Date of Issue: April 25, 1998
Affected Publication: API Recommended Practice 13K, *Recommended Practice for Chemical Analysis of Barite*, Second Edition, February, 1996.

ERRATA

Page 17, Table 2: Replace Table 2 with the following:

Sulfide Range, ppm	Sample Volume, g	Dräger Tube Identification (see tube body)	Tube Factor (used in calculation)
1.2–24	10.0	H_2S 100/a	0.12[a]
2.4–48	5.0	as above	as above
4.8–96	2.5	as above	as above

[a] Tube Factor of 0.12 applies to new tubes, H_2S 100/a (catalog number CH-291-01), with 100–2000 scale. Old tubes with 1–20 scale, use Tube Factor of 12.

Page 19, section 3.16.4 and equation 28: Change the paragraph to read as follows:
... and the Tube Factor (0.12) from Table 2...

Replace Equation 28 with the following:

$$\text{GGT, Acid-Soluble Sulfides, ppm} = \frac{0.12\ \text{(Darkened Length)}}{\text{Sample Wt., g}}$$

Recommended Practice for Chemical Analysis of Barite

API RECOMMENDED PRACTICE 13K
SECOND EDITION, FEBRUARY 1996

ANSI American National Standard ANSI/API 13K-1990

American
Petroleum
Institute

Recommended Practice for Chemical Analysis of Barite

Exploration and Production Department

API RECOMMENDED PRACTICE 13K
SECOND EDITION, FEBRUARY 1996

EFFECTIVE DATE: APRIL 1, 1996

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Suggested revisions are invited and should be submitted to the director of the Exploration and Production Department, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005.

CONTENTS

IMPORTANT INFORMATION CONCERNING USE OF ASBESTOS OR ALTERNATIVE MATERIALS

Asbestos is specified or referenced for certain components of the equipment described in some API standards. It has been of extreme usefulness in minimizing fire hazards associated with petroleum processing. It has also been a universal sealing material, compatible with most refining fluid services.

Certain serious adverse health effects are associated with asbestos, among them the serious and often fatal diseases of lung cancer, asbestosis, and mesothelioma (a cancer of the chest and abdominal linings). The degree of exposure to asbestos varies with the product and the work practices involved.

Consult the most recent edition of the Occupational Safety and Health Administration (OSHA), U.S. Department of Labor, Occupational Safety and Health Standard for Asbestos, Tremolite, Anthophyllite, and Actinolite, 29 *Code of Federal Regulations* Section 1910.1001; the U.S. Environmental Protection Agency, National Emission Standard for Asbestos, 40 *Code of Federal Regulations* Sections 61.140 through 61.156; and the U.S. Environmental Protection Agency (EPA) rule on labeling requirements and phased banning of asbestos products (Sections 763.160–179).

There are currently in use and under development a number of substitute materials to replace asbestos in certain applications. Manufacturers and users are encouraged to develop and use effective substitute materials that can meet the specifications for, and operating requirements of, the equipment to which they would apply.

SAFETY AND HEALTH INFORMATION WITH RESPECT TO PARTICULAR PRODUCTS OR MATERIALS CAN BE OBTAINED FROM THE EMPLOYER, THE MANUFACTURER OR SUPPLIER OF THAT PRODUCT OR MATERIAL, OR THE MATERIAL SAFETY DATA SHEET.

Recommended Practice for Chemical Analysis of Barite

1 Scope

1.1 INTRODUCTION

Barite is used to increase the density of oil well drilling fluids. It is a mined product that can contain significant quantities of minerals other than barium sulfate, which is its main component.

1.2 MINERALOGICAL COMPOSITION

A list of some of the minerals commonly associated with barite ores is given in Table 1 with the chemical formulas, mineralogical names, and the densities of the mineral grains.

1.3 DRILLING FLUID PERFORMANCE

The performance of barite in a drilling fluid is related in part to the percentage and type of non-barite minerals distributed in the barite ore. Some of these minerals have little or no effect on drilling fluid properties, but others can degrade these properties and even be harmful to rig personnel.

1.4 OBJECTIVE

It is the objective of this publication to provide a comprehensive, detailed description of the chemical analytical procedures for quantitatively determining the mineral and chemical constituents of barite. These procedures are quite elaborate and will normally be carried out in a well-equipped laboratory.

2 References

Unless otherwise specified, the most recent editions or revisions of the following standards shall, to the extent specified herein, form a part of this standard.

API
- RP 13B-1 *Water-Based Drilling Fluids*
- *Manual of Petroleum Measurement Standards*
  - Chapter 15, "Guidelines for Use of the International System of Units (SI) in the Petroleum and Allied Industries"

EPA[1]
- 40 *Code of Federal Regulations* Section 61.140–156
- 51 *Federal Register* 3738–3759 (January 29, 1986)

OSHA[2]
- 29 *Code of Federal Regulations* Section 1910:1001

3 Wet Methods of Analysis

3.1 GENERAL DESCRIPTION

3.1.1 Classical wet chemical methods of analysis are commonly used to analyze barites. This is because the crystallographic technique of X-ray diffraction, used to determine individual crystalline mineral structures, does not work well with barite ores, due to the strong absorption of X-rays by barium atoms.

3.1.2 These classical wet methods can determine the elements present in the sample but will not supply all of the details of the association of elements to form specific minerals. Iron, for example, may be present as the oxide, carbonate, or sulfide, or be incorporated into the structure of a clay mineral. A few chemical analysis procedures are selective for certain minerals or compounds, but most of the mineral composition must be deduced from the total results of chemical analysis. (See Appendix A.)

3.1.3 The chemical analysis methods employed have the following advantages:

a. If directions are carefully followed, most trained chemists or technicians can get good results within a reasonable time.
b. The methods are selective, sensitive (usually in the parts per million (ppm) range), accurate, and reproducible (accuracy and reproducibility are usually 1 percent or less).
c. From the results of the wet analyses, one can usually determine approximate mineral (and/or compound) composition.
d. These methods have been thoroughly tested over a period of many years; most of them are the final test result of a long evolution of trial-and-error techniques.

3.1.4 The following outline shows the general analytical methods used for the analysis of barite:

a. Barium Sulfate ($BaSO_4$)—sodium carbonate (Na_2CO_3) fusion, gravimetric analysis.
b. Strontium Sulfate ($SrSO_4$)—sodium carbonate (Na_2CO_3) fusion, atomic absorption (AA) analysis.
c. Silica (SiO_2) and Alumina (Al_2O_3)—sodium hydroxide (NaOH) fusion, colorimetric analysis.

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[1] Environmental Protection Agency. The *Code of Federal Regulations* is available from the U.S. Government Printing Office, Washington, D.C. 20402.

[2] Occupational Safety and Health Administration, U.S. Department of Labor. The *Code of Federal Regulations* is available from the U.S. Government Printing Office, Washington, D.C. 20402.

Table 1—Minerals Associated with Barite Ore Bodies

Mineral	Chemical Formula	Mineralogical Name	Specific Gravity	Hardness (MOHS)
Barium Sulfate	$BaSO_4$	Barite, Barytes	4.50	2.5–3.5
Strontium Sulfate	$SrSO_4$	Celestite	3.97	3.5
Barium Carbonate	$BaCO_3$	Witherite	4.3	3.5
Barium Silicate	$BaSi_2O_5$	Sanbornite	—	—
Calcium Carbonate	$CaCO_3$	Calcite	2.72	3
Calcium, Magnesium Carbonate	$CaMg(CO_3)_2$	Dolomite	2.87	3.54–4
Magnesium Carbonate	$MgCO_3$	Magnesite	3.0	4
Calcium Sulfate	$CaSO_4 \cdot 2H_2O$	Gypsum	2.3	2
Calcium Fluoride	CaF_2	Fluorite	3.0	4
Iron Oxide	Fe_2O_3	Hematite	5.2	5.5
Iron Oxide	$Fe_2O_3 \cdot nH_2O$	Goethite	3.3–4.0	5.5
Iron Oxide	Fe_3O_4	Magnetite	5.16–5.17	5.5–6.5
Iron Carbonate	$FeCO_3$	Siderite	3.7–3.9	3.5–4.0
Iron Sulfide	FeS_2	Pyrite	5.0	6–6.5
Iron Sulfide	$Fe_{1-x}S$	Pyrrhotite	4.6	4
Iron/Copper Sulfide	$CuFeS_2$	Chalcopyrite	4.2	4
Lead Sulfide	PbS	Galena	7.5	2.5
Zinc Sulfide	ZnS	Sphalerite	3.9	3.5–4
Ferrous Titinate	$FeTiO_3$	Ilmenite	4.7	5.5
Silicon Dioxide	SiO_2	Quartz, sand, chert	2.65	7.0
Aluminosilicates:				
Feldspar	$(Na,K)\,AlSi_3O_8$	Albite, Microcline	2.6	6
Micas	$KAl_2(OH)_2AlSi_3O_{10}$	Muscovite	2.85	2.5
Clays	$Al_2Si_2O_5(OH)_4$	Kaolinite	2.6	2.0
	$(Al,Mg)\,(OH)_2Si_4O_{10}$ $(Na,Ca)_x \cdot 4H_2O$	Montmorillonite	2–3	1–2

d. Sodium (Na) and Potassium (K)—hydrochloric acid (HCl) and/or hydrofluoric/sulfuric/nitric acid ($HF/H_2SO_4/HNO_3$) extraction, then flame emission analysis. The only HCl insoluble Na and K compounds sometimes found in barite are Na and K feldspars, which, however, are soluble in $HF/H_2SO_4/HNO_3$ mixed acids. The difference in Na and K values obtained by these two extractions can give an estimate of Na and K feldspars in barite.

e. Calcium (Ca), Magnesium (Mg), and Iron (Fe)—hydrochloric acid (HCl) and/or hydrofluoric/sulfuric/nitric acid ($HF/H_2SO_4/HNO_3$) extraction, then atomic absorption (AA) analysis. Following are compounds of Ca, Mg, or Fe sometimes found in barite, that are insoluble in HCl, but soluble in the $HF/H_2SO_4/HNO_3$ mixture; the difference in Ca, Mg, and Fe values obtained by these two extractions can give estimates of these compounds in barite: fluorite (CaF_2), talc (hydrous magnesium silicate), montmorillonite (Mg is usually present in lattice structure), and pyrite (FeS_2).

f. Alternative Methods for Iron (Fe)—extract with nitric (HNO_3) or with hydrochloric nitric (HCl-HNO_3) acid, or fuse with carbonate, then determine by AA analysis.

g. Copper (Cu), Manganese (Mn), Lead (Pb), and Zinc (Zn)—hydrochloric acid (HCl) extraction, then AA analysis.

h. Siderite ($FeCO_3$)—EDTA—caustic extraction, then AA analysis.

i. Total Carbonate (CO_3)—3N perchloric acid treatment, then adsorption of evolved CO_2 by ascarite in a gas train.

j. Sulfide—determined by the Garrett Gas Train method; alternate procedure: treat with 1N HCl or 1N H_2SO_4, analyze H_2S evolved with lead acetate paper.

k. Water Soluble Analysis—barite is extracted with an equal weight of deionized water, and filtrate is analyzed for elements of interest by titration, AA, and flame emission techniques.

l. Loss on Ignition—weight loss after heating to 1000°C, due to combined water lost from clays, decomposition of carbonates or organic and carbonaceous matter.

m. Calcium hydroxide (lime) or cement content—extract with an aqueous solution of sugar, and the lime/cement content titrated with standard HCl.

3.2 BARIUM SULFATE AND STRONTIUM SULFATE

3.2.1 Description

This procedure determines barium sulfate ($BaSO_4$) and strontium sulfate ($SrSO_4$) in barites by fusion with sodium carbonate (Na_2CO_3), dissolution with hydrochloric acid (HCl), and measurement of barium (Ba) gravimetrically by sulfate precipitation, and strontium (Sr) by atomic absorption spectrophotometry.

3.2.2 Equipment

a. Mortar and pestle.

b. A 149-micrometer (μm) sieve.

c. Stirring rod, one end fitted with a rubber policeman.

d. Atomic absorption spectrophotometer: any atomic absorption unit is suitable. Instrument settings recommended by the manufacturer should be followed.

e. Analytical balance: precision of ± 0.001 gram (g).

f. Platinum crucibles and lids: two 25-cubic centimeters (cm^3).

g. Crucible tongs: one 10-inch (in.) (25-centimeter (cm)) or one 20-in. (51-cm).

h. Muffle furnace: regulated to 1000 ± 20°C.

i. Beakers: four 250-cm^3, two 400-cm^3, and two 600-cm^3.

j. Watch glasses: two for 250-cm^3 and two for 600-cm^3 beakers.

k. Hot plate.

l. Glass funnels: two 65-millimeter (mm), long stem.

m. Filter paper: 11.0-cm Whatman #40 or equivalent, 11.0-cm Whatman #541 or equivalent.

n. Volumetric flasks (TC): two 250-cm^3.

o. Annealing cups: two size #1 (12-cm^3, 33 mm × 35 mm).

p. Desiccator.

q. Volumetric pipette (TD): one 10-cm^3.

r. Sodium carbonate (CAS #497-19-8): anhydrous, ACS grade powder.

s. Sodium carbonate solution (0.2%): 1 g Na_2CO_3/500 cm^3.

t. Hydrochloric acid solution (CAS #7647-01-0) (1:4): 20 cm^3 HCl/80 cm^3 of water.

u. Hydrochloric acid solution (6N): 50 cm^3 HCl/50 cm^3 of water.

v. Sulfuric acid solution (CAS #7664-93-9) (1:19): 5 cm^3 H_2SO_4/95 cm^3 of water.

w. Sulfuric acid solution (1%): 5 cm^3 H_2SO_4/495 cm^3 water.

x. Strontium AA standards: 5, 10, 15 milligrams per liter (mg/L) Sr in 1% HCl.

y. Ammonium hydroxide (CAS #1336-21-6): concentrated, ACS reagent grade.

z. Methyl orange indicator solution (CAS #547-58-0): 0.1 g methyl orange/100 cm^3 of water.

