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Petroleum and natural gas industries — Completion fluids and materials —

Part 3:
Testing of heavy brines

Industries du pétrole et du gaz naturel — Fluides de complétion et matériaux —

Partie 3: Essais de saumures denses

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document 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, well cements and treatment fluids*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 12, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO13503-3:2005), which has been technically revised.

This document supplements API RP 13J, 5th edition (2014).

The technical requirements of this document and API RP 13J used to be identical. In the meantime API RP 13J has been technically revised as API RP 13J, 5th edition (2014). The purpose of this edition of ISO 13503 is to bring this document up-to-date, by referencing the current edition of API RP 13J and including supplementary content.

The main changes are as follows:

- the method for measuring crystallization temperature in formate brines is described in this document and differs from the method described in API RP 13J, 5th edition (2014) due to the specific nature of formate brines;
- the method for measuring pH in formate brines is described in this document and differs from the method described in API RP 13J, 5th edition (2014), since the API recommended method is unsuitable for formate brines;
- the method for determining carbonate and bicarbonate concentrations in formate brines is described in this document and differs from the buffer capacity method described in API RP 13J, 5th edition (2014), since the API recommended method is unsuitable for formate brines.

A list of all parts in the ISO 13503 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Crystallization temperature is an important property of well construction and intervention fluids used in cold weather conditions and/or under high pressure. API RP 13J, 5th edition (2014) defines the crystallization temperature of a brine as the temperature at which crystals will appear in a brine solution of a given density as it cools. The definition of the crystallization temperature of a brine provided by API RP 13J, 5th edition (2014) is not suitable for formate brines because of prominent supercooling and metastable phase potassium formate crystals that can form at temperatures much lower than crystallization temperature for stable potassium formate crystals.

Formate brines, especially potassium and cesium formate brines and their blends, behave very differently from most other brines due to strong kinetic effects that complicate crystallization temperature measurements. The following factors complicate crystallization temperature measurements in formate brines:

— crystallization temperatures can be very low and can be lower than the cooling capability of the measuring equipment;

— a significant amount of supercooling;

— existence of metastable potassium formate crystals that form in potassium-rich formate brines.

API RP 13J, 5th edition (2014) recommends measuring pH of a concentrated brine on a neat sample, despite multiple reasons indicating this approach is wrong, including Debye-Hückel and pH buffers (standards) formulas being well outside of its validity range. This causes inconsistent, ambiguous, and meaningless pH results. Diluting a brine sample before a pH measurement eradicates these issues.

API RP 13J, 5th edition (2014) recommends measuring buffer capacity of a concentrated brine by a standard carbonate titration to measure the combined carbonate and hydroxide buffering capacity, followed by titration to a randomly selected ‘target pH’ of 7,5 (measured in undiluted brine). By selecting such a low target pH (corresponding to a pH of around 6 in diluted brine) the second titration endpoint is at the part of the titration curve masked by the formate/formic acid equilibrium and is unsuitable for determining the bicarbonate concentration, resulting in inconsistent and ambiguous bicarbonate concentration results. These issues can be eradicated by using a method compromising a pH measurement, a possible pH adjustment, and a phenolphthalein titration. Carbonate/bicarbonate concentrations are calculated based on this data.

Petroleum and natural gas industries — Completion fluids and materials —

Part 3:
Testing of heavy brines

1 Scope

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

This document supplements API RP 13J, 5th edition (2014), the requirements of which are applicable with the exceptions specified in this document.

This document provides more suitable method descriptions for determining the formate brines pH, carbonate/bicarbonate concentrations and crystallization temperature at ambient pressure compared to the methods provided by API RP 13J, 5th edition (2014).

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 RP 13J, 5th edition (2014), *Testing of heavy brines*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in API RP 13J, 5th edition (2014) and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
crystallization temperature
CT
temperature at which crystals and liquid exists in stable equilibrium

Note 1 to entry: This definition takes precedence over the description of Crystallization Temperature in API RP 13 J, 5th edition (2014), 7.1.3.

4 Supplements to API RP 13J, 5th edition (2014)

4.1 General

The requirements specified in API RP 13J, 5th edition (2014) shall apply, with the exceptions specified in 4.2 to 4.5.

4.2 Method for determining iron content

The requirements specified in API RP 13J, 5th edition (2014), Clause 11 shall apply with the following exception:

The method for determining iron content shall not be used on formate brines.

