

# Recommended Practice for Laboratory Testing of Drilling Fluids

ANSI/API RECOMMENDED PRACTICE 13I  
EIGHTH EDITION, MARCH 2009

REAFFIRMED, MARCH 2016

**ISO 10416:2008 (Identical), Petroleum and natural gas  
industries—Drilling fluids—Laboratory testing**



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.

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 recommended practice 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 publications may be used by anyone desiring to do so. Every effort has been made by the Institute to assure 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, 1220 L Street, N.W., Washington, D.C. 20005.

*Copyright © 2009 American Petroleum Institute*

## **API 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.

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

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

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, 1220 L Street, N.W., Washington, D.C. 20005. 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, 1220 L Street, N.W., Washington, D.C. 20005.

Suggested revisions are invited and should be submitted to the Standards Department, API, 1220 L Street, NW, Washington, D.C. 20005, [standards@api.org](mailto:standards@api.org).

# Contents

Page

API Foreword .....	ii
Foreword .....	vii
Introduction .....	viii
1 Scope .....	1
2 Normative references .....	1
3 Terms and definitions .....	2
4 Symbols and abbreviations .....	3
5 Barite .....	6
5.1 Principle .....	6
5.2 Reagents and apparatus .....	6
5.3 Sampling .....	7
5.4 Calculation of moisture content .....	7
5.5 Sieve analysis .....	7
5.6 Sedimentation analysis .....	8
6 Barite performance .....	12
6.1 Principle .....	12
6.2 Reagents and apparatus .....	12
6.3 Base drilling fluid preparation .....	13
6.4 Rheology test .....	13
6.5 Calculation .....	14
7 Abrasiveness of weighting materials .....	14
7.1 Principle .....	14
7.2 Reagents and apparatus .....	15
7.3 Determination of abrasion .....	15
8 Mercury in drilling fluid barite .....	17
8.1 Principle .....	17
8.2 Reagents and apparatus .....	17
8.3 Preparation of standards .....	19
8.4 Sample digestion .....	19
8.5 Check for recovery of Hg during digestion .....	20
8.6 Analysis of standards and samples .....	20
8.7 Calculation .....	20
9 Cadmium and lead in drilling fluid barite .....	21
9.1 Principle .....	21
9.2 Reagents and apparatus .....	21
9.3 Preparation of combined cadmium and lead standards .....	22
9.4 Sample digestion .....	22
9.5 Analysis of standards and samples .....	22
9.6 Calculation .....	23
10 Arsenic in drilling fluid barite .....	23
10.1 Principle .....	23
10.2 Reagents and apparatus .....	24
10.3 Preparation of standards .....	25
10.4 Sample digestion .....	25
10.5 Analysis of standards and samples .....	26
10.6 Calculation .....	26
11 Bridging materials for regaining circulation .....	26
11.1 Principle .....	26

11.2	Apparatus .....	27
11.3	Preparation of test drilling fluid .....	27
11.4	Static slot test .....	27
11.5	Dynamic slot test .....	28
11.6	Static marble bed test.....	28
11.7	Dynamic marble bed test .....	28
11.8	Static ball bearings (BB shot) bed test.....	29
11.9	Dynamic ball bearings (BB shot) bed test .....	29
12	Filtration-control agents.....	29
12.1	Principle .....	29
12.2	Reagents and apparatus .....	29
12.3	General instructions for preparation of base drilling fluids.....	31
12.4	Salt-saturated drilling fluid .....	31
12.5	High-hardness, salt-saturated drilling fluid .....	32
12.6	10 % potassium chloride (KCl) drilling fluid .....	32
12.7	Pre-hydrated bentonite slurry .....	33
12.8	Modified seawater drilling fluid .....	33
12.9	Low-salinity drilling fluid .....	33
12.10	Lime-treated drilling fluid.....	34
12.11	Low solids, non-dispersed drilling fluid.....	34
12.12	Freshwater lignosulfonate drilling fluid .....	35
12.13	Initial performance test .....	35
12.14	Performance after heat ageing .....	36
13	Methylene blue test for drilled solids and commercial bentonite .....	36
13.1	Methylene blue capacity of drill solids .....	36
13.2	Methylene blue capacity of commercial bentonite.....	39
13.3	Solids content .....	40
14	Deflocculation test for thinner evaluation.....	41
14.1	Principle .....	41
14.2	Reagents and apparatus .....	42
14.3	Procedure for moisture content .....	43
14.4	Calculation of moisture content.....	43
14.5	Preparation of drilling fluid base.....	43
14.6	Calculation.....	44
14.7	Determination of rheological properties .....	44
14.8	Calculation of thinner efficiency .....	46
15	Testing base oils used in drilling fluids .....	46
15.1	General.....	46
15.2	Reagents and apparatus .....	46
15.3	Density, relative density (specific gravity), or API gravity-hydrometer method (see ISO 3675).....	46
15.4	Density and relative density of liquids using a digital density meter (see ASTM D 4052) .....	47
15.5	Kinematic viscosity of transparent and opaque oils — Calibrated capillary tube method (see ISO 3104).....	47
15.6	Distillation (see ISO 3405) .....	47
15.7	Aniline point and mixed aniline point (see ISO 2977:1997) .....	48
15.8	Pour point (see ISO 3016) .....	48
15.9	Flash point by Pensky-Martens closed tester (see ISO 2719).....	49
15.10	Aromatics content (see IP 391 or ASTM D 5186).....	49
16	Potassium ion content — Ion-selective electrode method.....	50
16.1	Principle.....	50
16.2	Reagents and apparatus .....	50
16.3	Preparation of electrodes .....	51
16.4	Operational check of electrode system.....	51
16.5	Measurements using a meter with direct concentration readout capability .....	52
16.6	Measurements with instruments that provide either a digital or an analogue readout in millivolts .....	52
17	Calcium ion content — Ion-selective electrode method.....	53
17.1	Principle.....	53
17.2	Reagents and apparatus .....	53

