

INTERNATIONAL STANDARD

ISO 10416

First edition
2002-12-01

Petroleum and natural gas industries — Drilling fluids — Laboratory testing

*Industries du pétrole et du gaz naturel — Fluides de forage — Essais en
laboratoire*

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Reference number
ISO 10416:2002(E)

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Printed in Switzerland

Contents

	Page
Foreword	vii
Introduction.....	viii
1 Scope	1
2 Normative references	1
3 Terms, definitions and abbreviated terms	2
3.1 Terms and definitions	2
3.2 Symbols and abbreviated terms	2
4 Particle size analysis for fines in barite	3
4.1 Principle	3
4.2 Reagents and apparatus	3
4.3 Sampling	4
4.4 Calculation of moisture content	4
4.5 Sieve analysis	5
4.6 Sedimentation analysis	5
5 Barite performance	10
5.1 Principle	10
5.2 Reagents and apparatus	10
5.3 Base drilling fluid preparation	11
5.4 Rheology test	11
5.5 Calculation	12
6 Abrasiveness of weighting materials	12
6.1 Principle	12
6.2 Reagents and apparatus	13
6.3 Determination of abrasion	13
7 Standard method for determination of mercury in drilling fluid barite	15
7.1 Principle	15
7.2 Reagents and apparatus	15
7.3 Preparation of standards	17
7.4 Sample digestion	17
7.5 Check for recovery of Hg during digestion	17
7.6 Analysis of standards and samples	18
7.7 Calculation	18
8 Standard method for determination of cadmium and lead in drilling fluid barite	19
8.1 Principle	19
8.2 Reagents and apparatus	19
8.3 Preparation of combined cadmium and lead standards	20
8.4 Sample digestion	20
8.5 Analysis of standards and samples	20
8.6 Calculation	21
9 Standard method for determination of arsenic in drilling fluid barite	21
9.1 Principle	21
9.2 Reagents and apparatus	22
9.3 Preparation of standards	23
9.4 Sample digestion	23
9.5 Analysis of standards and samples	24
9.6 Calculation	24
10 Bridging materials for regaining circulation	24