4.3 Method for determining carbonate and bicarbonate concentrations

4.3.1 General

The requirements specified in API RP 13J, 5^{th} edition (2014), Clause 13 shall apply with the following exception:

— The method for determining buffer capacity shall not be used in formate brines.

— Carbonate and bicarbonate determination method described in 4.3.2 to 4.3.6 shall be applied.

Carbonate and bicarbonate concentrations can be measured in all formate single-salt and blended brines:

— sodium formate brine;

— potassium formate brine;

— cesium formate brine;

— sodium/potassium formate brine blends;

— potassium/cesium formate brine blends.

4.3.2 Determination of carbonate concentration by titration in the titration sample

The following steps shall be executed:

a) If the sample is contaminated with solids, it shall be filtered using an API filter press and nitrogen gas. CO2 gas shall not be used.

b) The sample shall be diluted with 9 parts of deionized water. Using 5 ml of brine and 45 ml of deionized water is recommended.

c) pH of the diluted brine sample (original sample) shall be measured with a calibrated glass electrode and recorded.

d) If the measured pH is not between 9,5 and 11,0, it shall be adjusted to 10,0 to10,5 range with a strong acid (HCl, HNO3 or H2SO4) or a strong base (NaOH or KOH). If it is between 9,5 and 11,0, but outside 10,0 to 10,5, it is recommended to adjust it to 10,0 to 10,5 range with a strong acid (HCl, HNO3 or H2SO4) or a strong base (NaOH or KOH).

e) The sample, referred to as the titration sample, shall be titrated down to pH=8,2 using HCl, HNO3 or H2SO4 (0,2 N is the most suitable concentration for formate brines used in the field). It is achieved by dropwise adding an acid solution and recording the volume added and the sample's pH.

f) Once the sample's pH reaches 8,2, the volume acid used shall be recorded (Vacid). The carbonate concentration in the titration sample shall be calculated from Formula (1).

$$[\mathrm{CO_3}^{2-}]_{\mathrm{titr}} = C_{\mathrm{acid}} \times V_{\mathrm{acid}} / V_{\mathrm{brine}} \qquad (1)$$

where

$[\mathrm{CO_3}^{2-}]_{\mathrm{titr}}$	is the carbonate concentration, in mol/l, in the titration sample;
C_{acid}	is the concentration of HCl, HNO_3 or H_2SO^4, in N used for titration;
V_{acid}	is the volume of HCl, HNO_3 or H_2SO^4, in ml, used for titration;
V_{brine}	is the volume of brine, in ml, before dilution.

4.3.3 Calculation of carbonate fraction in the titration sample

4.3.3.1 General

Carbonate fraction in the titration sample shall be calculated and the calculation depends on the type of formate brine.

4.3.3.2 Single salt formate brines

Carbonate fraction shall be calculated using Formula (2):

$$f_{\mathrm{carb}}(\mathrm{pH}) = e^{[a+(b\times\ln(\mathrm{pH})+c)/\mathrm{pH}^2]} \qquad (2)$$

where $f_{\mathrm{carb}}(\mathrm{pH})$ is the molar fraction of buffer that is carbonate as a function of pH.

Table 1 — Formula (2) a, b, and c parameters for sodium, potassium, and cesium formate brines

Brine type	a	b	c	Validity pH-range
sodium formate	-2,36	12,22	-23 029	8,20 to 11,48
potassium formate	-12,29	3 383	-6 638	
cesium formate	-12,55	3 496	-6 881	

4.3.3.3 Na/K formate blend

The following steps shall be followed:

a) Carbonate fraction shall be calculated using Formula (3):

$$f_{\mathrm{carb}}(\mathrm{pH}) = \varphi_{\mathrm{NaHCOO}} \times f_{\mathrm{carb(NaHCOO)}}(\mathrm{pH}) + \varphi_{\mathrm{KHCOO}} \times f_{\mathrm{carb(KHCOO)}}(\mathrm{pH}) \qquad (3)$$

where

$f_{\mathrm{carb(NaHCOO)}}(\mathrm{pH})$	is the molar fraction of carbonate in Na formate brine, calculated by Formula (2);
$f_{\mathrm{carb(KHCOO)}}(\mathrm{pH})$	is the molar fraction of carbonate in K formate brine, calculated by Formula (2);
$\varphi_{\mathrm{NaHCOO}}$	is the volume-fraction of Na formate brine (1,30 g/cm^3 / 10,9 lb/gal) in the blended brine;

φ_{KHCOO}	is the volume-fraction of K formate brine (1,57 g/cm^3 / 13,1 lb/gal) in the blended brine.

b) If the volume fractions of the Na formate and K formate brines are unknown, they shall be estimated based on the density of the blended brine using Formula (4) and Formula (5) [or Formula (6) and Formula (7) if using field units], provided the brine is not heavily diluted with water.

