

Chemical Analysis of Barite

API RECOMMENDED PRACTICE 13K
FOURTH EDITION, MARCH 2022



Special Notes

API publications necessarily address problems of a general nature. With respect to particular circumstances, local, state, and federal laws and regulations should be reviewed. The use of API publications is voluntary. In some cases, third parties or authorities having jurisdiction may choose to incorporate API standards by reference and may mandate compliance.

Neither API nor any of API's employees, subcontractors, consultants, committees, or other assignees make any warranty or representation, either express or implied, with respect to the accuracy, completeness, or usefulness of the information contained herein, or assume any liability or responsibility for any use, or the results of such use, of any information or process disclosed in this publication. Neither API nor any of API's employees, subcontractors, consultants, or other assignees represent that use of this publication would not infringe upon privately owned rights.

API publications may be used by anyone desiring to do so. Every effort has been made by the Institute to ensure the accuracy and reliability of the data contained in them; however, the Institute makes no representation, warranty, or guarantee in connection with this publication and hereby expressly disclaims any liability or responsibility for loss or damage resulting from its use or for the violation of any authorities having jurisdiction with which this publication may conflict.

API publications are published to facilitate the broad availability of proven, sound engineering and operating practices. These publications are not intended to obviate the need for applying sound engineering judgment regarding when and where these publications should be used. The formulation and publication of API publications is not intended in any way to inhibit anyone from using any other practices.

Any manufacturer marking equipment or materials in conformance with the marking requirements of an API standard is solely responsible for complying with all the applicable requirements of that standard. API does not represent, warrant, or guarantee that such products do in fact conform to the applicable API standard.

All rights reserved. No part of this work may be reproduced, translated, stored in a retrieval system, or transmitted by any means, electronic, mechanical, photocopying, recording, or otherwise, without prior written permission from the publisher. Contact the Publisher, API Publishing Services, 200 Massachusetts Avenue, NW, Suite 1100, Washington, DC 20001-5571.

Foreword

Nothing contained in any API publication is to be construed as granting any right, by implication or otherwise, for the manufacture, sale, or use of any method, apparatus, or product covered by letters patent. Neither should anything contained in the publication be construed as insuring anyone against liability for infringement of letters patent.

The verbal forms used to express the provisions in this document are as follows.

Shall: As used in a standard, “shall” denotes a minimum requirement to conform to the standard.

Should: As used in a standard, “should” denotes a recommendation or that which is advised but not required to conform to the standard.

May: As used in a standard, “may” denotes a course of action permissible within the limits of a standard.

Can: As used in a standard, “can” denotes a statement of possibility or capability.

This document was produced under API standardization procedures that ensure appropriate notification and participation in the developmental process and is designated as an API standard. Questions concerning the interpretation of the content of this publication or comments and questions concerning the procedures under which this publication was developed should be directed in writing to the Director of Standards, American Petroleum Institute, 200 Massachusetts Avenue, NW, Suite 1100, Washington, DC 20001. Requests for permission to reproduce or translate all or any part of the material published herein should also be addressed to the director.

Generally, API standards are reviewed and revised, reaffirmed, or withdrawn at least every five years. A one-time extension of up to two years may be added to this review cycle. Status of the publication can be ascertained from the API Standards Department, telephone (202) 682-8000. A catalog of API publications and materials is published annually by API, 200 Massachusetts Avenue, NW, Suite 1100, Washington, DC 20001.

Suggested revisions are invited and should be submitted to the Standards Department, API, 200 Massachusetts Avenue, NW, Suite 1100, Washington, DC 20001, standards@api.org.

Important Information Concerning Use of Asbestos or Alternative Materials

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

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

Consult the most recent edition of the Occupational Safety and Health Administration (OSHA), U.S. Department of Labor, Occupational Safety and Health Standards for Asbestos, Tremolite, Anthophyllite, and Actinolite, 29 *Code of Federal Regulations*, Section 1910.1001; the U.S. Environmental Protection Agency, National Emission Standard for Asbestos, 40 *Code of Federal Regulations*, Section 61.140 through 61.156; and the U.S. Environmental Protection Agency (EPA) rule on labeling requirements and phased banning of asbestos products (Sections 763.160-170).

