Recommended Practice for Chemical Analysis of Barite

API RECOMMENDED PRACTICE 13K
THIRD EDITION, MAY 2011

AMERICAN PETROLEUM INSTITUTE

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Upstream Segment

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1 Scope

1.1 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 A list of some 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 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 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 Normative References

The following referenced documents are indispensable for the application of this document. For dated references, on the edition cited applies. For undated references, the latest edition of the reference document (including any amendments) applies.

API Recommended Practice 13B-1, *Recommended Practice for Field Testing Water-based Drilling Fluids*

API Recommended Practice 13I-2009, *Recommended Practice for Laboratory Testing of Drilling Fluids*

API *Manual of Petroleum Measurement Standards* (*MPMS*), Chapter 15, *Guidelines for Use of the International System of Units (SI) in the Petroleum and Allied Industries*

ISO 10416:2008 [1], *Petroleum and natural gas industries—Drilling fluids—Laboratory testing*

U.S. *29 Code of Federal Regulations* (*CFR*) [2], Section 1910.1001

U.S. *40 Code of Federal Regulations* (*CFR*), Section 61.140 through Section 61.156

U.S. *51 Federal Register* (*FR*), 3738-3759 (January 29, 1986)

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[1] International Organization for Standardization, 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, www.iso.org.

[2] The *Code of Federal Regulations* and *Federal Register* are available from the U.S. Government Printing Office, Washington, DC 20402.

Table 1—Minerals Associated With Barite Ore Bodies

Mineral	Chemical Formula	Mineralogical Name	Specific Gravity	Hardness (Mohs)
Barium sulfate	$BaSO_4$	Barite, barites, barytes	4.50	2.5–3.5
Strontium sulfate	$SrSO_4$	Celestite	3.97	3.5
Barium carbonate	$BaCO_3$	Witherite	4.30	3.5
Barium silicate	$BaSi_2O_3$	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	$CaSO_4 \bullet 2H_2O$	Gypsum	2.3	2
Calcium fluoride	CaF_2	Fluorite	3.0	4
Iron oxide	Fe_2O_3	Hematite	5.2	5.5
Iron oxide	$Fe_2O_3 \bullet nH_2O$	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	3.5–4.0
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	6.0
Mica	$KAl_2(OH)_2AlSi_3O_{10}$	Muscovite	2.85	2.5
Clays	$Al_2Si_2O_5(OH)_4$	Kaolinite	2.6	2.0
	$(Al,Mg)(OH)_2Si_4O_{10}(Na,Ca)_x \bullet 4H_2O$	Montmorillonite	2–3	1–2

3 Terms and Definitions

For the purposes of this document, the following definitions apply.

3.1
ACS reagent grade

Grade of chemical meeting the purity standards specified by the American Chemical Society, with impurities measured in parts per million.

3.2
spectral grade

Grade of chemical exceeding the purity standards specified by the American Chemical Society, with impurities measured in parts per billion.

4 Acronyms, Abbreviations, and Symbols

For the purposes of this document, the following acronyms, abbreviations, and symbols apply.

AA	atomic absorption (spectrophotometer)
ACS	American Chemical Society
GGT	Garrett Gas Train
IC	ion chromatography
ICP	inductively coupled plasma (spectrometry), can be ICP-AES, ICP-MS, or ICP-OES
ICP-AES	inductively coupled plasma atomic emission spectrophotometer
ICP-MS	inductively coupled plasma mass spectrometry
ICP-OES	inductively coupled plasma optical emission spectrophotometer
NIST	National Institute of Standards and Technology
PTFE	polytetrafluoroethylene
UV	ultraviolet (spectrophotometer)
$c_{\mathrm{BaSO_4},\%}$	concentration of barium sulfate, expressed as a %
$c_{\mathrm{CaCO_3}\%}$	concentration of calcium carbonate, expressed as a %
$c_{\mathrm{CaOH_2},\%}$	concentration of calcium hydroxide (lime), expressed as a %
c_{Cl}	concentration of chloride ion, expressed in mg/l
$c_{\mathrm{CO_3}}$	concentration of carbonate ion, expressed in mg/l
$c_{\mathrm{CO_3},\%}$	concentration of carbonate ion, expressed as a %
$c_{\mathrm{CEMENT},\%}$	concentration of cement, expressed as a %
$c_{\mathrm{ELEMENT1},\%}$	concentration of element, as received basis, expressed as a %
$c_{\mathrm{ELEMENT2},\%}$	concentration of element, ashed basis, expressed as a %
c_{Fe}	concentration of iron in filtrate, expressed in ml/l
$c_{\mathrm{Fe},\%}$	concentration of iron, expressed as a %
$c_{\mathrm{FeCO_3},\%}$	concentration of iron carbonate, expressed as a %
$c_{\mathrm{HCl\text{-}SOLUBLE\,CA},\%}$	concentration of HCl-soluble calcium, expressed as a %, see Section 8
$c_{\mathrm{HCO_3}}$	concentration of bicarbonate ion, expressed in mg/l

$c_{\text{Metal ion},\%}$	concentration of a metal ion, expressed as a %
c_{OH}	concentration of hydroxyl ion, expressed in mg/l
c_{Pb}	concentration of lead in filtrate, expressed in mg/l
$c_{\text{PbCO}_3,\%}$	concentration of lead carbonate, expressed as a %
c_{PO_4}	concentration of phosphate, expressed in mg/l
c_{S}	acid soluble sulfide concentration, expressed in mg/kg
c_{SO_4}	concentration of sulfate ion, expressed in mg/l
$c_{\text{Sr},\%}$	concentration of strontium, expressed as a % from 6.6
$c_{\text{SrSO}_4,\%}$	concentration of strontium sulfate, expressed as a %
$c_{\text{WATER-SOLUBLE Ca},\%}$	concentration of water-soluble calcium, expressed as a %, see Section 11
c_{Zn}	concentration of zinc in filtrate, expressed in mg/l
$c_{\text{ZnCO}_3,\%}$	concentration of zinc carbonate, expressed as a %
$f_{\text{Al}_2\text{O}_3}$	correction factor for alumina
f_{CO3}	tube factor, i.e. 2.5 for Dräger No. 8101811
f_{S}	tube factor from Table 2
f_{SiO_2}	correction factor for silica
k	correction factor taken from Figure 1
l_{ST}	Dräger tube’s maximum darkened length, expressed in units marked on the tube
LOI	loss on ignition, expressed as a %
m	mass of the barite sample, expressed in g
m_1	mass of the precipitate, expressed in g
m_1	mass of the precipitate, expressed in g
m_2	mass of standard sample, expressed in g
m_3	mass of the crucible, expressed in g
m_4	mass of barite sample and crucible, expressed in g
m_5	mass of ignited sample and crucible, expressed in g
n_1	absorbance value for silica in standard, expressed in nm

n_2	absorbance value for silica in sample, expressed in nm
n_3	absorbance value for alumina in standard, expressed in nm
n_4	absorbance value for alumina in sample, expressed in nm
R	AA or ICP reading value, expressed in mg/l
R_{IC}	measured value from ion chromatograph, expressed in mg/l
R_{M}	meter reading, expressed in mg/l
$R_{\mathrm{PO_4}}$	phosphate ion concentration reading from the calibration curve, expressed in mg/l
V	volume of barite-water leachate filtrate, expressed in ml
V_{AgNO3}	volume of silver nitrate used, expressed in ml
V_{HCl}	volume of 0.1N HCl, expressed in ml
V_{M}	volume of 0.2N H_2SO_4 used in titration to reach methyl purple endpoint, expressed in ml
V_{P}	volume of 0.2N H_2SO_4 used in titration to reach phenolphthalein endpoint, expressed in ml
φ	mass fraction of element in sample, mg/l

5 Wet Analysis Methods

5.1 Principle

5.1.1 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.

5.1.2 These classical wet methods can determine the elements present in the sample but will not supply all 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 Annex A).

5.2 Advantages of Wet Chemical Analysis Methods

5.2.1 If directions are carefully followed, most trained chemists or technicians can get good results within a reasonable time.

5.2.2 The methods are selective, sensitive (usually in the milligrams per liter or milligrams per kilogram [parts per million] range), accurate, and reproducible (accuracy and reproducibility are usually 1% or less).

5.2.3 From the results of the wet analyses, one can usually determine approximate mineral (and/or compound) composition.

5.2.4 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.

5.3 Summary of General Analytical Methods

5.3.1 Barium sulfate ($BaSO_4$)—sodium carbonate (Na_2CO_3) fusion, gravimetric analysis.

5.3.2 Strontium sulfate ($SrSO_4$)—sodium carbonate (Na_2CO_3) fusion, atomic absorption (AA) analysis or inductively coupled plasma (ICP).

5.3.3 Silica (SiO_2) **and alumina** (Al_2O_3)—sodium hydroxide (NaOH) fusion, colorimetric analysis.

5.3.4 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 or ICP. The only HCl insoluble Na and K compounds sometimes found in barite are Na and K feldspars, which 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.

5.3.5 Calcium (Ca), **magnesium** (Mg), and **iron** (Fe)—hydrochloric acid (HCl) and/or hydrofluoric/sulfuric/nitric acid ($HF/H_2SO_4/HNO_3$) extraction, then AA or ICP 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).

5.3.6 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 or ICP analysis.

5.3.7 Copper (Cu), **manganese** (Mn), **lead** (Pb), and **zinc** (Zn)—hydrochloric acid (HCl) extraction, then AA analysis.

5.3.8 Siderite ($FeCO_3$)—ethylene diaminetetraacetic acid dihydrate (EDTA)/sodium hydroxide (NaOH) extraction, then AA or ICP analysis.

5.3.9 Total carbonate (CO_3^{-2})—determine by the Garrett Gas Train (GGT) method.

5.3.10 Sulfide (S^{-})—determined by the GGT method; alternative procedure—treat with 1N HCl or 1N H_2SO_4, analyze H_2S evolved with lead acetate paper.

5.3.11 Water soluble analysis—barite is extracted with an equal weight of deionized water, and filtrate is analyzed for elements of interest by titration, AA, ICP and flame emission techniques.

5.3.12 Loss on ignition (LOI)—weight loss after heating to 1000 °C (1832 °F), due to combined water lost from clays, decomposition of carbonates or organic and carbonaceous matter.

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

6 Barium Sulfate and Strontium Sulfate

6.1 Principle

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 AA spectrometry or ICP.

6.2 Reagents and Materials

6.2.1 Deionized or distilled water.

6.2.2 Sodium carbonate (CAS #497-19-8), anhydrous, Na_2CO_3, American Chemical Society (ACS) grade powder.

6.2.3 Sodium carbonate solution (0.2 %), 1 g Na_2CO_3 diluted to 500 ml with deionized water.

6.2.4 Hydrochloric acid (CAS #7647-01-0) **solution** (1:4), 20 ml HCl diluted with 80 ml of deionized water.

6.2.5 Sulfuric acid (CAS # 7664-93-9) s**olution** (1:19), 5 ml H_2SO_4 diluted with 95 ml deionized water.

6.2.6 Sulfuric acid solution (1 %), 5 ml H_2SO_4 diluted with 495 ml deionized water.

6.2.7 Strontium AA or ICP standards, 5 mg, 10 mg, and 15 mg per liter (mg/l) Sr in 1 % HCl.

6.2.8 Ammonium hydroxide (CAS #1336-21-6), NH_4OH, concentrated, ACS reagent grade.

6.2.9 Methyl orange (CAS #547-58-0) **indicator solution**, 0.1 g methyl orange diluted to 100 ml with deionized water.

6.3 Apparatus

6.3.1 Mortar and pestle.

6.3.2 Sieve, 149 µm.

6.3.3 AA spectrophotometer or ICP spectrometer, any AA or ICP unit is suitable. Instrument settings recommended by the manufacturer should be followed.