3.2.3 Procedure

3.2.3.1 Use a representative sample ground such that 100 percent passes through the sieve.

3.2.3.2 Accurately weigh a 1.0 g sample on the analytical balance.

Note: It is advisable to perform this analysis in duplicate. If this is done, a slightly smaller sample (e.g., 0.8 g) of barite should be used to check accuracy and precision of the procedure.

3.2.3.3 Transfer sample to a 25-cm^3 platinum crucible containing 6 g Na_2CO_3. Mix thoroughly with stirring rod. Cover the mixture with an additional 2 g Na_2CO_3.

3.2.3.4 Fuse for 1 hour at 1000°C in the muffle furnace. Have a crucible lid on crucible during fusion.

Note: When beginning the fusion, the furnace may be hot or cold.

3.2.3.5 Remove from furnace with crucible tongs and while contents are still molten, give a slow rotary motion so that melt will spread over the sides and solidify as a thin shell over the interior. This procedure later facilitates the removal of the contents.

CAUTION: Use proper safety precautions while handling hot crucible and melt.

3.2.3.6 Allow to cool. Place crucible and lid in a 250-cm^3 beaker containing 150 cm^3 water. Digest on warm hot plate until melt has completely disintegrated and can easily be removed from crucible.

Note: Digesting overnight is preferable.

3.2.3.7 Remove crucible from beaker with rubber policeman and wash inside and out with water. Remove crucible lid and wash also.

3.2.3.8 Filter through Whatman #40 Filter Paper, or equivalent, transferring all of solids to filter paper. Wash filter paper and solids 12 times with hot 0.2% Na_2CO_3 solution. Discard filtrate.

Note: If Fe content is less than 5 percent, proceed to 3.2.3.10. If Fe content is more than 5 percent, then precipitation of iron hydroxide along with barium sulfate can cause an error. In this case, go to 3.2.3.9, which gives a method for removing Fe and Al.

3.2.3.9 Use the following method for removing iron and aluminum from barite.

3.2.3.9.1 Dissolve the carbonates with hot 6N HCl, catching the solution in a 250-cm^3 beaker. The funnel must be covered with a watch glass while adding the acid dropwise with a medicine dropper. Raise the watch glass just enough to insert the dropper.

Note: If Sr is to be run, catch solution in 250-cm^3 volumetric flask and dilute to volume mark. With a dry pipette remove a 10-cm^3 aliquot for Sr and transfer remainder into a 400-cm^3 beaker.

3.2.3.9.2 Remove Fe and Al by adding NH_4OH dropwise until fumes of NH_3 are given off.

3.2.3.9.3 Filter through Whatman #541 Filter Paper or equivalent, catching the filtrate in a 600-cm^3 beaker.

3.2.3.9.4 Redissolve the precipitate with 6N HCl, catching the filtrate in a 250-cm^3 beaker.

3.2.3.9.5 Reprecipitate the Fe and Al with NH_4OH and filter into 600-cm^3 beaker from 3.2.3.9.3.

3.2.3.9.6 Bring filtrate up to approximately 400 cm^3. Add a few drops of methyl orange and titrate with 6N HCl. Add 1 cm^3 excess 6N HCl. Filtrate should now be at the correct pH to precipitate $BaSO_4$ according to 3.2.3.14, 3.2.3.15, 3.2.3.16, and 3.2.3.17.

3.2.3.10 Dissolve the carbonates (Ba, Sr, Cr) from the paper with warm HCl solution (1:4), catching the solution in a 600-cm^3 beaker. The funnel must be covered with a watch glass while adding the acid *dropwise* with a medicine dropper. Raise the watch glass just enough to insert the dropper.

Note: If a strontium analysis is to be performed, the solution should be caught in a 250-cm^3 volumetric flask and, after washing filter paper, diluted to mark with distilled water. With a dry pipette, a 10-cm^3 aliquot is pipetted out for Sr determination by atomic absorption and the remainder transferred to a 600-cm^3 beaker for Ba determination.

3.2.3.11 After carbonates are dissolved, rinse the crucible and lid with hot 6N HCl and pour through filter paper.

Note: There may be a few solids undissolved, such as iron oxide, which may be disregarded.

3.2.3.12 Wash paper thoroughly with distilled water.

3.2.3.13 Dilute filtrate to about 400 cm^3 and boil using stirring rod instead of boiling chips.

3.2.3.14 To the boiling solution, add 10 cm^3 H_2SO_4 solution (1:19) *dropwise* and boil for 15 minutes. Allow to stand for at least 4 hours. Keep hot but do not boil.

3.2.3.15 Filter through Whatman #40 Filter Paper, or equivalent, transferring all of precipitate to filter paper. Wash 6 or 7 times with 1% H_2SO_4.

3.2.3.16 Place filter paper in a tared annealing cup. Heat slowly from cold to 850°C in the muffle furnace and hold at that temperature for 1 hour.

Note: The $BaSO_4$ should be white.

3.2.3.17 Remove from furnace, and cool to room temperature in desiccator. Reweigh the $BaSO_4$ precipitate and annealing cup on the analytical balance to the nearest 0.1 milligram (mg).

3.2.4 Procedure—Strontium Analysis

To determine strontium, analyze the 10-cm^3 aliquot (taken in 3.2.3.9, see note) for Sr on an AA spectrophotometer using prepared standards for Sr and manufacturer's recommendations for AA instrument settings.

3.2.5 Calculation

3.2.5.1 If no Sr is present, the following formula is used:

$$BaSO_4,\ \% = \frac{100\ (\text{weight of precipitate (ppt), g})}{\text{weight of sample, g}} \qquad (1)$$

Note: If a 10-cm^3 aliquot was taken for Sr determination, use 104.17 instead of 100 in equation (1).

3.2.5.2 If Sr is present, the following formula is used to calculate the amounts of $BaSO_4$ and $SrSO_4$ that are coprecipitated:

$$BaSO_4 + SrSO_4,\ \% = \frac{100\ (\text{weight of ppt, g})}{\text{weight of sample, g}} \qquad (2)$$

Note: If a 10-cm^3 aliquot was taken for Sr determination, use 104.17 instead of 100 in equation (2).

$$SrSO_4,\ \% = 2.0963\ (\%\ \text{Sr, from AA}) \qquad (3)$$

Note: Compare this amount of $SrSO_4$ to correction curve in Figure 1 to obtain correction used in calculation.

$$BaSO_4,\ \% = \%\ (BaSO_4 + SrSO_4)\ (\text{coppt}) - \text{correction factor} \qquad (3a)$$

3.3 SILICA AND ALUMINA

3.3.1 Description

Silica and alumina occur in barite ores mostly as aluminosilicates (for example, clay, feldspars, micas, and so forth) and quartz. These two oxides are determined by fusing the barite with NaOH and then measuring the Al_2O_3 and SiO_2 colorimetrically.

3.3.2 Equipment

a. Ultraviolet (UV)/visible spectrophotometer: any UV/visible spectrophotometer with photometric precision of 0.001 absorbance is suitable. Use 1-cm glass or quartz cells.
b. Graduated cylinder, plastic: one 10-cm^3.
c. Nickel crucibles and lids: two 75-cm^3.
d. Hot plate.
e. Analytical balance: precision of ± 0.0001 g.
f. Bunsen burner, tripod, and triangle.
g. Beakers: four or more 600-cm^3.
h. Stirring rods: plastic or PTFE (Teflon® or equivalent).
i. Volumetric flasks (TC): one 1000-cm^3 and one 100-cm^3.
j. Volumetric pipettes (TD): one 10-cm^3, one 2-cm^3, one 5-cm^3, and one 10-cm^3.
k. Serological pipettes (TD): one 1-cm^3, one 2-cm^3, one 5-cm^3, and one 10-cm^3.
l. Graduated cylinder (TC): one 10-cm^3.
m. Ammonium molybdate solution (CAS #12027-67-7): 7.5 g of $[(NH_4)_6Mo_7O_{24} \cdot 4H_2O]$ with 75 cm^3 water, 25 cm^3 of 20% H_2SO_4 and mix. Store in plastic bottle.
n. Tartaric acid solution (CAS #87-69-4) (10%): 50 g tartaric acid/450 cm^3 of water. Store in plastic bottle.
o. Reducing solution: 0.7 g sodium sulfite (CAS #7757-83-7), 10 cm^3 water. Add 0.15 g 1-amino-2-naphthol-4-sulfonic acid (CAS #90-51-7) and stir until dissolved. Dissolve 9 g of sodium bisulfite (CAS #7631-90-5) in 90 cm^3 of water and add this solution to the first solution and mix.

Note: This solution is not stable, and should be prepared fresh just prior to use.

p. Hydroxylamine hydrochloride solution (CAS #5470-11-1) (10%): 50 g $NH_2OH \cdot HCl$/450 cm^3 water.

Note: This solution is not stable, and should be prepared fresh just prior to use.

q. Calcium chloride (CAS #10043-52-4) solution: transfer 7 g $CaCO_3$ to a 250-cm^3 beaker, add 100 cm^3 water and 15

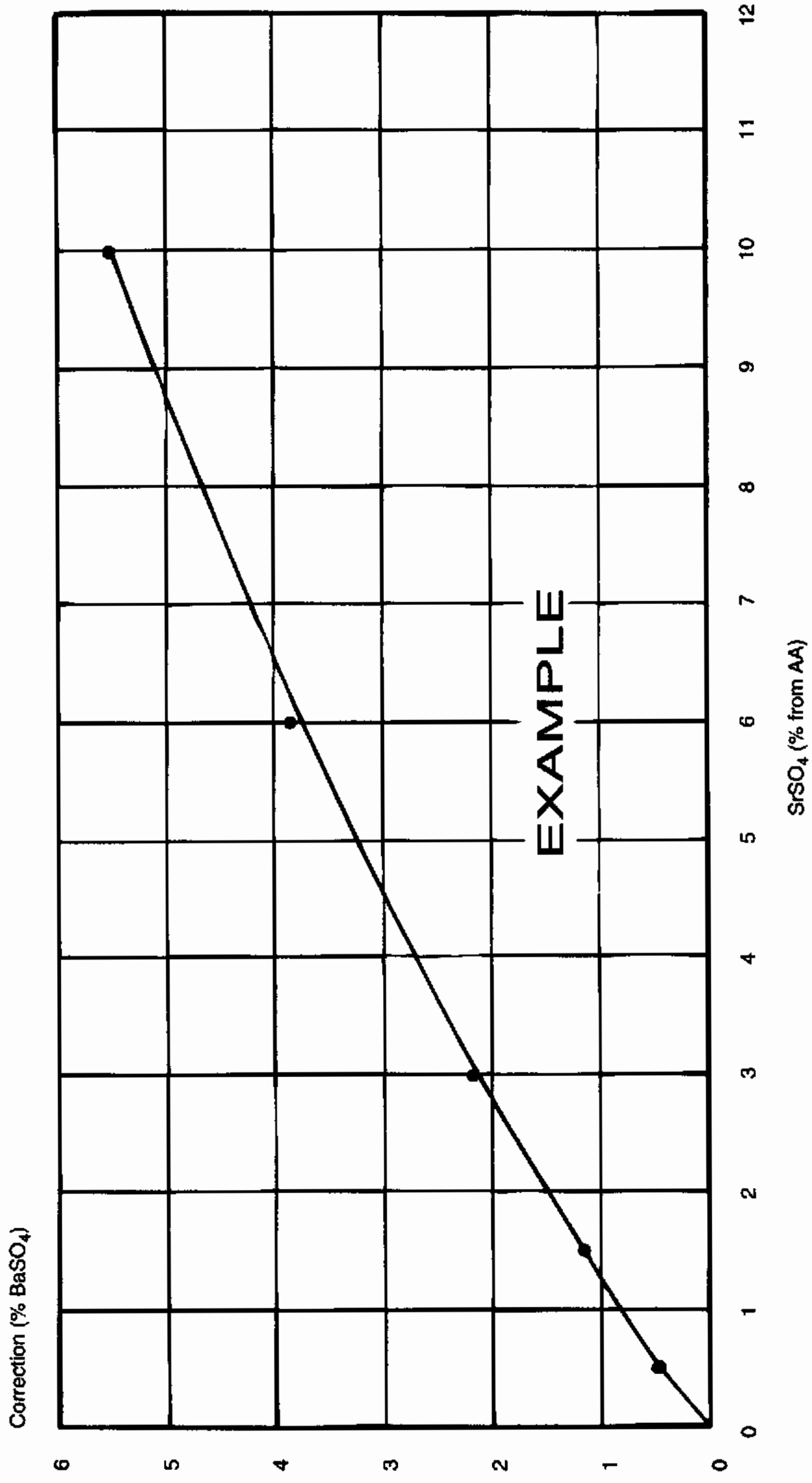

Figure 1—Correction Curve (for $SrSO_4$ in $BaSO_4$)

cm^3 concentrated HCl. Heat to boiling and boil for a few minutes. Cool to room temperature and dilute to 500 cm^3.
r. Potassium ferricyanide (CAS #13746-66-2) solution (0.75%): add 0.375 g $K_3Fe(CN)_6$ to 50 cm^3 water just before using. DO NOT STORE.

CAUTION: Potassium ferricyanide is quite toxic; avoid contact or any chance of ingestion.

s. Buffer solution: 100 g sodium acetate (CAS #127-09-3) ($NaC_2H_3O_2{:}H_2O$)/200 cm^3 water, add 30 cm^3 glacial acetic acid (CAS #64-19-7) and dilute to 500 cm^3 with water.

CAUTION: Avoid skin contact with glacial acetic acid.

t. Alizarin Red S (CAS #130-22-3) solution (0.05%): 0.25 g Alizarin Red S (sodium alizarine sulfonate)/500 cm^3 of water, stir, and filter.
u. Thioglycolic acid solution (CAS #68-11-1) (4%): 4 g $HSCH_2COOH$/96 cm^3 of water just before using. DO NOT STORE.
v. Sodium hydroxide (CAS #1310-73-2) solution (30%): 30 g NaOH/70 cm^3 of water. Store in plastic bottle.
w. Hydrochloric acid (CAS #7647-01-0) solution (6N): 50 cm^3 HCl/50 cm^3 water.
x. National Institute of Standards and Technology sample number 99 (feldspar).