10.1	Principle	24
10.2	Apparatus	25
10.3	Preparation of test drilling fluid	25
10.4	Static slot test	25
10.5	Dynamic slot test	26
10.6	Static marble bed test	26
10.7	Dynamic marble bed test	26
10.8	Static ball bearings (BB) bed test	26
10.9	Dynamic ball bearings (BB) bed test	27
11	Filtration control agents	27
11.1	Principle	27
11.2	Reagents and apparatus	27
11.3	General instructions for preparation of base drilling fluids	29
11.4	Salt-saturated drilling fluid	29
11.5	High-hardness, salt-saturated drilling fluid	29
11.6	10 % potassium chloride (KCl) drilling fluid	30
11.7	Prehydrated bentonite slurry	30
11.8	Modified seawater drilling fluid	31
11.9	Low salinity drilling fluid	31
11.10	Lime-treated drilling fluid	31
11.11	Low solids, non-dispersed drilling fluid	32
11.12	Freshwater lignosulfonate drilling fluid	32
11.13	Initial performance test	33
11.14	Performance after heat ageing	33
12	Methylene blue test for drill solids and commercial bentonite	34
12.1	Methylene blue capacity of drill solids	34
12.2	Methylene blue capacity of commercial bentonite	36
12.3	Solids content	38
13	Deflocculation test for thinner evaluation	38
13.1	Principle	38
13.2	Reagents and apparatus	39
13.3	Procedure for moisture content	40
13.4	Calculation of moisture content	40
13.5	Preparation of drilling fluid base	40
13.6	Calculation	41
13.7	Determination of rheological properties	41
13.8	Calculation of thinner efficiency	43
14	Standard methods for testing base oils used in drilling fluids	43
14.1	Principle	43
14.2	Reagents and apparatus	43
14.3	Density, relative density (specific gravity), or API gravity-hydrometer method — ISO 3675	43
14.4	Density and relative density of liquids using a digital density meter — ASTM D4052	44
14.5	Kinematic viscosity of transparent and opaque oils — Calibrated capillary tube method — ISO 3104	44
14.6	Distillation — ISO 3405	44
14.7	Aniline point and mixed aniline point — ISO 2977	45
14.8	Pour point — ISO 3016	45
14.9	Flash point by Pensky-Martens closed tester — ISO 2719	46
14.10	Aromatics content — IP 391 or ASTM D5186	46
15	Potassium content — Ion-selective electrode method	47
15.1	Principle	47
15.2	Reagents and apparatus	47
15.3	Preparation of electrodes	48
15.4	Operational check of electrode system	48
15.5	Measurements using a meter with direct concentration readout capability	49
15.6	Measurements with instruments which provide either digital or analog readout in millivolts	49
16	Calcium 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 which provide either digital or analog readout in millivolts.....	52
17	Density of solids — Stereopycnometer method	53
17.1	Principle	53
17.2	Apparatus.....	53
17.3	Procedure — Stereopycnometer method	53
17.4	Calculation — Stereopycnometer method.....	54
18	Density of solids — Air comparison pycnometer method.....	55
18.1	Principle	55
18.2	Apparatus.....	55
18.3	Procedure — Air comparison pycnometer method.....	55
18.4	Calculation — Air comparison pycnometer method	55
19	Sodium in water-based drilling fluids — Ion-selective electrode method	56
19.1	Principle	56
19.2	Reagents and apparatus.....	56
19.3	Preparation and operational check of electrode system	57
19.4	Measurements using a meter with direct concentration readout capability.....	58
19.5	Measurements using a meter with readout in millivolts	58
20	Ageing of water-based drilling fluids	59
20.1	Principle	59
20.2	Practices common to preparation, handling and testing over all temperature ranges	59
20.3	Drilling fluid sample preparation and ageing at ambient temperature.....	60
20.4	Drilling fluid ageing at moderate temperatures (ambient to 65 °C)	61
20.5	Drilling fluid ageing at substantially elevated temperatures (over 65 °C).....	62
20.6	Inertness and chemical compatibility in high-temperature ageing cells	65
20.7	Obtaining supplies and services for the ageing of drilling fluid samples	66
21	Ageing of oil-based drilling fluids	66
21.1	Principle	66
21.2	Apparatus.....	67
21.3	Practices common to preparation, handling and testing over all temperature ranges	68
21.4	Drilling fluid ageing at ambient temperatures.....	69
21.5	Drilling fluid ageing at moderate temperatures (ambient to 65 °C)	70
21.6	Drilling fluid ageing at substantially elevated temperatures (over 65 °C).....	71
21.7	Inertness and chemical compatibility in high-temperature ageing cells	72
21.8	Obtaining supplies and services for the ageing of drilling fluid samples	73
22	Shale particle disintegration test by hot rolling.....	73
22.1	Principle	73
22.2	Reagents and apparatus.....	74
22.3	Procedure.....	75
22.4	Calculation	75
23	Drilling fluid materials — High viscosity polyanionic cellulose (PAC-HV) (regular).....	76
23.1	Principle	76
23.2	Determination of moisture content	76
23.3	Procedures with test fluid containing PAC-HV	77
24	Drilling fluid materials — Low viscosity polyanionic cellulose (PAC-LV).....	79
24.1	Principle	79
24.2	Determination of moisture content	80
24.3	Procedures with test fluid containing PAC-LV	80
25	Preparation and evaluation of invert emulsion drilling fluids	83
25.1	Principle	83
25.2	Reagents and apparatus.....	83

25.3	Mixing of initial drilling fluid.....	84
25.4	Testing of properties of initial drilling fluid	85
25.5	Preparation of sample contaminated by seawater	85
25.6	Preparation of sample contaminated by Base Evaluation Clay	86
25.7	Preparation of sample contaminated by mixed-salt brine	86
25.8	Procedure for hot rolling	86
25.9	Procedure for static ageing.....	86
25.10	Procedure for testing after heat ageing	87
26	Permeability plugging in cells with set-screw-secured end caps (HTHP filtration)	87
26.1	Principle	87
26.2	Safety considerations	87
26.3	Apparatus — Permeability plugging apparatus (PPA) with set-screw-secured end caps	89
26.4	Procedure for high temperature, high pressure (HTHP) filtration.....	91
26.5	Test conclusion and disassembly	94
26.6	Data reporting	95
27	Permeability plugging in cells with threaded end caps (HTHP)	96
27.1	Principle	96
27.2	Safety considerations	96
27.3	Apparatus — Permeability plugging apparatus (PPA) with threaded end caps	98
27.4	Procedure for high temperature, high pressure (HTHP) filtration.....	100
27.5	Test conclusion and disassembly	102
27.6	Data reporting	104
	Bibliography.....	105

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ISO 10416:2002

<https://standards.iteh.ai/catalog/standards/sist/f408072b-73c4-4aac-a72a-31429fac14e2/iso-10416-2002>

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 3.