6.3.4 Balance, with accuracy of 0.001 g.

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

6.3.6 Crucibles and lids, platinum, two 25 ml.

6.3.7 Crucible tongs, one 25 cm (10 in.) or one 50 cm (20 in.).

6.3.8 Muffle furnace, regulated to 1000 °C ± 20 °C (1832 °F ± 72 °F).

6.3.9 Beakers, four 250 ml, two 400 ml, and two 600 ml.

6.3.10 Watch glasses, two fitted to 250-ml beaker and two fitted for 600-ml beaker.

6.3.11 Hot plate.

6.3.12 Funnels, glass, two 65-mm long stem.

6.3.13 Filter paper, 11.0 cm Whatman #40® [3], 11.0 cm Whatman #541®, or equivalent.

6.3.14 Volumetric flasks (TC), two 250 ml.

6.3.15 Annealing cups, two size #1 (12 ml, 33 mm × 35 mm).

6.3.16 Desiccator.

6.3.17 Volumetric pipette (TD), one 10 ml.

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[3] Whatman #40® and Whatman #541® are examples of suitable products available commercially. This information is given for the convenience of users of this part of API 13K and does not constitute an endorsement by API of these products.

6.3.18 Medicine droppers, two.

6.4 Sampling

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

6.5 Procedure—Barium Sulfate Determination

6.5.1 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 may be used to check accuracy and precision of this procedure.

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

6.5.3 Fuse for 1 h at 1000 °C (1832 °F) in the muffle furnace. Have a crucible lid on crucible during fusion.

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

6.5.4 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.

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

NOTE Digesting overnight is preferable.

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

6.5.7 Filter through Whatman #40 filter paper, or equivalent, transferring all solids to filter paper. Wash filter paper and solids twelve (12) times with hot 0.2 % Na_2CO_3 solution. Discard filtrate.

NOTE If iron content is less than 5 %, proceed to 6.5.9. If iron content is more than 5 %, then precipitation of iron hydroxide along with barium sulfate can cause an error. In this case, go to 6.5.8, which gives a method for removing iron and aluminum.

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

6.5.8.1 Dissolve the carbonates with hot 6N HCl, catching the solution in a 250-ml 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 strontium is to be run, catch solution in a 250-ml volumetric flask and dilute to volume mark. With a dry pipette, remove a 10-ml aliquot for strontium and transfer remaining volume into a 400-ml beaker.

6.5.8.2 Remove iron and aluminum by addition NH_4OH dropwise until fumes of NH_3 are given off.

6.5.8.3 Filter through Whatman #541 filter paper or equivalent, catching the filtrate in a 600-ml beaker.

6.5.8.4 Redissolve the precipitate with 6N HCl, catching the filtrate in a 250-ml beaker.

6.5.8.5 Reprecipitate the iron and aluminum with NH_4OH and filter into a 600-ml beaker from 6.5.8.3.

6.5.8.6 Bring the filtrate to approximately 400 ml. Add a few drops of methyl orange and titrate with 6N HCl. Add 1 ml excess 6N HCl. Filtrate should now be at the correct pH to precipitate the $BaSO_4$ according to 6.5.13 to 6.5.16.

6.5.9 Dissolve the carbonates (Ba, Sr, Cr) from the paper with warm HCl solution (20 %), catching the solution in a 600-ml 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 strontium is to be run, catch solution in a 250-ml volumetric flask, wash the filter paper and dilute to volume mark. With a dry pipette, remove a 10-ml aliquot for strontium and transfer remaining volume into a 600-ml beaker for barium analysis.

6.5.10 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.

6.5.11 Wash paper thoroughly with distilled water.

6.5.12 Dilute filtrate to about 400 ml and boil using stirring rod instead of boiling chips.

6.5.13 To the boiling solution, add 10 ml H_2SO_4 solution (1:19) dropwise and boil for 15 min. Allow to stand for at least 4 h. Keep hot but do not boil.

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

6.5.15 Place filter paper in a tared annealing cup. Heat slowly from cold to 850 °C (1562 °F) in the muffle furnace and hold at that temperature for 1 h.

NOTE The $BaSO_4$ should be white.

6.5.16 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 mg.

6.6 Procedure—Strontium Sulfate Determination

To determine strontium, analyze the 10-ml aliquot taken in 6.5.9 for Sr on an AA or ICP spectrophotometer using prepared standards for Sr and manufacturer's recommendations for AA or ICP instrument settings.

6.7 Calculation

6.7.1 If no strontium is present, the amount of barium sulfate present can be calculated.

$$c_{\mathrm{BaSO_4},\%} = \frac{100\, m_1}{m} \tag{1}$$

where

$c_{\mathrm{BaSO_4},\%}$ is the concentration of barium sulfate, expressed as a %;

m is the mass of the barite sample, expressed in g;

m_1 is the mass of the precipitate, expressed in g.

NOTE If a 10-ml aliquot was taken for strontium determination, use 104.17 rather than 100, in Equation (1).

6.7.2 If strontium is present, the amount of barium sulfate and strontium sulfate that are co-precipitated can be calculated.

$$c_{\mathrm{BaSO_4},\%} + c_{\mathrm{SrSO_4},\%} = \frac{100 \times m_1}{m} \tag{2}$$

where

$c_{\mathrm{BaSO_4},\%}$ is the concentration of barium sulfate, expressed as a %;

$c_{\mathrm{SrSO_4},\%}$ is the concentration of strontium sulfate, expressed as a %;

m is the mass of the barite sample, expressed in g;

m_1 is the mass of the precipitate, expressed in g.

NOTE If a 10-ml aliquot was taken for Sr determination use 104.17 rather than 100, in Equation (2).

$$c_{\mathrm{SrSO_4},\%} = 2.0963 \times c_{\mathrm{Sr},\%} \tag{3}$$

where

$c_{\mathrm{SrSO_4},\%}$ is the concentration of strontium sulfate, expressed as a %;

$c_{\mathrm{Sr},\%}$ is the concentration of strontium, expressed as a % from 6.6.

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

$$c_{\mathrm{BaSO_4},\%} = \left[c_{\mathrm{BaSO_4},\%} + c_{\mathrm{SrSO_4},\%}\right] - k \tag{4}$$

where

$c_{\mathrm{SrSO_4},\%}$ is the concentration of strontium sulfate, expressed as a %;

$c_{\mathrm{BaSO_4},\%}$ is the concentration of barium sulfate, expressed as a %;

k is the correction factor taken from Figure 1.

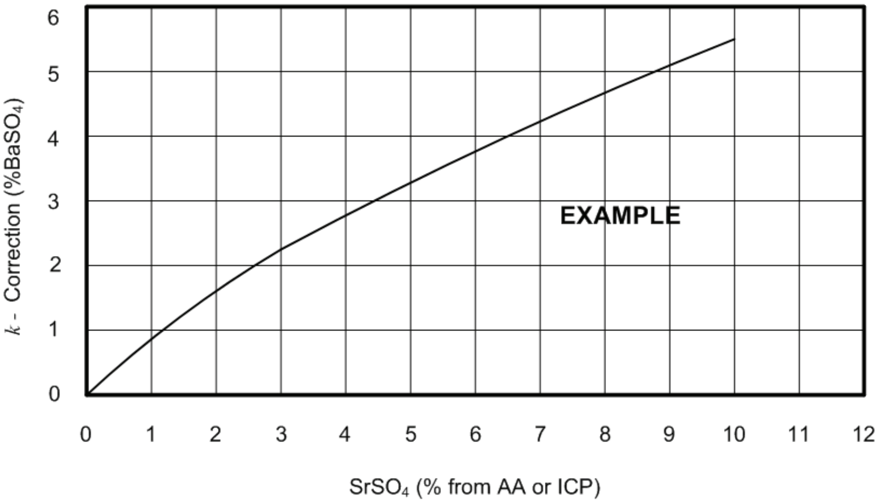

Figure 1—Correction Curve for $SrSO_4$ in $BaSO_4$ (k)

7 Silica and Alumina

7.1 Principle

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

7.2 Reagents and Materials

7.2.1 Distilled or deionized water.

7.2.2 Sulfuric acid (CAS # 7664-93-9) **solution** (1:19 or 20 %), 5 ml H_2SO_4 diluted with 95 ml distilled water.

Caution—Concentrated sulfuric acid is a strong, potentially harmful acid. Use proper safety precautions.

7.2.3 Ammonium molybdate (CAS #12027-67-7) **solution**, 7.5 g of $(NH_4)_6Mo_7O_{24} \bullet 4H_2O$ dissolved in 75 ml water and 25 ml 20 % H_2SO_4, stored in plastic bottle.

7.2.4 Tartaric acid (CAS #87-69-4) **solution** (10 %), 50 g tartaric acid dissolved in 450 ml water, store in plastic bottle.

7.2.5 Reducing solution. Dissolve 0.7 g sodium sulfite (CAS #7757-83-7) in 10 ml water. Add 0.15 g 1-amino-2-naphthol-4-sulfonic acid (CAS #90-51-7) and stir until dissolved. Dissolve 9 g sodium bisulfite (CAS #7631-90-5) in 90 ml 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.

7.2.6 Hydroxylamine hydrochloride (CAS #5470-11-1) **solution** (10 %), 50 g $NH_2OH{\bullet}HCl$/450 ml water.

7.2.7 Calcium chloride (CAS #10043-85-4) **solution**, transfer 7 g $CaCO_3$ to a 250-ml beaker. Add 100 ml water and 15 ml concentrated HCl. Heat to boiling and boil for a few minutes. Cool to room temperature and pour into 500-ml volumetric flask. Dilute to 500-ml mark.

7.2.8 Potassium ferricyanide (CAS #13746-66-2) **solution** (0.75 %), 0.375 g $K_3Fe(CN)_6$ to 50 ml water just before using. DO NOT STORE SOLUTION.

Caution—Potassium ferricyanide is toxic though inhalation, ingestion or skin contact; avoid contact or any chance of ingestion.

7.2.9 Buffer solution, 100 g sodium acetate (CAS #127-09-3) ($NaC_2H_3O_2 \bullet H_2O$)/200 ml water, add 30 ml glacial acetic acid (CAS #64-19-7) and dilute to 500 ml with water.

Caution—Avoid skin contact with glacial acetic acid.

7.2.10 Alizarin Red S (CAS #130-22-3) **solution** (0.05 %), 0.25 g Alizarin Red S (sodium alizarine sulfonate)/500 ml water, stirred and filtered.

7.2.11 Thioglycolic acid (CAS #68-11-1) **solution** (4 %), 4 g $HSCH_2COOH$/96 ml water just before using. DO NOT STORE SOLUTION.

7.2.12 Sodium hydroxide (CAS #1310-73-2) **solution** (30 %), 30 g NaOH/70 ml water, stored in plastic bottle.

7.2.13 Hydrochloric acid (CAS #7647-01-0) s**olution** (6N), 50 ml HCl/50 ml water.

Caution—Concentrated hydrochloric acid is a strong, potentially harmful acid. Use proper safety precautions.

7.2.14 National Institute of Standards and Technology (NIST) Standard Reference Materials 70a (feldspar, potash).

7.3 Apparatus

7.3.1 Ultraviolet (UV)/visible spectrophotometer, any UV/visible spectrophotometer with photometric precision of 0.001 absorbance is suitable.

7.3.2 UV cells, 1 cm, glass or quartz.

7.3.3 Graduated cylinder, plastic, one 10 ml.

7.3.4 Crucibles and lids, nickel, several 75 ml.

7.3.5 Hot plate.

7.3.6 Balance, accuracy to 0.0001 g.

7.3.7 Bunsen burner, tripod and triangle.

7.3.8 Stirring rods, plastic or PTFE (Teflon®) [4].

7.3.9 Beakers, glass, four or more 600 ml.

7.3.10 Volumetric flasks (TC), one 1000 ml, one 500, ml and one 100 ml.

7.3.11 Volumetric pipettes (TD), one 1 ml, one 2 ml, one 5 ml, and one 10 ml.

7.3.12 Serological pipettes (TD), one 1 ml, one 2 ml, one 5 ml, and one 10 ml.

7.3.13 Graduated cylinder (TC), glass, one 10 ml.

7.3.14 Filter paper.

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[4] Teflon® is an example of a suitable product available commercially. This information is given for the convenience of users of this part of API 13K and does not constitute an endorsement by API of these products.

7.4 Procedure—Sample Preparation

7.4.1 Clean each nickel crucible with dilute HCl prior to use.

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

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

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

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

Caution—The hot melt is potentially harmful, so use good laboratory practices (safety precautions) while working with it.

