High-Viscosity Index Lubricating Oils from EPR Destruction Products

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SYNOPSIS

Polyolefinic oils obtained from olefin oligomerization or copolymerization involve the most common synthetic hydrocarbon oils. Research and experimental results concerning high viscosity index synthetic polyolefinic oils and viscosity index improvers' production by ethylene–propylene copolymer (EPR) and thermal destruction of EPRs noted for various molecular weight and composition resulted in high viscosity index polyolefinic oils and thickeners. The effect of structure, composition, and molecular weight on the destruction process has been discussed for various EPRs. The resulting polyolefinic oils and viscosity index improvers were studied. © 1992 John Wiley & Sons, Inc.

INTRODUCTION

When petroleum oils cannot meet the increasing demands upon modern motor oils, synthetic and semisynthetic oils may be used.[1-3] The application of these oils is intended for the producing of all-weather oils with extremely low volatility, which contributes to lowering of oil consumption approximately by a factor of two.[1]

Usually, motor oil bases are prepared from polyolefin and ester mixtures.[2] The composition of polyolefin oils is similar to that of high-purity paraffin oils. The length of the main carbon chain and the side-chain position provide good viscosity and temperature characteristics. High thermostability and low volatility are inherent in polyolefin oils. Commercial oils of a such basis correspond to the oil classification of ISO VG and range from 5 to 1000.[3]

Polyolefinic oils are usually obtained by polymerization of high molecular olefins with the number of carbon atoms of $C_8—C_{10}$. The production of polyolefinic oils based on petroleum gases (ethylene and propylene) is extremely promising, because the lack of petroleum raw material makes them the only source of providing industry with lubricant materials. The present paper deals with the results of the study of this problem.

EXPERIMENTAL

Ethylene–propylene copolymer was synthesized over Ziegler–Natta organometallic catalytic systems in liquid propylene as the medium.[4,5] Production of additives and lubricating oils on its basis was discussed in Refs. 6–8. In ethylene-propylene copolymer synthesis the following systems were used: vanadium triacetyl acetonate + diisobutyl aluminum chloride $[(AcAc)_3V + Al(iC_4H_9)_2Cl]$ and vanadyl trichloride + diethyl aluminum chloride $[VOCl_3 + Al(C_2H_5)_2Cl]$. High-purity ethylene and propylene (99.0 and 99.6 vol %, respectively) were used as reactants.

The copolymerization process was carried out in a 7 L bench-scale batch reactor and in a continuous pilot plant with a capacity of 20 kg/h. The copolymer samples thus obtained contained from 25 to 60 mol % of propylene units and exhibited elastomeric properties and viscosity-average molecular weight (M_v) of 30,000–500,000.

Thermal destruction of EPR was carried out in a 1.5 L bench-scale glandless batch autoclave equipped with an electric heater, a stirrer, and a temperature and residual pressure controller.

The autoclave was charged with 0.7 kg of lump polymer and heated at a rate of 3–5°C/min. The

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experimental temperature was maintained during the time interval required for obtaining products with the desirable molecular weight. Destruction distillates were fed to a separator via a cooler and further to a distillate receiver.

The higher molecular weight product for obtaining additives was removed from the reactor bottom to a product receiver. Thermal destruction kinetics of EPR was studied by TGA with the aid of the OD-102 MOM derivatograph at a progressive temperature rise to 500°C and higher.

RESULTS AND DISCUSSION

As the result of the studies, it was established that polyisobutylene and polyolefinic oils demonstrate a wide range of viscosity of 5–100 mm/s and a viscosity index of 120–140, with congelation temperature from −20 to −50°C.

The yield of the desirable products—polymer additives and oils—during the EPR thermal destruction shows a high value of 94–98%. The destruction distillates consist mainly of isoolefins and isoparaffins.

The change of the concentration of propylene in EPR from 25 to 60 mol % results in the increase of the yield of distillate products from 16 to 80 mol %. At the same time, a fivefold increase of the destruction rate was observed.[9]

Destruction of EPR starts at temperatures above 200°C and is characterized by weight loss, lower molecular weight, and changing MWD. As seen from the TGA results, major weight loss is typical for temperatures above 270°C and increases rapidly with temperature rising to 400°C. Total destruction is observed at 480–500°C.

