

# ELEMENTAL COMPOSITION OF ZEOLITES BY ICP-AES

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## UOP Method 961-98

### SCOPE

This method is for the determination of components in zeolites by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES). Although the determination of silicon (Si), aluminum (Al), sodium (Na), potassium (K), calcium (Ca), iron (Fe), titanium (Ti), magnesium (Mg), phosphorus (P) and lithium (Li) is specifically addressed, many other elements, including trace contaminants, can also be determined. However, barium (Ba) and lead (Pb) cannot be determined in the digest due to the formation of insoluble sulfates. Barium can be determined from the fusion solution with a quantitation limit of approximately 0.01 mass-%. This method is suitable for samples containing less than 40 mass-% alumina ( $\text{Al}_2\text{O}_3$ ) and less than 65 mass-% silica ( $\text{SiO}_2$ ); however, it can be readily expanded to samples with other silica/alumina ratios utilizing the same approach as described herein. Zirconium (Zr) at levels above about one mass-% can interfere with the analysis for aluminum when using some instruments, unless an alternative wavelength is used. Precision is about 1 to 2% relative standard deviation for elements present at concentrations greater than 0.1 mass-%, and mass balances between 98.5 and 101.5 mass-% are reported on a volatile-free basis. Precision below 0.1 mass-% does not exceed 10% relative down to the quantitation limits listed in Table 2.

### OUTLINE OF METHOD

The elements of interest are determined utilizing two dissolution techniques. For Si and Al, a portion of the zeolite sample is fused with lithium tetraborate and the resulting melt is dissolved in 10% hydrochloric acid. The resulting solution is analyzed by ICP and the concentrations of Si and Al are determined by comparison to a NIST standard prepared in a manner the same as the unknowns. The other components are determined following a digestion with sulfuric and hydrofluoric acids. The resulting solution is analyzed by ICP and the concentrations of Na, K, Ca, Fe, Ti, Mg, P and Li are determined by comparison to a standard solution of similar elemental and sulfuric acid concentrations. Results are reported on a volatile-free basis, with the loss on ignition (LOI) determined by UOP Method 275 (LOI at 900°C).

### APPARATUS

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

**IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).**

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*Balance*, readability 0.1-mg

*Balance*, top-loading, readability 0.01-g

*Beaker*, Vycor™, 250-mL, Fisher Scientific, Cat. No. 02-541A

*Beaker*, Teflon™, 250-mL, Fisher Scientific, Cat. No. 02-593-5B

*Bottles*, 125- and 250-mL, wide mouth, round, polypropylene, Fisher Scientific, Cat. Nos. 02-893-5C and -5D, respectively

*Bottle*, washing, one-liter, polypropylene, Fisher Scientific, Cat. No. 03-409-14C

*Burner*, blast, Merker MFG. No. H-5020, VWR Scientific, Cat. No. 18216-000

*Crucible*, high form, platinum-gold, 30-mL, Alfa Aesar, Cat. No. 35375

*Cylinder*, graduated, polypropylene, 10-mL, Fisher Scientific, Cat. No. 08-570-21A

*Cylinders*, graduated, 50- and 100-mL, Fisher Scientific, Cat. Nos. 08-552-4B and -4C, respectively

*Flasks*, volumetric, Class A, 100-, 200-, 250-, 500- and 1000-mL, Fisher Scientific, Cat. Nos. 10-210C, -210D, -210E, -210F and -210G, respectively, several of each required

*Flasks*, volumetric, 200-mL, polypropylene, Nalgene™, Mfg. No. 4000-0200, Fisher Scientific, Cat. No. 10-198-50C

*Funnel*, disposable, 55-mm diameter, Fisher Scientific, Cat. No. 10-320A

*Fluxer*, Claisse (optional), with necessary crucibles and other equipment, available from SPEX CertiPrep, Model Fluxy-30, Cat. No. 7121

*Hot plate*, stirring, variable heat, Corning Model PC-420, Fisher Scientific, Cat. No. 11-497-7A

*Hot plate*, variable heat, Corning Model PC-600, Fisher Scientific, Cat. No. 11-497-2A

*Inductively Coupled Plasma-Atomic Emission Spectrometer* (ICP-AES), Perkin-Elmer Optima 3000 spectrometer, or equivalent (see *NOTE*)

*Mortar and pestle*, agate

*Muffle furnace* (optional), capable of 1000°C. The interior surface of the furnace must be of non-flaking material, or sample contamination may result.

