 - Test Results for Initial Characterization - Moisture Content and Total Chlorine Bench Tests on Secondary Smelter Waste
Halaco Engineering Company Superfund Site
Oxnard, California

Material description	*MC* (%)	*tCl* (wt %)
Sample TXIWMU1		
Grey silty sand	16.6	2.67
Sample TXIWMU4		
Brown silty sand	15.7	0.173
Sample TXIWMU9		
Grey silty sand	27.1	3.01
Sample TXIWMU12		
Grey silty sand	27.1	2.91

Abbreviations

MC - Moisture content
% - Percent weight
tCl - Total chlorine
wt % - Percent dry weight
XRF - X-ray fluorescence

Notes

1. tCl is the direct measurement from the XRF instrument on the dried material.

Table 3 - Test Results for Water/Waste Mixtures
Bench Tests on Secondary Smelter Waste
Halaco Engineering Company Superfund Site
Oxnard, California

Ratio (1)	*Aqueous tCl* (wt %) (2)	*Slurry MC* (%) (3)	*Total solids* (%) (4)	*Redeposited tCl* (wt %) (5)	*Residual tCl* (wt %) (6)	*Net tCl removed* (wt %) (7)	*tCl mass balance check* (wt %) (8)	*Overall tCl recovered* (wt %) (9)
Sample TXIWMU1 (initial tCl = 2.67 wt %)								
1:1	0.760	38.9	61.1	0.484	2.19	0.276	2.47	92.5
2:1	0.837	47.2	52.8	0.374	2.21	0.463	2.67	100
3:1	0.829	42.7	57.3	0.206	2.26	0.623	2.88	108
10:1	1.27	46.8	53.2	0.111	1.85	1.15	3.00	113
20:1	1.48	44.5	55.5	0.059	1.66	1.42	3.09	116
Sample TXIWMU9 (initial tCl = 3.01 wt %)								
1:1	0.862	48.1	51.9	0.799	2.52	0.0631	2.58	85.8
2:1	0.953	49.2	50.8	0.461	2.40	0.492	2.89	96.0
3:1	0.974	51.1	48.9	0.339	2.63	0.635	3.26	108
10:1	1.35	51.8	48.2	0.146	1.99	1.21	3.20	106
20:1	1.44	55.6	44.4	0.090	1.87	1.35	3.22	107
Sample TXIWMU12 (initial tCl = 2.91 wt %)								
1:1	0.938	43.5	56.5	0.722	2.51	0.216	2.73	93.9
2:1	1.07	51.9	48.1	0.575	2.50	0.490	2.99	103
3:1	1.07	49.1	50.9	0.343	2.40	0.724	3.12	107
10:1	1.53	48.7	51.3	0.145	2.00	1.38	3.38	116
20:1	1.77	51.0	49.0	0.0920	1.93	1.68	3.61	124

Abbreviations

MC - Moisture content
% - Percent weight
tCl - Total chlorine
wt % - Percent dry weight
XRF - X-ray fluorescence

Notes

1. Aqueous tCl (Column 2) is the direct measurement from the XRF machine on the aqueous (supernatant) fraction.
2. Redeposited tCl (Column 5) is the chlorine that was solubilized during agitation with water, but redeposited onto the solid phase during drying.
3. Residual tCl (Column 6) is the direct measurement from the XRF machine on the solid dried slurry fraction.

Table 4 - Turbidity Results for Supernatants from Water/Waste Mixtures
Bench Tests on Secondary Smelter Waste
Halaco Engineering Company Superfund Site
Oxnard, California

Water-to-waste ratio **(1)**	***T = 0 min*** **(2)**	***T = 15 min*** **(3)**	***T = 30 min*** **(4)**	***T = 45 min*** **(5)**	***T = 60 min*** **(6)**
Sample TXIWMU1					
1:1	NA	210	170	170	170
2:1	120	110	90.0	76.0	66.0
3:1	93.0	89.0	85.0	120	77.0
10:1	150	145	130	125	120
Sample TXIWMU9					
1:1	320	390	360	300	250
2:1	77.0	63.0	52.0	42.0	34.0
3:1	54.0	45.0	43.0	62.0	46.0
10:1	56.0	55.0	47.0	45.0	42.0
Sample TXIWMU12					
1:1	NA	NA	NA	NA	NA
2:1	120	100	80.0	52.0	43.0
3:1	50.0	43.0	43.0	68.0	51.0
10:1	130	120	110	96.0	96.0

Abbreviations

min - Minutes
NA - Not available (insufficient fluid to obtain a turbidity reading).
T - Settling time

Notes

1. All results are in nephelometric turbidity untis (NTUs).
2. Turbidity measurements were not conducted on supernatants from the 20:1 water-to-waste ratio.

