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Measurement of the Density of Biphenyl / Diphenyl Oxide Mixtures at Isobaric Conditions

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Abstract. The density of the eutectic mixture of biphenyl and diphenyl oxide was measured at 12 to 40 bar and up to 400 °C for the first time. An unused quality as delivered and a used quality from a parabolic trough plant were tested. Significant compressibility was observed for both qualities giving rise to density increases of 3% for the unused fluid at 40 bar and 4.8% for the used fluid compared to the technical data sheet of Dowtherm A.

Keywords: Biphenyl, Diphenyl Oxide, Dowtherm A, Density, Pressure, Compressibility

1. Introduction

The density of heat transfer fluids (HTFs) used in parabolic trough plants needs to be known within the temperature and pressure range applied for operation of the fluid. At increasing temperature, the density of HTFs is decreasing which causes considerable expansion that has to be compensated by providing sufficiently large vessels in the HTF system. Moreover, density is relevant for pressure loss and pumping power considerations. Hence, precise density data are relevant for the design of parabolic trough plants.

In parabolic trough plants HTFs are operated at pressures above their vapor pressure. There is a nitrogen blanket used to prevent oxidation but also any boiling of the fluid. In addition, there is mechanical pressure on the fluid by the HTF pumps forcing the fluid through the solar field. Design total pressure of a parabolic trough plant is up to 41 bar.

Most parabolic trough systems are operated with the eutectic mixture of biphenyl (BP) and diphenyl oxide (DPO). Technical data sheets provided by typical manufacturers of this fluid do only provide density data for saturation pressure. [1-3] *Cabaleiro* et al. [4] studied the compressibility of BP/DPO mixtures at pressures up to 450 bar but only at temperatures up to 90 °C.

Up to now there is no study addressing the impact of pressure on the density of BP/DPO mixtures at temperatures up to 400 °C giving rise to some uncertainty of total volume changes caused by heating and cooling of the fluid and regarding volumetric heat capacity. This study addresses density measurements covering the upper operating conditions of HTFs with respect to temperature and pressure. It is also addressed whether significant changes may be caused by extensive use of the fluid. An unused (fresh) BP/DPO quality is tested as well as a used quality that had been in operation in a parabolic trough plant for more than ten years.

2. Approach

The experiment used for this study is based on measuring the volume of the fluid to be tested at defined pressure and temperature.

The set-up is shown in Figure 1. It includes a steel cylinder located within a circulating air furnace which provides stable thermal conditions up to 600 °C. The cylinder is connected to a syringe pump outside the furnace. This pump allows for adjusting and maintaining enhanced pressure up to several 100 bar as well as precise measurement of volume changes. The cylinder is equipped with a temperature sensor inside the volume. A pressure transducer is included into the pump head for pressure control and an additional precise pressure transducer (with 0.02% uncertainty) is included into the fluid connections for monitoring. Several temperature sensors (calibrated within less than 0.5 K uncertainty) are mounted all over the piping to monitor the actual temperature of the fluid in each section outside the furnace.