3.3.3 Procedure—Sample Preparation

3.3.3.1 Transfer 5-cm^3 portions of 30% NaOH solution measured with a plastic graduated cylinder to a series of 75-cm^3 nickel crucibles. One crucible will be needed for each sample, two for standards, and one for a blank.

Note: Each crucible should be cleaned with diluted HCl before use.

3.3.3.2 Evaporate the NaOH solution to dryness on a hot plate.

3.3.3.3 Accurately weigh a 0.10- to 0.15-g sample of each barite and transfer to a crucible containing NaOH. Use 50-mg portions for standards.

3.3.3.4 Cover and heat the crucibles over Bunsen burner to a dull redness for about 5 minutes. Remove each crucible from the heat and swirl the melt around the sides. Allow melt to cool.

CAUTION: This hot melt is potentially harmful, so use safety precautions while working with it.

3.3.3.5 Add 50 cm^3 of water to each crucible, cover, and allow to stand until melt disintegrates completely. Time can vary from one hour, if solutions are stirred occasionally, to overnight.

3.3.3.6 Rinse each 600-cm^3 beaker with 6N HCl. Place in each beaker an acid solution of 400 cm^3 water and 20 cm^3 of 6N HCl.

3.3.3.7 Transfer the contents of each crucible to a 600-cm^3 beaker prepared in 3.3.3.6. PTFE stirring rod should be used and care should be taken so that the alkaline solutions will not contact the side of the beaker but drain directly into the acid solution. Police and wash each crucible.

3.3.3.8 Transfer contents of each beaker to a 1000-cm^3 volumetric flask and dilute to mark with deionized water. The solutions containing barite will be cloudy.

3.3.3.9 Centrifuge a 40- to 50-cm^3 portion to obtain clear solution for SiO_2 and Al_2O_3 analyses. If time permits, allow samples to settle and pipette aliquots from top of sample.

Note: If solutions are not to be used the same day, they should be transferred to tightly stoppered plastic bottles for storage, to avoid evaporation and possible contamination with silica from the glass flask.

3.3.4 Procedure—Silica Determination

3.3.4.1 Rinse a 100-cm^3 volumetric flask for each sample prepared in 3.3.3 with 6N HCl followed by water before using.

3.3.4.2 Transfer 10 cm^3 of each solution prepared in 3.3.3 to volumetric flasks. Add approximately 50 cm^3 deionized water to each flask.

3.3.4.3 Add 2 cm^3 ammonium molybdate solution with a pipette. Swirl the flask during the addition. Allow to stand for 10 minutes.

3.3.4.4 Add 4 cm^3 tartartic acid solution with a pipette. Swirl the flask while adding.

3.3.4.5 Add 1 cm^3 reducing solution with a pipette. Swirl the flask while adding.

3.3.4.6 Dilute to volume with deionized water. Mix well and allow to stand for at least 30 minutes. Fresh reducing solution should be used each time the test is run.

3.3.4.7 Determine the absorbance for each solution at 640 nanometers (nm). Use the reagent blank solution as the reference. Record the value for each solution.

3.3.5 Procedure—Alumina Determination

3.3.5.1 Transfer 15 cm^3 of each solution prepared in 3.3.3 to 100-cm^3 volumetric flasks.

3.3.5.2 Add 2 cm^3 calcium chloride solution to each flask.

3.3.5.3 Add 1 cm^3 of 10% hydroxylamine hydrochloride solution to each flask and swirl to mix.

3.3.5.4 Add 1 cm^3 potassium ferricyanide solution and swirl to mix.

3.3.5.5 Add 2 cm^3 thioglycolic acid solution (mercaptoacetic acid) and mix. Allow to stand 5 minutes.

3.3.5.6 Add 10 cm^3 Alizarin Red S solution with a pipette.

3.3.5.7 Dilute to mark with deionized water. Mix and allow to stand 45 to 75 minutes.

3.3.5.8 Determine the absorbance at 475 nm for each solution. Use the reagent blank solution as a reference. Record the value for each solution.

3.3.6 Calculation

3.3.6.1 Calculate percent silica (SiO_2):

$$\text{Factor} = \frac{(SiO_2 \text{ in Std., \%}) (\text{Std. Wt. in Soln., g})}{\text{Absorbance of Std.}} \quad (4)$$

$$SiO_2\,\% = \frac{(\text{Factor}) (\text{Absorbance of Sample})}{\text{Sample Wt., g}} \quad (5)$$

3.3.6.2 Calculate percent alumina (Al_2O_3):

$$\text{Factor} = \frac{(Al_2O_3 \text{ in Std., \%}) (\text{Std. Wt. in Soln., g})}{\text{Absorbance of Std.}} \quad (6)$$

$$Al_2O_3\,\% = \frac{(\text{Factor}) (\text{Absorbance of Sample})}{\text{Sample Wt., g}} \quad (7)$$

3.4 HYDROCHLORIC ACID SOLUBLE METALS (Na, K, Ca, Mg, Fe, Cu, Mn, Pb, and Zn)

3.4.1 Description

3.4.1.1 The hydrochloric acid soluble metals usually analyzed in barite are sodium, potassium, calcium, magnesium, iron, copper, manganese, lead, and zinc. Most of the compounds of these metals found in barite are soluble under the conditions of this test.

3.4.1.2 Exceptions to acid solubility are pyrite (FeS_2), and feldspars of sodium and potassium. Fluorite (CaF_2) slowly dissolves but may not be completely dissolved during the digestion time called for in the procedure. The magnesium in montmorillonite and talc is also not analyzed by this procedure. For a total analysis of all these metals, see 3.5: Hydrofluoric, Sulfuric, Nitric, Perchloric Acid Soluble Metals.

3.4.2 Equipment

a. A 149-μm sieve.
b. Atomic absorption spectrophotometer: any atomic absorption unit is suitable. Instrument settings recommended by the particular manufacturer should be followed.
c. Analytical balance: precision of ± 0.001 g.
d. Graduated cylinder (TD): one 25-cm^3.
e. Beakers: ten 250-cm^3.
f. Hot plate/magnetic stirrer.
g. Stirring bar.
h. Deionized or distilled water.
i. Volumetric flasks: ten 100-cm^3.
j. Atomic absorption standards prepared in 1% HCl.
k. Hydrochloric acid (CAS #7647-01-0): ACS reagent grade.

3.4.3 Procedure

3.4.3.1 Use a representative sample of barite ground such that 100 percent passes through the sieve.

3.4.3.2 Weigh a 1.0- to 1.5-g sample into a 250-cm^3 beaker.

3.4.3.3 Add 20 cm^3 concentrated HCl. Place a magnetic stirring bar into beaker, and cover with a watch glass.

3.4.3.4 Boil while stirring for 30 minutes. Add water 2 or 3 times during this period to maintain 20-cm^3 volume.

3.4.3.5 Cool and transfer to a 100-cm^3 volumetric flask.

3.4.3.6 Dilute to mark with deionized water.

3.4.3.7 Allow solids to settle. If solids do not settle, filter or centrifuge the sample.

3.4.3.8 Analyze the clear supernate using an AA spectrophotometer. Run the standards prepared in 1% HCl. Record all values for the solutions.

3.4.4 Calculation

The concentration of each metal is calculated by multiplying the results obtained on the AA spectrophotometer by the correction factor for the dilution used (dilution factor), corrected for weight of sample.

$$\text{Metal in sample, ppm} = \frac{100\ (\text{AA value, mg/L})}{\text{Sample Wt., g}} \quad (8)$$

3.5 HYDROFLUORIC, SULFURIC, NITRIC, PERCHLORIC ACID SOLUBLE METALS (Na, K, Ca, Mg, Fe, Cu, Mn, Pb, and Zn)

3.5.1 Description

The hydrofluoric/sulfuric/nitric/perchloric acid soluble metals are sodium, potassium, calcium, magnesium, iron, copper, manganese, lead, and zinc.

This acid extraction dissolves all minerals in barite containing these metals, including pyrite, feldspars, fluorite, talc, and clays. By comparing the results of this analysis to the results of the hydrochloric acid soluble metals (see 3.4), an approximation of the pyrite, feldspars, etc., in the barite sample may be made.

3.5.2 Equipment

a. A 149-μm sieve.
b. Analytical balance: precision of ± 0.001 g.
c. Atomic absorption spectrophotometer: any atomic absorption unit is suitable. Instrument settings recommended by the particular manufacturer should be followed.
d. Graduated cylinder (TD): one 25-cm^3.
e. Beakers: ten 100-cm^3 PTFE (Teflon®, or equivalent) beakers with covers.
f. Hot plate/magnetic stirrer.
g. Stirring bar.
h. Beakers: ten 400-cm^3.
i. Stirring rod, one end fitted with a rubber policeman.
j. Dropping bottle: one 25-cm^3.

k. Volumetric flasks: ten 250-cm^3.
l. Polyethylene bottle: one 1000-cm^3.
m. Fume hood.
n. Atomic absorption standards prepared in 1% HCl.
o. Deionized or distilled water.
p. Nitric acid (CAS #7697-37-2): ACS reagent grade (70%).

CAUTION: Concentrated nitric acid is a strong, potentially harmful acid. Use proper safety precautions.

q. Acid mixture A: transfer 454 g (392 cm^3) of hydrofluoric acid (CAS #7664-39-3) (48%) to a 1000-cm^3 polyethylene bottle. Cool the bottle in ice water. While the bottle is in the ice water, add 165 cm^3 sulfuric acid (CAS #7664-93-9) (98%) mix, and allow to cool. Add 40 cm^3 nitric acid (CAS #7697-37-2) (70%) and mix.

CAUTION: Strong acid mixture; use all proper safety precautions.

CAUTION: Hydrofluoric acid is quite corrosive and toxic. Avoid skin contact, wear protective clothing and eye/face protection, and work under a fume hood.

CAUTION: Perchloric acid is a strong acid that can cause skin burns; it is also toxic. It can also form explosive perchlorates when reacted with other chemicals. Use eye protection and work under a fume hood.

CAUTION: Concentrated sulfuric acid is very dangerous—avoid any contact with skin or clothing, and wear protective eye wear, face wear, and garments.

CAUTION: Strong acid mixture; use all proper safety precautions.

r. Acid mixture B: mix 100 cm^3 nitric acid (70%) and 100 cm^3 perchloric acid (CAS #7601-90-3) (72%).
s. Hydrazine sulfate (CAS #10034-93-2) solution (0.2%): 0.2 g hydrazine sulfate/100 cm^3 of water.

3.5.3 Procedure

3.5.3.1 Use a representative sample of barite ground such that 100 percent passes through the sieve.

3.5.3.2 Accurately weigh a 0.5-g sample on the analytical balance and transfer into a 100-cm^3 PTFE (Teflon® or equivalent) beaker.

3.5.3.3 Under a fume hood, add 15 cm^3 of acid mixture A (3.5.2, Item q). Swirl the beaker to wet the sample and cover the beaker with PTFE cover.

3.5.3.4 Place on the hot plate/stirrer, set on low heat, and allow to heat overnight (16 hours).

3.5.3.5 Remove cover from the beaker and turn heat on hot plate/stirrer to about 212–302°F (100–150°C). Heat for 1 hour or until fumes (of HF) are no longer released.

3.5.3.6 Cool and transfer to a 400-cm^3 beaker, using a rubber policeman and a minimum of deionized water.

3.5.3.7 Place on the hot plate/stirrer set on medium heat about 212–302°F (100–150°C).

3.5.3.8 Heat until fumes (of SO_3) start to evolve, then remove beaker from hot plate/stirrer.

3.5.3.9 After SO_3 fumes have stopped evolving, add about 4 drops of acid mixture B (3.5.2, Item r) using the dropping bottle.

3.5.3.10 Replace the beaker on the hot plate/stirrer and heat until strong fumes evolve and any color due to organic matter has disappeared.

3.5.3.11 Remove beaker from hot plate and allow to cool for a few minutes. Then add 225 cm^3 water, 5 cm^3 HNO_3, and 1 cm^3 of 0.2% hydrazine sulfate solution.

3.5.3.12 Replace on the hot plate/stirrer and heat to boiling. If a brown precipitate of MnO_2 remains after the solution has boiled for a few minutes, add an additional 1 cm^3 of 0.2% hydrazine sulfate.

Note: The residue remaining should consist of $BaSO_4$ and $SrSO_4$, and should be white.

3.5.3.13 Cool to room temperature. Then transfer contents of beaker to a 250-cm^3 volumetric flask, dilute to volume with deionized water, and mix.

3.5.3.14 Allow the solids to settle. If solids do not settle, filter or centrifuge the sample.

3.5.3.15 Analyze the clear supernate using an AA spectrophotometer. Run the standards prepared in 1% HCl. Record all values for the solutions.

3.5.4 Calculation

The concentration of each metal is calculated by multiplying the results obtained on the AA spectrophotometer by the correction factor for the dilution used (dilution factor), corrected for the weight of sample.

$$\text{Metal in sample, ppm} = \frac{100\ (\text{AA value, mg/L})}{\text{Sample Wt., g}} \qquad (9)$$

3.6 ALTERNATIVE METHODS FOR IRON

3.6.1 Description

Several procedures other than those given in 3.4 and 3.5 are commonly used to determine iron in barite ore bodies. The three procedures given below include two that determine acid soluble iron (one in nitric acid, the second in aqua regia) and one that determines total iron, including lattice-substituted iron (carbonate fusion).

3.6.2 Equipment

a. Acid digestion bomb—PTFE (Teflon® or equivalent) lined, 25-cm^3 (Parr bomb or equivalent).

b. Analytical balance: precision of ± 0.0001 g.
c. Platinum crucibles and lids: two 25-cm^3.
d. Crucible tongs: one 10-in (25-cm).
e. Filter paper: 11.0-cm Whatman #40, or equivalent; 11.0-cm Whatman #42 or #44, or equivalent.
f. Muffle furnace: regulated to 1000 ± 20°C.
g. Oven: regulated to 176 ± 2°F (80 ± 1°C).
h. Volumetric flasks: five 25-cm^3.
i. Volumetric flasks: five 100-cm^3.
j. Beakers: five 250-cm^3.
k. Filter: 2.5-cm glass fiber.
l. Hot plate.
m. Graduated cylinder (TD): one 25-cm^3.
n. Sodium carbonate (CAS #497-19-8): ACS reagent grade.
o. Potassium carbonate (CAS #584-08-7): ACS reagent grade.
p. Atomic absorption spectrophotometer: any atomic absorption unit is suitable. Instrument settings recommended by the manufacturer should be followed.
q. A 149-μm sieve.
r. Deionized or distilled water.
s. Sodium carbonate (CAS #497-19-8) solution (0.2%):1 g Na_2CO_3/500 cm^3 of water.
t. Hydrochloric acid (CAS #7647-01-0): ACS reagent grade.