Table 5 - Results for Temperature Variation and Detergent Tests on Sample TXIWMU9
Bench Tests on Secondary Smelter Waste
Halaco Engineering Company Superfund Site
Oxnard, California

Aqueous tCl (wt %)			*Fraction retained on 200 sieve (after T = 3 hrs)*					
T = 1 hr (1)	*T = 2 hr* (2)	*T = 3 hr* (3)	*Total solids (%)* (4)	*MC (%)* (4)	*Redeposited tCl* (wt %) (5)	*Residual tCl* (wt %) (6)	*Net tCl removed* (wt %) (7)	*tCl mass total* (wt %) (8)
Temperature = 20ºC								
1.28	1.32	1.39	64.4	64.2	0.580	2.02	0.815	2.83
Temperature = 35ºC								
1.45	1.57	1.71	57.8	52.3	0.435	2.06	1.28	3.34
Temperature = 70ºC								
1.58	1.37	1.37	52.0	55.2	0.390	1.94	0.976	2.92
1 percent Triton X solution (at ambient temperature)								
1.22	1.28	1.34	62.6	58.9	0.445	2.08	0.893	2.97

Abbreviations

ºC - Degrees Celsius
hr - Hour(s)
MC - Moisture content
T - Mixing time
tCl - Total chlorine
wt % - Percent dry weight
% - Percent weight
XRF - X-ray fluorescence

Notes

1. The mixture used for these tests was sample TXIWMU9 with a water-to-waste ratio of 3:1.
2. The MC of sample TXIWMU9 was measured to be 30.45 percent.
3. Redeposited tCl (Column 7) represents the chlorine that was solubilized during agitation with water, but redeposited onto the solid phase during drying.
4. Residual tCl (Column 8) is the direct measurement from the XRF instrument on the dried waste sample.

Table 6 - Test Results of Extracts Prepared from Pulverized Samples and Water at 70ºC
Bench Tests on Secondary Smelter Waste
Halaco Engineering Company Superfund Site
Oxnard, California

T = 1 hr		T = 3 hr		T = 4 hr		T = 72 hr	
Aqueous tCl (wt %)	***Residual tCl (wt %)***	***Aqueous tCl* (wt %)**	***Residual tCl (wt %)***	***Aqueous tCl* (wt %)**	***Residual tCl (wt %)***	***Aqueous tCl* (wt %)**	***Residual tCl (wt %)***
Sample TXIWMU1							
1.54	1.13	1.68	0.99	1.65	1.02	1.65	1.02
Sample TXIWMU9							
2.10	0.91	2.05	0.96	1.96	1.05	1.96	1.05
Sample TXIWMU12							
1.95	0.96	2.20	0.71	2.17	0.74	1.90	1.01
Average values							
1.86	**1.00**	**1.98**	**0.89**	**1.93**	**0.94**	**1.84**	**1.03**

Abbreviations

ºC - Degrees Celsius
hr - Hour(s)
T - Mixing time
tCl - Total chlorine
wt % - Percent dry weight
XRF - X-ray fluorescence

Notes

1. Each mixture was prepared using a water-to-waste ratio of 20:1 with 70ºC water.
2. Aqueous tCl is the direct measurement from the XRF instrument on a water sample.
3. Residual tCl is the result derived by subtracting the aqueous tCl from the original tCl of each sample (Table 2).

Table 7 - Test Results of Acidification Experiment
Bench Tests on Secondary Smelter Waste
Halaco Engineering Company Superfund Site
Oxnard, California

	***Aqueous tCl at T = 24 hrs* (wt %), water-only test**	***Aqueous tCl at T = 1 hr* (wt %), acid test**	***Residual tCl* (wt %)**	***Net tCl* (wt %)**
Sample TXIWMU9				
	1.63	0.82	0.64	3.09

Abbreviations

hr- Hour(s)
T - Mixing time
tCl - Total chlorine
wt % - Percent dry weight
ºC - Degrees Celsius
XRF - X-ray fluorescence

Notes

1. Aqueous tCl is the direct measurement from the XRF instrument on the aqueous fraction.
2. For the water-only test, a mixture of 5:1 water-to-waste ratio was prepared using 70ºC water.
3. For the acid test, the mixture tested was 3 parts 1% nitric acid to 1 part dried, retained solids from the water-only test.
4. Residual tCl is the direct measurement from the XRF machine on dried material.