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 International Standard 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 and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

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Introduction

This International Standard, which establishes testing methodologies for drilling fluid materials, is based on API RP 13I, fifth edition, June 1, 1995 [1]. This International Standard was developed in response to 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 would yield reproducible and accurate results. The tests are anticipated to be performed in a laboratory setting, but could 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.

Users of this International Standard should be aware that further or differing requirements may 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 and reagents, and sometimes the supplier(s) of those materials which are available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO 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 intended 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 normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 2977, *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 3839, *Petroleum products — Determination of bromine number of distillates and aliphatic olefins — Electrometric method*

ISO 10414-1:2001, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 1: Water-based fluids*

ISO 10414-2:2002, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 2: Oil-based fluids*

ISO 13500:1998, *Petroleum and natural gas industries — Drilling fluid materials — Specifications and tests*

ASTM D422, *Standard Test Method for Particle-Size Analysis of Soils*

ISO 10416:2002(E)

ASTM D1141, *Standard Practice for Substitute Ocean Water*

ASTM D4052, *Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter*

ASTM D5186, *Standard Test Method for Determination of Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels by Supercritical Fluid Chromatography*

ASTM E100, *Standard Specification for ASTM Hydrometers*

IP 391, *Test Method for Determination of Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels by Supercritical Fluid Chromatography*

3 Terms, definitions and abbreviated terms

For the purposes of this International Standard, the following terms, definitions and abbreviated terms apply.

3.1 Terms and definitions

3.1.1

ACS reagent grade

chemical which meets purity standards as specified by the American Chemical Society (ACS)

3.1.2

flash side

side containing residue ("flash") from stamping and with concave indentations

3.1.3

darcy

k

permeability of a porous medium, where one darcy is the flow of a single-phase fluid of 1 cP viscosity that completely fills the voids of the porous medium, flowing through the medium under conditions of viscous flow at a rate of $1 \text{ cm}^3 \cdot \text{s}^{-1} \cdot \text{cm}^{-2}$ cross-sectional area, and under a pressure or equivalent hydraulic gradient of $1 \text{ atm} \cdot \text{cm}^{-1}$

NOTE 1 cP = 1 mPa·s.

3.1.4

quarter, verb

mix and divide into four specimens to assure homogeneity of specimens

3.1.5

spurt loss

volume of fluid that passes through the filtration medium before a filter cake is formed

3.1.6

tube sampling

sampling method comprising withdrawal of powdered sample from bag or bulk via a cylindrical device pushed into the sample, locked shut and withdrawn

3.2 Symbols and abbreviated terms

d inner diameter

D outer diameter

AA atomic absorption spectroscopy

ACS American Chemical Society

API American Petroleum Institute

ASTM	American Society for Testing and Materials
BB	ball bearings
CAS	Chemical Abstracts Service
DCP	direct current plasma
DS	drill solids
EDTA	ethylenediaminetetraacetic acid
HHP	high temperature, high pressure
ICP	inductively coupled plasma
ISA	ionic strength adjuster
ISE	ion-selective electrode
LGS	low gravity solids
MBT	methylene blue test
PAC-HV	high viscosity polyanionic cellulose
PAC-LV	low viscosity polyanionic cellulose
PPA	permeability plugging apparatus
PPT	permeability plugging test
PTFE	polytetrafluoroethylene

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4 Particle size analysis for fines in barite (standards.iteh.ai)

4.1 Principle

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Fines are the particles in the range of 2 μm to 10 μm equivalent spherical diameter and are considered to be detrimental to drilling fluids at high concentrations. Both sieve analysis and sedimentation methods for determining fines concentration are described below.

4.2 Reagents and apparatus

4.2.1 Dispersant solution.

Prepare a solution of 40 g sodium hexametaphosphate and approximately 3,6 g sodium carbonate diluted to 1 l with deionized or distilled water. The sodium carbonate is used to adjust the pH of the solution to 9,0 or slightly less. After the initial pH adjustment, check the pH each day the solution is used. When the pH falls below 8,0, discard the solution.

4.2.2 Oven, capable of maintaining a temperature of $105 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$.

4.2.3 Mixer, capable of operation at $11\,500 \text{ r/min} \pm 300 \text{ r/min}$ under load, with single corrugated impeller approximately 25,4 mm in diameter¹⁾.