*Oven*, drying, capable of 105°C

*Pipets*, total volume, Class A, 5-, 10-, 15-, 20- and 25-mL, Fisher Scientific, Cat. Nos. 13-650-2F, -2L, -2M, -2N and -2P, respectively, several of each required

*Regulator*, argon, two stage, high purity, Matheson Gas Products, Model 3122-580

*Stir bar*, magnetic, Teflon coated, 40 x 120 mm, Fisher Scientific, Cat. No. 14-511-60A

*Tongs*, crucible, stainless steel, lock joint, platinum shoes, Fisher Scientific, Cat. No. 15-205

*Triangle*, nickel-chromium, 2-inch (51-mm), Fisher Scientific, Cat. No. 15-260B

*Vacuum Desiccator*, Corning No. 3118-160, Fisher Scientific, Cat. No. 08-632

*Watch glass*, Pyrex™, 90-mm diameter, Fisher Scientific, Cat. No. 02-612C

*Watch glass*, Teflon, 100-mm, Fisher Scientific, Cat. No. 02-610-5E

## REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified. References to water mean distilled, deionized water.

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

*Argon*, 99.995% minimum purity, plasma and nebulizer gas

*Cesium Iodide*, CsI (optional), releasing agent, SPEX CertiPrep, Cat. No. FF-0730

*Gloves*, nitrile latex, Fisher Scientific, Cat. No. 11-393-64A through -64E, corresponding to hand sizes 7 through 11

*Hydrochloric acid*, concentrated

*Hydrochloric acid*, 10% (vol/vol). Mix one part concentrated hydrochloric acid with nine parts water. Make one liter at a time and store in a one-liter wash bottle.

*Lithium tetraborate*, Ultra Pure, SPEX CertiPrep, Cat. No. FF-0601

*Nitric acid*, concentrated

*Nitrogen*, 99.99% minimum purity, water and oil free, ICP optics purge gas

*Phosphorus solution*, 10,000-µg/mL, SPEX CertiPrep, Cat. No. PLP9-3X

*Soda feldspar*, National Institute of Standards and Technology (NIST), Cat. No. 99a

*Standard solutions of metals*, Sc, Fe, Ca, Mg, Li and Ti solutions of 1000 µg/mL concentration each, SPEX CertiPrep, Cat. Nos. PLSC2-2X, PLFE2-2X, PLCA2-2X, PLMG2-2X, PLLI2-2X and PLT19-2X, respectively

*Standard solutions of metals*, Al, P, K and Na solutions of 10,000 µg/mL concentration each, SPEX CertiPrep, Cat. Nos. PLAL1-3X, PLP9-3X, PLK1-3X and PLNA1-3X, respectively

## PROCEDURE

**CAUTION:** All subsequent steps involving lithium tetraborate fusion and handling of concentrated acids must be performed in a properly operating fume hood, and appropriate personal protective equipment must be worn (see MSDS for each material used).

### Preparation of ICP Calibration Standards and Blanks

Scandium is used as an internal standard; other elements and/or concentrations can be utilized; adjust accordingly (see *NOTE*). Label and date all standard and blank solution containers.

### Lithium Tetraborate Calibration Standard for Aluminum and Silicon

A solution of fused NIST 99a is used to calibrate the ICP for the determination of aluminum and silicon. This is used because of difficulties encountered with the accuracy and stability of commercial aqueous silicon standards.

