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INVESTIGATION ON PRECISION OF DETERMINATION OF SARA ANALYSIS OF VACUUM RESIDUAL OILS FROM DIFFERENT ORIGIN

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Abstract
This study examines the repeatability of SARA analysis performed in LUKOIL Neftohim Burgas Research Laboratory of straight run and H-Oil unconverted vacuum residual oils (VROs), which have hydrogen content between 10.5 and 12.8%, and saturate content between 26.5 and 62.9%. The repeatability was found to be 1.8% for saturates; 2.4% for aromatics; 1.2% for resins; and 0.7% for asphaltenes. Hydrogen content was found to correlate with the VRO saturates content and this correlation could be used for assessment of the correctness of the performed SARA analysis. SARA separation of the VROs showed to be a useful technique for further more detailed analysis of the VRO SARA fractions that may help unveil the ultra-complex chemical nature of the vacuum residual oils.

Keywords: vacuum residual oil; SARA composition; repeatability; SARA fraction element composition.

1. Introduction

The characterization of residual oils by their separation into the fractions: saturates, aromatics, resins (polars) and asphaltenes (SARA) has been widely used in residue process studies [1]. The vacuum residual oil SARA analysis data has been reported by Xu et al. to correlate with the yields of the residue fluid catalytic cracking (RFCC) [2]. The vacuum residual oil SARA analysis data have been also reported to correlate with the thermal cracking conversion and the thermal condensation conversion [3]. The SARA analysis data of oils have been used by Watkinson *et al.* [4] to introduce the colloidal instability index CII [4] that was found to be a useful empirical parameter for correlating fouling and precipitation results [5-7]. Therefore SARA analysis could be considered as an important method for characterization of residual oils. There are numerous variations of this type of analysis [8]. However little is published about the precision of the SARA analysis method applied to vacuum residual oils from different origin. The LUKOIL Neftohim Burgas (LNB) Research laboratory adapted a group hydrocarbon method, originally developed for analysis of heavy distillate, for SARA analysis of residual oils [9]. This method has been employed in several resid process studies of the LNB Chief Process Engineer department [10-13]. It has been used in this study to define the precision of the SARA analysis method by multiple analyses of three straight run vacuum residual oils (VROs) from the crude oils Vald'Agri (Italy), Kazakh heavy (Kazakhstan), and Caspian heavy (Azerbaidjan), and of one H-Oil unconverted vacuum residual oil. This study also includes data for SARA analysis of 20 vacuum residual oils – feeds and unconverted products from the LNB ebullated bed residue hydrocracking H-Oil unit. The aim of this paper is to determine the precision (repeatability) of the SARA analysis method applied to the vacuum residual oils mentioned above and specify the indicators for evaluation of the correctness of the performed SARA composition measurements.

2. Experimental

2.1 Deasphaltization of the vacuum residual oils under study

Before the liquid chromatography separation of the VROs in saturates, aromatics and resins they were deasphalted. The deasphaltization was carried out by the following procedure: 35 grams of vacuum residue are put in a flask of 1 liter inserted in a heater and equipped with a reflux condenser. 700 ml of n-heptane is added to the residue in the flask. The obtained mixture is homogenized by the use of a mechanical mixer and when the temperature of the blend reaches 98°C (the boiling point of n-heptane) it remains at that temperature for an hour. Then the heater is switched off and the mixture stays in a dark place for 12 hours at ambient temperature to settle down the asphaltene fraction. Afterwards the mixture is filtrated to remove asphaltenes after their washing with n-heptane till the solution becomes colorless. The mixture of deasphalted oil with n-heptane is separated by distilling the lower boiling n-heptane. The asphaltene yield is calculated by the equation:

$$Asphaltene_{yield} = \frac{M_1 - M_0}{M_{VRO}} \cdot 100$$

(1)

where: asphaltene$_{yield}$ = the yield of asphaltenes in wt.%; M_1 = the weight of the filter with asphaltenes on the filter, g; M_0 = the weight of the empty filter, g.