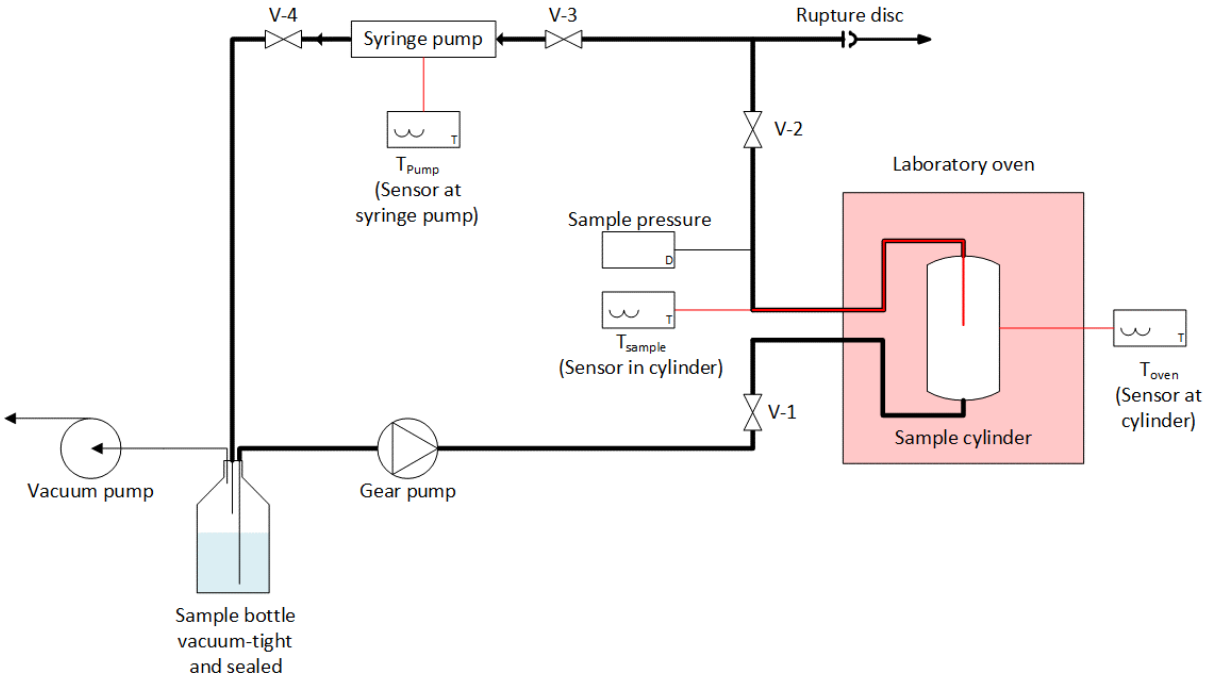

Figure 1. *Experimental set-up with most relevant components.*

The exact volume of the cylinder and of each piping section assigned to a temperature sensor is determined volumetrically at ambient temperature using deionized water and counterchecked by filling the system with eutectic BP/DPO at ambient temperature.

The apparatus is completely filled by pumping the fluid to be tested from a vacuum-tight sample bottle with a gear pump through the sample cylinder and the piston pump within a circuit back to the sample bottler. The latter is connected to a vacuum pump. The filling process is performed until all gas is removed from the apparatus and the fluid. The mass of fluid in the system is determined gravimetrically by measuring the weight change of the sample bottle supplying the system during the filling process.

The density of the fluid between 20 – 120 °C is determined with a SVM 3001 *Stabinger* device from *Anton Paar* (with less than 1% uncertainty). Compression tests at ambient temperature confirmed that the fluids under investigation show negligible compressibility around 25 °C. Hence, calculation of HTF masses in the syringe pump and the piping outside the furnace can be done without any pressure correction as these parts of the system maintain at ambient temperature throughout the tests.

3. Results and Discussion

Isobaric expansion of unused Dowtherm A (>99.9% BP/DPO, provided from ThermoFisher) was tested up to 400 °C and at 12 - 40 bar. The density of the fluid inside the cylinder is calculated from the total mass of the fluid in the experiment, the fluid amount in the syringe pump as well as in the connection piping and the volume of the cylinder corrected by its thermal expansion.

Accordingly, the density at 12 bar differ from Dow's data sheet which is referring to saturation pressure only within +0.3/-0.4% (see Figure 2). At higher pressures, significantly higher densities are found at temperatures around the normal boiling point of the fluid. Within 300 – 400 °C significantly higher densities are found at pressures between 20 – 40 bar. At 20 bar the density is 0.5 – 1% higher, at 30 bar the increase is 1 – 2% and at 40 bar densities are 1 – 3% higher compared to the densities in the data sheet.

