

## Operation of HELISOL®5A in a parabolic trough test loop

Christoph Hilgert<sup>a,\*</sup>, Christian Jung<sup>a</sup>, Kai Schickedanz<sup>b</sup>, Guillaume Saliou<sup>a</sup>,  
Anne Schlierbach<sup>a</sup>, Marc Röger<sup>a</sup>, Loreto Valenzuela<sup>c</sup>, Erich Schaffer<sup>b</sup>,  
Christoph Wasserfuhr<sup>d</sup>

<sup>a</sup> German Aerospace Center (DLR), Linder Höhe, 51147 Cologne, Germany

<sup>b</sup> Wacker Chemie AG, Gisela-Stein-Straße 1, 81671 Munich, Germany

<sup>c</sup> CIEMAT Plataforma Solar de Almería, Ctra. de Senés km 4.5, E-04200 Tabernas, Almería, Spain

<sup>d</sup> TÜV NORD Systems GmbH & Co. KG, Grosse Bahnstrasse 31, 22525 Hamburg, Germany

### ARTICLE INFO

#### Keywords:

CSP  
Silicone-based heat transfer fluid  
Prototype demonstration  
Parabolic trough test loop  
HELISOL®5A  
PDMS

### ABSTRACT

Polydimethylsiloxanes (PDMS / silicone oils) are commonly used as heat transfer fluids (HTF) at temperatures up to 400 °C in various industries, but the applications in large scale parabolic trough collector fields for thermal power generation has not been established due to higher prices compared to the commonly used mixture of diphenyl oxide and biphenyl. This paper describes the first system prototype demonstration in operational environment (loop scale) of a new silicone-based heat transfer fluid called HELISOL® 5A available at a competitive price level compared to the state of the art. Technical details of the parabolic trough test loop operated with HELISOL® 5A are presented. Solar operation at the state-of-the-art temperature of 400 °C for 150 h and at 425 °C for 480 h, demonstrated HELISOL® 5A's loop scale functionality in analogy to DIN 51528. The tolerance of HELISOL® 5A to temperatures above 425 °C was demonstrated by an operation period of 50 h at 450 °C. The degradation of the HTF is determined by the degree of cross-linking between macromolecules. Based on accompanying HTF analysis correlated with sample-based gas analysis, no measurable HTF degradation after 480 h at 425 °C was found. Only the stress test at 450 °C reveals an onset of moderate thermal degradation reaching 0.1 % mole fraction of cross-linked molecules. The heat transfer performance of HELISOL® 5A was confirmed at 400 °C by solar blind infrared absorber tube temperature measurements indicating a steady temperature increase of 0.2 K/m along the receiver tubes at 6.2 kg/s mass flow.

### 1. Introduction

Parabolic-trough collector (PTC) solar power plants using organic heat transfer fluids represent the majority of today's concentrating solar power (CSP) plants. The overall efficiency of such power plants depends largely on the solar field outlet temperature. Up to now, the maximum operating temperature is determined by the thermal stability of the used HTF. The state-of-the-art HTF, the eutectic mixture of diphenyl oxide (DPO) and biphenyl (BP) is limited to 400 °C [1]. HELISOL® 5A can be operated at 425 °C [2], thus enabling a more efficient energy conversion by means of the steam Rankine cycle. A techno-economic comparison incorporating further benefits and drawbacks of (this) silicone-based HTF, like the low pour point, improved occupational and environmental safety, its low hydrogen formation rate as well as the increased

operation pressure has been reported [3]. While silicone oils [4] have been used in the past as HTF in medium scale installations at temperatures up to 400 °C, e.g. PTC test loops at Plataforma Solar de Almería (PSA) [5 6] and elsewhere, the application on a significantly larger scale has not yet been realized. Against this background, the presented demonstration of the loop scale functionality and applicability of HELISOL® 5A in a relevant technical environment – including associated parabolic trough collector components at temperatures of 425 °C – represents the necessary step to reach the technological readiness level 7. Therefore, the objective of this study is the closely monitored loop scale demonstration of HELISOL® 5A at 425 °C for 480 h in a relevant technical environment. This paper presents the setup of the loop-scale demonstration using an existing PTC test loop with the implementation of four additional features: a retention line capable of holding a volume of 3.3 m<sup>3</sup> of HTF at the test loop outlet temperature, a cross-flow

\* Corresponding author.

E-mail addresses: [christoph.hilgert@dlr.de](mailto:christoph.hilgert@dlr.de) (C. Hilgert), [loreto.valenzuela@psa.es](mailto:loreto.valenzuela@psa.es) (L. Valenzuela), [erich.schaffer@wacker.com](mailto:erich.schaffer@wacker.com) (E. Schaffer), [cwasserfuhr@tuv-nord.de](mailto:cwasserfuhr@tuv-nord.de) (C. Wasserfuhr).

<https://doi.org/10.1016/j.solener.2025.113301>

Received 12 August 2024; Received in revised form 5 January 2025; Accepted 21 January 2025

Available online 7 March 2025

0038-092X/© 2025 The Authors. Published by Elsevier Ltd on behalf of International Solar Energy Society. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

Nomenclature	
<i>Abbreviations</i>	
ASTM	ASTM International, formerly known as American Society for Testing and Materials
BO	boil out
BOP	balance of plant
BP/DPO	biphenyl and diphenyl oxide eutectic mixture
CH <sub>4</sub>	methane
C <sub>2</sub> H <sub>6</sub>	ethane
CIEMAT	The Centre for Energy, Environmental and Technological Research / Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas
CSP	concentrating solar power
CO	carbon monoxide
CO <sub>2</sub>	carbon dioxide
DIN	The German Institute for Standardisation / Das Deutsche Institut für Normung
DLR	Deutsches Zentrum für Luft- und Raumfahrt
DSC	differential scanning calorimetry
EV	expansion vessel
GUM	guide to the expression of uncertainties in measurement
H <sub>2</sub>	hydrogen
HCE	heat collecting element
HELISOL <sup>®</sup> 5A	a linear, non-reactive polydimethylsiloxane produced by WACKER
HT	high temperature
HTF	heat transfer fluid
IR	infrared
N <sub>2</sub>	nitrogen
O <sub>2</sub>	oxygen
PDMS	polydimethylsiloxane
PROMETEO	parabolic test facility at Plataforma Solar de Almería
PSA	Plataforma Solar de Almería
PTC	parabolic trough collector
REPA	rotation and expansion performing assembly
RL	retention line
RTJ	ring type joints
SITEF	Silicone Fluid Test Facility – project title
<sup>29</sup> Si NMR spectroscopy	silicon nuclear magnetic resonance spectroscopy
SYLTHERM <sup>™</sup> 800	a linear, non-reactive polydimethylsiloxane produced by the company Dow
3MH	tris(trimethylsilyl)silane
T-Group	trifunctional unit / branched structures
TMS	tetramethylsilane
TRL	technical readiness level
SCHOTT PTR <sup>®</sup> 70	parabolic trough receiver with 70 mm diameter (formerly produced by the company Schott)
UV-Vis spectroscopy	ultraviolet and visible spectroscopy
<i>Symbols</i>	
<i>c</i>	specific heat capacity (J/kgK)
<i>D</i>	diameter (m)
<i>d<sub>i</sub></i>	inner diameter (m)
<i>f</i>	function (–)
<i>f<sub>1</sub></i>	empiric factor considering pipe curvature (–)
<i>f<sub>0</sub></i>	empiric factor considering tangential heat flow dissipation in pipe wall (–)
<i>L</i>	length (m)
<i>m</i>	mass (kg)
<i>P</i>	power (W)
<i>Pr</i>	Prandtl number (–)
<i>q̇</i>	heat flux (W/m <sup>2</sup> )
<i>Re</i>	Reynolds number (–)
<i>T</i>	temperature (°C)
<i>t</i>	time (s)
<i>V</i>	volume (m <sup>3</sup> )
<i>Greek symbols</i>	
<i>α</i>	heat transfer coefficient (W/m <sup>2</sup> K)
<i>Δ</i>	difference
<i>λ</i>	thermal conductivity (W/mK)
<i>σ</i>	coverage factor
<i>θ̂</i>	film HTF temperature (°C)
<i>θ̄</i>	bulk HTF temperature (°C)
<i>Subscripts</i>	
<i>ap</i>	approximated
<i>c</i>	combined
<i>i</i>	inner
<i>m</i>	mean
<i>o</i>	outer

sampling station to allow representative collection of HTF samples including associated volatile/dissolved gases for laboratory analysis, the development and implementation of appropriate flexible interconnections, and the introduction of appropriate heat collecting elements (HCE). These modifications were made in order to enable solar operation with silicone-based HTF up to 450 °C. Under “test program” three different operation phases are described in terms of the operation parameters. The section “results” is split in two sections. The HTF related findings section covers heat transfer, thermal degradation, and gas formation. The operational findings section covers technical procedures, leaks, or technical incidents. Finally, the technical applicability of HELISOL<sup>®</sup> 5A is discussed.

## 2. State-of-the-art HTF in parabolic-trough collector applications

Heat transfer fluid is a general term for substances used as heat transfer media. HTFs can be classified by their state of matter under normal operating conditions. In addition to the three standard states (gaseous, liquid, solid), phase change HTFs and supercritical fluids are

also possible. Suitable HTFs for CSP applications can be evaluated by their thermal and transport properties. Several authors have presented CSP specific reviews of HTF [7–9]. The state-of-the-art HTF currently used in most commercial PTC power plants is the eutectic mixture of biphenyl and diphenyl oxide [10]. Therminol<sup>®</sup> VP1 [1] Dowtherm<sup>™</sup> A [11] and Diphyl<sup>®</sup> [12] are the established brands in the market. Despite of the widespread use of BP/DPO, alternative HTFs are requested for CSP applications as the maximum operating temperature of 400 °C limits the efficiency of the plants. Furthermore, associated occupational and environmental risks are significant and publicly recognized [13]. With molten salt maximum temperatures of 550 °C are possible but so far, no salts with low melting points around or below ambient temperatures are available [14]. Consequently, extensive freeze protection measures have to be foreseen. Corrosion is also an issue for molten salts in contrast to organic HTFs. Water is considered as an alternative as well but its high vapor pressure increases the costs for storage and the two phase flow in direct steam generation systems enhances the control demand considerably [15]. The upper temperature limits of gaseous HTFs in CST systems are generally limited only by the materials of the receivers and the components that carry the HTF. Gases, however, have

the disadvantage of low heat transfer coefficients and densities [8]. Solidification temperatures below 0 °C and boiling temperatures above 1600 °C are possible for liquid metals and their alloys. Nevertheless, liquid metals are considered only for central receiver systems because of the operational risks. Heller [8] offers a very comprehensive overview of possible heat transfer media in CST and presents the corresponding technical data. In contrast to these alternatives, CSP plants with organic HTFs are easy to scale as proven technology is applied. Organic HTFs for high temperature applications are predominantly produced from aromatic hydrocarbons and aromatic ethers due to their high thermal stability in comparison to aliphatic compounds. The eutectic mixture of DPO and BP was patented in 1932 [16,17]. Since then no other fluid with carbon backbone has been described with higher stability despite intense investigation for the heat transfer in industrial heating or power generation [18]. Still, two significant disadvantages of current thermal oils have to be mentioned: their environmental and operational hazards due to the formation of the carcinogenic degradation product benzene and the increasing formation of hydrogen [3] over time. Hydrogen affects the vacuum annulus and thus the heat losses especially of the hot HCEs (towards the collector end) operating close to 400 °C if not removed meticulously [19]. BP/DPO has a melting point of 12 °C which may require freeze protection in some power plant locations. Silicone oils [4] in the context of CSP have been used as HTF in the past only in medium scale research CSP installations at temperatures up to 400 °C [5,6]. The application on a much larger scale was not economically feasible. Only with the introduction of a cheaper silicone-based HTF, further CSP-related research was initiated, starting with the evaluation of a suitable fluid composition [20], the techno-economical evaluation in the context of CSP [21], a guideline for the qualification of silicone-based HTF for CSP applications [22] and a specific operational risk assessments [23] were published. However, the system (silicone-based HTF in PTC) still needed to be demonstrated in a relevant environment at 425 °C, which is covered by this work. Fig. 1 and Table 1 show a comparison of the molecular structure and key technical data of the state-of-the-art HTF and HELISOL® 5A.