2.2. Determination of the content of saturates, aromatics, and resins in the de-asphalted vacuum residual oils

The hydrocarbon group composition of the deasphalted vacuum residual oils was determined by the use of liquid adsorption chromatography on silica gel following the procedure: The sample oil in amount of about 6g is diluted in n-hexane in ratio 1:3 and charged to a glass percolation column containing 80-85 g silica gel (silica gel 60 Fluka, particle size 35-70 mesh ASTM). After the whole sample quantity soaks the silica gel 350-450 ml of n-hexane (99% Fluka) is charged to the column for desorption of saturates, 200 ml of toluene (98.5-99.9%) for desorption of aromatics, and 200 ml ethanol (99.7%)-toluene mixture 50 to 50 by volume for desorption of resins. Then 100 ml ethanol is charged to the column for final washing. The effluent is collected in glass bottles (20 – 25 pieces). The solvents are completely removed from the recovered n-hexane, toluene, ethanol-toluene (50/50 vol./vol.) fractions by distillation and residues are weighed. The eluted compounds with the n-hexane could be considered as saturates. Those eluted with toluene could be considered as aromatics, and the compounds eluted with the ethanol-toluene (50/50 vol./vol.) blend are classified as resins. More precise determination of the content of saturates and aromatics can be made by the use of refraction. Saturates are these fractions that have nd$_{20}$ ≤ 1.49. Light aromatics are the fractions that have nd$_{20}$ between 1.49 and 1.53. Middle aromatics are the fractions that have nd$_{20}$ between 1.53 and 1.59. Heavy aromatics are the fractions that have nd$_{20}$ > 1.59. Resins fraction follows the heavy aromatics fraction and its refraction cannot be determined because of the dark colour. That is why the resins content is determined on the base of the balance. The total weight of all the recovered fractions must be between 97 and 103% of the sample charged. If this recovery is not obtained, the test is repeated.

The experimental method of HTSD (ASTM D 7169) applied in this work is described in detail in [20]. The element composition of the VROs and their fractions studied in this work was measured in accordance with ASTM D 5291 (hydrogen, carbon, and nitrogen content), and ASTM D 1552 (sulphur content).

3. Results and discussion

There are two ASTM standards (ASTM D 2007, and ASTM D 2549) which employ clay-silica gel chromatography to separate and determine hydrocarbon group fractions in heavy oils [14-15]. The ASTM D 2007 is used to classify oil samples of initial boiling point of at least 260°C (500°F) into the hydrocarbon types of polar compounds, aromatics, saturates, and asphaltenes [14].The

ASTM D 2549 covers the separation and determination of representative aroma-tics and non aromatics fractions from hydrocarbon mixtures that boil between 232 and 538°C [15]. From these two methods only ASTM D-2007 is applicable for SARA analysis of residual oils, since typically the residual oils contain considerable amount of components boiling above 538°C. Unfortunately the ASTM D 2007 standard does not provide data for the precision in determination of the different SARA fractions in asphaltene containing heavy oils. For that reason we measured four times the SARA composition of the four vacuum residual oils mentioned in Introduction section by the silica-gel chromatographic method described in Experimental section. The results of the measured SARA compositions of the four studied VROs are presented in Table 1. By the use of equations 2, 3, and 4 and the data from Table 1the statistic parameters standard deviation (SD) and confidence limits (CL) were estimated.

$$SD = \pm\sqrt{\sum_i^n \frac{(xi - x_{mean})^2}{df}} \qquad (2)$$

where, SD=standard deviation; xi =ith measurement of saturates, aromatics, resins, and asphaltenes, wt.%; df = degree of freedom.

$$df = n - a \qquad (3)$$

n = number of measurements (in our case n = 16)
a = number of estimated parameters (in our case the estimated parameters were four = arithmetic means of the four SARA compositions of the four studied VROs.)

$$CL = \frac{SD \cdot t}{\sqrt{n}} \qquad (4)$$

t = t-value from t-distribution at probability of 95%.