4.2.4 Container for mixing, 180 mm deep, $d = 97 \text{ mm}$ at top and 70 mm at bottom²⁾.

1) Multimixer Model 9B with B29 impeller is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

2) Hamilton Beach Mixer Cup No. M110-D is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

- 4.2.5 Sieves**, of mesh sizes 75 µm, 45 µm and 30 µm, having a diameter of 76 mm and a depth of 64 mm from top of the frame to the wire cloth.
- 4.2.6 Stopwatch**, with direct-reading counter and an accuracy of 0 min to 25 min over the test interval.
- 4.2.7 Rubber stopper**, size 13 (diameters 68 mm top and 58 mm bottom).
- 4.2.8 Wash bottles**, one containing 125 ml dispersant solution diluted to 1 l with deionized water, and one with deionized water.
- 4.2.9 Laboratory balance**, of sensitivity 0,01 g.
- 4.2.10 Thermometer**, with scale reading including 16 °C to 32 °C, with an accuracy of 0,5 °C.
- 4.2.11 Beaker**, of capacity 250 ml.
- 4.2.12 Water bath or constant temperature room**, capable of maintaining a convenient constant temperature at or near 20 °C.
- 4.2.13 Glass sedimentation cylinder**, 457 mm high and 63,5 mm diameter, and marked for a volume of 1 l (see ASTM D422).
- 4.2.14 Hydrometer**, ASTM No. 151H, conforming to ASTM E100, graduated to read specific gravity of the suspension.
- 4.2.15 Evaporating dishes.** iTeh STANDARD PREVIEW
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- 4.2.16 Laboratory spatulas**, of assorted sizes.
- 4.2.17 Desiccator**, with calcium sulfate (CAS number 7778-18-9) desiccant, or equivalent.
- 4.2.18 Spray nozzle**³⁾. <https://standards.iteh.ai/catalog/standards/sist/f408072b-73c4-4aac-a72a-31429fac14e2/iso-10416-2002>

4.3 Sampling

Obtain four samples of approximately 10 g and one sample of approximately 80 g of the barite by tube sampling and quartering.

4.4 Calculation of moisture content

- 4.4.1** Weigh 10 g ± 0,01 g of barite obtained in 4.3.
- 4.4.2** Dry to constant mass at a temperature of 105 °C ± 3 °C.
- 4.4.3** Cool the sample in a desiccator and weigh.
- 4.4.4** Calculate the moisture content from equation (1), as a percent (mass fraction)

$$w_h = 100 \frac{m_o - m_d}{m_o} \quad (1)$$

3) Spraying Systems Company No. TG 6.5 tip with 1/4 TT body is the trade name of a suitable product supplied by Spraying Systems. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

where

w_h is the moisture content, in percent (mass fraction);

m_o is the mass of original sample, in grams;

m_d is the mass of dry sample, in grams.

4.5 Sieve analysis

4.5.1 Weigh $10\text{ g} \pm 0,01\text{ g}$ of barite obtained in 4.3, and place in a mixing container. Add 44 ml of dispersant solution. Hand-stir the sample and dilute to approximately 350 ml with deionized water. Stir 5 min on mixer.

4.5.2 Wash the sample with the diluted dispersant solution onto a $75\text{ }\mu\text{m}$ mesh sieve. Continue to wash with approximately 400 ml of the dilute dispersant solution using a wash bottle. Then wash the material on the screen using tap water from a spray nozzle at 70 kPa for 2 min. While washing, allow the elbow bend of the nozzle to rest on the rim of the sieve and move the spray of water repeatedly over the surface of the screen. After tap-water washing, wash the sample at least twice with deionized water; then transfer the residue from the screen to a tared evaporating dish, using deionized water to remove the residue from the screen.

4.5.3 Dry the residue in the oven to constant mass, cool in a desiccator. Weigh to $\pm 0,01\text{ g}$.

4.5.4 Repeat 4.5.1, 4.5.2, and 4.5.3 using $45\text{ }\mu\text{m}$ and $30\text{ }\mu\text{m}$ mesh sieves with separate barite samples.

4.5.5 Calculate m_d , w_r and w_f from equations (2), (3) and (4).

$$m_d = m_o \frac{100 - w_h}{100} \quad (2)$$

$$w_r = 100 \frac{m_r}{m_d} \quad (3)$$

$$w_f = 100 - w_r \quad (4)$$

where

w_h is the moisture content, in percent (mass fraction), as determined in 4.4.4;

m_o is the mass of original sample, in grams;

m_d is the mass of dry sample, in grams;

m_r is the mass of residue, in grams;

w_r is the residue remaining on the sieve, in percent (mass fraction);

w_f is the part of material finer than the sieve, in percent (mass fraction).