### 2.1. Heat transfer coefficient

The heat transfer coefficient  $\alpha_i$  at the inner diameter  $d_i$  of the absorber tube wall can be calculated according to section B6.2 of DIN 4754 [24] see Eq. (1).

$$\alpha_i = f_1 \frac{\lambda}{d_i} 0.012 Re^{0.87} Pr^{0.4} \quad (1)$$

$$10^4 < Re < 10^6$$

$$1.5 < Pr < 500$$

$$d_i \ll L$$

$$f_1 = 0.8$$

$f_1$  considers the influence of pipe curvature and the direction of heat flow.

The inner absorber tube wall film temperatures  $\hat{\vartheta}_i$  are calculated based on a heat flux  $\dot{q}$  of 58 kW/m<sup>2</sup> at the outer absorber tubes which represents the average calculated flux of the *IberTrough* collector. Film temperatures are calculated based on the increase in temperature  $\Delta\vartheta$  and the bulk HTF temperature  $\bar{\vartheta}$  according to section B6.1 of DIN 4754 [24] see Equation (2) and (3).

$$\hat{\vartheta}_i = \Delta\vartheta + \bar{\vartheta} \quad (2)$$

$$\Delta\vartheta = \frac{\dot{q}}{\alpha_i} \frac{d_o}{d_i} f_0 \quad (3)$$

$$f_0 = 0.9$$

$f_0$  considers the tangential heat flow dissipation in the pipe wall

### 3. A new silicone-based HTF for parabolic-trough collector applications: HELISOL® 5A

Another HTF – developed by Wacker Chemie AG and marketed under the name of HELISOL® 5A – is a linear, non-reactive polydimethylsiloxane with a kinematic viscosity of approx. 5 mm<sup>2</sup>/s (20 °C). It is a clear, odorless and colorless liquid when supplied. In contrast to organic HTFs as BP/DPO, the polymer backbone of all siloxanes (e.g., polydimethylsiloxanes) consists of alternating silicon and oxygen atoms (–Si – O – Si – O – Si –), see Fig. 1, right.

The overall hydrogen formation is significantly lower and does not increase exponentially over time [3]. Furthermore, the hydrogen formation is not affected by the presence of degradation products unlike the state-of-the-art HTF [21]. Its pour point < -55.0 °C does not require any type of freeze protection in typical CSP locations. And most importantly, under typical PTC conditions polydimethylsiloxanes can be operated at 425 °C loop outlet temperature during 25 years without any degradation induced HTF exchange see section 6.1.3.

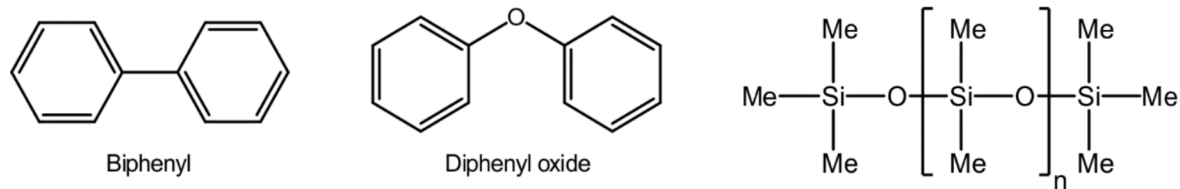
#### 3.1. Thermal degradation of silicone-based heat transfer fluids

Generally, heat transfer fluids change their chemical composition and consequently their properties during operation at temperatures above 250 °C. Thus, the properties of the “HTF in use” must rather be considered than those of the “unused” HTF. The method how to determine the degradation level of polydimethylsiloxane-based heat transfer fluids is given by the IEC Standard 62862-1-6:2024 [25]. In general, polymeric mixtures of linear siloxanes consisting of M- and D-units. The thermal stability of silicone-based HTF is assessed by determining the amount of T-units formed after exposure to elevated temperatures. An increasing number of T-groups may lead to branching points between individual polymer chains. Ultimately, if each polymer chain of a polydisperse mixture has at least two T-units, the system is theoretically completely cross-linked. Crosslinking in general will increase the mean molar mass of the polymer mixture and eventually lead to a long-term increase in viscosity. It is possible to find a correlation between the number of T-groups and the viscosity increase.

The maximum tolerable amount of T-groups depends on the fluid composition and has to be defined by the fluid manufacturer. This happens based on the tolerable maximum viscosity under given technical boundary conditions. For the application of HELISOL® 5A the correlation of T-groups and viscosity is not disclosed by the manufacture but the limit is set. In case of HELISOL® 5A 10 % T-groups are tolerable in the given context. For the purposes of analysis, the samples taken from PROMETEO were referenced against laboratory scale thermally aged samples using an autoclave in combination with high-temperature industrial ovens. To quantify the degree of degradation of the HELISOL® 5A samples the molar amount of T-groups was analyzed via <sup>29</sup>Si NMR spectroscopy, results see Table 8. This method differentiates the siloxane monomers and the M-structures, D-structures, and T-structures of the oligomers, which are the common unit structures for siloxanes.

#### Equilibration

At temperatures above 250 °C, silicone-based heat transfer fluids (polydimethylsiloxanes) undergo rearrangement reactions (equilibration) of their silicone-oxygen bonds when used as intended (under inert conditions). Such equilibration is a temperature-dependent equilibrium between linear and cyclic siloxanes depending on the working temperature the ratio between cyclic and linear components may vary [25].



**Fig. 1.** left: representation of the molecular structure of biphenyl (BP) and diphenyl oxide (DPO); right: chemical structure of a trimethylsilyloxy-encapped polydimethylsiloxane (PDMS).

**Table 1**  
Technical data of BP/DPO and HELISOL<sup>®</sup> 5A.

	BP/DPO	HELISOL <sup>®</sup> 5A
Working temp.:	60 °C – 400 °C	– 50 °C – 425 °C
Freezing point:	12 °C	– 55 °C
Vapor pressure (400 °C):	11 bar	15.8 bar
Self-ignition temp.:	599 °C	358 °C
Density (25 °C)	App. 1050 kg/m <sup>3</sup>	App. 920 kg/m <sup>3</sup>

#### 4. Experimental setup – PROMETEO parabolic troughs test loop

The so-called PROMETEO test loop consists of two parallel, east–west aligned parabolic-trough collectors named *IberTrough* with eight 12 m-long trough modules each, all parameters see Table 2. The *IberTrough* collector has an aperture width of 7334 mm and a focal length of 2172 mm. It is intended to use 90 mm receiver tubes but it was equipped with 70 mm diameter receiver tubes of the type “PTR 70”<sup>1</sup> [26] instead. The mirror shape deviations and the receiver tube positions of both collectors were measured using QFly [27] but not fed into a ray tracer for intercept factor calculations. Still, it became evident that the reduction in receiver tube diameter from 90 mm (design diameter) to 70 mm affected the intercept factor significantly. The total thermal power reaches about 600 kW at solar noon with DNI of 950 W/m<sup>2</sup> and an average HTF temperature in the solar field of 400 °C. Both collectors are connected in series while the corresponding balance of plant includes a pump, an expansion vessel, an air cooler and a drain tank see Fig. 2. PROMETEO was erected in 2010 by *Iberdrola Ingeniería y Sistemas* in collaboration with CIEMAT and operated for one year for the qualification of the *IberTrough* collector [28]. Later on, it was refurbished in 2016 in preparation of HTF demonstration activities during the SITEF project [29]. Said modifications were introduced to enable continuous operation with fluid temperatures up to 450 °C at the second / southern collector outlet. To cope with this requirement, new REPAs and 70 mm diameter HCEs were installed alongside with the exchange of the existing pipes made of steel grade ASTM A106 against ASTM A335 P11 piping. This was done solely at sections that potentially could contain HTF at temperatures above 400 °C. Due to the fact that the installed

**Table 2**  
Technical data of PROMETEO test loop located at Plataforma Solar de Almería.

Parabolic trough collector type	IberTrough
Collector aperture	7334 mm
Focal length	2172 mm
Collector module length	12 m
Solar field	2 collectors, 100 m length each
Thermal power (70 mm HCE / DNI 950 W/m <sup>2</sup> )	App. 600 kW
HTF volume	6.7 m <sup>3</sup> (expansion vessel excluded and retention line included).
Heat collecting elements	SCHOTT PTR <sup>®</sup> 70 (steel grade 1.4910)

*IberTrough* collector is a prototype, the east–west orientation of the collectors was chosen in the first place in order to facilitate collector performance testing / demonstration. When it comes to HTF testing, the accumulated time at the desired operating temperature is most relevant. Thus, PROMETEO comprises the less favorable east–west orientation, which results in significantly longer heat up ramps compared to a north–south orientation. This drawback was accepted due to the fact that no other facility was available by the time the activities were started.

##### 4.1. Mixing cooler

The PROMETEO test loop was originally set up to be operated at a maximum temperature of 400 °C, thus the components of the balance of plant (air cooler, pump, expansion vessel, valves sensors etc.) cannot be operated at significantly higher temperatures, while the loop outlet temperature can reach 450 °C. Consequently, the returning HTF from the collector outlet must be cooled down. This is done by mixing a fraction of colder HTF from the feed line side to the return line side. A static mixer makes sure that the two streams mix properly e.g. preventing temperature layering at low flows. Fig. 2 shows the simplified flow diagram of the PROMETEO installation indicating the mixing cooler.

##### 4.2. Heat collecting elements

In total 48 heat collecting elements (HCE) of the type SCHOTT PTR<sup>®</sup>70 made of austenitic stainless steel (1.4910) with 70 mm outer diameter and 2.2 mm wall thickness were installed to both *IberTrough* collectors. As the collector type was intended to use 90 mm diameter HCEs of 4100 mm length, a set of two adapters was mounted at each HCE support to guarantee a concentric HCE position within the original HCE clamps. Furthermore, the length of the SCHOTT PTR<sup>®</sup>70 receiver tubes is 4060 mm ± 2 mm and had to be adapted. Thus, a 40 mm piece of pipe of the identical material (same batch 1.4910) was welded to all HCEs. The expected increase in thermal HCE expansion from 323 mm (400 °C) to 371 mm (450 °C) in the axial direction does not exceed the range of movement of the HCE supports and was considered in the design of the REPA system.