Table 1 Data for SARA analysis of four different VROs measured four times each

VRO	Exp., №	SARA, wt., %			
		Saturates	Aromatics	Resins	Asphaltenes
Val'd Agri	1	25.1	61	8.8	5.2
	2	26.7	59.7	8.7	4.9
	3	27.1	58.8	9	5.1
	4	27.2	59.5	8.3	4.9
Average for Val'd Agri		26.5	59.7	8.7	5
Kazakh	5	48.6	40.9	8	2.6
	6	47.1	40	10	2.9
	7	49.3	38	10.1	2.7
	8	48.9	38.3	9.8	3
Average for Kazakh		48.5	39.3	9.5	2.8
Caspian Heavy	9	32	50	9.6	8.5
	10	32	49.8	9.7	8.6
	11	32.6	49	9.9	8.5
	12	32.1	48.5	10.3	9.1
Average for Caspian Heavy		32.2	49.3	9.9	8.7
H-Oil VTB	13	62.5	31.6	3.7	2.3
	14	62.3	30.6	3.7	3.4
	15	62.9	30.5	3.5	3.1
	16	64	28.8	3.9	3.4
Average for H-Oil VTB		62.9	30.4	3.7	3

Table 2 summarizes the estimated by eqs. 2-4 statistical parameters SD, 2SD, and CL. The parameter CL can be considered as an indicator for the repeatability of the studied in this work SARA analysis method. In some cases 2SD has been used as an indicator for the repeatability [16]. In fact 2SD equals to CL when the number of measurements is not lower than 60. However as evident from the data in Table 2 the ratio between CL and 2SD is very close to unity = 1.1. Therefore the assumption that 2SD ≈ repeatability could be considered correct. Table 2 also contains data of repeatability of the ASTM D 2007 method, applied to oil samples which do not contain asphaltenes. These data indicate that both methods ASTM D 2007 and that investigated in this work have almost the same repeatability. However the question about the proper separation between the fractions in the SARA analysis still remains open. In order to assess the precision of the separation between the saturate and the aromatic fractions in the studied method we used refraction (n_d^{20}) of the fractions as a criterion. For most samples it was impossible to measure the refraction of the fractions. That is why we use the methodology described in ref.9, where the fractions were blended with hydrotreated VGO (HTVGO) and their nd_{20} was estimated from the refraction of the blend and that of the HTVGO as described in [9]. It should be noted here that the use of the refraction elongated the time for performance of the SARA analysis from four to five days. Data for SARA composition of the four studied VROs by the use of the refraction are presented in Table 3.

Table 2 Standard deviation (SD), double standard deviation (2SD), and confidence limits (CL) estimated by eqs. 1-3 for the method studied in this work, and repeatability of ASTM D 2007

Hydrocarbon group	SD	2 SD	CL	Repeatability according to ASTM D-2007
Saturates, wt.%	0.8	1.6	1.8	2.1
Aromatics, wt.%	1.1	2.1	2.4	2.3
Resins, wt.%	0.6	1.1	1.2	1.2
Asphaltenes, wt.%	0.3	0.6	0.7	-

Table 3 SARA composition of the four studied VROs with the use of refraction for defining the separation between saturates and aromatics.

VRO	Saturates, wt.%	Light Aromatics, wt.%	Middle + Heavy Aromatics, wt.%	Resins, wt.%	Asphaltenes, wt.%
Val'd Agri	10.0	6.8	69.9	8.3	4.9
Kazakh	25.7	17.4	44.0	9.8	3.0
Caspian Heavy	16.8	9.8	55.0	9.9	8.5
H-Oil VTB	40.1	16.7	36.6	3.5	3.1

It is evident from these data that without use of the refraction the saturate content of the VROs gets overestimated by the applied SARA method. Light aromatics and minor amount of middle aromatics can be eluted with the saturate fraction, if no refraction is applied to define the separation. Figure 1 presents a graph of the dependence of the difference between saturate content measured without refraction and with refraction on the saturate content of the VRO measured without using the refraction. Seven VRO SARA analyses were used to make this graph. The regression line from Figure 1 could be applied to correct the saturate content if no refraction is employed.

