Quest for a reference standard for viscosity at high temperatures and high pressures

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General Aim

To find a suitable viscosity reference fluid for

- high temperatures,
- high pressures
- high viscosity

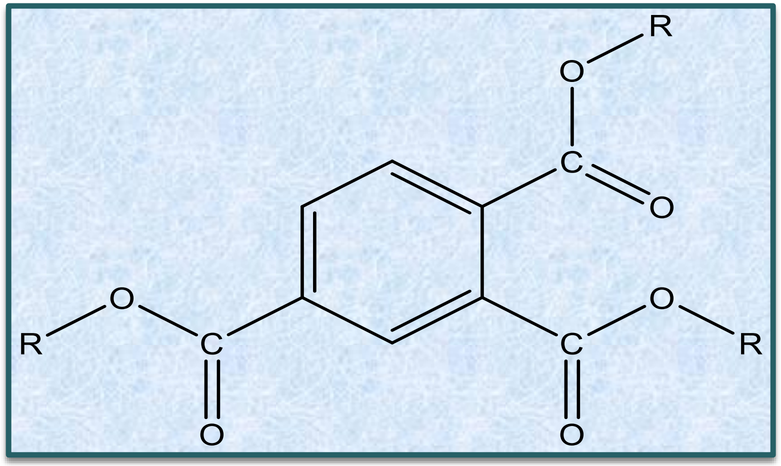
For industrial use in oil extraction and tribology

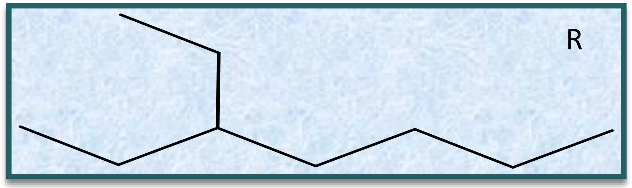
Proposal: **TOTM -** *Tris(2-ethylhexyl) trimellitate*

Tris(2-ethylhexyl) trimellitate – TOTM

Appropriate for use as high viscosity reference liquid for high temperatures and high pressures:

- Low vapour pressure;
- Large temperature range in the liquid state: (-50 to 414) ºC
- Available with high purity: ≅ 99%
- High viscosity: near the ultimate target for oil exploration industry
- Commercial availability at reasonably low cost
- Not classified as dangerous, according to European Directive 67/548/EEC - harmful in contact with skin, and susceptible to cause serious eye irritation





The ultimate target for oil exploration industry (473 K, 200 MPa)

In pursuit of a high temperature, high pressure, high viscosity standard: The case of TOTM:

W.A. Wakeham, M. J. Assael, H.M.N.T. Avelino, S. Bair, H.O. Baled, B.A. Bamgbade, J.-P. Bazile, F.J.P. Caetano, M.J.P. Comuñas, J.-L. Daridon, J.C.F. Diogo, R.M. Enick, J.M.N.A. Fareleira, J. Fernández, M.C. Oliveira, T. Santos, Ch.M. Tsolakidou; J. Chem. Eng. Data 2017 (in press for special issue in honor of Ken Marsh).

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In pursuit of a high temperature, high pressure, high viscosity standard: the case of TOTM
By W.A. Wakeham et al.

TOTM [Tris(2-ethylhexyl) trimellitate] CAS-No. 3319-31-1

Calculation of Viscosity and Density for interpolation of the values in Table 9

Input

T/K	p/MPa
473	200

Output

ρ / kg.m^{-3}	η / mPa.s
992.60	10.2

Change values ONLY in the green highlighted cells.

Excel™ executable file

TOTM: Studies carried out so far (results incorporated in the joint article) (4 different lots)

- Viscosity;
- Density;
- Effect of impurities on the viscosity and density;
- Surface Tension /taking into account capillary measurements;
- Effect of water contamination;
- Examination of Newtonian behaviour over a wide range of shear rates at high pressure;
- Thermal Stability;
- Speed of sound.