— Metric unit:

$$\varphi_{\mathrm{NaHCOO}} = \frac{1{,}57 - d}{0{,}27} \tag{4}$$

$$\varphi_{\mathrm{KHCOO}} = \frac{d - 1{,}30}{0{,}27} \tag{5}$$

where d is the density of the blend, in g/cm^3 or specific gravity.

— Field unit:

$$\varphi_{\mathrm{NaHCOO}} = \frac{13.1 - d}{2.2} \tag{6}$$

$$\varphi_{\mathrm{KHCOO}} = \frac{d - 10.9}{2.2} \tag{7}$$

where d is the density of the blend, in lb/gal.

4.3.3.4 Cs/K formate blend

The following steps shall be followed:

a) Carbonate fraction shall be calculated using Formula (8):

$$f_{\mathrm{carb}}(\mathrm{pH}) = \varphi_{\mathrm{CsHCOO}} \times f_{\mathrm{carb(CsHCOO)}}(\mathrm{pH}) + \varphi_{\mathrm{KHCOO}} \times f_{\mathrm{carb(KHCOO)}}(\mathrm{pH}) \tag{8}$$

where

$f_{\mathrm{carb(CsHCOO)}}(\mathrm{pH})$	is the molar fraction of carbonate in Cs formate brine, calculated by Formula (2);
$f_{\mathrm{carb(KHCOO)}}(\mathrm{pH})$	is the molar fraction of carbonate in K formate brine, calculated by Formula (2);
$\varphi_{\mathrm{CsHCOO}}$	is the volume-fraction of Cs formate brine (2,20 g/cm^3 18,4 lb/gal) in the blended brine;
φ_{KHCOO}	is the volume-fraction of K formate brine (1,57 g/cm^3 / 13,1 lb/gal) in the blended brine.

b) If the volume fractions of Cs formate and K formate brines are unknown, they shall be estimated based on the density of the blended brine using Formula (9) and Formula (10) [or Formula (11) and Formula (12) if using field units], provided the brine is not heavily diluted with water:

— Metric unit:

$$\varphi_{\mathrm{KHCOO}} = \frac{2{,}20 - d}{0{,}63} \tag{9}$$

$$\varphi_{\text{CsHCOO}} = \frac{d - 1{,}57}{0{,}63} \tag{10}$$

where d is the density of the blend, in g/cm^3 or specific gravity.

— Field unit:

$$\varphi_{\text{KHCOO}} = \frac{18.4 - d}{5.3} \tag{11}$$

$$\varphi_{\text{CsHCOO}} = \frac{d - 13.1}{5.3} \tag{12}$$

where d is the density of the blend, in lb/gal.

4.3.4 Calculation of bicarbonate concentration in the titration sample

Bicarbonate concentration in the titration sample shall be calculated using Formula (13):

$$[\text{HCO}_3^{\ -}]_{\text{titr}} = [\text{CO}_3^{\ 2-}]_{\text{titr}} \frac{1 - f_{\text{carb}}(\text{pH})}{f_{\text{carb}}(\text{pH})} \tag{13}$$

where

$[\text{HCO}_3^{\ -}]_{\text{titr}}$	is the bicarbonate concentration, in mol/l, in the titration solution;
$[\text{CO}_3^{\ 2-}]_{\text{titr}}$	is the carbonate concentration, in mol/l, in the titration solution calculated in 4.3.2;
$f_{carb}(\text{pH})$	is the molar fraction of buffer that is carbonate in the titration sample calculated in 4.3.3.

