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SILICA GEL PLASTIC GREASES RESISTANT TO CORROSIVE MEDIA

E. D. Makeeva, et al

Foreign Technology Division
Wright-Patterson Air Force Base, Ohio

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by

Ye. D. Makeyeva, T. K. Ostrovskaya, et al.

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13. ABSTRACT

Two greases were synthesized; VNII NP-264 and VNII-279. The dispersion medium for the first grease are polymers of trifluorochloroethylene which boil when the range of 140-200°C at 10 torr, and for the second grease -- no. 30 synthetic oil. Used as thickeners for these greases were special synthesized organosilica gels - alkylsilyl derivative, and in alkyl alcoxysilyl derivative of silicic acid gel. Treated with the thickeners, these two greases are found to be exceedingly moisture resistant; they can withstand boiling in water for 100 hr without the separation of the dispersion medium from the thickener; they also possess high chemical and colloidal stability and do not cause corrosion of metal, and do not melt. They have found use as antifriction and thickener agents in bearings of electric motors, pumps which transfer acid, are used in blowers, and as stoppers for containers, and used in valves and threaded couplings operating in corrosive media in the range of ±50°C.

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З з	З з	Z, z	Ч ч	Ч ч	Ch, ch
И и	И и	I, i	Ш ш	Ш ш	Sh, sh
Й й	Й й	Y, y	Щ щ	Щ щ	Shch, shch
К к	К к	K, k	Ъ ъ	Ъ ъ	"
Л л	Л л	L, l	Ы ы	Ы ы	Y, y
М м	М м	M, m	Ь ь	Ь ь	'
Н н	Н н	N, n	Э э	Э э	E, e
О о	О о	O, o	Ю ю	Ю ю	Yu, yu
П п	П п	P, p	Я я	Я я	Ya, ya

* ye initially, after vowels, and after ъ, ь; e elsewhere. When written as ё in Russian, transliterate as yё or ё. The use of diacritical marks is preferred, but such marks may be omitted when expediency dictates.

SILICA GEL PLASTIC GREASES RESISTANT TO CORROSIVE MEDIA

Ye. D. Makeyeva, T. K. Ostrovskaya,
O. V. Atayeva, B. S. Gurenkov, and
Z. P. Slugina

Recently an especially acute need arose for ingredients in lubricants of mechanisms in contact with concentrated acids and alkalis.

The durability of plastic greases in contact with corrosive media can be achieved, firstly, by introducing substances in the composition of the grease (dispersion media and thickeners), chemically inert to acids and alkalis, and secondly, by formulating such a colloidal system of a grease that it is not destroyed due to the action of these reagents.

It was shown in [1-4] that the organofluorine compounds and lubricating materials using them as a base possess high chemical inertness to chromic, perchloric, chlorosulfonic, fuming nitric and other acids. Our investigations indicated that plastic greases, inert to the action of concentrated alkalis, as well as to the action of certain acids (for example, sulfuric, hydrochloric, acetic acids), can also be produced using other synthetic oils as a base. Silica gel, modified with surface-active substances –

alkyhalo derivatives of silane, alcohol-telomeres, higher alcohols of the aliphatic series and others [6] can be used as a thickening component of such oils.

As a result of investigations two greases were synthesized: VNII NP-264 and VNII NP-279. The dispersion medium for the first grease are the polymers of trifluorochloroethylene which boil in the range of 140-200°C at 10 torr, and the second – No. 30 synthetic oil (Table 1).

Table 1. Characteristics of the dispersion media.

(1) Дисперсионная среда	ρ, г/см³ (4)	(2) Температура, °C		(3) ν, сст		
		(5) кипения	потери подвижности (6)	(7) при −20 °C	(7) при +50 °C	(7) при +100 °C
(8) Полимеры трифторхлорэтилена . . .	1,9360	140 — 200 (10 тор)	— 44	4 540	66,6	12,1
(9) Масло № 30	0,8534	>520	— 42	40 000	167	26,1

KEY: (1) Dispersion medium; (2) Temperature, °C; (3) ν, cSt; (4) ρ, g/cm^3; (5) boiling point; (6) loss of mobility; (7) at; (8) Polymers of trifluorochloroethylene; (9) No. 30 oil.
[тор = torr]

Figure 1 shows the curves which characterize the antiwear properties of the dispersion media of both greases determined from a four-ball bearing frictional testing machine at 50°C, 1500 r/min and a d of the ball bearing, 12.7 mm. From the figure it is apparent that for polymers of trifluorochloroethylene with an increase in load from 100 to 300 kgf, the diameter of the wear spot practically remains constant (1.2 mm). No. 30 synthetic oil under the given testing conditions only withstands a load up to 70 kgf.

