Guidelines

Silicone-Based Heat Transfer Fluids (SiHTF) in Line Focusing Concentrating Solar Power Applications

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Glossary

AAS	Atomic absorption spectroscopy
BP/DPO	Biphenyl/diphenyl oxide in eutectic mixture
CSP	Concentrating solar power
DLR	Deutsches Zentrum für Luft - und Raumfahrt
DSC	Differential scanning calorimetry
DTA	Differential thermal analysis
GC-MS	Gas chromatography–mass spectrometry
GPC	Gel permeation chromatography
HCE	Heat collecting element (parabolic trough receiver)
HPDSC	High pressure differential scanning calorimeter
HPDTA	High pressure differential thermal analysis
HSE	Health and safety executive
HTF	Heat transfer fluid
ICP-OES	Inductively coupled plasma-optical emission spectrometry
NMR	Nuclear magnetic resonance spectroscopy
O&M	Operation and maintenance
PTC	Parabolic trough collector
SiHTF	Silicone based heat transfer fluid
TAN	Total acid number
T-unit	Branching siloxane group resulting from long-term thermal stress
XRS	X-ray spectrometry

Preface

SolarPACES is an international cooperative network bringing together teams of national experts from around the world to focus on the development and marketing of concentrating solar power (CSP) systems (also known as solar thermal power systems). It is one of a number of collaborative programs, called Implementing Agreements, managed under the umbrella of the International Energy Agency to help find solutions to worldwide energy problems. Within SolarPACES several international Task-activities coordinate the work.

The objectives of Task III "Solar Technology and Advances Applications" deal with the advancement of technical and economic viability of emerging solar thermal technologies and their validation with suitable tools by proper theoretical analyses and simulation codes as well as by experiments in special arrangements and adapted facilities. For this purpose, procedures and techniques are defined for the design, evaluation and use of the components and subsystems to optimize concentration, reception, transfer, storage and application of solar thermal energy. In essence, the goals are to investigate innovative multi-discipline advances needed for the further development of concentrating solar thermal systems. This also concerns, among others, process heat applications, the utilization of solar concentration for the development of improved materials, and the introduction of hybrid solar/fossil power plant concepts.

A group of experts in the field of silicone-based heat transfer fluids (SiHTF) has been working together as members of Task III to create this guideline document covering silicone-based heat transfer fluids for line focusing concentrating solar power applications. The solar community is called to work towards consensus of procedures and to promote the guidelines to be transformed into international IEC standards through the national organizations like UNE, IEC, DKE, ASME, ASTM, etc.

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

The guideline is open to amendments and updating as the state of the art of measurement instruments and procedures advances. Please send comments, amendments, suggestions to christoph.hilgert@dlr.de (Mr. Christoph Hilgert, DLR).

Introduction to the document

This guideline document covers general information, without specific limits, for selecting standard and non-standard test methods for evaluating silicone-based heat transfer fluids in line focusing concentrating solar power applications.

In order to distinguish between "standards listing/citing or referring to further standards" and standards describing methods, this document uses the word "standard" for those listing or citing further standards and the word "methods" for standardized methods (those standards describing test methods).

There are existing standards covering similar applications, but none of them fully covers the application of silicone-based heat transfer fluids in line focusing concentrating solar power applications due to e.g. none suitable or missing methods. Six relevant standards were selected to function as a reference in terms of the individual requirements or methods they refer to. The six "reference standards" are listed below:

- UNE 2016015:2018 (Spain)   Heat transfer fluids for solar thermal power plants with parabolic trough collector technology. Requirements and tests.
- DIN 51522:1998-11 (Germany)   Heat transfer fluids Q - Specifications, test
- DIN 51529:2000-11 (Germany)   Testing of mineral oils and related products, Testing and evaluation of used heat transfer fluids
- GB 23971-2009 (China)   Heat transfer fluids – Requirements and testing, based on DIN 51522-1998
- ASTM D5372-17 (USA)   Standard Guide for Evaluation of Hydrocarbon Heat Transfer Fluids
- ASTM D7665-17 (USA)   Standard Guide for Evaluation of Biodegradable Heat Transfer Fluids

All (established) methods required by these "reference standards" are listed under the section "Existing test methods", while the section "Additional test methods" lists non-standard test methods or additional methods which are considered particularly useful for evaluating silicone-based heat transfer fluids for line focusing concentrating solar power applications.

Consequently, each method no matter if existing or additional has its own section which is divided in six sub-sections:

1) "Relevance to CSP applications" points out the influence of a specific HTF property to line focusing concentrating solar power applications.

2) "Summary of standards" indicates if the "reference standards" are referring to one or several methods for the determination of a specific property. The newest versions of the relevant test methods are listed unless the corresponding standard refers to a specific version of a method. This is the case for the UNE 2016015 standard.
For the sake of simplicity, if available, it is referred to the English version of a method.

3) "Description of methods" gives a brief explanation of the standard and its purpose.

4) "Differences of methods", In many cases the above-mentioned standards are listing different methods for the determination of the same property. In these cases, a comparison between the different methods is presented, resulting in one of the following statements:

"identical"   different standards are referring to the identical method -> no further comment

"equivalent"   different standards are referring to equivalent methods leading to equivalent results

"similar"   different standards are referring to somewhat different procedure leading to somewhat different results. In this case the reasons for different results are mentioned and an evaluation of the differences in the context of CSP is presented

In some cases, a comparison is not presented even though there are different methods available, for the reason that the particular parameter has comparably little relevance in the given context.

5) "Applicability of methods" evaluates whether the corresponding method(s) can be applied to silicone-based HTF.

6) "Alternatives / more suitable methods", are presented in case existing standards cannot be applied conveniently to silicone-based HTF.

Terms and definitions

Heat transfer fluids: This is a general term for substances used as heat transfer carriers.

Unused heat transfer fluids: Heat transfer fluids as delivered that have not been used in a heating system.

Heat transfer fluids in use: According to the UNE 2016015:2018 standard, heat transfer fluids in use refers to heat transfer media which have been filled in the heating system and heated-up at least one time. According to DIN 51522 (German standard) or GB 24747 (Chinese standard) standard, heat transfer fluids in use have been used in a heating system, without further remarks. However, in the case of silicone-based HTF a clear distinction is necessary:

> **SiHTF in use:** Silicone-based heat transfer fluids that have been used in the heating system at high temperatures: in the case of silicone-based heat transfer fluids this state is reached after operation for 30 days (720 hours) at a temperature of approx. 425 °C. After this the physical properties and chemical composition of the fluid remain stable and do not change if further heated.

Thermal stability: The ability of heat transfer fluids to resist chemical decomposition under high temperature conditions. Note: with increase of temperature, heat transfer fluids will go through chemical reaction and/or molecular rearrangement, and the resulting gaseous and non-volatile decomposition products will influence the performance of heat transfer fluids.

Working temperature (bulk temperature): The average main flow temperature of the heat transfer fluid measured at the hot solar collector outlet under the heat transfer system operating condition is the working temperature.

Maximum permitted working (bulk) temperature: Under the operating conditions of the heating system, the maximum mainstream temperature of the heat transfer fluid permitted at the hot solar collector outlet (boiler) is the maximum permitted working temperature. This permissible temperature aims at limiting degradation rate and to avoid significant change of the boiling range.

Maximum permitted film temperature: The film temperature refers to the heat transfer fluid temperature at the heating surface of the hot solar collector outlet, namely the boundary layer temperature of heat transfer fluid in contact with the inner tube wall. The maximum permitted temperature in said boundary layer is the maximum permitted film temperature. The temperature of the heat transfer fluid on any part of the heat transfer system shall not exceed this temperature [1]. The permissible film temperature aims at limiting degradation rate and formation rate of insoluble material which would e.g. give rise to fouling at exchanging surfaces when applying organic HTFs.

Closed heating system: Heating system of heat transfer fluids in which the expansion tank is isolated from the atmosphere. Note: Closed systems generally use inert gas or cold oil seals to isolate the expansion tank from the atmosphere.

Open heating system: Heating system of heat transfer fluids in the expansion tank which connects with the atmosphere.

Introduction to silicone-based heat transfer fluids

The silicone-based heat transfer fluids mentioned in this guideline are linear, clear, odorless and non-reactive polydimethylsiloxanes with a broad viscosity range and they are in general characterized by a very high thermal stability.
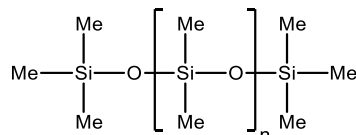

Figure 1: Molecular structure of polydimethylsiloxane heat transfer fluids.

Due to their chemical structures, polydimethylsiloxane heat transfer fluids have a property profile which sets them apart from organic materials such as mineral oils and aromatic heat transfer fluids. At temperatures above 200 °C they undergo rearrangement reactions (equilibration) of their silicone-oxygen bonds when used as intended (under inert conditions). The rate of this molecular rearrangement is directly related to the temperature applied. As a result, low molecular-weight linear and cyclic siloxanes are formed until an equilibrium fluid composition is reached that remains stable. This rearrangement reaction is not a degradation reaction and does not affect fluid lifetime. The newly formed low molecular-weight linear and cyclic siloxanes are part of the heat transfer fluid itself and therefore require operation in closed heating systems. There is no need to separate these low-boiling components from the fluid.
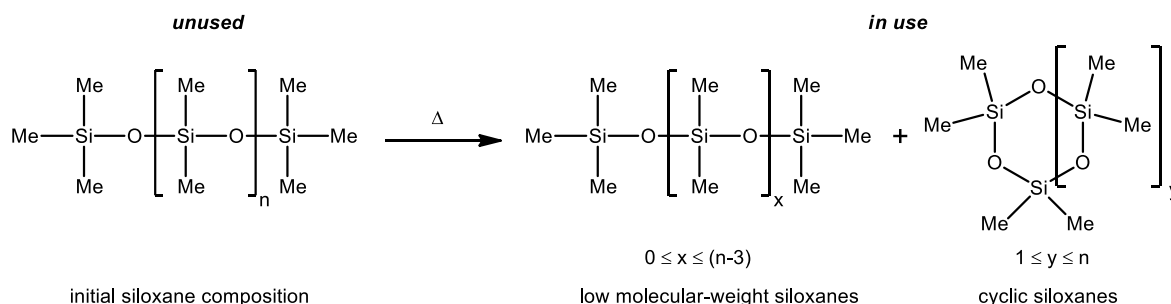
Figure 2: Representation of the equilibrium reaction of typical polydimethylsiloxanes at high temperatures.

Because of the rearrangement reaction some physical properties of the fluids will change within the initial phase of operation. Especially, viscosity and flashpoint decrease, while the equilibrium vapor pressure increases. Once the equilibrium fluid composition is reached the physical properties remain stable (in general this is completed after 30 days of operation at the desired temperature of approx. 425 °C[1]). For comparison reasons, the typical product data and the physical property tables should be provided in the technical relevant pressure and temperature range both, for the heat transfer fluid unused and the heat transfer fluid in use.

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[1] A parabolic trough power plant does not provide such conditions. About 25% of the HTF is at maximum temperature for about 8 to 11 hours of an operational day. Thus, equilibration takes several months.

Existing test methods

1 Fluid composition general and fluid characteristics

1.1 Composition

The composition of an HTF is the information of the chemical identity of the medium or its individual components. For complex mixtures this can be also the chemical family instead of individual compounds.

1.1.1 Relevance to CSP applications

The physical and chemicals properties of the HTF are very relevant for CSP applications. These properties and the HSE classification are determined by the type of chemistry of the HTF. This factor is fundamental for the CSP industry. Accordingly, at least the chemical family is required.

Compounds present in unused and in use HTF have to be known in order to design plants properly in terms of environmental and occupational health issues. Rates of compositional changes during operation are required for laying out processing systems for removal of degradation products.

The chemical identity of the medium or its components shall be indicated in the safety data sheet. For clear identification, the indication of as many designations as possible according to the usual nomenclature systems should be noted.

Due to the long exposure times typically at the upper range of an HTF's thermal stability, for the operation of a plant the "composition after extended use" should be reflected in data sheets. This is not the case today for many products.

1.1.2 Summary of standards

- UNE 206015   manufacturer data sheet of HTF
- DIN 51522   no method mentioned
- DIN 51529   determination of impurities and decomposition products by Gas chromatography-mass spectrometry (GC-MS)
  determination of reactive impurities by differential thermal analysis (DTA)
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.1.3 Description of methods

The composition is required by the UNE 206015 standard only with the remark to the data sheet of the manufacturer`s. There is no existent standard to determine the composition of HTF.

DIN 51529 references to GC-MS for analyzing impurities and degradation products in mg/kg but gives no details on suitable procedures. For testing reactive impurities e.g. via evolution of heat in kJ/kg the same standard references to DTA but again without any details on suitable procedures.

1.1.4 Differences of methods

No comparison is presented.

1.1.5 Applicability of methods

There is no existent standard to determine the composition.

1.1.6 Alternatives / more suitable methods

Silicone fluids are multi-component mixtures of polymeric molecules (classification: polymer). The monomeric unit is siloxane [Me2SiO]n. The fluid composition for silicone fluids can be determined via general polymer analytic methods e.g. GC-MS, GPC, to evaluate important key parameters:

- Mean molecular weight M_W

- Polydispersity D

- Mean chain length

1.2   Chlorine content in %

The term chlorine content is used to designate the amount of chlorine in the HTF.

1.2.1   Relevance to CSP applications

As the corrosiveness of the HTF increases with increasing chlorine concentration, the chlorine content of the heat transfer medium must be known.

1.2.2   Summary of standards

- UNE 2016015   EN ISO 15597:2006-01: Petroleum and related products - Determination of chlorine and bromine content - Wavelength-dispersive X-ray fluorescence spectrometry
- DIN 51522   DIN 51577-3:1990-06 (withdrawn) → DIN ISO 15597:2006-01
- DIN 51529   DIN 51577-3:1990-06 (withdrawn) → DIN ISO 15597:2006-01
- GB 23971   Heat Transfer Fluids or organic heat transfer fluid Annex B (Normative) Determination of Total Chlorine in Crude Petroleum by Coulometric Method
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.2.3   Description of methods

GB 23971: By burning the sample in the presence of oxygen and nitrogen, chlorine in the sample is converted into chloride ions. By titration, the total chlorine content of the sample can be determined.

1.2.4   Differences of methods

SN／T 4570-2016 (GB 23971, Annex B) and EN ISO 15597:2006-01: are identical. Petroleum and related products - Determination of chlorine and bromine content - Wavelength-dispersive X-ray fluorescence spectrometry

1.2.5   Applicability of methods

The methods listed under 1.6.2 are applicable to SiHTFs.

1.2.6   Alternatives / more suitable methods

No alternative methods are required.

1.3   Metal content

The term metal content is used to designate the amount of metals in the HTF.

