INVESTIGATION OF THERMAL STABILITY OF VNII NP-300A GREASE BY THERMOGRAVIMETRIC ANALYSIS

L. I. Nazarova, E. M. Nikonorov, and N. K. Volobuev

UDC 543.876:621.892

The development of gas chromatography methods depends to a considerable extent on the proper selection of stationary liquid phases. This is particularly true in the analysis of high-boiling compounds, which requires thermally stable liquid phases with extremely low volatilities. The Apiezon greases are used widely for this purpose [1-3].

As reported previously [4, 5], we are proposing the replacement of Apiezon Grade L and M greases by the grease VNII NP-300A, which is a high-viscosity mineral oil thickened with high-molecular-weight solid hydrocarbons.

Here we are reporting results from a study of the thermal-oxidative properties of various dispersion media and thickening agents that were approved when the VNII NP-300A grease was developed.

Samples of thermally stable liquid phases used in other countries were used as standards of comparison.

To determine the maximum allowable operating temperature (MAOT) of stationary liquid phases in the range up to 250-300°C, chromatographic and thermogravimetric methods of analysis are being used [6], more commonly the latter [7-9]. In this method, the change in mass of the high-boiling compound under study is followed continuously.

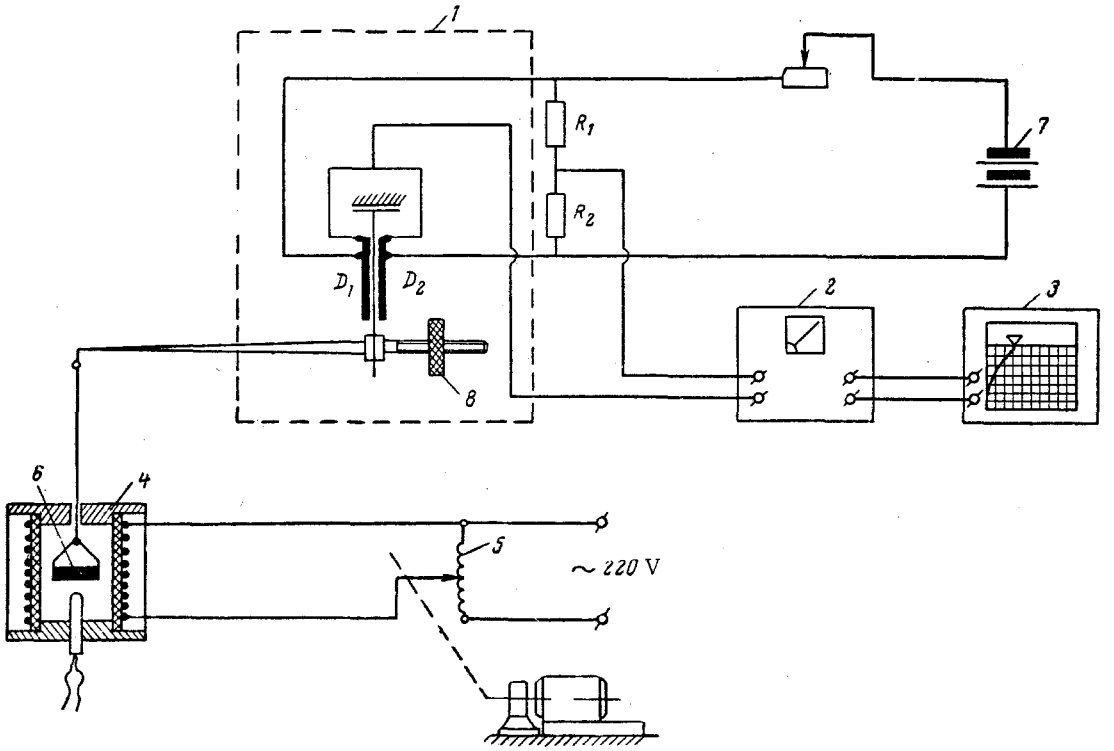

Fig. 1. Diagram of unit for thermogravimetric analysis of dispersion media and thickening agents for VNII NP-300A grease.

All-Union Scientific-Research Institute for Petroleum Processing (VNII NP). Translated from Khimiya i Tekhnologiya Topliv i Masel, No. 12, pp. 49-51, December, 1973.