CAUTION: Strong acid; use proper safety precautions.

u. Nitric acid (CAS #7697-37-2): ACS reagent grade.

CAUTION: Strong acid; use proper safety precautions.

v. Aqua regia: 30 cm^3 HNO_3/90 cm^3 HCl.

CAUTION: Strong acid; use proper safety precautions.

w. Hydrochloric acid solution (6N): 50 cm^3 HCl/50 cm^3 of water.
x. Iron AA standards: prepared in 1% HCl.
y. Iron AA standards: prepared in diluted aqua regia (15 cm^3 aqua regia diluted to 25 cm^3 with water).
z. Iron AA standards: prepared in 1.36N nitric acid (2 cm^3 HNO_3 diluted to 25 cm^3 with water).

3.6.3 Procedure—Nitric Acid Digestion

3.6.3.1 Use a representative sample of barite ground such that 100 percent passes through the sieve.

3.6.3.2 Weigh a 50-mg sample into a 25-cm^3 acid digestion bomb

3.6.3.3 Add 2 cm^3 nitric acid. Cover the bomb, and mount inside the steel shell. Tighten steel lid onto the bottom section.

CAUTION: This is a strong acid, and proper safety precautions should be used when handling it, particularly when it is used in a digestion bomb.

3.6.3.4 Heat for 1½ to 2 hours in oven at 176°F (80°C).

3.6.3.5 Cool, reheat at 176°F (80°C) for 1½ hours, and cool again to room temperature.

3.6.3.6 Carefully open the bomb and dilute with deionized water by spraying a few cm^3 water on the inner walls of the PTFE (Teflon® or equivalent) cell.

3.6.3.7 Filter through Whatman #42 or #44 filter paper or equivalent, into a 25-cm^3 volumetric flask. Wash sparingly with deionized water to effect a quantitative transfer. Dilute to mark with water.

3.6.3.8 Analyze for iron using an AA spectrophotometer. Run the standards prepared in 1.36N nitric acid. Record all values.

3.6.4 Procedure—Aqua Regia Digestion

3.6.4.1 Use a representative sample of barite ground such that 100 percent passes through the sieve.

3.6.4.2 Weigh a 2.0-g sample into a 25-cm^3 acid digestion bomb.

3.6.4.3 Add 15 cm^3 freshly prepared aqua regia. Put PTFE (Teflon® or equivalent) cover on the bomb and mount inside the steel shell. Seal by screwing the steel lid onto the bottom section.

CAUTION: This is a strong acid, and proper safety precautions should be used when handling it, particularly when it is used in a digestion bomb.

3.6.4.4 Heat for 1½ hours in oven at 176°F (80°C).

3.6.4.5 Cool to room temperature.

3.6.4.6 Carefully open the bomb and filter the contents quantitatively through a 2.5-cm glass fiber filter into a 25-cm^3 volumetric flask.

3.6.4.7 Wash bomb, filter, and dilute to the mark with water.

3.6.4.8 Analyze for iron using the AA spectrophotometer. Run standards prepared in diluted aqua regia.

3.6.5 Procedure—Carbonate Fusion

3.6.5.1 Use a representative sample of barite ground such that 100 percent passes through the sieve.

3.6.5.2 Weigh a 0.5- to 1.0-g sample into a 25-cm^3 platinum crucible. Mix with either 2 g sodium carbonate/1 g potassium carbonate mixture, or with 3 g sodium carbonate alone. Add another 2 g of carbonates to cover the mixture.

3.6.5.3 Cover with a platinum lid and fuse for 1 hour at 1000°C in the muffle furnace.

3.6.5.4 Remove the crucible with crucible tongs while hot and swirl gently to spread melt evenly inside the crucible.

CAUTION: Use proper safety precautions while handling hot crucible and melts.

3.6.5.5 Cool crucible, and place it in a 250-cm^3 beaker. Add sufficient deionized water to cover crucible.

3.6.5.6 Place beaker on a hot plate, and digest several hours on low heat until melt has completely disintegrated and separated from the platinum crucible.

3.6.5.7 Completely rinse the crucible and lid with water, combining the wash with the solution in the beaker.

3.6.5.8 Filter through Whatman #40 or equivalent filter paper. Wash filter paper and solids residue thoroughly with hot 0.2% sodium carbonate solution.

3.6.5.9 Dissolve the solid residue by adding hot 6N hydrochloric acid to the filter paper and filtering into a 100-cm^3 volumetric flask.

3.6.5.10 Wash filter paper with water and dilute to volume mark.

3.6.5.11 Analyze for iron using the AA spectrophotometer. Run standards prepared in 1% HCl. Record all values.

3.6.6 Calculations

3.6.6.1 For nitric acid digestion,

$$\% \text{ Fe} = \frac{2.5 \text{ (AA value, mg/L)}}{\text{Sample Wt., mg}} \qquad (10)$$

3.6.6.2 For aqua regia digestion,

$$\% \text{ Fe} = \frac{2.5 \text{ (AA value, mg/L)}}{\text{Sample Wt., mg}} \qquad (11)$$

3.6.6.3 For carbonate fusion,

$$\% \text{ Fe} = \frac{10 \text{ (AA value, mg/L)}}{\text{Sample Wt., mg}} \qquad (12)$$

3.7 WATER-SOLUBLE MATERIALS IN BARITE

3.7.1 Description

The water-soluble material in most barite is less than 0.1 percent (1000 ppm) and consists mostly of the chlorides, sulfates, and carbonates of Na and K and of the chlorides and sulfates of Mg and Ca. As far as the use of barite in drilling fluids is concerned, only the soluble alkaline earth metals (primarily Mg and Ca) are of essential importance. Specification for Ca and Mg reported as Ca is 250 ppm maximum. Soluble Zn, Mn, Cu, Pb, and Cr are often determined for environmental purposes.

3.7.2 Equipment

a. A 149-μm sieve.
b. Atomic absorption spectrophotometer: any atomic absorption unit is suitable. Instrument settings recommended by the particular manufacturer should be followed.
c. Analytical balance: precision to ± 0.01 g.
d. Mechanical shaker, or magnetic stirrer or field mixer with powerstat.
e. Graduated cylinder (TD): one 100-cm^3.
f. Erlenmeyer flask: one 250-cm^3.
g. API low-pressure filter cell.
h. Volumetric flask: one 200-cm^3.
i. Deionized or distilled water.
j. Cork or rubber stopper, to fit Erlenmeyer flask.

3.7.3 Procedure for Calcium, Magnesium, Zinc, Manganese, Copper, Lead, Chromium, Sodium, and Potassium

3.7.3.1 Use a representative sample of barite ground such that 100 percent passes through the sieve.

3.7.3.2 Weigh 100 ± 0.05 g barite sample and transfer to a 250-cm^3 Erlenmeyer flask.

3.7.3.3 Add 100 cm^3 of deionized water.

3.7.3.4 Stopper and shake on a mechanical shaker for 30 minutes.

3.7.3.5 Filter the suspension using an API low-pressure filter cell (as described in Recommended Practice 13B-1, Water-Based Drilling Fluids, Section 3) into a graduated cylinder.

Note: Filtrate is also used for chlorides, sulfates, carbonates, bicarbonates, hydroxyls, and phosphates. Procedures follow in 3.8 through 3.11.

3.7.3.6 Transfer filtrate to volumetric flask, wash sides of graduated cylinder, transferring washings to flask and make up to volume. Mix well.

3.7.3.7 Analyze the filtrate for calcium, magnesium, zinc, manganese, copper, lead, and chromium using an AA spectrophotometer.

3.7.3.8 Analyze the filtrate for sodium and potassium using a flame photometer or an AA spectrophotometer in the flame emission mode.

Notes:
1. Make the necessary dilutions, pH adjustments, and matrix matching between samples and standards.
2. For very low metal concentrations, it may be necessary to use a graphite furnace.

3.7.4 Calculation

$$\text{Metal in sample, ppm} = \frac{100 \text{ (AA value, mg/L)}}{\text{Sample Wt., g}} \qquad (13)$$

Note: If dilution was made in analyzing the samples, multiply the results on the AA spectrophotometer by the correction factor for the dilution used.

3.8 WATER-SOLUBLE CHLORIDES

3.8.1 Description

The water-soluble chlorides are measured by silver nitrate titration.

3.8.2 Equipment

a. Silver nitrate (CAS #7761-88-8) solution (0.0282N): dissolve 4.7910 g ACS reagent grade silver nitrate (equivalent to 0.001 g chloride-ion/cm^3) in deionized water and make up the volume to 1 liter (L). Store in an amber or opaque bottle.
b. Potassium chromate (CAS #7789-00-6) indicator solution: 5 g K_2CrO_4/100 cm^3 of water.
c. Sulfuric (CAS #7664-93-9) or nitric acid (CAS #7697-37-2) solution: standardized 0.02 normal (N/50).
d. Phenolphthalein indicator (CAS #77-09-8) solution: 1 g phenolphthalein/100 cm^3 of 50% ethyl alcohol/water solution.
e. Deionized or distilled water.
f. Serological (graduated) pipettes (TD): one 1-cm^3 and one 10-cm^3.
g. Titration vessel: 100–150-cm^3, preferably white.
h. Stirring rod or magnetic stirrer.
i. Buret: one 10-cm^3.

3.8.3 Procedure

3.8.3.1 Pipette one or more cm^3 of filtrate into the titration vessel.

3.8.3.2 Add 2–3 drops of phenolphthalein solution. If the indicator turns pink, add 0.02N sulfuric acid drop by drop from a pipette, while stirring, until the color is discharged.

3.8.3.3 Add 25 cm^3 of deionized water and 5–10 drops of potassium chromate indicator.

3.8.3.4 While stirring continuously, titrate the solution slowly (dropwise) with standard silver nitrate solution until the color changes from yellow to orange-red and persists for 30 seconds. Record the volume of silver nitrate solution required to reach the endpoint.

Note: Adjust the filtrate volume used in analysis so that the volume of silver nitrate used in titration is between 1 and 10 cm^3.

3.8.4 Calculation

$$\text{Chloride, ppm} = \frac{10^5\,(\text{Volume of Silver Nitrate, cm}^3)}{(\text{Volume of Filtrate, cm}^3)} \quad (14)$$

3.9 WATER-SOLUBLE SULFATES

3.9.1 Description

Water-soluble sulfates may be analyzed turbidimetrically using the reaction of soluble sulfates with barium chloride.

3.9.2 Equipment

a. Spectrophotometer, filter photometer, or turbidimeter. Use spectrophotometer at a wave length of 420 nm.
b. Hydrochloric acid (CAS #7647-01-0): ASC reagent grade.
c. Hydrochloric acid solution (6N): 50 cm^3 HCl/50 cm^3 of water.
d. Sodium sulfate, anhydrous (CAS #7757-82-6): ACS reagent grade.
e. Barium chloride (CAS #10361-37-2) crystals.

CAUTION: Barium chloride is extremely toxic. Avoid any contact with it that could lead to ingestion.

f. Volumetric flasks: one 1000-cm^3, four 100-cm^3.
g. Beakers: seven 50-cm^3.
h. Graduated cylinder (TD): one 10-cm^3.
i. Timer.
j. Pipettes: one 10-cm^3, one 5-cm^3, one 20-cm^3.
k. Cuvettes or appropriate container for transmission or turbidimetric analysis.
l. Graph paper, linear.
m. Ceramic spoon: Coors No. 65001, or equivalent.

3.9.3 Procedure

3.9.3.1 Preparation of Calibration Curve

3.9.3.1.1 Weigh out 0.1480 g sodium sulfate. Transfer quantitatively to a 1000-cm^3 volumetric flask. Add about 100 cm^3 deionized water, mix to dissolve, and dilute to the mark. Mix well. This is the stock solution containing 100 mg/L SO_4.

3.9.3.1.2 Prepare 20-, 40-, 60-, and 80-mg/L SO_4 standard solutions by pipetting 20, 40, 60, and 80 cm^3 of stock solution into separate 100-cm^3 volumetric flasks and making up to volume with deionized water. Mix well.

3.9.3.1.3 Pipette 15 cm^3 of each standard and the stock solution into separate dry, clean 50-cm^3 beakers. Add 1 cm^3 6N HCl to each solution. Swirl gently to mix.

3.9.3.1.4 Add one spoonful (0.3 g) of barium chloride crystals to each beaker. Swirl gently to mix.

3.9.3.1.5 Let samples stand 4 minutes.

3.9.3.1.6 Prepare a reagent blank by pipetting 15 cm^3 deionized water into another clean, dry 50-cm^3 beaker, add 1 cm^3 6N HCl, and swirl to mix. Add one spoonful (0.3 g) of barium chloride crystals. Swirl to mix.

3.9.3.1.7 Transfer the blank solution to a sample cell, insert cell in instrument, and set the absorbance to zero.

3.9.3.1.8 Rinse and fill cell with first standard solution (20 mg/L), insert cell in instrument, and record the absorbance reading.

3.9.3.1.9 Repeat 3.9.3.1.8 with the other standard solutions.

3.9.3.1.10 Plot the absorbance readings versus mg/L SO_4, using a linear graphing paper.

3.9.3.2 Analysis for Water-Soluble Sulfates

3.9.3.2.1 Pipette 15 cm^3 of sample (barite water leachate) into a clean 50-cm^3 beaker. Add 1 cm^3 6N HCl and one spoonful of barium chloride crystals. Swirl gently to mix.

3.9.3.2.2 Let sample stand for 4 minutes.

3.9.3.2.3 Transfer the solution to cell, insert cell in instrument, and record the absorbance reading.

3.9.3.2.4 Use the calibration chart to convert the absorbance reading to mg/L SO_4. If sample concentration is higher than highest standard, make the proper dilution and repeat the analysis.