A sample of VRO from Urals crude was analyzed in three laboratories applying the SARA procedure described in this work (LNB Research laboratory (RL)), ASTM D 2007 (Laboratory Nr.2) and the SARA procedure exploited in ref. 17 (Laboratory Nr.3). The results from the SARA analyses performed in the three laboratories are given in Table 4. It is evident from

these data that LNB RL (the studied procedure with application of refraction) and Laboratory Nr.3 obtained practically the same saturate content (the difference of 0.7% is within uncertainty of the measurement) and the same asphaltene content. However a difference exists between determination of aromatics and resins between LNB RL and Laboratory Nr.3.
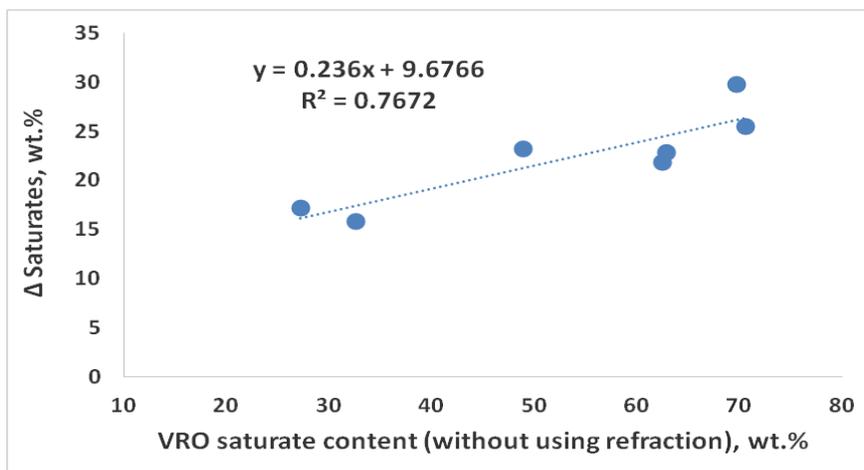

Figure 1 Dependence of the difference between VRO saturate content without and with the use of refraction on the VRO saturate content measured without using refraction.

Table 4 Results from SARA analysis of Urals VRO performed in three different laboratories

Hydrocarbon group	LNB RL		Laboratory Nr.2 ASTM D 2007	Laboratory Nr.3
	without refraction	with refraction		
Saturates, wt.%	29.8	13.1	17.1	12.4
Aromatics, wt.%	52.9	69.6	44.6	45.1
Resins, wt.%	12.3	12.3	20.0 (30.0*)	35.9
Asphaltenes,wt.%	5.0^1	5.0^1	18.3^2 (8.3*)	4.7^1

[1] C_7 asphaltenes; [2] C_5 asphaltenes
* Note: The asphaltene fraction content was corrected to account that asphaltenes in ASTM D 2007 are C_5 asphaltenes. The resin fraction content was corrected with addition of the difference between C_5 and C_7 asphaltenes

The aromatics content measured in accordance with ASTM D 2007 (Laboratory Nr.2) and that measured in Laboratory Nr.3 is practically the same. However, the difference in the asphaltene content measured in Laboratory Nr.2 and that measured in Laboratory Nr.3 is more than three times as high as that in in Laboratory Nr.3. It should be noted here that asphaltenes according to ASTM D 2007 procedure are C_5 asphaltenes while those measured in Laboratory Nr.3 and in LNB RL are C_7 asphaltenes. If a ratio of 2.2 as determined in ref. 13 between C_5 and C_7 asphaltenes for Urals VRO is assumed then asphaltenes in the sample analyzed in Laboratory Nr.2 would become 8.3% and the difference between C_5 and C_7 asphaltenes would be added to the resin (polar) fraction. Then the difference between the content of resin fraction from Laboratory Nr.3 and from Laboratory Nr.2 would become 5.9% which is again higher than the reproducibility of the ASTM D 2007 method (polars reproducibility = 1.8%). Obviously the separation between aromatics and resins is different in the three compared methods (the one studied in this work, the ASTM D 2007 and that used in Laboratory Nr.3). ASTM D-2007 uses the attapulgus clay and eluting solvent for oils, but a mixture of toluene and acetone 50/50 vol/vol to desorb resins. The Laboratory Nr.3 method uses activated alumina and silica gel and n-heptane to elute the saturates fraction, a blend of 2/1; vol./vol.; n-heptane/ toluene to elute aromatics fraction, and a blend of 1/1/1 ; vol./vol./vol. toluene/methylene