1.3.1   Relevance to CSP applications

The metal content is relevant because high contents e.g. of chromium can generate environmental and health risks. Furthermore, heat transfer oils could contain traces of metals, either caused by corrosion processes in case of high water levels in the HTF system, abrasion in the production process or by ongoing operation in heat transfer systems. Some metals could have a catalytic effect on the degradation of the HTF itself or could clog filters. So regular oil analyses must be carried out. The concentration of dissolved metals can be evaluated for example through spectrometry.

1.3.2   Summary of standards

- UNE 2016015   ASTM D 5185-18 Standard Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- DIN 51522   no method mentioned
- DIN 51529   determination required by inductively coupled plasma-optical emission spectrometry (ICP-OES) or Atomic absorption spectroscopy (AAS)
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.3.3   Description of methods

DIN 51529 references to ICP-OES and AAS to analyze metal content in mg/kg without indicating any details on suitable procedures.

ASTM D5185-18: This test method determines the concentration of elemental metals present in an oil sample. The oil sample is mixed first with a solvent (organic solvent e.g. Xylol) and then with argon gas to form a fine mist. This mist is introduced into a plasma flame. Elemental metals present in the sample will emit energy in the form of light when introduced to the plasma. Each element emits light at known wavelengths. By measuring the intensity of the light emitted at these known wavelengths the calibrated instrument can determine the concentration in ppm for the metallic elements present in the sample. This only works for dissolved metals, particles are not detected.

1.3.4   Differences of methods

No comparison is presented.

1.3.5   Applicability of methods

The ASTM D 5185-18 is not applicable to SiHTFs as the solvent used for organic HTFs cannot be used for SiHTFs.

1.3.6   Alternatives / more suitable methods

After microwave digestion of the sample with a mixture of hydrofluoric and nitric acid (HF/HNO_3), the digestions are mixed with mannitol, evaporated in the flow box and, after resumption, the sample is measured with ICP-OES or ICP-MS.

1.4 Sulfur content (mass fraction) in %

Sulfur is an element commonly found in crude oil and petroleum products. Sulfur is considered an undesirable contaminant because, when burned, it generates sulfur oxides. Consequently, most finished petroleum products have a limit on how much sulfur they can contain, making sulfur removal an important part of the overall refinery process. Sulfur is used in many extreme pressure oils and metal-working fluids as a pressure additive. The basic way this works is for the sulfur to react at elevated temperatures to form a sulfide layer which improves sliding but prevents scuffing or galling.

1.4.1 Relevance to CSP applications

HTF grades with chloride and sulfur levels in the ppm range may not be suitable for use in CSP plants due to the risk of leaks and expensive equipment replacements from stress corrosion cracking.

1.4.2 Summary of standards

- UNE 2016015   ISO 20846:2011, Petroleum products - Determination of sulfur content of automotive fuels - Ultraviolet fluorescence method.
  ISO 20884:2004, Petroleum products - Determination of sulfur content of automotive fuels - Wavelength-dispersive X-ray fluorescence spectrometry.

- DIN 51522   DIN 51400-6:1990-12, Testing of mineral oils and fuels; determination of sulfur content (total sulfur); analysis by wavelength dispersive X-ray spectrometry (XRS)

- DIN 51529   ISO 14596:1998, Petroleum products — Determination of sulfur content — Wavelength-dispersive X-ray fluorescence spectrometry

- GB 23971   GB/T388, Determination of sulfur content in petroleum products (oxygen bomb method)
  GB/T11140, Standard test method for sulfur in petroleum products by wavelength-dispersive X-ray fluorescence spectrometry
  GB/T17040, Standard test method for sulfur in petroleum and petroleum products by energy-dispersive X-ray fluorescence spectrometry
  SH/T0172, Determination of sulfur content in petroleum products (high-temperature method)
  SH/T0689 e, Standard test method for determination of total sulfur in light hydrocarbons, motor fuels and oils by ultraviolet fluorescence

- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.4.3 Description of methods

After combustion the sulfur is determined either by X-ray spectrometry or UV fluorescence detection.

1.4.4 Differences of methods

The methods use either X-ray spectrometry or UV fluorescence detection and are widely equivalent.

1.4.5 Applicability of methods

All above mentioned methods can be applied to SiHTF. As there is no sulfur involved in the production process of SiHTF the sulfur content is typically very low <10ppm.

1.4.6 Alternatives / more suitable methods

ISO 14596; Petroleum products - Determination of sulfur content - Wavelength-dispersive X-ray fluorescence spectrometry is considered more simple in terms of its applicability.

1.5 Water content

Water content is the quantity of water contained in a material. It can be given on a volumetric or mass (gravimetric) basis.

1.5.1 Relevance to CSP applications

High water content leads to high vapor pressure (see section 1.18 Vapor pressure). It may also impact the corrosivity and other parameters like aging rate of an HTF and hydrogen formation rate and thus the hydrogen concentration. Concerning SiHTFs, T-units are formed as consequence of water-induced degradation reactions. In consequence fluid viscosity will increase significantly faster.

1.5.2 Summary of standards

- UNE 2016015   ISO 12937:2000, Petroleum products - Determination of water - Coulometric Karl Fischer titration method
- DIN 51522   DIN 51777:2020-04, Petroleum products - Determination of water content using titration according to Karl Fischer
- DIN 51529   DIN 51777
- GB 23971   GB/T 11133, Liquid petroleum products – Determination of water – Karl Fischer reagent method
- ASTM D5372   ASTM D95 - 13(2018), Standard Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
- ASTM D7665   ASTM D95

1.5.3 Description of methods

According to ISO 12937:2000, DIN 51777:2020-04 and GB/T 11133, water content of an aliquot of the HTF sample is determined with the Karl Fischer titration. Due to low solubility of water most commonly the coulometric version of the method is applied.

According to ASTM D95 water is boiled out of an aliquot of the sample. After condensation it is collected in a graduated cylinder. The method is recommended for water content up to 25 % by volume but cross sensitivity to evaporable and water-soluble compounds has to be considered. Water content and particles content are determined after filling an aliquot of sample into a glass jar. The sample is then examined visually for clarity. After swirling visual sediment on or water drops below the vortex are checked.

1.5.4 Differences of methods

Karl Fischer titration quantifies any dissolved or undissolved water within the sample aliquot. Visual inspections can only detect undissolved water. The latter can be particularly helpful when large amounts of water (like several percent) are present in the sample. In such case Karl Fischer might cause minor findings as the aliquots might not be collected from the sample in a representative manner.

1.5.5 Applicability of methods

Coulometric Karl Fischer titration works very well for unused and used biphenyl and diphenyl oxide mixtures. It is also suitable for silicone oils but the most appropriate variety of the method should be defined for analyzing silicone HTF. This includes the choice for separated or non-separated cell design, gas extraction or direct injection, suitable anolyte and catholyte. Coulometric Karl Fischer normally only presents dissolved or dispersed water. This is as large amounts of undissolved water will not normally be transferred into this analysis when no special attention is paid on carefully mixing the sample while collecting aliquots for evaluation e.g. by stirring.

Visual inspection according to ASTM D4176 should work for silicone fluids but interference might happen for eutectic mixtures of biphenyl and diphenyl oxide. The latter is as undissolved high boilers might be mixed up with water droplets using the proposed procedure. Large amounts of undissolved water can be identified as liquid droplets below the HTF sample in the case of silicone fluids and as droplets swimming on the HTF sample in the case of mixtures of biphenyl and diphenyl oxide.

1.5.6 Alternatives / more suitable methods

In case of large amounts of undissolved water, the sample can be filled into a graduated cylinder to determine the amount of water volumetrically. Hence, ASTM D4176-04(2019), Standard Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures) is suitable.

1.6 Insoluble compounds

Insoluble products can be defined by lack of solubility of solid compounds in specific solvents like e. g. acetone, toluene or pentane. Another definition is lack of solubility of solid compounds in the HTF at a specific temperature. Such compounds can be referred to as particles. See section 1.7 Particle count and section 1.8, Precipitation number.

1.6.1 Relevance to CSP applications

Insoluble compounds can be caused by degradation products of the HTF, by corrosion products of steel or by contamination with dirt e. g. from welding processes. Hence, the kind of insoluble products may indicate contamination by dirt, corrosion products e. g. due to enhanced humidity, or significant thermal stress of the HTF. Insoluble products may decrease heat transfer on surfaces. They may also cause clogging or blockage of pipelines e. g. in cases of prolonged overheating or wear and clogging of seals and valves.

1.6.2 Summary of standards

- UNE 2016015   ASTM D893 - 14(2018) Standard Test Method for Insolubles in Used Lubricating Oils
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned GB 23971   no method mentioned
- ASTM D5372   ASTM D893 - 14(2018)
- ASTM D7665   ASTM D893 - 14(2018)

1.6.3 Description of methods

ASTM D893 aims at quantitation of particles in used lubricating oil. In "procedure A" a representative sample is mixed with pentane and centrifuged. The oil solution is decanted and the precipitate is washed with pentane. After drying it is weighed giving the pentane insolubles. A similar procedure is based on dissolving an oil sample in toluene giving the toluene insolubles. In "procedure B" a coagulant is added to the solvents.

The pentane insolubles are considered to include oil-insoluble materials and resinous matter from oil or additive degradation. The toluene insolubles are expected to origin from external contamination, wear and corrosion materials.

1.6.4 Differences of methods

No comparison is presented.

1.6.5 Applicability of methods

ASTM D893 can be applied to silicone-based oils.

1.6.6 Alternatives / more suitable methods

DIN EN 12662 also can be applied to silicone-based HTF`s.

As silicone fluids are poor solvents particles might be simply quantified from an oil sample without mixing with additional solvent. Centrifuging as described in ASTM D893 or microfiltration could be used. Remains of silicone oil might be sufficiently removed from the isolated particles with isopropanol.

1.7 Particle count

The term particle count is used to designate the amount of particles in the HTF.

1.7.1 Relevance to CSP applications

The reason for testing particle count is because hard particles in the oil can cause damage to machines like pumps. Particles can lead to abrasion and consequently to wear or damages of valves and other elements exposed to the fluid stream. The particle count gives one of many indications of the abrasivity. In addition, particles such as tar can cause fluid degradation and particles such as metal oxides or carbonaceous material might have catalytical activity and thus cause increased degradation rates of HTF.

1.7.2 Summary of standards

- UNE 2016015   ASTM D7647 - 10, Standard Test Method for Automatic Particle Counting of lubricating and Hydraulic Fluids Using Dilution Techniques to Eliminate the Contribution of Water and Interfering Soft Particles by light Extinction.
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.7.3 Description of methods

ASTM D7647 - 10, test method covers the characterization of particle size in the range of 4 – 200 µm and concentration in lubricants or hydraulic fluids. It is based on using specific optical particle counters based on the light extinction principle. The instrument has to be capable of recording the size and number of particles as they pass across the detector.

1.7.4 Differences of methods

No comparison is presented.

1.7.5 Applicability of methods

ASTM D7647 addresses petroleum products and synthetic based products like polyalpha olefin, polyalkylene glycol, or phosphate ester. Fluids with visible particles or opaque fluids after dilution are beyond the standard method. The test method is specific to automatic particle counters that use the light extinction principle.

The standard is not qualified for silicone fluids. Most likely the standard will not work with the required diluents due to the poor solubility of silicone fluids.

1.7.6 Alternatives / more suitable methods

Filtering off any undissolved particles from the HTF and analyzing the precipitate in terms of organic and inorganic / metallic constituents is more meaningful than summarizing any precipitates as no interference with dispersed droplets or compounds precipitated by diluents is to be expected. Counting particles precipitated by diluents is not directly relevant to wear if these compounds dissolve well in the HTF.

1.8   Precipitation number

Precipitation is defined as the creation of a solid from a solution. When the reaction occurs in a liquid solution, the solid formed is called the 'precipitate'. The chemical that causes the solid to form is called the 'precipitant'. Without sufficient force of gravity (settling) to bring the solid particles together, the precipitate remains in suspension.

Precipitation number as well as insoluble definition is - an index of the proportional amount of solid matter precipitated from oil under test. See section 1.6 Insoluble compounds.

1.8.1   Relevance to CSP applications

Particle precipitation and insolubles may harm pumps and valves. The values should be very low in CSP plants.

1.8.2   Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   ASTM D91 - 02(2019) Test Method for Precipitation Number of Lubricating Oils
- ASTM D7665   ASTM D91

1.8.3   Description of methods

ASTM D91 is on the determination of the precipitation number of steam cylinder stocks and black oils and lubricating oils. The test measures the amount of naphtha-insoluble material in the oil and reports it as the precipitation number. A representative sample is mixed with hexane and centrifuged using specifically graduated centrifuge tubes that allow for measuring the volume of precipitate.

1.8.4   Differences of methods

While ASTM D91 works with the addition of hexane, the procedures described in ASTM D893 (see section 1.6 Insoluble compounds) aim at measuring the volume of particles after adding pentane or toluene. The volume is measured with specifically graduated cone-shaped centrifuge tubes after separating the particles by centrifuging at specific rotating speeds.

1.8.5   Applicability of methods

ASTM D91 and ASTM D893 are both applicable to SiHTFs.

1.8.6   Alternatives / more suitable methods

SiHTFs without additional stabilizers are not known to form precipitates under operation conditions in the range of -50°C to 450°C, thus there is no need for a more suitable method.

1.9   Acid number

Acid number and neutralization number are equivalent measures. The acid number (or neutralization number or acid value or acidity) is the amount of potassium hydroxide (KOH) (in mg/g) needed for neutralization of the acids in an oil.

1.9.1   Relevance to CSP applications

The oxidative state of the HTF is indicated by the total acid number (TAN) of the fluid and increases as it oxidizes. During oxidation, less stable products may be formed and the TAN, an indicator of oxidative state, will start to increase. This property can be monitored using routine sampling and chemical analysis by comparing carbon residues, light- and heavy-chain hydrocarbons over time.

1.9.2   Summary of standards

- UNE 206015   DIN ISO 6618:2015-07: Petroleum products and lubricants – Determination of acid or base number – color-indicator titration method
- DIN 51522   DIN 51558-1 (withdrawn) → DIN ISO 6618:2015-07
- DIN 51529   DIN 51558-1 (withdrawn) → DIN ISO 6618:2015-07
- GB 23971   GB/T4945: Standard test method for acid and base number of petroleum products and lubricants by color-indication titration
  GB/T7304: Petroleum products and lubricants – Determination of acid number – Potentiometric titration method
- ASTM D5372   ASTM D664 - 11a: Test Method for Acid Number of Petroleum Products by Potentiometric Titration
- ASTM D7665   ASTM D664 - 11a

1.9.3   Description of methods

According to all the standards listed above, the acid number is the required amount of base (expressed in milligrams of potassium hydroxide) to neutralize acidity in one gram of heat transfer fluid (mg KOH / g).

