Addendum to the article "Viscosity data for kukersite shale gasoline fractions"

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Received 18 January 2023, accepted 14 April 2023, available online 10 May 2023

Abstract. The evaluation of experimental data is based on the disclosure of essential information related to data measurement. A recent paper published in the journal Oil Shale presented experimental viscosity data on narrow boiling range fractions, prepared by distillation from a wide gasoline fraction of Kukersite oil shale pyrolysis oil (from an industrial plant). However, the article suffers from a deficiency of experimental description coupled with somewhat of an oversimplification of derivation of viscosity data from capillary viscometer measurements. Therefore, this addendum or short commentary supplemental article provides additional experimental information desirable for data evaluation and interpretation, along with corresponding corrections to the data.

Keywords: *kukersite oil shale retort oil, straight run naphtha boiling range fraction, kinematic viscosity, dynamic viscosity, viscosity estimation methods.*

Introduction

The present short paper is intended to complement the article by Baird et al. "Viscosity data for kukersite shale gasoline fractions" [1] (whose author list includes also the author of this paper) and provides missing experimental information related to the evaluation and interpretation of the data and viscosity estimation methods presented in the original article. The current paper also contains slightly corrected (refined) viscosity data based on additional information made available. Therefore, it is essential that this paper should be viewed as an addendum, rather than a stand-alone article.

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Supplement to subchapter 2.1. Sample preparation

Narrow boiling range fractions (distillation cuts) for viscosity measurements were prepared by distillation from a straight run naphtha boiling range fraction of Kukersite oil shale retort oil (pyrolysis oil from Galoter process [2, 3, 4]). The straight run naphtha boiling range fraction, or the so-called technical gasoline fraction (about 20% by weight of the total oil and with a molecular weight ranging from about 50 to about 150 g/mol), was separated by a fractional condensation of the total retort oil (with a molecular weight of about 50 to 900 g/mol [5, 6]) in the aboveground industrial plant. Technical gasoline fractions taken at two different times from the plant, which operated under the same plant regime, were used for sample preparation by distillation. The distillations to narrow boiling range fractions were performed at atmospheric pressure. One set of samples (from the technical fraction with a density of 0.78998 g/cm³ at 20 °C) was prepared using a simple batch distillation equipment (non-standard setup due larger glass flask/sample size, otherwise following the basic procedure of ASTM D86 [7, 8]). The second set of samples (from the technical fraction with a density of 0.79025 g/cm³ at 20 °C) employed a batch rectification equipment (following the basic procedure of ASTM D2892 [9, 10]). A cooling temperature between about a few degrees and -10 °C was applied for sample collection to minimise the loss of volatiles. After preparation, the narrow boiling range samples were stored in an inert gas environment (in hermetically sealable sample bottles, closed in an inert gas glove-box) in a dark cabinet at room temperature or below to minimize sample aging. The narrow boiling point fractions were viewed as pseudocomponents and were characterised by average properties [11–14]. The "pseudo" concept is a simplified approach where a fraction with a narrow boiling range is viewed as a single substance.

Supplement to subchapter 2.2 Viscosity measurements

The kinematic viscosity measurement setup, designed to measure the kinematic viscosity of one sample at a time, was constructed in-house. A Fungilab Cannon-Fenske routine capillary viscometer (size 25; recommended measurement range 0.5-2 cSt) with a manufacturer certificate (conversion factors at +40 °C and +100 °C for calculation of the kinematic viscosity directly from the flow time; stated viscometer expanded uncertainty of 0.3%) was applied to measure the kinematic viscosity of Kukersite gasoline narrow boiling range fractions (fluids with Newtonian flow behaviour). The viscometer was filled at room temperature. The flow time of each sample was the generally average of 2–3 consecutive measurements. Flow time was taken manually with a digital stopwatch with a resolution of 10 ms (measurement uncertainty of 0.1 s, taking into account the human factor).

To minimise chemical changes of the samples at elevated temperatures, the setup was supplemented by providing an inert gas environment above the capillary viscometer (nitrogen with a purity of 99.999% was used as an inert gas). For this, the same balloon (a common party balloon) was connected to both viscometer tubes (a capillary tube and a filling tube) using silicone tubes. The pressure, under which the measurements were performed, was therefore the sum of the atmospheric pressure and the pressure created by the balloon (ca 0.07 bar). It also allowed the silicone tubes to be closed (with clamps) near the viscometer tubes to minimize compositional changes due to evaporation during sample thermal equilibration periods.