chloride/methanol to desorb resins. The method studied in this work (LNB RL) most probably underestimates the content of resins since toluene is used to elute aromatics. Goual and Firoozabadi [18] have shown in their work that toluene elutes not only aromatics but also some resins. This can explain the big difference between resins content in the LNB RL method and that of the other two methods: ASTM D 2007 and that of Laboratory Nr.3. The repeatability of the method used by Laboratory Nr.3 is 1.5% for the three fractions: saturates, aromatics, and resins [19], and 1% for asphaltenes [21], which is close to the SARA method studied in this work and ASTM D 2007. This suggests that regardless of the difference in the procedures to perform SARA analysis the precision of the measurements is almost the same. As far as resins content measurement is concerned it is difficult to assess which method provides more correct values – ASTM D 2007 or that of Laboratory Nr.3.

Based on the results from LNB RL SARA analysis (without refraction) of 22 vacuum residual oils obtained from individual crude oils, or their blends in different ratio – feeds for the LNB EBR H-Oil hydrocracking unit and unconverted vacuum tower bottom product a graph was made that relates the VRO saturate content with the VRO hydrogen content (Figure 2).
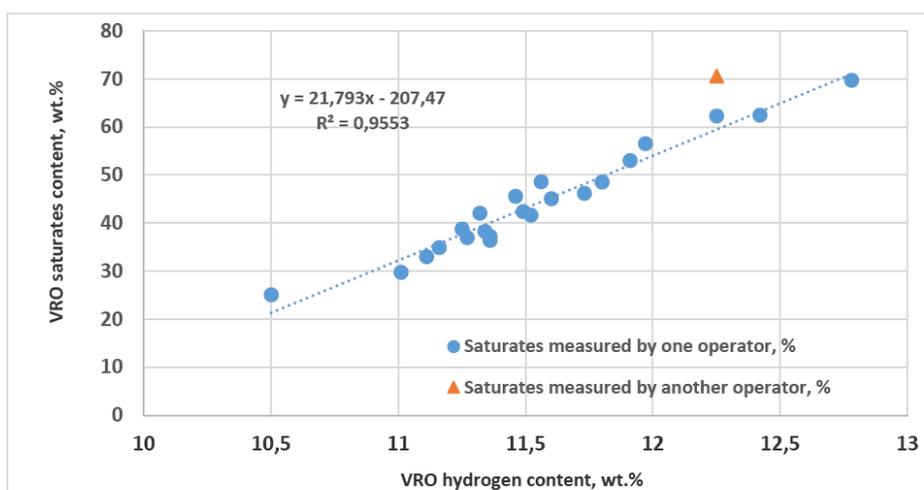

Figure 2 Relation between VRO hydrogen content and VRO saturates content (without using refraction)

The regression line from Figure 2 was used as a tool to assess the correctness of the performed SARA analysis. The H-Oil VTB sample from Table 1 was analyzed for SARA composition by a new operator and the result of this measurement was following:

Saturates, %	Aromatics, %	Resins, %	Asphaltenes, %
70.6	24.1	3.2	1.7

A comparison between these data and those in Table 1 for the H-Oil VTB shows that the difference between saturates, aromatics, and asphaltenes is bigger than the confidence limits from Table 2. The saturate content versus the hydrogen content of this sample apparently deviates from the regression line as shown in Figure 2. A repeating of the SARA analysis of the sample from the same operator gave results similar to those shown in Table 1, which were within the confidence limits. Therefore it could be concluded that a relation like this shown in Figure 2 can be used as an indicator for the assessment of the correctness of the performed SARA analysis. It deserves mentioning here that the vacuum residual oils are the most difficult part of the crude oils for characterization. As shown in an earlier study even simple properties like density and viscosity require special attention when VROs are analyzed [13]. Therefore besides SARA analysis, properties like hydrogen content, density, and Conradson carbon content are recommended to measure and all these properties should exhibit consistency

between each other [13]. In this way error in measurement of SARA analysis or in any of the properties mentioned above could be detected.