1.9.4   Differences of methods

- GB/T 7304 and ASTM D664-11a are identical.
- GB/T 4945 and ASTM D974 are considered equivalent, although there are some differences in the preparation of standard solution of potassium hydroxide-isopropanol between ASTM D974 and GB/T4945. The sample weight of potassium hydroxide used and the preparation process show some differences.
- DIN ISO 6618:2015-07 and ASTM D974 are considered equivalent and are recommended indistinctly. These methods follow the equivalent procedure even though the preparation processes of the samples comprise some differences.

1.9.5   Applicability of methods

All methods listed under 1.1.2 are applicable to SiHTFs.

The inflection point (detected using a color change) is commonly used on fresh HTF; however, for HTF in use the inflection may become indistinguishable requiring the use of the buffer point as the end point.

1.9.6   Alternatives / more suitable methods

ASTM D974 – 12: Standard Test Method for Acid and Base Number by Color-Indicator Titration can also be applied to SiHTF.

1.10 pH-value

The pH scale is used to specify how acidic or basic a water-based solution is. A pH of 7 is neutral. A pH less than 7 is acidic. A pH greater than 7 is basic. See section 1.9, Acid number.

1.10.1 Relevance to CSP applications

The pH value influences the corrosion behavior of the materials in contact with the HTF. HTFs with high TAN should also cause a low pH-value when mixed with water.

1.10.2 Summary of standards

- UNE 2016015   Annex A (informative), Determination of pH-value of organic heat transfer fluids. (Reference value: 6-8)
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.10.3 Description of methods

To determine the pH value of a sample, an aliquot of the HTF sample is extracted with ten volumes of (neutral) distilled water. The pH-value of the aqueous phase (upper layer) of the solution formed after the extraction is determined.

1.10.4 Differences of methods

No comparison is presented.

1.10.5 Applicability of methods

The determination of the pH-value is applicable to SiHTFs.

1.10.6 Alternatives / more suitable methods

See section 1.9 Acid number.

1.11 Water-soluble acid and alkali

Examination of the water-soluble acid or alkali fractions in the specimen by means of the pH-value. See section 1.10 pH-value.

1.11.1 Relevance to CSP applications

Water-soluble acid and alkali would lead to an acidic or basic HTF system which would have an impact on the corrosion behavior (see section 1.10 pH-value). For CSP only pressurized systems with nitrogen blankets are in use. Hence, any contact with oxygen that could give rise to acid formation is rather unlikely. Nevertheless, testing acid, neutralization number, pH-value or water-soluble acid is basically suitable for confirming the absence of air.

1.11.2 Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   GB/T 259 Petroleum products--Determination of water-soluble acids and alkalis
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.11.3 Description of methods

GB/T 259: Extract the water-soluble acids or alkalis in the test sample with distilled water or ethanol water solution, then check the color change of the extracted fluid with a methyl orange or phenolphthalein indicator respectively or determine the pH value of the extractive with a pH meter so as to determine if there is any water-soluble acid or alkali.

1.11.4 Differences of methods

No comparison is presented.

1.11.5 Applicability of methods

GB/T 259 is applicable to SiHTFs.

1.11.6 Alternatives / more suitable methods

No alternative methods are required.

1.12 Density at 15 °C / Specific gravity

The density of an HTF is its mass per unit volume.

1.12.1 Relevance to CSP applications

In itself, density is of low relevance. High density means more mass, and thus more static load of pipes and vessels. It influences the transport volume and thus possibly the logistic costs.

1.12.2 Summary of standards

- UNE 206015   ISO 3675:1999-11: Crude petroleum and liquid petroleum products - Laboratory determination of density - Hydrometer method
  ISO 12185:1996: Crude petroleum and petroleum products – Determination of density – Oscillating U-tube method.

- DIN 51522   DIN 51757:2011-01: Testing of mineral oils and related materials - Determination of density

- DIN 51529   no method mentioned

- GB23971-2009   GB/T 1884:2000: Crude petroleum and liquid petroleum – Laboratory determination of density – Hydrometer method

- ASTM D5372   ASTM D1298 - 12b (2017): Standard Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
  ASTM D4052 - 18a: Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

- ASTM D7665   ASTM D1298 - 12b (2017), ASTM D4052 - 18a:

1.12.3 Description of methods

The ISO 3675 and ASTM D1298 provide test methods for laboratory determination of density by mean of the hydrometer method. The sample is brought to the specified temperature of the test and a test portion transferred to a hydrometer cylinder that has been brought to approximately the same temperature. The appropriate hydrometer, whose temperature has also been regulated, is lowered into the test portion and allowed to settle. After temperature equilibrium has been reached, the hydrometer scale is read, the temperature of the test portion taken and the observed hydrometer reading reduced to 15°C using standard measurement tables.

The ISO 12185 and ASTM D4052 provide test methods for laboratory determination of density by oscillating U-tube method.

1.12.4 Difference of standards

- ISO 3675:1999-11, and ASTM D1298 are equivalent.
- ASTM D4052 and ISO 12185 are considered equivalent for the determination of density of middle distillates when expressed in kg/m3 and different for other products according to the guidance given in ISO/TR 19686-100.
- GB/T 1884 is equivalent to DIN 51757. The only difference is that the temperature is 20°C (GB/T 1884) whereas the density is tested at 15℃ (DIN 51757).

1.12.5 Applicability of methods

All five methods are applicable to SiHTFs.

1.12.6 Alternatives / more suitable methods

No alternative methods are required.

1.13   Specific gravity

Specific gravity is an expression of density in relation to the density of a standard or reference (usually water) at a certain temperature.

1.13.1 Relevance to CSP applications

Specific gravity is covered by the section density (see section 1.12 Density at 15 °C / Specific gravity and A.6 Density at operating conditions).

1.13.2 Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   ASTM D1298 - 12b (2017), Standard Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
                     ASTM D4052 - 18a, Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
- ASTM D7665   ASTM D1298 - 12b (2017), ASTM D4052 - 18a:

1.13.3 Description of methods

See section 1.12 Density at 15 °C / Specific gravity.

1.13.4 Differences of methods

See section 1.12 Density at 15 °C / Specific gravity.

1.13.5 Applicability of methods

Specific gravity is a parameter used to analyze the pumpability of the fluid, as density. Hydraulic shock during pumping can be predicted via the use of a combination of density and compressibility data.

1.13.6 Alternatives / more suitable methods

See section 1.12 Density at 15 °C / Specific gravity.

1.14   Normal boiling point

The normal boiling point of a liquid is the special case in which the vapor pressure of the liquid equals the defined atmospheric pressure at sea level, one atmosphere.

1.14.1 Relevance to CSP applications

The boiling temperature at ambient pressure influences in particular the handling of the heat transfer medium during commissioning. During operation, all areas of the plant that carry the heat transfer medium are inertized and pre-stressed with a pressure above the vapor pressure of the heat transfer medium at maximum process temperature. If the medium can be contaminated with water, it is advantageous if the nominal boiling point of the medium is higher than the one of water, so that the water can be separated by boiling.

1.14.2 Summary of standards

- UNE 2016015   ISO 3405:2011-04, Petroleum products - Determination of distillation characteristics at atmospheric pressure
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   SH/T0558, GB/T6536
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.14.3 Description of methods

According to ISO 3405, the boiling range is measured by distillation of the sample. The substance is evaporated in a defined apparatus under controlled heating. The current boiling temperature is recorded, the vapors are cooled by a cooler, the condensate volume collected is determined using a measuring cylinder. As soon as the first drop condenses here, the temperature is recorded as the start of boiling. This creates a boiling temperature / condensate volume ratio, called as boiling curve.

GB/T6536 is the standard test method for distillation of petroleum products at atmospheric pressure, which is modification of ASTM D86:2007. This test method covers the atmospheric distillation of petroleum products and liquid fuels using a laboratory batch distillation unit to determine quantitatively the boiling range characteristics of such products as light and middle distillates (e.g. aviation gasolines. diesel fuels etc.).

SH/T0558 is the standard test method for boiling range distribution of petroleum fractions by gas chromatography, which is modification of ASTM D2887-15 the Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography. The test method is applicable to petroleum products and fractions having a final boiling point of 538 °C or lower at atmospheric pressure as measured by this test method. This test method is limited to samples having a *boiling range* greater than 55.5 °C, and having a vapor pressure sufficiently low to permit sampling at ambient temperature

1.14.4 Differences of methods

No comparison is presented.

1.14.5 Applicability of methods

As SiHTF is a multi-component mixture of molecules with various molecular weights there is no single boiling point but a boiling range (see Introduction to silicone-based heat transfer fluids). Nevertheless, ISO 3405 is applicable to determine at least the starting point of the boiling range.

1.14.6 Alternatives / more suitable methods

No alternative methods are available that could be applied to mixtures at high temperature and pressure levels.

1.15   Pour point

The pour point of a liquid is the temperature below which the liquid loses its flow characteristics. It is defined as the minimum temperature at which the oil has the ability to pour down from a beaker.

1.15.1 Relevance to CSP applications

The pour point is the parameter to define the lowest operating temperature. If the pour point of the HTF is above the ambient temperature, special technical precautions must be taken, especially during commissioning and longer standstills of the plant or parts of the plant. For example, trace heating of the pipeline system or a back-up heater may be required. No measures are required if the pour point is below the minimum ambient temperature at the CSP location.

1.15.2 Summary of standards

- UNE 2016015   ISO 3016:1994, Petroleum products. Determination of pour point
- DIN 51522   ISO 3016:2019, Petroleum and related products from natural or synthetic sources - Determination of pour point
- DIN 51529   no method mentioned
- GB 23971   GB/T 3535-2006, Petroleum products – Determination of pour point ISO 3016:1994 (This standard has been revised by ISO 3016:2019)
- ASTM D5372   ASTM D97 - 17b
- ASTM D7665   ASTM D97 - 17b

1.15.3 Description of methods

According to ASTM D97 - 17b the specimen is cooled inside a cooling bath to allow the formation of paraffin wax crystals. At about 9 °C above the expected pour point, and for every subsequent 3 °C, the test jar is removed and tilted to check for surface movement. When the specimen does not flow when tilted, the jar is held horizontally for 5 sec. If it does not flow, 3 °C is added to the corresponding temperature and the result is the pour point temperature.

ISO 3016:2019-09: After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3 °C for flow characteristics. The lowest temperature at which movement ('pour' or 'flow') of the sample is observed is recorded as the pour point.

1.15.4 Differences of methods

GB/T 3535-2006, DIN ISO 3016 and ASTM D97 are equivalent.

Applicability of methods

1.15.5 GB/T 3535-2006, DIN ISO 3016 and ASTM D97 can be applied for SiHTF.

1.15.6 Alternatives / more suitable methods

No alternative methods are required.

1.16 Surface tension

Surface tension is the tendency of liquid surfaces to shrink into the minimum surface area possible. Surface tension of a liquid represents the surface stress and is a measure of intermolecular interaction energy. Thus, the molecular properties can be investigated by the experimental surface tension. Interfacial tension measurements of oils provide a sensitive means of detecting small amounts of soluble polar contaminants and products of oxidation.

1.16.1 Relevance to CSP applications

In CSP surface tension is of no relevance.

1.16.2 Summary of standards

- UNE 2016015   ASTM D971-12 Standard test method for interfacial tension of oil against water by the ring method
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.16.3 Description of methods

ASTM D971 covers the measurement of the interfacial tension between mineral oil and water, under non-equilibrium conditions. The test is applied to service-aged oils as an indication of the degree of deterioration. In the interfacial tension measurement, a metallic ring (platinum) is lifted through the water-oil interface. The oil is above the water surface due to the density difference of these two liquids. Water has a higher surface tension that the oil and therefore force is needed to lift up the ring from the water surface. The force measured is used to calculate the interfacial tension between the oil and the water.

1.16.4 Differences of methods

No comparison is presented.

1.16.5 Applicability of methods

ASTM D971 is used to evaluate the interfacial tension but not the surface tension; in principle this method is applicable to SiHTFs.

Most of the standards are carried out with a commercially available tensiometer. Measurement with plates or ring method (different measuring bodies), and in each case the force that is necessary to pull the ring out of the liquid (or from the interface of two liquids), or the force that acts on the plate at the wetting by the liquid (upward) is exerted.

1.16.6 Alternatives / more suitable methods

DIN EN 14370: Determination of surface tension for surface active agents; The force is measured which is either exerted by the surface tension on a plate brought vertically into contact with the liquid and completely wetted by it (static method) or which is required to pull a horizontally suspended bracket or ring out of the liquid surface (quasi-static method). In the static method, the plate is immobile, so that an equilibrium value is obtained. The quasi-static method requires movement of the ring or bracket. Sufficiently small and slow changes in the position of the ring or yoke during measurement minimize the deviations from equilibrium. This method is applicable for SiHTF. DIN EN 14370 refers to ISO 304:1985

ISO 304:1985: Determination of surface tension by drawing up liquid films (Surface active agents): The maximum force is measured which is necessary to act vertically on a stirrup or

a ring, in contact with the surface of the liquid, in order to separate it from this surface. This also can be done with a plate in contact with the surface. The surface tension of pure liquids or other solutions can also be measured by this method.

1.17   Thermal expansion coefficient at 200 °C

Thermal expansion is the tendency of matter to change its shape, area, and volume in response to a change in temperature.

1.17.1 Relevance to CSP applications

It is a material property that is indicative of the extent to which a material / substance expands upon heating. The way in which material/substance properties change due to thermal expansion is of great importance both in the design of plant processes and equipment and, therefore, in plant costs. The diameter of the pipes and the size of the equipment required for their correct operation depend partly on this point.

The most import impact is the dimensioning of expansion and overflow vessels, which may become substantially larger and more expensive as the expansion increases. Therefore, it is important for the design of the process and for the equipment.

1.17.2 Summary of standards

- UNE 2016015   ASTM C531 - 00(2012), Standard Test Method for linear Shrinkage and Coefficient of Thermal Expansion of Chemical-Resistant Mortars, Grouts, Monolithic Surfacings, and Polymer Concretes.
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

1.17.3 Description of methods

ASTM C531 covers the measurement of the linear shrinkage during setting and curing and the coefficient of thermal expansion of chemical-resistant mortars, grouts, monolithic surfaces, and polymer concretes.

A bar of square cross-section is cast to a prescribed length in a mold that holds measuring studs that are captured in the ends of the finished casting.

The change in length after curing is measured and used to calculate shrinkage. The change in length at a specific elevated temperature is measured and used to calculate the coefficient of thermal expansion.

This test method is limited to materials with aggregate size of 0.25 in. (6 mm) or less.

1.17.4 Differences of methods

No comparison is presented.

1.17.5 Applicability of methods

ASTM C531 is not applicable to SiHTFs because ASTM C531 is applicable for solid / cured materials but HTF´s in general are in a fluid condition in the operation temperature range.