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

7.4.7 Rinse each 600-ml beaker with 6N HCl. Place in each beaker an acid solution of 400 ml water and 20 ml of 6N HCl.

7.4.8 Transfer the contents of each crucible to a 600-ml beaker prepared in 7.3.7. 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.

7.4.9 Transfer contents of each beaker to a 1000-ml volumetric flask and dilute to mark with distilled or deionized water. The solutions containing barite will be cloudy.

7.4.10 Centrifuge a 40 ml to 50 ml portion to obtain clear solution for SiO_2 and Al_2O_3 analyses. If time permits, allow sample to settle and pipette aliquots from top of sample.

NOTE If solutions are not to be used the same day, transfer to tightly stoppered plastic bottle for storage, to avoid evaporation and possible contamination with silica from the glass flask.

7.5 Procedure—Silica Determination

7.5.1 Rinse a 100-ml volumetric flask for each sample prepared in 7.4 with 6N HCl followed by water before using.

7.5.2 Transfer 10 ml of each solution prepared in 7.4 to volumetric flasks. Add approximately 50 ml deionized water to each flask.

7.5.3 Add 2 ml ammonium molybdate solution with a pipette. Swirl the flask during the addition. Allow to stand for 10 min.

7.5.4 Add 4 ml tartaric acid solution with a pipette. Swirl the flask while adding.

7.5.5 Add 1 ml reducing solution with a pipette. Swirl the flask while adding.

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

7.5.7 Determine the absorbance for each solution at 640 nanometers (nm). Use the reagent blank solution as the reference. Record the value for each solution, as n_1.

7.6 Procedure—Alumina Determination

7.6.1 Transfer 15 ml of each solution prepared in 7.4 to 100-ml volumetric flasks.

7.6.2 Add 2 ml calcium chloride solution to each flask.

7.6.3 Add 1 ml 10 % hydroxylamine hydrochloride solution to each flask and swirl to mix.

7.6.4 Add 1 ml potassium ferricyanide solution and swirl to mix.

7.6.5 Add 2 ml thioglycolic acid solution (mercaptoacetic acid) and mix. Allow to stand 5 min.

7.6.6 Add 10 ml Alizarin Red S solution with a pipette.

7.6.7 Dilute to mark with deionized water. Mix and allow to stand 45 min to 75 min.

7.6.8 Determine the absorbance at 475 nm for each solution. Use the reagent blank solution as a reference. Record the value for each solution, as n_2.

7.7 Calculation

7.7.1 Calculate percent silica (%SiO_2) in each sample.

$$f_{\mathrm{SiO_2}} = \frac{\left(c_{\mathrm{SiO_2\ STD,\%}}\right) \times \left(m_2\right)}{n_1} \tag{5}$$

$$c_{\mathrm{SiO_2,\%}} = \frac{f_{\mathrm{SiO_2}}\left(n_2\right)}{m} \tag{6}$$

where

$f_{\mathrm{SiO_2}}$ is the correction factor for silica;

m_2 is the mass of standard sample, expressed in g;

m is the mass of the barite sample, expressed in g;

n_1 is the absorbance value for silica in standard, expressed in nm;

n_2 is the absorbance value for silica in sample, expressed in nm.

7.7.2 Calculate percent alumina (Al_2O_3).

$$f_{\mathrm{Al_2O_3}} = \frac{\left(c_{\mathrm{Al_2O_3\ STD,\%}}\right) \times \left(m_2\right)}{n_3} \tag{7}$$

$$c_{\mathrm{Al_2O_3,\%}} = \frac{f_{\mathrm{Al_2O_3}}\left(n_4\right)}{m} \tag{8}$$

where

$f_{\mathrm{Al_2O_3}}$ is the correction factor for alumina;

m_2 is the mass of standard sample, expressed in g;

m is the mass of the barite sample, expressed in g;

n_3 is the absorbance value for alumina in standard, expressed in nm;

n_4 is the absorbance value for alumina in sample, expressed in nm.

8 Hydrochloric Acid Soluble Metals—Sodium, Potassium, Calcium, Magnesium, Iron, Copper, Manganese, Lead and Zinc

8.1 Principle

8.1.1 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.

8.1.2 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 these metals, see Section 9.

8.2 Reagents and Materials

8.2.1 Deionized or distilled water.

8.2.2 Hydrochloric acid (CAS #7647-01-0), ACS reagent grade.

8.2.3 AA or ICP standards, prepared in 1 % HCl.

NOTE Potential matrix interferences are seen for some metals when using HCl and ICP may be avoided by substituting 1 % HNO_3.

8.3 Apparatus

8.3.1 Sieve, 149 µm.

8.3.2 AA spectrophotometer or ICP spectrometer, any AA or ICP unit is suitable. Instrument settings recommended by the manufacturer should be followed.

8.3.3 Balance, with accuracy of 0.001 g.

8.3.4 Graduated cylinder (TD), one 25 ml.

8.3.5 Beakers, glass, ten 250 ml.

8.3.6 Hot plate/magnetic stirrer.

8.3.7 Stirring bar.

8.3.8 Watch glasses, to cover 250-ml beakers.

8.4 Procedure

8.4.1 Use a representative sample of barite ground such that 100 % passes through sieve.

8.4.2 Weigh a 1.0 g to 1.5 g sample into a 250-ml beaker.

8.4.3 Add 20 ml concentrated HCl. Place a magnetic stirring bar into beaker, and cover with a watch glass.

8.4.4 Boil while stirring for 30 min. Add water 2 or 3 times during this period to maintain 20 ml volume.

8.4.5 Cool and transfer to a 100-ml volumetric flask.

8.4.6 Dilute to mark with deionized water.

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

8.4.8 Analyze the clear supernatant using an AA or ICP spectrophotometer. Run the standards prepared in 1 % HCl. Record all values for the solutions.

8.5 Calculation

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

$$\varphi = \frac{100\,R}{m} \qquad (9)$$

where

R is the AA or ICP reading value, expressed in mg/l;

m is the mass of the barite sample, expressed in g;

φ is the mass fraction of element in sample, mg/l.

9 Procedure—Hydrofluoric, Sulphuric, Nitric, Perchloric Acid Soluble Metals—Sodium, Potassium, Calcium, Magnesium, Iron, Copper, Manganese, Lead, and Zinc

9.1 Principle

The hydrofluoric/sulphuric/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 result of this analysis to the results of the hydrochloric acid soluble metals (see Section 8), an approximation of the pyrite, feldspars, etc., in the barite sample may be made.

9.2 Reagents and Materials

9.2.1 Deionized or distilled water.

9.2.2 Nitric acid (CAS #7697-37-2) (70 %), ACS reagent grade.

Caution—Concentrated nitric acid is a strong, potentially harmful acid. Use proper safety precautions.

9.2.3 AA or ICP standards, prepared in 1 % HCl.

NOTE Potential matrix interferences are seen for some metals when using HCl and ICP may be avoided by substituting 1 % HNO_3.

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

Caution 1—Strong acid mixture, use all proper safety precautions.

Caution 2—Hydrofluoric acid is corrosive and toxic. Avoid skin contact, wear protective clothing and eye/face protection, and work under a fume hood.

Caution 3—Concentrated sulfuric acid is very dangerous. Avoid skin contact, wear protective clothing and eye/face protection.

9.2.5 **Acid mixture B**, mix 100 ml nitric acid (70 %) and 100 ml perchloric acid (CAS #7601-90-3) (72 %).

Caution 1—Strong acid mixture, use all proper safety precautions.

Caution 2—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.

9.2.6 **Hydrazine sulfate** (CAS #10034-93-2) **solution** (0.2 %), 0.2 g hydrazine sulfate/100 ml of water.

9.3 Apparatus

9.3.1 **Sieve**, 149 µm.

9.3.2 **AA or ICP spectrophotometer**, any AA or ICP unit is suitable. Instrument settings recommended by the manufacturer should be followed.

9.3.3 **Balance**, with accuracy of 0.001 g.

9.3.4 **Graduated cylinder** (TD), one 25 ml.

9.3.5 **Beakers**, with covers, PTFE or equivalent, ten 100 ml.

9.3.6 **Hot plate/magnetic stirrer.**

9.3.7 **Stirring bar.**

9.3.8 **Beakers,** glass, ten 400 ml.

9.3.9 **Stirring rod,** one end fitted with a rubber policeman.

9.3.10 **Dropper bottle**, one 25 ml.

9.3.11 **Volumetric flasks**, ten 250 ml.

9.3.12 **Polyethylene bottle**, one 1000 ml.

9.3.13 **Fume hood.**

9.3.14 **Ice bath**, for cooling acid mixture.

9.4 Procedure

9.4.1 Use a representative sample of barite ground such that 100 % passes through the sieve.

9.4.2 Accurately weigh a 0.5 g sample on the analytical balance and transfer into a 100-ml PTFE beaker.

9.4.3 Under a fume hood, add 15 ml of acid mixture A. Swirl the beaker to wet the sample and cover the beaker with PTFE cover.

9.4.4 Place on the hot plate/stirrer set on low heat, and allow beaker to heat overnight (16 h).

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

9.4.6 Cool and transfer to a 400-ml beaker, using a rubber policeman and a minimum of deionized water.

9.4.7 Place on the hot plate/stirrer set on medium heat, about 100 °C to 150 °C (212 °F to 302 °F). Heat for 1 h or until fumes of HF are no longer released.

9.4.8 Heat until fumes of SO_3 start to evolve, then remove beaker from the hot plate/stirrer.

9.4.9 After SO_3 fumes have stopped evolving, add about 4 drops of acid mixture B using the dropper bottle.

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

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

9.4.12 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 ml of 0.2 % hydrazine sulfate.

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

9.4.13 Cool to room temperature. Then transfer contents of the beaker to a 250-ml volumetric flask, dilute to volume with deionized water, and mix.

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

9.4.15 Analyze the clear supernatant using an AA or ICP spectrophotometer. Run the standards prepared in 1 % HCl or HNO_3. Record all values for the solutions.

9.5 Calculation

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

$$\varphi = \frac{100\,R}{m_1} \qquad (9)$$

where

R is the AA or ICP reading value, expressed in mg/l;

m is the mass of the barite sample, expressed in g;

φ is the mass fraction of element in sample, mg/l.

10 Alternative Methods for Iron

10.1 Principle

Several procedures other than those given in Section 8 and Section 9 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).

10.2 Reagents and Materials

10.2.1 Deionized or distilled water.

10.2.2 Sodium carbonate (CAS #497-19-8), ACS reagent grade.

10.2.3 Potassium carbonate (CAS #584-08-7), ACS reagent grade.

10.2.4 Sodium carbonate (CAS #497-19-8) **solution** (0.2 %), 1 g Na_2CO_3/500-ml water.

10.2.5 Hydrochloric acid (CAS #7647-01-0), ACS reagent grade.

Caution—Strong acid, use proper safety precautions.

10.2.6 Nitric acid (CAS #7697-01-0), ACS reagent grade.

Caution—Strong acid, use proper safety precautions.

10.2.7 ***Aqua regia,*** 30 ml HNO_3/90 ml water.

Caution—Strong acid, use proper safety precautions.

10.2.8 Hydrochloric acid (CAS #7647-01-0) **solution** (6N), 50 ml HCl/50 ml water.

10.2.9 Iron AA or ICP standards, prepared in 1 % HCl.

10.2.10 Iron AA or ICP standards, prepared in diluted *aqua regia* (15 ml *aqua regia* diluted to 25 ml with water).

10.2.11 Iron AA or ICP standards, prepared in 1.36N nitric acid (2 ml HNO_3 diluted to 25 ml with water).

10.3 Apparatus

10.3.1 Acid digestion bomb, PTFE lined, 25 ml. Parr bomb, or equivalent.

10.3.2 Balance, with accuracy of 0.0001 g.

10.3.3 Crucibles with lids, platinum, two 25 ml.

10.3.4 Crucible tongs, one 25 cm (10 in.).

10.3.5 Filter paper, Whatman #40, 11.0 cm, or equivalent; and Whatman #42 or #44, 11.0 cm, or equivalent.

10.3.6 Muffle furnace, regulated to 1000 °C ± 20 °C (1832 °F ± 72 °F).

10.3.7 Oven, regulated to 80 °C ± 1 °C (176 °F ± 2 °F).

10.3.8 Volumetric flasks, five 25 ml.

10.3.9 Volumetric flasks, five 100 ml.

10.3.10 Beakers, glass, five 250 ml.

10.3.11 Filter, glass fiber, 2.5 cm.

10.3.12 Hot plate.

10.3.13 Graduated cylinder (TD), one 25 ml.

10.3.14 Sieve, 149 µm.

10.3.15 AA spectrophotometer or ICP spectrophotometer, any AA or ICP unit is suitable. Instrument settings recommended by the manufacturer should be followed.