##### 4.3. Rotation and expansion performing assemblies

A total number of four rotation and expansion performing assemblies (REPA) was manufactured and installed to the test loop, located at inlet and outlet of both parabolic-trough collectors. Each REPA was assembled of two 2.5” corrugated flexible hoses and a swivel joint designed to cope with an operating temperature of 450 °C and a max pressure of 41 bar. The design process was completed after a series of material and leak tests by a Senior Flexonics in-house fatigue test making use of the test rig #2 described by [30]. Fig. 3 shows one of the installed REPA systems at the northern collector inlet.

##### 4.4. Retention line

A retention line (RL) was integrated between the collector outlet and

<sup>1</sup> Material: 1.7335, DIN: 13 CrMo 4 4, ISO: 13CrMo4-5.

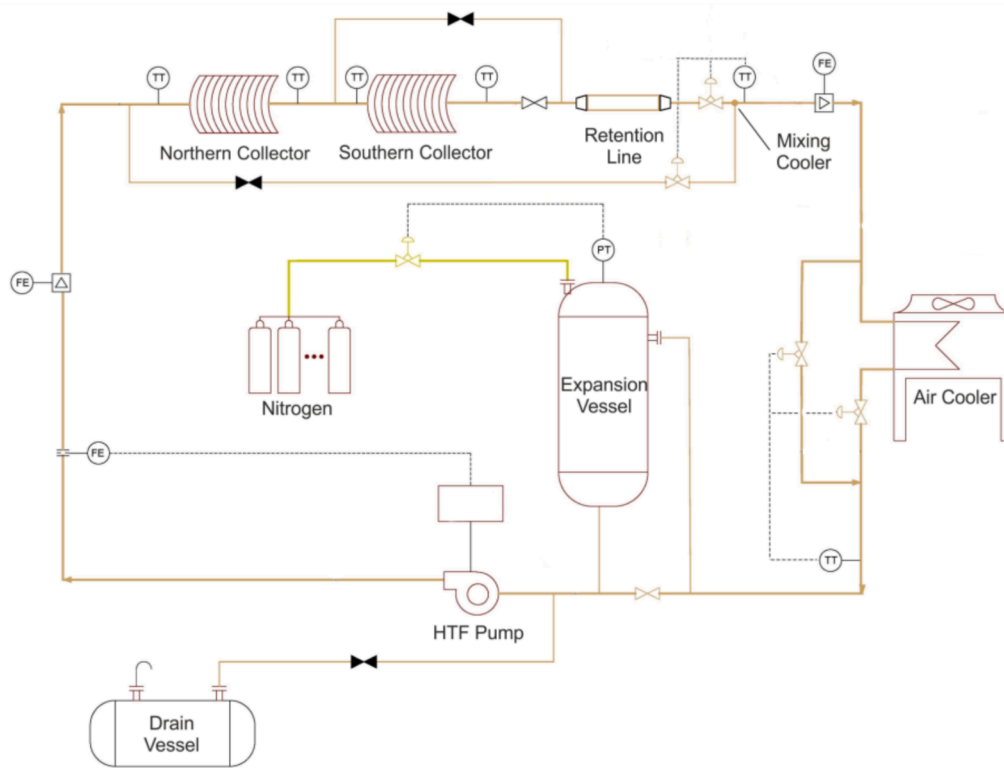


Fig. 2. Simplified flow diagram of the PROMETEO test facility indicating, the essential components (pump, solar collectors, retention line, mixing cooler, air cooler and expansion vessel) and the flow directions.



Fig. 3. Installed REPA (feedline hose, swivel joint and flex hose) at PROMETEO, thermal insulation not installed.

the air cooler inlet in order to increase the total amount of heated HTF at loop outlet temperature, see Fig. 2. The retention line is a 102 m-long 8" pipe replacing an initially installed 4" line in the same location. It has a volume of 3.3 m<sup>3</sup> while the entire facility comprises a total circulating HTF volume of 6.7 m<sup>3</sup> (expansion vessel excluded, retention line included). Depending on the mass flow HTF takes 4 to 9 min to travel through the RL while at the same time its temperature drops by about 2 K due to thermal losses. Said retention time represents the time of HTF flowing through a typical header pipe in a 50 MW<sub>e</sub> commercial PTC solar power plant.

#### 4.5. Sampling station / HTF sample extraction

In order to examine both the HTF and its reaction products, small but representative amounts of HTF (including the corresponding amounts of dissolved gases) in terms of sampling temperature, pressure and location inside the loop must be made available to be analyzed in a lab. Thus, a flow-through sampling station was implemented to take samples at operating temperature and pressure from the feed pipe connecting the solar collectors with the balance of plant (BOP). Fig. 4 presents the flow diagram and the picture of the sampling station.

For sample extraction, one or two sampling containers can be hydraulically connected in between the return and feedline linking the BOP with the solar collectors. Consequently, HTF can travel from the feedline (higher pressure) to the return line running through the sampling containers, hence, bypassing the collectors. As dirt and air have undesired influences in both, the HTF under operation and the sample, the sampling containers and the sampling lines are cleaned and later flushed with nitrogen, before running hot HTF through them. In order to take a sample, HTF is fed through the sampling containers for a defined period of time (15 min) in order to heat up the sampling equipment and remove potential nitrogen accumulations. Finally, all valves are closed in order of opposite flow direction. Once cooled down, the containers are dismantled, sealed with blind plugs and shipped to laboratory for gas and HTF degradation analysis. The determination of dissolved gases is done accordingly to [31]. Two improvements to the sampling station were identified in order to reduce possible systematic influences. Firstly, by installing a thermal insulation the temperature drop of the cross-flow can be reduced, possibly affecting the amount of dissolved gases in the HTF as the gas solubility is temperature dependent. Secondly, an 45° inclined orientation / mounting of the sampling containers helps to remove nitrogen bubbles in flow direction.

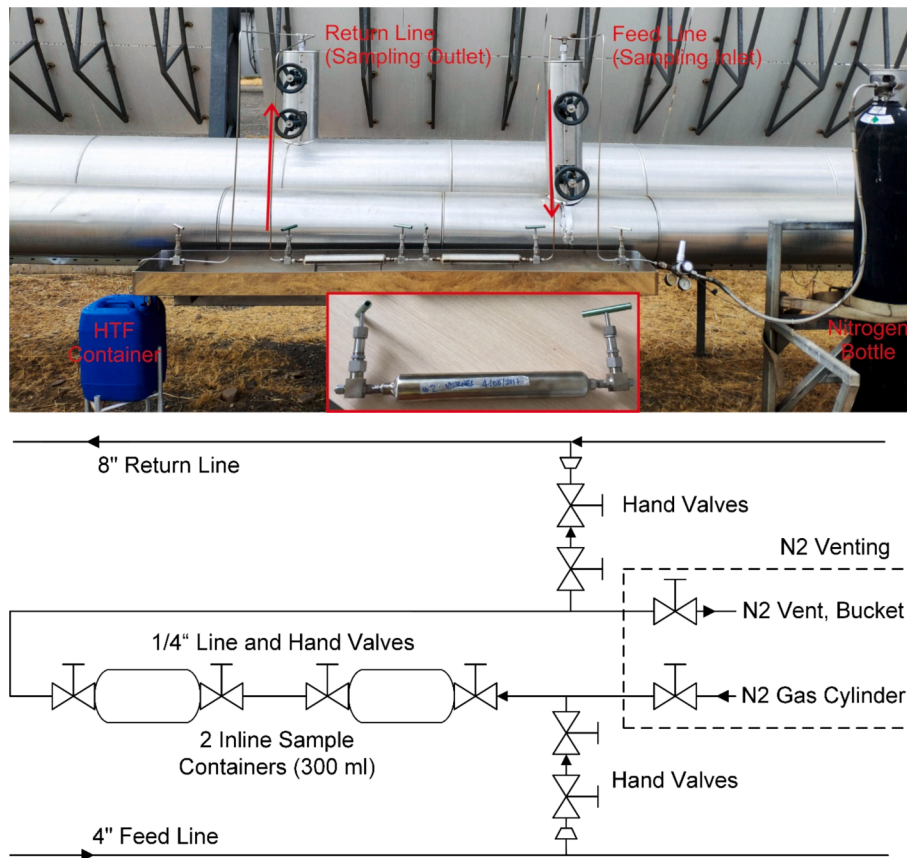


Fig. 4. Sampling device; top: picture of sampling station indicating components, close up of sampling container; bottom: piping diagram of cross-flow sampling station.

**Table 3**  
Operation parameters during typical operation day, June 7th 2016.

Action	$T_{BOP}$	$T_{in\ Loop}$	$T_{out\ Loop}$	$P_{EV}$	$P_{out\ Loop}$	EV level	$\dot{m}_{Loop}$
Operation start 9:00	48 °C	64 °C	60 °C	9.8 bar	11 bar	640 mm	7.7 kg/s
Reaching set temp. 12:37	390 °C	385 °C	400 °C	15.7 bar	17.2 bar	1609 mm	6.3 kg/s
Shut down 18:30	225 °C	255 °C	255 °C	12.8 bar	13.2 bar	1180 mm	0 kg/s

**5. Experimental proof of concept: Test plan**

The system prototype demonstration in operational environment (TRL 7) was achieved by operating the new silicone-based HTF HELLSOL® 5A in the PROMETEO test loop. Hence, the hydraulic circuit was drained, cleaned and filled with 6.3 m<sup>3</sup> of this new HTF. During testing, at a typical operation day, the HTF was circulated through both solar collectors in series and heated up to the desired loop outlet temperature. Once the loop outlet temperature ± 10 K was reached, the operation hours achieved in this temperature range were considered for the test. Meanwhile, the loop outlet temperature was held as steady as possible, mainly by adjusting the mass flow through the collectors. The test plan defined for the demonstration of the new HTF was composed of three

phases.