Table 8 - Test Results of Residue from Supernatants
Bench Tests on Secondary Smelter Waste
Halaco Engineering Company Superfund Site
Oxnard, California

Chemical	*Weight percent in solid residue*
Water-to-waste ratio = 5:1, temp = 70ºC, T = 1 hr	
$CaCl_2$	27.2
KCl	20.0
$MgCl_2$	18.5
NaCl	33.3
Total	***99.0***
1% nitric acid solution, solution-to-waste ratio = 3:1, temp = 20ºC, T = 1 hr	
$CaCl_2$	0.3
$Ca(NO_3)_2$	6.3
$MgCl_2$	4.7
$Mg(NO_3)_2$	89.6
Total	***100.9***

Abbreviations

ºC - Degrees Celsius
hr - Hour(s)
T - Mixing time
temp - Temperature

ATTACHMENT 1
SAMPLING INFORMATION

STANDARD OPERATING PROCEDURE 6-6

Surface Soil/Hand-auger Soil and Sediment Sampling

Purpose and Scope

This Standard Operating Procedure (SOP) provides the Equipment, Materials, Procedures, and Guidelines to collect surface and shallow subsurface soil, sediment, and waste samples using surface scoops and hand-augering methods. Surface and shallow subsurface soil, sediment, and waste samples will be collected from most study areas, including the following:

- Smelter Area
- Waste Management Area
- Nature Conservancy Land
- Hueneme Parcel
- Loading Dock Area
- Wetland Area
- Beach Dunes Area

The surface and shallow subsurface sampling may be performed by a subcontractor. The procedures in this SOP do not replace more detailed specifications developed or provided by the subcontractor. The subcontractor's procedures will be reviewed for consistency with this SOP before implementation of the work.

References

Field Sampling Plan		
Section	6.1.4	Task 4 – Solid Matrix Sampling
Table	5-3	Request for Analysis, Solid Matrix Samples
Figures	A-1	Solid Matrix Sample Locations, Overview
	A-2	Solid Matrix Sample Locations, Smelter Parcel
	A-3	Solid Matrix Sample Locations, Waste Management Area
	A-4	Solid Matrix Sample Locations, Ditch South of WMU
	A-5	Solid Matrix Sample Locations, NCL East
	A-6	Solid Matrix Sample Locations, NCL North
Appendix	B	GPS Coordinates for Sample Locations

Equipment and Materials

- Stainless steel or disposable plastic scoops that can be used to remove soil from the ground and shallow subsurface.

- Stainless steel hand auger that can be used to remove soil from the ground and shallow subsurface.
- Pre-cleaned sample containers
- Clean nitrile gloves
- Sample coolers and ice
- Log book

Procedures and Guidelines

- Use a decontaminated stainless-steel scoop/trowel to scrape away surficial organic material (grass, leaves, etc.).
- Obtain the subsurface soil sample using an auger down to the appropriate depth. To facilitate a hand-auger sample collection, measure the length of the auger bucket and place tape at the appropriate intervals on the hand-auger extensions.
- Empty the contents of the scoop/trowel into a decontaminated stainless-steel pan.
- Repeat this procedure until sufficient soil is collected to meet volume requirements. Homogenize the soil sample as described in SOP 6-7: *Homogenization of Soil Samples*.
- Homogenize cuttings in the pan using a disposable scoop as specified in SOP 6-7: *Homogenization of Soil Samples.*
- Transfer sample for analysis into appropriate containers with the disposable scoop used for homogenization.
- Prepare samples for laboratory submission. Sample IDs, types, sampling area, and depth are presented in Section 5 of the FSP.

Decontamination

Non-disposable sampling equipment will be decontaminated before and after use. Decontamination procedures are detailed in SOP 6-24: *Decontamination of Sampling Equipment*.

Investigation Derived Waste Decontamination

Soil and water generated during sampling will be contained and managed as detailed in Section 6.4 of the FSP.