10.4 Procedure—Nitric Acid Digestion

10.4.1 Use a representative sample of barite ground such that 100 % passes through the sieve.

10.4.2 Weigh a 50 mg sample into a 25-ml acid digestion bomb.

10.4.3 Add 2 ml nitric acid. Cover the bomb, and mount inside the steel shell. Tighten steel lid onto the bottom section.

Caution—Nitric acid is a strong acid, and proper safety precautions should be used when handling it, particularly when it is used in a digestion bomb.

10.4.4 Heat for 1.5 h to 2.0 h in oven at 80 °C ± 1 °C (176 °F ± 2 °F).

10.4.5 Cool, reheat at 80 °C ± 1 °C (176 °F ± 2 °F) for 1.5 h, and cool again to room temperature.

10.4.6 Carefully open the bomb and dilute with deionized water by spraying a few ml of water on the inner walls of the PTFE cell.

10.4.7 Filter through Whatman #42 or #44 filter paper, into a 25-ml volumetric flask. Wash sparingly with deionized water to make a quantitative transfer. Dilute to mark with water.

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

10.5 Calculation—Nitric Acid Digestion

$$c_{\text{Fe,\%}} = \frac{2.5(R)}{m} \tag{10}$$

where

$c_{\text{Fe,\%}}$ is the concentration of iron, expressed as a %;

R is the AA or ICP reading value, expressed in mg/l;

m is the mass of the barite sample, expressed in g.

10.6 Procedure—*Aqua Regia* Digestion

10.6.1 Use a representative sample of barite ground such that 100 % passes through the sieve.

10.6.2 Weigh 2.0 g sample into a 25-ml acid digestion bomb.

10.6.3 Add 15 ml freshly prepared *aqua regia*. Put PTFE cover on the bomb and mount inside the steel shell. Seal by screwing the steel lid onto the bottom section.

Caution—*Aqua regia* is a strong acid, and proper safety precautions should be used when handling it, particularly when it is used in a digestion bomb.

10.6.4 Heat for 1.5 h in oven at 80 °C ± 1 °C (176 ° F ± 2 °F).

10.6.5 Cool to room temperature.

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

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

10.6.8 Analyze for iron using the AA or ICP spectrophotometer. Run standards prepared in diluted *aqua regia*. Record all values.

10.7 Calculation—*Aqua Regia* Digestion

$$c_{\text{Fe},\%} = \frac{2.5\left(R\right)}{m} \tag{11}$$

where

$c_{\text{Fe},\%}$ is the concentration of iron, expressed as a %;

R is the AA or ICP reading value, expressed in mg/l;

m is the mass of the barite sample, expressed in g.

10.8 Procedure—Carbonate Fusion

10.8.1 Use a representative sample of barite ground such that 100 % passes through the sieve.

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

10.8.3 Cover with a platinum lid and fuse for 1 h at 1000 °C (1832 °F) in the muffle furnace.

10.8.4 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.

10.8.5 Cool crucible, and place it in a 250-ml beaker. Add sufficient deionized water to cover crucible.

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

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

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

10.8.9 Dissolve the solid residue by adding hot 6N hydrochloric acid to the filter paper and filtering into a 100-ml volumetric flask.

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

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

10.9 Calculation—Carbonate Fusion

$$c_{\text{Fe},\%} = \frac{10\ \left(R\right)}{m} \tag{12}$$

where

$c_{\mathrm{Fe,\%}}$ is the concentration of iron, expressed as a %;

R is the AA or ICP reading value, expressed in mg/l;

m is the mass of the barite sample, expressed in g.

11 Water-soluble Materials in Barite

11.1 Principle

The water-soluble material in most barite is less than 0.1 % (1000 mg/l) and consists mostly of the chlorides, sulfates, and carbonates of sodium and potassium, and of the chlorides and sulfates of calcium and magnesium. As far as the use of barite in drilling fluid is concerned, only the soluble alkaline earth metals (primarily calcium and magnesium) are of essential importance. Specification for calcium and magnesium reported as calcium is 250 mg/l maximum. Soluble zinc, manganese, copper, lead, and chromium are often determined for environmental purposes.

11.2 Apparatus

11.2.1 Sieve, 149 µm.

11.2.2 AA spectrophotometer or ICP spectrometer, any AA or ICP unit is suitable. Instrument settings recommended by the manufacturer should be followed.

11.2.3 Balance, with accuracy of 0.01 g.

11.2.4 Mechanical shaker, or magnetic stirrer with stirring bar, or field mixer with powerstat.

11.2.5 Graduated cylinder (TD), one 100 ml.

11.2.6 Erlenmeyer flask, one 250 ml.

11.2.7 API low-pressure filter cell, as described in API 13B-1.

11.2.8 Volumetric flask, one 200 ml.

11.2.9 Deionized or distilled water.

11.2.10 Cork or rubber stopper, to fit Erlenmeyer flask.

11.3 Procedure

11.3.1 Use a representative sample of barite ground such that 100 % passes through the sieve.

11.3.2 Weigh 100 g ± 0.05 g barite sample and transfer to a 250-ml Erlenmeyer flask.

11.3.3 Add 100 ml of deionized water.

11.3.4 Stopper and shake on a mechanical shaker for 30 min.

11.3.5 Filter the suspension using an API low-pressure filter cell into a graduated cylinder.

NOTE Filtrate is also used for chlorides, sulfates, carbonates, bicarbonates, hydroxyls, and phosphates in Section 12 through Section 15.

11.3.6 Transfer filtrate to volumetric flask, wash sides of graduated cylinder, transferring washings to flask and dilute to volume with deionized water.

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

11.3.8 Analyze the filtrate for sodium and potassium using a flame photometer, an AA spectrometer in the flame emission mode or ICP spectrometer.

NOTE 1 Make the necessary dilutions, pH adjustments, and matrix matching between samples and standards.

NOTE 2 For very low metal concentrations, it may be necessary to use a graphite furnace or ICP spectrometer.

11.4 Calculation

$$c_{\text{Metal ion},\%} = \frac{100\left(R\right)}{m} \tag{13}$$

where

$c_{\text{Metal ion},\%}$ is the concentration of a metal ion, expressed as a %;

R is the AA or ICP reading value, expressed in mg/l;

m is the mass of the barite sample, expressed in g.

NOTE If dilution was made in analyzing the sample, multiply the results on the AA spectrophotometer by the correction factor for the dilution made.

12 Water-soluble Chlorides

12.1 Principle

The water-soluble chlorides are measured by silver nitrate titration or ion chromatography (IC).

12.2 Reagents and Materials

12.2.1 Silver nitrate (CAS #7761-88-8) **solution** (0.0282N), ACS reagent grade. Dissolve 4.7910 g silver nitrate (equivalent to 0.001 g chloride-ion/ml) in deionized water, and dilute to a volume of one liter. Store in an amber or opaque bottle.

12.2.2 Potassium chromate (CAS #7664-00-6) **indicator**, 5 g K_2CrO_4/100 ml water, stored in dropper bottle.

12.2.3 Sulfuric (CAS #7664-93-9) **or nitric acid** (CAS #7697-37-2) **solution**, (0.02N or N/50) ACS reagent grade.

Caution—Strong acid, use proper safety precautions.

12.2.4 Phenolphthalein (CAS #77-09-8) **indicator**, 1 g phenolphthalein/100 ml of 50:50 ethyl alcohol:water mix, stored in dropper bottle.

12.2.5 Deionized or distilled water.

12.2.6 Chloride ion chromatograph standards, 1 mg/l, 5 mg/l, and 10 mg/l chloride in deionized water.

12.3 Apparatus

12.3.1 Serological (graduated) pipettes (TD), one 1 ml and one 10 ml.

12.3.2 Titration vessel, 100 ml to 150 ml, preferably white.

12.3.3 Stirring rod or magnetic stirrer with stirring bar.

12.3.4 Burette, one 10 ml.

12.3.5 Ion chromatograph, any ion chromatograph unit with settings recommended by manufacturer, is suitable

12.4 Procedure—Water-soluble Chlorides by Titration

12.4.1 Pipette one or more ml of filtrate (from Section 11) into the titration vessel.

12.4.2 Add 2 drops or 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.

12.4.3 Add 25 ml of deionized water and 5 drops to 10 drops of potassium chromate indicator.

12.4.4 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 s. 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 ml and 10 ml.

12.5 Calculation—Water-soluble Chlorides by Titration

$$c_{\mathrm{Cl}} = \frac{10^5 \left(V_{\mathrm{AgNO_3}}\right)}{V} \tag{14}$$

where

c_{Cl} is the concentration of chloride ion, expressed in mg/l;

V_{AgNO3} is the volume of silver nitrate used, expressed in ml;

V is the volume of barite-water leachate filtrate, expressed in ml.

12.6 Procedure—Water-soluble Chlorides by Ion Chromatography

12.6.1 Use a suitable amount of filtration, from Section 11, for the analyses.

12.6.2 Analyze the filtrate for chlorides by IC using manufacturer’s accepted instrument settings and procedures.

NOTE Water-soluble sulfates (13.7) may also be determined at the same time.

12.6.3 Analyze standards prepared in deionized water.

12.6.4 Record all values and correct for any dilutions made.

12.7 Calculation—Water-soluble Chlorides by Ion Chromatography

$$c_{\mathrm{Cl}} = R_{\mathrm{IC}} \tag{15}$$

where

c_{C1} is the concentration of chloride ion, expressed in mg/l;

R_{IC} is the measured value from ion chromatograph, expressed in mg/l.

13 Water-soluble Sulfates

13.1 Principle

The water-soluble sulfates are analyzed turbidimetrically using the reaction of soluble sulfates with barium chloride or IC.

13.2 Reagents and Materials

13.2.1 Hydrochloric acid (CAS #7647-01-0), ACS reagent grade.

Caution—Concentrated hydrochloric acid is a strong, potentially harmful acid. Use proper safety precautions.

13.2.2 Hydrochloric acid (CAS #7647-01-0) **solution** (6N), 50 ml HCl/50 ml of water.

13.2.3 Sodium sulfate, **anhydrous** (CAS #7757-82-6), ACS reagent grade.

13.2.4 Barium chloride (CAS #10361-37-2), ACS reagent grade crystals.

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

13.2.5 Deionized or distilled water.

13.2.6 Sulfate ion chromatograph standards, 1 mg/l, 5 mg/l, and 10 mg/l sulfates in deionized water.

13.3 Apparatus

13.3.1 Spectrophotometer, filter photometer, or turbidimeter, with proper cells for the meter of choice. Use spectrophotometer at a wave length of 420 nm.

13.3.2 Volumetric flasks, one 1000 ml, four 100 ml.

13.3.3 Beakers, seven 50 ml.

13.3.4 Graduated cylinder (TD), one 10 ml.

13.3.5 Timer.

13.3.6 Pipettes, one 10 ml, one 5 ml, one 20 ml.

13.3.7 Cuvettes or appropriate container for transmission or turbidimetric analysis.

13.3.8 Graph paper, linear.

13.3.9 Ceramic spoon, Coors No. 65001, or equivalent.

13.3.10 Ion chromatograph, any ion chromatograph unit with settings recommended by manufacturer, is suitable.

13.4 Procedure—Preparation of Calibration Curve for Turbidity Method

13.4.1 Weigh out 0.1480 g of sodium sulfate. Transfer quantitatively to 1000-ml volumetric flask. Add about 100 ml deionized water, mix to dissolve, and dilute to mark. Mix well. This is the stock solution containing 100 ml/l SO_4^{-2}.

13.4.2 Prepare 20 ml/l, 40 ml/l, 60 ml/l, and 80 ml/l SO_4 standard solution by pipetting 20 ml, 40 ml, 60 ml and 80 ml of stock solution into separate 100-ml volumetric flasks and making up to volume with deionized water. Mix well.