One of the essential factors which determines the service properties of the plastic greases is its loss of mass which in the absence of thermal decomposition during the preparation and

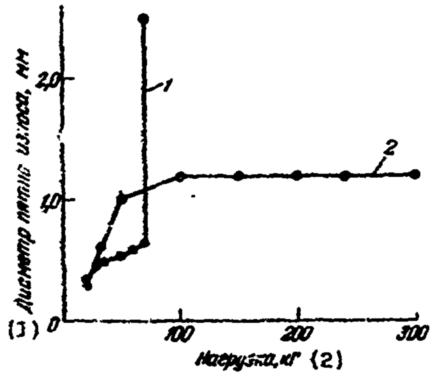

Fig. 1. The characteristics of the antiwear properties of the dispersion media of greases: 1 – No. 30 oil; 2 – low molecular polymers of trifluorochloroethylene. KEY: (1) Diameter of the wear spot, mm; (2) Load, kgf.

application of the grease, is determined by the losses of the mass of the dispersion medium. This index is determined on a PIM-2 device [?] using a liquid layer 20 μm thick, and an evaporation surface of 6 cm^2. It was established that the greatest losses of the mass are characteristic for polymers of trifluorochloroethylene: over a 120 min period at 50°C the losses of the mass amounted to 3?%. Such high losses of the mass (a substantial deficiency for all of the class of organofluorine compounds) are determined from their nature [4]. The losses of the mass of No. 30 oil at a temperature up to 100°C are insignificantly small – less than 1% over 180 min. Hardly noticeable losses of the mass of this oil only commence at a temperature above 150°C and at 170°C over 120 min the losses reach 28.4%.

Used as thickening components for VNII NP-264 and VNII NP-279, plastic greases were special synthesized organosilica gels – alkylsilyl derivative (I) and alkyl alcoxysilyl derivative of the gel of silicic acid (II) in the form of white or gray granules. From the characteristics presented in Table 2 it is apparent that the thickeners have a large specific surface and the high moisture resistance; the content of silicon – 40%.

The indices of the properties of greases using these thickeners as a base are given below:

Index	VNII NP-264	VNII NP-279
Ultimate strength at 50°C, gf/cm^2, not less than........	3	1.9
Effective viscosity, poise		
at -40°C, D = 100 s^{-1}, not more than.............	3800	3600
at +50°C and D = 1000 s^{-1}, not less than.............	10.0	10.0
Syneresis (50°C, 24 hr), wt. %, not more than.............	0.2	0.2
Losses of the mass (50°C, 24 hr) wt. %, not more than..	1.5	0.0
Colloidal stability (20°C, P = 300 gf), wt. %, not more than....................	4.0	4.0

Table 2. Characteristics of the thickening components.

(1) Загуститель	(2) Насыпная плотность г/см³	(3) Удельная поверхность* м²/г	(4) Элементарный состав вес. %				(5) Влагостойкость %
			C	H	Si	O	
I	0,12—0,13	340—400	9—10	—	40	—	99—100
II	0,25—0,35	250—330	20—26	4,0—5,5	31—3[illegible]	37—[illegible]	99—100

*After ignition at 500°C.

KEY: (1) Thickener; (2) Bulk density g/cm^3; (3) Specific surface area*, m^2/g; (4) Elementary composition; (5) Moisture resistance, %.

The greases are exceedingly moisture resistant: they can undergo boiling in water for 100 hr without the separation of the dispersion medium from the thickener, they possess high chemical and colloidal stability, do not cause corrosion of metals, do not melt; the lowering of the oxygen pressure (at 100°C for 100 hr) is equal to zero.

The curves of the effective viscosity and the ultimate strength during heating (determined on a PVR-1, ГОСТ 9127-59 [ГОСТ = GOST = All-Union State Standard] device) indicate that with an increase in temperature from 50 to 80°C the values of these indices for both of the greases increased by at least 2-4 times (Fig. 2). At temperatures below 0°C, the viscosity-temperature characteristics of the greases is determined by the characteristics of the dispersion media.

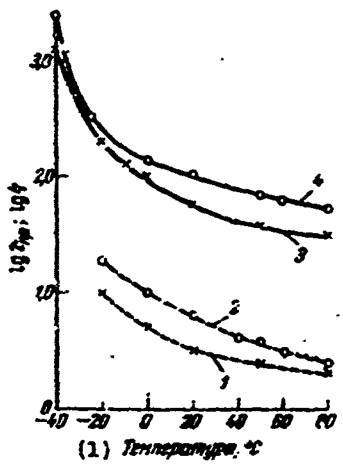

Fig. 2. Curves of the viscosity-strength properties of greases: 1, 3 – VNII NP-279; 2, 4 – VNII NP-264: Solid line – $\tau_{пр} f(t)$; dashed line – $\eta f(t)$. KEY: (1) Temperature, °C.