Although SARA analysis of VROs is a long analysis – four (without using refraction) or five (with the use of refraction) days are needed to accomplish one SARA analysis, it is a useful means to further investigate the different SARA fractions. In our study one individual straight run VRO (Vald'Agri) and another blended straight run VRO (H-Oil feed) and an unconverted H-Oil VRO product (H-Oil VTB) were separated in saturates, aromatics, resins and asphaltenes and these fractions were analyzed for their element composition (Table 5) and high temperature simulated distillation (HTSD). Figures 3, 4 and 5 present high temperature simulation distillation curves of the fractions saturates, aromatics and resins of the three VROs mentioned above.
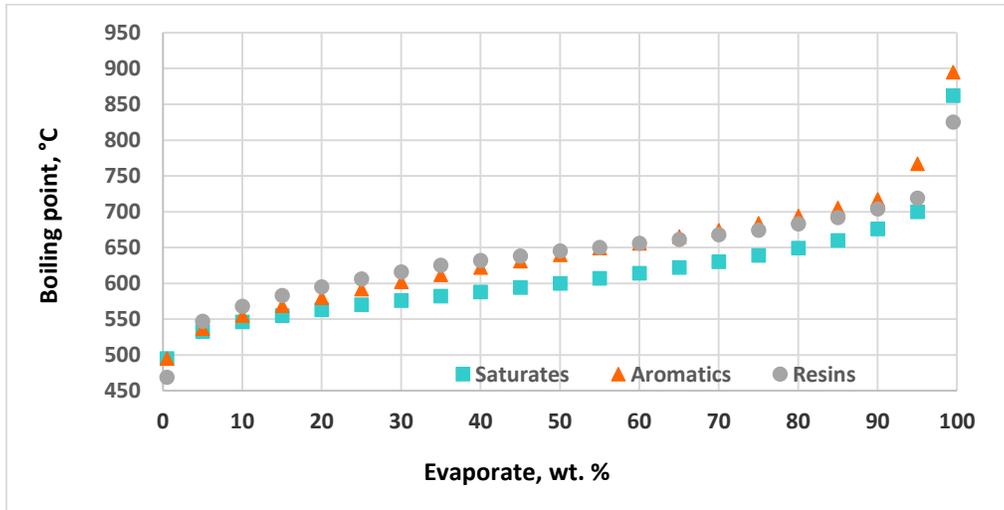

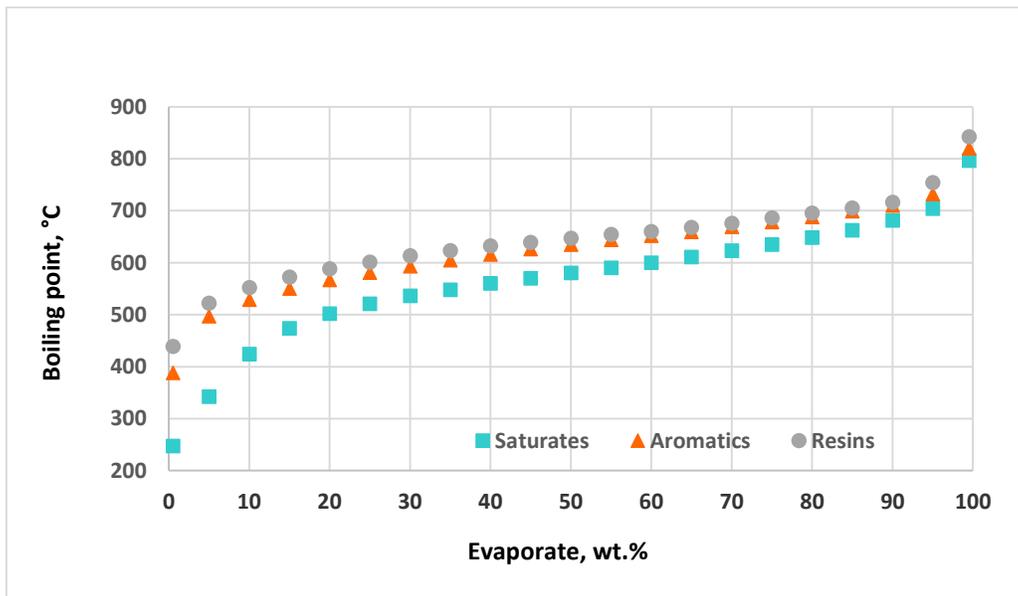Figure 3 HTSD of Vald'Agri VRO saturates, aromatics, and resins fractions