Documentation

The field documentation requirements will include recording all observations made during field activities to document the work and potential issues that could affect the quality of field data. The documentation should be entered in a field logbook with consecutively

numbered pages and/or the data collection forms. Documentation should include at a minimum:

- Work/sample location
- Date of field activity
- Personnel on-site
- Time of day
- PPE level
- Field methods and procedures
- Field readings and measurements
- Field observations and comments
- Unusual conditions
- Decontamination procedures
- Investigation derived waste procedures

Attachments

None.

STANDARD OPERATING PROCEDURE 6-7

Homogenization of Soil Samples

Purpose and Scope

This Standard Operating Procedure (SOP) provides the Equipment, Materials, Procedures, and Guidelines for the homogenization of soil samples. The homogenization of soil is performed to minimize bias of sample representativeness introduced by the natural stratification of constituents within the sample and to collect a representative soil samples for sampling interval. Soil will be composited and homogenized to yield one sample for each depth and each area where direct push sampling techniques are not feasible.

References

Refer to SOP 6-5: *Direct Push Soil Sample Collection* and SOP 6-6: *Surface Soil/Hand Auger Sampling* for instructions on sample collection.

Equipment and Materials

- Sample containers
- Stainless-steel spoons or spatulas
- Stainless-steel pans

Procedures and Guidelines

Soil and sediment samples should be homogenized in the field. After a sample is taken, a dedicated disposable scoop should be used to remove the sample from the auger. The sampler should not use fingers to do this, as gloves may introduce organic interferences into the sample.

Prior to homogenizing the soil samples, any rocks, stems, tree roots, seeds, or other debris should be removed from the sample. The sample should be placed in a decontaminated stainless-steel pan and thoroughly mixed using a dedicated disposable scoop. The soil or sediment material in the pan should be scraped from the sides, corners, and bottom, rolled into the middle of the pan, and initially mixed. Cohesive soil should be chopped to reduce the size of the chunks. The sample should be thoroughly mixed until it appears to be uniform color, texture, and particle size. The sample should then be quartered and moved to the four corners of the pan. Each quarter of the sample should be mixed individually and then rolled to the center of the pan and mixed with the entire sample again. The sample should be collected from the pan by taking small quantities of soil from quadrants around the pan. If more than one container is to be filled, the soil should be remixed and sampled from quadrants around the pan until the next container is filled. Continue until the required volume of soil has been collected.

TABLE 1 - XY COORDINATES FOR WMU SAMPLING LOCATIONS - 04 OCTOBER 2012				
SAMPLE ID	***POINT_X***	***POINT_Y***	***COORDINATE_SYSTEM***	***COORDINATE_UNIT***
TXI-WMU-1	298913.9127	3779598.0099	NAD_1983_UTM_Zone_11N	METERS
TXI-WMU-4	298991.4300	3779589.4832	NAD_1983_UTM_Zone_11N	METERS
TXI-WMU-9	298903.4617	3779540.5757	NAD_1983_UTM_Zone_11N	METERS
TXI-WMU-12	298987.0259	3779531.5549	NAD_1983_UTM_Zone_11N	METERS

