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Donald G. Graczyk, Alice M. Essling, Edmund A. Huff,
Florence P. Smith, and Christine T. Snyder

Analytical Chemistry Laboratory
Chemical Technology Division
Argonne National Laboratory
9700 South Cass Avenue
Argonne, IL 60439

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Analytical Chemistry Laboratory/Chemical Technology Division
Argonne National Laboratory
9700 S. Cass Avenue
Argonne, Illinois 60439

Abstract

Chemical characterization of aluminum salt cake, or of intermediate or final products from processing salt cake, offers interesting challenges to the analytical chemist because the component phases resist dissolution (a key first step in most analysis procedures) and because process chemists and engineers need information on specific components such as leachable salts or aluminum metal in addition to overall elemental composition. Our laboratory had an opportunity to meet some of these challenges first-hand while providing analysis support to a project studying conversion of salt cake fines to value-added oxide products. In the course of this work, we adapted or devised analysis methods for determining leachable salt, total halides (Cl and F), aluminum metal, and elemental composition. Leaching of salt cake fines was accomplished by ultrasonic agitation with deionized water. We analyzed the leachate for anions (chloride, fluoride, nitrate, phosphate, sulfate) by ion chromatography and for cations by inductively coupled plasma atomic emission spectrometry (ICP-AES). Only chloride appeared at measurable concentrations among the anions. Charge balances between cations and chloride were near unity, indicating that all major dissolved species were chloride salts. For the total halides determination, we first brought the chloride and fluoride components into solution by decomposition with a potassium hydroxide fusion. The dissolved chloride and fluoride were measured by ion chromatography. Aluminum metal present in the fines was determined by a hydrogen evolution procedure, which we adapted to permit determination of sub-milligram quantities of metallic aluminum. In our approach, we reacted the aluminum metal with hydrochloric acid in a closed system containing a measured amount of high-purity helium. After reaction, we measured the hydrogen to helium ratio by mass spectrometry to determine the quantity of H_2 evolved. Recoveries of aluminum metal standards (approximately 30 mg Al) averaged 93%. Comparison of the acid evolution with caustic reaction of the aluminum metal showed results from the two methods to be virtually identical but reaction was faster in the acid medium. Metallic elements were determined in the salt cake materials by total dissolution and ICP-AES measurement of dissolved metals. Decomposition of the salt cake with mineral acids left undissolved residues that had to be brought into solution by fusion with sodium carbonate. A better dissolution was obtained by fusing the salt cake materials with lithium tetraborate, although this approach does not permit determination of lithium or boron in the dissolved sample. We found that the solution from lithium tetraborate fusion could be used as a starting point for accurate aluminum assay of salt cake materials by classical 8-hydroxyquinolate gravimetry.

Introduction

The mixture of salts, aluminum metal, and refractory oxides that constitutes aluminum salt cake poses many challenges to the analytical chemist who is asked to provide chemical information on its composition. Our laboratory had an opportunity to meet some of these challenges while providing analysis support to a project studying conversion of salt cake fines to value-added oxide products. The study involved a pilot-scale process in which, first, salt cake feed material was leached to separate water-soluble salts from insoluble residues so the salt fraction could be recovered and, then, the insoluble residues were treated, calcined, and converted into forms useful in producing potentially marketable products. Process engineers needed data from chemical analysis to determine the chemical composition of the salt cake feed material and to track the salt cake constituents through the processing steps. Of interest to the project were not only the overall elemental composition of the process feed and intermediate materials, but also individual phase components such as residual leachable salts, halides (fluoride and chloride) that did not leach with water and stayed with the insoluble fraction of the salt cake feed, and aluminum present as metal. For this work, we developed or adapted methods that took advantage of equipment, instrumentation, and expertise available in our facility, particularly classical and advanced decomposition techniques for sample preparation, inductively coupled plasma atomic emission spectrometry (ICP-AES) for multielement determination of metals in solution (1, 2), Ion Chromatography (IC) for determination of anions in solution (3), and mass spectrometry for analysis of gases.

In this paper, we describe the methods we applied to determining leachable salt, total halides, aluminum metal, and elemental composition of the salt cake and salt-cake-processing intermediates. We present some of the reasons we selected the methods we used, discuss the performance of the individual methods, and comment on the potential usefulness of these methods for emerging salt-cake-recycling industries.