1. 150 h of operation at the state-of-the-art HTF solar field outlet temperature of 400 °C
2. 480 h of operation at the pre-defined nominal outlet temperature of 425 °C to achieve the proof of concept in analogy to DIN 51528
3. 50 h of operation at 450 °C to demonstrate the robustness of the system

During all three test periods, hot pressurized HTF samples were taken monthly to analyze the gas composition and the HTF degradation. Between phase 1 and 2 about 3 m<sup>3</sup> of new HTF were added to the circuit, as the retention line implementation increased the overall system

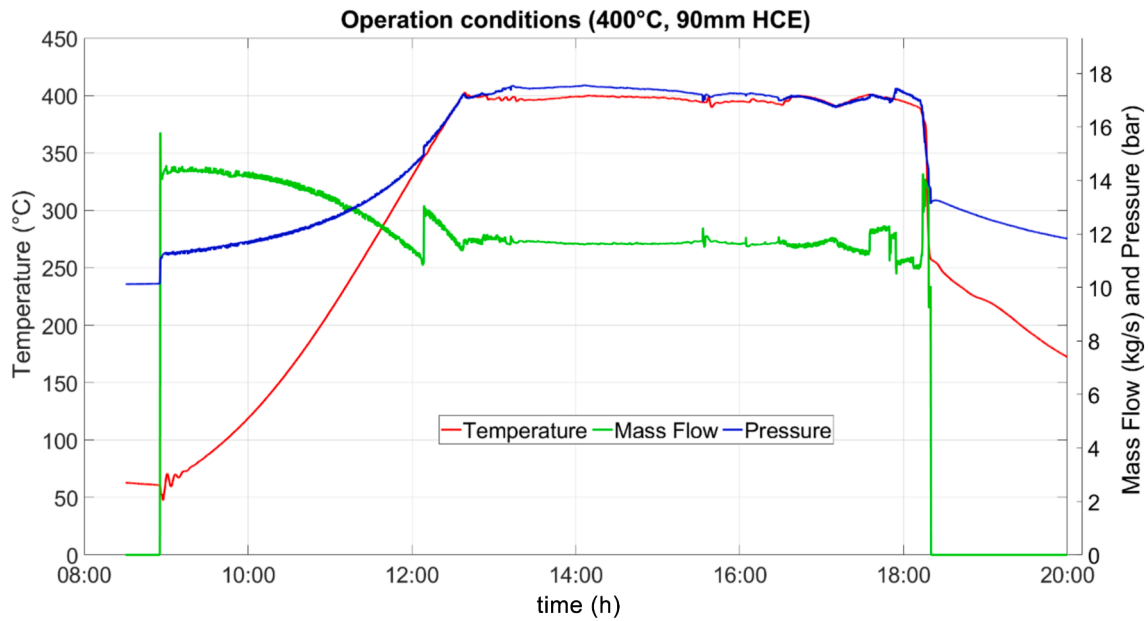


Fig. 5. Solar collector outlet HTF temperature, mass flow and pressure over time; heat up and operation at 400 °C. Date: June 7th 2016.

Table 4

Operation parameters during typical operation day, July 14th 2017.

Action	T <sub>BOP</sub>	T <sub>in Loop</sub>	T <sub>out Loop</sub>	P <sub>EV</sub>	P <sub>out Loop</sub>	EV level	m <sub>Loop</sub>
Operation start 9:10	45 °C	90 °C	70 °C	8.8 bar	11.2 bar	1000 mm	4 kg/s
Reaching set temp. 14:30	385 °C	382 °C	426 °C	17.8 bar	22.5 bar	2250 mm	5 kg/s
After shut down 17:51	220 °C	375 °C	375 °C	17.8 bar	18.2 bar	2050 mm	0 kg/s

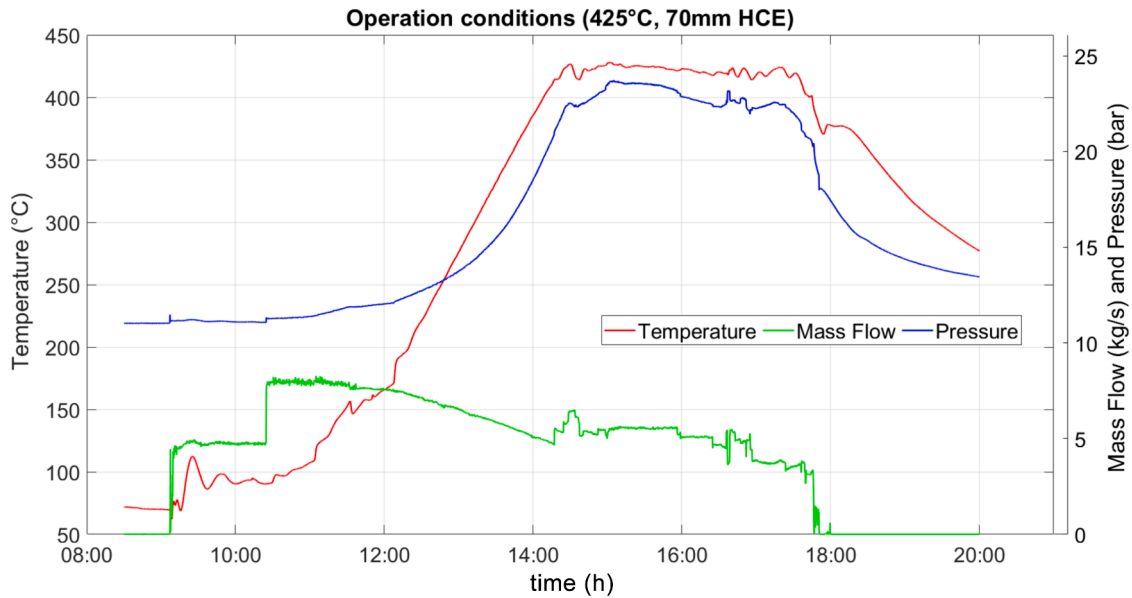


Fig. 6. Solar collector outlet HTF temperature, mass flow and pressure over time; heat up and operation at 425 °C. Date: July 14th 2017.

Table 5

Operation parameters during typical overheating operation day, July 26th 2018.

Action	T <sub>BOP</sub>	T <sub>in Loop</sub>	T <sub>out Loop</sub>	P <sub>EV</sub>	P <sub>out Loop</sub>	EV level	m <sub>Loop</sub>
Operation start 9:25	40 °C	62 °C	67 °C	10.5 bar	11 bar	550 mm	7.7 kg/s
Reaching set temp. 13:10	385 °C	387 °C	435 °C	22.2 bar	23.2 bar	1915 mm	5.8 kg/s
After shut down 16:12	243 °C	360 °C	375 °C	18.8 bar	19.3 bar	1690 mm	0 kg/s

volume.

### 5.1. Operation of HELISOL<sup>®</sup> 5A at PROMETEO at 400 °C

In order to confirm the performance of HELISOL<sup>®</sup> 5A in a typical parabolic trough loop environment, it was operated at state-of-the-art conditions at 400 °C for 150 h. The corresponding operation parameters are exemplarily presented in Table 3 and Fig. 5 below.

### 5.2. Operation of HELISOL<sup>®</sup> 5A at PROMETEO at 425 °C – “Proof of Concept”

In order to achieve the proof of concept in analogy to DIN 51528, the PROMETEO test loop was operated for 480 h at an average loop outlet temperature of 425 °C. During operation, the system pressure was varied stepwise from the upper limit towards the lower pressure limit of the facility. Accordingly, pressure values of 27 bar (425 °C) to 15 bar (425 °C) were measured at the collector outlet. Especially operation at 15 bar did not lead to a noticeable HTF phase change even though the vapor pressure is given with 19.5 bar at 425 °C. For typical operation parameters see Table 4 and Fig. 6 below.

### 5.3. Operation of HELISOL<sup>®</sup> 5A at PROMETEO at 450 °C – “Stress Test”

In order to demonstrate the robustness of HELISOL<sup>®</sup> 5A in this operational environment and to assess the corresponding ageing of the HTF when operated at temperatures significantly above the recommended operating temperature (overheated), the PROMETEO test loop was operated for 50 h at a set outlet temperature of 450 °C. For typical operation parameters see Table 5 and Fig. 7 below.

## 6. Results

### 6.1. HTF related findings

#### 6.1.1. Comparison of heat transfer coefficients and film temperatures

The comparison of the heat transfer coefficient  $\alpha_i$  at the inner diameter  $d_i$  of the absorber tube wall between HELISOL<sup>®</sup> 5A and the state-of-the-art HTF BP/DPO (Dowtherm<sup>™</sup> A) is calculated according to Equation (1) and presented in Table 6. For the sake of completeness, the comparison is presented up to 425 °C based on the information given in the technical data sheets of both fluids, although Dowtherm<sup>™</sup> A is not intended to be used at temperatures above 400 °C. An independent investigation of the heat transfer coefficient based on an infrared measurement is presented in section 6.2.4. and serves to verify the sufficiently high heat transfer for this specific application with HELISOL<sup>®</sup> 5A. The inner absorber tube wall film temperatures  $\hat{\vartheta}_i$  are calculated based on a heat flux  $\dot{q}$  of 58 kW/m<sup>2</sup> at the outer absorber tubes which represents the average calculated flux of the *IberTrough* collector. The film temperatures are calculated based on the temperature increase  $\Delta\vartheta$  and the bulk HTF temperature  $\bar{\vartheta}$  according to Equation (2) and (3). Table 6 shows the calculated film temperature results of both fluids at identical mass flow rates over temperatures from 300 to 425 °C.

A look at Table 6 shows that the heat transfer coefficients calculated for HELISOL<sup>®</sup> 5A with identical mass flows at 300 to 425 °C HTF temperature deviate between 3 % at 425 °C and 21 % at 350 °C relative to the state-of-the-art HTF. This can be explained with a significantly lower density of HELISOL<sup>®</sup> 5A while the lower viscosity of HELISOL<sup>®</sup> 5A at 400 °C and above compensates some of this. When looking at the calculated film temperatures, at the same temperature range, one sees that the calculated film temperatures for HELISOL<sup>®</sup> 5A are only 1 % to 2

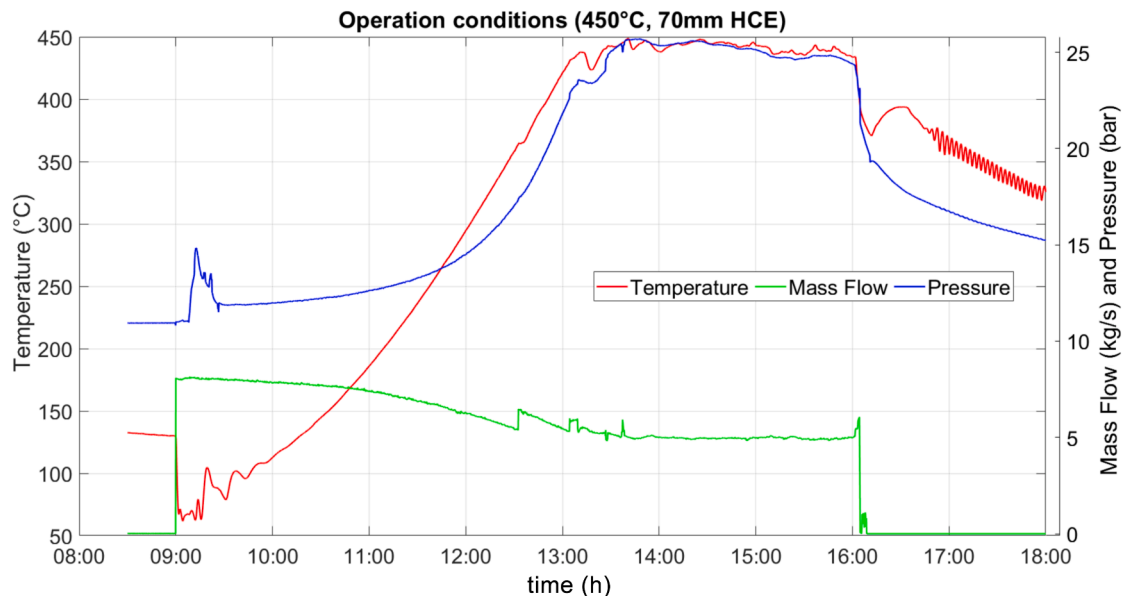


Fig. 7. Solar collector outlet HTF temperature, mass flow and pressure over time; heat up and operation at 450 °C. Date: July 26th 2018.