1. Dry a portion of NIST 99a in an oven at 105°C for 2 hours, remove and place in a desiccator to cool.
2. Weigh  $0.125 \pm 0.005$  g, to the nearest 0.1-mg, of dried NIST 99a into a platinum crucible.
3. Weigh  $1.25 \pm 0.02$  g, to the nearest 0.01 g, of lithium tetraborate into the platinum crucible, using a top-loading balance. Rotate and tilt the crucible by hand to mix the sample with the lithium tetraborate flux.
4. Place the crucible on a triangle and heat with a blast burner for a minimum of 30 minutes, occasionally checking for unfused sample as evidenced by white specks in the melt. The fusion is complete when the melt is entirely clear. The fusion can also be performed in a muffle furnace set at 1000°C, or by using an automated fusion apparatus (fluxer) with the addition of approximately 100 mg of CsI as a releasing aid.
  - If unfused sample is detected, mix the hot melt by rotating and tilting the hot crucible with tongs, and continuing fusion until it is complete.
5. Remove the crucible from the flame using the platinum tipped tongs, place it upright in a 250-mL Vycor beaker and allow to cool for about 5 minutes.
6. Place a stirring bar inside the crucible and add 10% hydrochloric acid so that the crucible is submerged by about 10 mm.

7. Cover the beaker with a watch glass and place it on a stirring hot plate, adjust the heat to achieve mild boiling and the stirrer to achieve a vortex in the solution.
8. Continue heating until the fusion melt has completely dissolved, usually within 10 minutes after boiling begins.
  - Carefully check the crucible and the beaker for any particulates, indicating undissolved NIST 99a. If any doubt remains as to the completeness of the fusion or dissolution of the melt, discard the solution and start over at Step 1.
9. Remove the beaker from the hot plate and allow it to cool. Using the 10% hydrochloric acid from the wash bottle and a funnel, quantitatively transfer the beaker contents to a 250-mL volumetric flask.
  - The crucible is to remain in the beaker. Use the wash bottle to wash the interior and exterior walls of the crucible as well as the beaker walls during transfer.
10. Pipet 5 mL of the 1000- $\mu\text{g/mL}$  scandium solution into the volumetric flask.
11. Allow the solution to cool to ambient temperature and then dilute to the mark with 10% hydrochloric acid. Stopper and invert several times to mix thoroughly and then transfer the solution to a labeled and dated 250-mL polypropylene bottle for storage.
12. Calculate the aluminum and silicon concentrations using Eq. 1:

$$\text{Si or Al Concentration, } \mu\text{g/mL} = 10,000 \frac{CW}{250} \quad (1)$$

where:

**C** = certified value for Si or Al in NIST 99a, mass-%

**W** = mass of dried NIST 99a taken (Step 2), g

**250** = volumetric flask volume, mL

**10,000** = factor to convert mass-% to  $\mu\text{g/g}$

13. Calibrate the ICP with the new NIST 99a standard solution as described in *Sample Analysis* and compare to the old, previously utilized, standard solution. The results should agree within 2% (relative) of certified values.
  - For the initial establishment of a calibration curve, also fuse and analyze a portion of NIST 99a as an unknown. The values should be within 2% relative of the certified values. If the values returned for the unknown NIST 99a portion are too low, the fusion of the unknown portion was incomplete. If the values are too high, the fusion for the Calibration Standard was incomplete.

### Lithium Tetraborate Calibration Standard Blank

1. Weigh  $1.25 \pm 0.02\text{g}$ , to the nearest 0.01 g, of lithium tetraborate into a 250-mL volumetric flask.
2. Add approximately 100 mL of 10% hydrochloric acid to dissolve the powder.
  - Some heating may be required to dissolve the lithium tetraborate.

3. Pipet 5 mL of 1000- $\mu\text{g/mL}$  scandium solution into the volumetric flask.
4. Allow the solution to cool to ambient temperature and dilute to the mark with 10% hydrochloric acid. Stopper, mix thoroughly and transfer into a labeled and dated 250-mL polypropylene bottle for storage.
  - The calibration standard and blank are stable for three months.
  - Make up new calibration solutions when the ones in use are about 75% consumed or are older than three months.
  - A blank is necessary due to trace levels of silicon and aluminum found in commercial batches of lithium tetraborate and for matrix matching. This blank is also to be used for diluting samples where the Al or Si concentration is above the concentration in the NIST 99a standard calibration solution.
  - Because of varying contamination levels, the blank and calibration solutions are to be made using the same lot of lithium tetraborate.
  - When starting to use a new lot of lithium tetraborate, make up a new calibration standard and blank.