For the study of the changes in the effective viscosity and ultimate strength of the greases during storage, a layer of grease (5-6 mm) was applied on the outer surface of small glass cups which were then stored in a thermostat at 50°C for a period of one year. The viscosity and ultimate strength of the thermostatically controlled greases were periodically determined. The results of the determinations indicated that the viscosity-strength properties of VNII NP-279 grease did not change over a period of one year (Fig. 3) whereas the viscosity and ultimate strength of VNII NP-264 increased under these conditions by 7-8 and 5-6 times, respectively. Evidently, the latter can be explained by the high volatility of the dispersion medium of the grease, since during the storage of this grease in a sealed tare the viscosity-strength properties hardly change at all.

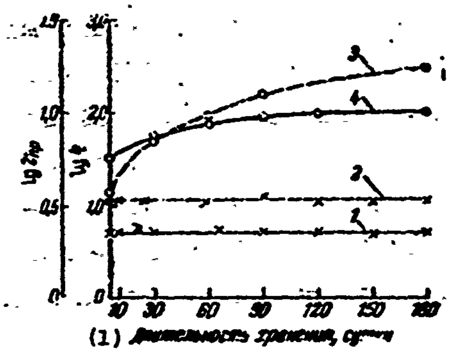

Fig. 3. Curves of the change in effective viscosity (solid lines) and the ultimate strength (dashed lines) depending on the length of storage of the greases: 1, 2 — VNII NP-279; 3, 4 — VNII NP-264.
KEY: (1) Length of storage, 24 hour periods.

One of the important service properties of plastic greases is its physical stability. Machining of the grease can cause a sharp change in the original volumetric-physical properties of the grease which results in the blowout of the grease from the friction joint.

The physical stability of the greases were studied on a PVR-1 device, by determining the changes in the shear stress arising in the greases during a successive increase and reduction in the strain rate. A successive increase and decrease in the strain rate results in non-coinciding flow curves (Fig. 4) [7, 8]. The physical stability of the greases were evaluated by the ratio of the shear stress (τ, gf/cm^2) of the first cycle[1] to the shear stress of the 20th cycle with $D = 100\ s^{-1}$.

As seen from Fig. 4, both greases have high physical stability. In order to achieve such a state whereby the rate of destruction of the structural elements of the grease and their rate of restoration during the transition from one flow regime to another and the reverse, remain practically constant, 4-6 cycles are sufficient. During this shear stress in greases when $D = 100\ s^{-1}$, it is lowered by more than one-half. For comparison the curves are presented on this same graph for the flow of TsIATIM-221 grease thickened with a component which is a soap — a complex of stearate and calcium acetate. Agreement of the flow curves of this grease

[1]A cycle — one successive increase and reduction of the strain rate.

begins only after 26 cycles, and the shear stress when D = 100 s^{-1} is lowered by one-half.