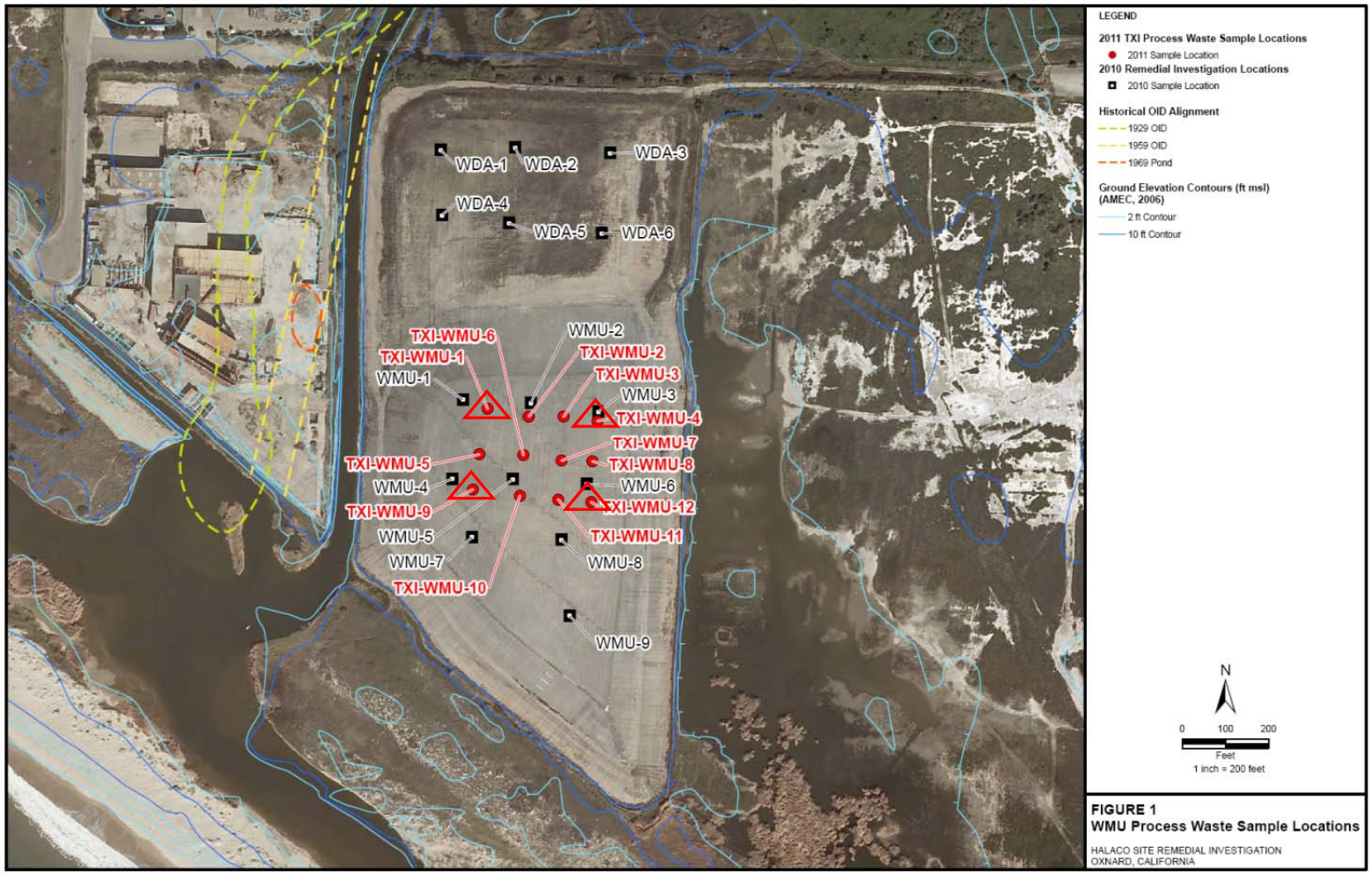

Figure 1. Sampling Locations 04 October 2012 (TXI-WMU 1, 4, 9, 12, shown as red triangles)

CH2MHILL Applied Sciences Lab
CHAIN OF CUSTODY RECORD
AND AGREEMENT TO PERFORM SERVICES

CVO 2300 NW Walnut Boulevard
Corvallis, OR 97330-3638
(541) 752-4271 FAX (541) 752-0276

COC # _____

Project #	Purchase Order #
Project Name: Hanaco	
Company Name: CH2M HILL	
Project Manager or Contact & Phone #: 805-371-7817 x 3803 Mark Watkins	Report Copy to:
Requested Completion Date:	Site ID:
Sample Disposal: Dispose ☑ Return ☐	

Sampling Date	Sampling Time	Type COMP	Type GRAB	Matrix WATER	Matrix SOIL	Matrix AIR	CLIENT SAMPLE ID (9 CHARACTERS)	LAB QC	TOTAL # OF CONTAINERS	Requested Analytical Method # (Chloride transmissivity Study EPA/17 Brown)	Preservative (NONE)									THIS AREA FOR LAB USE ONLY: Lab # / Page / of	EPA Tier QC Level (1 (Screening) 2 3 4)	Alternate Description	Lab ID
10/4/12	1040	X			X		TXIWMU4		1	X							brown silty sand						
	1120	X			X		TXIWMU12		1	X							gray silty sand						
	1145	X			X		TXIWMU9		1	X							gray silty sand						
10/4/12	1250	X			X		TXIWMU1		1	X							gray silty sand						