4.6 Sedimentation analysis

4.6.1 Weigh $80\text{ g} \pm 0,1\text{ g}$ of barite obtained in 4.3, and place in a mixing container. Add $125\text{ ml} \pm 2\text{ ml}$ of dispersant solution, hand-stir the sample and dilute to approximately 400 ml with deionized water. Stir 5 min on mixer.

4.6.2 Transfer the mixture to a 1 l sedimentation cylinder, washing completely the sample into the cylinder and adding deionized water to the 1 l mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 s while holding a rubber stopper in the top of the cylinder.

4.6.3 When the cylinder is set on the countertop, start the timer immediately. Hang the thermometer in the sample suspension.

4.6.4 Take hydrometer and thermometer readings after 5 min, 10 min, 20 min, 40 min, 90 min, 180 min and 360 min. (This should give particle sizes ranging from less than 2 µm to over 10 µm).

When taking a hydrometer reading, carefully and slowly insert the hydrometer, about 20 s to 25 s before the reading is due, to approximately the depth at which the reading is taken. As soon as the reading has been taken, carefully and slowly remove the hydrometer. Clean hydrometer in deionized or distilled water, and dry.

4.6.5 Calculate the amount (w_d) and particle diameter (D) of the fines material from equations (5) and (6) (see example data sheet and calculation below).

$$w_d = \frac{100\,000\rho}{m_d(\rho-1)} \times (H_c - 1) \tag{5}$$

where

w_d is the mass fraction of sample in suspension, in percent;

m_d is the mass of dried sample, in grams;

ρ is the density of the barite sample, in grams per millilitre (determined according to ISO 13500:1998, clause 7);

H_c is the corrected hydrometer reading [the hydrometer reading minus composite correction (see 4.6.7 and 4.6.8)];

D is the particle diameter (equivalent spherical diameter), in micrometres;

$$D = 100 \sqrt{\frac{30\eta \cdot l}{980(\rho-1)t}} = 17,5 \sqrt{\frac{\eta \cdot l}{(\rho-1)t}} \tag{6}$$

where

η is the viscosity of water at test temperature, in centipoise (cP) (see Table 1);

l is the effective depth of hydrometer, in centimetres (see Table 2);

t is the time interval from start of sedimentation to taking of reading, in minutes.

Table 1 — Viscosity of water at various temperatures

Temperature θ °C	Viscosity η cP	Temperature θ °C	Viscosity η cP
15,6	1,1225	22,2	0,9533
16,1	1,1061	22,8	0,9399
16,7	1,0911	23,3	0,9291
17,2	1,0773	23,9	0,9163
17,8	1,0611	24,4	0,9058
18,3	1,0479	25,0	0,8937
18,9	1,0324	25,6	0,8815
19,4	1,0197	26,1	0,8717
20,0	1,0050	26,7	0,8601
20,6	0,9904	27,2	0,8507
21,1	0,9785	27,8	0,8396
21,7	0,9646	28,3	0,8305

NOTE 1 1 cP = 1 mPa.s.

NOTE 2 Values were calculated by the formula:

$$\eta = \frac{1}{0,021482 \left[(\theta - 8,435) + \sqrt{8078,4 + (\theta - 8,435)^2} \right] - 1,2}$$

where

η is the viscosity, in centipoise;

θ is the temperature, in degrees Celsius.

NOTE 3 See references [3] and [4].

EXAMPLE A typical data sheet for barite and calculation is given below.

Data sheet for barite, SG 4,30				
Time t min	Hydrometer reading ρ g/ml	Temperature θ °C	Hydrometer correction from curve	Corrected hydrometer reading H_c
5	1,035 0	25,5	- 0,001 9	1,033 1
10	1,028 0	25,5	- 0,001 9	1,026 1
20	1,021 0	25,5	- 0,001 9	1,019 1
40	1,014 0	25,5	- 0,001 9	1,012 1
90	1,012 0	25,5	- 0,001 9	1,010 1
180	1,008 5	25,0	- 0,002 0	1,006 5
360	1,007 0	25,0	- 0,002 0	1,005 0

$$w_d = \frac{100\,000(4,30)(1,0261 - 1)}{80(4,30 - 1)} = 42,5 \%$$

$$D = 17,5 \sqrt{\frac{\eta l}{(\rho - 1)t}} = 17,5 \sqrt{\frac{(0,8837) 8,9}{(4,30 - 1)10}} = 8,5$$