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

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

13.4.5 Let samples stand 4 min.

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

13.4.7 Transfer the blank solution to a sample cell, insert cell in instrument, and record the absorbance reading.

13.4.8 Repeat 13.4.7 with the other standard solutions.

13.4.9 Plot the absorbance readings versus the ml/l SO_4, using linear graph paper.

13.5 Procedure—Analysis for Water-soluble Sulfates by Turbidity Method

13.5.1 Pipette 15 ml of sample (barite-water leachate from Section 11) into a clean 50-ml beaker. Add 1 ml 6N HCl and one spoonful of barium chloride crystals. Swirl gently to mix.

13.5.2 Let sample stand 4 min.

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

13.5.4 Use the calibration chart (see 13.4) 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.

13.6 Calculation—Water-soluble Sulfates by Turbidity Method

$$c_{\mathrm{SO_4}} = \frac{100 R_{\mathrm{M}}}{m} \qquad (16)$$

where

$c_{\mathrm{SO_4}}$ is the concentration of sulfate ion, expressed in mg/l;

m is the mass of barite sample, expressed in g;

R_{M} is the meter reading, expressed in mg/l.

13.7 Procedure—Analysis for Water-soluble Sulfate by Ion Chromatography

13.7.1 Use a suitable amount of filtrate from Section 11 for the analysis.

13.7.2 Analyze the filtrate for sulfate by IC using manufacturers' accepted instrument settings and procedures. Analyze standards prepared in deionized water.

13.7.3 Record all values and correct for any dilutions made.

13.8 Calculation—Water-soluble Sulfates by Ion Chromatography

$$c_{\mathrm{SO_4}} = R_{\mathrm{IC}} \tag{17}$$

where

$c_{\mathrm{SO_4}}$ is the concentration of sulfate ion, expressed in mg/l;

R_{IC} is the measured value from ion chromatograph, expressed in mg/l.

14 Water-soluble Carbonates, Bicarbonates, and Hydroxyl Ions

14.1 Principle

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.

14.2 Reagents and Materials

14.2.1 Sulfuric acid (CAS #7664-93-9) **solution** (0.02N or N/50)

Caution—Concentrated sulfuric acid is a strong, potentially harmful acid. Use proper safety precautions.

14.2.2 Phenolphthalein (CAS #77-09-8) **indicator**, 1 g phenolphthalein/100 ml of 50:50 ethyl alcohol:water solution, stored in dropper bottle.

14.2.3 Methyl purple (CAS #1340-02-9) **indicator**, 0.1 g methyl purple/100 ml water, stored in dropper bottle.

14.3 Apparatus

14.3.1 pH meter (optional, but more accurate).

14.3.2 Titration vessel, 100 ml to 150 ml, preferably white.

14.3.3 Serological (graduated) pipette (TD), one 10 ml.

14.3.4 Volumetric pipettes, one 10 ml and one 25 ml.

14.3.5 Stirring rod or magnetic stirrer with stirring bar.

14.4 Procedure

14.4.1 Pipette 10 ml of barite-water leachate from Section 11 into an appropriate sized beaker. (Use a 25 ml sample, if necessary).

14.4.2 Add 2 drops of phenolphthalein indicator and titrate to the colorless ends point with 0.02N H_2SO_4. This is a pH 8.3 if using a pH meter. Record the number of ml of acid required, as V_P.

14.4.3 Add 2 drops of methyl purple indicator and, without refilling the pipette, continue the titration to the methyl purple endpoint, noted by the solution changing from green to purple. This is a pH4.3 if using a pH meter. Record the number of ml of acid required, as V_M.

14.5 Calculation

14.5.1 Determine relationship of V_P and V_M.

If $V_P = 0$,	the alkalinity is due to bicarbonate ion alone;
If $V_P = V_M$,	the alkalinity is due to hydroxyl ion alone;
If $2\,V_P = V_M$,	the alkalinity is due to carbonate ion alone;
If $2\,V_P > V_M$,	the alkalinity is due to a mixture of carbonate and hydroxyl ions;
If $2\,V_P < V_M$,	the alkalinity is due to a mixture of carbonate and bicarbonate ions.

14.5.2 Ion concentrations.

$$c_{OH} = \frac{(2 - V_P V_M)(1.7\text{x}10^4)}{V \text{ x } m} \tag{18}$$

$$c_{HCO_3} = \frac{(V_M - 2V_P)(6.1\text{x}10^4)}{V \text{ x } m} \tag{19}$$

If hydroxide is present:

$$c_{CO_3} = \frac{(V_M - V_P)(6 \text{ x } 10^4)}{V \text{ x } m} \tag{20}$$

If hydroxide is absent:

$$c_{CO_3} = \frac{V_P \text{ x } 6 \text{ x } 10^4}{V \text{ x } m} \tag{21}$$

where

c_{OH} is the concentration of hydroxyl ion, expressed in mg/l;

c_{HCO3} is the concentration of bicarbonate ion, expressed in mg/l;

c_{CO3} is the concentration of carbonate ion, expressed in mg/l;

V is the volume of barite-water leachate sample, expressed in ml;

m is the mass of the barite sample, expressed in g;

V_P is the volume of 0.2N H_2SO_4 used in titration to reach phenolphthalein endpoint, expressed in ml;

V_M is the volume of 0.2N H_2SO_4 used in titration to reach methyl purple endpoint, expressed in ml.

15 Water-soluble Phosphates

15.1 Principle

Water-soluble phosphates, both ortho and condensed, are determined colorimetrically by use of the Hach® [5] premixed reagents PhosVer III® [6], or equivalent. A sample of the filtrate from the water extraction of the barite sample (Section 11) is heated in the presence of excess acid to convert all phosphates to orthophosphate. Color is developed by the addition of PhosVer III 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. IC may be used as an alternative for phosphate determinations.

15.2 Reagents and Materials

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

Caution—Concentrated sulfuric acid is a strong, potentially harmful acid. Use proper safety precautions.

15.2.2 Sulfuric acid (CAS #7664-93-9) **solution** (5.25N), 143 ml H_2SO_4/1 l solution.

15.2.3 Sodium hydroxide (CAS #1310-73-2), ACS reagent grade.

Caution—Concentrated sodium hydroxide is a strong, potentially harmful acid. Use proper safety precautions.

15.2.4 Sodium hydroxide (CAS #1310-73-2) **solution** (5.0N), 200 g NaOH/1 l solution.

15.2.5 Sodium phosphate (CAS #10361-03-2), ACS reagent grade, ($NaPO_4 \bullet 12H_2O$).

15.2.6 Standard phosphate stock (CAS #10361-03-2) **solution** (1000 mg/l PO_4^{-3}), 1.726 g sodium phosphate/1 l solution.

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

15.2.8 Distilled water.

15.3 Apparatus

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

15.3.2 Erlenmeyer flasks, several 125 ml.

15.3.3 Graduated mixing cylinders, with stoppers, several 25 ml.

15.3.4 Volumetric pipettes, several of various sizes.

15.3.5 Volumetric flasks, several 100 ml.

15.3.6 Hot plate.

15.3.7 Graph paper, linear.

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[5] Hach colorimeter® is an example of suitable product available commercially. This information is given for the convenience of the users of this part of API 13K and does not constitute an endorsement by API of this product.

[6] PhosVer III® is an example of a suitable product available commercially. This information is given for the convenience of the users of this part of API 13K and does not constitute an endorsement by API of this product.

15.4 Procedure—Preparation of Calibration Curve

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

15.4.2 Transfer 25 ml of each solution to a 125-ml Erlenmeyer flask. Prepare a reagent blank using 25 ml of distilled water.

15.4.3 Add 2.0 ml of 5.25N sulfuric acid and boil gently on a hot plate for 30 min, maintaining sample volume at 15 ml to 20 ml by additions of distilled water.

15.4.4 After 30 min, remove from the hot plate, cool to room temperature, add 2.0 ml of 5.0N sodium hydroxide solution, and transfer to the 25-ml graduated mixing cylinders.

15.4.5 Add the contents of one PhosVer III, reagent pillow, dilute to the 25-ml mark with distilled water. Stopper and mix.

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

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

15.5 Procedure—Analysis of Samples

15.5.1 Transfer 5 ml of the barite-water leachate from Section 11 to a 125-ml Erlenmeyer flask and add sufficient distilled water to bring total volume to 25 ml.

15.5.2 Proceed with steps from above, 15.4.3 through 15.4.6.

15.5.3 Determine the phosphate content, in mg/l, in the final 25 ml solution by reference to the previously prepared calibration curve.

15.5.4 If the phosphate ion content is below the range of the calibration curve, take a larger portion of the barite-water leachate from Section 11. 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 filtration that was present in the solution in which the color was developed.

15.6 Calculation

$$c_{\mathrm{PO_4}} = \frac{25 \times R_{\mathrm{PO_4}}}{V} \qquad (23)$$

where

$c_{\mathrm{PO_4}}$ is the concentration of phosphate, expressed in mg/l;

$R_{\mathrm{PO_4}}$ is the phosphate ion concentration reading from the calibration curve, expressed in mg/l;

V is the volume of barite-water leachate sample, expressed in ml.

16 Loss on Ignition

16.1 Principle

The LOI of barite ores is the weight loss after heating to 1000 °C (1832 °F). The LOI can be due to several things, including the following.

a) Moisture—rarely over 0.1 % to 0.2 %, and usually not separately determined.

b) Combined water—from clays, hydrated iron oxides, gypsum, and other accessory minerals.

c) Organic and carbonaceous matter.

d) Thermal decomposition of carbonate minerals. This can be offset by the metal oxides formed combining with sulfur oxides to form metal sulfides.

e) Loss of sulfur from decomposition of pyrite. This can be offset by the iron taking up oxygen.

16.2 Reagents and Materials, and Apparatus

16.2.1 Desiccant, silica gel (CAS #7631-86-9), indicating.

16.2.2 Sieve, 149 µm.

16.2.3 Balance, with accuracy of 0.0001 g.

16.2.4 Crucibles and lids, platinum, two 25 ml.

16.2.5 Muffle furnace, regulated to 1000 °C (1832 °F).

16.2.6 Dessicator, glass.

16.3 Procedure

16.3.1 Use a representative sample of barite ground such that 100 % passes through the sieve.

16.3.2 Accurately weigh a 1.0 g to 1.5 g sample on the analytical balance into an ignited platinum crucible. Record this as m_6.

16.3.3 Cover with lid and place crucible into a cold muffle furnace. Heat to 1000 °C (1832 °F) and hold at 1000 °C (1832 °F) for 30 min.

16.3.4 Cool in desiccator. Remove lid and reweigh on the analytical balance. Record this as m_7.

16.4 Calculation

$$\%\text{LOI} = \frac{100\ (m_4 - m_5)}{m_4 - m_3} \qquad (24)$$

where

LOI is the loss on ignition, expressed as a %;

m_3 is the mass of the crucible, expressed in g;

m_4 is the mass of barite sample and crucible, expressed in g;

m_5 is the mass of ignited sample and crucible, expressed in g.

17 Siderite Content

17.1 Principle

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 AA, ICP spectrometer, or colorimetry.

17.2 Reagents and Materials

17.2.1 Distilled or deionized water.

17.2.2 Sodium hydroxide (CAS #1310-73-2), ACS reagent grade.

Caution—Concentrated sodium hydroxide is a strong, potentially harmful acid. Use proper safety precautions.

17.2.3 Sodium hydroxide (CAS #1310-73-2) **solution** (8N), 320 g NaOH/l distilled water.

17.2.4 EDTA solution, 40 g Na_4EDTA (CAS #64-02-8)/100 ml water.

NOTE If tetrasodium salt is not available, solution may be prepared from disodium salt. Into a 500-ml beaker, add 325 ml deionized water, 196.0 g $Na_2H_2EDTA \bullet 2H_2O$ (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 ml.