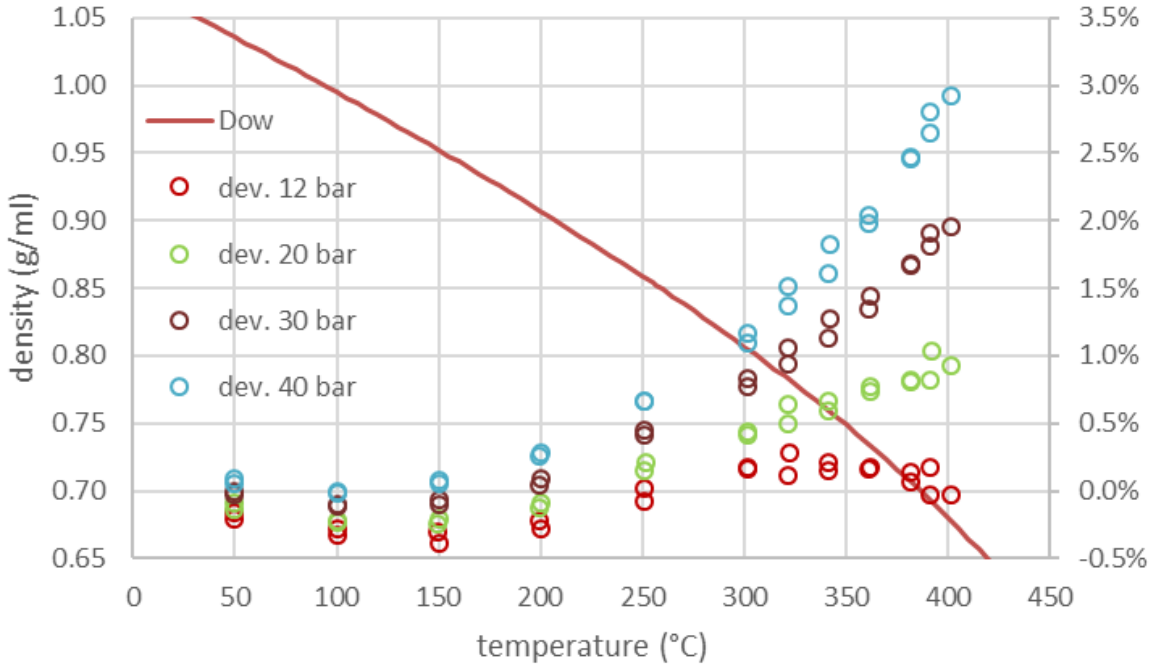

Figure 2. *Measured deviation of isobaric density of unused Dowtherm A at 12 – 40 bar from technical data sheet.*

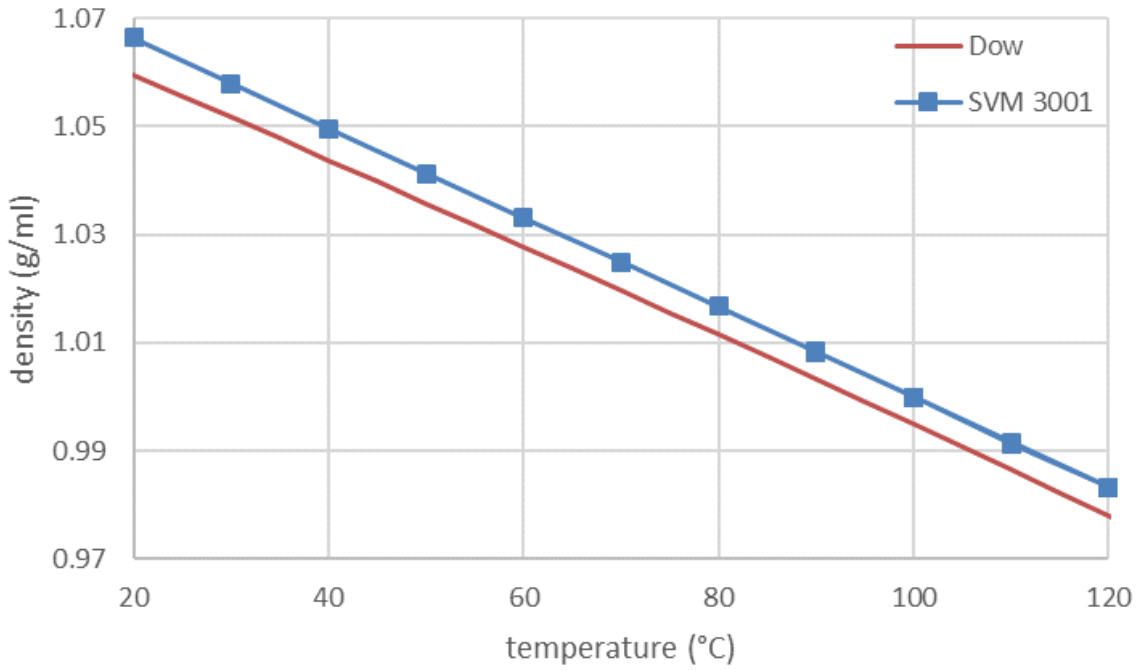

Figure 3. *Density of used BP/DPO mixture with 0.5% low boilers and 6% high boilers (blue dots are measured data, blue line is interpolated) in comparison to unused Dowtherm A (red line) according to data sheet.*