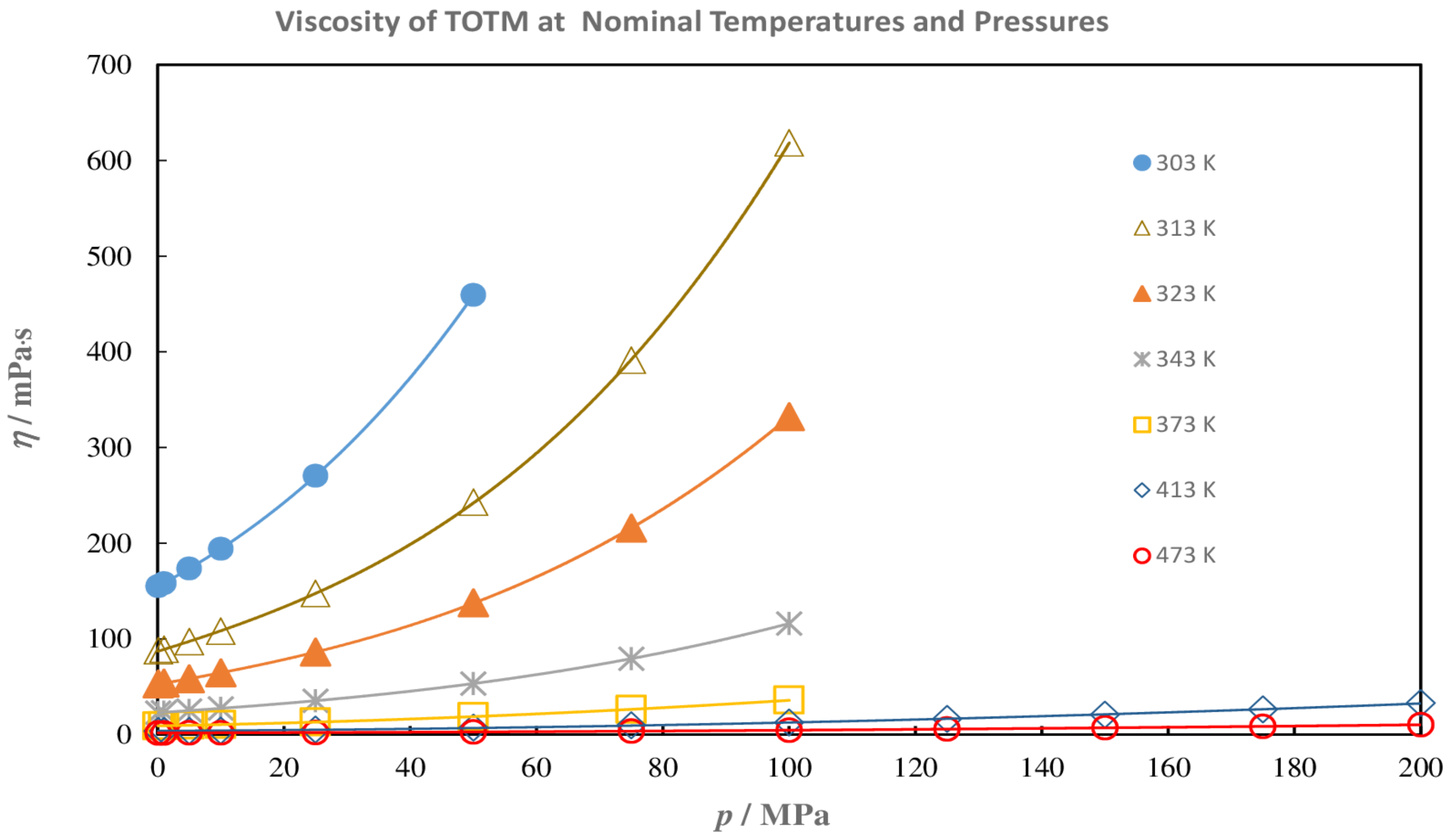
VISCOSITY

<u>Standard reference viscosity valid in:</u>

Temperature range : 303 to 473 K

Pressure range: 0.1 to 200 MPa

Viscosity range: 1.6 to 755 mPa.s



Present work has the particular aim:

- To present new density data included in the earlier paper (special issue) that have not yet been published.