17.3	Preparation of electrodes .....	54
17.4	Operational check of electrode system .....	55
17.5	Measurements using a meter with direct concentration readout capability .....	55
17.6	Measurements with instruments that provide either a digital or an analogue readout in millivolts .....	55
18	Sodium ion content — Ion-selective electrode method .....	56
18.1	Principle.....	56
18.2	Reagents and apparatus.....	57
18.3	Preparation and operational check of the electrode system .....	57
18.4	Measurements using a meter with a direct concentration-readout capability.....	58
18.5	Measurements using a meter with readout in millivolts .....	58
19	Density of solids — Stereopycnometer method .....	59
19.1	Principle.....	59
19.2	Apparatus .....	59
19.3	Procedure — Stereopycnometer method .....	59
19.4	Calculation — Stereopycnometer method.....	60
20	Density of solids — Air comparison pycnometer method .....	61
20.1	Principle.....	61
20.2	Apparatus .....	61
20.3	Procedure — Air comparison pycnometer method .....	61
20.4	Calculation — Air comparison pycnometer method.....	61
21	Ageing of water-based drilling fluids .....	62
21.1	Principle.....	62
21.2	Practices common to preparation, handling and testing over all temperature ranges .....	62
21.3	Drilling fluid sample preparation and ageing at ambient temperature .....	63
21.4	Drilling fluid ageing at moderate temperatures [ambient to 65 °C (150 °F)].....	64
21.5	Drilling fluid ageing at substantially elevated temperatures [over 65 °C (150 °F)].....	66
21.6	Inertness and chemical compatibility in high-temperature ageing cells.....	68
21.7	Obtaining supplies and services for the ageing of drilling fluid samples.....	69
22	Ageing of oil-based drilling fluids.....	69
22.1	Principle.....	69
22.2	Apparatus .....	70
22.3	Practices common to preparation, handling and testing over all temperature ranges .....	71
22.4	Drilling fluid ageing at ambient temperatures .....	72
22.5	Drilling fluid ageing at moderate temperatures [ambient to 65 °C (150 °F)].....	73
22.6	Drilling fluid ageing at substantially elevated temperatures [over 65 °C (150 °F)].....	74
22.7	Inertness and chemical compatibility in high-temperature ageing cells.....	75
22.8	Obtaining supplies and services for the ageing of drilling fluid samples.....	76
23	Shale-particle disintegration test by hot rolling .....	76
23.1	Principle.....	76
23.2	Reagents and apparatus .....	77
23.3	Procedure .....	77
23.4	Calculation .....	78
24	Drilling fluid materials — High-viscosity polyanionic cellulose (PAC-HV) (regular).....	79
24.1	Principle.....	79
24.2	Determination of moisture content.....	79
24.3	Procedures with test fluid containing PAC-HV .....	80
25	Drilling fluid materials — Low-viscosity polyanionic cellulose (PAC-LV).....	82
25.1	Principle.....	82
25.2	Determination of moisture content.....	83
25.3	Procedures with test fluid containing PAC-LV .....	83
26	Preparation and evaluation of invert-emulsion drilling fluids .....	86
26.1	Principle.....	86
26.2	Reagents and apparatus .....	86
26.3	Mixing of the initial drilling fluid .....	87
26.4	Testing the properties of the initial drilling fluid.....	88
26.5	Preparation of the sample contaminated by seawater .....	88