1.17.6 Alternatives / more suitable methods

The thermal expansion coefficient can be calculated by means of the TDS of the HTF based on density data.

1.18   Vapor pressure

Vapor pressure or equilibrium vapor pressure is defined as the pressure exerted by a vapor in thermodynamic equilibrium with its condensed phases (solid or liquid) at a given temperature in a closed system. The equilibrium vapor pressure is an indication of a liquid's evaporation rate. It relates to the tendency of particles to escape from the liquid (or a solid). A substance with a high vapor pressure at normal temperatures is often referred to as volatile.

1.18.1 Relevance to CSP applications

The vapor pressure of the HTF together with the pressure losses (and additional safety measures) determines the design pressure of the CSP subsystems. At vapor pressures (operation temperature) lower than the ambient pressure, a heat transfer medium may also be suitable as a storage medium. High vapor pressures lead to increased plant costs. If the medium in its area of application has a vapor pressure above the ambient pressure, pressure vessels are required which cause considerable costs.

When evaluating the resulting design pressures, there are threshold values, the exceeding of which leads abruptly to additional costs. For example, valves, flanges and measurement technology are offered in pressure classes. In combination with the materials used and the design temperature, this results in a maximum value for the design pressure of the system in the selected pressure class. In addition, the main components of the receivers and the flexible connection element of the parabolic trough collectors are currently manufactured in series for a design pressure of 40 bar only. Exceeding this value leads to considerable additional costs.

1.18.2 Summary of standards

- UNE 2016015   determination required, no method mentioned
- DIN 51522   determination required, no method mentioned
- DIN 51529   no method mentioned
- GB 23971   determination required, no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   ASTM D2879 – 18, Standard Test Method for Vapor Pressure-Temperature Relationship and Initial Decomposition Temperature of Liquids by Isoteniscope

1.18.3 Description of methods

ASTM D2879 is a test method for vapor pressure-temperature relationship of pure liquids by isoteniscope. It is applicable to liquids that are compatible with borosilicate glass and that have a vapor pressure between 133 Pa (0.0013 bar) and 101.3 kPa (~1 bar) at the selected test temperatures (from ambient up to 623 K). ASTM 2879 measures vapor pressure of pure liquids (or eutectic mixtures). For regular mixtures it produces readings for a specific filling level of the test device.

1.18.4 Differences of methods

No comparison is presented,

1.18.5 Applicability of methods

ASTM E1782 is suitable for determining vapor pressures in typical operating temperature range of parabolic trough plants. Nevertheless, the ASME E1782 test method is limited to vapor pressure of pure liquids or melts that exhibit a single sharp boiling endotherm under the conditions outlined in this test method. Mixtures in general will cause enhanced uncertainty as partly evaporation of low boilers will make the results less accurate.

Thus, the method does not work for SiHTF within the operational temperature range of CSP applications.

1.18.6 Alternatives / more suitable methods

Due to the issues with mixtures repeated measurements with varyingly high filling levels of the test devices can be used to countercheck the significance of the results achieved with ASTM E1782. The procedure has to be described in detail in order to find readings relevant with respect to CSP application. This is subject to current investigations.

1.19 Kinematic viscosity at 0 °C, 40 °C, 100 °C, and at operating conditions

Viscosity can be conceptualized as quantifying the internal frictional force that arises between adjacent layers of fluid that are in relative motion. For instance, when a fluid is forced through a tube, it flows more quickly near the tube's axis than near its walls. In such a case, experiments show that some stress (such as a pressure difference between the two ends of the tube) is needed to sustain the flow through the tube. This is because a force is required to overcome the friction between the layers of the fluid which are in relative motion: the strength of this force is proportional to the viscosity.

1.19.1 Relevance to CSP applications

The kinematic viscosity is a relevant parameter for the evaluation of the flowing and pumping behavior of the HTF. It influences the pressure losses.

In the temperature range in which the parabolic trough power plant is operated (considering the day and night cycle), the viscosity of HTF concerning the state of the art has little influence on the pressure losses in the system. Even the viscosity at ambient temperature, which is increased by a factor of about 2, does not pose a problem. A critical question regarding viscosity is, whether too low values are a problem for the pumps.

1.19.2 Summary of standards

- UNE 2016015   ISO 3104:1994, Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity

- DIN 51522   DIN 51562-1:1999-01, Viscometry - Measurement of kinematic viscosity by means of the Ubbelohde viscometer
  DIN 53015:2019-06, Viscometry - Measurement of viscosity using the Hoeppler Falling-Ball Viscometer

- DIN 51529   DIN 51562-1:1999-01, DIN 53015:2019-06

- GB 23971   GB/T265: Petroleum Products - Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity

- ASTM D5372   ASTM D445 – 19a: Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

- ASTM D7665   ASTM D445 – 19a

1.19.3 Description of methods

DIN 51562-1, ASTM D445-19a and GB/T265:

The viscosity of the test liquid is determined by measuring the time it takes for the sample, whose volume is defined by two ring-shaped measuring marks, to flow laminarly through a capillary under the influence of gravity.

This method is suitable for measuring kinematic viscosities within a range of 0.35 to 100 000 mm^2/s with flow times ranging from 200 s to 1 000 s at temperatures from 10 °C to 100 °C.

When using the viscometer at temperatures or flow times outside the above ranges, additional errors of measurement may occur which are not included in the uncertainty of measurement specified in clause 14 in DIN 51562-1.

DIN 53015: A ball moves in rolling and sliding motion in an inclined cylindrical tube filled with the fluid to be tested. The time required for the ball to travel a defined measuring distance is measured. By swiveling the measuring part, the return of the ball can also be used for measurement. With the help of Stokes' sedimentation law the dynamic viscosity is calculated, which is given in mPa*s (10^{-3} Pa*s).

1.19.4 Comparison of standards

No comparison is presented.

1.19.5 Applicability of methods

All methods are applicable for SiHTF.

1.19.6 Alternatives / more suitable methods

Alternatively, the dynamic viscosity according to DIN 53019 can be measured and converted to achieve the kinematic viscosity.

1.19.7 Viscosity at operating conditions

Currently no methods exist to measure the viscosity under operating conditions. However, the company flucon GmbH developed a sensor enabling an in-situ measurement of the dynamic viscosity in a pipe system up to 300 °C. [2][2]. The device is currently under testing in relevant environment with SiHTF and other fluids.

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[2] Equipment for >400 °C is currently under testing and available soon.

1.20 Viscosity index

The viscosity index (VI) is an arbitrary, unit-less measure of a fluid change in viscosity relative to temperature change ranging from 0 to 100. It is mainly used to characterize the viscosity-temperature behavior of lubricating oils. The higher the VI value, the lower the decrease of viscosity at higher temperatures that means the higher the VI, the more stable the viscosity remains over temperature fluctuations. Normally the viscosity index is used to indicate the lubricity of oils in a certain temperature range.

1.20.1 Relevance to CSP applications

The viscosity index is not relevant for CSP applications because normally the viscosity index is used to indicate the lubricity of oils in a certain temperature range e.g. for engine oils whereas for CSP plants it is essential to know the viscosity of the HTF at a certain temperature. Furthermore, thermal fluids operate in closed circuit systems without continuous exposure to air, unlike lubricating oils or hydraulic fluids that operate in open systems with continuous exposure to air. Hence, oxidation has a neglectable influence in the viscosity in CPS applications.

1.20.2 Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   ASTM D2270 - 10(2016), Standard Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 °C and 100 °C
- ASTM D7665   ASTM D2270 - 10(2016)

1.20.3 Description of methods

The ASTM D2270 method covers the procedure for calculating the viscosity index of petroleum products, such as lubricating oils, and related materials from their kinematic viscosities at 40 °C and 100 °C. This practice does not apply to petroleum products with kinematic viscosities less than 2.0 mm^2/s at 100 °C.

The determination of the kinematic viscosity of a petroleum product shall be carried out in accordance with test methods D445, D7042, IP 71, or ISO 3104. If viscosity index calculated for a given sample using kinematic viscosity measurements from different test methods are in disagreement, the values calculated from test method D445 measurements shall be accepted.

1.20.4 Differences of methods

No comparison is presented.

1.20.5 Applicability of methods

ASTM D2270 is applicable to SiHTFs.

1.20.6 Alternatives / more suitable methods

DIN 51563:2011-04 and DIN ISO 2909:2004-08 describe as well how to calculate viscosity index from kinematic viscosity.

2 Thermal stability

2.1 Fluid thermal stability

According to UNE 206015 the fluid thermal stability is the resistance of a heat transfer fluid against cracking within a temperature range for which it is specified.

According to GB 23971 the thermal stability is the ability of heat transfer fluids to resist chemical decomposition under high temperature condition.

Note: with increase of temperature, heat transfer fluids will go through chemical reaction or molecular rearrangement, and the resulting gaseous decomposition products, low-boiling components, high-boiling components and nonvolatile decomposition products will influence the performance of heat transfer fluids.

2.1.1 Relevance to CSP applications

The thermal stability of an HTF typically limits and thus determines the maximum operating temperature (see section 2.7 Maximum permitted bulk temperature) of parabolic trough power plants. This value is also used to estimate the life span and exchange rate (if needed) of the HTF under operating conditions.

2.1.2 Summary of standards

- UNE 2016015   DIN 51528:1998-07, Testing of mineral oils and related products - Determination of thermostability of unused heat transfer fluids

- DIN 51522   DIN 51528:1998-07

- DIN 51529   DIN 51528:1998-07

- GB 23971   GB/T23800, Heat transfer fluids – Determination of thermal stability

- ASTM D5372   ASTM D6743 - 11(2015), Standard Test Method for Thermal Stability of Organic Heat Transfer Fluids

- ASTM D7665   ASTM D6743 - 11(2015)

2.1.3 Description of methods

DIN 51528, GB/T23800: To assess the thermal stability of the HTF mass fractions of high and low boilers together with gases and nonvolatile decomposition products of the heat-stressed fluid are used to detect the entire degree of decomposition of the heat carrier. The mass fraction of gaseous reaction products is generally negligible. The heat transfer medium to be tested is melted into a borosilicate glass ampoule or filled into a stainless-steel test vessel and sealed gas-tight. For safety reasons, the glass ampoules are inserted in a metallic protective tube. The glass ampoules in the protective tube or the test vessel are tempered for a specified period of time at a test temperature of ± 1 K. The test temperature is advantageous to be set in the upper application temperature of the individual heat carrier. For each test an unused heat transfer medium has to be used. After completion of the annealing and opening of the glass ampoules or test vessels, the mass fraction is evaluated. Low and high boilers of the test substance determined by gas chromatography based on DIN 51435. All non-evaporable products which cannot be detected by gas chromatography shall be determined separately in a ball tube distillation apparatus (at 250 °C and 0.1 mbar). The test results of the annealed samples are compared with those of the unused heat carrier.

ASTM D6743: This test method covers the determination of the thermal stability of unused organic heat transfer fluids. The procedure is applicable to fluids used for the transfer of heat at temperatures both above and below their boiling point. It is applicable to fluids with maximum bulk operating temperature between 260 °C (500 °F) and 454 °C (850 °F). The procedure shall not be used to test a fluid above its critical temperature. In this test method, the volatile decomposition products are in continuous contact with the fluid during the test.

This test method will not measure the thermal stability threshold (the temperature at which volatile oil fragments begin to form), but instead will indicate bulk fragmentation occurring for a specified temperature and testing period.

2.1.4 Differences of methods

GB/T 23800, DIN 51528 and ASTM D6743 are equivalent. According to GB/T 23800 the capacity of the glass ampoules is 15 ml, the testing time is 720 h and the determination of low and high boilers of the test substance by gas chromatography (DIN 51435) is required. DIN 51528 defines the glass ampoules to be at least 5 ml, a testing time of at least 480 h and requires gas chromatographical analysis of low and high boilers (DIN 51435) as well as determination of gases via weight loss between closed and opened ampoules and nonvolatile products via ball-tube distillation up to 250 °C and 0.1 mbar. ASTM D6743 requires at least 27 g HTF and a test period of at least 500 h. GB/T added the requirement of weighting accuracy and the report of the change in appearance after and before testing.

2.1.5 Applicability of methods

DIN 51528 and ASTM D6743 presuppose that all formed low and high boilers (higher and lower as the HTF) are degradation products. In both standards it is stated that they have not been evaluated for silicone fluids. SiHTF equilibrates and forms high and low boilers during operation. The formed low and high boilers are still part of SiHTF and therefore cannot be considered as degradation products. In other words, equilibrated SiHTF consists of high and low boiling components.

2.1.6 Alternatives / more suitable methods

Silicone-based heat transfer fluids (SiHTF) in general are polymeric mixtures of linear siloxanes. Concerning the thermal stability of SiHTF, the determination of the T-unit amount can be applied:

Unused SiHTF contain low values of T-units (in the lower ppm range < 1000 ppm). During operation above 250 °C, T-units are continuously formed and will slowly lead to branching of the former separate siloxane molecules. The amount of T-units present in the SiHTF is therefore linked with the viscosity of the fluid. In the end, higher amounts of T-units could lead to a viscosity increase.

The amount of T-units can be determined via NMR-spectroscopy - the maximum tolerable amount of T-units depend on the fluid composition and has to be defined by the fluid manufacturer.

In addition, the formation of gases should be analyzed not only in terms of mass loss but as amount of the individual gases per mass of HTF. Longer aging times like 3000 – 5000 h (compared to existing methods) should be considered in order to represent a larger fraction of HTF life span in CSP applications. This is as aging rates are not necessarily constant in time. For BP/DPO they can increase on pro-longed aging while at least gas formation rate can decrease for silicone fluid. [3] [4]

2.2 Ash content (mass fraction) in %:

The ash content of an HTF is a measure of the amount of inorganic noncombustible material it contains.

2.2.1 Relevance to CSP applications

For organic HTF the ash content (micron-size spherical particles) can be used for the determination of inorganic contamination of the fluid. Concerning SiHTFs determining the ash content is not relevant as potential inorganic contamination of the fluid will be mixed with the combustion product SiO_2.

2.2.2 Summary of standards

- UNE 206015   no method mentioned
- DIN 51522   ISO 6245:1995-11 (withdrawn) →ISO 6245:2001: Petroleum products — Determination of ash
- DIN 51529   no method mentioned
- GB 23971   GB/T 508: Petroleum products – Determination of ash (GB/T 508-1985, eq. ISO 6245:1982) (withdrawn) → ISO 6245:2001
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

2.2.3 Description of methods

A test portion contained in a suitable vessel is ignited and allowed to burn until only ash and carbon remain. The carbonaceous residue is reduced to an ash by heating in a muffle furnace at 775 °C, allowed to cool and weighed.

2.2.4 Differences of methods

GB/T 508 and ISO 6245 1982 are identical

2.2.5 Applicability of methods

ISO 6245:2001 is applicable for petroleum products and SiHTFs. Nevertheless, as the remaining material (after burning/oxidation) of SiHTFs will consist of large fractions of SiO_2 and potential ashes resulting from inorganic contamination, it does not result in significant values.