- To complement the rheological studies presented in the special issue, giving particular attention to the shear rate range of greatest interest for the proposed use of TOTM as an industrial reference fluid for viscosity – check of the Newtonian behaviour under atmospheric pressure at shear rates attainable in capillary measurements.

Measurements of the Density of Liquid TOTM in the joint article.

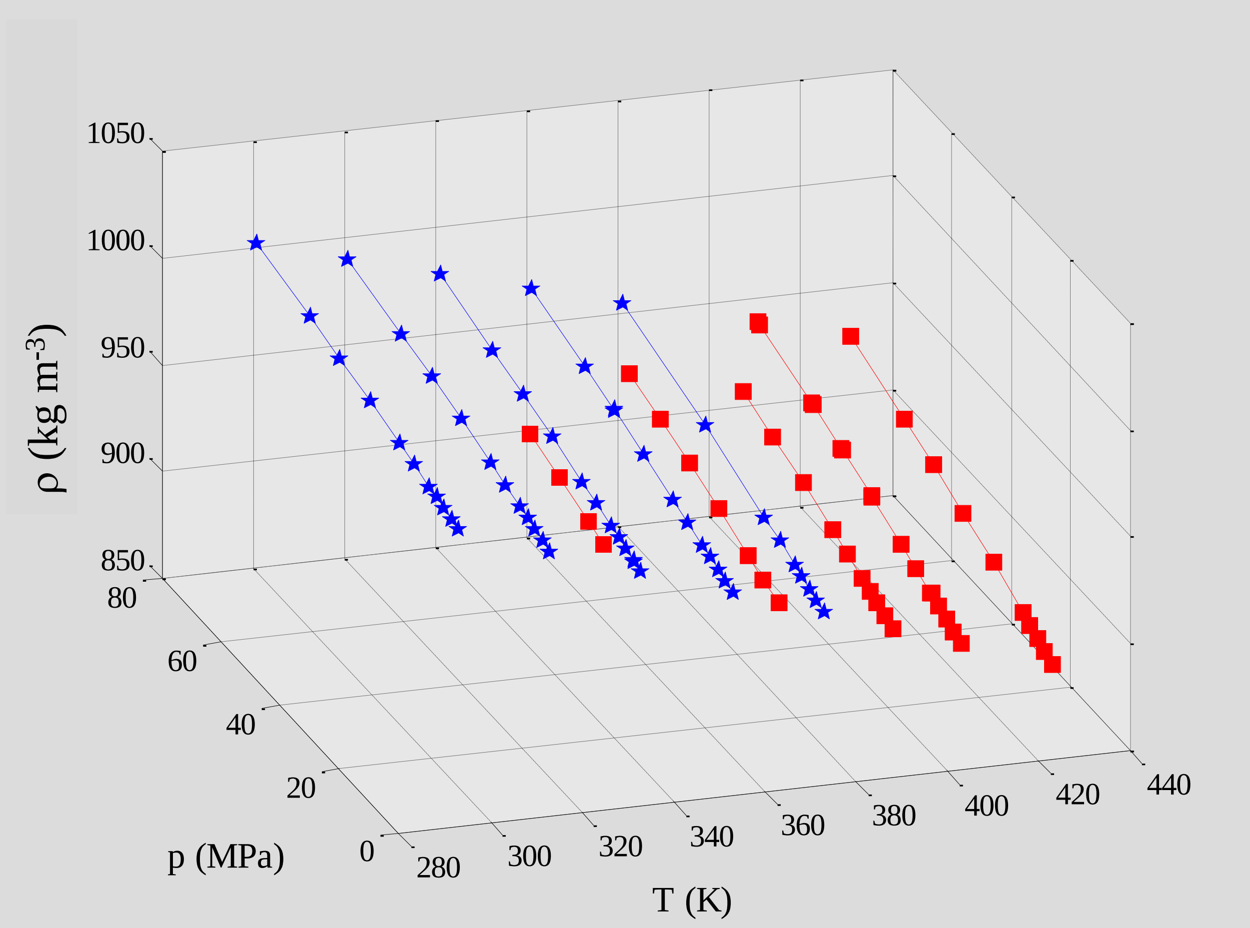
Year	First author	Ref. nº	Temperature Range / K	Pressure Range / MPa	Number of data	Method	lot number	Purity %	Nominal uncertainty[a)]	
1998	Lorenzi	29	288-358	0.1	10	U-tube	NA	99	0.04 kg·m^{-3}	
2014	Diogo	16	293-373	0.1-68	66	U-tube	(A)	99	0.2%	
2015	Diogo	23	293-373	0.1-68	16	U-tube	(B)	99	0.2%	
2015	**Avelino**	**30**	**388-403**	**0.1-68**	**22**	**U-tube**	**(B)**	**99**	**0.2%**	**Present work**
2015	Bair	12	313-410	0.1-350	18	Bellows piezometer	(B)	99	0.2%	
2015	**Avelino**	**30**	**328-423**	**0.1-68**	**50**	**U-tube**	**(A)**	**99**	**0.2%**	**Present work**
2016	Bamgbade	22	314-523	3.5-270	79	Variable-Volume	(C)	99	0.7%	
2016	Bazile	24	293-373	0.1-200	182	Speed of Sound	(C)	99	(0.1-0.2)%	
2016	Bazile	24	293-373	0.1-140	135	U-Tube	(C)	99	0.1%	