3.9.4 Calculation

$$\text{Sulfate, ppm} = \frac{100\ (\text{mg/L } SO_4)}{\text{Sample Wt., g}} \qquad (15)$$

Note: Multiply mg/L SO_4 by dilution factor, if needed.

3.10 WATER-SOLUBLE CARBONATES, BICARBONATES, AND HYDROXYL IONS

3.10.1 Description

Carbonates, bicarbonates, and hydroxyl ion concentrations are determined from the amount of standard acid required to titrate a given amount of barite water leachate to the phenolphthalein and methyl purple endpoints.

3.10.2 Equipment

a. Sulfuric acid (CAS #7664-93-9) solution: standardized 0.02 Normal (N/50).

b. Phenolphthalein indicator (CAS #77-09-8) solution: 1 g phenolphthalein/100 cm³ of a 50% alcohol/water solution.

c. Methyl purple indicator (CAS #1340-02-9) solution (0.1 g methyl purple/100 cm³ of water).

d. pH meter (optional).

e. Titration vessel: 100–150-cm³, preferably white.

f. Serological (graduated) pipette (TD): one 10-cm³.

g. Volumetric pipettes: one 10-cm³ and one 25-cm³.

h. Stirring rod or magnetic stirrer.

3.10.3 Procedure

3.10.3.1 Pipette 10 cm³ of filtrate from 3.7.3.5 into an appropriate-size beaker. (Use a 25 cm³ sample, if necessary.)

3.10.3.2 Add 2 drops of phenolphthalein indicator and titrate to the colorless endpoint (pH 8.3 with a pH meter is more accurate) with 0.02N H_2SO_4. Record the number of cm³ of acid required as P.

3.10.3.3 Add 2 drops of methyl purple indicator and, without refilling the pipette, continue the titration to the methyl purple endpoint (turns from green to purple at pH 4.3). Record the total number of cm³ of acid required as M.

3.10.4 Calculations

If $P = 0$, the alkalinity is due to bicarbonate alone.

$P = M$, the alkalinity is due to hydroxyl ion alone.

$2P = M$, the alkalinity is due to carbonate alone.

$2P > M$, the alkalinity is due to a mixture of carbonate and hydroxyl ion.

$2P < M$, the alkalinity is due to a mixture of carbonate and bicarbonate.

$$\text{Hydroxide, ppm} = \frac{(2 - PM)(1.7 \times 10^4)}{(\text{Vol. of sample, cm}^3) \times (\text{Wt. barite sample, g})} \qquad (16)$$

$$\text{Bicarbonate, ppm} = \frac{(M - 2P)(6.1 \times 10^4)}{(\text{Vol. of sample, cm}^3) \times (\text{Wt. barite sample, g})} \qquad (17)$$

If hydroxide is present:

$$\text{Carbonate, ppm} = \frac{(M - P)(6 \times 10^4)}{(\text{Vol. of sample, cm}^3) \times (\text{Wt. barite sample, g})} \qquad (18)$$

If hydroxide is absent:

$$\text{Carbonate, ppm} = \frac{P \times 6 \times 10^4}{(\text{Vol. of sample, cm}^3) \times (\text{Wt. barite sample, g})} \qquad (19)$$

3.11 WATER-SOLUBLE PHOSPHATES

3.11.1 Description

Water-soluble phosphates, both ortho and condensed, are determined colorimetrically by use of the Hach pre-mixed reagents PhosVer III, or equivalent. A sample of the filtrate from the water extraction of the barite sample (3.7.3.5) is heated in the presence of excess acid to convert all phosphates to orthophosphate. Color is developed by the addition of the PhosVer III or equivalent reagent, which contains ammonium molybdate and ascorbic acid. An intense blue color is formed if phosphate is present, and the intensity of the color is measured with a spectrophotometer at a wavelength of 700 nm.

3.11.2 Equipment

a. Spectrophotometer or filter photometer, capable of measuring absorbance at a wavelength of 700 nm.

b. Sulfuric acid (CAS #7664-93-9): ACS reagent grade (98%).

CAUTION: Concentrated sulfuric acid is very dangerous—avoid any contact with skin or clothing, and wear protective eye wear, face wear, and garments.

c. Sodium hydroxide (CAS #1310-73-2): ACS reagent grade.

d. Sulfuric acid solution (5.25N): 143 cm³ H_2SO_4/1 L of solution.

e. Sodium hydroxide solution (5.0N): 200 g NaOH/1 L of solution.

f. Hach PhosVer III, or equivalent, phosphate reagent powder pillows.

g. Standard phosphate stock solution (1000 mg/L PO_4^{-3}): 1.726 g $Na_3PO_4 \cdot 12H_2O$/L (CAS #10361-03-2) of solution.

h. Erlenmeyer flasks: several 125-cm³.

i. Stoppered graduated mixing cylinders: several 25-cm³.

j. Volumetric pipettes: several, various sizes.
k. Volumetric flasks: several 100-cm^3.

3.11.3 Procedure

3.11.3.1 Preparation of Calibration Curve

3.11.3.1.1 Prepare standard solutions containing 0.25, 0.50, 1.00, and 2.00 mg/L PO_4^{-3} by serial dilution of the stock phosphate standard solution.

3.11.3.1.2 Transfer 25 cm^3 of each solution to 125-cm^3 Erlenmeyer flasks. Prepare a reagent blank using 25 cm^3 of distilled water.

3.11.3.1.3 Add 2.0 cm^3 of 5.25N sulfuric acid and boil gently on a hot plate for 30 minutes, maintaining sample volume at 15–20 cm^3 by additions of distilled water.

3.11.3.1.4 After 30 minutes remove from the hot plate, cool to room temperature, add 2.0 cm^3 of 5.0N sodium hydroxide, and transfer to the 25-cm^3 graduated mixing cylinders.

3.11.3.1.5 Add the contents of one PhosVer III, or equivalent, reagent pillow, dilute to the 25-cm^3 mark with distilled water, stopper, and mix.

3.11.3.1.6 Wait at least 2 minutes but no more than 10 minutes, then measure the absorbance at the wavelength of 700 nm, using a sample cell or tube with a path length of 1 inch (25 mm). Zero the instrument with the reagent blank.

3.11.3.1.7 Prepare a calibration curve by plotting phosphate ion concentration versus absorbance on linear graph paper.

3.11.3.2 Analysis of Samples

3.11.3.2.1 Transfer 5 cm^3 of the filtrate obtained in 3.7.3.5 to a 125-cm^3 Erlenmeyer flask and add sufficient distilled water to bring total volume to 25 cm^3.

3.11.3.2.2 Proceed as directed in "Preparation of Calibration Curve," 3.11.3.1.3 through 3.11.3.1.6.

3.11.3.2.3 Determine the phosphate content, in mg/L, in the final 25 cm^3 solution by reference to the previously prepared calibration curve.

3.11.3.2.4 If the phosphate ion content is below the range of the calibration curve, take a larger portion of the filtrate from 3.7.3.5. If the phosphate ion content is above the range of the calibration curve, prepare a volumetric dilution and take an appropriate aliquot. In any case, record the actual volume of the original filtrate from 3.7.3.5 that was present in the solution in which the color was developed.

3.11.4 Calculation

$$\text{Phosphate, ppm} = \frac{25\ (\text{mg/L } PO_4^{-3} \text{ from calib. curve})}{\text{Volume of filtrate used, cm}^3} \quad (20)$$

3.12 LOSS ON IGNITION

3.12.1 Description

The loss on ignition (LOI) of barite ores is the weight loss after heating to 1000°C. The LOI can be due to several things, including:

a. Moisture—rarely over 0.1–0.2% and usually not separately determined.
b. Combined water—from clays, hydrated iron oxides, gypsum, and so forth.
c. Organic and carbonaceous matter.
d. Thermal decomposition of carbonate minerals; this can be offset, however, by the metal oxides formed combining with sulfur oxides to form metal sulfates.
e. Loss of sulfur from decomposition of pyrite; this can, however, be offset by the iron taking up oxygen.

3.12.2 Equipment

a. A 149-μm sieve.
b. Analytical balance: precision of ± 0.0001 g.
c. Platinum crucibles and lids: two 25-cm^3.
d. Muffle furnace: regulated to 1000°C ± 20°C.
e. Desiccator, glass.
f. Desiccant: silica gel (CAS #7631-86-9) (indicating).

3.12.3 Procedure

3.12.3.1 Use a representative sample of barite ground such that 100 percent passes through the sieve.

3.12.3.2 Accurately weigh a 1.0–1.5-g sample on the analytical balance into an ignited and tared platinum crucible.

3.12.3.3 Place crucible into a cold muffle furnace. Heat to 1000°C and hold at 1000°C for 30 minutes.

3.12.3.4 Remove from furnace with crucible tongs. Place crucible in desiccator.

3.12.3.5 Cool in desiccator and reweigh on the analytical balance.

3.12.4 Calculation

$$\%\ \text{Loss on Ignition} = \frac{100\ (\text{Ignition loss, g})}{\text{Sample Wt., g}} \quad (21)$$

3.13 SIDERITE CONTENT

3.13.1 Description

Siderite ($FeCO_3$) occurs in barite as a natural impurity. Siderite can be dissolved from the barite matrix by refluxing the barite with an alkaline solution of ethylenediamine tetraacetic acid (EDTA) or its salt, and then analyzing by either atomic absorption or colorimetry.

3.13.2 Equipment

a. Balance: precision of ± 0.1 g.

b. Flask, round bottom: 250-cm^3.

c. EDTA solution: 40 g Na_4EDTA (CAS #64-02-8)/100 cm^3 water.

Note: If tetrasodium salt is not available, solution may be prepared from disodium salt. Into a 500-cm^3 beaker, add 325 cm^3 deionized water, 196.0 g Na_2H_2 EDTA.$2H_2O$ (ethylenediamine tetraacetic acid) CAS #6388-92-6 and 37.5 g NaOH. Dissolve and adjust pH to 11.0 with 8N NaOH. Dilute to 500 cm^3.

d. Sodium hydroxide (CAS #1310-73-2) solution (8N): 320 g NaOH/L distilled water.

e. Condenser: water-cooled.

f. Heating mantle: to fit 250-cm^3 round-bottom flask.

g. Variable power regulator.

h. Funnel: 3-in. (77 mm).

i. Filter paper: Whatman #1 or equivalent, 11-cm.

j. Deionized water.

k. Atomic absorption spectrophotometer, or Hach Test Kit for Iron, or equivalent.

l. Colorimeter or color comparator wheel.

m. Volumetric flask: 1-L.

n. Hydrochloric acid (CAS #7647-01-0) solution (1N): 8.3 cm^3 HCl/91.7 cm^3 water.

o. FerroVer Powder Pillows: Hach Catalog Number 854, or equivalent, range 0–3 ppm Fe for water analysis.

3.13.3 Procedure—Extraction

3.13.3.1 Add 20 g barite to 100 cm^3 extraction solution in 250-cm^3 round-bottom flask.

3.13.3.2 Adjust pH to 11.0 with 8N NaOH.

3.13.3.3 Attach condenser and reflux gently for 2 hours.

3.13.3.4 Filter solution.

3.13.4 Procedure—Iron Content by Atomic Absorption

Use manufacturer's accepted instrument settings and procedures to analyze for iron.

3.13.5 Procedure—Iron Content by Colorimetric Determination

3.13.5.1 Transfer 10 cm^3 of filtrate from 3.13.3.4 to a 1-L volumetric flask.

Note: Use 1 cm^3 of filtrate from 3.13.3.4 if the iron content is known to be high.

3.13.5.2 Add about 900 cm^3 deionized water, and adjust the pH with dilute HCl to pH 5.0 ± 0.2.

3.13.5.3 Dilute to volume with deionized water.

3.13.5.4 Measure 25 cm^3 of solution into clean colorimeter bottle.

3.13.5.5 Add contents of FerroVer Powder Pillow, or equivalent, to sample. Swirl the bottle to mix. If iron is present, an orange color will develop.

3.13.5.6 Let sample stand for 2 minutes, but no longer than 10 minutes before measuring the color.

3.13.5.7 Fill a clean colorimeter bottle with deionized water and place it in the light cell.

3.13.5.8 Insert the Iron (FerroVer Method, or equivalent) Meter Scale in the meter and use the 4445 Color Filter, or equivalent. Adjust the light control for a reading of zero mg/L.

3.13.5.9 Place the prepared sample in the light cell and read the ppm iron.

Notes:

1. Copper may interfere by forming a yellow color. One mg/L of cupric copper has been found to cause a 0.2 mg/L (as iron) positive error.
2. A large excess of iron will interfere by inhibiting full color development. If there is any doubt about the validity of a given result, a diluted sample should be tested and the result compared with the original result. At least a 1:1 dilution should be tried first, followed by a 9:1 dilution if the original and the 1:1 dilution results do not coincide.
3. FerroVer has a limited shelf life. Under ideal (cool, dry) conditions, it will keep for several years. Under poor (warm, moist) conditions, it may fail after six months. Periodically perform a test on a sample containing Fe^{++}. If the characteristic orange color develops, the FerroVer is still in satisfactory condition. If no orange color develops, the FerroVer Powder Pillows should be discarded.

3.13.6 Calculation

a. If AA method is used,

$$\text{Iron carbonate, \%} = 0.00104\ (Fe^{++} \text{ in filtrate, mg/L}) \qquad (22)$$

b. If colorimetric method is used,

For 10 cm^3 diluted to 1000 cm^3:

$$\text{Iron carbonate, \%} = 0.104\ (Fe^{++} \text{ in filtrate, mg/L}) \qquad (23)$$

For 1 cm^3 diluted to 1000 cm^3:

$$\text{Iron carbonate, \%} = 1.04\ (Fe^{++} \text{ in filtrate, mg/L}) \qquad (24)$$

3.14 ZINC CARBONATE AND LEAD CARBONATE

3.14.1 Description

Two of the carbonates commonly present as impurities in drilling fluid barite are zinc carbonate and lead carbonate. These compounds are both soluble in 10% acetic acid, where the other common lead and zinc salts found in barite, sphalerite (ZnS) and galena (PbS), are not soluble in this acid.