Determination of Leachable Salt

One parameter of interest to the processing of aluminum salt cake is the amount of sodium chloride/potassium chloride salt that leaches with water and might thereby be made available for recovery and recycling as smelter flux. To determine the leachable salt in salt cake and in intermediate products from processing the salt cake, we began by placing a weighed amount of sample material (typically 1 g) in a glass, screw-cap vial with 20 mL laboratory deionized water and agitating the mixture in an ultrasonic bath for 2 hours. Then, we filtered the leaching solution, washed the solid residue on the filter with additional deionized water, and brought the combined filtrate and washings to a known volume. Finally, we analyzed the solution by ICP-AES to determine dissolved metals and by IC to determine anions including chloride, fluoride, and sulfate.

Although we generally analyzed for as many as 25 elements with ICP-AES, the only cations that were typically found at greater than trace concentrations were sodium and potassium; calcium and magnesium were usually also present, but in lesser amounts. Among the anions, only chloride appeared at measurable concentrations in the leachates. The charge ratio of cations to chloride typically fell in the range 0.95 to 1.05, indicating that chloride salts were the predominant species that had dissolved during leaching. For salt cake feed materials, leachable salts represented a major portion of the sample composition. Some feed materials we analyzed contained as much as 30 or 40 wt% leachable salt. By contrast, processed materials we analyzed early in the project showed lower concentrations (5 to 20 wt%). As the processes were optimized, we saw processed materials for which the leachable salt content approached the limits of detection for the methods we applied.

These instrumental analysis methods provide very sensitive detection of dissolved species. The ICP-AES measurements for cations have detection limits corresponding to 0.001 wt% or less of most elements relative to the mass of material that was leached. The detection limits for the IC anions determination correspond to 0.004 wt% for chloride and fluoride and to 0.01 wt% for sulfate. This level of sensitivity was quite useful in assessing the effectiveness of leachable salt removal during certain steps of the salt-cake processing.

Determination of Total Halides

In addition to the leachable alkali chlorides present in the aluminum salt cake materials we handled, other halide-containing components such as CaF_2, Na_3AlF_6, and $Ca(OCl)_2$ were also present, as indicated by X-ray diffraction analysis. Because water-insoluble halides could influence process requirements and product properties, the process engineers asked us to determine total halides in the salt cake materials. For the determination of total halides in many materials, alkali hydroxide fusion is often used to decompose the sample material. Some evidence exists that potassium hydroxide gives better retention of fluoride in the melt than lithium or sodium hydroxide (4). In setting up our procedure for the determination of total fluorine and chlorine in the salt cake materials, we adapted a potassium hydroxide fusion procedure used by the U.S. Environmental Protection Agency (EPA) for decomposing industrial waste materials for metals analysis (5). In this method, 0.25 g of sample is fused with 2 g KOH in a pyrolytically coated graphite crucible. The fusion is carried out stepwise, with the crucible first heated in a block heater to 160°C and then to 360°C, and then in a muffle furnace to 525°C. In the EPA method for metals, the cooled fusate is dissolved in acid and hydrogen peroxide. For the anions determination, we found that we could simply leach the fusate with deionized water to obtain a caustic solution that contained the desired halides, leaving some insoluble residues that could be filtered off or merely allowed to settle out of solution. After diluting the caustic leachate to an appropriate volume, we measured fluoride and chloride ions in the solution by IC. For this measurement, a portion of the solution was first put through a pretreatment column (OnGuard-H, Dionex Corporation, Sunnyvale, CA) that neutralizes excess hydroxide with hydrogen ions produced by ion exchange from a cation resin in the hydrogen form. With this pretreatment, the caustic fusate solution could be analyzed under the same IC conditions as were used for analysis of the water-leachable anions.

Typical concentrations of fluorine in the untreated or leached salt cake materials were around 2 wt%. Normally, total chlorine concentrations were indistinguishable from the leachable chloride (15 to 30 wt% in the untreated salt cake). In performing the IC analyses of the KOH fusates, we noted that not only fluoride and chloride were present in the dissolved fusates, but also sulfate ion. This was not particularly surprising because alkali hydroxide fusions are known to provide oxidizing conditions that should be able to convert most sulfur-containing species in a sample to sulfate. When we compared the quantities of sulfur equivalent to the sulfate measured in the KOH fusate with sulfur concentrations measured in corresponding samples by the conventional combustion method applied in a commercial sulfur determinator (LECO Corporation, St. Joseph, MI), we discovered that the two results were nearly identical at values near 0.2 wt% S. Thus, it appears that the KOH fusion procedure could be useful for determining sulfur in salt cake materials, as well as total halides.