Explanation: Silicone-based heat transfer fluids are multi-component mixtures of oligomeric molecules (classification: polymer). The monomeric unit is siloxane $[Me_2SiO]_n$, (see Introduction to silicone-based heat transfer fluids).

The oxidation of silicone fluids in the presence of oxygen starts at about 200 – 250 °C by the formation Si-O-Si bridges and crosslinking. This reaction rapidly leads to higher molecular weights and branched siloxanes and will strongly increase viscosity. Finally, SiO_2, CO_2 and water are formed as the main combustion products. The chemical reaction of the combustion of silicones in the presence of oxygen is

$[Si(CH_3)_2O]_n + 4 \cdot n\ O_2 \rightarrow n \cdot SiO_2 + 2 \cdot n\ CO_2 + 3 \cdot n\ H_2O.$
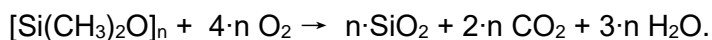
ASTM D 482-03: Standard Test Method for Ash from Petroleum Products, is an alternative method, but still not applicable to SiHTFs.

2.2.6 Alternatives / more suitable methods

Metal content testing can be used as an alternative to inorganic contamination testing of SiHTFs.

2.3 Carbon residue (mass fraction) in %:

The term carbon residue is used to designate the carbonaceous residue formed after evaporation and pyrolysis of a petroleum product under specific conditions.

2.3.1 Relevance to CSP applications

During thermal cracking of hydrocarbons, carbon accumulates in high boiling degradation products. Excessive accumulation high boilers gives rise to formation of carbonaceous material which can lead to fouling [5] of heat exchanger surfaces.

2.3.2 Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   DIN 51551-1:2009-04, Testing of lubricants and liquid fuels - Determination of the carbon residue - Part 1: Conradson method
DIN EN ISO 10370:2015-03, Petroleum products - Determination of carbon residue - Micro method
- DIN 51529   DIN EN ISO 10370:2015-03
- GB 23971   GB/T268 e: Petroleum products, Determination of carbon residue, Conradson method (GB/T 268-1987, neq ISO 6615:1983
SH/T0170: Petroleum products Determination of carbon residue Electric process
GB/T17144: Petroleum products, Determination of carbon residue, Micro method (GB/T 17144-1997, eqv ISO 10370:1993)
- ASTM D5372   ASTM D189 – 06 (2019) Standard Test Method for Conradson Carbon Residue of Petroleum Products
ASTM D524 – 15 (2019): Standard Test Method for Ramsbottom Carbon Residue of Petroleum Products
- ASTM D7665   ASTM D189 – 06 (2019), ASTM D524 – 15 (2019)

2.3.3 Description of methods

These test methods cover the determination of the amount of carbon residue left after evaporation and pyrolysis of an oil and are intended to provide some indication of relative coke-forming propensities. This test method is generally applicable to relatively nonvolatile petroleum products which partially decompose on distillation at atmospheric pressure. The term carbon residue is used throughout this test method to designate the carbonaceous residue formed after evaporation and pyrolysis of a petroleum product under the conditions specified in this test method. The residue is not composed entirely of carbon but is a coke which can be further changed by pyrolysis.

2.3.4 Differences of methods

No comparison is presented, as the mentioned methods cannot be applied to SiHTFs.

2.3.5 Applicability of methods

None of the methods is applicable to SiHTFs.

Silicone fluids show no coke-forming properties, therefore only small amounts of carbon content can be found in the residual material. The main oxidation product of silicone fluids is SiO_2.

2.3.6 Alternatives / more suitable methods

No alternative methods are required.

2.4   Determination of **boiling range**

A boiling range is the temperature range involved in the distillation of oil (complex mixture), from the start to the time when the component with the highest boiling point evaporates.

2.4.1   Relevance to CSP applications

For hydrocarbon-based HTFs, the fluid degradation depends on the concentration of the degradation products. Therefore, when degraded HTF is purified by distillation, the degradation rate is almost reduced to the original rate. If the fluid's stability is independent of the high and low boiling content, the distillation thereof is of little relevance.

High and low boilers affect CSP plants because the new materials generated (with more or less molecular weight than the initial one) may reduce the thermal stability of the HTF. High boilers are known to cause fouling (see A.3 Fouling).

2.4.2   Summary of standards

- UNE 206015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   DIN 51435:2010-03, Testing of petroleum products - Determination of boiling range distribution - Gas chromatography method
- GB23971-2009   no method mentioned
- ASTM D5372   ASTM D86 – 19, Standard Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure
ASTM D1160 – 18, Standard Test Method for Distillation of Petroleum Products at Reduced Pressure
ASTM D2887 – 19, Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography (cross-referenced by ASTM D 6743)
- ASTM D7665   ASTM D86 - 19, ASTM D1160 - 18

2.4.3   Description of methods

ASTM D86 covers the atmospheric distillation of petroleum products and liquid fuels using a laboratory batch distillation unit to determine quantitatively the boiling range characteristics of such products as light and middle distillates, automotive spark-ignition engine fuels with or without oxygenates.

ASTM D1160 is used for the determination of the distillation characteristics of petroleum products, biodiesel, and fractions that may decompose if distilled at atmospheric pressure. The method covers the determination, at reduced pressures, of the range of boiling points for petroleum products and biodiesel that can be partially or completely vaporized at a maximum liquid temperature of 400 °C. Both a manual method and an automatic method are specified.

DIN 51435 and ASTM D2887 describe gas chromatographic methods for determining boiling ranges from chromatograms.

2.4.4   Differences of methods

No comparison is presented.

2.4.5   Applicability of methods

ASTM D1160 is not applicable for silicone fluids because maximum distillation temperature is at 400 °C. Above 200 °C silicone fluids show an equilibration reaction which always form low boiling components, see section Introduction to silicone-based heat transfer fluids. The low molecular-weight linear and cyclic siloxanes which form during heating in silicone fluids are a

part of the heat transfer fluid itself (fluid composition). Therefore, distillation for the separation of high- and low boilers is not applicable in the case of silicone-based HTFs.

DIN 51435 and ASTM D2887 are barely applicable to SiHTFs because due to the equilibration reaction erroneous readings would be concluded from the compositional changes relative to unused HTF.

For organic HTFs all formed low and high boilers (higher and lower than the HTF) are degradation products and can be separated from the fluid.

2.4.6 Alternatives / more suitable methods

In general, there is no need to determine high and low boilers in SiHTFs, as they are part of the heat transfer fluid itself and must not be separated during operation.

2.5 High-boiler content

Thermal degradation or thermal cracking of HTFs is the breaking of bonds in the fluid molecules at temperatures close to or above the recommended maximum bulk temperature or film temperature. In heat transfer terminology, degradation products are known as "low boilers" if they have boiling points lower than the unused fluid and "high boilers" if they have boiling points higher than the unused HTF.

2.5.1 Relevance to CSP applications

High boiling compounds may indicate contamination, oxidation or thermal stress in the fluid. Formation of high boilers may give rise to increasing viscosity and decrease of thermal stability. Minor changes of other properties like density and heat capacity may be caused by high boilers as well. In an advanced phase, there may even be fouling, i.e. loss of heat transfer on surfaces and solid matter formation.

2.5.2 Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   DIN 51528:1998-07, Testing of mineral oils and related products - Determination of thermostability of unused heat transfer fluids
- DIN 51529   DIN 51528:1998-07 -> DIN 51435:2010-03, Testing of petroleum products - Determination of boiling range distribution - Gas chromatography method
- GB 23971   no method mentioned
- ASTM D5372   ASTM D6743 -> ASTM D2887
- ASTM D7665   ASTM D2887

2.5.3 Description of methods

DIN 51528 describes the determination of high boiler fraction in HTF samples with simulated distillation via gas chromatography (GC) according to DIN 51435 after thermally aging of new HTF. HTF samples are separated on a non-polar GC column. The fractions of low and high boilers are determined by referencing to the boiling range of the unused fluid. While DIN 51528 references to DIN 51435 for determining degradation products of samples from thermal stability tests, DIN 51529 references to DIN 51528 for determining low and high boiler content of used HTFs. Likewise, ASTM D6743 and ASTM D7665 reference to the simulated distillation method described in ASTM D2887 for determining the fractions of low and high boilers.

2.5.4 Differences of methods

There are no relevant differences among the methods, thus no comparison is presented.

2.5.5 Applicability of methods

High boiler determination according to DIN 51528, DIN 51529, ASTM D 6743 and ASTM D7665 is not validated for silicone-based HTFs. Due to the equilibration reactions of polydimethylsiloxanes the definition of high boilers is not a viable approach as fresh fluid in the equilibrated condition would have large readings (large fractions of high boilers) without showing any technical issues. Therefore, the above-mentioned methods are not applicable to determine the thermal stability of SiHTFs.

2.5.6 Alternatives / more suitable methods

The high-boiler content cannot be considered to evaluate the thermal stability of SiHTFs and therefore the determination of T-units via NMR shall rather be used, see section 2.1 Fluid thermal stability.

2.6   Low boiler content

Thermal degradation or thermal cracking of HTFs is the breaking of bonds in the fluid molecules by heat in excess of the fluid's recommended maximum bulk temperature or film temperature. Smaller molecules than previously existed can be formed – or the fragments may react with each other to form polymeric molecules larger than previously existed in the fluid. Smaller molecules typically belong to the group of "low boilers" while larger ones typically are "high boilers". The presence of low boilers is evident with a measured decrease in the flash point and viscosity of the thermal fluid as well as an increase in vapor pressure.

2.6.1   Relevance to CSP applications

Concerning the state of the art HTF, low boiling compounds indicate contamination or thermal stress in the fluid. Low molecular weight components may cause cavitation at pumps and they could lower the viscosity value. Low boilers also increase the vapor pressure level in the HTF system. Low boilers are typically partially released from the system when the gas cover of the expansion vessel is vented. Accordingly, a treatment or filter (activated carbon filter or similar) is typically used for exhaust gas treatment. For deliberate removal of low boilers, ullage systems are used that are based on flash distillation.

2.6.2   Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   DIN 51528:1998-07: Testing of mineral oils and related products - Determination of thermostability of unused heat transfer fluids
- DIN 51529   DIN 51528:1998-07 -> DIN 51435:2010-03, Testing of petroleum products - Determination of boiling range distribution - Gas chromatography method
- GB 23971   no method mentioned
- ASTM D5372   ASTM D6743 – 20 -> ASTM D2887 - 19ae2
- ASTM D7665   ASTM D2887 - 19ae2

2.6.3   Description of methods

DIN 51528 describes the determination of low boiler fraction in HTF samples with simulated distillation via gas chromatography according to DIN 51435 after thermally aging of new HTF. HTF samples are separated on a non-polar GC column. The fractions of low and high boilers are determined by referencing to the boiling range of the unused fluid. While DIN 51528 references to DIN 51435 for determining degradation products of samples from thermal stability tests, DIN 51529 references to DIN 51528 for determining low and high boiler content of used HTFs. Likewise, ASTM D6743 and ASTM D7665 reference to the simulated distillation method described in ASTM D2887 for determining the fractions of low and high boilers.

2.6.4   Differences of methods

No comparison is presented.

2.6.5   Applicability of methods

Low boiler determination according to DIN 51528, DIN 51529, ASTM D 6743 and ASTM D7665 is not validated for silicone-based HTFs. Due to the equilibration reactions of polydimethylsiloxanes the definition of low boilers is not a viable approach as fresh SiHTF in the equilibrated condition would have a large high- and low boiler content. Most of those hypothetic low boilers are part of the HTF and cannot be associated with technical issues. Therefore, the above-mentioned methods are not applicable to determine the thermal stability of SiHTFs.

2.6.6 Alternatives / more suitable methods

Instead of low boilers a definition of specific low boiling degradation compounds could be used for silicone-based HTFs. For polydimethylsiloxanes low boiling degradation products are less important than the formation of methane which is the mayor degradation product of this class of compounds. However, the evaluation of low boiling compounds cannot be used for the determination of the thermal stability of a SiHTF and therefore the determination of T-units via NMR shall rather be used, see section 2.1 Fluid thermal stability.

2.7 Maximum permitted bulk temperature

(see section 2.1 Fluid thermal stability)

The concept of the bulk temperature is that adiabatic mixing of the fluid from a given cross section of the duct will result in some equilibrium temperature that accurately reflects the average temperature of the moving fluid. Maximum bulk temperature is the fluid's highest permitted average temperature. This definition is equivalent to the definition of maximum working temperature.

2.7.1 Relevance to CSP applications

Maximum permitted working (bulk) temperature: under the operating conditions of the solar heating system, the average temperature of the heat transfer fluid measured at the outlet of the solar collector (boiler) is the working temperature. It is limited by the heat stability (degradation) of the fluid. Thus, it is the most relevant parameter in order to avoid higher degradation of the HTF than calculated.

As explained in section 2.1 Fluid thermal stability, this is the key design parameter for parabolic trough power plants. The bulk temperature of the HTF directly impacts the storage temperature spread and its mass-specific energy content. In short: High HTF temperature, low storage cost. Also, the steam temperature driving the steam turbine cycle is impacted. From Carnot's law follows: high HTF temperature, high conversion efficiency in power cycles.

2.7.2 Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   DIN 51528:1998-07, Testing of mineral oils and related products - Determination of thermostability of unused heat transfer fluids
- DIN 51529   DIN 51528:1998-07
- GB 23971   GB/T 23800, Heat transfer fluids - Determination of thermal stability
- ASTM D5372   ASTM D6743 - 11(2015), Standard Test Method for Thermal Stability of Organic Heat Transfer Fluids
- ASTM D7665   ASTM D6743 - 11(2015)

2.7.3 Description of methods

According to GB/T 23800 the maximum permitted bulk temperature corresponds to the maximum test temperature at which the deterioration rate of heat transfer fluids being tested does not exceed 10 % (mass fraction). Additionally, it is mentioned that the maximum working temperature shall be at least 10 °C lower than the maximum permitted bulk temperature in actual use.

DIN 51528 and ASTM D6743: After exposing the test fluid to heat, the percentage by mass of high-and low-boiling components is determined by gas chromatography.

2.7.4 Differences of methods

Whereas in GB/T 23800 there is a limit of 10 % of degradation products (mass fraction) for the maximum permitted bulk temperature no limit is set in DIN 51528 and ASTM D6743. Therefore, the supplier of the HTF has to set the limit of the maximum permitted bulk temperature according to the latter methods.

2.7.5 Applicability of methods

SiHTFs equilibrate and form high and low boilers during operation as intended. The formed low and high boilers are still part the SiHTF and therefore are not considered degradation products. For silicone-based HTFs the determination of the thermal stability via low and high

boilers cannot be applied as silicone fluids undergo an equilibration reaction, a rearrangement of molecules takes place anyway which is not a degradation indicator of the fluid.