17.2.5 Hydrochloric acid (CAS #7647-01-0), ACS reagent grade.

17.2.6 Hydrochloric acid (CAS #7647-01-0) **solution** (1N), 8.3 ml HCl/91.7 ml water.

17.2.7 FerroVer powder pillows® [7], Hach Catalog Number 854, or equivalent, 0 mg/l to 3 mg/l range.

17.2.8 AA or ICP iron standards.

17.3 Apparatus

17.3.1 Balance, with accuracy of 0.1 g.

17.3.2 Flask, round bottom, 250 ml.

17.3.3 Condenser, water-cooled.

17.3.4 Heating mantle, to fit 250-ml round-bottom flask.

17.3.5 Variable power regulator.

17.3.6 Funnel, 77 mm (3 in.).

17.3.7 Filter paper, Whatman #1 or equivalent, 11 cm.

17.3.8 AA spectrophotometer, ICP spectrometer, or Hach test kit for iron, or equivalent.

17.3.9 Colorimeter, or color comparator wheel.

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[7] FerroVer powder pillows® are an example of a suitable product available commercially. This information is given for the convenience of users of this part of API 13K and does not constitute an endorsement by API of these products.

17.3.10 Volumetric flask, one 1 l.

17.4 Procedure—Extraction

17.4.1 Add 20 g barite to 100 ml extraction solution in the round-bottom flask.

17.4.2 Adjust pH to 11.0 with 8N NaOH.

17.4.3 Attach condenser and reflux gently for 2 h.

17.4.4 Filter solution.

17.5 Procedure—Iron Content by Atomic Absorption or Inductively Coupled Plasma

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

17.6 Calculation—Extraction

$$c_{\mathrm{FeCO_3},\%} = 0.00104 \times c_{\mathrm{Fe}} \tag{25}$$

where

$c_{\mathrm{FeCO_3},\%}$ is the concentration of iron carbonate, expressed as a %;

c_{Fe} is the concentration of iron in filtrate, expressed in ml/l.

17.7 Procedure—Iron Content by Colorimetric Determination

17.7.1 Transfer 10 ml of filtrate from 17.4 to a 1 l volumetric flask.

NOTE Use 1 ml of filtrate from 17.4 if the iron content is known to be high.

17.7.2 Add about 900 ml deionized water, and adjust the pH with dilute HCl to pH 5.0 ± 0.2.

17.7.3 Dilute to volume with deionized water.

17.7.4 Measure 25 ml of solution into clean colorimeter bottle.

17.7.5 Add contents of FerroVer powder pillow to sample. Swirl the bottle to mix. If iron is present, an orange color will develop.

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

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

17.7.8 Insert the Iron Meter Scale in the meter and use the 4445 Color Filter. Adjust the light control for a reading of 0 mg/l.

17.7.9 Place the prepared sample in the light cell and read the mg/l iron.

NOTE 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.

NOTE 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, test a diluted sample and compare the result with the original result. Begin with a 1:1 dilution first, followed by a 9:1 dilution if the original 1:1 dilution results do not coincide.

NOTE 3 FerroVer has a limited shelf life. Under ideal (cool, dry) conditions, it will keep for several years. Under poor (warm, moist) condition, it fails after six months. Periodically perform a test on a sample containing Fe^{+2}. If the characteristic orange color develops, the FerroVer is still in satisfactory condition. IF no orange color develops, the FerroVer powder pillow should be discarded.

17.8 Calculation—Iron Content by Colorimetric Determination

17.8.1 For 10 ml diluted to 1000 ml:

$$c_{\mathrm{FeCO_3},\%} = 0.104 \times c_{\mathrm{Fe}} \tag{26}$$

17.8.2 For 1 ml diluted to 1000 ml:

$$c_{\mathrm{FeCO_3},\%} = 1.04 \times c_{\mathrm{Fe}} \tag{27}$$

where

$c_{\mathrm{FeCO_3},\%}$ is the concentration of iron carbonate, expressed as a %;

c_{Fe} is the concentration of iron in filtrate, expressed in ml/l.

18 Zinc Carbonate and Lead Carbonate

18.1 Principle

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.

18.2 Reagents and Materials, and Apparatus

18.2.1 **Acetic acid** (CAS #64-19-7) **solution**, 10 % by weight.

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

18.2.3 **Desiccator.**

18.2.4 **Mortar and pestle**.

18.2.5 **Sieve**, 75 µm.

18.2.6 **Balance**, with accuracy of 0.0001 g.

18.2.7 **Beaker**, one 250 ml.

18.2.8 **Funnel**, one 77 mm (3 in.).

18.2.9 **Filter paper**, Whatman #1, or equivalent, 11 cm.

18.2.10 **Volumetric flask**, one 100 ml.

18.2.11 **Magnetic stirrer**, with stirring bar.

18.2.12 **AA spectrophotometer or ICP spectrometer**, any AA or ICP unit is suitable. Instrument settings recommended by the manufacturer should be followed.

18.2.13 AA or ICP standards, Pb and Zn, prepared in 10 % by weight acetic acid.

18.3 Procedure—Extraction

18.3.1 Dry a sample of barite for 4 h in oven. Use a representative sample of barite ground such that 100 % passes through the sieve.

18.3.2 Weigh a 1 g sample of barite into the 250-ml beaker.

18.3.3 Add 80 ml of 10 % acetic acid.

18.3.4 Place stirring bar into beaker, and place beaker on stirrer. Stir for 10 min.

18.3.5 Filter through filter paper into a 100-ml volumetric flask.

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

18.3.7 Dilute to volume with 10 % acetic acid.

18.4 Procedure—Lead and Zinc Determination by Atomic Absorption or Inductively Coupled Plasma

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

18.5 Calculation

$$c_{\mathrm{ZnCO_3},\%} = \frac{0.0192 \text{ x } c_{\mathrm{Zn}}}{m} \tag{28}$$

$$c_{\mathrm{PbCO_3},\%} = \frac{0.0129 \text{ x } c_{\mathrm{Pb}}}{m} \tag{29}$$

where

$c_{\mathrm{ZnCO_3},\%}$ is the concentration of zinc carbonate, expressed as a %;

c_{Zn} is the concentration of zinc in filtrate, expressed in mg/l;

m is the mass of the barite sample, expressed in g;

$c_{\mathrm{PbCO_3},\%}$ is the concentration of lead carbonate, expressed as a %;

c_{Pb} is the concentration of lead in filtrate, expressed in mg/l.

19 Total Carbonate

19.1 Principle

19.1.1 Total carbonate can be released from barite by treating a sample with acid. The evolved carbonate is measured with the GGT and carbon dioxide Dräger Tube, or equivalent. The procedure is performed in an air-tight gas train with nonreactive carrier gas.

19.1.2 The barite sample appropriate to this procedure is a commercial barite of drilling fluid grade that has been ground at a mill to a size to meet fluid requirements. No further (laboratory) grinding of the sample is recommended.

19.2 Reagents and Materials

19.2.1 Bottled carrier gas, nitrogen source, with gauge and pressure regulator.

19.2.2 Sulfuric acid (CAS #7664-93-9), ACS reagent grade, 2.5 mols/liter (5N)—139 ml H_2SO_4/liter of solution.

Caution—Sulfuric acid is a strong acid, and proper safety precautions should be used when handling it.

19.2.3 Distilled or deionized water.

19.2.4 Defoamer, in a dropper bottle.

19.3 Apparatus

19.3.1 Balance, with accuracy of 0.0001 g.

19.3.2 GGT apparatus, consisting of a transparent plastic gas train, and inert gas supply and pressure regulator, and a Dräger tube, or equivalent. See API 13B-1.

Specifications of the GGT are as follows.

a) Body.

Chamber 1:		
	depth	90 mm (3.54 in.),
	diameter	38 mm (1.52 in.).
Chambers 2 and 3:		
	depth	90 mm (3.54 in.),
	diameter	30 mm (1.18 in.).
Passages between chambers:		
	diameter	2.0 mm (0.08 in.).
Material:		
	transparent material or glass that is inert to acid, sulfides and hydrogen sulfide gas.	

b) Dispersion Tube.

Stem:		
	diameter	8.0 mm (0.315 in.),
	length	150 mm (5.9 in.).
Dispersion frit (bell-shaped, fine):		
	diameter	30 mm (1.18 in.).
Material:		
	low coefficient of expansion, heat-resistant glass.	

c) Flow meter, floating-ball type preferred, capable of measuring 300 ml/min of CO_2 gas.

d) Flexible tubing, type inert to hydrogen sulfide and carrier gas. Latex rubber or equivalent, is preferred.

e) Fittings and rigid tubing, type inert to hydrogen sulfide and acid.

f) Rubber septum.

19.3.3 Dräger CO_2 analysis tube® [8], marked Carbon Dioxide 100/a (No. 8101811) 100 to 3000 mg/l.

19.3.4 Hypodermic syringe, with 21-gauge needle, one 10 ml (for acid).

19.3.5 Magnetic stirrer, with plastic or glass covered stirring bar, 0.6 cm × 2.5 cm ($^1/_4$ in. × 1 in.).

19.3.6 Dräger Alcotest gas bag, No. 7626425, or equivalent.

19.3.7 Dräger Accyri hand-operated bellows gas-detector vacuum pump® [9], Model 31, or equivalent.

19.3.8 Stopcock, 2-way bore, 8 mm (0.315 in.) glass with PTFE plug.

19.4 Procedure

19.4.1 Ensure that the gas train is clean, dry and on a level surface, with the top removed.

19.4.2 Adjust the magnetic stirrer below the gas train body so that the stirring bar will freely rotate and vigorously agitate contents in Chamber 1.

19.4.3 Weigh 0.1 g to 0.3 g of barite sample to 0.1 mg. Transfer sample into Chamber 1. Record sample mass as m.

19.4.4 Add 20 ml of deionized water and a few drops of defoamer.

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

19.4.6 Attach the flexible tubing from the nitrogen source to the glass dispersion tube of Chamber 1.

19.4.7 Adjust the dispersion tube in Chamber 1 to approximately 2.9 cm ($^3/_4$ in.) above the bottom, just sufficient to clear the stirring bar.

19.4.8 Gently flow carrier gas for 1 min to purge air from the system. Check for leaks. Shut off carrier gas.

19.4.9 Fully collapse the gas bag and simultaneously check for leaks. To do this, connect the gas bag and stopcock to the hand pump. Open stopcock, fully depress and release the hand pump. When the gas bag is completely empty and free of leaks, the pump will remain depressed for several minutes. If leakage is detected, check the pump and all connections. To check the pump alone, insert a sealed Dräger tube into the pump opening and depress the bellows. It will remain depressed if pump does not leak.

19.4.10 With the gas bag fully collapsed, install flexible tubing from the stopcock and gas bag onto the outlet of Chamber 3.

19.4.11 Slowly inject 10 ml of 2.5 mol/l (5N) sulfuric acid solution into Chamber 1 through the rubber septum. Use a clean syringe and needle, and start rapid stirring of Chamber 1.

19.4.12 Open the stopcock on the gas bag. Restart the nitrogen gas flow and allow gas bag to fill steadily during a 10 min interval. When the gas bag is firm to the touch (do not burst it) shut off the nitrogen gas flow, and close the stopcock. Immediately proceed to 19.4.13.

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[8] Dräger CO_2 analysis tube® is an example of a suitable brand available commercially. This information is given for the convenience of users of API 13K and does not constitute and endorsement by API of these products.

[9] Dräger Accyri hand-operated bellows gas detector vacuum pump® is an example of suitable brand available commercially. This information is given for the convenience of users of this part of API 13K and does not constitute an endorsement by API of these products.

19.4.13 Remove the gas bag from the gas train. Break the top off each end of CO_2 Dräger tube. Observe that an arrow on the tube indicates gas flow direction. Attach the Dräger hand pump to the downstream end of the Dräger tube.

19.4.14 Open the stopcock valve on the bag. With steady hand pressure, fully depress the and pump. Release the pump so that the gas flows out of the bag and through the Dräger tube. Operate the pump and count the strokes until the bag is empty.

NOTE Ten strokes should empty the bag. More than ten strokes indicates that leakage has occurred and the test results will not be correct.

19.4.15 Observe that purple/blue stain shows on the Dräger tube when CO_2 is present in the gas bag. If the Dräger tube becomes almost filled with stain, shut off the valve and quickly replace the tube with a new one. Reopen the valve and continue the analysis.