Determination of Aluminum Present as Metal

The amount of aluminum present as metal in the salt cake feed and processed materials that we were asked to analyze was of considerable interest to the process engineers because the metal was implicated in certain process difficulties they were trying to resolve. Because the determination of metallic aluminum in drosses and powders is important to the aluminum industry, a number of procedures have been developed to make such measurements (6). For our work, we elected to determine the metal by a hydrogen evolution procedure using hydrochloric acid to effect the gas-generation reaction. Although it has been argued in some references (7) that HCl is not as selective for aluminum as a caustic alkali like sodium hydroxide and that HCl has no particular advantage over the alkali, we found the reaction with 6<u>N</u> HCl to be much more rapid. We also found that for a set of samples treated both ways, results we obtained were the same within experimental uncertainty, as shown in Table I.

In our analysis, a weighed subsample of salt cake material was placed in a closed system fitted with an addition funnel and connected to a vacuum manifold, which included a calibrated section of known volume and a Baratron (MKS Instruments, Inc., Burlington, MA) capacitive manometer for pressure measurement. The entire system was evacuated and then filled with an amount of high-purity helium that was measured in the calibrated volume by recording its pressure and temperature before expanding it into the manifold section that contained the sample. After expansion, the helium pressure over the sample was typically 0.5 atmosphere. Next, approximately 30 mL of 6<u>N</u> hydrochloric acid was slowly added with stirring to the sample by means of the addition funnel. After all the acid was added, the reaction mixture was vigorously

Table I. Results for Aluminum Metal Content of Salt Cake Materials Using Hydrochloric Acid or Sodium Hydroxide to Evolve Hydrogen

Sample Designation	Aluminum Metal Equivalent to Hydrogen from HCl Evolution, wt%	Aluminum Metal Equivalent to Hydrogen from NaOH Evolution, wt%
95-0151-01	15.5 ± 1.6	15.3 ± 1.5 12.3 ± 1.2[a]
95-0151-02	17.6 ± 1.8 19.5 ± 2.0[a]	22.2 ± 2.2
95-0151-03	2.23 ± 0.22	2.59 ± .026
95-0151-04	12.1 ± 1.2	13.5 ± 1.4

[a] Replicate measurement using separate subsample.

stirred and the gases in the manifold were allowed to mix and equilibrate for a period of at least one hour. Then, a portion of the cover gas was expanded into a previously evacuated gas-sampling bulb for subsequent analysis by mass spectrometry to determine the volume composition of the gas.

The mass spectrometric analysis measured not only the volume fractions of hydrogen and helium in each gas sample, but also the concentrations of carbon monoxide and dioxide, nitrogen, oxygen, methane and ethane, and other gases. From the volume ratio of each gas to helium and the known amount of helium that was added to the system before reaction, we calculated the volume [in units of cubic centimeters at standard temperature and pressure, cc(STP)] of each gas that was present in the system. We then converted these volumes to equivalent moles of gas by assuming ideal gas behavior [22.414 cc(STP) per millimole of gas], and equated the hydrogen to a mass of aluminum metal by assuming 1.5 moles of H_2 produced for each mole of aluminum that reacted; that is,

$$Al^0 + 3\,H^+ \rightarrow Al^{3+} + 1.5\,H_2 \qquad (1)$$

With our system, to which we normally added about 80 cc(STP) helium, and the ability of the mass spectrometric measurement to detect a H_2/He ratio of less than 0.0005, it would in principle be possible to detect the hydrogen produced by reaction of as little as 0.03 mg of aluminum, as shown by the following calculation:

$$\begin{gathered} 80\ \text{cc(STP) He} \times 0.0005\ \text{cc(STP) H}_2/\text{cc(STP) He} \\ \div 22.414\ \text{cc(STP) H}_2/\text{mmol H}_2 \times 1\ \text{mmol Al}/1.5\ \text{mmol H}_2 \\ \times 26.9815\ \text{mg Al}/\text{mmol Al} = 0.03\ \text{mg Al} \end{gathered} \qquad (2)$$

Such low detection limits cannot actually be achieved, however, because limitations such as the finite solubility of hydrogen in the aqueous reaction matrix and incomplete mixing of the evolved hydrogen and helium cover gas complicate interpretation of very small amounts of H_2 gas. Hence, unless a sample produced an amount of H_2 corresponding to 0.3 mg or more of aluminum, we reported the concentration of Al metal as being less than this amount. Typical subsamples that we analyzed with the gas evolution method were in the mass range from 0.1 g to about 5 g, with the larger masses taken for samples that evolved the least gas. With a 5 g subsample, the 0.3 mg minimum reporting limit corresponds to an aluminum metal concentration of only 0.006 wt%. Thus, the procedure provided a very sensitive test for aluminum metal in the process materials.