The yield of destruction products varies directly with the reaction temperature. Higher pressure values result in lower yields of both distillates and gaseous products.

The energy of activation of the destruction reaction is 247–264 kJ/mol within the temperature range of 380–460°C, wherein the major weight loss of the copolymer was found to occur. With increasing propylene unit concentration in the copolymer and molecular weight, the activation energy drops by 8–12 kJ/mol. The reaction is of the first order and is irrespective of the copolymer properties.

A critical characteristic of the copolymer is the variation of its molecular weight within a temperature range of 350–360°C. Molecular weight of the copolymer declines smoothly to 5000–6000, which is accompanied by the formation of as little as 2 wt % of gaseous products. This can be traced to the fact that the energy barrier is surmounted and the macromolecule is broken over its total length and primarily at its weak bonds, which may be caused by tertiary carbon atoms in propylene units.

The changes in molecular weight with destruction time, i.e., the exposure to heat at the given temperature, appear at curves that decline with a limited molecular weight.

EPR thermal destruction above 370°C involves the formation of low molecular products that are separated under experimental conditions into a distillate and a residual product. The separation proceeds intensively at of 400°C and higher temperatures. Increased yields of distillate and the gaseous product are attended by their lower molecular weights and residual yields. As a result, the residual product changes almost completely to distillate (up to 92 wt %) and gaseous (up to 8 wt %) products. The rate of the destruction to distillates grows with the higher content of propylene units in the copolymer.

Rheological properties of the product are determined from its molecular weight and composition; viscosity of boiling above 350°C fractions increases with the lower isoparaffin content.[10,11] The distillate is a light-green liquid with a faint odor of pyrolisate; it has a molecular weight of 250–350, specific gravity of 803–804 kg/m, and boiling range from 70°C to 500–520°C.

An increase in copolymer destruction temperature from 380 to 430°C results in a greater yield of fractions boiling within 250–400°C. An increase in pressure from 2.66 to 101.3 kPa in the course of destruction at 400°C brings about a greater yield (from 30 to 85 wt %) of the fractions boiling below 350°C. Fractional composition and yield of the distillate is substantially independent of the molecular weight and composition of the starting copolymer.

Gaseous hydrocarbons are formed mainly at the onset of the destruction. As the temperature increases from 380 to 430°C, the yield of gases tends to increase from 5 to 8 wt %; however, the pressure rise from 2.6 to 101.3 kPa brings about a drop in the yield of gases from 8 to 3 wt %. The growth of the propylene unit content in the copolymer favors the yield of gases only slightly and it is unaffected by the molecular weight of the starting copolymer. The gaseous product composition is, in turn, almost unaffected by the pressure and the composition and molecular weight of the copolymer.

The major components of the gaseous products are ethylene, propylene, ethane, propane, and butane/butene fraction (isobutane, *n*-butane, 1-bu-

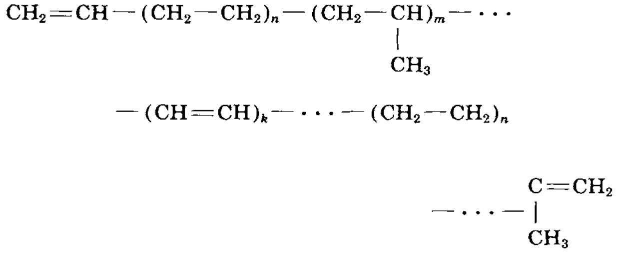
Table I The Change of Molecular Mass of EPC Depending on the Destruction Temperature

T (°C)	Destruction Time (Min)	Additive Yield (Wt %)	Specific Gravity (kg/m^3)	Molecular Weight ($\bar{M}_v$)
340	20	98.0	860	9000
350	20	98.0	854	7000
360	10	98.0	854	6000
370	10	98.0	850	4000
380	10	98.0	850	2000

tene, and isobutene). The share of ethylene, propylene, and isobutene increases with the higher destruction temperature.