2.7.6 Alternatives / more suitable methods

Determination of T-units via NMR See section 2.1.6 Alternatives / more suitable methods "T-unit method".

2.8 Thermal oxidation stability (175 °C, 72 h)

In open systems, heat transfer oils react with oxygen under thermal stress. Therefore, the resistance to oxidation is important for the life span of the fluid.

2.8.1 Relevance to CSP applications

Thermal oxidation stability is not relevant as parabolic trough power plants operate HTF under inert conditions in a closed system.

2.8.2 Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   Annex C, see section 2.8.3 Description of methods
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

2.8.3 Description of methods

GB 23971, Annex C: this method is used to determine the thermal oxidation stability of heat transfer fluids under natural convection conditions. It is also used to simulate the contact of the expansion tank in the heat transfer system of heat transfer fluids with the air so as to assess the thermal oxidation stability of heat transfer fluids used in open heating systems. This method is applicable to mineral and synthetic liquid phase heat transfer fluids. Heating the sample in the presence of steel bar catalyst at a certain temperature for at least 72 h, and then assess the thermal oxidation stability of the sample through analytic determination of the sample before and after the thermal test.

2.8.4 Differences of methods

No comparison is presented.

2.8.5 Applicability of methods

GB 23971, Annex C is applicable to SiHTFs.

The oxidation of silicone fluids in the presence of oxygen can be determined. It starts at about 200 – 250 °C by the formation Si-O-Si bridges and crosslinking. This reaction rapidly leads to higher molecular weights and branched siloxanes and will strongly increase viscosity. Finally, SiO_2, CO_2 and water are formed as the main combustion / oxidation products. The chemical reaction of the combustion of silicones in the presence of oxygen is presented below.

$$[Si(CH_3)_2O]_n + 4 \cdot n \ O_2 \rightarrow n \cdot SiO_2 + 2 \cdot n \ CO_2 + 3 \cdot n \ H_2O$$
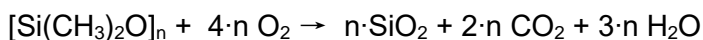
2.8.6 Alternatives / more suitable methods

No alternative methods are required because thermal oxidation stability is not relevant for CSP.

3 Heat transfer

3.1 High-temperature heat capacity

Heat capacity is a physical property of an HTF, defined as the amount of heat to be supplied to a given mass of an HTF to produce a unit change in its temperature.

3.1.1 Relevance to CSP applications

A high specific heat capacity results in a comparably low required mass flow to transport a certain amount of heat. A lower mass flow results in a reduced pressure loss, and consequently in a decreased internal electrical power consumption of the corresponding pump(s). Furthermore, a high specific heat capacity reduces the overall HTF quantity, thus reduces cost.

3.1.2 Summary of standards

- UNE 2016015   ASTM E1269 - 11(2018), Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry
ASTM E2716 - 09(2014), Standard Test Method for Determining Specific Heat Capacity by Sinusoidal Modulated Temperature Differential Scanning Calorimetry
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   ASTM D2766-95(2009), Standard Test Method for Specific Heat of Liquids and Solids (Withdrawn 2018) - No Replacement
- ASTM D7665   ASTM D2766-95(2009)

3.1.3 Description of methods

ASTM E1269 describes the determination of the specific heat capacity by differential scanning calorimetry (DSC) in general. It provides details on the sensing system and the procedure including the amount of sample but interestingly it does not recommend the size of the crucibles to be used.

ASTM E2716 describes the sinusoidal modulated technique.

3.1.4 Differences of methods

ASTM E1269 and ASTM E2716 use different thermal programs for determining heat capacities from DSC measurements.

3.1.5 Applicability of methods

The method is basically not suitable for obtaining isobaric heat capacities of heat transfer fluids as the measurement is performed at saturation pressure. As the amount of HTF per volume of crucible is not defined erroneous results are possible due to heat of evaporation when low masses of HTF are used.

Non-compressible fluids below the normal boiling point have practicably identical values of isobaric heat capacity (c_p) and heat capacity at saturation pressure (c_{sat}). Above the boiling point these parameters tend to deviate but less than the uncertainty of measurement of most commercial DSC instruments (~3-5 %).

For silicone-based, fluids c_p and c_{sat} deviate considerably above the normal boiling point. Moreover, the critical point of these mixtures might be within or slightly above the range of the measurement. As c_p is relevant for the application of HTFs, DSC in closed crucibles might be applied for relative comparison but not for determining isobaric heat capacities.

3.1.6 Alternatives / more suitable methods

For determining real isobaric heat capacity calorimeters with open cells combined with pressure regulation of the HTF can be applied up to 300 °C. [6]

Isobaric heat capacity might also be concluded from isobaric enthalpy change measurements if suitably dimensioned. The latter experiment requires a pressurized flow of hot HTF that is cooled down to relevant temperatures with a reference fluid like water. Precise measurements of mass flows, temperatures and pressures are required as well as minimizing and calibrating heat losses from the measurement system. [7]

3.2 Thermal conductivity

The thermal conductivity of a material is a measure of its ability to conduct heat.

3.2.1 Relevance to CSP applications

Low thermal conductivity helps to reduce heat losses, when the HTF cools off on cold surfaces. This is a minor effect though. More importantly, the thermal conductivity affects the Prandtl number and the heat transfer coefficient. The Prandtl number describes the heat transport by momentum exchange in relation to the heat transport by heat conduction within a fluid. This means that a high thermal conductivity increases the heat transport (at the same flow velocity) and ensures a low temperature gradient in the laminar boundary layer (from the core temperature to the film temperature of the flow). In solar field operation, the heat transfer from the solar receiver to the heat transfer medium should always take place in turbulent flow and therefore a certain flow rate is required which is connected to the concept of thermal conductivity as this influences the minimum mass flow at which a parabolic trough can operate. A low thermal conductivity tends to lead to high surface temperatures of the absorber tube and thus to increased heat losses in the solar field. If the solar radiation is low, the design temperature cannot be reached at minimum mass flow. The collected energy has to be discarded as "under-specified". Energy, even if only small amounts, is lost. Thus, the thermal conductivity has a moderate influence on the performance of the system and thus on its economic efficiency.

3.2.2 Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   ASTM D2717-95 (2009), Standard Test Method for Thermal Conductivity of Liquids (Withdrawn 2018, no replacement)
- ASTM D7665   ASTM D2717-95 (2009)

3.2.3 Description of methods

ASTM 2717 is withdrawn without replacement. The only method which can be applied is ASTM D2896 – 19 (see section 3.2.6). It covers the use of a transient hot wire liquid thermal conductivity method and associated equipment for the determination of thermal conductivity, thermal diffusivity and volumetric heat capacity of aqueous engine coolants, non-aqueous engine coolants, and related fluids. The system is intended for use in a laboratory. The system directly measures thermal conductivity and thermal diffusivity without the requirement to input any additional properties. Volumetric heat capacity is calculated by dividing the thermal conductivity by the thermal diffusivity of the sample measured. This test method can be applied to any aqueous or non-aqueous engine coolant or related fluid with thermal conductivity in the range of 0.1 to 1.0 W/(m·K). The range of temperatures applicable to this test method is –20 to 100 °C.

3.2.4 Differences of methods

No comparison is presented.

3.2.5 Applicability of methods

ASTM D7896 – 19 can be applied to SiHTFs but just up to 100 °C.

3.2.6 Alternatives / more suitable methods

An alternative method for temperature up to 400 °C is presented under A.5 Thermal conductivity at operating conditions.

3.3 Latent heat

Latent heat is the energy (enthalpy) released or absorbed, by a body or a thermodynamic system, during a constant-temperature process — usually a phase transition.

3.3.1 Relevance to CSP applications

In normal operation, the latent heat is of no relevance as the HTF is always kept well above its solidification temperature, so that no phase change should take place. In case of pump trips, a high latent heat delays the freezing of the HTF and may give important time to recover operation (see section 1.15 Pour point). This is relevant, if the ambient temperature is below the solidification temperature of the HTF.

3.3.2 Summary of standards

- UNE 2016015   ASTM D 3418:2015, Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

3.3.3 Description of methods

ASTM D 3418: Thermal analysis provides a rapid method for measuring transitions due to morphological or chemical changes, in a polymer as it is heated/cooled through a specified temperature range. Change in specific heat capacity, heat flow and temperature values are determined for these transitions. Differential scanning calorimetry is used to assist in identifying specific polymers, polymer alloys, and certain polymer additives, which exhibit thermal transitions.

3.3.4 Differences of methods

There is no significant difference to be expected between ASTM D 3418 and ISO 11357-3:2018-07. Hence, one can conclude that "no alternative methods are required". Still the method is mentioned.

3.3.5 Applicability of methods

The method is applicable to SiHTFs. The expected phase changes of SiHTFs happen at temperatures below -50 °C and therefore it has no relevancy to CSP applications.

3.3.6 Alternatives / more suitable methods

Alternatively, ISO 11357-3:2018-07 (Plastics - Differential scanning calorimetry (DSC) - Part 3: Determination of temperature and enthalpy of melting and crystallization) can be applied to SiHTFs.

3.4 Specific resistivity at 20 °C

Electrical resistivity and its inverse, electrical conductivity, is a fundamental property of a material that quantifies how strongly it resists or conducts electric current. A low resistivity indicates a material that readily allows electric current.

3.4.1 Relevance to CSP applications

The parameter is relevant when a liquid is used as fire-resistant fluid in industrial equipment. The parameter can give an indication of the content of free ions and ion-forming particles, which could be relevant of liquids in service if there are present conductive contaminants. This parameter is of little relevance for CSP plants, but it could be required to establish safety, health and environmental practices and determine the applicability of regulatory limitations prior to use. This parameter is not relevant for heat transfer fluids used in parabolic troughs plants.

3.4.2 Summary of standards

- UNE 2016015   ASTM D1169-11, Standard test method for specific resistance (resistivity) of electrical insulating liquids
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

3.4.3 Description of methods

ASTM D 1169 provides a method that covers the determination of specific resistance (resistivity) applied to new electrical insulating liquids, as well as to liquids in service. This method covers a procedure for making referee tests with DC potential. The procedure defined involves measuring the resistance between the terminals of a test cell when a specified voltage is applied. Accurate measurement of the test temperature is relevant as resistivity is very sensitive to this property. The ASTM procedure allows different values in the voltage gradient to be applied.

3.4.4 Differences of methods

No comparison is presented.

3.4.5 Applicability of methods

The method is applicable to SiHTFs.

3.4.6 Alternatives / more suitable methods

No alternative methods are required.

4 Safety relevant parameters

4.1 Auto-ignition temperature:

The value for the auto-ignition temperature is needed to estimate the behavior of an HTF to ignite spontaneously in the presence of air. Thus, it serves to evaluate the risk level of using an HTF.

4.1.1 Relevance to CSP applications

The auto-ignition temperature of a medium is required to specify equipment suitable for the use in potentially explosive atmospheres (areas prone to leakages). The process related surface temperatures must either be limited to a safe value below the auto-ignition temperature, or other measures to prevent fire in accordance with the results of a hazard analysis have to be taken. Under certain circumstances, this can lead to considerable changes or call the process as a whole into question.

4.1.2 Summary of standards

- UNE 2016015   ASTM E659 – 15, Standard test method for auto-ignition temperature of chemicals
- DIN 51522   DIN 51794:2003-05, Testing of mineral oil hydrocarbons - Determination of ignition temperature
- DIN 51529   DIN 51522:1998-11
- GB 23971   SH/T0642, Petroleum and petro chemical products, determination of auto-ignition temperature
- ASTM D5372   ASTM E 659:2015, Test Method for auto-ignition Temperature of Chemicals
- ASTM D7665   ASTM E 659:2015

4.1.3 Description of methods

In every ignition test, a small portion of the flammable substance under test is introduced into an open conical flask. The flask is heated in an electrical furnace and it is observed whether ignition takes place at the prevailing temperature. Finally, any vaporizable components left in the flask are blown out by air. The lowest ignition temperature is determined from a series of ignition tests in which temperature and sample size are varied. The ignition temperature is then reported as the result of a selection from the lowest values obtained in a number of test series.

4.1.4 Differences of methods

- SH/T0642 and DIN 51794:2003-05 are equivalent.
- ASTM E659 is not compared to SH/T0642 and DIN 51794:2003-05.

4.1.5 Applicability of methods

In DIN 51794:2003-05 it is stated: "It should be borne in mind that the ignition temperature of flammable substances is not a physico-chemical constant but depends on the prevailing conditions in a particular case. The determination of the auto ignition temperature according to above mentioned methods (EN 14522, ASTM E659, SH/T0642) is suitable for silicone oils.

4.1.6 Alternatives / more suitable methods

EN 14522:2005: Determination of the auto-ignition temperature of gases and vapors. As the auto-ignition temperature is measured under conditions where the ambient temperature corresponds to the bulk temperature (of the HTF under testing) it has to be considered that

the auto-ignition temperature is not representing typical CSP conditions as the ambient temperature at CSP plants is much lower than the bulk temperature of the fluid.

A release test performed at Plataforma Solar de Almería (PSA) under realistic conditions [3] clearly shows that in case of a leakage at operation temperature (420 °C) there is no self-ignition of the leaking SiHTF.

4.2 Fire point

The fire point of an HTF is the lowest temperature at which the vapor of that HTF will continue to burn for at least 5 seconds after ignition by an open flame (defined ignition source).

4.2.1 Relevance to CSP applications

The fire point determines the susceptibility of the fluid to ignite and continue to burn from area sources, such as exposed electrical contacts, sparks, open flames, etc. A good design is to provide sloping of underlying surfaces such that any liquid release can drain away from process equipment. A HAZOP study can also lead to explosion hazard zones, where explosion-safe equipment must be used.

Therefore, special care is taken in CSP plants where leakage may occur, such as ball joints, as well as where the HTF may come into contact with the synthetic insulation.

4.2.2 Summary of standards

- UNE 2016015   ISO 2592:2000, Petroleum and related products - Determination of flash and fire points - Cleveland open cup method
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB23971-2009   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

4.2.3 Description of methods

At constant pressure the fire point is some Kelvin above the flashpoint. Above this temperature the flame continues burning even though the ignition source is removed.

4.2.4 Differences of methods

No comparison is presented.

4.2.5 Applicability of methods

The method is applicable to SiHTFs.

The setup of the open cup test is used, see section 4.4 Flash point, open cup.

4.2.6 Alternatives / more suitable methods

No alternative methods are required. The flashpoint is more significant than the fire point because it defines the lowest temperature where ignition can occur.

