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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

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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.


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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.

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# 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, 5$^{th}$ 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, 5$^{th}$ 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.


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# 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.

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