Density

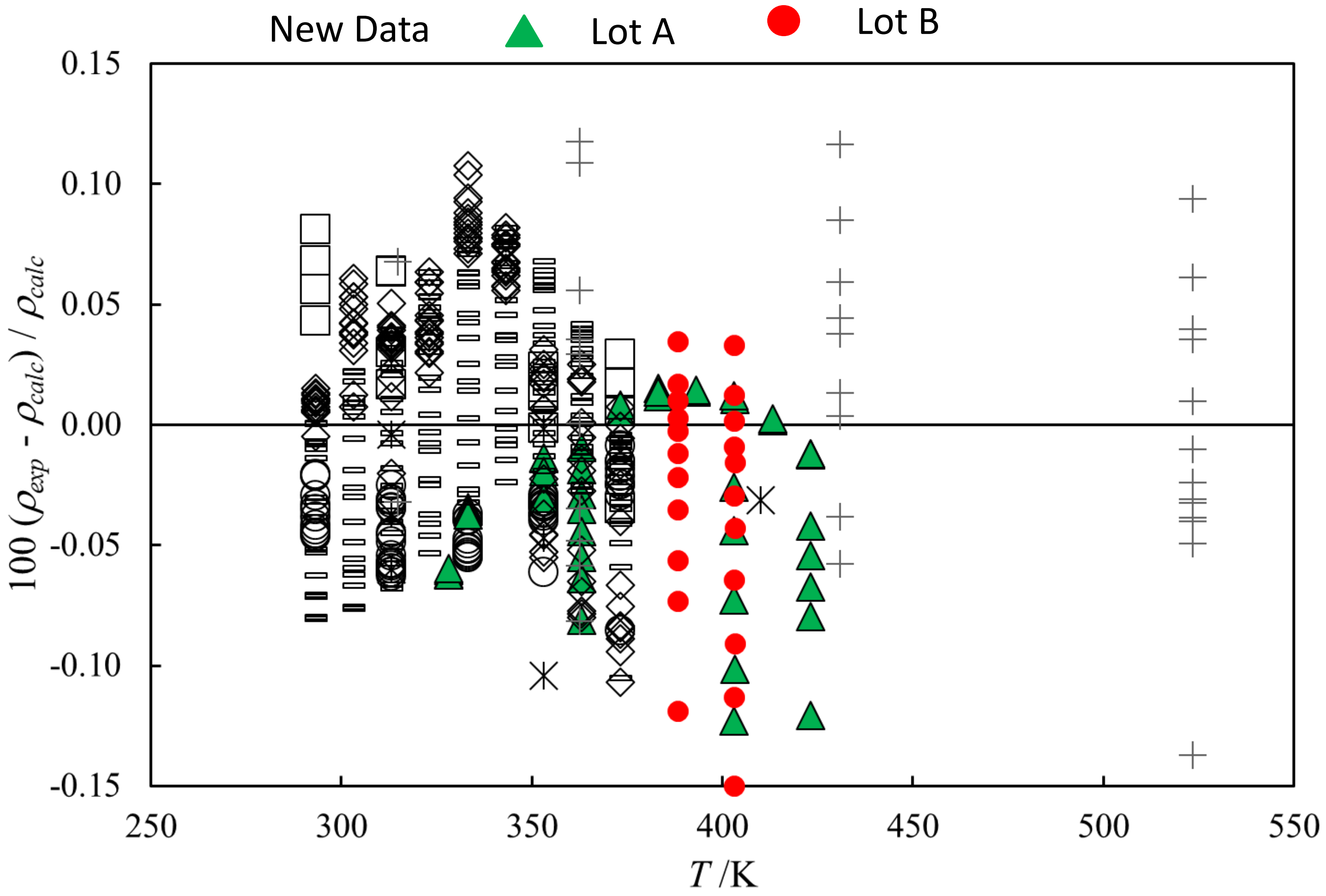
New density measurements have been performed using an Anton Paar vibrating U-tube model HP, using Model 5000 as a reading unit