Figure 4 HTSD of H-Oil VRO feedstock saturates, aromatics, and resins fractions
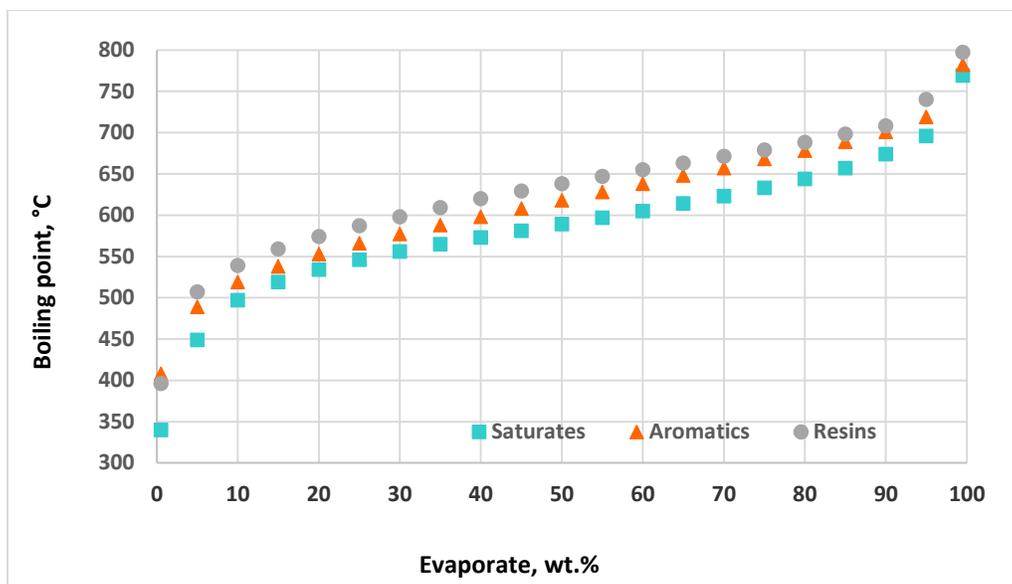
Figure 5 HTSD of H-Oil unconverted VRO (VTB) saturates, aromatics, and resins fractions

Unfortunately with the applied procedure for measurement of HTSD the asphaltene fraction distillation curve was not possible to determine. The data in Figures 3-5 indicate that satura-tes are the lowest boiling point group hydrocarbon fraction. The aromatics have higher than saturates boiling points and lower or equal to the boiling points of the resins. The difference between boiling point of resins and aromatics can be distinct as is the case with the H-Oil unconverted VRO (Figure 5) or slightly perceivable like that in Vald'Agri VRO (Figure 3). If a graph is made on the base of the data for hydrogen content in the SAR fractions and their HTSD T_{50} one can see that there is a relationship between the difference of hydrogen content of the SAR fractions and the difference between HTSD T_{50} of the SAR fractions (Figure 6).

Table 5 SARA composition and element composition of SARA fractions of the three studied VROs