4.3.5 Calculation of total carbonate and bicarbonate concentration

The total carbonate and bicarbonate concentration, which is pH-independent in the relevant pH-range, is the same in the original sample and the titration sample. It shall be calculated using Formula (14):

$$\left[\text{CO}_3^{\ 2-} + \text{HCO}_3^{\ -}\right] = [\text{CO}_3^{\ 2-}]_{\text{titr}} + [\text{HCO}_3^{\ -}]_{\text{titr}} \tag{14}$$

4.3.6 Calculation of carbonate and bicarbonate concentrations in the original sample (before pH adjustment)

4.3.6.1 General

The method of calculating of carbonate and bicarbonate concentrations ($\left[\text{CO}_3^{\ 2-}\right]$, $\left[\text{HCO}_3^{\ -}\right]$) in the original sample depends on the original sample's pH and whether a pH adjustment was performed before the titration.

4.3.6.2 No pH adjustment was performed before the titration

If no pH adjustment was performed [see 4.3.2 d)] before the titration, the original sample is identical to the titration sample and Formulas (15) and (16) apply:

$$\left[\text{CO}_3^{\ 2-}\right] = [\text{CO}_3^{\ 2-}]_{\text{titr}} \tag{15}$$

$$\left[\mathrm{HCO_3}^-\right]=[\mathrm{HCO_3}^-]_{\mathrm{titr}} \tag{16}$$

4.3.6.3 pH adjustment was performed before titration

If a pH adjustment was performed [see 4.3.2 d)] before the titration, the molar carbonate and bicarbonate concentrations in the original sample depend on the original sample's pH and can be calculated by Formulas (17) and (18):

a) If original pH larger than or equal to 11,48:

$$[\mathrm{CO_3}^{2-}]=[\mathrm{CO_3}^{2-}+\mathrm{HCO_3}^-] \tag{17}$$

$$[\mathrm{HCO_3}^-]=0 \tag{18}$$

b) If original pH is less than 11,48 and larger than 8,2:

1. Carbonate fraction for the original sample shall be determined according to the method in 4.3.3, but the pH value of the original sample shall be used instead of the pH value of the titration sample.
2. The molar carbonate and bicarbonate concentrations in the original sample shall be calculated using Formulas (19) and (20):

$$[\mathrm{CO_3}^{2-}]=[\mathrm{CO_3}^{2-}+\mathrm{HCO_3}^-]\times f_{\mathrm{carb}}\left(\mathrm{pH_{orig}}\right) \tag{19}$$

$$[\mathrm{HCO_3}^-]=[\mathrm{CO_3}^{2-}+\mathrm{HCO_3}^-\left]-[\mathrm{CO_3}^{2-}\right] \tag{20}$$

where $\mathrm{pH_{orig}}$ equals the pH of the original sample [before adjustment, see 4.3.2 c)].

c) If original pH is less than 8,2, the molar carbonate and bicarbonate concentrations in the original sample shall be calculated using Formulas (21) and (22):

$$[\mathrm{CO_3}^{2-}]=0 \tag{21}$$

$$[\mathrm{HCO_3}^-\left]+[\mathrm{CO_2}]_{\mathrm{aq}}=\right[\mathrm{CO_3}^{2-}+\mathrm{HCO_3}^-] \tag{22}$$

where $[\mathrm{CO_2}]_{\mathrm{aq}}$ is the dissolved carbon dioxide concentration, in mol/l.

Low $\mathrm{CO_2}$ solubility in formate brines means that most carbonate/bicarbonate buffer in Formula (22) is $\mathrm{HCO_3}^-$, but its exact concentration cannot be determined.

4.4 Method for determining CT

4.4.1 General

The requirements specified in API RP 13J, 5th edition (2014), Clause 7 shall apply with the following exception.

The method for determining CT of formate brines shall be in accordance with 4.4.2 to 4.4.6.

4.4.2 Application and procedure

The CT can be measured in all formate single-salt and blended brines:

- sodium formate brine;
- potassium formate brine;
- cesium formate brine;
- sodium/potassium formate brine blends;
- potassium/cesium formate brine blends.

The overall procedural steps for measurement of CT of formate brines shall be as follows:

a) prepare seeding material (see 4.4.3);

b) select seeding material (see 4.4.4);

c) determine approximate CT (see 4.4.5);

d) determine accurate CT (see 4.4.6).

4.4.3 Preparation of seeding material

4.4.3.1 General

Formate single-salt and blended brines' CT shall be measured with one or more of the following seeding materials:

- sodium formate crystals;
- potassium formate stable crystals;
- cesium formate crystals.

Formate brine seeding crystals may be stored in plastic containers in the freezer. Formate brines or crystals should never be stored in glass containers.