4.3 Flash point, closed cup

The flash point of an HTF is the lowest temperature at which its vapors (volatile substance) ignite if exposed to an ignition source. The flash point in the closed cup method is conducted inside a closed vessel which is not open to the outside atmosphere. The lid is sealed and the ignition source is introduced into the vessel itself, allowing for a closer approximation to real-life conditions (such as those found inside an HTF tank).

4.3.1 Relevance to CSP applications

The flash point determines the flammability classification of the liquid and thus the transport regulations (hazardous goods), as well as measures for occupational and plant safety.

The relevance for the operation of a CSP plant is limited, as the fluid is typically operated always significantly above the flash point. Accordingly, the system is designed for it, and all surfaces of the fluid are covered with inert gas (nitrogen). Based on the flash point, measures have to be considered e.g. definition of EX-areas.

4.3.2 Summary of standards

- UNE 2016015   ISO 2719:2016-11, Determination of flash point - Pensky-Martens closed cup method
- DIN 51522   DIN EN 22719:1993 (withdrawn) → ISO 2719:2016-11
- DIN 51529   DIN EN 22719:1993 (withdrawn) → ISO 2719:2016-11
- GB23971-2009   GB/T 261-2008: Determination of flash point – Pensky-Martens closed cup, ISO 2719:2016-11
- ASTM D5372   ASTM D93 – 19, Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
- ASTM D7665   ASTM D93 – 19

4.3.3 Description of methods

The test portion is placed into the test cup of a Pensky-Martens apparatus and heated to give a constant temperature increase with continuous stirring. An ignition source is directed through an opening in the test cup lid at regular temperature intervals with simultaneous interruption of stirring. The lowest temperature at which the application of the ignition source causes the vapor of the test portion to ignite and a flame propagate over the surface of the liquid is recorded as the flash point at the absolute barometric pressure. This temperature is corrected to standard atmospheric pressure using a specified formula.

4.3.4 Differences of methods

All methods are identical.

4.3.5 Applicability of methods

All standards are applicable to SiHTFs

4.3.6 Alternatives / more suitable methods

No alternative methods are required.

4.4 Flash point, open cup

The flash point of an HTF is the lowest temperature at which its vapors (volatile substance) ignite if given an ignition source. Measuring a flash point using an open cup method is conducted in a vessel which is exposed to the air outside. The temperature of the substance is gradually raised and an ignition source is passed over the top of it, until it reaches a point where it "flashes" and ignites.

4.4.1 Relevance to CSP applications

The flash point determines the flammability classification of a liquid and thus the transport regulations (hazardous goods), as well as measures for occupational and plant safety. The relevance for the operation of a CSP plant is limited, as the fluid is most of the time operated at temperatures significantly above the flash point. Accordingly, the system is designed for it, and all surfaces of the fluid are covered with inert gases (nitrogen). The open cup flash point is of relevance to indicate the fluids behavior in case of accidental leakages. At points where leakages potentially could occur in CSP, explosion (see 4.2 Fire point) hazard zones are foreseen. Therefore, no ignition source are present.

4.4.2 Summary of standards

- UNE 2016015   ISO 2592:2000, Petroleum and related products – Determination of flash and fire points – Cleveland open cup method
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB23971-2009   GB/T 3536-2008, Petroleum products, Determination of flash and fire points. Cleveland open cup method
  ISO 2592:2000 (withdrawn) → ISO 2592:2017
- ASTM D5372   ASTM D92-18, Standard Test Method for Flash and Fire Points by Cleveland Open Cup Tester
- ASTM D7665   ASTM D93 – 19

4.4.3 Description of methods

The test crucible is filled with the sample up to a certain level. The temperature of the sample may be increased rapidly (5 °C / min to 17 °C / min) at the beginning; as the flash point is approached, the sample is slowly heated further at a constant heating rate (5 °C / min to 6 °C / min). A small test flame is swung over the test crucible at defined temperature intervals.

The lowest temperature at which the test flame ignites the vapor phase above the surface of the liquid is considered the flash point at ambient air pressure. To determine the fire point, the test continues until the vapor phase above the sample is ignited by the test flame and burns for at least 5 s. The flash point and fire point determined at ambient air pressure are corrected to atmospheric normal pressure using an equation.

4.4.4 Differences of methods

All methods are identical.

4.4.5 Applicability of methods

All standards are applicable to SiHTFs.

4.4.6 Alternatives / more suitable methods

No alternative methods are required.

5 Compatibility

5.1 Biodegradation

Biodegradation is the breakdown of organic matter by microorganisms, such as bacteria and fungi.

5.1.1 Relevance to CSP

A high degree of biodegradability can alleviate the requirements for system tightness. If HTF leaks from its containing system, the environmental effects can be reduced if organisms decompose its components.

5.1.2 Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D7665   ASTM D5864 - 18, Standard Test Method for Determining Aerobic Aquatic Biodegradation of Lubricants or Their Components
- ASTM D5372   no method mentioned

5.1.3 Description of methods

ASTM D 5864 covers the determination of the degree of aerobic aquatic biodegradation of fully formulated lubricants or their components on exposure to an inoculum under laboratory conditions.

5.1.4 Differences of methods

No comparison is presented, as there is only a single method.

5.1.5 Applicability of methods

ASTM D5864 is applicable to SiHTFs.

5.1.6 Alternatives / more suitable methods

No alternative methods are required.

5.2   Corrosion

Corrosion is a natural process that converts a refined metal into a more chemically-stable form such as oxide, hydroxide, or sulfide. It is the gradual destruction of materials by chemical and/or electrochemical reaction with their environment (HTF).

5.2.1   Relevance to CSP applications

The corrosiveness of the heat transfer medium influences the choice of material in the solar field and HTF system and thus significantly influences costs. A high proportion of chromium and nickel as alloying elements leads to high corrosion resistance. Proportions of titanium or niobium are added to stabilize steels, i.e. to prevent a depletion of chromium due to diffusion effects at high temperatures. The alloying elements mentioned are very expensive in relation to carbon steel (base material) and therefore the material A106 is normally used. Heat transfer oils are typically non-corrosive, thus corrosion is of minor importance in parabolic trough applications.

5.2.2   Summary of standards

- UNE 206015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB23971   no method mentioned
- ASTM D5372   ASTM G4 - 01(2014): Standard Guide for Conducting Corrosion Tests in Field Applications
- ASTM D7665   ASTM G4 - 01(2014)

5.2.3   Description of methods

This guide (ASTM G4 - 01(2014)) covers procedures for conducting corrosion tests in plant equipment or systems under operating conditions to evaluate the corrosion resistance of engineering materials. It does not cover electrochemical methods for determining corrosion rates.

5.2.4   Differences of methods

No comparison is presented, as there is only a single method.

5.2.5   Applicability of methods

ASTM G4 – 01 is applicable to SiHTFs.

5.2.6   Alternatives / more suitable methods

No alternative methods are required.

5.3   Copper corrosion

The copper corrosion test examines the corrosiveness of the HTF in contact with copper.

5.3.1   Relevance to CSP applications

The corrosiveness of the heat transfer medium influences the choice of material in the solar field and HTF system and thus significantly influences material costs. Copper is a metal with high thermal conductivity and has a good initial corrosion behavior but cannot be used for this application because of their poor mechanical properties at high temperatures and pressures required for this solar application, hence steel is commonly used. Thus, copper corrosion is today of no relevance in the CSP industry.

5.3.2   Summary of standards

- UNE 206015   ISO 2160:1998, Petroleum products – corrosiveness to copper – copper strip test
- DIN 51522   ISO 2160:1998
- DIN 51529   no method mentioned
- GB23971   GB/T5096:2017, Test method for corrosiveness to copper from petroleum products by copper strip test
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

5.3.3   Description of methods

The copper strip test is a qualitative test method used to determine the level of corrosion of petroleum products. In the test, a polished copper strip is suspended in the heat transfer fluid and its effect observed. At elevated temperatures, a copper strip that has been polished is immersed in a sample of the heat transfer fluid, usually 30 ml. The strip is then removed and test for corrosion and a classification number is given according to a color table.

5.3.4   Differences of methods

GB/T5096:2017 and ISO 2160:1998 are equivalent.

5.3.5   Applicability of methods

Both methods are applicable to SiHTFs.

5.3.6   Alternatives / more suitable methods

No alternative methods are required.

5.4 Effect on rubber or elastomeric seals

The ability of rubber and rubber-like seals /components to withstand the effect of liquids (HTFs) is evaluated.

5.4.1 Relevance to CSP applications

This characteristic is relevant for different equipment, such as the draining equipment, personal safety equipment, filling equipment, etc. No power plant components have rubber parts in contact with hot HTF during regular operation. Hence the relevance is very limited.

5.4.2 Summary of standards

- UNE 206015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB23971-2009   no method mentioned
- ASTM D5372   ASTM D471 - 16a, Standard Test Method for Rubber Property – Effect of Liquids
- ASTM D7665   ASTM D471 - 16a

5.4.3 Description of methods

ASTM D471 covers the required procedures to evaluate the comparative ability of rubber and rubber-like compositions to withstand the effect of liquids. It is designed for testing: (1) specimens of vulcanized rubber cut from standard sheets (see Practice D3182), (2) specimens cut from fabric coated with vulcanized rubber (see Test Methods D751), or (3) finished articles of commerce (see Practice D3183). This test method includes the following:

- Change in mass (after immersion)
- Change in volume (after immersion)
- Dimensional-change method for water-insoluble liquids and mixed liquids
- Change in mass with liquid on one surface only
- Determining mass of soluble matter extracted by the liquid
- Change in tensile strength, elongation and hardness (after immersion)
- Change in breaking resistance, burst strength, tear strength and adhesion for coated fabrics

5.4.4 Differences of methods

No comparison is presented.

5.4.5 Applicability of methods

The method is applicable to SiHTFs.

5.4.6 Alternatives / more suitable methods

No alternative methods are required.

5.5 Foam

Foam is an object formed by trapping pockets of gas in liquids (HTFs).

5.5.1 Relevance to CSP applications

The aspect of foam formation is not yet finally evaluated. Foaming may lead to problems at open surfaces, i.e. the expansion and overflow vessels of a CSP plant. If foam builds up massively, HTF foam may be pressed into the venting system and enter the exhaust-gas treatment system, typically only designed for treating low boilers. When draining used HTF from CSP facilities shortly after depressurization, the fluid shows effervescence due to dissolved nitrogen. Typically, no foam is formed.

5.5.2 Summary of standards

- UNE 2016015   ISO 6247:1998, Petroleum products - Determination of foaming characteristics of lubricating oils
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

5.5.3 Description of methods

ISO 6247 specifies a method for the determination of the foaming characteristics of lubricant oils at specific moderate temperatures. It is applicable to lubricants which may or may not contain additives to modify or suppress the tendency to form stable foams. The rating used to describe the foam tendency and or/stability is empirical. The test portion, maintained at 24 °C, is blown with air at a constant rate for 5 minutes, then allowed to settle for 10 minutes. The volume of foam is measured at the end of the period. The test is repeated at a second portion at 93.5 °C, and then, after collapsing the foam, at 24 °C.

5.5.4 Differences of methods

No comparison is presented.

5.5.5 Applicability of methods

The standard is applicable to SiHTFs.

5.5.6 Alternatives / more suitable methods

No alternative methods are required.

Additional test methods

A.1   Hydrogen formation

Hydrogen content is the quantity of molecular hydrogen (H_2) contained in a material. It can be given on a volumetric or mass basis. During operation, especially near the maximum operation temperatures HTF components undergo very slow degradation reactions that produce hydrogen as a by-product.

Relevance to CSP applications

Heat collecting elements also called receiver tubes are located in the focal line of the PTC. They comprise a metal tube which conducts the HTF and a glass envelope-tube. The annulus between metal tube and glass envelope is evacuated. This vacuum functions as a thermal insulation which is transparent and thus can be penetrated by concentrated sunlight reflected from the parabolic shaped mirrors. The vacuum insulation is affected by molecular hydrogen inside the HTF system. During operation, especially near maximum temperatures HTF components undergo very slow degradation reactions that produce hydrogen as a by-product. Hydrogen permeates through the metal tube. To prevent any hydrogen accumulation getter materials are located inside the glass-envelope that bind the gas. Getter materials ensure low hydrogen pressures until specific hydrogen loading of the material is reached. Accumulation of gaseous hydrogen inside the envelope causes substantial degradation of the thermal performance of the receivers. This happens when the absorption capacity of the getter material is exhausted.

For maintaining full thermal performance of the receivers over the lifetime most HCE manufacturers specified hydrogen pressure limits. The concentration of hydrogen has to be kept below these limits during operation. Accordingly, the plant should be designed in an appropriate way to remove hydrogen sufficiently. For laying out removal of hydrogen the formation rate of hydrogen should be specified for an HTF under consideration. The long term-behavior of hydrogen formation rate should be addressed in tests on thermal stability according to 2.1 as data on the unused condition of an HTF can differ significantly from the conditions in use.

For controlling hydrogen limits during plant operation an analytical procedure is required.

Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

Summary of available methods

- ASTM D3612 – 02 (2017)   Standard Test Method for Analysis of Gases Dissolved in Electrical Insulating Oil by Gas Chromatography
- ASTM D831 / D831M - 12   Standard Test Method for Gas Content of Cable and Capacitor Oils
- ASTM D923 – 15   Standard Practices for Sampling Electrical Insulating Liquids
- ASTM D2759 – 00 (2017)   Standard Practice for Sampling Gas from a Transformer Under Positive Pressure

- ASTM F307 - 13   Standard Practice for Sampling Pressurized Gas for Gas Analysis

Description of methods

- ASTM D3612 describes gas chromatographical analysis of dissolved gases in insulating oil. The sample is handled at ambient pressure within the evaluation process.
- ASTM D831 describes an apparatus and a procedure for determining the total amount of dissolved gas in cable or transformer oil. The standard is not applicable without major adaptations to test pressurized samples with high gas content.
- ASTM D923 describes a practices for collecting samples of unused electrical insulating liquids from non-pressurized systems and storage containers.
- ASTM D2759 describes the collection of gases with some positive pressure from transformers. The procedure and materials (like plastic or glass) cannot be used for collecting safely and representatively samples from the HTF system of a CSP plant. Moreover, the procedure addresses only the collection of gaseous samples and not collecting oil samples with representative content of dissolved gas.
- ASTM F307 describes a procedure for obtaining a sample of pressurized gas for gas analysis from a system or component using an evacuated sample pressure vessel. Collecting HTF samples with representative amounts of dissolved gas is not addressed.

Differences of methods

- Some of the standards address collecting samples. While ASTM D923 concentrates on low pressure oil samples, ASTM D2759 and ASTM F307 are only intended for obtaining gaseous samples.
- ASTM D831 describes a procedure and technique for determining the total amount of dissolved gas for oil samples from transformers that contain up to 0,10 % dissolved gas (by volume at 760 mm and 0 °C).

Applicability of methods

The procedures and equipment listen under Summary of available methods are not applicable for testing HTFs at the pressures and temperatures relevant for CSP plants.