Kinematic viscosity measurements were performed in the overall temperature range of from -10 to 160 °C. The highest measurement temperature for a particular sample was conventionally determined by the minimum flow time of about 200 seconds (about 20% lower than the value recommended by the manufacturer). The temperature control and measurement subsystem consisted of a glycerin-filled temperature-controlled bath. The bath was a glass vessel containing a cooling coil, a mixer, a heating element and two temperature measuring probes. The temperature of the bath was controlled by heating (temperature stability in the vicinity of the viscometer better than ± 0.1 °C, by Julabo temperature controller LC4) and the temperature in the vicinity of the viscometer was measured with a PT100 (standard uncertainty ± 0.1 °C; temperature recording resolution 0.01 °C). For the given test setup, the standard uncertainty of the viscosity measurement system was tested against distilled water and n-octane, which supported the uncertainty of 0.2%.

Considering the above, the supplementary commentary article provides slightly corrected kinematic viscosity data in Appendix. The kinematic viscosity data in Appendix (Tables A1 and A2) are derived using the temperature dependence of the viscometer conversion factor (determined from the manufacturer provided conversion factors at 40 °C and 100 °C). In the initial article [1], only the manufacturer provided conversion factor factor factor at 40 °C was used to derive kinematic viscosity data over the temperature range measured and therefore a systematic error larger than "The expanded uncertainty of the capillary viscometer was +/-0.3% [1]" was introduced.

Kinematic viscosity was converted to dynamic viscosity based on density data measured with a DMA 5000 M densiometer (Anton Paar GmbH, Switzerland). The density measurement temperature range for samples with a molecular weight greater than 100 g/mol (or specific gravity greater than 0.76) was between 15.6 °C and 80 °C, but the measurement range for lighter fractions (i.e. the first three fractions) was shifted to a lower temperature. To find the density at the viscosity measurement temperature, a density temperature dependence equation (empirical equation) was derived from the data for each sample. The estimated standard uncertainty of the densities found by curve fitting was 0.0002 g/cm³. (For gasoline fractions, the estimated standard uncertainty of the densities measured was 0.0001 g/cm³ [15]).

Supplement to subchapter 2.3. Other characterisation data

Different properties were measured/determined for pseudocomponents, i.e. fractions with a narrow boiling range obtained during distillation. In connection with the derivation of viscosity determination equations, number average molecular weight, carbon number, hydrogen-carbon ratio, density (as specific gravity) and refractive index (as refractive index parameter) could be highlighted from the measured properties of all fractions [11]. In addition, average boiling points were determined experimentally for all the fractions obtained by rectification. The average boiling point, a definitionbased property for pseudocomponent characterisation, was calculated for the rectification samples as arithmetic means of initial and final collecting temperatures (condensing temperatures) of the fractions with narrow boiling ranges during rectification (i.e. as arithmetic means of temperatures measured in the condenser by a K-type thermocouple, giving a standard uncertainty of these average boiling points of ± 0.5 °C) [14, 16]. Note that neither the average boiling point nor the initial collecting temperature of the cut has a direct quantitative relation with the boiling point of the fraction, which is also a parameter applied in viscosity estimation methods [15, 17]. Note also that for some fractions obtained by rectification, the boiling points have been determined from measured vapor pressure curves as well [17]. For simple batch distillation narrow boiling range fractions, average boiling points were not given as the above approach is not reliable for simple batch distillation (more specifically, without an empirical correcting equation [11]), although the initial and final collecting temperatures of fractions were recorded [18].