19.4.16 Record total stain reading from Dräger(s) tube in units marked on the tube. Include the faint blue tinge in the purple stain length reading. If the stain reading is too low, try starting with a larger sample size. If the stain reading fills two complete tubes, repeat with a smaller sample size.

NOTE To clean the gas train, remove the flexible tubing and remove the top. Wash with warm water and mild detergent, using a soft brush. Use a pipe cleaner to clean the passage between the chambers. Wash, rinse, and then blow out the dispersion tube with gas. Rinse the unit with deionized water and allow to drain dry.

19.5 Calculation

$$c_{\mathrm{CO_3},\%} = \frac{l_{\mathrm{ST}} \times f_{\mathrm{CO3}}}{m \times 10\,000} \tag{30}$$

where

$c_{\mathrm{CO_3},\%}$ is the concentration of CO_3, expressed as a %;

m is the mass of the barite sample, expressed in g;

l_{ST} is the Dräger tube's maximum darkened length, expressed in units marked on the tube;

f_{CO3} is the tube factor, i.e. 2.5 for Dräger No. 8101811.

20 Acid-soluble Sulfides

20.1 Principle

20.1.1 The concentration of acid-soluble sulfides in drilling fluid barite can be determined by acidizing with HCl and measuring the evolved H_2S with the GGT. The GGT separates the gas from the liquid, preventing contamination of the H_2S detector.

20.1.2 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 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.

20.1.3 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 will occur.

20.2 Reagents and Materials

20.2.1 Carrier gas, inert to hydrogen sulfide, acid and Dräger tube reagents. Nitrogen is preferred but helium is acceptable. Avoid air, nitrous oxide or other oxygen-containing gases.

20.2.2 Hydrochloric acid (CAS #7647-01-0) **solution**, (5N), 410 ml HCl/l of solution.

Caution—Concentrated hydrochloric acid is a strong, potentially harmful acid. Use proper safety precautions.

20.2.3 Defoamer, in a dropper bottle.

20.2.4 Distilled or deionized water.

20.3 Apparatus

20.3.1 Balance, with accuracy of 0.0001 g.

20.3.2 GGT apparatus, consisting of a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flow meter and a Dräger tube. See API 13B-1.

Specifications of the GGT are as follows.

a) Body.

Chamber 1:		
	depth	90 mm (3.54 in.),
	diameter	38 mm (1.52 in.).
Chambers 2 and 3:		
	depth	90 mm (3.54 in.),
	diameter	30 mm (1.18 in.).
Passages between chambers:		
	diameter	2.0 mm (0.08 in.).
Material:		
	transparent material or glass that is inert to acid, sulfides and hydrogen sulfide gas.	

b) Dispersion tube.

Stem:		
	diameter	8.0 mm (0.315 in.),
	length	150 mm (5.9 in.).
Dispersion frit (bell-shaped, fine):		
	diameter	30 mm (1.18 in.).
Material:		
	low coefficient of expansion, heat-resistant glass.	

c) Flow meter, floating-ball type preferred, capable of measuring 300 ml/min of CO_2 gas.

d) Flexible tubing, type inert to hydrogen sulfide and carrier gas. Latex rubber or equivalent is preferred.

e) Fittings and rigid tubing, type inert to hydrogen sulfide and acid.

f) Rubber septum.

20.3.3 Dräger H_2S Analysis Tubes

20.3.3.1 Low range: marked H_2S 100/a (No. CH 29101, 100 mg/l to 200 mg/l H_2S);

20.3.3.2 High range: marked H_2S 0.2 %/A (No. CH 28101, 0.2 volume % to 7 volume % H_2S).

20.3.4 Hypodermic syringe, one 20 ml for hydrochloric acid.

20.3.5 Magnetic stirrer, with plastic- or glass-covered stirring bar—0.6 cm × 2.5 cm ($^1/_4$ in. × 1 in.).

20.4 Procedure

20.4.1 Ensure that the gas train is clean, dry and on a level surface, with the top removed.

NOTE Moisture in the train can cause the ball in the flow meter to float erratically and can affect the accuracy of the Dräger tube reading.

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

20.4.3 Bottle nitrogen or helium shall be used as a carrier gas.

Caution—Do not use nitrous oxide cartridges or air as pressure sources for GGT analysis. Under pressure, nitrous oxide or air may result in oxidation of hydrogen sulfide. Nitrous oxide cartridges shall be used only for GGT carbonate analysis.

20.4.4 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. Record as original sample mass, m.

Table 2—Dräger Tube Identification, Sample Volume, and Tube Factor to be Used for Various Sulfide Ranges

Sulfide Range	Sample Mass m_S	Dräger Tube Identification	Tube Factor [a] F
mg/l	G	(see tube body)	(used in calculation)
1.2 to 24	10.0	H_2S 100/a	0.12 [b]
2.4 to 48	5.0	H_2S 100/a	0.12 [b]
4.8 to 96	2.5	H_2S 100/a	0.12 [b]
30 to 1050	10.0	H_2S 0.2 %/a	1450 [c]
60 to 2100	5.0	H_2S 0.2 %/a	1450 [c]
120 to 4200	2.5	H_2S 0.2 %/a	1450 [c]

[a] If other tubes are used, it is necessary to change the tube factors in Table A.1 in accordance with the manufacturer's specification.

[b] Tube factor 0.12 applies to tubes marked H_2S 100/a (Cat. No. CH 29101) with 100 mg/l to 2000 mg/l scale. For older tubes with 1 to 20 scale, use a tube factor of 12.

[c] Tube factor 1450 applies to tubes marked H_2S 0.2%/a (Cat. No. CH 28101) with 0.2 vol% to 7.0 vol% scale. For older tubes with 1 to 17 cubic centimeters scale, use tube factor 600 times the ratio: Batch Factor/0.40.

20.4.5 Add 10 ml of deionized water and a few drops of defoamer.

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

20.4.7 Carefully break the tip off both ends of the 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.

20.4.8 Attach the flexible tubing to the dispersion tube and to the Dräger tube.

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 provide pressure relief in the event of over pressurization.

20.4.9 Adjust the dispersion tube in Chamber 1 to approximately 2.0 cm ($^3/_4$ in.) above the bottom, just sufficient to clear the stir bar.

20.4.10 Gently flow carrier gas for a 10 s period to purge air from the gas train. Check for leaks. Shut off carrier gas.

20.4.11 Add 20 ml of 5N hydrochloric acid solution into Chamber 1 and start rapid stirring of Chamber 1.

20.4.12 Immediately restart carrier gas flow. Adjust the rate between 200 ml/min and 400 ml/min. Keep floating ball in flowmeter between the two marks.

20.4.13 Observe changes in appearance of the Dräger tube. Record the maximum Darkened Length (in units marked on tube) after flowing for a total of 60 min. If first Dräger tube becomes almost filled with stain, shut off flow and quickly replace with a new tube. Restart the gas flow and continue the analysis. Add the two stain lengths and use the sum in the calculation.

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.

20.5 Calculation

The acid-soluble sulfide concentration, c_{S^-}, expressed in milligrams per kilogram, in the barite sample can be calculated as given in Equation (30).

$$c_{\text{S}^-} = \frac{l_{\text{ST}} \text{ x } f_{\text{S}}}{m} \tag{30}$$

where

c_{S^-} is the acid-soluble sulfide concentration, expressed in mg/kg;

l_{ST} is the Dräger tube’s maximum darkened length, expressed in units marked on the tube;

m is the mass of the barite sample, expressed in g;

f_{S} tube factor from Table 2.

21 Calcium Hydroxide (Lime) or Cement

21.1 Principle

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.

21.2 Reagents and Materials

21.2.1 **Sucrose** (CAS #57-50-1), crystalline, any granular table sugar will suffice.

21.2.2 Hydrochloric acid (CAS #7647-01-0) **solution** (0.1N).

21.2.3 Phenolphthalein indicator (CAS #77-09-8), 1 g phenolphthalein/100 ml of 50:50 alcohol:water solution.

21.2.4 Deionized or distilled water.

21.3 Apparatus

21.3.1 Sieve, 149 µm.

21.3.2 Balance, with accuracy of 0.001 g.

21.3.3 Magnetic stirrer.

21.3.4 Stirring bar.

21.3.5 Erlenmeyer flasks, several 250 ml.

21.3.6 Burrette, one 25 ml.

21.3.7 Graduated cylinder (TC), one 100 ml.

21.4 Procedure

21.4.1 Use a representative sample ground such that 100 % passes through the sieve.

21.4.2 Accurately weigh a 1.0 g sample into the 250-ml Erlenmeyer flask.

21.4.3 Add 65 ml to 75 ml deionized water and the magnetic stirring bar.

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

21.4.5 Turn off the stirrer and let the solution stand for 2 min.

21.4.6 Add 2 drops to 3 drops phenolphthalein indicator solution and titrate with 0.1N HCl to the phenolphthalein endpoint. Record the volume of 0.1N HCl required, as V_{HCl}.

21.5 Calculation

24.5.1 If the contamination is thought to be only lime, use Equation (29).

$$c_{\mathrm{CaOH_2},\%} = \frac{0.370 \times V_{\mathrm{HCl}}}{m} \quad (31)$$

where

$c_{\mathrm{CaOH_2},\%}$ is the concentration of calcium hydroxide (lime), expressed as a %;

V_{HCl} is the volume of 0.1N HCl, expressed in ml;

m is the mass of the barite sample, expressed in g.

21.5.2 If the contamination is thought to be cement, the value is multiplied by two, as most oilfield cements are about 50 % lime.

$$c_{\text{CEMENT},\%} \approx 2 \text{ x } c_{\text{CaOH}_2,\%} \tag{32}$$

where

$c_{\text{CEMENT},\%}$ is the concentration of cement, expressed as a %;

$c_{\text{CaOH}_2,\%}$ is the concentration of CaOH (lime), expressed as a %.

22 X-ray Fluorescence Analysis

22.1 Principle

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

22.1.2 These element spectra are then separated electronically or optically into individual X-ray lines, each line being characteristic of a given element.

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

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

22.1.5 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.

22.1.6 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 (e.g. particle size, surface smoothness, and element concentration) of the sample has a significant effect on the results (response) of the analysis.

22.1.7 Two sample preparation procedures are used.

22.1.7.1 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 resolved.

22.1.7.2 In the pressed powder method, 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 standard samples analyzed by wet chemical techniques, and consisting of elements in matrix material similar to the sample.

22.2 Reagents and Materials

22.2.1 Lithium tetraborate (CAS #12007-60-2), anhydrous, “spectral” grade powder.

22.2.2 Boric acid (CAS #10043-35-3), “spectral” grade powder.

22.2.3 Germanium oxide (CAS #20619-16-3), “spectral” grade powder.

22.2.4 Lithium carbonate (CAS #584-13-2), anhydrous, “spectral” grade powder.

22.2.5 Lithium nitrate (CAS #7790-69-4), anhydrous, “spectral” grade powder.

22.2.6 Lanthanum oxide (CAS #1312-81-8), “spectral” grade powder.

22.2.7 Hydrobromic acid (CAS #10035-10-6) solution (10 %), 10 g HBr/90 ml water.

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

22.2.9 Flux mixture B, 90 % lithium tetraborate, 10 % lithium nitrate.

22.2.10 Synthetic standards, several made from “pure” element compounds, such as $BaSO_4$, $SrSO_4$, SiO_2, Fe_2O_3, and others, to cover all elements being analyzed. The range of concentrations should cover the anticipated ranges in the unknowns.

22.2.11 Barite ore standards, several, all analyzed by conventional wet chemical techniques. Again, the concentration ranges of $BaSO_4$, $SrSO_4$, SiO_2, Fe_2O_3, and others, should cover anticipated levels of the unknowns.

22.3 Apparatus

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

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

22.3.3 Muffle furnace, regulated to 1100 °C ± 20 °C (2010 °F ± 72 °F).

22.3.4 Drying oven, regulated to 110 °C ± 5 °C (230 °F ± 10 °F).

22.3.5 Meker-type burner, two or more, capable of producing heating up to 1200 °C (2200 °F).

22.3.6 95 % platinum–5 % gold crucibles and covers, several 20 ml to 25 ml.

NOTE Graphite crucibles may also be used as an alternative.

22.3.7 95 % platinum–5 % gold molds, several sized to fit XRF sample holder.