26.6	Preparation of the sample contaminated by base evaluation clay.....	89
26.7	Preparation of the sample contaminated by mixed-salt brine .....	89
26.8	Procedure for hot-rolling .....	89
26.9	Procedure for static ageing .....	89
26.10	Procedure for testing after heat ageing.....	90
27	High-temperature/high-pressure filtration testing of drilling fluids using the permeability plugging apparatus and cells with set-screw-secured end caps .....	90
27.1	Principle .....	90
27.2	Safety considerations.....	90
27.3	Apparatus — Permeability-plugging apparatus (PPA) with set-screw-secured end caps.....	92
27.4	Procedure for high-temperature/high-pressure (HTHP) filtration.....	94
27.5	Test conclusion and disassembly .....	97
27.6	Data reporting .....	99
28	High-temperature/high-pressure filtration testing of drilling fluids using the permeability-plugging apparatus and cells with threaded end caps .....	100
28.1	Principle .....	100
28.2	Safety considerations.....	100
28.3	Apparatus — Permeability-plugging apparatus (PPA) with threaded end caps .....	102
28.4	Procedure for high-temperature/high-pressure (HTHP) filtration.....	104
28.5	Test conclusion and disassembly .....	106
28.6	Data reporting .....	108
	Bibliography .....	110

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10416 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

This second edition cancels and replaces the first edition (ISO 10416:2002), which has been technically revised.

## Introduction

This International Standard, which establishes testing methodologies for drilling fluid materials, is based on API RP 13I, seventh edition/ISO 10416:2002 [2]. This International Standard was developed in response to a demand for more exacting testing methodologies. The tests contained herein were developed over several years by a group of industry experts and were identified as being those which can yield reproducible and accurate results. The tests are anticipated to be performed in a laboratory setting, but can be applicable in a field situation with more rigorous apparatus and conditions than normally found in a drilling fluid field-test kit.

These tests are designed to assist in the evaluation of certain parameters for drilling fluids, with these properties not necessarily used for the maintenance of a drilling fluid in field use. The tests provide either more precision or different properties than those given in the field-testing standards ISO 10414-1 and ISO 10414-2.

It is necessary that users of this International Standard be aware that further or differing requirements can be needed for individual applications. This International Standard is not intended to inhibit a vendor from offering, or the purchaser from accepting, alternative equipment or engineering solutions for the individual application. This may be particularly appropriate where there is innovative or developing technology. Where an alternative is offered, the vendor should identify any variations from this International Standard and provide details.

As with any laboratory procedure requiring the use of potentially hazardous chemicals, the user is expected to have received proper knowledge and training in the use and disposal of these chemicals. The user is responsible for compliance with all applicable local, regional, and national regulations for worker and local health, safety and environmental liability.

This International Standard contains footnotes giving examples of apparatus, reagents and sometimes the supplier(s) of those materials that are available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO/API of the products named. Equivalent products may be used if they can be shown to lead to the same results.

# Petroleum and natural gas industries — Drilling fluids — Laboratory testing

## 1 Scope

This International Standard provides procedures for the laboratory testing of both drilling fluid materials and drilling fluid physical, chemical and performance properties. It is applicable to both water-based and oil-based drilling fluids, as well as the base or “make-up” fluid.

It is not applicable as a detailed manual on drilling fluid control procedures. Recommendations regarding agitation and testing temperature are presented because the agitation history and temperature have a profound effect on drilling fluid properties.

## 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.

ISO 91-1:1992, *Petroleum measurement tables — Part 1: Tables based on reference temperatures of 15 °C and 60 °F*

ISO 2719, *Determination of flash point — Pensky-Martens closed cup method*

ISO 2977:1997, *Petroleum products and hydrocarbon solvents — Determination of aniline point and mixed aniline point*

ISO 3007, *Petroleum products and crude petroleum — Determination of vapour pressure — Reid method*

ISO 3016, *Petroleum products — Determination of pour point*

ISO 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity*

ISO 3405:2000, *Petroleum products — Determination of distillation characteristics at atmospheric pressure*

ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 3839, *Petroleum products — Determination of bromine number of distillates and aliphatic olefins — Electrometric method*

ISO 10414-1:2008, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 1: Water-based fluids*

ISO 10414-2:—<sup>1)</sup>, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 2: Oil-based fluids*

---

1) To be published. (Revision of ISO 10414-2:2002)