

Testing of Heavy Brines

API RECOMMENDED PRACTICE 13J
SIXTH EDITION, JANUARY 2023



American
Petroleum
Institute

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.

Users of this standard should not rely exclusively on the information contained in this document. Sound business, scientific, engineering, and safety judgment should be used in employing the information contained herein.

API is not undertaking to meet the duties of employers, manufacturers, or suppliers to warn and properly train and equip their employees, and others exposed, concerning health and safety risks and precautions, nor undertaking their obligations to comply with authorities having jurisdiction.

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 utilized. 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.

Copyright © 2023 American Petroleum Institute

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 in order to conform to the standard.

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

Contents

	Page
1 Scope.....	1
2 Normative References	1
3 Terms, Definitions, Acronyms, Abbreviations, and Symbols	1
3.1 Terms and Definitions.....	1
3.2 Acronyms, Abbreviations, and Symbols	3
4 Requirements	7
4.1 Quality Control Instructions.....	7
4.2 Records Retention.....	7
5 Calibration of Equipment	7
5.1 General Requirements	7
5.2 Reagents and Materials for Calibration and Verification	7
5.3 General Requirements for Calibration of Particular Apparatus	8
5.4 Frequency of Calibration/Verification	8
5.5 Calibration/Verification—Temperature-measuring Device	9
5.6 Calibration/Verification—Density-measuring Device	9
6 Density	11
6.1 General	11
6.2 Apparatus	12
6.3 Measurement of Relative Density or Density Using a Hydrometer	14
6.4 Density Corrections for Hydrometers and Brines	15
6.5 Calculation of Density in Units Other than g/mL or kg/m ³	22
6.6 Calculations for Pressure and Temperature Compensation	24
7 Crystallization Temperature.....	28
7.1 Principle	28
7.2 Apparatus	33
7.3 Determination of Crystallization Temperature of Halide Brines.....	35
8 Brine Clarity	35
8.1 Principle	35
8.2 Apparatus	36
8.3 Determination of Brine Clarity	36
9 Solids Evaluation by Gravimetric Procedure	37
9.1 Principle	37
9.2 Apparatus	37
9.3 Preparation of Glass-fiber Filter Disk.....	38
9.4 Selection of Sample Volume	39
9.5 Determination of Total Suspended Solids	39
9.6 Calculation of Total Suspended Solids.....	40
9.7 Calculation of Precision and Bias	40
9.8 Determination of Volatile and Non-volatile Solids	41
9.9 Calculation of Non-volatile and Volatile Solids	41

Contents

	Page
10 pH.....	41
10.1 Principle	41
10.2 Reagents and Materials	42
10.3 Apparatus	43
10.4 Measurement of pH of Halide Brines.....	44
10.5 Precision Statement	45
10.6 Care of the Electrode	45
11 Iron Contamination	45
11.1 Principle	45
11.2 Reagents and Materials	46
11.3 Apparatus	46
11.4 Preparation of Colorimetric Standards	47
11.5 Measurement of Iron Contamination.....	47
11.6 Precision Statement	48
12 Buffering Capacity of Brines.....	48
12.1 Principle	48
12.2 Equipment.....	49
12.3 Reagents and Materials	50
12.4 Procedure—Determination of Buffer Concentrations of Halide Brines.....	51
12.5 Calculation—First pH Endpoint	53
12.6 Calculation—Second pH Endpoint	55
13 Daily Completion Fluid Report.....	56
13.1 Principle	56
13.2 Daily Report Header	57
13.3 Wellbore Geometry, Pumps, and Zone Data	57
13.4 Completion Fluid Properties	58
13.5 Fluids and Products Accounting.....	59
13.6 Usage and/or Cost Accounting.....	59
13.7 Daily Operational Commentary	59
13.8 Vendor Representative Identification.....	59
13.9 Environmental and Safety Information	60
Annex A (informative) Example of Daily Completion Fluid Report	61
Annex B (informative) Gas Hydrates	62
Annex C (informative) Pressure Crystallization of Brines	64
Annex D (informative) Brine Viscosity.....	66
Annex E (informative) Principle of Corrosion Testing	67
Bibliography	80
Figures	
1 Hydrometer Reading at Bottom of Meniscus.....	15
2 Calcium Chloride Crystallization Temperature.....	29
3 Typical Crystallization Curve for Brine.....	33