### Sulfuric Acid Digestion Calibration Solution

1. Prepare 500 mL of the sulfuric acid digestion calibration solution in 5% sulfuric acid by pipeting the volumes of the element concentrate solutions indicated in Table 1 into a 500-mL volumetric flask.

**Table 1**  
**Calibration Standard Volumes and Concentrations**

| Element | Volume,<br>mL | Element in<br>Concentrate,<br>$\mu\text{g/mL}$ | Element<br>Concentration<br>in the Working<br>Calibration<br>Standard,<br>$\mu\text{g/mL}$ | Approximate<br>Concentration in Solid,<br>mass-% |          |
|---------|---------------|------------------------------------------------|--------------------------------------------------------------------------------------------|--------------------------------------------------|----------|
|         |               |                                                |                                                                                            | As Element                                       | As Oxide |
| Al      | 30            | 10,000                                         | 600                                                                                        | 24                                               | 45       |
| Na      | 15            | 10,000                                         | 300                                                                                        | 12                                               | 16       |
| K       | 15            | 10,000                                         | 300                                                                                        | 12                                               | 14.4     |
| P       | 15            | 10,000                                         | 300                                                                                        | 12                                               | 27.5     |
| Ca      | 40            | 1000                                           | 80                                                                                         | 3.2                                              | 4.5      |
| Li      | 25            | 1000                                           | 50                                                                                         | 2                                                | 4.3      |
| Ti      | 25            | 1000                                           | 50                                                                                         | 2                                                | 3.3      |
| Fe      | 25            | 1000                                           | 50                                                                                         | 2                                                | 2.9      |

2. Pipette 10 mL of 1000- $\mu\text{g/mL}$  scandium solution in to the 500-mL flask.
3. Using a graduated cylinder, add 25 mL of concentrated sulfuric acid, then carefully add water to fill the flask half full and swirl to mix the contents.
  - Gloves are to be used at all times when handling hydrofluoric and sulfuric acids.
4. Allow to cool, fill to mark with water, stopper and invert to mix.
  - Make a new calibration solution when approximately 75% of the old one has been consumed or it is three months old.

## Calibration Blank for Sulfuric Acid Digestions

Prepare a calibration blank analogous to the working standard above, add the internal standard, but not aluminum or any of the other elements that are to be determined.

## Sample Preparation

Samples are generally provided in powder form; if as pill (sphere) or extrudate, they must be ground. A mortar and pestle or any commercial grinding apparatus may be used.

## Lithium Tetraborate Fusion

1. Weigh  $0.10 \pm 0.01$  g of powder sample, to the nearest 0.1-mg, into a platinum crucible.
2. Weigh  $1.0 \pm 0.02$  g of lithium tetraborate, to the nearest 0.01-g, into the crucible. Rotate and tilt the crucible by hand to mix the sample with the lithium tetraborate.
  - Weigh a sample portion for LOI by UOP Method 275 at the same time the sample is weighed for the metal determinations.
  - Note: use 100 mg sample, 1g of flux and 4 mL of 1000- $\mu$ g/mL scandium solution into 200 mL, all ratios as for the NIST 99a standard. A larger amount for NIST 99a results in a greater amount of the calibration standard.
3. Fuse as for the NIST 99a standard above, but add only 4 mL of 1000- $\mu$ g/mL scandium solution and transfer into a 200-mL volumetric flask.