3.14.2 Equipment

a. Oven, regulated to 220 ± 5°F (104 ± 2°C).

b. Desiccator.

c. Mortar and pestle.

d. A 75-μm sieve.

e. Balance: precision of ± 0.0001 g.

f. Beaker: one 250-cm^3.

g. Acetic acid solution (CAS #64-19-7): 10% by weight.

h. Funnel: one 3-in. (77-mm).

i. Filter paper: Whatman #1, or equivalent, 11-cm.

j. Volumetric flask: one 100-cm^3.
k. Magnetic stirrer, with stirring bar.
l. Atomic absorption spectrophotometer.
m. Atomic absorption standards: Pb and Zn prepared in 10% by weight acetic acid.

3.14.3 Procedure—Extraction

3.14.3.1 Dry a sample of barite for 4 hours in oven. Use a representative sample ground such that 100 percent passes through the sieve.

3.14.3.2 Weigh a 1-g sample of barite into the 250-cm^3 beaker.

3.14.3.3 Add 80 cm^3 of 10% acetic acid.

3.14.3.4 Place stirring bar into beaker, and place beaker on stirrer. Stir for 10 minutes.

3.14.3.5 Filter through filter paper into a 100-cm^3 volumetric flask.

3.14.3.6 Wash twice with small portions of 10% acetic acid.

3.14.3.7 Dilute to volume with 10% acetic acid.

3.14.4 Procedure—Lead and Zinc Determination by Atomic Absorption

Use manufacturer's accepted instrument settings and procedures to analyze for lead and zinc.

3.14.5 Calculation

$$\text{Zinc carbonate, \%} = 0.0192\,\frac{(\text{Zn in filtrate, mg/L})}{\text{Sample Wt., g}} \qquad (25)$$

$$\text{Lead carbonate, \%} = 0.0129\,\frac{(\text{Pb in filtrate, mg/L})}{\text{Sample Wt., g}} \qquad (26)$$

3.15 TOTAL CARBONATE

3.15.1 Description

Total carbonate can be released from barite by treating a sample with acid. The evolved carbonate is absorbed by a suitable absorbent (Ascarite) and measured as the weight gained in the absorbent. The procedure is performed in an air-tight gas train with a nonreactive carrier gas.

3.15.2 Equipment

a. Balance: precision of ± 0.0001 g.
b. A 149-μm sieve.
c. Erlenmeyer flasks: two 125-cm^3.
d. Erlenmeyer flasks: two 250-cm^3.
e. Flask, round bottom: one 250-cm^3.
f. Bottles: three 250-cm^3.
g. Ascarite (CAS #1310-73-2): NaOH impregnated asbestos

CAUTION: Asbestos is a hazardous material. See warnings and precautions on use of asbestos in Policy Statement.

h. Calcium chloride (CAS #10043-52-4): ACS reagent grade.
i. Carrier gas: bottled nitrogen, with gauge and pressure regulator.
j. Perchloric acid (CAS #7601-90-3): ACS reagent grade.

CAUTION: Strong acid; use proper safety precautions.

k. Perchloric acid solution: 1:3 (weight/weight) concentrated $HClO_4$:H_2O.
l. Sulfuric acid (CAS #7664-93-9): ACS reagent grade.

CAUTION: Strong acid; use proper safety precautions.

m. Glass tubing: 6–10 mm outside diameter.
n. Rubber stoppers to fit bottles and flasks.
o. Heating mantle to fit round-bottom flask with variable rheostat.

Notes:
1. Gas train consisting of seven sequential bottles/flasks should be assembled as in Figure 2 with glass tubing and rubber stoppers.
2. Ascarite absorption tubes should be protected from the atmosphere when disconnected for weighing or when not in use. Disconnect rubber tubing connections and place a rubber policeman over the exposed glass tubing.
3. Desiccant must follow Ascarite in final absorption tube to absorb water of reaction from:

$$2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$$

3.15.3 Procedure

3.15.3.1 Use a representative sample ground such that 100 percent passes through the sieve.

3.15.3.2 Weigh a suitably sized sample (5 to 10 g) of barite and place in flask #4.

3.15.3.3 Assemble train and purge with nitrogen for 20 minutes.

3.15.3.4 Disconnect Ascarite tube #8 and weigh to 0.5 mg. Reconnect to train.

3.15.3.5 While nitrogen is still flowing, unstopper flask #3. Quickly add 120 cm^3 perchloric acid solution and restopper.

3.15.3.6 Nitrogen will drive acid over into flask #4, which contains the sample. Boil flask #4 for 10 minutes.

3.15.3.7 Purge with nitrogen for an additional 20 minutes.

3.15.3.8 Disconnect Ascarite tube #8 and weigh to 0.5 mg. Gain in weight represents carbon dioxide evolved from the sample.

3.15.4 Calculation

$$\text{Carbonate } (CO_3)\text{, \%} = \frac{135\,(\text{Weight gain, g})}{\text{Sample Wt., g}} \qquad (27)$$

3.16 ACID-SOLUBLE SULFIDES

3.16.1 Description

3.16.1.1 The concentration of acid-soluble sulfides in

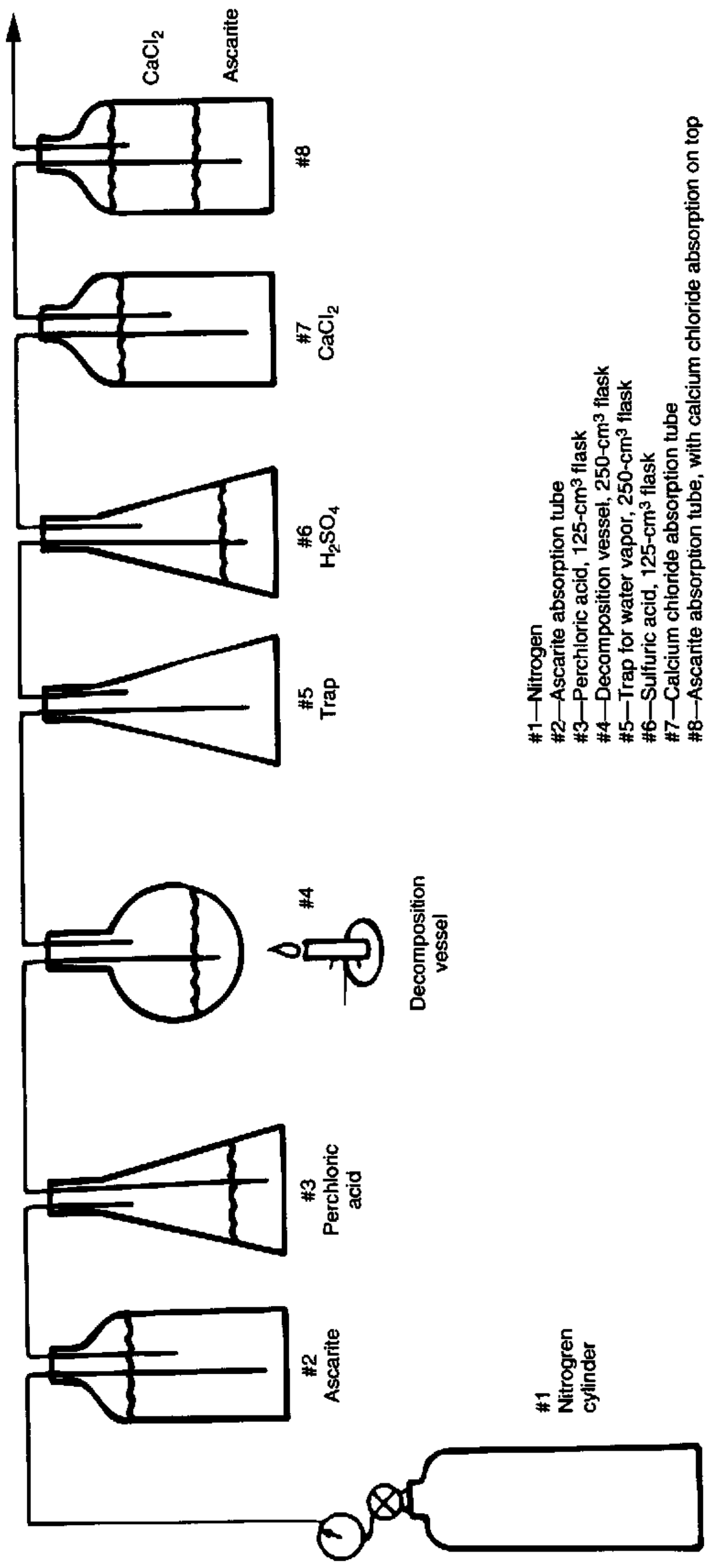

Figure 2—Diagram of Ascarite Gas Train for Total Carbonate

drilling fluid barites can be determined by acidizing with HCl and measurement of evolved H_2S with the Garrett Gas Train (GGT). The GGT separates the gas from the liquid, preventing contamination of the H_2S detector. This detector is a Dräger tube or equivalent, which is the preferred detector, although a lead acetate paper disk can be accommodated in the GGT for positive or negative indication of the presence of H_2S. The Dräger (or equivalent) tube responds quantitatively to H_2S by progressively darkening along its length as the H_2S reacts with the reagent. The low-range Dräger tube is white until H_2S turns it brownish-black.

3.16.1.2 The barite sample appropriate to this test is a commercial barite of drilling fluid grade that has been ground to size at a mill to meet fluid requirements. No further (laboratory) grinding of the sample is recommended because air oxidation of freshly exposed sulfide mineral surfaces can occur.

3.16.2 Equipment

a. The Garrett Gas Train (GGT) apparatus, as shown in Figure 3, consists of a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flowmeter, and a dispersion tube and frit and is described in Recommended Practice 13B-1, Water-Based Drilling Fluids, Appendix A.17a.

b. Carrier gas. Type inert to: hydrogen sulfide, acid, and Dräger, or equivalent, tube's reagents. Nitrogen or helium is preferred. Avoid air, nitrous oxide, or oxygen-containing gases.

c. Dräger, or equivalent, H_2S-Analysis Tube: one low-range—marked "H_2S 100/A"—(No. CH-291-10).

d. Hydrochloric acid solution (CAS #7647-01-0) (5N): 410 cm^3 HCl/L of solution.

e. Defoamer in a dropper bottle.

f. Hypodermic syringe: one 20-cm^3 for hydrochloric acid.

g. Magnetic stirrer with plastic- or glass-covered stirring bar—$^1/_4$ in. × 1 in. (0.6 cm × 2.5 cm) to fit into GGT Chamber 1.

h. Analytical balance: precision of ± 0.0001 g.

3.16.3 Procedure

3.16.3.1 Adjust the magnetic stirrer below the gas train body so that the stirbar will freely rotate and vigorously agitate contents of Chamber 1.

3.16.3.2 Bottled nitrogen or helium should be used as a carrier gas.

Note: Do not use nitrous oxide or air as a carrier gas. Use of either of these gases may result in oxidation of H_2S.

3.16.3.3 Weigh the appropriate amount of barite sample to 0.1 mg based on expected sulfides, using sample size suggested in Table 2. Transfer sample into Chamber 1.

Table 2—Dräger Tube, or Equivalent, Identification, Sample Volumes, and Tube Factors to Be Used for Various Sulfide Ranges

Sulfide Range, mg/L	Sample Volume, cm^3	Dräger Tube Identification (see tube body)	Tube Factor (used in calcualtion)
1.2–24	10.0	H_2S 100/a	0.12[a]
2.4–48	5.0	as above	as above
4.8–96	2.5	as above	as above
60–1020	10.0	H_2S 0.2%/A	1500[b]
120–2040	5.0	as above	as above
240–4080	2.5	as above	as above

[a] A tube factor of 0.12 applies to new tubes, H_2S 100/a (Catalog No. CH-291-01), with a scale from 100 to 2000. For old tubes, use a tube factor of 12.

[b] A tube factor of 1500 applies to new tubes, H_2S 0.2%/A (Catalog No. CH-281-01), with a scale from 0.2 to 7.0. For old tubes, use tube factor 600 times ratio: batch factor/0.40.

3.16.3.4 Install the top of the gas train and hand-tighten evenly to seal O-rings.

3.16.3.5 Carefully break the tip off both ends of Dräger tube and install the tube with the arrow pointing downward into the bored receptacle. Likewise, install the flowmeter tube with the word "Top" upward. Be sure O-rings seal around the body of each tube.

3.16.3.6 Attach the flexible tubing to the dispersion tube and to the Dräger, or equivalent, tube as shown in Figure 3.

Note: As H_2S reacts with many types of rubber and plastic, use only latex rubber or inert plastic tubing. Do not clamp flexible tubing; unclamped tubing provides pressure relief in event of overpressurization.

3.16.3.7 Adjust the dispersion tube in Chamber 1 to approximately 2.0 cm above the bottom, just sufficient to clear the stir bar.

3.16.3.8 Gently flow carrier gas for a 10-second period to purge air from the gas train. Check for leaks. Shut off carrier gas.

3.16.3.9 Add 20 cm^3 of 5N hydrochloric acid solution into chamber and start rapid stirring of Chamber 1.

3.16.3.10 Immediately restart carrier gas flow. Adjust the rate between 200–400 cm^3 per minute. (Keep floating ball in flowmeter between the two marks.)

3.16.3.11 Observe changes in appearance of the Dräger tube. Note and record the maximum Darkened Length (in units marked on the tube) after flowing for a total of 60 minutes. (If first Dräger tube becomes almost filled with stain, shut off flow and quickly replace with another new tube, restart flow and continue the analysis. Add the two stain lengths and use the sum in the calculation below.)

Note: To clean the gas train, remove the flexible tubing and remove the tops. Take the Dräger tube and flowmeter out of their receptacles and plug the holes. Wash with warm water and mild detergent, using a soft brush. Use a pipe cleaner to clean the passages between chambers. Wash, rinse, and then blow out the dispersion tube with gas. Rinse the unit with deionized water and allow to drain dry.