Results obtained with aluminum foil standards (nominally 30 mg Al) that we analyzed in the same manner as samples averaged 93% recovery of the aluminum with a standard deviation of about 6%. Duplicate measurements on salt cake materials sometimes showed somewhat greater variability (up to about 15% of the measured value), which could be attributed to non-uniformity in the metal content of the samples at the small sample size necessary to accommodate the capacity of the manifold we had available. Because the manifold could only handle an amount of hydrogen equivalent to about 50 mg of aluminum metal, while some samples contained as much as 22 wt% Al as metal, we could not always take a large enough subsample to fully represent the material being analyzed. Results from these samples nevertheless provided data sufficient for the process studies to which they contributed, even with the sampling uncertainty.

Gases other than hydrogen were often observed in the gases we analyzed by mass spectrometry. With salt cake feed materials in particular, measurable amounts of methane and ethane were present, which we presume arose from decomposition of reactive carbides present in the materials, as illustrated by eq. 3:

$$Al_4C_3 + 12\,H_2O \rightarrow 4\,Al(OH)_3 + 3\,CH_4 \qquad (3)$$

Quantities of methane were sometimes on the order of 2.5 cc(STP) per gram of salt cake but were lower in many cases; ethane was almost always lower than the methane by a factor of 5 or more. These amounts of hydrocarbons translate to reactive carbide concentrations of about 0.1 wt% or less, as carbon. Another gas frequently evolved from the samples we analyzed was carbon dioxide from acid decomposition of carbonates in the salt cake materials. Amounts of CO_2 were occasionally substantial, reaching levels of 15 cc(STP) per gram of sample or, equivalently, 4 wt% carbonate in the solid. The observed CO_2 can only provide a minimum value for the carbonate content of these samples because conditions of the gas generation were not fashioned to ensure that dissolved CO_2 was completely released to the gas phase. Nevertheless, tracking the observed CO_2 provided positive evidence of a substantial carbonate presence in the salt cake feed material and evidence that carbonates were lower in processed salt cake materials.

Determination of Elemental Composition

Many methods exist for the determination of individual element concentrations in real-world materials. One of the challenges faced by the analytical chemist is to select the method or set of methods that will provide reliable information on those elements that might be important within the context of the sample being analyzed and the use to which the data will be put. In our elemental characterization of the salt cake materials, we made use of several techniques. Measurements for total chlorine and fluorine by KOH fusion and IC analysis of the fusate were described earlier in this paper. For some other nonmetallic elements, we used commercial elemental analysis instruments designed for the elements of interest. These included a LECO WR-12 Carbon Determinator (LECO Corporation, St. Joseph, MI) for measuring carbon; a LECO CHN-

900 Analyzer for carbon, hydrogen, and nitrogen; and a LECO 518 Sulfur Determinator for sulfur. It was quite satisfying that, when data were available from more than one method for a given element, the results were in good agreement. Comparison between the sulfur indicated by the KOH fusion/IC procedure and that obtained with the oxygen-combustion method used in the LECO system was noted already. We also saw agreement between the carbon results from the two LECO analyzers we applied (see Table II) even though the two systems operate under different conditions-- the WR12 employs combustion of the sample in flowing oxygen at 1500°C while the CHN-900 uses combustion in limited oxygen at 1000°C.

Table II. Comparison of Results for Carbon from Two Different Commercial Instruments

Sample Designation	Carbon Measured with LECO WR-12, wt%	Carbon Measured with LECO CHN-900, wt%
96-0200-01	3.50 ± 0.18	3.73 ± 0.28
95-0103-01	1.41 ± 0.07	1.52 ± 0.07
95-0103-02	1.67 ± 0.08	1.57 ± 0.07
95-0103-03	0.76 ± 0.04	0.68 ± 0.07
95-0103-04	0.23 ± 0.05	0.16 ± 0.05
95-0103-05	0.05 ± 0.03	0.05 ± 0.05