## Sulfuric/Hydrofluoric Acid Digestion

1. Weigh  $0.50 \pm 0.05$  g of powder sample, to the nearest 0.1-mg, into a 250-mL Teflon beaker.
2. Wet the contents with water and then add an additional 20 mL of water.
3. Using a graduated cylinder, add  $10 \pm 0.5$  mL of sulfuric acid and swirl to mix.
4. Using a polypropylene graduated cylinder add  $10 \pm 1$  mL of hydrofluoric acid.
  - Gloves are to be used at all times when handling hydrofluoric and sulfuric acids.
5. Place the uncovered beaker on a hot plate and heat until white fumes of sulfuric acid appear.
  - Do not overheat as evaporation of sulfuric acid will make the matrix matching problematic.
6. Remove the beaker from the hot plate and allow it to cool. Check to see if the contents are dark-colored due to the presence of organic materials or coke that may have been in the sample. If dark, add approximately 2 mL of concentrated nitric acid and place the beaker back on the hot plate until white fumes of sulfuric acid evolve and the solution is no longer dark (repeat the addition of nitric acid if required). Remove the beaker from the hot plate and allow it to cool.
  - Do not overheat as evaporation of sulfuric acid will make the matrix matching problematic.
7. Add, slowly, about 40 mL of water to the sample, cover the beaker with a Teflon watch glass and return it to the hot plate. Let the sample boil until the solution is clear, remove and allow to cool.

8. Transfer, quantitatively, the contents of the beaker into a 200-mL Nalgene volumetric flask. Pipet 4 mL of 1000- $\mu\text{g/mL}$  scandium solution into the flask.
9. Allow to cool to ambient temperature and fill to the mark with water. Stopper and invert the flask several times to mix.

### Sample Analysis

It is assumed that the analyst is familiar with the routine set up and operation of an ICP spectrometer. Set up the ICP-AES instrument in accordance with the manufacturer's guidelines. Table 2 lists the recommended wavelengths for each element. Other wavelengths may be used if it can be shown that precision is not adversely affected. If the concentration of Al is less than 0.5  $\mu\text{g/mL}$  in the fusion solution (equal to 0.1 mass-% Al or 0.2 mass-%  $\text{Al}_2\text{O}_3$  in the sample), determine the Al concentration from analysis of the sulfuric acid digest.

1. Standardize the instrument by aspirating the blank, followed by the standards. Allow sufficient wash-out time between aspirations.
  - Wash-out times depend on instrument conditions, such as the pump rate, and on the concentration levels being measured, and can vary between 30 and 120 seconds.
2. Aspirate the samples, after entering the dilution factors into the computer. Allow sufficient wash-out time between aspirations.
3. Check how well the calibration is holding by aspirating the calibration standards, treating them as unknowns with no dilution factors. Check after every fifth sample, and following the final sample.
  - Any calibration checks and the final standard reading should return the concentrations of the elements in the standards within  $\pm 2\%$  relative. If not, re-calibrate and re-analyze the sample solutions.

**Table 2**  
**Recommended Wavelengths and Quantitative Limits**

| Element | Wavelength, nm | Quantitation Limits,<br>$\mu\text{g/mL}$ in solution | Approximate<br>mass-ppm in solid, |          |
|---------|----------------|------------------------------------------------------|-----------------------------------|----------|
|         |                |                                                      | As Element                        | As Oxide |
| Al      | 396.152**      | 0.2                                                  | 40                                | 80       |
| Ca*     | 317.933        | 0.1                                                  | 20                                | 30       |
| Fe*     | 238.204        | 0.04                                                 | 8                                 | 10       |
| K*      | 766.491        | 0.4                                                  | 80                                | 100      |
| Li*     | 610.364        | 0.2                                                  | 40                                | 90       |
| Mg*     | 285.213        | 0.04                                                 | 8                                 | 15       |
| Na*     | 589.592        | 0.2                                                  | 40                                | 50       |
| P*      | 213.618        | 0.4                                                  | 80                                | 200      |
| Sc      | 424.683        | -----Internal Standard-----                          |                                   |          |
| Si      | 251.611        | 0.2                                                  | 40                                | 90       |
| Ti*     | 334.941        | 0.02                                                 | 4                                 | 7        |

\* Based on 0.5 g of sample diluted to 200-mL volume.