Supplement to chapter 3. Results and discussion

The viscosity temperature dependence of the measured samples follows well the Arrhenius-type behaviour (a two-parameter exponential equation known as Andrade viscosity equation [19]) over the temperature ranges measured. This linear relationship, when the natural logarithm of viscosity is plotted against the reciprocal value of the temperature, is illustrated in the Figure. It should be noted here that for several fractions, only the viscosity values at the highest measurement temperatures of these fractions slightly deviated from the straight line. Since this occurred randomly among the samples studied, these deviations were likely experimental artifacts caused by changes in the composition of the samples due to the more intense evaporation near the boiling point in these cases. Therefore, in general, the application of a three-parameter exponential equation (an Antoine-type correlation) or double-exponential equations (i.e. double-logarithmic regressions) could be considered as somewhat of an overcomplication of the analysis.

To develop easy to use empirical equations for estimating viscosity, the corresponding constants of Andrade's equation or other more complex

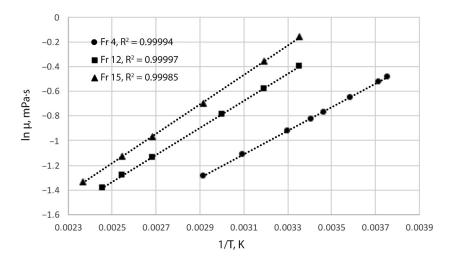


Figure. Natural logarithm of dynamic viscosity of rectification fractions 4, 12 and 15 as a function of the reciprocal value of temperature. The coefficient, R-Squared, is shown as goodness-of-fit.

empirical forms, which are applied to represent the variation of liquid viscosity upon temperature, can be given as a function of the properties/characteristics of the fraction (one or more properties/characteristics, which may include among others also viscosity at a certain temperature or combined/calculated properties such as the Watson characterisation factor or API gravity). There are several dozen regression equations that have been proposed to model viscosity of liquids, including petroleum fractions, from which to choose [19–22]. Conventionally, the best-fitting model with the simplest form (that adequately describes the change in viscosity with temperature) is chosen. When applying these empirical estimation methods (or evaluating their application), one should consider not only statistical evaluation parameters (for example, percent average absolute deviation), but also applicationspecific recommendations and information, such as the application range and background information about the data used to develop these methods (type or family of compounds, number of data points, range and distribution of their values, etc.).

Note that due to the different separation efficiencies of simple distillation and rectification, these distillation methods give samples (narrow boiling range gasoline fractions) with somewhat different chemical composition and properties. Firstly, while the distribution of boiling temperatures of the fractions obtained by rectification is narrower and more like a Gaussian distribution, then the fractions obtained by simple distillation have a wider distribution of boiling temperatures and the shape is more skewed to the right [8, 15, 16]. Secondly, unlike fractions from simple distillation, there is no monotonic change in the so-called energy properties (such as density or refractive index [11, 13]) in the successive series of rectification fractions, because different classes of compounds dominate the fractions as a result of separation efficiency. It is more dominant in the lower boiling fractions and levels off in the higher boiling fractions. Therefore, based on the above, empirical equation constants that are a function of more than one pseudocomponent's property are in principle more appropriate, on the basis of improving the predictability of viscosity estimation [11, 13].

Supplement to the Acknowledgement section

The research was supported by the National Research and Development Program "Energy" through the Project No. AR10129 "Investigation of thermodynamic properties important for the future of the oil shale industry". The corresponding financial support is gratefully acknowledged. Estonian Energy Ltd. is thankfully acknowledged for providing straight run naphtha fractions of Kukersite oil shale retort oil.

The PI of the Project No. AR10129 (V. Oja) is very grateful to the participants of the project from the former Chemical Engineering Department of Tallinn University of Technology: in the context of the viscosity article of Kukersite oil shale gasoline fractions, especially to Dr Alexey Yanchilin for viscosity measurements and constructing the viscosity measurement setup, to MSc Sven Kamenev and Dr Eduard Tearo for their help in constructing the viscosity measurement setup, to Dr Alfred Elenurm for his scientific advice, and to Mr Einart Lindaru, Mrs Ilme Rohtla and Dr Rivo Rannaveski for their contribution to sample characterization.

The publication costs of this Addendum were covered by the Estonian Academy of Sciences.

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Appendix

Table A1. Experimental viscosity data for Kukersite oil shale gasoline fractions prepared by the simple distillation at atmospheric pressure (i.e. for the samples given in the article [1] with the identifier GD0513). Standard uncertainty for temperature $(T, ^{\circ}C)$ is $\pm 0.1 ^{\circ}C$, for kinematic viscosity (v, cSt) is $\pm 0.2\%$ and for dynamic viscosity (μ , mPa·s) is $\pm 0.2\%$.