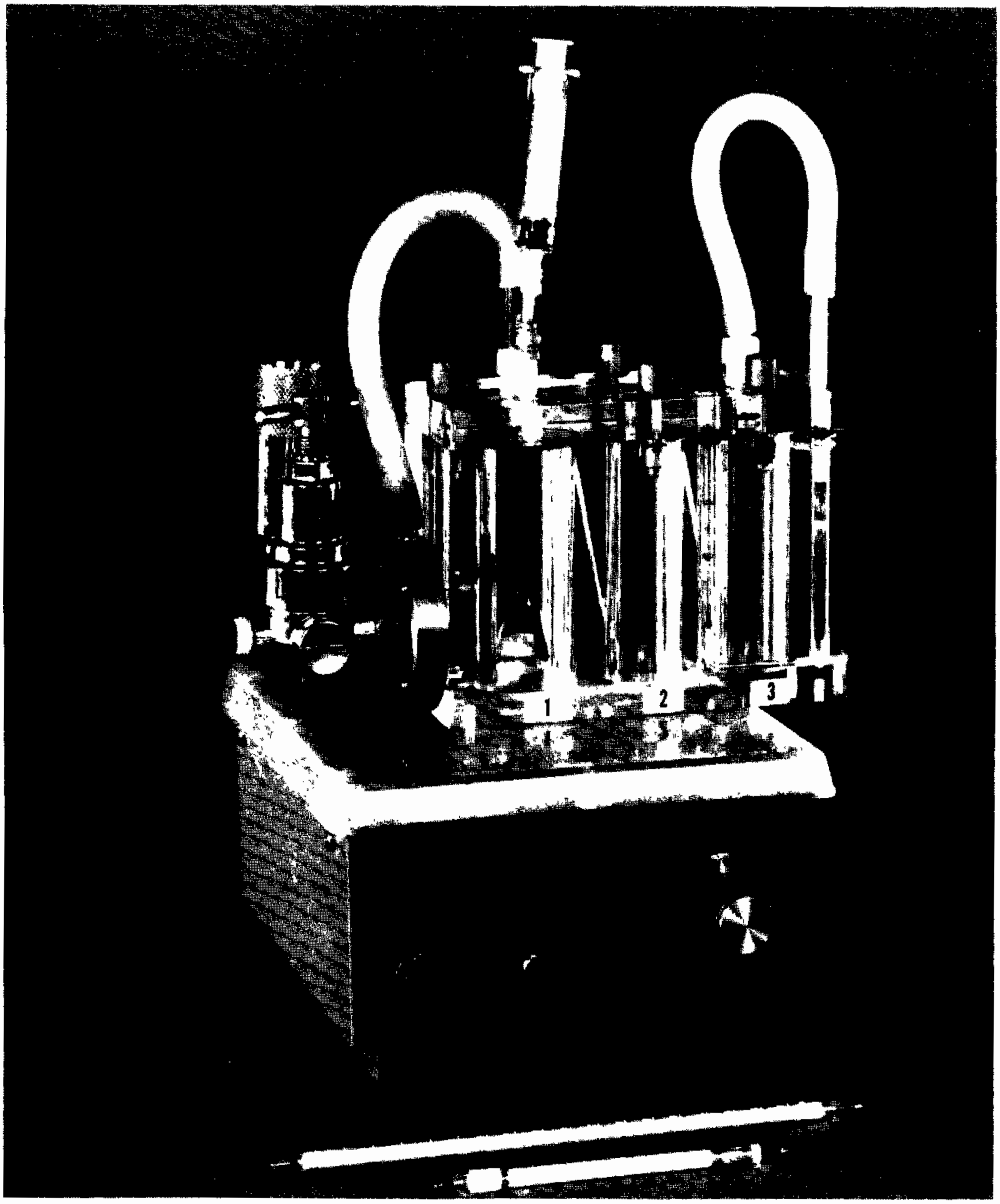

Figure 3—Garrett Gas Train Sulfide Analyzer on a Magnetic Stirrer Set Up for Barite Analysis

3.16.4 Calculations

Using the accurately measured Sample Weight, the Dräger, or equivalent, tube's maximum Darkened Length and the Tube Factor (12) from Table 2, calculate sulfides in the sample:

$$\begin{matrix}\text{GGT, Acid-Soluble} \\ \text{Sulfides, ppm}\end{matrix} = \frac{\text{12 (Darkened Length)}}{\text{Sample Wt., g}} \qquad (28)$$

Note: Darkened length is in units marked on the tube.

3.17 CALCIUM HYDROXIDE (LIME) OR CEMENT

3.17.1 Description

Some field samples of barite are contaminated with lime (calcium hydroxide) or cement, usually due to improper cleaning of bulk barite bins or tanks. This contamination is measured by extracting the barite sample with an aqueous solution of sucrose (sugar), which solubilizes the lime. The solution is then titrated with 0.1N HCl to the phenolphthalein endpoint. The calculation depends on whether the contaminant is known to be lime or cement. (This can be known from the history of the barite or from X-ray diffraction data.)

3.17.2 Equipment

a. Analytical balance: precision of ± 0.001 g.
b. Magnetic stirrer.
c. Stirring bar.
d. Erlenmeyer flasks: several 250-cm^3.
e. Burette: one 25-cm^3.
f. A 149-μm sieve.
g. Graduated cylinder (TC): one 100-cm^3.
h. Sucrose (CAS #57-50-1): crystalline (any granular table sugar will suffice).
i. Hydrochloric acid (CAS #7647-01-0) solution: standardized 0.1N.
j. Phenolphthalein indicator (CAS #77-09-8) solution: 1 g phenolphthalein/100 cm^3 of a 50% alcohol/water solution.

3.17.3 Procedure

3.17.3.1 Use a representative sample of barite ground such that 100 percent passes through the sieve.

3.17.3.2 Accurately weigh a 1.0-g sample on the balance into the 250-cm^3 Erlenmeyer flask.

3.17.3.3 Add 65–70 cm^3 deionized water and the magnetic stirring bar.

3.17.3.4 Add 15 g sucrose (commercial crystalline sugar) and mix at medium speed on the magnetic stirrer for 1 minute.

3.17.3.5 Turn off the stirrer and let the solution sit for 2 minutes.

3.17.3.6 Add 2–3 drops phenolphthalein indicator solution and titrate with 0.1N HCl to the phenolphthalein endpoint. Record the volume of 0.1N HCl required.

3.17.4 Calculations

3.17.4.1 If the contamination is thought to be lime,

$$\text{Lime (CaOH}_2\text{), \%} = \frac{0.370 \times (\text{vol. of 0.1N HCl, cm}^3)}{\text{Sample Wt., g}} \qquad (29)$$

3.17.4.2 If the contamination is thought to be cement, since most oil field cements are about 50% lime,

$$\text{Cement, \% (approx)} = 2\ (\text{\% Lime}) \qquad (30)$$

4 X-Ray Fluorescence Analysis

4.1 DESCRIPTION

4.1.1 In conventional X-ray fluorescence (XRF) spectroscopy, X-rays generated from a tube (or occasionally from radioisotopes) are used to irradiate a specimen of a barite ore. This incident X-ray beam excites elements in the specimen, which then emit (fluoresce) their characteristic line X-ray spectra.

4.1.2 These element spectra are then separated electronically or optically into individual X-ray lines, each line being characteristic of a given element. Elements may then be identified qualitatively by the wavelength (or energies) of those lines, and concentrations may be determined by the line intensities.

4.1.3 This nondestructive XRF method may be used for analysis of all elements with an atomic number greater than 10. The time of analysis is short—in fact, it is a small fraction of the time required for conventional wet chemical methods.

4.1.4 One limitation is that analysis is for elements, not for compounds. Several rational schemes of analysis, however, may be employed to determine mineral or compound compositions.

4.1.5 Two basic types of XRF instruments are employed to measure spectral lines and intensities. One is wavelength-dispersive XRF spectroscopy, in which the spectrum is resolved by analyzing crystals. The second instrument is energy-dispersive XRF spectroscopy, which utilizes a treated silicon crystal and a multichannel analyzer to resolve energies of XRF line spectra.

4.1.6 Since the incident X-ray beam penetrates only a few microns into the specimen surface, sample preparation is critical to obtaining good results. Both the incident (primary) X-rays and the emitted (secondary) X-rays are attenuated by the neighboring particles or atoms; this is known as a matrix effect. Thus the physical form (for example, particle size, surface smoothness, and element concentration) of the sample has a significant effect on the results (response) of the analysis.

4.1.7 Generally, two sample preparation procedures are used. In the fusion method, the sample is mixed with a flux, followed by fusion at high temperature to form a stable glass bead.

A major advantage of this method is that all of the elements are in the same, well-defined chemical environment as a glass. The sample is homogeneous and particle size effects are eliminated. At the same time, the sample is diluted with flux, so matrix effects are small and can be accurately accounted for. Also, calibration standards can be prepared from pure chemicals.

4.1.8 In the second procedure, the pressed powder technique, the barite powder (either neat or mixed with a small amount of binder) is pressed under high pressure into a pellet, which is then analyzed by XRF spectroscopy. This method is much faster and simpler than the fusion technique, and if carefully carried out can be reasonably accurate. Analysis of the lower atomic number elements such as aluminum and silicon is less accurate. This procedure also requires an extensive library of samples (standards) analyzed by wet chemical techniques, and consisting of elements in matrix material similar to the sample.

4.2 EQUIPMENT

a. X-ray fluorescence spectrometer: any XRF unit is suitable. Instrument settings recommended by the manufacturer should be followed.

b. Computer software for XRF spectrometer (usually supplied by spectrometer manufacturer), optional.

c. Muffle furnace: regulated to 1100 ± 20°C.

d. Drying oven: regulated to 230 ± 10°F (110 ± 5°C).

e. Meker-type burners: two or more.

f. 95% platinum–5% gold crucibles and covers: several 20–25 cm^3.

Note: Graphite crucibles may also be used as an alternative.

g. 95% platinum–5% gold molds: Several sized to fit XRF sample holder.

Note: Graphite molds may also be used as an alternative.

h. Hydraulic pellet press: capable of pressures of at least 40,000 pounds per square inch (psi) (276,000 kilopascals [kPa]).

i. Die for pellet press: sized to fit XRF sample holder.

j. Impact grinder and grinding vials.

k. Timer.

l. Hot plate.

m. Analytical balance: precision of ± 0.001 g.

n. A 45-μm sieve.

o. Crucible tongs.

p. Platinum wire stirrer.

q. PTFE, Teflon® or equivalent stirring rod.

r. Plastic sample cups and plastic (for example, Mylar® or equivalent) film: sized to fit XRF spectrometer.

s. Lithium tetraborate (CAS #12007-60-2): anhydrous, "spectral" grade powder.

t. Boric acid (CAS #10043-35-3): "spectral" grade powder.

u. Germanium oxide (CAS #20619-16-3): "spectral" grade powder.

v. Lithium carbonate (CAS #584-13-2): anhydrous, "spectral" grade powder.

w. Lithium nitrate (CAS #7790-69-4): anhydrous, "spectral" grade powder.

x. Lanthanum oxide (CAS #1312-81-8): "spectral" grade powder.

y. Hydrobromic acid (CAS #10035-10-6) solution (10%): 10 g HBr/90 cm^3 water.

z. Flux mixture A: 47% lithium tetraborate, 37% lithium carbonate, 16% lanthanum oxide.

aa. Flux mixture B: 90% lithium tetraborate, 10% lithium nitrate.

bb. Synthetic standards: several, made up of "pure" element compounds such as $BaSO_4$, $SrSO_4$, SiO_2, Fe_2O_3, and so forth, to cover all of the elements being analyzed. The range of concentrations should cover the anticipated ranges in the unknowns.

cc. Barite ore standards: several, all analyzed by conventional wet chemical techniques. Again, the concentration ranges (of $BaSO_4$, SiO_2, Fe_2O_3, and so forth) should cover anticipated levels of the unknowns.

4.3 PROCEDURE—SAMPLE PREPARATION

4.3.1 Dry a representative sample of barite for at least 2 hours at 110°C.

4.3.2 Grind the dried sample in the impact grinder such that 100 percent passes through a 45-μm sieve.

4.3.3 Place part of the ground sample in a platinum–gold or graphite crucible and ash in the muffle furnace for 30 minutes at 1000°C.

CAUTION: Use proper safety precautions when handling hot crucible and melt.

4.3.4 Grind the ashed sample in the impact grinder to 100 percent passing through a 45-μm sieve.

4.3.5 One or more of the four following methods of sample preparation (4.3.5.1 through 4.3.5.4) may now be used.

4.3.5.1 Pressed Powder Method

4.3.5.1.1 Approximately 20 g of the ground (but not ashed) sample is placed in a standard die and pressed to 40,000 psi with a hydraulic press. The resulting pellet is saved for XRF analysis.

Note: As an alternative procedure, the loose barite powder may be placed in a plastic sample cup, using a plastic (for example, Mylar, or equivalent) film to hold the sample and to act as a "window" to the incident X-ray beam. The accuracy and precision of this method, however, are not as high as for the pressed pellet procedure.

4.3.5.1.2 Several standards (both synthetic and analyzed standards may be used) are prepared in the same manner.

4.3.5.1.3 If the pellet does not hold together after it is made, a small amount (for example, 10%) of a binder such as boric acid may be added to the sample, mixed thoroughly, and pressed into a pellet. If this is done, then the standards must be prepared in the same manner.

4.3.5.2 Fused Glass Disc Technique: Flux A

4.3.5.2.1 Accurately weigh on the analytical balance 0.500 g ground, ashed barite sample with 0.050 g germanium oxide and 2.000 g flux mixture A into a platinum–gold crucible.

4.3.5.2.2 Mix with PTFE (Teflon® or equivalent) rod.

4.3.5.2.3 Fuse in the muffle furnace for 15 minutes at 980°C. Swirl melt, using the crucible tongs, to ensure mixing of products after 5 minutes.

CAUTION: Use proper safety precautions when handling hot crucible and melt.

4.3.5.2.4 Pour the melt into the platinum–gold mold that is kept at about 428°F (220°C) on the hot plate.

4.3.5.2.5 After the melt cools for 10 minutes, remove it with the tongs to a piece of unglazed tile.

4.3.5.2.6 The glass bead should shrink as it cools and pop free of the mold. The bead may be used as is (using the surface that was next to the bottom of the mold), or it may be further polished before XRF analysis.

4.3.5.2.7 Prepare several standards (synthetic and/or analyzed barite standards) in the same manner.

4.3.5.3 Fused Glass Disc Technique: Flux B

4.3.5.3.1 Accurately weigh on the analytical balance 1.000 g ground, ashed barite with 4.350 g flux mixture B into a platinum–gold crucible.