TABLE 1. Thermal Stability of Various Dispersion Media and Thickening Agents

Substance tested	Mass loss (%) at indicated temperature				
	100 °C	150 °C	200 °C	250 °C	300 °C
Dispersion media					
Naphthenic-base mineral oils					
VM-1	0,5	1,0	3,0	13,3	25
VM-3	N	N	0,25	3,75	22,5
VM-4	0,6	1,5	1,75	2,0	15,0
Synthetic hydrocarbon oils					
MS-1	N	N	N	1,25	8,5
MS-2	N	N	N	1,0	7,5
Aromatic-base mineral oils					
B-29	N	0.25	0,75	2,0	8,7
B-35			0,75	1,0	6,0
Hydrocarbon fractions from B-35 oil					
FB-1	N	N	N	1,75	5,0
FB-2	N	N	0,5	1,0	2,5
Solid hydrocarbons of various molecular weights					
TU-1	N	N	0,50	8,25	25
TU-2	N	N	0,25	2.5	14,0
TU-3	N	N	0,20	1,9	9,0
TU-4	N	N	N	0,5	5,0
TU-5	N	N	N	0,2	2,0

Note. The letter "N" indicates that there was essentially no loss in mass of the test sample.

TABLE 2. Thermal Stability and MAOT of Various Greases Used as High-Temperature Stationary Liquid Phases

Substance tested	Mass loss (%) at indicated temp.		MAOT, °C
	250°C	300°C	
Apiezon M	0,3	4,8	250
Apiezon L	0,0	2,5	300
Apiezon N	0,4	3,5	250
VNII NP-300A	0,0	4,5	300

Note. No loss in mass detected at 100, 150, or 200°C.

Here we assembled a special unit for thermogravimetric analysis (Fig. 1), consisting of the strain-gage attachment 1, dc amplifier 2, recorder 3, cylindrical electric furnace 4, and controlled power source 5, by which the test sample 6 could be heated at various rates. The strain gages D_1 and D_2 and the wire-wound resistors R_1 and R_2 are the arms of a balanced bridge with the dc amplifier and the recorder included in the measuring diagonal. The instrument power supply is the dc source 7, with a voltage of 6.5 V that is monitored during operation with a voltmeter.

When the mass of the test sample changes, the metal strip to which the strain gages are attached is elastically deformed, thus changing the resistance of the strain gages, the potential in the measuring diagonal of the bridge, and the reading of the recorder. The sensitivity of the unit to change in sample mass is determined by the dimensions of the elastic strip, the length of the balance arm, and the sensitivities of the strain gages and the metering instruments.

The sample weight may be a few milligrams or as much as several grams; the weight 8 is used to counterbalance the sample weight. The scale of the instrument can thus be chosen for any limits desired, independent of

the original sample size or the mass of the auxiliary parts. In our case, the registration scale of the KSP-4 instrument (250 mm) was balanced for 250 mg.

The error in determining the weight was at most 2% of full scale.

The test procedure is quite simple. A weighed test sample (1 g) in a glass beaker 25 mm in diameter, previously counterbalanced by means of the weight 8, is placed in the electric furnace. Then all instruments and the furnace heater are switched on. The temperature is raised at 6°C/min. Changes in mass during the experiment (about 2 h) are recorded on the strip-chart of the instrument.

Thermogravimetric data are listed in Table 1 for the thermal-oxidative stability of various oils and hydrocarbons. The best in this respect are dispersion media of the B-35 type (high-viscosity aromatic mineral oil) and thickening agents of the TU-5 type (high-molecular-weight solid hydrocarbons boiling above 500°C).

These components were used as the basis of development for the hydrocarbon grease VNII NP-300A, which has a high thermal-oxidative stability and a high maximum allowable operating temperature (MAOT). As can be seen from the data listed in Table 2, the VNII NP-300A grease is on a par with the imported Apiezon greases.

LITERATURE CITED

1. S. Dal Nogare and R. S. Juvet, Gas-Liquid Chromatography, Wiley, New York (1962).
2. M. Shinglyar, Gas Chromatography in Practice [in Russian], Khimiya, Moscow (1964).
3. Manual on Gas Chromatography [Russian translation], Mir, Moscow (1969).
4. L. I. Nazarova, E. D. Makeeva, N. I. Lulova, et al., Khim. Tekhnol. Topl. Masel, No. 7, 56 (1969).
5. L. I. Nazarova, N. I. Lulova, E. M. Nikonorov, et al., Khim. Tekhnol. Topl. Masel, No. 2, 51 (1971).
6. J. Takacs, J. Bella, and L. Masor, Chromatography, 16, 218 (1964).
7. G. Liptay, S. Bekassy, and J. Takacs, Chromatography, 24, 22 (1966).
8. G. Liptay and J. Takacs, Chromatography, 34, 1 (1968).
9. V. L. Davison and D. L. Moore, Gas Chromatog., 6, 540 (1968).