4.4.3.2 Sodium and cesium formate brine seeding crystals

a) Prepare a sample with density in the range:

  - sodium formate brine: 1,25 g/cm^3 to 1,33 g/cm^3;
  - cesium formate brine: 2,15 g/cm^3 to 2,35 g/cm^3.

b) Cool the sample to a temperature of approximately −40 °C and let it stay for minimum 1 hour.

c) Verify visually that crystallization has occurred in the sample; these are the seeding crystals that shall be stored at a temperature lower than −10 °C.

d) If crystallization is not observed, shake the sample by hand to attempt initiating crystallization.

e) If shaking by hand does not initiate crystallization, transfer the cold sample to a glass container.

f) If the sample still has not crystallized, increase the brine concentration by either evaporation or by adding more salt in powder form to the sample without exceeding the limits stated in the first step, and return to b).

4.4.3.3 Potassium formate brine

a) Prepare potassium formate brine sample with density in the range 1,50 g/cm^3 to 1,59 g/cm^3.

b) Cool the sample to a temperature of approximately −40 °C and let it stay for minimum 1 h.

c) Observe visually whether crystallization occurs in the sample; if crystals are observed, proceed to g).

d) If crystallization is not observed, shake the sample by hand to attempt initiating crystallization.

e) If shaking by hand does not initiate crystallization, transfer the cold sample to a glass container.

f) If the sample still has not crystallized, increase the brine concentration by either evaporation or by adding more salt in powder form to the sample without exceeding the limits stated in the first step, and return to b).

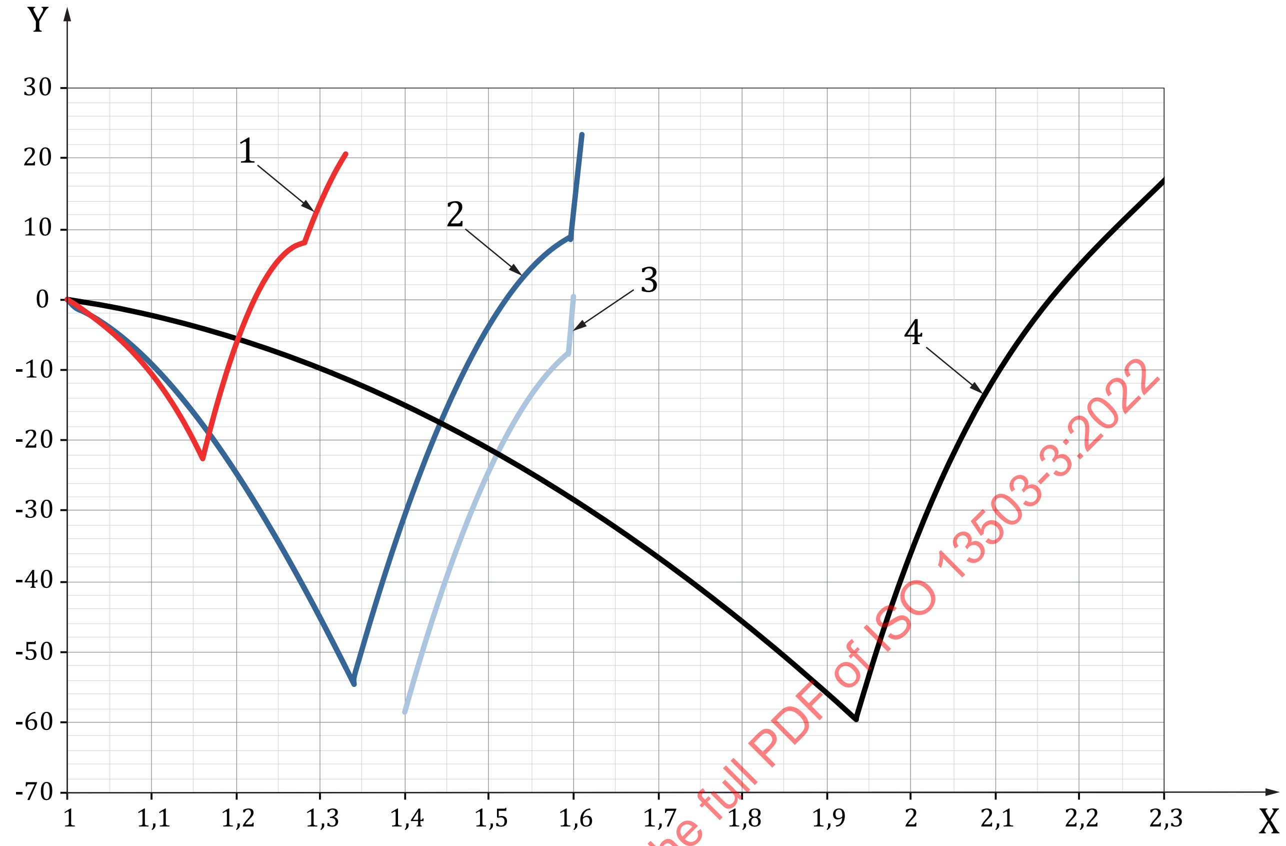
g) Produce stable potassium formate crystals by stirring a new sample of the potassium formate [a)] with the metastable crystals formed in c) at the approximate metastable CT.