For the determination of metallic elements, the ICP-AES system provides an enormously powerful tool, capable of measuring most metals at concentrations ranging from parts per million to weight percent in a given material. There is a catch, however, because materials to be analyzed by ICP-AES must first be brought into solution for introduction into the instrument. Most often, dissolution is accomplished by decomposition with mineral acids or by fusing the material with an appropriate fluxing agent that promotes solubilization of the material components. In our laboratory, we generally prefer mineral acid decomposition when it can be used because the fusion techniques result in solutions that contain higher levels of dissolved solids, which can interfere with instrument performance, and also add components that might preclude measurement of one or more elements of interest. Consequently, when confronted with the need to analyze salt cake materials, our first attempts at preparing solutions for the ICP-AES system utilized acid-decomposition techniques. We had some success using mixtures of nitric, hydrochloric and hydrofluoric acids in Teflon-lined, closed digestion vessels (Parr Acid Digestion Bombs, Parr Instrument Co., Moline, IL) heated to 140°C overnight. However, we found that almost every sample behaved a little differently and that adjustments to the acid proportions had to be made to accommodate these differences. Moreover, a number of samples left residues that simply could not be decomposed with acid treatment. In some cases, we isolated the undissolved material and subjected it to fusion with sodium carbonate to effect dissolution. This approach suffered three drawbacks. First, the two-step dissolution took a lot of effort and required analysis of two solutions to get the desired information. Second, sodium in the flux prevented reliable determination of sodium in the sample, and this element was an important one for characterizing these materials. And third, the sodium carbonate fusion was not always successful in decomposing the residues. X-ray diffraction analysis of the most resistant residues usually indicated that they contained Al_2O_3 in the form of corundum and/or SiO_2 in the form of tridymite or cristobolite.

A much better and, in our experience, universally successful, method for dissolving the salt cake materials is based on fusion with lithium tetraborate. In the procedure we use, a weighed amount (approximately 100 mg) of salt cake sample is mixed with approximately 250 mg of lithium tetraborate flux in a platinum crucible and then the mixture is covered with a layer of 150 mg additional flux. Next, the crucible is placed in a muffle furnace, preheated to 1000°C, for 20 to 30 minutes. After the fused sample cools, we place a stirring bar in the crucible and add 1:1 HCl (4 mL) and water (4 mL). The fusate is then dissolved on a stirring hotplate, and the solution is transferred to a volumetric flask for dilution to a known volume; our experience indicates that the solution should be transferred and diluted while warm or some solids might precipitate out of solution and not redissolve easily. The only elements normally measured by ICP-AES that this preparation interferes with are lithium and boron. In the materials we worked with, the acid-decomposition procedures we applied gave ample evidence that these elements were not of significance; lithium was seldom detected and boron concentrations were only on the order of a few hundredths of a weight percent.

The ICP-AES analysis laboratory normally reported concentration data for up to 26 elements, including Al, B, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Si, Sn, Sr, Ti, V, Zn, and Zr. Together with data from other methods for Cl, F, C, S, N, and H, these results generally provided sufficient compositional information to satisfy the needs of process chemists and engineers. To illustrate the levels at which these elements might occur in salt cake process materials, the results we obtained for one sample of washed salt-cake fines (leachable salts removed) are shown in Table III. As is conventional for such analyses, the balance of material not accounted for by the listed elements is presumed to consist of oxygen, which was not measured.

Typical uncertainties on the order of 5 to 10% of the reported value are associated with the elemental concentrations measured by the methods discussed above. Sometimes, however, more accurate values are needed for particular elements in a given situation. In most cases, requirements for high accuracy can only be met by application of classical methods involving gravimetric or titrimetric procedures. Because such procedures determine only one element and are typically quite labor intensive and expensive, they normally are used sparingly. We did have an opportunity arise in our salt cake analysis work, however, where accurate assay of the aluminum content in one product was needed. From an earlier ICP-AES analysis of the material, it was known to contain about 34 wt% aluminum (duplicate analyses gave values of 33.6 and 34.0%). For this assay, we applied the classical gravimetric aluminum assay in which aluminum in a dissolved portion of sample is precipitated with 8-hydroxyquinoline and determined from the mass of precipitate obtained (8). Based on our experience with dissolving samples for the mutielement analysis by ICP-AES, we used the lithium tetraborate fusion procedure to bring a portion (about 100 mg) of sample into solution. The procedure we followed (9) utilizes an iron hydroxide precipitation to remove elements that