	Fr2			Fr3			Fr4			Fr5	
T, ⁰C	v, cSt	μ, mPa·s	T, ∘C	v, cSt	μ, mPa·s	T, ⁰C	v, cSt	μ, mPa·s	T, ∘C	v, cSt	μ, mPa·s
0.9	0.6440	0.4892	0.8	0.7327	0.5692	30.1	0.5964	0.4546	25.1	0.6297	0.4910
20.4	0.5288	0.3919	25.1	0.5663	0.4272	50.0	0.5001	0.3722	40.0	0.5490	0.4208
39.9	0.4544	0.3283	50.0	0.4593	0.3358	6.69	0.4296	0.3123	64.9	0.4493	0.3345
59.9	0.3978	0.2799	74.8	0.3857	0.2732	89.7	0.3767	0.2670	89.7	0.3796	0.2743
79.7	0.3684	0.2523	89.8	0.3583	0.2489	109.6	0.3475	0.2398	109.7	0.3461	0.2440
	Fr6			Fr7			Fr8				
T, °C	v, cSt	μ, mPa·s	T, ⁰C	v, cSt	μ, mPa·s	T, ⁰C	v, cSt	μ, mPa·s			
25.1	0.6969	0.5532	25.1	0.9463	0.7644	25.1	1.104	0.9073			
40.0	0.6023	0.4704	40.0	0.7945	0.6319	40.0	0.9128	0.7389			
64.9	0.4868	0.3698	69.8	0.5943	0.4578	69.8	0.6677	0.5241			
89.7	0.4074	0.3007	99.7	0.4711	0.3511	99.7	0.5210	0.3962			
109.7	0.3596	0.2594	139.4	0.3691	0.2629	139.4	0.4019	0.2925			

(i.e. for the samples given in the article [1] with the identifier GR060613). Standard uncertainty for temperature (T, °C) is ± 0.1 °C, for kinematic viscosity (v, cSt) is $\pm 0.2\%$ and for dynamic viscosity (µ, mPa·s) is $\pm 0.2\%$. For some fractions, normal boiling points (T_b, Table A2. Experimental viscosity data for Kukersite oil shale gasoline fractions prepared by the rectification at atmospheric pressure °C) that were available (measured with the vapor pressure tester ERAVAP [10,15]) are also given.