4.3.5.3.2 Mix with PTFE (Teflon® or equivalent) rod.

4.3.5.3.3 Add 10 drops of the 10% HBr solution to the mixture and cover the crucible.

4.3.5.3.4 Place the crucible over a Meker-type burner and heat on low flame for a few minutes.

4.3.5.3.5 Increase the flame and fuse the sample for 8 minutes at high temperature (approximately 1000°C).

4.3.5.3.6 Swirl the crucible occasionally, using the crucible tongs, to aid in forming a homogeneous melt and to assist the release of any gas phase that may form during fusion.

CAUTION: Use proper safety precautions when handling hot crucible and melt.

4.3.5.3.7 Place the platinum–gold mold over a second Meker-type burner and heat to high temperature.

4.3.5.3.8 When the fused melt is clear and free of bubbles, pour it into the heated mold.

4.3.5.3.9 Place the filled mold on a piece of unglazed tile and allow it to cool.

4.3.5.3.10 As the glass cools, it will pop free of the mold. It may be analyzed as is (using the bottom surface for analysis) or further polished before XRF analysis.

4.3.5.3.11 Prepare several standards (synthetic and/or analyzed barite standards) in the same manner.

4.3.5.4 Fusion/Pressed Disc Techniques

4.3.5.4.1 Accurately weigh on the analytical balance 0.500 g barite sample and 1.000 g lithium tetraborate into a platinum–gold crucible.

4.3.5.4.2 Mix with PTFE (Teflon® or equivalent) rod.

4.3.5.4.3 Place into muffle furnace and fuse for 15 minutes at 1050–1100°C with occasional swirling of the melt to ensure thorough mixing.

CAUTION: Use proper safety precautions when handling hot crucible and melt.

4.3.5.4.4 Cool bead and add boric acid to total weight of 1.600 g.

4.3.5.4.5 Grind the mixture for 10 minutes in the impact grinder.

4.3.5.4.6 Press in the hydraulic press at 40,000 psi using boric acid as backing to the disc.

4.3.5.4.7 Repeat with several synthetic and/or analyzed standards.

4.4 PROCEDURE—SPECTRAL ANALYSIS

4.4.1 Using the instrument settings recommended by the manufacturer, collect spectra of the elements of interest for both standards and unknown barite samples.

Note: Eight elements are routinely analyzed: barium, iron, silicon, aluminum, calcium, sulfur, strontium, and magnesium. Other "minor" elements such as lead, zinc, manganese, potassium, and so forth, can also be analyzed, but are usually present at low or trace concentrations.

4.4.2 Spectral data must be collected long enough for adequate precision in the final results. This can usually be determined by following instructions that come with the instrument or the computer software.

4.4.3 Most instrument manufacturers provide computer software that accomplishes data manipulation automatically. This includes storage of standard spectra; linear and nonlinear regression analysis; statistical analysis; and matrix correction programs.

4.5 CALCULATIONS

4.5.1 Fused glass discs—there is little or no interelement or matrix correction. The flux A method contains lanthanum ion; this effectively dilutes the sample to a level where the concentration/X-ray intensity relationship is linear. The analysis is compared to calibrated standards, either graphically or by computer software.

4.5.2 Fusion/pressed disc method—the results are compared with standards and fitted to curves drawn from concentration/intensity values. The concentration/X-ray response is nonlinear and is corrected from the calibration curves.

4.5.3 For both fusion methods, the concentrations of elements obtained from XRF analysis are for ashed samples, not for as-received samples. To correct for this loss on ignition (LOI), the following equation is used:

$$\text{Element (as received), \%} = \frac{\text{\% element (ash basis)}\,(100 - \text{\% LOI})}{100} \qquad (31)$$

4.5.4 Pressed powder method—here the matrix effects are large, and consequently a large number of analyzed samples must be examined in order to obtain accurate and reproducible results. Calibration curves will be nonlinear, but computer programs can be used for both nonlinear regression analysis or to correct for matrix effects.

APPENDIX A—RATIONAL ANALYSIS

A.1 Introduction

The chemical analysis results obtained from wet chemical analyses and X-ray fluorescence analysis of barite ores yield no details of the different mineral species that make up these ores. If limited assumptions are made, however, then the mineralogy or mineral composition may be deduced. This type of "logical" analysis to obtain the mineralogy of the barite ores is called rational analysis.

A.2 Procedure and Calculation

A.2.1 Assume all barium occurs as the sulfate, and all strontium as the sulfate. If wet chemical analysis is used, then these compounds are obtained directly. If X-ray fluorescence is used to obtain the oxides, then:

$$BaSO_4, \% = 1.522 \% \; BaO \qquad (A\text{-}1)$$

$$SrSO_4, \% = 1.773 \% \; SrO \qquad (A\text{-}2)$$

A.2.2 Acid-soluble (HCl) iron is either siderite ($FeCO_3$) or hematite (Fe_2O_3). Pyrite, being insoluble in HCl but soluble in HNO_3, is obtained by:

$$FeS_2, \% = 2.148 \times (\% \text{ Fe from } HNO_3 - \% \text{ Fe from HCl}) \qquad (A\text{-}3)$$

A.2.3 Fe_2O_3 may be obtained from the HCl soluble iron by:

$$Fe_2O_3, \% = 0.699 \times (\% \text{ Fe from HCl} - 0.482 \times \% \text{ Siderite}) \qquad (A\text{-}4)$$

A.2.4 Aluminosilicate minerals include feldspars, clays, micas, and so forth. These may be approximated from alumina content, as follows:

$$\text{Aluminosilicates}, \% = 2.7 \times \% \; Al_2O_3 \qquad (A\text{-}5)$$

A.2.5 Free silica or quartz may be approximated by subtracting the silica in aluminosilicates from total silica:

$$\text{Quartz}, \% = \text{Total} \% \; SiO_2 - 0.47 \times \% \text{ Aluminosilicates} \qquad (A\text{-}6)$$

A.2.6 Calcium sulfate (for example, gypsum) is obtained from the water-soluble Ca:

$$\text{Gypsum}, \% = 4.295 (\% \text{ W.S. Ca}) \qquad (A\text{-}7)$$

(This is assuming that there is no $CaCl_2$ present.)

A.2.7 Calcium carbonate may be approximated from the difference between the HCl-soluble Ca and the water-soluble Ca (assuming no lime or cement was detected):

$$CaCO_3, \% = 2.497 (\% \text{ HCl-Sol. Ca} - \% \text{ W.S. Ca}) \qquad (A\text{-}8)$$

A.2.8 Loss on ignition is due primarily to loss of CO_2 by carbonates, and secondarily to loss of water from clays.

A.2.9 The above "scheme" of rational analysis is strictly for purposes of an example; other schemes may be used, as long as each scheme is chemically correct. Also, it would be fairly simple to write computer programs that could make the calculations from such rational analyses very easy and fast to perform.

APPENDIX B—METRIC "SI" UNIT CONVERSION TABLE

Property	Traditional Unit	Recommended SI unit	Symbol	Conversion Factor	Example
Mechanical Properties				**Multiply by:**	
Depth	feet	meter	m	0.3048	10,000 ft = 3048 m
Hole Diameter	inch	millimeter	mm	25.4	12 $^{1}/_{4}$ in. = 311 mm
Pipe Diameter	inch	millimeter	mm	25.4	4 $^{1}/_{2}$ in. = 114 mm
Bit Size	inch	millimeter	mm	25.4	12 $^{1}/_{4}$ in. = 311 mm
Weight on Bit	pound	newton	N	4.4	20,000 lb = 88,000 N
Rotary Table Speed	rpm	revolutions per minute[a]	r/min	1	45 rpm = 45 r/min
Nozzle Size	$^{1}/_{32}$ inch	millimeter	mm	0.79	$^{10}/_{32}$ in. = 7.9 mm
Nozzle Velocity	feet/sec	meter/sec	m/s	0.3048	400 ft/sec = 122 m/s
Drilling Rate	feet/hour	meter/hour[a]	m/h	0.3048	30 ft/h = 9 m/h
Volume	barrels	cubic meter	m^3	0.159	3000 bbl = 477 m^3
Liner Size	inch	millimeter	mm	25.4	6 $^{1}/_{2}$ in. = 165 mm
Rod Diameter	inch	millimeter	mm	25.4	2 $^{1}/_{4}$ in. = 57 mm
Stroke Length	inch	millimeter	mm	25.4	16 in. = 406 mm
Pump Output	barrel/minute	cubic meter/minute[a]	m^3/min	0.159	8.5 bbl/min = 1.35 m^3/min
	gallon/minute	cubic meter/minute[a]	m^3/min	0.00378	357 gpm = 1.35 m^3/min
Pump Pressure	pound/sq inch	kilopascal	kPa	6.9	2500 psi = 17,300 kPa
Annular Velocity	feet/min	meter/minute[a]	m/min	0.3048	200 ft/min = 61 m/min
Slip Velocity	feet/min	meter/minute[a]	m/min	0.3048	20 ft/min = 6.1 m/min
Temperature	degree Fahrenheit	degree Celsius	°C	$\frac{(°F-32)}{1.8}$	80°F = 27°C
	degree centigrade	degree Celsius	°C	1.0	
Funnel Viscosity	seconds/quart	seconds/liter	s/L	Units cannot normally be converted	
	seconds/1000 cm^3	seconds/liter	s/L	1.0	
Mud Weight	pounds/gallon	kilogram/cubic meter	kg/m^3	120	10 lb/gal = 1200 kg/m^3
	pounds/ft^3	kilogram/cubic meter	kg/m^3	16	74.8 lb/ft^3 = 1200 kg/m^3
Pressure Gradient	psi/ft	kilopascal/meter	kPa/m	22.6	0.52 psi/ft = 11.8 kPa/m
Hydrostatic Head	psi	kilopascal	kPa	6.9	4000 psi = 27,600 kPa
Shear Stress	lbf/100 ft^2	pascal	Pa	0.48	20 lb/100 ft^2= 960 Pa
	dynes/cm^2	pascal	Pa	0.100	10 dynes/cm^2 = 1.0 Pa
Shear Rate	reciprocal second	reciprocal second	s^{-1}	1.0	
Apparent, Plastic and Effective Viscosity	centipoise	centipoise[a]	cP		
Yield Point	lbf/100 ft^2	pascal	Pa	0.48	15 lb/100 ft^2 = 7.2 Pa
Gel Strength	lbf/100 ft^2	pascal	Pa	0.48	3 lb/100 ft^2 = 1.44 Pa
Dial Reading, Standard V-G Meter	$\frac{\text{lbf/100 ft}^2}{1.065}$	pascal	Pa	0.51	dial reading – 10 = 5.1 Pa
Power Law Constants					
n	dimensionless				
K	dynes s^n/cm^2	millipascal seconds^n/ square centimeter	mPa•s^n/cm^2	100	10 dynes s^n/cm^2 = 100 mPa•s^n/cm^2
	lbf s^n/100 ft^2	millipascal seconds^n/ square centimeter	mPa•s^n/cm^2	479	1.2 lb s^n/100 ft^2 = 575 mPa•s^n/cm^2
API Filtrate	cubic centimeter/ 30 min	cubic centimeter[a]/ 30 min	cm^3/30 min		
Filter Cake	$^{1}/_{32}$ inch	millimeter	mm	0.8	$^{3}/_{32}$ in. = 2.4 mm
Sand Content					
Solids Content	Volume percent	cubic meter/cubic meter	m^3/m^3	0.01	10% = 0.1 m^3/m^3
Oil Content					
Water Content					
Particle Size	micron	micrometer	μm	1.0	

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[a] Denotes acceptable deviation from API 2564 preferred unit.

Property	Traditional Unit	Recommended SI unit	Symbol	Conversion Factor	Example
Chemical Properties				**Multiply by:**	
Ionic Concentration	grains/gallon	milligram/liter[a]	mg/L	17.1 × specific gravity	500 grains/gal = 8600 mg/L
	parts/million	milligram/liter[a]	mg/L		100,000 ppm of NaCl × 1.0707 = 107,070 mg/L
Alkalinity P_f, M_f, P_1, P_2	cubic centimeter/ cubic centimeter	cubic centimeter/ cubic centimeter	cm^3/cm^3		
MBT Methylene Blue Content	cubic centimeter/ cubic centimeter	cubic centimeter/ cubic centimeter	cm^3/cm^3		
Miscellaneous				**Multiply by:**	
Additive Concentration	pound/barrel	kilogram/cubic meter	kg/m^3	2.85	10 lb/bbl = 28.5 kg/m^3
Corrosion Rate	$lb/ft^2/yr$	kilogram/square meter/year	$kg/m^2/yr$	4.9	0.9 lb/ft^2/year = 4.3 kg/m^2/year
	mils/year	millimeter/year	mm/a	0.0254	200 mils/year = 5.08 mm/a
Bentonite Yield	barrels/US ton	cubic meter[a]/metric tonne	m^3/t	0.175	100 bbl/ton = 17.5 m^3/t
Hydraulic HP	horsepower	kilowatt	kW	0.746	600 hhp = 450 kW
Screen Size					
Mesh	openings/inch	openings/centimeter	openings/cm	0.254	100 mesh = 25.4 openings/cm
Aperture	micron	micrometer	μm	1	
Open Area	area percent	square meter/square meter	m^2/m^2	0.01	30% = 0.3 m^2/m^2
Weight of Drill Pipe	pound/foot	kilogram/meter	kg/m	1.49	19.5 lb/ft = 29.1 kg/m
Oil Muds				**Multiply by:**	
Oil Content Water Content	volume percent	cubic meter/cubic meter	m^3/m^3	0.01	10% = 0.1 m^3/m^3
Emulsion Stability	volts	volts	V		
Activity	dimensionless				
Salinity	ppm	milligrams[a]/liter	mg/L	specific gravity	250,000 ppm of $CaCl_2$ × 1.24 = 310,000 mg/L
Aniline Point	degree Fahrenheit	degree Celsius	°C	$\frac{(°F-32)}{1.8}$	150°F = 66°C

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[a] Denotes acceptable deviation from API 2564 preferred unit.

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