Alternatives / more suitable methods

For collecting and quantitatively analyzing representatively composed samples of HTF with gases a procedure described by Jung et al. can be used. [8]

The method is based on collecting HTF including all gases during operating the HTF system directly from the solar field e. g. from a drain valve. After cooling down to ambient temperature, the gas content in the pressurized cylinder is determined by expanding into a calibrated volume. The composition of the gas is analyzed via gas chromatography. Taking volume, mass and density of the HTF, pressure, temperature and composition into account the amount of gases like methane, ethane, carbon monoxide, carbon dioxide, oxygen and hydrogen per mass of HTF is obtained.

It was also proposed to locate hydrogen selective pressure measurement devices in the expansion vessel of the HTF system. [9] This approach requires to ensure equilibrium between the HTF of the expansion vessel and the solar field. Otherwise no representative readings can be expected.

A.2 Toxicity

Toxicity describes the adverse effects of a substance that result either from a single exposure or from multiple exposures in a short period of time.

Relevance to CSP applications

Toxicity of an HTF and its degradation products is relevant as large-scale installations with thousands of individual components cannot be considered hermetic. Leakages no matter if liquid or in gas phase can affect the health of exposed staff and the environment. [10] These undesirable consequences are associated with direct and indirect cost and furthermore negatively influence the image and acceptance of the technology. This feature is very important at all times of the process in both cases unused or HTF in use since this characteristic is considered from the transport of the fluid from the factory to site until after prolonged use.

Summary of standards

The supplier of chemicals in general is obliged to register and test the products according to the international standards. The Globally Harmonized System of Classification and Labelling of Chemicals (GHS) is an internationally agreed-upon standard managed by the United Nations that was set up to replace the assortment of hazardous material classification and labelling schemes previously used around the world. Core elements of the GHS include standardized hazard testing criteria, universal warning pictograms, and harmonized safety data sheets which provide users of dangerous goods with a host of information. The GHS system also regulates transport, storage and disposal of chemical substances and mixtures. Therefore, there is no need to define new standards how to test the toxicity of heat transfer fluids. National authorities must transfer the international regulations to national standards (in Europe this is the European parliament and council). GHS has been already adopted to varying degrees in nearly all major countries.

Applicable UN regulations:

- GLOBALLY HARMONIZED SYSTEM OF CLASSIFICATION AND LABELLING OF CHEMICALS (GHS), United Nations, ISBN No. 978- 92-1-117042-9

Applicable EU regulations:

- REGULATION (EC) No 1272/2008: on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006.
- REGULATION (EC) No 1907/2006: concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH), establishing a European Chemicals Agency, amending Directive 1999/45/EC and repealing Council Regulation (EEC) No 793/93 and Commission Regulation (EC) No 1488/94 as well as Council Directive 76/769/EEC and Commission Directives 91/155/EEC, 93/67/EEC, 93/105/EC and 2000/21/EC.

Description of methods

Various methods are defined in the regulations (mentioned above) as the methods depend on the specific substances. No specific method is mentioned here.

Differences of methods

No comparison is presented.

Applicability of methods

All methods are applicable to SiHTFs.

Alternatives / more suitable methods

No alternative methods are required.

A.3   Fouling

Fouling is the accumulation of unwanted material on solid surfaces to the detriment of function. In the context of transferring heat from or to an HTF, fouling represents an additional resistance to the heat transfer, thus lowering the amount of transferred heat per surface area and Kelvin. In other words, fouling is hindering heat transfer. Fouling depends on temperature, time and flow conditions. In general, there are three ways in which data on fouling may be obtained, namely from experience, from laboratory studies and by the use of plant data.

Relevance to CSP applications

Some organic HTFs are known to cause fouling. The fouling material which deposits onto the surfaces results from the degradation of the heat transfer fluids at high temperatures. Hence, applications making use these fluids usually incorporate a fouling factor of 0.18–0.35 (m^2K/kW) [5] leading to up to 30 % larger sizes of heat transferring equipment. Thus, fouling directly increases the cost of heat transferring equipment. Furthermore, fouling also affects the general need and the frequency of revisions of heat transferring equipment, which is directly associated with maintenance cost and downtime.

Fouling hinders the transfer of heat as its presence in the heat exchangers generates additional resistances to heat transfer decreasing the thermal efficiency and causing an increase of the pressure drop.

Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

Summary of available methods

- ASTM D4778 – 15   Standard Test Method for Determination of Corrosion and Fouling Tendency of Cooling Water Under Heat Transfer Conditions
- ISO 16784-2:2006   Corrosion of metals and alloys — Corrosion and fouling in industrial cooling water systems — Part 2: Evaluation of the performance of cooling water treatment programmes using a pilot-scale test rig
- ASTM D8002 - 15e1   Standard Test Method for Modified Fouling Index (MFI-0.45) of Water
- ASTM D 8253   Standard Test Method for Determination of the Asphaltene Solvency Properties of Bitumen, Crude Oil, Condensate and/or Related Products for the Purpose of Calculating Stability, Compatibility for Blending, Fouling, and Processibility (Manual Microscopy Method)

Description of methods

ASTM D4778 describes a method for easily determining the corrosion and fouling tendency of a particular water under heat transfer conditions. This will allow the evaluation of changes in the various system variables such as heat flux, flow velocity, metallurgy, cycles-of-concentration, and treatment schemes on heat exchanger performance.

ISO 16784-2:2006 Corrosion of metals and alloys — Corrosion and fouling in industrial cooling water systems — Part 2: Evaluation of the performance of cooling water treatment programs using a pilot-scale test rig.

ASTM D 8253 covers the use of a basic microscope to determine the asphaltene solvency properties of bitumen, crude oil, condensate or related products, or the combinations thereof. These properties can be used to calculate the solubility parameters required to calculate and predict asphaltene stability for blending purposes, refinery unit fouling, processibility, emulsion stabilization, pipeline and tank deposition, and equipment fouling. If the solubility properties are known for two products, then the compatibility of potential blend ratios can be predicted.

Differences of methods

No comparison is presented.

Applicability of methods

The applicability ASTM D 8253 to SiHTFs under CSP typical operation conditions has not been examined. The methods using water are not applicable to SiHTFs.

Alternatives / more suitable methods

There is no method available to determine the fouling of heat transfer oils. Fouling is a function of the system and the equipment used. Parameters which influence the fouling rate in a system include: the type of fluid, the type of heat exchanger, the temperatures, the velocities and the materials of construction. The actual fouling rate is different for each application.

There are numerous resources for fouling factor values in literature [5]. The most reliable source is from existing operating facilities. When production data is available, it can be used to calculate the actual fouling factors for the system. Together with the maintenance and cleaning history, this provides the best resource for selecting the fouling factors for a particular application.

A.4   Gas solubility

The solubility of gas is the amount of gas per HTF. This can be expressed with various coefficients (like according to Bunsen, Oswald or Henry).

Relevance to CSP applications

In HTF systems that are operated with blanket gas atmosphere some of the nitrogen used is also dissolved in the fluid depending on temperature, pressure and the characteristics of the fluid. The solubility of nitrogen in the HTF determines the required amount of nitrogen to build up a specific blanket pressure. This holds as the HTF in the solar field is exchanged with the HTF at the point of nitrogen supply. In such configuration the HTF of the solar field will be saturated with nitrogen. Thus, for building up specific nitrogen pressure in the HTF system, not only the amount of nitrogen in the gas phases of the vessels will be required but also the amount of nitrogen that is dissolved in the HTF inside the vessels and in the complete HTF system.

The solubility of gaseous degradation products like hydrogen, methane or others is also relevant as it determines the transfer into the gas phase of separator units. The latter normally is the atmosphere of vessels from which gas can be released out of the HTF system.

Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

Summary of available methods

- ASTM D3827-92   Standard test method for estimation of solubility of gases in petroleum and other organic liquids

Description of methods

ASTM D3827-92 covers a procedure for calculating equilibrium solubility of nitrogen, hydrogen, methane and several other gases in lubricants, fuels and solvents between -273.15 °C and 214.85 °C.

Differences of methods

No comparison is presented

Applicability of methods

ASTM D3827-92 is also intended for dimethyl and methyl phenyl silicone but limited to a maximum temperature of 214.85 °C.

Alternatives / more suitable methods

Grove et al. describe measurements of the solubilities of hydrogen, nitrogen, methane and other gases in terphenyl between 236,85 °C – 406,85 °C and 1.3 bar – 5.4 bar using a pressure cell. [11]

A.5 Thermal conductivity at operating conditions

The thermal conductivity of a material is a measure of its ability to conduct heat.

Relevance to CSP applications

See section 3.2. Thermal conductivity.

Summary of standards

- UNE 2016015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB 23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

Summary of available methods

- ASTM D7896-19:   Standard Test Method for Thermal Conductivity, Thermal Diffusivity, and Volumetric Heat Capacity of Engine Coolants and Related Fluids by Transient Hot Wire Liquid Thermal Conductivity Method.

Description of methods

ASTM D7896 covers the use of a transient hot wire liquid thermal conductivity method and associated equipment (the System) for the determination of thermal conductivity, thermal diffusivity and volumetric heat capacity of aqueous engine coolants, non-aqueous engine coolants, and related fluids. The System is intended for use in a laboratory. It directly measures thermal conductivity and thermal diffusivity without the requirement to input any additional properties. Volumetric heat capacity is calculated by dividing the thermal conductivity by the thermal diffusivity of the sample measured. This test method can be applied to any aqueous or non-aqueous engine coolants or related fluid with thermal conductivity in the range of 0.1 to 1.0 W/m·K. This test method excludes fluids that react with platinum.The range of temperatures applicable to this test method is –20 to 100 °C.

Differences of methods

No comparison is presented.

Applicability of methods

ASTM D7896 – 19 is not applicable because the maximum temperature is limited to 100 °C.

Alternatives / more suitable methods

The company flucon GmbH developed a sensor enabling an in-situ measurement of the thermal conductivity based on the "hot wire method" which can be operated up to 300 °C. [12] [3] The increase in measurement temperature is currently under testing.

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[3]Equipment for >400 °C is currently under testing and available soon.

A.6   Density at operating conditions

The density at operating conditions of an HTF is its mass per unit volume at operating conditions.

Relevance to CSP applications

The density at operation conditions is relevant, because the increase of density at elevated temperature affects the sizing of the expansion vessel and the overflow vessels.

At a certain mass flow in the system, the density of the heat transfer medium determines the volume flow. A high volume flow results in a high flow rate in the piping system. The flow velocity is used as a quadratic factor in the calculation of the pressure loss in the system. A high density thus has a positive effect on the pressure loss in the system. High pressure losses in the system lead to a high power consumption of the pumps, which reduces the net electricity production and thus the yield of a solar power plant.

If the density of the heat transfer medium changes strongly with the temperature, the piping system of the parabolic trough power plant requires a large compensating volume, which is associated with higher investment costs.

Summary of standards

- UNE 206015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

Summary of available methods

There is no existent standard method to determine the density at operating conditions.

Description of methods

No description is presented.

Differences of methods

No comparison is presented.

Applicability of methods

In contrast to organic fluids and due to the nature of multi-component silicone fluids the density is not only a function of temperature but also of the pressure applied. Because of this behavior in the high temperature region densities have to be measured for the full range of operation conditions (temperature and pressure). For these measurements no standard method exists.

Alternatives / more suitable methods

Densities of compressed liquids and supercritical fluids up to 250 °C (pressures up to 400 bar) can be experimentally determined with a thermostatted vibrating tube densimeter (e.g. DMA HPM Anton Paar, USP 841 - Specific gravity). Densities at higher temperatures can be measured via a high temperature and pressure static-synthetic cell which may be applied up to temperatures of 600 °C and pressures of 400 bar. For thermo-regulation the equilibrium cell should be mounted in a stainless-steel block which can be heated with an electrical heating unit inside a high temperature insulation cage.

A.7   Enthalpy change at operating conditions

The enthalpy change within two temperature levels of an HTF is the heat that it takes up or releases per unit mass.

Relevance to CSP applications

The enthalpy change of an HTF at operation conditions is relevant, because it determines the mass flow that is required to transfer the heat from the solar field to the power block or thermal storage within the designed thermal range of a system. The flow velocity is used as a quadratic factor in the calculation of the pressure loss in the system. A high enthalpy change thus has a positive effect on the pressure loss in the system. High pressure losses in the system lead to higher power consumption of the pumps, which reduces the net electricity production and thus the yield of a solar power plant.

Summary of standards

- UNE 206015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

Summary of available methods

There is no existent standard method to determine the enthalpy change at operating conditions.

Description of methods

No description is presented.

Differences of methods

No comparison is presented.

Applicability of standards

In contrast to organic fluids and due to the nature of multi-component silicone fluids the enthalpy change is not only a function of temperature but also of the pressure applied. Because of this behavior in the high temperature region enthalpy changes have to be measured for the full range of operation conditions (temperature and pressure). For these measurements no standard method exists.

Alternatives / more suitable methods

Isobaric enthalpy change measurements are possible with a pressurized flow of hot HTF that is cooled down to relevant temperatures with a reference fluid like water. Precise measurements of mass flows, temperatures and pressures are required as well as minimizing and calibrating heat losses from the measurement system. [7]

A.8   Kinematic viscosity at operating conditions

Viscosity can be conceptualized as quantifying the internal frictional force that arises between adjacent layers of fluid that are in relative motion. For instance, when a fluid is forced through a tube, it flows more quickly near the tube's axis than near its walls. In such a case, experiments show that some stress (such as a pressure difference between the two ends of the tube) is needed to sustain the flow through the tube. This is because a force is required to overcome the friction between the layers of the fluid which are in relative motion: the strength of this force is proportional to the viscosity.

Relevance to CSP applications

The kinematic viscosity is a relevant parameter for the evaluation of the flowing and pumping behavior of an HTF. It influences the pressure losses.

Summary of standards

- UNE 206015   no method mentioned
- DIN 51522   no method mentioned
- DIN 51529   no method mentioned
- GB23971   no method mentioned
- ASTM D5372   no method mentioned
- ASTM D7665   no method mentioned

Summary of available methods

There is no existent standard method to determine the kinematic viscosity at operating conditions.

Description of methods

No description is presented.

Differences of methods

No comparison is presented.

Applicability of standards

There are no standards available.

Alternatives / more suitable methods

Grzyll et al. report the determination of viscosities with hermetically closed capillary viscometers between 300 – 400 °C. [13] The procedure seems to be suitable for testing viscosity within the operational temperature range of CSP plants. Nevertheless, preparing and handling of the equipment can be considered as technically demanding due to the high temperatures and pressures that the glassware has to be exposed to.

A quartz viscometer has been developed recently by the company flucon GmbH. This device is operational at high temperatures and low viscosities that are relevant for the operating conditions of CSP plants. [12]

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