10/4/12

Relinquished By	Date/Time	Received By	Date/Time
Elizabeth [illegible]		FedEx Airbill # 800978562613	
Sampled By and Title (Please sign and print name)	Date/Time	Relinquished By (Please sign and print name)	Date/Time
Received By (Please sign and print name)	Date/Time	Relinquished By (Please sign and print name)	Date/Time
Received By (Please sign and print name)	Date/Time	Shipped Via: UPS Fed-Ex Other _____	Shipping # 800978562613

Special Instructions:

Instructions and Agreement Provisions on Reverse Side

DISTRIBUTION: Original - LAB, Yellow - LAB, Pink - Client
REV 09/2006 LAB FORM 340

ATTACHMENT 2
WAL'S SOP FOR XRF ANALYSIS (BASED ON ASTM C114)

Standard Operating Procedure

ASTM C114 - Standard Test Methods for Chemical Analysis of Hydraulic Cement

Copy Approved by: _____ Date:

Document Number: SOP 01

Distributed to:_____ Date

Copy #: _____

1. Procedure Section

1.1. Scope and Applicability

This standard operating procedure (SOP) details the chemical analysis of cements.

1.2. Summary of Method

This method details the chemical analysis of cement by X-ray fluorescence (XRF), determination of loss on ignition (LOI), and insoluble residue.

2. Definitions

- LOI - weight loss due to ignition at 1,000° C.
- Insoluble residue - the portion of cement that will not dissolve in acid or caustic solution.
- Ratio pellet - sample containing all elements in a program, used to monitor and correct changes in intensities.

3. Health and Safety Warnings

- Be aware that the acids and caustic solutions used in this procedure can cause serious burns.
- Very high temperatures are used in the preparation method.

4. Cautions

5. Interferences

6. Personnel Qualifications, Training and Proficiency

- All trained technicians can perform LOI, insoluble residue, and fusion preparation.
- X-ray technician must be responsible for chemical analysis and data processing.
- Training will consist of each section of this SOP and referenced SOPs
- Proficiency verification will consist of preparing and analyzing in duplicate a certified reference material (CRM) to the precision and accuracy requirements listed in ASTM C114.

7. Apparatus and Materials

- Analytical balance capable of reading 0.0001 g
- Ceramic crucible
- Gooch crucible
- Vacuum filter flask
- Ashless filter paper 2.4 cm
- 25% hydrochloric acid, (HCl) solution
- 10 g/L sodium hydroxide,(NaOH) solution
- 200 ppm lithium solution, 1.98 g. Lithium Borates 67-33 flux per 1,000 ml DI water (acidic solution)
- Methyl Red Indicator, 0.1 g per 50 ml methanol
- CCRL, NIST, or other CRM

8. Instrument or Method Calibration

- WDX-XRF calibrated by the senior X-ray technician. No change can be made to this instrument by anyone else. The calibration of this instrument requires multiple standards.
- Calibrate the flame photometer by the procedure defined in Sections 11.21-11.24.

9. Sample Collection

Cement is a very finely ground substance and can be treated as homogeneous.

10. Handling and Preservation

Samples should remain inside their original container until used, unless container has been compromised in shipping. No special preservation is required.

11. Sample Preparation and Analysis

11.1. Chemical analysis: each sample will be prepared in duplicate on an as received basis as outlined in the *Preparation of Fusion Pellets for Analysis by XRF* procedure.

11.2. A minimum of two standards will be prepared with each run. Standard pellets may be reused in concurrent runs, but new ones should be prepared each week as needed.

11.3. LOI: Make sure balance is zeroed. Weigh an empty ceramic crucible[1]. Record its weight and ID on log sheet or supplemental worksheet. Add 3-5 g of cement and record total initial weight (cement + crucible).

11.4. Place crucible in 950° C furnace for 30 minutes. Remove, and allow to cool in a desiccator.

11.5. Check that balance is zeroed. Weigh the ignited crucible and record the total ignited weight.

11.6. Percent LOI = (total initial weight - total ignited weight) / (crucible weight - total initial weight) * 100.

11.7. Insoluble Residue: Weigh out 1.000 g. of cement. Place in 250 ml beaker with 20 ml DI water and stir, being sure to break up all clumps. Add 25 ml of 25% HCl. Stir to break up any clumps.