\*\* If zirconium is present as a component, use Al 308.215 nm.

## CALCULATIONS

If the weight/volume dilution factor for the sample is entered into the instrument computer, the concentrations of the elements in the original sample, mass-% on an as-received (AR) basis, are reported automatically, but, with improper number of significant figures. Round and report to three significant figures for results equal to or greater than ten mass-%, to two significant figures between ten and one mass-%, and to one significant figure below one mass-%.

If the LOI value is available prior to the instrument analysis, the sample weight can be corrected for volatile content, the corrected value entered into the instrument computer, and the computer will calculate results on a volatile-free (VF) basis. If the LOI is not available, manually calculate the concentration of each element in the original sample on a volatile-free (VF) basis as follows:

$$\text{Concentration, mass-\% VF} = \frac{C}{\left(1 - \frac{D}{100}\right)} \quad (2)$$

where:

**C** = concentration of element on an as-received basis, mass-%

**D** = % LOI as determined by UOP Method 275, mass-%

**1** = factor to convert mass fraction LOI to mass fraction dry solids

**100** = factor to convert mass-% LOI to mass fraction, mass-%

## REPORT

*Every sample result must be checked for mass balance.* Use the gravimetric factors listed in Table 3 to convert elemental concentrations to oxides. Report results only when the mass balance is between 98.5 and 101.5 mass-% (include LOI if results are as-received), otherwise rerun the sample. If the mass balance is low, scan the sulfuric acid digest to determine if any missing elements (for example, Pt, Sn and La or other Rare Earths) are present. Through the use of appropriate standards, and using the procedures listed herein, determine the amount of any such elements found and include them in the final report.

**Table 3**  
**Gravimetric Conversion Factors**

| <u>To Convert</u>                    | <u>Multiply By</u> |
|--------------------------------------|--------------------|
| Al to Al <sub>2</sub> O <sub>3</sub> | 1.8895             |
| Ca to CaO                            | 1.3992             |
| Fe to Fe <sub>2</sub> O <sub>3</sub> | 1.4297             |
| K to K <sub>2</sub> O                | 1.2046             |
| Li to Li <sub>2</sub> O              | 2.1525             |
| Mg to MgO                            | 1.6583             |
| Na to Na <sub>2</sub> O              | 1.3480             |
| P to P <sub>2</sub> O <sub>5</sub>   | 2.2914             |
| Si to SiO <sub>2</sub>               | 2.1393             |
| Ti to TiO <sub>2</sub>               | 1.6680             |

## NOTE

In the performance of this method, either a simultaneous or sequential ICP instrument may be used. Simultaneous instruments generally do not require use of an internal standard. Internal standard use is specified here in order to produce the best possible precision from the instrument (Optima 3000) used in development of the method. If it can be shown that a particular simultaneous instrument can produce data of equal or better precision without use of an internal standard, use of the internal standard as specified herein can be dropped. Any procedural steps pertaining to use of the internal standard can then be ignored. When using a sequential instrument, the internal standard must be used.

## PRECISION

### UOP Repeatability

Based on single tests performed by each of three analysts on each of four different days (12 tests) in one laboratory, the within-laboratory estimated standard deviations (esd) were calculated for components at specific concentration ranges in a variety of products and are listed in Table 4. Two tests performed in one laboratory by different analysts on different days should not differ by more than the allowable differences in Table 4 at the concentrations listed (95% probability). There is insufficient data to calculate ASTM Repeatability at this time. A Perkin-Elmer Optima 3000 (simultaneous) ICP instrument and scandium internal standard were used to produce the data in Tables 4 and 5.