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

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

Contents

	Page
1 Scope.....	1
2 Normative References	2
3 Terms, Definitions, Acronyms, Abbreviations, and Symbols	2
3.1 Terms and Definitions	2
3.2 Acronyms and Abbreviations	2
3.3 Symbols	3
4 Wet Analysis Methods.....	6
4.1 Principle	6
4.2 Advantages of Wet Chemical Analysis Methods.....	6
4.3 Summary of General Analytical Methods.....	6
5 Barium Sulfate and Strontium Sulfate.....	7
5.1 Principle	7
5.2 Reagents and Materials	8
5.3 Apparatus	8
5.4 Sampling.....	9
5.5 Procedure—Barium Sulfate Determination.....	9
5.6 Procedure—Strontium Sulfate Determination.....	11
5.7 Calculation—Barium Sulfate and Strontium Sulfate Mass Fractions	11
6 Silica and Alumina	12
6.1 Principle	12
6.2 Reagents and Materials	12
6.3 Apparatus	13
6.4 Procedure—Sample Preparation	14
6.5 Procedure—Silica Determination.....	15
6.6 Procedure—Alumina Determination	15
6.7 Calculation—Silica and Alumina Mass Fractions.....	16
7 Hydrochloric Acid Soluble Metals—Sodium, Potassium, Calcium, Magnesium, Iron, Copper, Manganese, Lead, and Zinc	17
7.1 Principle	17
7.2 Reagents and Materials	17
7.3 Apparatus	17
7.4 Procedure	18
7.5 Calculation	18
8 Procedure—Hydrofluoric, Sulfuric, Nitric, Perchloric Acid Soluble Metals—Sodium, Potassium, Calcium, Magnesium, Iron, Copper, Manganese, Lead, and Zinc.....	18
8.1 Principle	18
8.2 Reagents and Materials	19
8.3 Apparatus	19
8.4 Procedure	20
8.5 Calculation	21
9 Alternative Methods for Iron.....	21
9.1 Principle	21
9.2 Reagents and Materials.....	21

Contents

	Page
9.3	Apparatus 22
9.4	Nitric Acid Digestion—Iron 23
9.5	Aqua Regia Digestion—Iron 23
9.6	Procedure—Carbonate Fusion 24
10	Water-soluble Materials in Barite 25
10.1	Principle 25
10.2	Apparatus and Materials 25
10.3	Procedure—Water-soluble Materials in Barite 26
10.4	Water-soluble Metals in Barite 27
10.5	Calculation—Water-soluble Materials in Barite 27
11	Water-soluble Chlorides 27
11.1	Principle 27
11.2	Reagents and Materials 27
11.3	Apparatus 28
11.4	Procedure—Water-soluble Chlorides by Titration 28
11.5	Procedure—Water-soluble Chlorides by Ion Chromatography 28
11.6	Calculation—Water-soluble Chlorides 29
12	Water-soluble Sulfates 29
12.1	Principle 29
12.2	Reagents and Materials 29
12.3	Apparatus 30
12.4	Procedure—Preparation of Calibration Chart for Turbidity Method 30
12.5	Procedure—Analysis for Water-soluble Sulfates by Turbidity Method 31
12.6	Procedure—Analysis for Water-soluble Sulfate by Ion Chromatography 31
12.7	Calculation—Water-soluble Sulfates 31
13	Water-soluble Carbonates, Bicarbonates, and Hydroxyl Ions 32
13.1	Principle 32
13.2	Reagents and Materials 32
13.3	Apparatus 32
13.4	Procedure 32
13.5	Calculation 33
14	Water-soluble Phosphates 34
14.1	Principle 34
14.2	Reagents and Materials 34
14.3	Apparatus 35
14.4	Procedure—Preparation of Calibration Curve 35
14.5	Procedure—Analysis of Sample 36
14.6	Calculation 36
15	Loss on Ignition (<i>LOI</i>) 36
15.1	Principle 36
15.2	Reagents, Materials, and Apparatus 37
15.3	Procedure 37
15.4	Calculation 37
16	Siderite Content 38