**Table 6**  
 Left: Comparison of heat capacity density and viscosity of HELISOL<sup>®</sup> 5A and BP/DPO. Right: Comparison of calculated heat transfer coefficients and film temperatures of HELISOL<sup>®</sup> 5A and Dowtherm A according to DIN 4754 at identical mass flow rates. Film temperatures are calculated based on a heat flux of 58 kW/m<sup>2</sup> as this value represents the highest fluxes on the 70 mm diameter HCE at the PROMETEO installation.

m <sup>3</sup>	kg/s	Temp. °C	Cp J/(kg K)	Density kg/m <sup>3</sup>	Visco. mPa s	Identical massflow							Average HTF velocity Vm								
						5	5.5	6	6.5	7	HTF Temp. °C	5	5.5	6	6.5	7	5	5.5	6	6.5	7
PDMS (HEL. 5A)	300	2,165	640	0.20	1,851	2,012	2,170	2,326	2,481	300	330	328	326	324	323	2.3	2.5	2.8	3.0	3.2	
	350	2,354	576	0.17	1,947	2,116	2,282	2,447	2,609	350	379	377	375	373	372	2.6	2.8	3.1	3.3	3.6	
	400	2,517	495	0.11	2,303	2,502	2,699	2,894	3,086	400	424	423	421	419	418	3.0	3.3	3.6	3.9	4.2	
	425	2,521	443	0.10	2,467	2,680	2,891	3,100	3,306	425	448	446	445	443	442	3.3	3.7	4.0	4.3	4.7	
m <sup>3</sup>	kg/s																				
	BP/DPO (Dowtherm A)	300	2,310	817	0.20	2,334	2,535	2,735	2,932	3,127	300	324	322	321	319	318	1.8	2.0	2.2	2.4	2.5
		350	2,455	760	0.17	2,444	2,656	2,864	3,071	3,276	350	373	371	370	368	367	1.9	2.1	2.3	2.5	2.7
		400	2,636	694	0.12	2,730	2,966	3,199	3,430	3,658	400	421	419	418	416	415	2.1	2.3	2.6	2.8	3.0
425		2,748	657	0.13	2,539	2,758	2,975	3,190	3,402	425	447	445	444	443	442	2.3	2.5	2.7	2.9	3.2	
m <sup>3</sup>	kg/s																				
	Deviation	300	6 %	22 %	1 %	21 %	300	2 %	28 %												
		350	4 %	24 %	2 %	20 %	350	1 %	32 %												
		400	4 %	29 %	3 %	16 %	400	1 %	40 %												
425		8 %	33 %	26 %	3 %	425	0 %	48 %													

% higher. At the same time HELISOL<sup>®</sup> 5A is much more tolerant to overheating and thus to higher film temperatures. This leads to the conclusion, that the heat transfer for both fluids is similar, at identical mass flows.

Film temperatures of both fluids at 5 - 7 kg/s and 300 to 425 °C HTF temperature deviate between 0 % and 2 % only marginally. BP/DPO's film temperature at 6 kg/s and 400 °C is calculated to have a 7 K margin to its maximum film temperature of 425 °C (Dowtherm<sup>TM</sup> A). HELISOL<sup>®</sup> 5A's film temperature at 6 kg/s and 425 °C has a margin of 5 K to its maximum film temperature of 450 °C. The maximum film temperature values differ for BP/DPO (Dowtherm<sup>TM</sup> A – 425 °C; Diphyll – 420 °C and Therminol<sup>®</sup> VP-1 – 430 °C) depending on the manufacturer [2,5,11,16].

When looking at Table 7 one can see, considering identical volume flow rates at 300 to 425 °C HTF temperature that the heat transfer coefficients calculated for HELISOL<sup>®</sup> 5A deviate between 31 % and 37 % relative to the state-of-the-art HTF. This can be explained by the considerably lower heat capacity and lower density of HELISOL<sup>®</sup> 5A compared to BP/DPO especially at elevated temperatures (see Table 6).

### 6.1.2. Gas Concentration in the HTF at PROMETEO

During all three operation phases, the HTF circuit of PROMETEO was kept closed in terms of unintended HTF and gas leakages in order to enable representative gas analysis and avoid contaminations of the environment. HTF and gas samples were taken as describes above in section 4.5. The results of the gas analyses in terms of decomposition-related gases (hydrogen, methane and ethane) together with nitrogen are presented in Fig. 8 while Fig. 9 presents oxygen together with carbon monoxide and carbon dioxide being oxidation products. Furthermore, the concentrations of the tri- and tetramethylsilane are presented, both substances are present in the gas phase due to the low boiling points of (26 °C TMH and 83 °C 3MH). After 144 h of operation at 425 °C, the PROMETEO system was decompressed to discharge gaseous decomposition products. Apart from nitrogen, methane was determined in particular, which can be expected according to the formation kinetics. Initially, larger concentrations of carbon monoxide, carbon dioxide and oxygen were also present. The last sample examined again shows slightly increased values for oxygen and carbon monoxide. Overall, the analysis results show that the initially higher concentrations of decomposition-related gases, such as methane in particular, are not reached again in a short time after removal. The gas tightness of various flange connections at the top of the expansion vessel was improved after 350 operating hours, which helps to explain the significantly higher hydrogen concentrations observed thereafter.

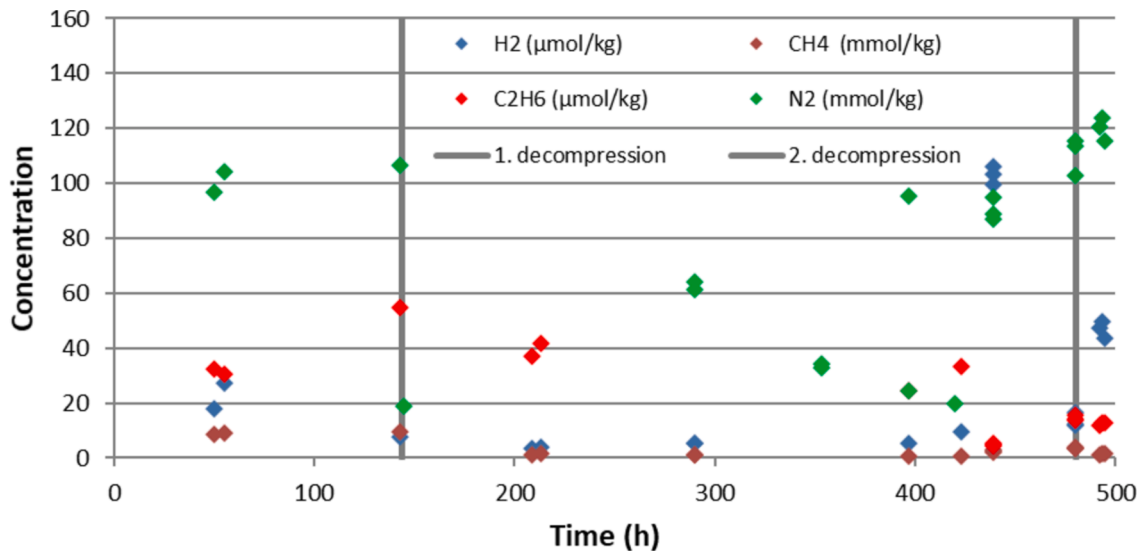
### 6.1.3. HTF analysis

HELISOL<sup>®</sup> 5A was sampled by DLR and investigated by WACKER over the operating period. The results are summarized in Table 8. Over the operating period of 373 h, including 150 h at temperatures above 390 °C, the viscosity dropped from the original 4.6 mPa-s to 3.7 mPa-s. The equilibration of the silicone oil led to a reduction of the flash point from the original 130 °C to 22 °C, and the water content also fell from 82 ppm to 66 ppm. After retrofitting the plant, app. 3 tons of new HELISOL<sup>®</sup> 5A were added, which is clearly reflected in the increase in both viscosity to 4.1 mPa-s and the flash point to 65 °C. The boil-out process can be recognized in a decrease in viscosity, lowering flash point and decrease in water content. Subsequent operation at 425 °C did not lead to a measurable increase in T-groups / degradation of the silicone oil. Viscosity and flash point dropped to an equilibrium level of 3.899 mPa-s and 38.0 °C respectively, and the water content dropped to below 70 ppm. The elemental analysis showed no detectable traces of metals in the silicone oil over the entire operating period. Operation at 425 °C was followed by an overheating scenario in which the HELISOL<sup>®</sup> 5A in the plant was heated to 450 °C, over a total period of 52 h. During this time three samples were taken. Overall, the overheating to 450 °C can be considered as largely unremarkable. The flash point dropped from 38.0

**Table 7**

Comparison of calculated heat transfer coefficients and film temperatures of HELISOL<sup>®</sup> 5A and Dowtherm<sup>™</sup> A according to DIN 4754 for identical volume flow rates. Film temperatures are calculated based on a heat flux of 58 kW/m<sup>2</sup> on a 70 mm diameter HCE.

V m <sup>3</sup> /s	Temp. °C	identical volume flow															
		Heat Transfer Coefficient α W/(m <sup>2</sup> K)					HTF Temp. °C	Film Temperature θ °C					Mass Flow m' kg/s				
		20	25	30	35	40		20	25	30	35	40	20	25	30	35	40
PDMS (HEL. 5A)	300	1,377	1,672	1,959	2,241	2,517	300	341	334	329	325	322	3.6	4.4	5.3	6.2	7.1
	350	1,322	1,605	1,881	2,150	2,415	350	393	385	380	376	373	3.2	4.0	4.8	5.6	6.4
	400	1,368	1,661	1,947	2,226	2,501	400	441	434	429	425	423	2.7	3.4	4.1	4.8	5.5
	425	1,332	1,617	1,895	2,167	2,435	425	467	460	455	451	448	2.5	3.1	3.7	4.3	4.9
V m <sup>3</sup> /s		20	25	30	35	40		20	25	30	35	40	20	25	30	35	40
BP/DPO (Dowtherm <sup>™</sup> A)	300	2,146	2,605	3,053	3,492	3,922	300	326	322	318	316	314	4.5	5.7	6.8	7.9	9.1
	350	2,109	2,561	3,001	3,432	3,855	350	377	372	369	366	365	4.2	5.3	6.3	7.4	8.4
	400	2,176	2,643	3,097	3,541	3,977	400	426	421	418	416	414	3.9	4.8	5.8	6.7	7.7
	425	1,930	2,344	2,746	3,141	3,527	425	454	449	446	443	441	3.6	4.6	5.5	6.4	7.3
V m <sup>3</sup> /s		20	25	30	35	40		20	25	30	35	40	20	25	30	35	40
Deviation	300			36 %			300			3 %					22 %		
	350			37 %			350			3 %					24 %		
	400			37 %			400			3 %					29 %		
	425			31 %			425			2 %					33 %		