The new results were aimed to extend the range of our previous experimental data.

★ Previous data ■ New data



Deviations of the available density data at high pressures from their correlation



○,Diogo 2014; □, Diogo 2015, ∗,Bair; ▬, Bazile (SS); ◇,Bazile (U-tube); ×, Bamgbade

Rheological studies –

- Previous work mentioned in the joint article by Scott Bair:
- Newtonian behaviour was observed at shear stresses below 5 MPa at high pressures
- and confirmed by Assael and Tsolakidou in the low shear domain, useful for normal industrial needs.

Present work investigates the following domains:

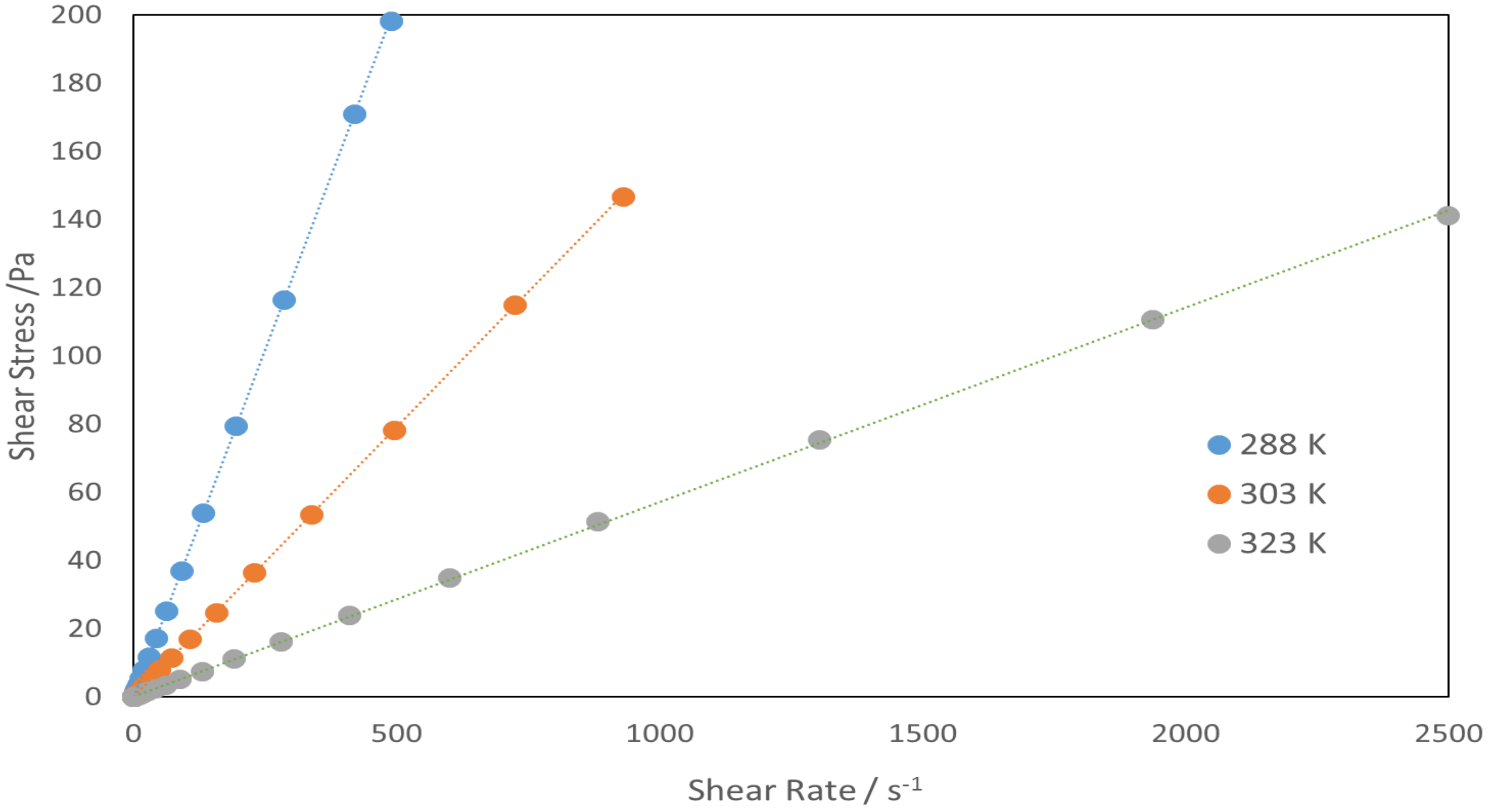
- shear stress up to 750 Pa and shear rate up to 4000 s^{-1} at atmospheric pressure,

Measurements with a parallel plate Rheometer -

➢ Rheometer AR1500ex10C4298 (40mm parallel plate, Peltier steel plate)