Contents

	Page
4 Typical Crystallization Temperature Measurement Setup.....	34
5 Example of pH vs Volume of HCl Acid Added	52
6 Example of $\Delta\text{pH}/\Delta\text{VHCl}$ vs pH for More Accurate Determination of Titration Endpoint	53

Tables

1 Temperature Correction Factors for Hydrometers due to Glass Expansion and Contraction	17
2 Estimated Brine Thermal Expansion/Contraction Factors (Conversion Factors)	21
3 Pressure Compensation Factor in SI Units.....	25
4 Temperature Compensation Factor in SI Units	25
5 Pressure Compensation Factors in USC Units	27
6 Temperature Compensation Factors in USC Units	27
7 Calcium Chloride Solutions—Crystallization Temperature	30
8 Sodium Chloride Solutions—Crystallization Temperature	31
E.1 Mechanical Properties of Selected Oil Country Tubular Goods	70

Introduction

This standard is based on API Specification 13J, 5th Edition and covers the testing of heavy brines commonly used in petroleum and natural gas completion, fracturing, workover, and drill-in fluids. These brines can be purchased or rented from multiple sources and are available worldwide. No single source or limited source of supply is included, either by inference or reference.

Annexes A through E are given for information.

In this standard, quantities are expressed in the International System (SI) of units and are also, where practical, expressed in U.S. Customary (USC) units in parentheses.

NOTE The units do not necessarily represent a direct conversion of SI units to USC units, or of USC units to SI units.

Consideration has been given to the precision of the instrument making the measurement. For example, thermometers are typically marked in one-degree increments; therefore, temperature values have been rounded to the nearest degree. However, for certain critical measurements (such as brine crystallization), thermometers are typically calibrated and used to 0.05 °C (0.1 °F).

Calibrating an instrument refers to assuring the accuracy of the measurement. Accuracy is the degree of conformity of a measurement of a quantity to its actual or true value. Accuracy is related to precision, or reproducibility of a measurement. Precision is the degree to which further measurements or calculations will show the same or similar results. Precision is characterized in terms of the standard deviation of the measurement. The results of calculations or a measurement can be accurate but not precise; precise but not accurate; neither; or both. A result is valid if it is both accurate and precise.

This document uses a format for numbers that follows the examples given in the API Document Format and Style Manual, October 2020. In this document, the decimal mark is a period and separates the whole part from the fractional part of a number. No spaces are used in the numbering format. The thousands separator is a comma and is only used for numbers greater than 10,000 (i.e. 5000 items, 12,500 bags).

Testing of Heavy Brines

1 Scope

This standard covers the physical properties, potential contaminants, and test procedures for heavy brine fluids manufactured for use in oil and gas well drilling, completion, fracturing, and workover fluids.

This standard provides methods for assessing the performance and physical characteristics of heavy brines for use in field operations. It includes procedures for evaluating the density or specific gravity, the clarity or amount of particulate matter carried in the brines, the crystallization point or the temperature at which the brines make the transition between liquid and solid at atmospheric pressure (a discussion of crystallization temperature under pressure is provided Annex C), the pH, iron contamination, and buffering capacity. This standard also contains a discussion of gas hydrate formation and mitigation (Annex B), brine viscosity (Annex D), corrosion testing (Annex E), and a daily completion fluids report with example (Annex A).

NOTE Procedures for evaluating the crystallization point, pH, and buffer capacity of brines are not applicable to formate brines. Procedures applicable to formate brines are found in ISO 13503-3:2022 [4].

This standard is intended for the use of manufacturers, service companies, and end users of heavy brines.

2 Normative References

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

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

ASTM E77 ¹, *Standard Test Method for Inspection and Verification of Thermometers*

ASTM E617, *Standard Specification for Laboratory Weights and Precision Mass Standards*

NIST SRM 185h ², *Potassium Hydrogen Phthalate, pH Standard*

NIST SRM 186g, *Potassium Dihydrogen Phosphate, pH Standard*

NIST SRM 191C, *pH Standards*

NIST SRM 3126a, *Iron (Fe) Standard Solution*

3 Terms, Definitions, Acronyms, Abbreviations, and Symbols

3.1 Terms and Definitions

For the purposes of this standard, the following terms and definitions apply.

3.1.1

ACS reagent grade

Grade of chemical that meets purity standards as specified by the American Chemical Society (ACS).

¹ ASTM International, PO Box C700, 100 Barr Harbor Drive, West Conshohocken, PA 19428, www.astm.org.

² National Institute of Standards and Technology, 100 Bureau Drive, Gaithersburg, MD 20899, www.nist.gov.