Table III. Elemental Composition of a Sample of Washed Salt-Cake Fines

Element	Total Concentration, wt%
Al	25.5[a]
Ba	0.09
Be	<0.002
Ca	1.23
Cd	<0.002
Cr	0.09
Co	<0.01
Cu	0.39
Fe	1.58
Mg	6.69
Mn	0.14
Mo	<0.005
Ni	0.02
Pb	0.10
Si	3.40
Sr	0.01
Sn	0.01
V	<0.02
K	0.49
Na	0.66
P	<0.10
Ti	0.55
Zn	0.25
Zr	<0.02
Cl	0.59
F	3.87
S	0.22
C	3.60
N	0.54
H	1.34

[a] Metallic aluminum by hydrogen evolution was 3.04 wt%.

interfere with the aluminum assay. In this case, we suspected that the hydroxide precipitate might also carry some aluminum because the sample material also had a substantial (7.9 wt%) magnesium content, and magnesium is known to promote aluminum losses in the hydroxide separation (10). Consequently, in addition to determining the major fraction of aluminum as the 8-hydroxyquinolate, we also redissolved the iron hydroxide precipitate that was isolated and determined the smaller amount of aluminum in it by ICP-AES. We then calculated the aluminum content as the sum of the aluminum in the 8-hydroxyquinolate and that in the hydroxide. The amount of aluminum in the hydroxide turned out to be larger than we had expected and corresponded to about 10% of the total aluminum. Data from duplicate samples of the processed salt cake are shown in Table IV.

Table IV. Distribution of Aluminum between Fractions Obtained during Assay by 8-Hydroxyquinoline Precipitation

Sample Replicate No.	Aluminum in Iron Hydroxide Precipitate, wt% of Sample	Aluminum in 8-Hydroxy-quinolate, wt% of Sample	Total Aluminum, wt% of Sample
1	3.46	29.68	33.14
2	3.19	29.94	33.13

As is evident from the agreement in total aluminum concentrations in the last column of this table, the assays that were performed in this way were quite precise. Given expected accuracies of the ICP-AES measurements (about 5%, relative) and the aluminum gravimetry (about 0.2%, relative), the total aluminum results may be assigned an accuracy on the order of 0.5%, relative. It is significant to note, however, that if corrections were not made for the aluminum carried in the iron hydroxide precipitation, the results would have shown fairly good precision but would have been low by about 10% on average. This observation exemplifies the pitfalls that exist in chemical analysis of multicomponent materials like aluminum salt cake and products derived from it. Even the experienced analysis laboratory will require time and resources to test their methods with representative materials if high confidence in the results is to be achieved.

Conclusions and Recommendations

Overall, the methods and procedures described in this paper served quite well in providing the information that was needed for the process studies that they supported. The success we observed in applying the ICP-AES and ion chromatographic measurements to determining individual salt cake components confirms our belief that these instruments represent two of the most powerful tools available to today's analytical chemist. In preparing samples for elemental analysis with the ICP-AES, the lithium tetraborate fusion procedure proved universally effective as long as data for lithium and boron were not needed. Similarly, the potassium-hydroxide fusion procedure appeared to be quite effective for solubilizing halides and provided a solution matrix that was easily amenable to ion chromatographic analysis. Although the hydrogen-evolution procedure we applied in the determination of aluminum metal was satisfactory for the work in this project, it has several disadvantages that would make it worthwhile to investigate alternative methods for frequent material characterization or process control. These disadvantages include the need for a special apparatus for gas handling and for a gas-analysis capability (at least if our approach is used), and long processing times. One possible alternative method for measuring aluminum present as metal, which we would like to propose, is a variation of the copper salt method described by Young (6). In Young's procedure, a sample is treated with a dissolved copper salt, which displaces the metallic aluminum and precipitates as copper metal. The quantity of aluminum brought into solution during the treatment is a measure of aluminum metal in the sample and could be conveniently measured by ICP-AES.

Chemical analysis of aluminum salt cake and related materials requires careful attention to dealing with potential interferences on individual methods as well as an ability on the part of the analyst to deal with the variable composition and properties of the materials. As processes emerge for recycling the components of salt cake waste, procedures will be needed that can be applied reliably by less-skilled analysts than are now needed for characterization measurements. Developing such procedures is both a challenge and an opportunity for analytical chemistry. We hope that the work described here will be helpful in the development of an established analytical chemistry of aluminum salt cake.

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