Contents

	Page
16.1 Principle	38
16.2 Reagents and Materials	38
16.3 Apparatus	38
16.4 Procedure—EDTA Extraction.....	39
16.5 Procedure—Iron Content by AA or ICP Spectrometry	39
16.6 Procedure—Iron Content by Colorimetric Determination	39
16.7 Calculation—Siderite by EDTA Extraction and AA/ICP.....	40
17 Zinc Carbonate and Lead Carbonate	40
17.1 Principle	40
17.2 Reagents, Materials, and Apparatus.....	41
17.3 Procedure—Acetic Acid Extraction	41
17.4 Procedure—Lead and Zinc Determination by AA or ICP Spectrometry.....	41
17.5 Calculations	42
18 Total Carbonate	42
18.1 Principle	42
18.2 Reagents and Materials.....	42
18.3 Apparatus	42
18.4 Procedure	44
18.5 Calculation	45
19 Acid-soluble Sulfides	45
19.1 Principle	45
19.2 Reagents and Materials.....	45
19.3 Apparatus	46
19.4 Procedure	46
19.5 Calculation	47
20 Calcium Hydroxide (Lime) or Cement.....	48
20.1 Principle	48
20.2 Reagents and Materials.....	48
20.3 Apparatus	48
20.4 Procedure	48
20.5 Calculation	49
21 X-ray Fluorescence Analysis	49
21.1 Principle	49
21.2 Reagents and Materials.....	50
21.3 Apparatus	50
21.4 XRF Analysis—Procedures	51
21.5 Procedure—Spectral Analysis	53
21.6 Calculation	54
22 Mercury in Drilling Fluid Barite.....	54
22.1 Principle	54
22.2 Reagents and Materials.....	54
22.3 Apparatus	56
22.4 Preparation of Standards.....	56
22.5 Sample Digestion.....	57
22.6 Recovery of Hg during Digestion—Check	57

Contents

	Page
22.7 Standards and Samples—Analysis	57
22.8 Calculation	58
23 Cadmium and Lead in Drilling Fluid Barite	59
23.1 Principle	59
23.2 Reagents and Apparatus	59
23.3 Preparation of Combined Cadmium and Lead Standards	60
23.4 Sample Acid Digestion	60
23.5 Analysis of Standards and Samples	61
23.6 Calculation	61
24 Arsenic in Barite.....	62
24.1 Principle	62
24.2 Reagents and Apparatus	62
24.3 Preparation of As Standards.....	64
24.4 Sample Digestion.....	64
24.5 Analysis of Standards and Samples	64
24.6 Calculation	65
Annex A (informative) Rational Analysis	66
Bibliography.....	69

Figures

1 Typical Correction Curve for SrSO in BaSO	12
---------------------------------------------------	----

Tables

1 Minerals Associated with Barite Ore Bodies	1
2 H ₂ S Dräger Tube Identification, Sample Volume, and Tube Factor to be Used for Various Sulfide Ranges	47

Chemical Analysis of Barite

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 performed in a well-equipped laboratory.

Table 1—Minerals Associated with Barite Ore Bodies

Mineral	Chemical Formula	Mineralogical Name	Relative Density	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 ₂ O ₅	Sanbornite	3.74	5
Calcium carbonate	CaCO ₃	Calcite	2.72	3
Calcium magnesium carbonate	CaMg(CO ₃) ₂	Dolomite	2.87	3.5–4
Magnesium carbonate	MgCO ₃	Magnesite	3.0	4
Calcium sulfate	CaSO ₄ •2H ₂ O	Gypsum	2.3	2
Calcium fluoride	CaF ₂	Fluorite	3.0	4
Iron (ferric) oxide	Fe ₂ O ₃	Hematite	5.2	5.5
Iron oxyhydroxide	Fe ₂ O ₃ •nH ₂ O	Goethite	3.3–4.0	5–5.5
Iron (II, III) oxide	Fe ₃ O	Magnetite	5.16–5.17	5.5–6.5
Iron carbonate	FeCO ₃	Siderite	3.7–3.9	3.5–4
Iron sulfide	FeS ₂	Pyrite	5.0	6–6.5
Iron sulfide	Fe _{1-x} S _(x=0 to 0.2)	Pyrrhotite	4.6	4
Iron/copper sulfide	CuFeS ₂	Chalcopyrite	4.2	4
Lead sulfide	PbS	Galena	7.5	2.5
Zinc sulfide	ZnS	Sphalerite	3.9	3.5–4
Ferrous titanate	FeTiO ₃	Ilmenite	4.7	5.5
Silicon dioxide	SiO ₂	Quartz, sand, chert	2.65	7
Aluminosilicates:				
Feldspar	(Na,K)AlSi ₃ O ₈	Albite, microcline	2.6	6
Mica	KAl ₂ (OH) ₂ AlSi ₃ O ₁₀	Muscovite	2.85	2.5
Clays	Al ₂ Si ₂ O ₅ (OH) ₄	Kaolinite	2.6	2
	(Al,Mg)(OH) ₂ Si ₄ O ₁₀ (Na,Ca) _x •4H ₂ O	Montmorillonite	2–3	1–2