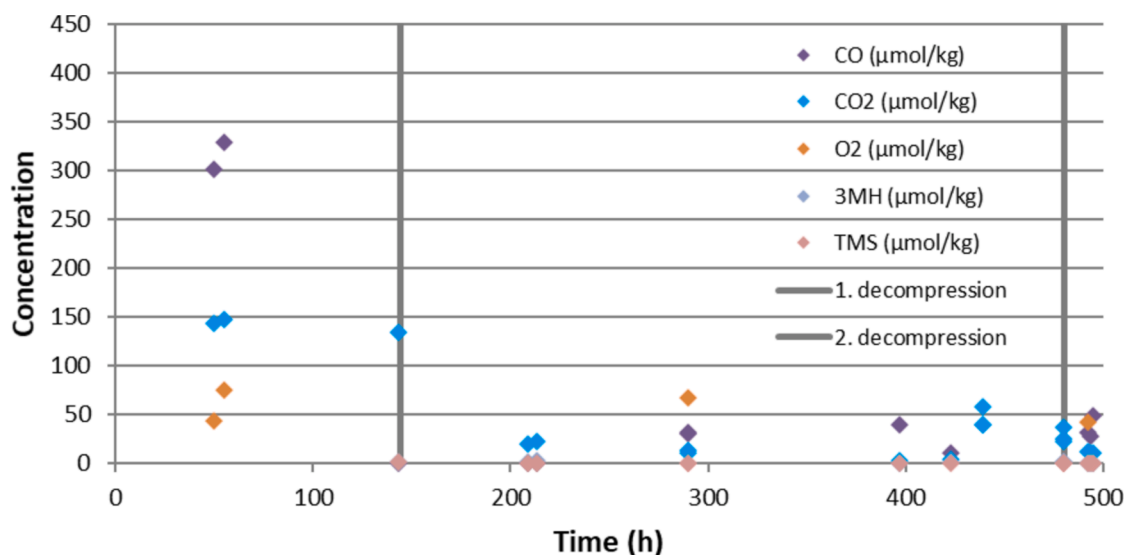
**Fig. 8.** Concentrations of hydrogen (H<sub>2</sub>), methane (CH<sub>4</sub>), ethane (C<sub>2</sub>H<sub>6</sub>) and nitrogen (N<sub>2</sub>) in HELISOL<sup>®</sup> 5A during 480 operation hours at 425 °C in PROMETEO.

°C to 28.0 °C, the viscosity of 3.7 mPa-s was only slightly lower than the previous operation at 425 °C. The water content dropped further to below 30 ppm, and elemental analysis still showed no detectable traces of common alloying metals. A degradation of the silicone oil due to the targeted overheating at 450 °C could not be detected for the test period by means of the T-group method.

**6.2. Operation related findings**

**6.2.1. Clean-out, filling HELISOL<sup>®</sup> 5A and heat-up**

The PROMETEO test facility was originally filled and operated with SYLTHERM<sup>™</sup> 800 [4]. This heat transfer medium was drained. Once all drain locations where opened and emptied, flushing with purified compressed air was applied to drive residue HTF towards drain locations where it was contained. When exchanging the HTF content of a circuit



**Fig. 9.** Concentrations of carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>), oxygen (O<sub>2</sub>), tri- and tetramethylsilane (3MH and TMS) in HELISOL<sup>®</sup> 5A during 480 operation hours at 425 °C in PROMETEO.

with the given extent including app. 500 m of tubing and about 7 m<sup>3</sup> of wetted volume the necessity of complete removal or cleaning correlates to significant effort. The option of flushing some kind of cleaning or intermediate fluid through the installation was excluded. Instead, the tolerance of HELISOL<sup>®</sup> 5A towards impurities or fractions of SYLTHERM<sup>™</sup> 800 was examined prior to the HTF exchange. See [section 6.2.2](#). Filling was performed using a mobile electric pump connecting its suction side to an interim bulk container filled with fresh HTF and its discharge side to the drain line at the lowest point of the installation. While filling, the vent of the expansion vessel was open to the environment in order to make sure contained gases could escape. Once gas suction of the HTF circulation pump could be excluded, HTF circulation through the expansion vessel served to expel gas bubbles from the pipe system. Finally, all vents were purged. In preparation of the boil-out, nitrogen was filled into the app. 7 m<sup>3</sup> gas space of the expansion vessel (EV) reaching a pressure of 2 bar before venting it to the environment. This procedure was repeated three times. Afterwards, HTF circulation through the EV was established and periodic solar operation begun maintaining an HTF temperature inside the EV at about 140 °C during 3 h. Parallel to the HTF circulation a steady flow of nitrogen was led through the EV escaping to the environment together with the contained moisture. Meanwhile, its pressure was kept measurable above ambient pressure. Eventually, the EV was pressurized with nitrogen to a level of 10 bar before the HTF temperature was gradually increasing to 425 °C during three days of operation while monitoring the entire installation closely.

#### 6.2.2. Determination of SYLTHERM<sup>™</sup> 800 concentration in HELISOL<sup>®</sup> 5A

After filling the loop with colorless HELISOL<sup>®</sup> 5A the mass fraction of the remaining colored SYLTHERM<sup>™</sup> 800 was determined by UV-Vis spectrometry. This comparative study was done with samples taken

from the newly filled PROMETEO and with non-contaminated HELISOL<sup>®</sup> 5A.

For the doping study, up to 5 % (m/m) of the SYLTHERM<sup>™</sup> 800 taken from PROMETEO was added to fresh HELISOL<sup>®</sup> 5A and the radiation absorption of the mixtures as well as of the pure substances from 190 nm was investigated with a UV-Vis spectrometer in 10 mm quartz cuvettes. According to the comparison of the radiation extinctions, a content of 1 % to 2 % (m/m) SYLTHERM<sup>™</sup> 800 was contained in the HELISOL<sup>®</sup> 5A after HTF exchange in the PROMETEO test facility (see [Fig. 10](#)).

#### 6.2.3. Infrared absorber temperature measurements between 400 °C and 425 °C

The aim of this measurement was to investigate whether a significant temperature difference (more than 10 K) can be measured between the surface of the coated steel absorber and the HTF bulk temperature inside the tube. This is especially relevant at the exit of the southern collector, where the fluid temperature reaches about 425 °C. A “solar blind” infrared (IR) camera setup was used to observe the surface temperature of 24 coated steel absorber tubes at different locations along the northern and southern collector during solar operation. The mass flow inside the HCEs was 6.2 kg/s during the measurements. The distance between measurement points was set to 8 m, hence every second tube was measured. Initially, a calibration with one defocused collector operating steady state at 400 °C was performed, whereby the camera parameters were adapted to the specific tube surface properties with respect to their emission values. The analysis of the temperature rise along the receiver tubes in flow direction makes it possible to check if there is any significant discontinuity or “jump” along the collector, indicating a possible change of thermodynamic properties in the heat transfer fluid itself affecting the heat transfer conditions. For both

**Table 8**  
 Overview of the investigated chemical/physical data of the obtained HELISOL® 5A samples during all three operation periods. a: Measurements with Strabinger viscometer from Anton Paar, b: Measurements with Seta-Flash Series 3 flash point meter from Stanhope-Seta, c: Water determination according to Karl-Fischer, d: Elemental analysis using ICP with a Perkin Elmer Optima 7300 DV.

Date of sampling	Operation condition	comment	Total operation time		Viscosity <sup>a</sup> at 25 °C [mPas]	Flashpoint <sup>b</sup> [°C]	Water content <sup>c</sup>			Elemental analysis metals <sup>d</sup>			
			[h]	Operation time > 390 °C [h]			Iron [ppm]	Chromium [ppm]	Copper [ppm]	Vanadium [ppm]	Tungsten [ppm]	T-group [mol-%]	
Mint condition			0	0	4.615	130.0	< 10	< 10	< 10	< 10	< 10	< 10	asd
27.05.2016	Operation at 400 °C		135	38	3.952	44.5	< 10	< 10	< 10	< 10	< 10	< 10	0
10.06.2016			177	57	3.854	44.5	< 10	< 10	< 10	< 10	< 10	< 10	0
28.06.2016			240	88	4.159	46.0	< 10	< 10	< 10	< 10	< 10	< 10	0
07.07.2016			281	108	3.809	22.0	< 10	< 10	< 10	< 10	< 10	< 10	0
28.07.2016			373	150	3.744	22.0	< 10	< 10	< 10	< 10	< 10	< 10	0
08./2016 to 03/2017: Refurbishment													
05.04.2017	Addition of fresh HELISOL® 5A	boil out	boil out		4.138	65.0	< 10	< 10	< 10	< 10	< 10	< 10	0
24.05.2017		before BO			4.105	67.0	< 10	< 10	< 10	< 10	< 10	< 10	0
06.07.2017		before BO			3.999	45.5	< 10	< 10	< 10	< 10	< 10	< 10	0
03.08.2017	Operation at 425 °C		157	50	3.756	35.5	< 10	< 10	< 10	< 10	< 10	< 10	0
03.08.2017			n.a.	55	3.666	28.0	< 10	< 10	< 10	< 10	< 10	< 10	0
11.09.2017			242	92	3.710	21.0	< 10	< 10	< 10	< 10	< 10	< 10	0
13.12.2017			598	290	3.701	37.5	< 10	< 10	< 10	< 10	< 10	< 10	0
24.01.2018			741	353	3.856	39.5	< 10	< 10	< 10	< 10	< 10	< 10	0
09.03.2018			861	397	3.899	38.0	< 10	< 10	< 10	< 10	< 10	< 10	0
Overheating													
02.08.2018	Operation at 450 °C		1192	23	3.726	39.0	< 10	< 10	< 10	< 10	< 10	< 10	0
08.08.2018			1220	31	3.753	38.0	< 10	< 10	< 10	< 10	< 10	< 10	0.1
07.09.2018			1258	41	3.777	28.0	< 10	< 10	< 10	< 10	< 10	< 10	0.1
04.10.2018			1275	52	3.750	28.0	< 10	< 10	< 10	< 10	< 10	< 10	0.1

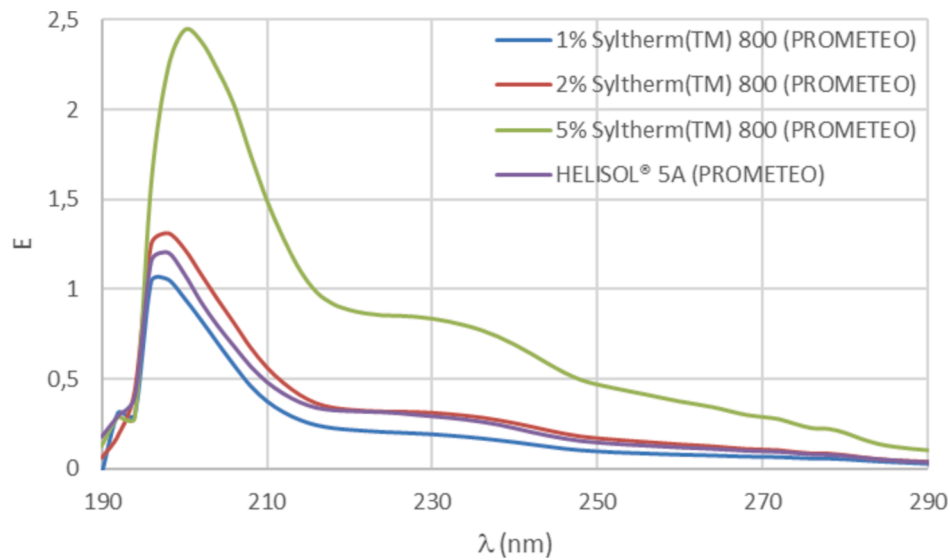


Fig. 10. Spectral extinction HELISOL<sup>®</sup> 5A sample from PROMETEO after HTF exchange and HELISOL<sup>®</sup> 5A with different concentrations of SYLTherm<sup>™</sup> 800 (sample from PROMETEO). Measurements of the samples filtered with Teflon (0.2  $\mu\text{m}$ ) against HELISOL<sup>®</sup> 5A.

collectors, the incremental change in the interpolated temperature values, between the Pt100 readings, are calculated and plotted in Fig. 11, blue dots. The red squares represent the surface temperatures measured with the IR camera.