11.8. Put on low heat for 15 minutes.

11.9. If determining Na and K by Flame Photometry, transfer analytically into a 100 ml volumetric flask and bring to volume. Remove a 5 ml aliquot for Na & K and place this into a 15 ml plastic vial and set aside. This will be used in step 11.18.

11.10. Weigh a pre-ignited Gooch crucible (in furnace for 10 minutes) and record weight and ID on log sheet or supplemental worksheet. Place an ashless filter paper into the crucible, and put on vacuum filter flask set-up.

11.11. Add some distilled water to seat filter. Pour sample from the volumetric flask or beaker. Try not to disturb the sediment on the bottom until the last pouring.

11.12. Rinse flask with DI water to get all particles out and into crucible. Transfer filter to a clean beaker, use spatula to dig out filter paper. Rinse tip of the spatula & Gooch with DI water into beaker[5].

11.13. Number the beaker the same as crucible. Add 25 ml of 10 g/L NaOH. Place beaker in microwave for 15 seconds per beaker.

11.14. Add 2-3 drops of Methyl Red Indicator, and 25% HCl until yellow goes to pink then add an extra squirt of acid.

11.15. Place a new filter paper in same crucible -wet with DI water and filter the liquid, include the earlier filter paper. Rinse with DI water.

11.16. Place crucible into furnace at 750°C for 15 minutes to burn off filter paper. Allow crucible to cool in desiccator. Weigh back crucible and record weight on log sheet or supplemental worksheet.

11.17. Percent insoluble residue = weight gain in grams * 100.

11.18. Determining sodium and potassium: To the 5 ml aliquot taken from volumetric flask in step 11.9, add 5 ml, of 100 ppm lithium solution. Shake to mix.

11.19. Prepare a similar vial using 5 ml from a cement standard and 5 ml of lithium solution.

11.20. Prepare a blank using 5 ml DI water, and 5 ml lithium solution.

11.21. Turn on flame photometer. Turn on gas first, then turn on air, then turn on switch. Let flame photometer warm up for several minutes.

11.22. Measure alkali blank – should be 0-0. Use zero knobs, put needle between arrows on left hand dial.

11.23. Measure cement alkali standard - should be 1.35 Na and 1.20 K. Adjust if needed with calibration knobs.

11.24. Run cement standard, record all values on the worksheet, turn gas off first until flame goes off, then air, then switch.

11.25. Determining chlorine, chromium oxide, and zinc oxide

11.26. Place ~5 g of sample in 34 mm sample cup and secure 4 um prolene film.

11.27. Place sample in 34 mm mask X-ray cup and analyze using Cement Powder Trace program. Do not use a 28 mm mask due to chromium in steel.

11.28. Run at least one cement standard and check for compliance with ASTM C114. If results are outside of acceptance ranges, run drift correction and recheck.

12. Deviations from ASTM C114

1. Ceramic crucible not platinum.
2. 3-5 grams instead of 1.
3. 30 minutes instead of 15 min and then to constant weight.
4. 25 ml 25% HCl instead of 5 ml conc.
5. Less rinsing required with gooch crucible.
6. Ceramic crucible not platinum, 750° C instead of 1,000° C.

13. Data Acquisition, Calculations and Data Reduction

- All data are entered into main computer under Excel cement program. Concentrations are fitted between the two standards. The program is equipped to let you know if any element in the standard is out of range, and it checks the quality of the duplicate. Save each fit under the log number for further reference.

14. Data Management and Records Management

All data are attached to the log sheet and filed in customer file, along with copy of report and invoice.

15. Quality Control and Quality Assurance Section

All cement analyses are run in duplicate with two standards. Standards are run to check short-term stability. A primary standard will be run with each insoluble residue sample, and/or sodium/potassium determination. Re-qualification of XRF test method is required every 2 years or when more than one constituent is found to be outside specified limits. (See Table 1 in ASTM C114).

16. References

- ASTM C114, *Standard Test Methods for Chemical Analysis of Hydraulic Cement*
- *Preparation of Fusion Pellets for Analysis by XRF* (Wyoming Analytical Laboratory SOP)

Prepared by Stephen Ellis Date: 05/12/03

Revision by Charles R. Wilson Date: 6/8/12

Reviewed and approved by Charles R. Wilson Date: 6/8/12