This can be achieved with the CT-measuring equipment shown in Figure 3. The transition is observed by a significant and rapid increase in temperature and increase in amount of visual crystals. If the transition does not take place after one hour, the stirring rate should be increased. Patience is often required, as transformation does not always take place easily. Stable potassium formate crystals cannot convert back to metastable crystals and can safely be stored at a temperature below −10 °C.

4.4.4 Selection of seeding material

4.4.4.1 General

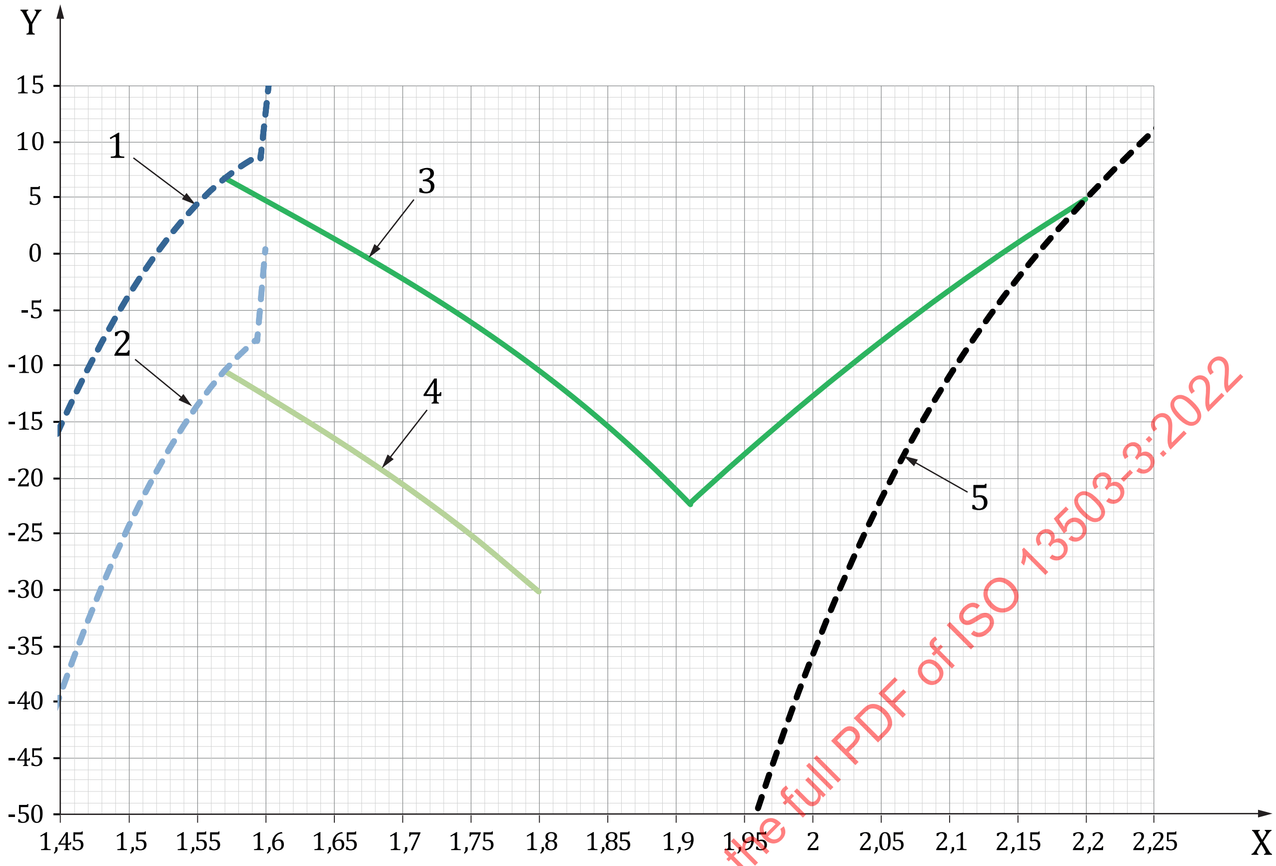
Seeding material should be selected based on brine composition. Figures 1 and 2 can be used as guidance.