NOTE Graphite molds may also be used as an alternative.

22.3.8 Hydraulic pellet press, capable of pressures of at least 276,000 kPa (40,000 lb/in.^{2}).

22.3.9 Die for pellet press, sized to fit XRF sample holder.

22.3.10 Impact grinder and grinding vials.

22.3.11 Timer.

22.3.12 Hot plate.

22.3.13 Balance, with accuracy of 0.001 g.

22.3.14 Sieve, 45 µm.

22.3.15 Crucible tongs.

22.3.16 PTFE stirring rod.

22.3.17 Sample cups, plastic, sized to fit XRF sample holder.

22.3.18 Mylar® [10] or plastic film to seal sample cups.

22.4 Procedure—Sample Preparation

22.4.1 Dry a representative sample of barite for at least 2 h at 110 °C (230 °F).

22.4.2 Grind the dried sample in the impact grinder such that 100 % passes through the sieve. This is the dried sample.

22.4.3 Place part of the ground sample in a platinum-gold or graphite crucible and ash in the muffle furnace for 30 min at 1000 °C (1832 °F).

Caution—Use proper safety precautions when handling hot crucible and melt.

22.4.4 Grind the ashed sample in the impact grinder to 100 % passes through the sieve. This is the ashed sample.

22.4.5 One or more of the following methods of sample preparation (22.4.5.1 through 22.4.5.4) may be used.

22.4.5.1 Procedure—Pressed Powder Method

a) Approximately 20 g of the ground (but not ashed) sample is placed in a standard die and pressed to 276,000 kPa (40,000 lb/in.^{2}) 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 (e.g. Mylar or equivalent) film to hold the sample and to act as a "window" to incident X-ray beam. The accuracy and precision of this method, however, are not as high as for the pressed pellet procedure.

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

c) If the pellet does not hold together after it is made, a small amount (e.g. 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.

22.4.5.2 Procedure—Fused Glass Disc Technique: Flux A

a) Accurately weigh on the analytical balance 0.500 g ground, ashed barite sample with 0.050 g germanium oxide and 2.0 g flux mixture A into a platinum-gold crucible.

b) Mix with PTF rod.

c) Fuse in the muffle furnace for 15 min at 980 °C (1800 °F). Swirl melt, using the crucible tongs, to ensure mixing of products after 5 min.

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[10] Mylar® is an example of suitable plastic brand available commercially. This information is given for the convenience of users of this part of API 13K and does not constitute an endorsement by API of these products.

Caution—Use proper safety precautions when handling hot crucible and melt.

d) Pour the melt into the platinum-gold mold that is kept at about 220 °C (425 °F) on the hot plate.

e) After the melt cools for 10 min, remove it with the tongs to a piece of unglazed tile.

f) 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.

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

22.4.5.3 Procedure—Fused Glass Disc Technique: Flux B

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

b) Mix with PTFE rod.

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

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

e) Increase the flame and fuse the samples for 8 min at high temperature, which is above 1000 °C (1832 °F).

f) 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.

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

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

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

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

22.4.5.4 Procedure—Fusion/Pressed Disc Technique

a) Accurately weigh on the analytical balance 0.500 g barite sample and 1.00 g lithium tetraborate into a platinum-gold crucible.

b) Mix with PTFE rod.

c) Place into muffle furnace and fuse for 15 min at 1050 °C to 1100 °C (1922 °F to 2012 °F) with occasional swirling of the melt to ensure thorough mixing.

Caution—Use proper safety precautions when handling hot crucible and melt.

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

e) Grind the mixture for 10 min in the impact grinder.

f) Press in the hydraulic press at 276,000 kPa (40,000 lb/in.^{2}) using boric acid as backing to the disc.

g) Repeat with several synthetic and/or analyzed standards.

22.5 Procedure—Spectral Analysis

22.5.1 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, aluminium, calcium, sulphur, strontium, and magnesium. Other “minor” elements such as lead, zinc, manganese, potassium, etc., can also be analyzed, but are usually present at low or trace concentrations.

22.5.2 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.

22.5.3 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.

22.6 Calculation

22.6.1 Fused glass discs—there is little or no inter-element 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.

22.6.2 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.

22.6.3 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 LOI, the following equation is used:

$$c_{\text{ELEMENT1,\%}} = \frac{c_{\text{ELEMENT2,\%}} \ \text{x} \ (100 - \%\text{LOI})}{100} \qquad (33)$$

where

$c_{\text{ELEMENT1,\%}}$ is the concentration of element, as received basis, expressed as a %;

$c_{\text{ELEMENT2,\%}}$ is the concentration of element, ashed basis, expressed as a %.

22.6.4 Pressed powder method—in this method 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.

23 Heavy Metals in Barite

23.1 Mercury in drilling fluids barite—see API 13I-2009 or ISO 10416:2008, Section 8.

23.2 Cadmium and lead in drilling fluids barite—see API 13I-2009 or ISO 10416:2008, Section 9.

23.3 Arsenic in drilling fluids barite—see API 13I-2009 or ISO 10416:2008, Section 10.

Annex A
(informative)

Rational Analysis

A.1 Principle

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” analyses 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:

$$c_{\mathrm{BaSO_4},\%} = 1.522 \text{ x } c_{\mathrm{BaO},\%} \tag{A.1}$$

$$c_{\mathrm{SrSO_4},\%} = 1.773 \text{ x } c_{\mathrm{SrO},\%} \tag{A.2}$$

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:

$$c_{\mathrm{FeS_2},\%} = 2.148 \text{ x } c_{\mathrm{Fe_{HNO_3}},\%} - c_{\mathrm{Fe_{HCl}},\%} \tag{A.3}$$

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

$$c_{\mathrm{Fe_2O_3},\%} = 0.699 \text{ x } \left(c_{\mathrm{Fe_{HCl}},\%} - 0.482 \text{ x } c_{\mathrm{SIDERITE},\%}\right) \tag{A.4}$$

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

$$c_{\mathrm{ALUMINOSILICATES},\%} = 2.7 \text{ x } c_{\mathrm{Al_2O_3},\%} \tag{A.5}$$

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

$$c_{\mathrm{QUARTZ},\%} = c_{\mathrm{TOTAL\ SiO_2},\%} - \left(0.47 \text{ x } c_{\mathrm{ALUMINOSILICATES},\%}\right) \tag{A.6}$$

A.2.6 Calcium sulfate (e.g. gypsum) is obtained from the water-soluble Ca:

$$c_{\mathrm{GYPSUM},\%} = 4.295 \text{ x } c_{\mathrm{WATER\text{-}SOLUBLE\ Ca},\%} \tag{A.7}$$

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):

$$c_{\mathrm{CaCO_3\,\%}} = 2.497 \text{ x } \left(c_{\text{HCl-SOLUBLE CA, \%}} - c_{\text{WATER-SOLUBLE Ca, \%}}\right) \tag{A.8}$$

where

$c_{\mathrm{CaCO_3\,\%}}$ is the concentration of $CaCO_3$, expressed as a %;

$c_{\text{HCl-SOLUBLE CA, \%}}$ is the concentration of HCl-soluble Ca, expressed as a %, see Section 8;

$c_{\text{WATER-SOLUBLE Ca, \%}}$ is the concentration of water-soluble Ca, expressed as a %, see Section 11.

A.2.8 LOI 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.

Annex B
(informative)

Metric “SI” Unit Conversion Table

Property	U.S Customary Unit	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	revolutions/minute (r/min)	revolutions/minute	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/second	meter/second	m/s	0.3048	400 ft/s = 122 m/s
Drilling rate	feet/hour	meter/hour	m/h	0.3048	30 ft/h = 9 m/h
Volume	barrels (42 gallons)	cubic meters	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	barrels/minute	cubic meters/minute	m^3/min	0.159	8.5 bbl/min = 1.35 m^3/min
	gallons/minute	cubic meters/minute	m^3/min	0.00378	357 gpm = 1.35 m^3/min
Pump pressure	pounds/inch^2	kilopascal	kPa	6.9	2500 psi = 17,300 kPa
Annular velocity	feet/minute	meter/minute	m/m	0.3048	200 ft/min = 61 m/min
Slip velocity	feet/minute	meter/minute	m/m	0.3048	200 ft/min = 61 m/min
Temperature	degree Fahrenheit	degree Celsius	°C	(°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 density (weight)	pounds/gallon	kilogram/cubic meter	kg/m^3	120	10 lb/gal = 1200 kg/m^3
	pounds/foot^3	kilogram/cubic meter	kg/m^3	16	74.7 lb/ft^3 = 1200 kg/m^3
Hydrostatic head	pounds/inch^2	kilopascal	kPa	6.9	4000 psi = 27,600 kPa
Pressure gradient	psi/foot	kilopascal/meter	kPa/m	22.6	0.52 psi/ft = 11.8 kPa/m^3
Shear stress	pound-force/100 feet^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}		
Apparent, plastic and effective viscosity	centipoise	centipoise			
Yield point	pound-force/100 feet^2	pascal	Pa	0.48	15 lb/100 ft^2 = 7.2 Pa
Gel strength	pound-force/100 feet^2	pascal	Pa	0.48	3 lb/100 ft^2 = 1.44 Pa
Dial reading, standard V-G meter	pound-force/100 feet^2/1.065	pascal	Pa	0.51	dial reading: 10 = 5.1 Pa

Power law constants:					
N	dimensionless				
K	dynes second/100 $feet^2$	millpascal seconds/ square centimeter	mPa•s/cm^2	100	10 dynes•s/cm^2 = 100 mPa•s/cm^2
	pound-force/100 $feet^2$	millpascal seconds/ square centimeter	mPa•s/cm^2	479	1.2 lb•s/100 ft^2 = 575 mPa•s/cm^2
API Filtrate	milliliters/30 minutes	milliliters/30 minute	ml/30 min		
Filter cake	$^1/_{32}$ inch	millimeter	mm	0.8	$^3/_{32}$ in. = 2.4 mm
Sand content	volume percent	$meter^3/meter^3$	$m^3 = m^3$	0.01	10 % = 0.1 m^3/m^3
Solids content	volume percent	$meter^3/meter^3$	$m^3 = m^3$	0.01	10 % = 0.1 m^3/m^3
Oil content	volume percent	$meter^3/meter^3$	$m^3 = m^3$	0.01	10 % = 0.1 m^3/m^3
Water content	volume percent	$meter^3/meter^3$	$m^3 = m^3$	0.01	10 % = 0.1 m^3/m^3
Particle size	micron	micrometer	µm	1.0	
Chemical properties:				**Multiply by:**	
Ionic concentration	grains/gallon	milligrams/liter	mg/l	17.1 × specific gravity	500 grains/gal = 8600 mg/l
	parts/million	milligrams/liter	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		
Methylene blue content: MBT	cubic centimeter/ cubic centimeter	cubic centimeter/cubic centimeter	cm^3/cm^3		
Miscellaneous:				**Multiply by:**	
Additive concentration	pounds/barrel	kilogram/cubic meter	kg/m^3	2.85	
Corrosion rate	$lb/ft^2/yr$	kilogram/square meter/year	$kg/m^2/yr$	4.9	
	mils/year	millimeter/year	mm/a	0.0254	
Bentonite yield	barrels/U.S. ton	cubic meter/metric tonne	m^3/t	0.175	
Hydraulic HP	horsepower	kilowatt	kW	0.746	
Screen size					
Mesh	openings/inch	opening/centimeter	openings/cm	0.254	
Aperture	micron	micrometer	µm	1	
Open area	area percent	square meter/square meter	m^2/m^2	0.01	
Weight of drill pipe	pound/foot	kilogram/meter	kg/m	1.49	
Oil drilling fluids:				**Multiply by:**	
Oil content	volume percent	cubic meter/cubic meter	m^3/m^3	0.01	10 % = 0.1 m^3/m^3
Water content	volume percent	cubic meter/cubic meter	m^3/m^3	0.01	
Emulsion stability	volts	volts	V		
Activity	dimensionless				
Salinity	ppm	milligrams/liter	mg/l	specific gravity	250,000 ppm of $CaCl_2$ × 1.24 = 310,000 mg/l
Aniline point	degree Fahrenheit	degree Celsius	°C	(°F − 32)/1.8	150 °F = 66 °C

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