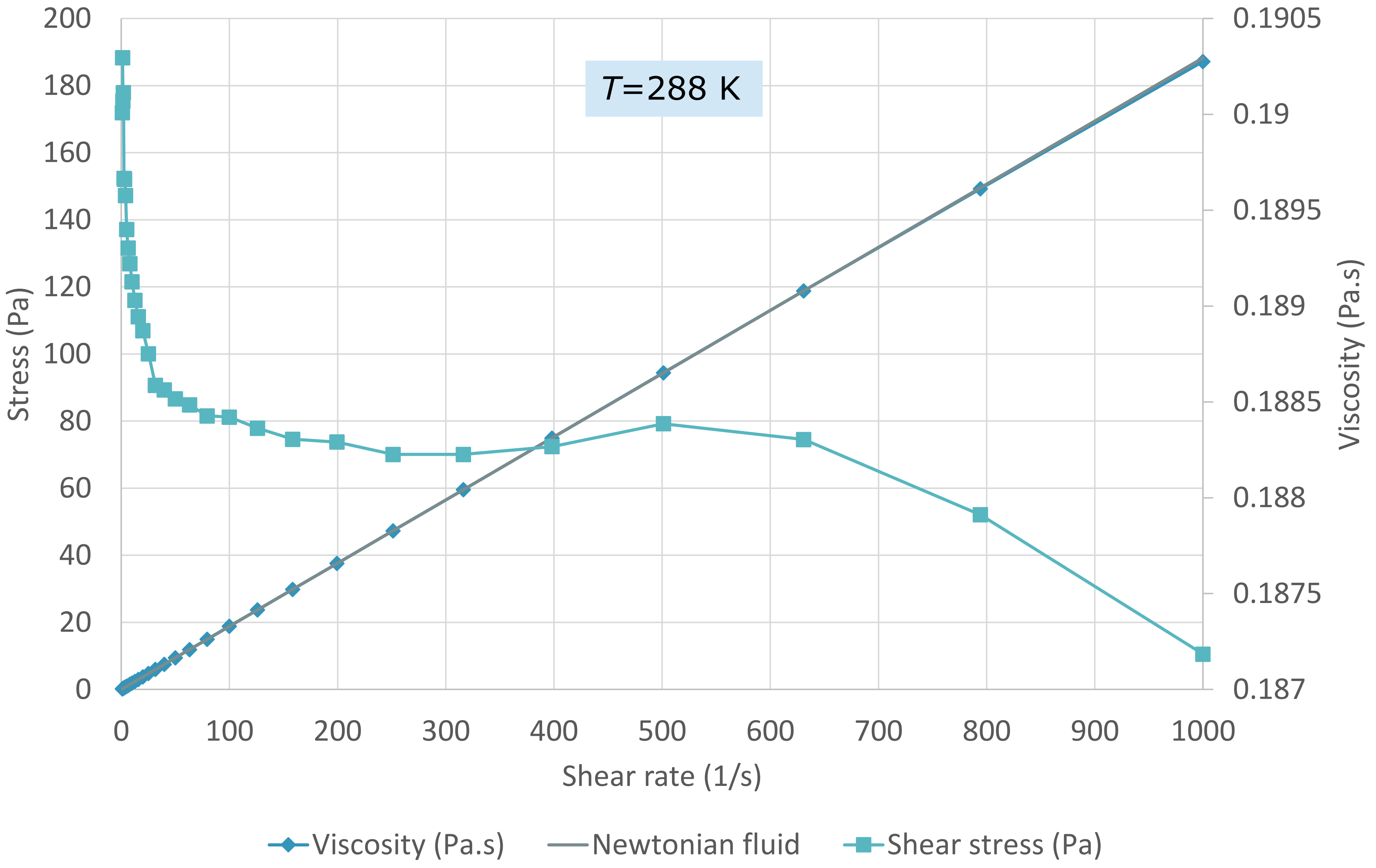
Results at three temperatures confirm Newtonian behaviour

Experiments with shear stress up to 200 Pa and shear rate up to 500 s^{-1} at 288 K



If the experiment at 288 K is continued to higher shear ratesNewtonian behaviour is observed until about 600 s^{-1}

For shear rates higher than 600 s^{-1} a shear-thinning behaviour is evidenced. No evidence of turbulence appears.-1



As a conclusion, TOTM has a rheological behaviour compatible with its use as an industrial reference fluid for viscosity.

These results confirm previous studies described in

W.A. Wakeham, M. J. Assael, H.M.N.T. Avelino, S. Bair, H.O. Baled, B.A. Bamgbade, J.-P. Bazile, F.J.P. Caetano, M.J.P. Comuñas, J.-L. Daridon, J.C.F. Diogo, R.M. Enick, J.M.N.A. Fareleira, J. Fernández, M.C. Oliveira, T. Santos, Ch.M. Tsolakidou; **In pursuit of a high temperature, high pressure, high viscosity standard: The case of TOTM,** J. Chem. Eng. Data 2017 (in press for special issue in honor of Ken Marsh).

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