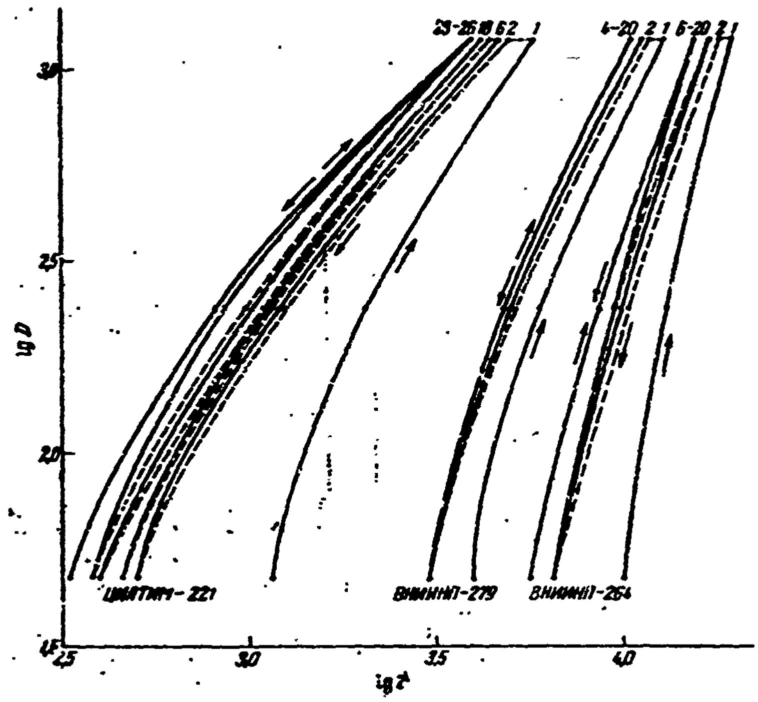

Fig. 4. The physical stability of VNII NP-279 grease. The figures on the curves indicate the number of cycles.
[ЦИАТИМ-221 = TsIATIM-221; ВНИИНП-279 = = VNIINP-279; ВНИИНП-264 = VNIINP-264]

Frequently in practice it is important to know just how the properties of the grease change during storage under conditions of high humidity (up to 98%). The use of greases with polymers of trifluorochloroethylene as a base under these conditions is not recommended since in the presence of water they cause the corrosion of the metallic surface. Therefore, investigations of the hydroscopic properties as well as the effect of the adsorption of water vapors and inversely, their evaporation, on the volumetric-physical properties were conducted only for VNII NP-279 grease. The grease was applied to glass plates with the aid of a stencil, the plates were then weighed and placed in a desiccator, the bottom of which is filled with water, and the plates held at room temperature for 300 hrs. After that the plates were removed and weighed again. Part of the plates were placed in a dry desiccator for the determination of the kinetics of evaporation of the adsorbed water. After the adsorption of water and its evaporation the viscosity and ultimate strength were determined.

As seen in Fig. 5 the adsorption of the water vapors by VNII NP-279 at room temperature for 300 hrs amounted to less than 0.5%; in this case the values of the effective viscosity (50°C, D = $= 1000\ s^{-1}$) and the ultimate strength at 50°C increased from 10.0 up to 10.7 poise and from 2.0 up to 2.5 gf/cm^2, respectively.

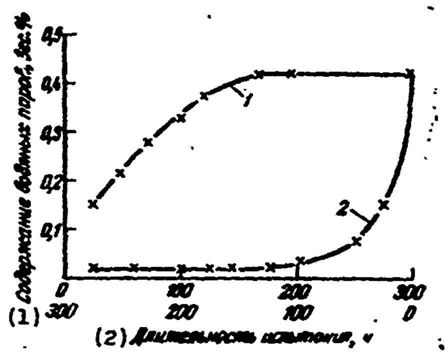

Fig. 5. The kinetics of adsorption and subsequent evaporation of water vapors: 1 – curve of adsorption of water vapors; 2 – curve of evaporation.
KEY: (1) Water vapor content, %; (2) Testing time, hr.

For VNII NP-279 grease, the reversibility of the process of adsorption of water is characteristic; with the lowering of the relative humidity of the surrounding medium, practically the entire amount of adsorbed water is evaporated, and the values of the effective viscosity and the ultimate strength of the grease become equal to the original (in the examined case, 0.7 poise and 1.9 gf/cm^2, respectively). The high values of the loss of mass of the dispersion medium of VNII NP-264 grease restricts the feasibility of its use at temperatures higher than 100°C, and also for a long period of time without replacement despite its high antiwear properties.

Data on the compatibility of the greases with acids, alkalis and certain solvents is presented in Table 3 (the testing time is 1 month).

Table 3. The compatibility of greases with corrosive media and solvents.

Medium	Temperature of testing, °C	VNII NP-264	VNII NP-279
Hydrochloric acid (conc.)	20	Does not dissolve	
	50	" " "	
Fuming nitric acid......	20	Does not dissolve	Dissolves
	50	"	Does not dissolve
Sulfuric acid (conc.)...	20	"	"
	50	"	"
Acetic acid............	20	"	"
Potassium hydroxide (density 1.4).........	20	-	"
Sodium hydroxide (density 1.4).........	20	-	"
Liquid ammonia (P = = 300 at)............	40	Dissolves	"
Ethyl alcohol...........	20	"	"
Acetone.................	20	"	"
B-70 gasoline...........	50	"	Dissolves
	20	"	"
Carbon tetrachloride....	20	"	"

VNII NP-264 and VNII NP-279 greases have found use as antifriction and thickener agents in bearings of electric motors, pumps which transfer acids, blowers, as stoppers, in valves and in threaded connections which operate in corrosive media in the range of ±50°C.

VNII NP-279 grease in an alkaline medium as well as in the atmosphere can be used over a broader range of temperatures – from -50 to 150°C. Owing to the low values of the loss of the mass of the dispersion medium, the grease does not require a change after a long period of time.

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