For both collectors, the incremental temperature rise between adjacent receiver tubes remains stable. For the northern collector the average increment is  $(0.229 \pm 0.004)$  K/m. For the southern collector the average increment is  $(0.210 \pm 0.007)$  K/m. Thus, no significant discontinuity or “jump” in temperature is observed for the above described setup.

#### 6.2.4. Incidents and repairs

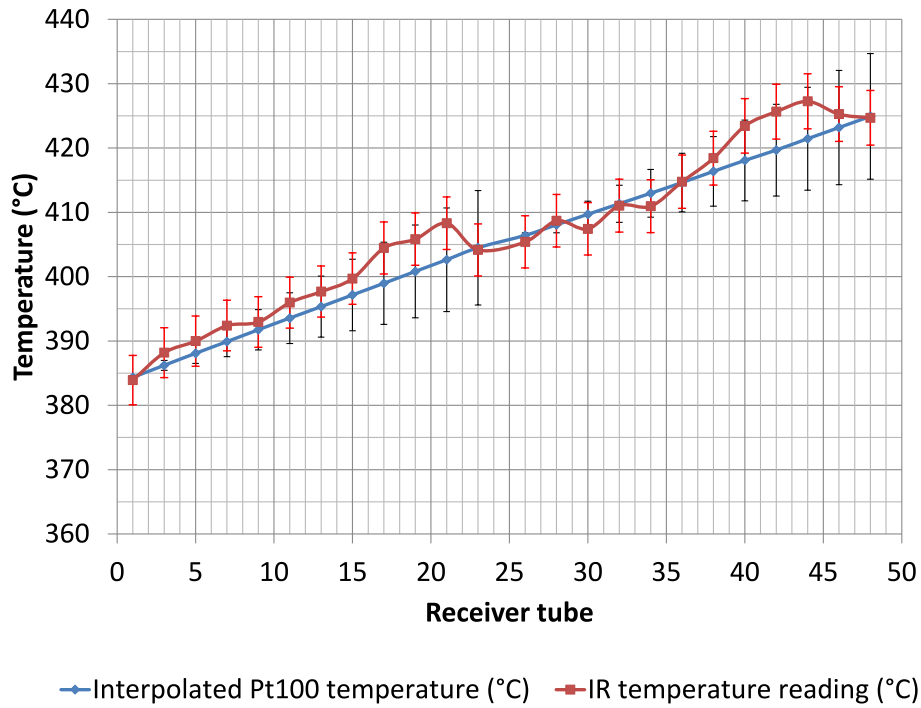
Several small leakage incidents are presented in the following, in order to give exemplary information about the leakage behavior of HELISOL<sup>®</sup> 5A. The root cause of the incident is also presented even though it shows the unintentional negligence of the authors regarding some components. It is mentioned that the plant was erected by Iberdrola and was subject to independent technical inspection afterwards. The demonstration operation took place later, while the leakages occurred in sections of the installation, which were not changed before or during the operation with HELISOL<sup>®</sup> 5A. Furthermore, the permitted operation parameters at these specific sections were not exceeded at any time.

**6.2.4.1. Incident 1 – Leaking fitting.** A first spontaneous leakage occurred April 26th 2016 at a pressure line fitting at the northern collector outlet at 374 °C and 23.8 bar under steady state operation conditions while the southern collector outlet was at 400 °C and 21.6 bar (see Fig. 12). To contain the leakage, operation was stopped, collectors were sent to stow position and the pump was switched off. The closest shut-off valves were closed to isolate the leakage from the rest of the plant to enable local draining and repairs. It was found, that the leakage

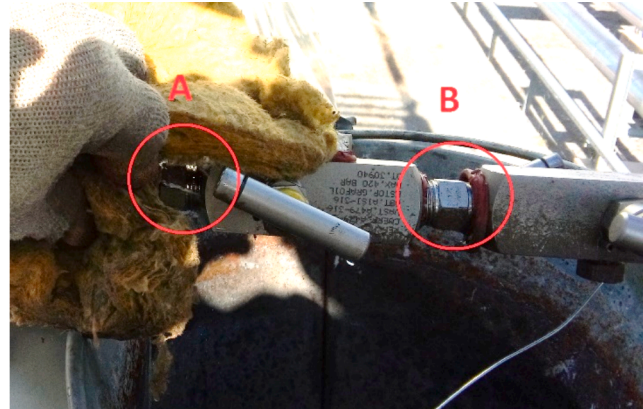
occurred at a connection fitting where the used sealant (HT silicone) was not suitable for applications at given temperatures and pressures.

**6.2.4.2. Incident 2 – Leaking 4” flange connection.** A second leakage occurred October 24th 2017 at a 4” ring type joints (RTJ) flange connection of a flow sensor at the collector feedline while the plant was under transient operation and decreasing the temperature by about 10 K/min to 370 °C and 15 bar at the sensor location (see Fig. 13). The fact that the insulation covered the flange connection / leakage prevented the operator from taking notice and at the same time enabled HTF accumulation inside the insulation material. This led to a reduced and delayed smoke formation and later to self-ignition inside the sheet metal cavity. The flame did not protrude beyond the sheet metal cavity, as an estimate of the flame size. The event of self-ignition can be perceived as the result of the combination of at least four factors: 1) the occurrence of an HTF leakage, 2) the condensation and accumulation of the leaking HTF 3) the accumulation of sufficient quantity of liquid HTF at a temperature close to the pipe temperature and 4) a sufficient long period of time to self-ignite. It was found, that screws suitable for lower temperature regimes were used and the tightening torque was not documented. Furthermore, both flanges were fully covered by insulation material. According to DIN 4754-1; Part 1 [24]: Safety requirements, test, “At locations with the risk of leakage (flanges, valves), the insulation must be applied in a way that leakages can be detected!” This was not respected, see Fig. 13 left side.

**6.2.4.3. Incident 3 – leaking 4” flange connection.** A third leakage occurred November 7th 2018 at the ring type joint flange connection of a second flow sensor located at the collector feedline while the plant was accidentally under transient operation decreasing the temperature by about 1 K/s to about 240 °C and 24 bar at the sensor location. The fact that the insulation was removed prior to the leakage made the leakage



**Fig. 11.** Temperature comparison of the interpolated Pt100 temperatures every 8 m (every second HCE) with IR temperature measurements at the same location, black error bars indicate the estimated measurement uncertainty (red IR measurement, black interpolation based on PT100). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



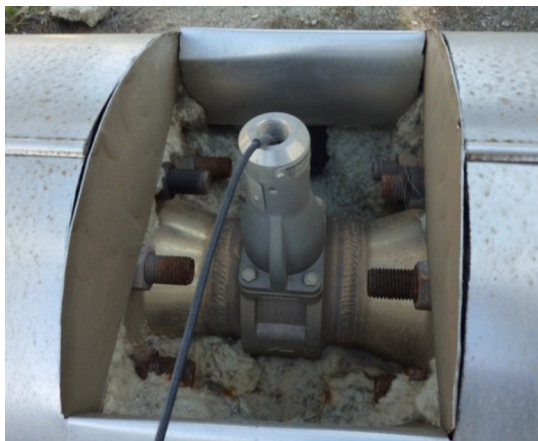
**Fig. 12.** Left: HELISOL<sup>®</sup> 5A vapor escaping from a leakage at a pressure indicator line at 374 °C and 23.8 bar, right: close up of the leaking fitting at cold unpressurized conditions, connection A presented the leak.

detectable immediately, see Fig. 14. It was later found out, that screws suitable for lower temperatures regimes were used and the tightening torques were not documented.

## 7. Conclusion

HELISOL<sup>®</sup> 5A successfully demonstrated its loop scale functionality achieving the proof of concept at Plataforma Solar de Almería (PSA)

after 480 h of operation at 425 °C in a parabolic trough test loop. In total app. 1100 h of solar operation were accumulated during a total number of 160 operation days. The monitored thermal HTF degradation did not change over time and thus was very low even though a stress test at temperatures between 440 and 450 °C during 50 h was performed. Requiring another 33 days of operation. The formation of methane and hydrogen due to HTF degradation was initially more pronounced and later reduced to a level close to the detection limit. This tendency was



**Fig. 13.** left: Sheet metal cavity where leakage became visible and self-ignition occurred, the flame did not protrude beyond the sheet metal cavity, right: flowmeter between two ring type joints flanges, thermal insulation removed after incident.



**Fig. 14.** HELISOL<sup>®</sup> 5A leaking from flow meter flanges at 240 °C and 24 bar during solar operation, the thermal insulation was removed prior to the incident.

independently confirmed by accompanying lab scale examinations [32]. In terms of operational safety and technical reliability heat collecting elements and rotation and expansion performing assemblies functioned without abnormalities. Still, three minor HTF leakages occurred at other locations due to local assembly failures. The heat transfer coefficients and film temperatures inside the HCE using HELISOL<sup>®</sup> 5A in comparison to the state-of-the-art HTF were calculated according to DIN 4754 for different mass flow rates and HTF temperatures. It can be concluded, that the heat transfer for both fluids is similar, as long as HELISOL<sup>®</sup> 5A is pumped with a similar mass flow compared to BP/DPO. When incorporating the significant difference in density at operation conditions, the use of HELISOL<sup>®</sup> 5A leads to about 30 % higher flow velocities and eventually to accordingly higher pumping parasitics at power plant

level. The HELISOL<sup>®</sup> 5A heat transfer performance was confirmed at state-of-the-art operation conditions at 400 °C by infrared camera-based absorber tube temperature field measurements.

#### **Uncertainty of the system demonstration**

In assessing the uncertainty of a system demonstration action, it is important to recognize that the outcome of the demonstration is not determined by means of a calculation. Therefore, a straightforward determination of the measurement uncertainty cannot be applied. Rather, one can translate “uncertainty” into “system robustness” or the “soundness” of the demonstration action. In this sense, a demonstration with low uncertainty must include a realistic setup, a high number of repetitions and also a variation of the most sensitive system parameters. In the present case, to assess the uncertainty from repetition the system was started and operated on 193 individual days. The maximum operating temperature was temporarily exceeded by 25 K and, in particular, critical low system pressures were implemented. The number of repetitions revealed uncertainties of the tubing system in terms of avoidable leakages and associated consequences, which are presented in section 6.2.4. The uncertainty resulting from exceeding the operating temperature was determined to be very low as the recorded HTF degradation was still at the lower detection limit. However, even after several months of operation, such a severe excess would not have any technical consequences. It rather indicates that the stated maximum operating temperature of 425 °C is a rather conservative value, this is underlined by the fact that there was no measurable wear of the HTF at this temperature during 480 h. The uncertainties resulting from operation at system pressures below the vapor pressure of the HTF are low as no functional consequences in terms of cavitation or reduced heat transfer were observed even at system pressures 4.5 bar below the given vapor pressure. To conclude, the overall uncertainty of the system demonstration can be considered low.