Fr1			Fr2			Fr3			Fr4	
, cSt	μ, mPa·s	T, ⁰C	v, cSt	μ, mPa·s	T, °C	v, cSt	μ, mPa·s	T, °C	v, cSt	μ, mPa·s
.4830	0.3472	-10.0	0.6032	0.4442	-10.0	0.7093	0.5480	-6.9	0.7817	0.6174
.4699	0.3364	-4.0	0.5663	0.4137	-4.0	0.6621	0.5077	-4.0	0.7545	0.5938
0.4309	0.3041	0.9	0.5384	0.3908	0.9	0.6268	0.4776	5.8	0.6712	0.5219
0.3976	0.2766	10.7	0.4909	0.3515	10.7	0.5665	0.4262	15.6	0.6043	0.4641
0.3826	0.2642	20.5	0.4512	0.3187	20.5	0.5172	0.3842	20.5	0.5759	0.4396
		30.1	0.4177	0.2912	30.1	0.4764	0.3494	30.1	0.5274	0.3977
		39.9	0.3879	0.2666	39.9	0.4396	0.3182	50.0	0.4467	0.3283
					50.0	0.4080	0.2913	6.69	0.3861	0.2763
$(T_{\rm b} = 95^{\circ})$	°C)	H	7 T6 (T _b = 105 4	C)		Fr7		H	$r8 (T_b = 119^{\circ})$	C)
v, cSt	μ, mPa·s	T, ∘C	v, cSt	μ, mPa·s	T, °C	v, cSt	μ, mPa·s	T, °C	v, cSt	μ, mPa·s
0.7344	0.5640	0.8	0.8037	0.6373	25.1	0.6526	0.5131	25.1	0.6756	0.5235
0.5955	0.4465	25.1	0.6201	0.4779	40.1	0.5657	0.4372	40.1	0.5850	0.4455
0.5003	0.3661	50.0	0.4941	0.3696	59.9	0.4791	0.3617	59.9	0.4934	0.3671
0.4290	0.3060	74.8	0.4111	0.2983	79.8	0.4154	0.3061	79.8	0.4254	0.3090
0.3742	0.2600	80.8	0.3725	0.2652	99.7	0.3655	0.2628	7.66	0.3728	0.2642
								109.6	0.3522	0.2465
	Fr1 v, cSt 0.4699 0.4699 0.4309 0.3976 0.3826 0.3826 0.3826 0.3826 0.3744 0.5955 0.5003 0.4290 0.3742		μ, mbass T, °C μ, mbass T, °C 0.3472 -10.0 0.3364 -4.0 0.3364 -4.0 0.2766 10.7 0.2642 20.5 0.2642 20.5 0.2642 30.1 39.9 39.9 μ, mPass T, °C μ, mPass T, °C 0.5640 0.8 0.3661 50.0 0.3060 74.8 0.2600 89.8	μ, mbass T, °C μ, mbass T, °C 0.3472 -10.0 0.3364 -4.0 0.3364 -4.0 0.2766 10.7 0.2642 20.5 0.2642 20.5 0.2642 30.1 39.9 39.9 μ, mPass T, °C μ, mPass T, °C 0.5640 0.8 0.3661 50.0 0.3060 74.8 0.2600 89.8	H, mPars T, $^{\circ}C$ $Fr2$ μ , mPars T, $^{\circ}C$ v , cSt 0.3472 -10.0 0.6032 0.3364 -4.0 0.5663 0.3364 -4.0 0.5663 0.3364 -4.0 0.5663 0.3364 0.9 0.5384 0.27666 10.7 0.4909 0.2642 20.5 0.4512 0.2642 30.1 0.49177 0.2642 30.1 0.4177 0.2642 30.1 0.4177 0.2642 30.1 0.4177 0.4417 0.44177 0.62037 0.6201 0.5640 0.8 0.5640 0.8 0.4465 25.1 0.6201 0.3661 50.0 0.4941 0.3060 74.8 0.4111 0.2600 89.8 0.3725	Hr2 Fr2 $\mu, m Pa:s$ $T, \circ C$ v, cSt $\mu, m Pa:s$ 0.3472 -10.0 0.6032 0.4137 0.3364 -4.0 0.5663 0.4137 0.3364 -4.0 0.5563 0.4137 0.3364 -4.0 0.5663 0.4137 0.3364 -4.0 0.5663 0.4137 0.3041 0.9 0.5384 0.3908 0.2766 10.7 0.4909 0.3515 0.2766 10.7 0.4909 0.3515 0.2642 20.5 0.4512 0.3187 0.2642 30.1 0.4177 0.2906 10.7 0.4512 0.3187 0.26666 $4, m Pa:s$ $T, \circ C$ v, cSt $\mu, m Pa:s$ $\mu, m Pa:s$ $T, \circ C$ v, cSt $\mu, m Pa:s$ 0.4446 0.56400 0.8037 0.6373 0.6373 0.4779 0.30601 50.0 0.49411	Fr2 Fr2 $\mu, m b a:s$ $T, \circ C$ $v, c St$ $\mu, m b a:s$ $T, \circ C$ 0.3472 -10.0 0.6032 0.4137 -10.0 0.3364 -4.0 0.5663 0.4137 -4.0 0.3364 -4.0 0.55663 0.4137 -4.0 0.3364 -4.0 0.55663 0.4137 -4.0 0.3364 -4.0 0.55633 0.4137 -4.0 0.3364 -4.0 0.55663 0.4137 -4.0 0.2766 10.7 0.4909 0.3515 10.7 0.2642 20.5 0.4512 0.3187 20.5 0.2642 30.1 0.4177 0.2915 30.1 0.2642 30.1 0.4177 0.2956 39.9 0.2642 30.1 0.4177 0.2956 39.9 0.2640 9.8379 0.26666 39.9 9.9 $\mu, m P a:s$ $T, \circ C$	H, mPa:s $Fr2$ $Fr2$ $Fr3$ $\mu, mPa:s$ $T, \circ C$ v, cSt $\mu, mPa:s$ $T, \circ C$ v, cSt 0.3472 -10.0 0.6032 0.4442 -10.0 0.7093 0.3364 -4.0 0.5663 0.4137 -4.0 0.7093 0.3364 -4.0 0.5663 0.4137 -4.0 0.7093 0.33041 0.9 0.5384 0.3908 0.9 0.6621 0.3041 0.9 0.5384 0.3908 0.9 0.6621 0.3041 0.9 0.5384 0.3908 0.9 0.6621 0.3041 0.9 0.5387 0.3187 20.5 0.5172 0.2642 20.5 0.4177 0.295 0.4396 0.4366 30.1 0.4177 0.2956 39.9 0.4396 0.6436 0.2640 0.3879 0.2366 39.9 0.4396 0.6436 1.11	H: Mindase Fr2 Fr3 Fr3 <th< td=""><td>Hars Fr2 Fr3 Fr3 Fr3 Hambars T, °C v, c5t µ, mbars T, °C 0.34137 -4.0 0.5663 0.4137 -4.0 0.5663 0.4776 5.8 0.303 0.33187 0.303 0.3515 10.7 0.5665 0.4766 5.8 0.4776 5.8 0.4764 5.8 0.1 0.0<</td></th<>	Hars Fr2 Fr3 Fr3 Fr3 Hambars T, °C v, c5t µ, mbars T, °C 0.34137 -4.0 0.5663 0.4137 -4.0 0.5663 0.4776 5.8 0.303 0.33187 0.303 0.3515 10.7 0.5665 0.4766 5.8 0.4776 5.8 0.4764 5.8 0.1 0.0<