| VROs	SARA, wt.%		C, %m/m	H, %m/m	N, %m/m	S, %m/m	HTSD T_{50}, °C
Val'd Agri	Sat.	26.5	85.13	13.20	< 0.01	-	600
	Aro.	59.7	82.72	9.49	0.02	7.96	640
	Res.	8.7	82.81	9.49	0.70	-	645
	Asp.	5.0	82.90	7.05	0.50	9.31	-
H-Oil Feed	Sat.	36.4	87.73	13.39	< 0.01	1.02	580
	Aro.	47.9	87.46	10.56	0.38	3.71	635
	Res.	7.4	84.01	10.14	1.34	3.36	647
	Asp.	8.5	86.10	7.50	1.20	4.30	-
H-Oil VTB	Sat.	52.6	88.01	13.68	0.01	0.33	589
	Aro.	37.5	89.40	11.83	0.47	1.26	618
	Res.	4.1	85.71	9.80	1.45	1.34	638
	Asp.	5.7	90.10	6.44	1.24	2.30	-
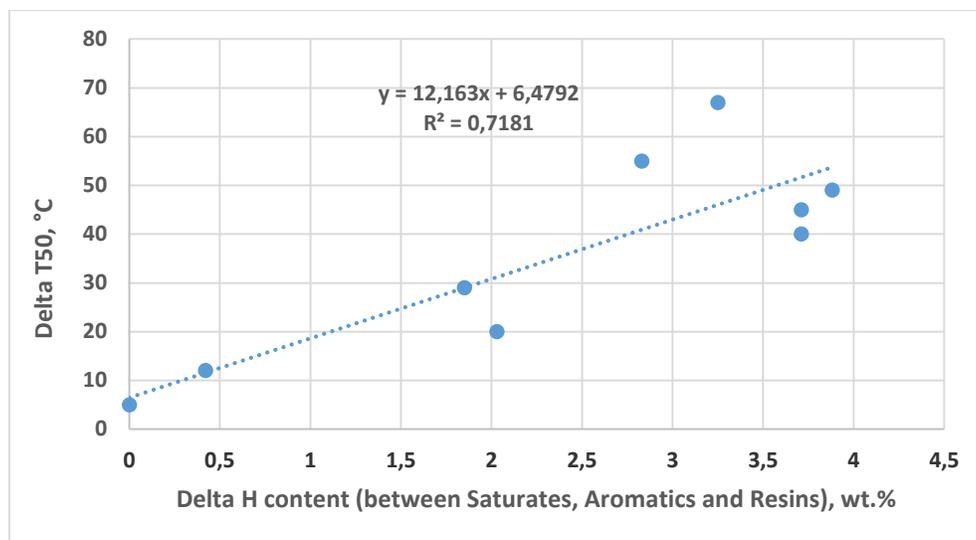
Figure 6 Dependence of the difference between hydrogen content of the SAR fractions on the difference between HTSD T_{50} of the SAR fractions

The data in Figure 6 indicate that the bigger the difference in the hydrogen content of the SAR fractions the bigger is the difference in their HTSD T_{50}. The difference between hydrogen content in resins and in aromatics of the H-Oil VTB sample is the biggest among the three studied VROs and as evident from Figure 5 the difference in the boiling points between resins and aromatics fractions is the most discernible. The data in Table 5 show that asphaltenes have the lowest hydrogen content of all SARA fractions. Therefore a conclusion could be made that asphaltenes should have the highest HTSD T_{50}. These data may illustrate the usefulness of the SARA separation for unveiling of the ultra-complex chemical nature of the vacuum residual oils. Moreover as was shown in this work it is characterized with a good repeatability.

4. Conclusions

Sixteen measurements of SARA composition of four vacuum residual oils from different origin which have hydrogen content between 10.5 and 12.8% were performed to assess the precision of the SARA analysis of vacuum residua. The estimated repeatability was almost the same as that of the ASTM D 2007 method for oils not containing asphaltenes. A comparison between SARA analysis of Urals VRO performed in three laboratories which employ different procedures indicated that the difference in the saturates content between the three laboratories was within uncertainty of the measurement (reproducibility), but the difference between the resins content was much higher than the precision of the measurements. Regardless of the significant difference in the resins content measured in the three laboratories the precision of the SARA methods applied in the three laboratories was almost the same.

Hydrogen content was found to correlate with the VRO saturates content and this correlation proved to be a useful tool for assessment of the correctness of the performed SARA analysis.

SARA separation of the VROs showed to be useful for further more detailed analysis of the VRO fractions. It was found that boiling points of the fractions saturates, aromatics, and resins increases in the order: saturates < aromatics ≤ resins. The higher the aromaticity (the lower hydrogen content) the higher the VRO fraction boiling point is. Based on these findings it could be concluded that asphaltenes, which are the most aromatic part of the VRO should have the highest boiling point among all other hydrocarbon group fractions.

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