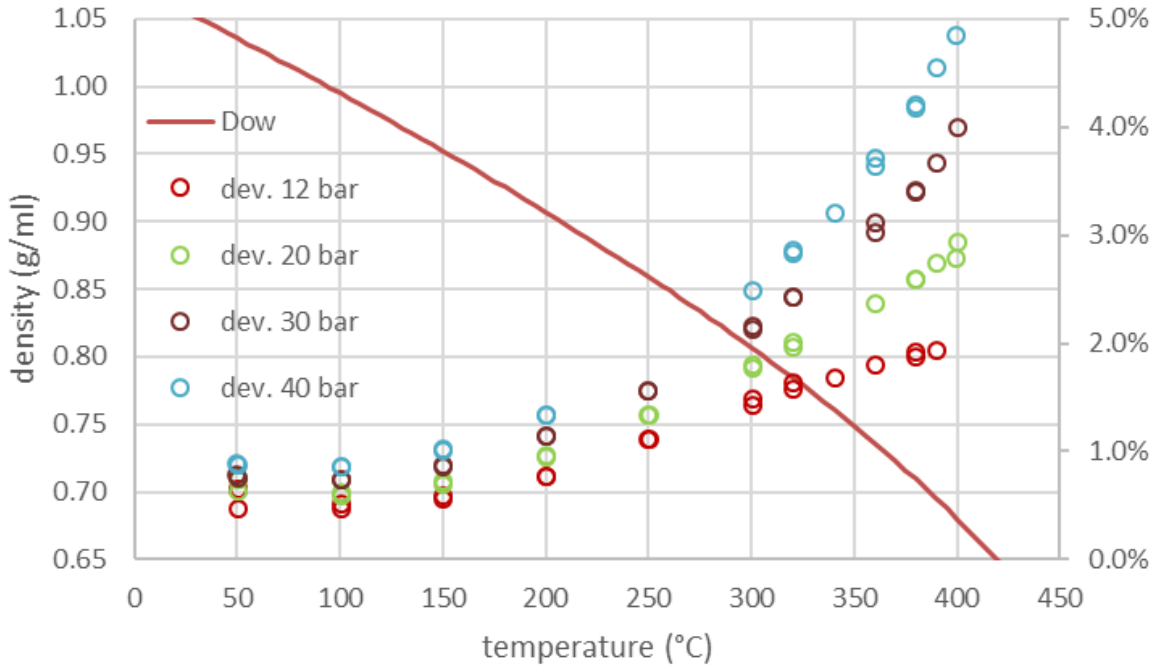

Figure 4. *Measured deviation of isobaric density of used eutectic BP/DPO mixture at 12 – 40 bar from technical data sheet of unused Dowtherm A.*

A sample of used BP/DPO mixture from a Spanish parabolic trough plant was tested as well. This used fluid was filtered with a PTFE membrane with 0.2 µm pore size to prevent any particle issues with the syringe pump. According to gas chromatographic analysis, the filtered sample contained 0.5% low boiling and 6% high boiling degradation products.

The density of the used fluid is significantly higher compared to unused BP/DPO. At ambient pressure the used fluid has 0.5% higher density compared to unused Dowtherm A according to the technical data sheet (see Figure 3). At 12 bar the density increase is up to 2.0%, at 20 bar it is up to 2.9%, at 30 bar up to 4.0% and at 40 bar up to 4.8% at 400 °C (see Figure 4).

4. Conclusion

The study indicates that eutectic BP/DPO mixture is significantly compressible within the typical operating temperature range of solar thermal parabolic trough plants. With density increases of 3% at 40 bar the effect is small for unused fluid but it increases to 4.8% significantly for used fluid containing 6% high and 0.5% low boiling degradation products. Used fluids from CSP plants can contain considerably higher concentrations of low and high boilers than investigated within this study. Accordingly, the impact on the density might be even higher for used fluids compared in this study.

The study illustrates that density data at high temperature according to manufacturer's data sheets differ significantly at enhanced pressure from actual fluid densities even in the unused condition of the fluid. Densities of used fluids can differ even stronger due to the presence of degradation products.

As the density of BP/DPO mixtures increases at higher pressures, no issues with expansion volumes are to be expected that rely on manufacturer's data sheets which suggest higher thermal expansion than measured in this study. Calculations of pressure drop and pumping power based on such data sheets can be expected to result in overestimations as well.

The used fluid investigated in this study was only moderately degraded. Hence, further studies with stronger degraded fluids have to be performed to provide an overview on the possible variation of the density in the used condition.

Data availability statement

No additional data is available.

Underlying and related material

There is no supplementary material available.

Author contributions

Carsten Spenke focused on the development of experiment, conducted the measurements and evaluated the results. Christian Jung supported setting up the experiment and data evaluation and took care for the manuscript.

Competing interests

The authors declare that they have no competing interests.

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