	μ, mPa·s	0.6505	0.5475	0.4053	0.3155	0.2728	0.2560	
Fr11	v, cSt	0.8098	0.6926	0.5300	0.4269	0.3778	0.3589	Fr15
	T, ⁰C	25.2	40.0	6.69	99.7	119.5	129.5	
°C)	μ, mPa·s	0.6028	0.5084	0.3797	0.2969	0.2575		
Fr10 (T _c = 129 °C)	v, cSt	0.7643	0.6553	0.5063	0.4101	0.3644		Fr14
FI	T, °C	25.2	40.0	6.69	99.7	119.5		
C)	μ, mPa·s	0.5512	0.4674	0.3840	0.3226	0.2762	0.2572	
Fr9 ($T_{h} = 123 \text{ °C}$)	v, cSt	0.7181	0.6193	0.5207	0.4480	0.3930	0.3705	Fr13
Ľ.	T, °C	25.1	40.0	59.9	79.8	7.99	109.6	

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1					
ι, mPa·s	0.6715	0.5618	0.4551	0.3209	0.2776

0.8399

25.1

0.7141 0.5911

40.0 59.9 99.7

v, cSt

T, °C

Fr12 ($T_{\rm h} = 141 \, {}^{\circ}\text{C}$)

0.2504

0.3545

0.3860

119.5 134.4

0.4360

Fr16

μ, mPa·s 0.9292 0.7615

v, cSt

T, °C

μ, mPa·s

v, cSt

T, °C

μ, mPa·s

v, cSt

T, °C

μ, mPa·s
 0.7112
 0.5917
 0.4767
 0.3347
 0.2858
 0.2581

T, °C

1.134

25.1

0.8520 0.6987 0.4994

1.038

25.1

0.7884

0.9723

25.1

0.5350 0.4003 0.3398 0.2673

0.9432 0.6832

40.0 69.9 99.7

0.8645

40.0 69.9 99.7

0.6499

0.8140 0.6638 0.5582

40.0 59.9 79.8 99.7

0.7562 0.6225 0.4570 0.3994 0.3670

40.0 59.9 99.7

v, cSt 0.8946

25.1

0.5190 0.4271

0.6373

0.2608

0.3672

0.3721

0.4576

119.5 149.4 159.3

0.4368

119.5 149.4

0.3589

0.4794

119.5 134.4

0.3668

0.5277

0.3796 0.3243 0.2633

0.5001