**Table 4**  
**UOP Repeatability**

| <u>Sample Type</u>      | <u>Component</u>                   | <u>Concentration or Range, mass-%</u> | <u>Within-Lab esd, mass-%</u> | <u>Allowable Difference, mass-%</u> |
|-------------------------|------------------------------------|---------------------------------------|-------------------------------|-------------------------------------|
| <b>Molecular Sieves</b> | <b>Al<sub>2</sub>O<sub>3</sub></b> | <b>19 to 38</b>                       | <b>0.28</b>                   | <b>0.9</b>                          |
|                         | <b>SiO<sub>2</sub></b>             | <b>36 to 64</b>                       | <b>0.47</b>                   | <b>1.5</b>                          |
|                         | <b>Na<sub>2</sub>O</b>             | <b>7 to 22</b>                        | <b>0.18</b>                   | <b>0.5</b>                          |
|                         | <b>K<sub>2</sub>O</b>              | <b>5 to 12</b>                        | <b>0.20</b>                   | <b>0.6</b>                          |
| <b>NIST 99a</b>         | <b>Al<sub>2</sub>O<sub>3</sub></b> | <b>20.6</b>                           | <b>0.15</b>                   | <b>0.6</b>                          |
|                         | <b>SiO<sub>2</sub></b>             | <b>65.2</b>                           | <b>0.53</b>                   | <b>1.9</b>                          |
|                         | <b>Na<sub>2</sub>O</b>             | <b>6.2</b>                            | <b>0.07</b>                   | <b>0.3</b>                          |
|                         | <b>K<sub>2</sub>O</b>              | <b>5.3</b>                            | <b>0.06</b>                   | <b>0.2</b>                          |
|                         | <b>CaO</b>                         | <b>2.1</b>                            | <b>0.04</b>                   | <b>0.1</b>                          |

The data listed in Table 4 were generated from control chart data covering a three week period and are considered to be an estimate of short-term repeatability of the method. When the test is run routinely in the field, a control standard and individual-range chart should be used to develop a better estimate of the long-term repeatability. If the silica/alumina ratio of the samples is significantly different from that of NIST 99a, use other NIST materials available that might make more appropriate standards.

## Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

## ACCURACY

Accuracy of the analysis was determined by analyzing the NIST 99a Standard Reference Material, and the data are listed in Table 5. The values agree within the experimental error of the test. There is no significant bias to the test.

**Table 5**  
**Accuracy**

| <b>Component</b>                   | <b>NIST Certified Values</b> |                    | <b>This Method</b> |                    |
|------------------------------------|------------------------------|--------------------|--------------------|--------------------|
|                                    | <b>mass-%</b>                | <b>esd, mass-%</b> | <b>mass-%</b>      | <b>esd, mass-%</b> |
| <b>Al<sub>2</sub>O<sub>3</sub></b> | <b>20.5</b>                  | <b>0.1</b>         | <b>20.6</b>        | <b>0.15</b>        |
| <b>SiO<sub>2</sub></b>             | <b>65.2</b>                  | <b>0.1</b>         | <b>65.2</b>        | <b>0.53</b>        |
| <b>Na<sub>2</sub>O</b>             | <b>6.2</b>                   | <b>0.1</b>         | <b>6.2</b>         | <b>0.07</b>        |
| <b>K<sub>2</sub>O</b>              | <b>5.2</b>                   | <b>0.1</b>         | <b>5.3</b>         | <b>0.06</b>        |
| <b>CaO</b>                         | <b>2.14</b>                  | <b>0.01</b>        | <b>2.1</b>         | <b>0.04</b>        |

## TIME FOR ANALYSIS

The elapsed time for one analysis is 5 hours. The labor requirement is 2.5 hours.

## REFERENCE

UOP Method 275, "Loss on Ignition of Catalysts at 900°C"

## SUGGESTED SUPPLIERS

Alfa Aesar, 30 Bond St., Ward Hill, MA 01835 (508-521-6300)  
 Fisher Scientific, 585 Alpha Dr., Pittsburgh, PA 15238 (412-490-8300)  
 Matheson Gas Products, Inc., P.O. Box 96, Joliet 60434 (815-727-4848)  
 National Institute of Standards and Technology, Standard Reference Materials Program, Room 204,  
 Building 202, Gaithersburg, MD 20899-0001 (301-975-6776)  
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