#### **CRedit authorship contribution statement**

**Christoph Hilgert:** Resources, Investigation. **Christian Jung:** Writing – review & editing, Supervision, Investigation. **Kai Schickedanz:** Writing – review & editing, Investigation. **Guillaume Saliou:** Writing – review & editing, Software, Resources, Investigation, Data curation. **Anne Schlierbach:** Resources, Investigation. **Marc Röger:** Supervision, Conceptualization. **Loreto Valenzuela:** Supervision, Resources, Investigation. **Erich Schaffer:** Resources, Investigation. **Christoph Wasserfuhr:** Resources, Investigation.

#### **Declaration of Competing Interest**

The authors declare that they have no known competing financial

interests or personal relationships that could have appeared to influence the work reported in this paper.

## Acknowledgements

The authors would like to thank the Federal German Ministry for Economic Affairs and Energy, the Ministry for Culture and Science of North Rhine-Westphalia, and the Spanish Ministry of Science and Innovation – Programa Estatal de I + D + I Orientada a los Retos de la Sociedad for funding the “SITEF” (Silicone Fluid Test Facility) project (0325846A, Germany), (PCIN-2014-083 (Spain), which included refurbishment of the PROMETEO test facility, its operation and accompanying scientific activities based on experiments carried out at the Plataforma Solar de Almeria. The authors thank Plataforma Solar de Almeria for providing access to its installations.

## References

- [1] Eastman, “Technical Data Sheet Therminol® VP-1,” <https://www.therminol.com>. [Online]. Available: [https://www.therminol.com/sites/therminol/files/documents/TF09A\\_Therminol\\_VP1.pdf](https://www.therminol.com/sites/therminol/files/documents/TF09A_Therminol_VP1.pdf).
- [2] W.C. Ag, Technical Data Sheet HELISOL 5A, // [www.wacker.com](http://www.wacker.com). [online]. Available // [www.wacker.com/h/en-de/medias/HELISOL-5A-en-2020.07.01.pdf](http://www.wacker.com/h/en-de/medias/HELISOL-5A-en-2020.07.01.pdf).
- [3] C. Jung, “Analyse und Reduktion der Wasserstoffbildung aus Wärmeträgern: Schlussbericht zum Forschungsvorhaben AREWa,” Deutsches Zentrum für Luft- und Raumfahrt e.V. (DLR), Institute of Solar Research, Cologne, 2014. [Online]. Available: <https://doi.org/10.2314/GBV:837>.
- [4] DOW, “Technical Data Sheet SYL THERM 800.” [Online]. Available: <https://www.dow.com/content/dam/dcc/documents/en-us/app-tech-guide/176/176-01435-01-syltherm-800-heat-transfer-fluid.pdf?iframe=true>.
- [5] E. Lüpfer et al., “EUROTROUGH Collector Qualification Complete - Performance Test Results FROM PSA,” in *ISES Solar World Congress*, Göteborg, 2003.
- [6] L. Valenzuela, R. López-Martín, E. Zarza, Optical and thermal performance of large-size parabolic-trough solar collectors from outdoor experiments: A test method and a case study, *Energy* 70 (2014/06/01/ 2014,) 456–464, <https://doi.org/10.1016/j.energy.2014.04.016>.
- [7] M. Becker, Comparison of heat transfer fluids for use in solar thermal power stations, *Electr. Pow. Syst. Res.* 3 (3) (1980) 139–150, [https://doi.org/10.1016/0378-7796\(80\)90001-2](https://doi.org/10.1016/0378-7796(80)90001-2).
- [8] L. Heller, “Literature Review on Heat Transfer Fluids and Thermal Energy Storage Systems in CSP Plants,” in “Solar Thermal Energy Research Group - Report,” Stellenbosch University, Stellenbosch South Africa, 2013. [Online]. Available: [https://sterg.sun.ac.za/wp-content/uploads/2011/08/HTF\\_TESmed\\_Review\\_2013\\_05\\_311.pdf](https://sterg.sun.ac.za/wp-content/uploads/2011/08/HTF_TESmed_Review_2013_05_311.pdf).
- [9] K. Vignarooban, X. Xinhai, A. Arvay, K. Hsu, A.M. Kannan, Heat transfer fluids for concentrating solar power systems – A review, *Appl. Energy* 146 (2015) 383–396, <https://doi.org/10.1016/j.apenergy.2015.01.125>.
- [10] N. National Renewable Energy Laboratory, “Concentrating Solar Power Projects,” <https://www.nrel.gov/>. [Online]. Available: <https://solarpaces.nrel.gov/>.
- [11] DOW, “Technical Data Sheet DOWTHERM A.” [Online]. Available: <https://www.dow.com/en-us/document-viewer.html?randomVar=2563939514817185210&docPath=/content/dam/dcc/documents/en-us/productdatasheet/176/176-01463-01-dowtherm-a-tds.pdf>.
- [12] LANXESS, “Technical Data Sheet Diphyll (Bruchure).” [Online]. Available: [https://advancedindustrialintermediates.com/fileadmin/user\\_upload/Diphyll\\_brochure\\_04.2017.pdf](https://advancedindustrialintermediates.com/fileadmin/user_upload/Diphyll_brochure_04.2017.pdf).
- [13] I. N. d. S. y. S. e. e. Tra., Ó. Lerma García, and M. Sánchez Fuentes, “NTP 1151 Exposición a HTF en centrales termosolares de concentradores cilíndrico parabólicos.” [Online]. Available: <https://www.insst.es/el-instituto-al-dia-ntp-1151-exposicion-a-htf-en-centrales-termosolares-de-concentradores-cilindrico-parabolicos>.
- [14] J.W. Raade, D. Padowitz, Development of molten salt heat transfer fluid with low melting point and high thermal stability, *J. Sol. Energy Eng.* (2011).
- [15] M. Eck, E. Zarza, M. Eickhoff, J. Rheinländer, L. Valenzuela, Applied research concerning the direct steam generation in parabolic troughs, *Sol. Energy* 74 (4) (2003/04/01/ 2003,) 341–351, [https://doi.org/10.1016/S0038-092X\(03\)00111-7](https://doi.org/10.1016/S0038-092X(03)00111-7).
- [16] J. J. Grebe, “Composition of matter,” USA Patent 1,882,809, 1932.
- [17] E.S. Blake, W.C. Hammann, J.W. Edwards, T.E. Reichard, M.R. Ort, Thermal Stability as a Function of Chemical Structure, *J. Chem. Eng. Data* 6 (1) (1961) 87–98.
- [18] J. Yeatts, L. B., J. E. Attrill, and J. Rainey, W. T., “GAS CHROMATOGRAPHIC ANALYSIS OF BIPHENYL PYROLYTIC PRODUCTS,”; Oak Ridge National Lab., Tenn., ORNL-TM-523 United States 10.2172/4723744 ORNL English, 1963. [Online]. Available: <https://www.osti.gov/servlets/purl/4723744>.
- [19] H. Price, R. Forristall, T. Wendelin, A. Lewand, T. Moss, C. Gummo, “Field Survey of Parabolic Trough Receiver Thermal Performance,” in *ASME International Solar Energy Conference*, Denver, CO, 2006.
- [20] A. G. Wacker Chemie and S. Dörrich, “Entwicklung umweltfreundlicher Hochtemperatur-Wärmeträgerfluide für solarthermische Parabolrinnenkraftwerke auf Siliciumbasis : Schlussbericht zum Forschungsvorhaben Si-HTF,” Wacker Chemie AG; München, 2015. [Online]. Available: <https://doi.org/10.2314/GBV:869413899>.
- [21] C. Jung, J. Dersch, A. Nietsch, M. Senholdt, “Technological perspectives of silicone heat transfer fluids for concentrated solar power,” in *SolarPACES Conference*, Beijing (2015), <https://doi.org/10.1016/j.egypro.2015.03.076>.
- [22] C. Hilgert, C. Jung, L. Valenzuela, E. Schaffer, D. Lei, “Silicone-Based Heat Transfer Fluids (SiHTF). In *Line Focusing Concentrating Solar Power Applications*, 2021.
- [23] C. Hilgert C. Jung C. Wasserfuhr J. Leon L. Valenzuela Qualification of silicone based HTF for parabolic trough collector applications SolarPACES 2019 Casablanca, Morocco. <https://doi.org/10.1063/1.5117598>.
- [24] D. 4754–1 (2015) 2015.
- [25] *Solar thermal electric plants – Part 1-6: Silicone-based heat transfer fluids for use in line-focus concentrated solar power applications (PRE-RELEASE VERSION)*, IEC 62862-1-6, I. E. C. (IEC), 2024. [Online]. Available: [https://webstore.iec.ch/preview/info\\_iecfdi62862-1-6%7Bed1.0%7Den.pdf](https://webstore.iec.ch/preview/info_iecfdi62862-1-6%7Bed1.0%7Den.pdf).
- [26] RIOGLASS. “Receiver Tubes for Linear CSP (Concentrated Solar Power) Applications.” <https://www.rioglass.com/our-products/hce-tubes.html> (accessed 28.07.2021, 2021).
- [27] P.C. Prah, *Photogrammetric Measurement of the Optical Performance of Parabolic Trough Solar Fields*, RWTH Aachen University, Aachen, 2019.
- [28] P. S. d. A. CIEMAT, PROMETEO, Parabolic Trough Tests Facility, accessed 28.07.2021, CIEMAT. (2021), [https://www.psa.es/en/facilities/parabolic\\_trough/prometeo.php](https://www.psa.es/en/facilities/parabolic_trough/prometeo.php).
- [29] C. Jung et al., “Abschlussbericht zum Verbundvorhaben SITEF - Silicone fluid test facility : Begleitende Laboruntersuchungen und Kollektorqualifizierung : Befüllung der Anlage und Testbetrieb ; Entwicklung und Einsatz einer flexiblen Rohrverbindung : Erhöhung der Betriebssicherheit und Beurteilung des Gefährdungspotenzials : Förderzeitraum: 01.01.2016-31.12.2017,” Deutsches Zentrum für Luft- und Raumfahrt (DLR); [Köln], 2018. [Online]. Available: <https://doi.org/10.2314/GBV:1663361460>.
- [30] F. Ortiz Vives, A. Kaufung, New Flexible Connection System For Parabolic Trough Collectors, *SolarPACES Conference*, Las Vegas (2008).
- [31] C. Jung, M. Senholdt, C. Spenke, T. Schmidt, and S. Ulmer, “Hydrogen Monitoring and Control in the Heat Transfer Fluid of Parabolic Trough Plants,” in *SolarPACES 2018*, online, 2019, doi: <https://doi.org/10.1063/1.5117599>.
- [32] A. G. Wacker Chemie. *HELISOL® – HEAT TRANSFER FLUIDS Technical Brochure*. (2020). Wacker Chemie, A. G. [Online]. Available: [https://www.wacker.com/h/en-th/medias/4\\_HELISOL\\_Technical\\_Product\\_Brochure.pdf](https://www.wacker.com/h/en-th/medias/4_HELISOL_Technical_Product_Brochure.pdf).