Key

X	density [g/cm^3]
Y	temperature [°C]
1	sodium formate
2	potassium formate – stable phase
3	potassium formate – metastable phase
4	cesium formate

Figure 1 — CT of sodium, potassium, and cesium formate single-salt brines as function of brine density



Key

X	density [g/cm³]
Y	temperature [°C]
1	potassium formate – stable phase
2	potassium formate – metastable phase
3	cesium/potassium formate – stable phase
4	cesium/potassium formate – metastable phase
5	cesium formate

Figure 2 — CT of blends of 1,57 g/cm^3 potassium formate and 2,20 g/cm^3 cesium formate brine as function of brine density

4.4.4.2 Single salt brines

If the brine's density is on the left side of the eutectic point (minimum in Figure 1), seeding is not required.

If the brine's density is on the right side of the eutectic point (minimum in Figure 1), a crystal of the same brine, produced as per 4.4.3 shall be used. If measuring CT of potassium formate brine, a stable crystal should be used [see 4.4.3.3 g)].

4.4.4.3 Blended brines

If measuring CT of a blended brine (cesium/potassium formate and sodium/potassium formate), the salt that crystallizes first when the brine is cooled down shall be used as seeding material. If it is not known which salt crystallizes first, it can be determined in the following way, using Figure 2 as guidance:

a) If the brine dilution causes water crystallization (freezing) before salt crystallization, seeding is not required.

b) For cesium/potassium formate blends not covered in a):

   1) If the density higher than 1,95 g/cm^3, cesium is the dominant cation and cesium formate crystals shall be used.

   2) If the density of the blend is in the range from 1,85 g/cm^3 to 1,95 g/cm^3, use cesium formate crystals and stable potassium formate crystals.

   3) If the density is lower than 1,85 g/cm^3 in a brine that has not been diluted with water, potassium is the dominant cation and stable potassium formate crystals shall be used.

   4) If the density of the brine is lower than 1,85 g/cm^3 and the brine could have been diluted with a significant amount of water, use cesium formate crystals and stable potassium formate crystals.

c) For sodium/potassium formate blends not covered in a):

   1) If it is known that sodium is the dominant cation, use sodium formate crystals.

   2) If it is known that potassium is the dominant cation, use stable potassium formate crystals.

   3) If the composition is unknown or sodium and potassium concentrations are similar, use sodium formate crystals and stable potassium formate crystals.

4.4.5 Determination of approximate CT

4.4.5.1 General

An approximate CT is needed for programming the temperature controller described in 4.4.6 and to determine when the seeding crystals should be added.

4.4.5.2 Single salt brines

Figure 1 shall be used to read the approximate CT for the relevant brine.

4.4.5.3 Blended cesium and potassium formate brines

The following provisions apply for blended cesium and potassium formate brines:

a) If it is a straight blend of concentrated cesium formate (density around 2,20 g/cm^3) and potassium formate (density around 1,57 g/cm^3) brines with no water added or removed, the approximate CT can be read from Figure 2.

b) If it is known that the brine can contain some extra water, 5 °C to 10 °C should be subtracted from the value read from Figure 2.

c) If it is known that water can have been removed from the blend, for example by evaporation or addition of dry salt, 5 °C to 10 °C should be added to the value read from Figure 2.

4.4.6 Accurate determination of CT

4.4.6.1 General

The sample should only be seeded once. For the subsequent cycles, no seeding is required. Figure 5 shows the sequence of three such cycles, indicating the various steps required for measurement.

If appropriate seeding crystals have been used, the three values FCTA, TCT and LCTD should be very close. If no seeding material was used, the first TCT is likely to be much too low because of supercooling, assuming the temperature of the cooling bath is low enough to overcome the supercooling. For potassium formate, if metastable potassium formate crystals, no seeding material at all, or seeding