Liquid destruction products comprise isoolefinic and isoparaffinic hydrocarbons. Their structure is similar to that of the starting copolymer macrochain and is characterized by methyl branches on propylene units and unsaturated vinyl structures. An increase in the destruction temperature from 380 to 400°C results in the growth of the unsaturated content from 75% to 95–98%. The olefin content grows from 50 to 60% in the light distillate fraction to 95–100% in heavy distillates. High unsaturation of these fractions indicates that there are one or more double bonds in the hydrocarbon molecules, predominantly in the end position.

The hydrocarbons obtained by EPR destruction are the fragments of the copolymer molecule in a wide range of their molecular length. Generally, the structure of such an olefinic hydrocarbon can be represented as



$$CH_2{=}CH{-}(CH_2{-}CH_2)_n{-}(CH_2{-}\underset{\displaystyle CH_3}{\underset{|}{CH}})_m{-}\cdots$$
$$-(CH{=}CH)_k{-}\cdots{-}(CH_2{-}CH_2)_n$$
$$-\cdots-\underset{\displaystyle CH_3}{\overset{\displaystyle C{=}CH_2}{|}}$$

where n = 2–4 on the average, m = 1–3 on the average, and k = 0–1. The hydrocarbons may have similar or different unsaturated structures or a methyl group at the end of the chain.

On the strength of both literature[12] and experimental data, it can be suggested that the thermal destruction of EPR is a free-radical reaction with a random splitting of macromolecules into macroradicals that continue the destruction through intermolecular and intramolecular chain transfer reactions or terminate the kinetic chain due to their recombination.

A target-oriented investigation of the products obtained by various degrees of destruction revealed that a great variety of both synthetic oils and viscosity improvers can be produced on their basis. An important feature of this destruction process is its freedom from wasters since various fractions from the destruction find extensive application in other spheres of the petroleum chemistry.[11,13,14]

The process of EPR destruction can be carried out along the following three basic paths:

1. Mild destruction to obtain polymer additives.
2. Moderate destruction to obtain high-viscosity (15–100 mm^2/s) and high-index (125–140) lubricating oils.
3. Deep destruction to obtain low-pour point, high-index oils (viscosity of 4–15 mm^2/s) and base oils for thickened all-weather lubricating oils.

An ethylene–propylene additive (EPA) with $\bar{M}_v$ = 2000–9000 was obtained by mild destruction of EPR with M_v = 60,000–500,000 and a propylene content of 42 wt % at a residual pressure of 6.6 kPa and a temperature varying in the range of 340–380°C (Table I).

Optimum molecular weight of the polymer additive was taken from the result of thickening low-viscosity, low-pour point M-4[†] oil obtained from the Neftyaniye Kamni (Oil Rocks) crude oil (Table II).

It is seen from Table II that the oils thickened with these polymer additives exhibit similar physical and chemical properties. EPA features good thickening ability, while the commercial bifunctional poly(methacrylate) additive (PMA-D) endows the oil with better viscosity–temperature characteristics. However, the thermal and mechanical stabilities of polymeric additives are evidently critical properties.

[†] All oil marks are given according to the USSR classification.

Table II Thickening Power of EPA of Different Molecular Mass

Oil or Additive	M_v	Consumption (wt %)	Specific Gravity (kg/m^3)	Viscosity, (mm^2/s)		VI	Pour Point (°C)	Flash Point (°C)	NPA Color
				100°C	−18°C				
M-4 base oil	—	—	870	3.8	1200	75	−42	184	1½
Thickened oils with									
EPA	2,000	15.5	870	9.91	4365	142	−42	180	4
EPA	4,000	8.0	870	9.62	4200	142	−42	180	4
EPA	6,000	6.5	870	10.42	4280	142	−42	180	4
EPA	7,000	4.5	871	9.68	5150	144	−42	180	3½
EPA	9,200	3.3	871	10.00	4100	145	−42	182	3½
PIB	10,000	6.8	870	9.6	4130	140	−42	182	4
PMA-D	20,000	4.6	869	9.5	1970	180	−42	182	2

In Table III, EPA is shown to be far superior to PMA-D and poly(isobutylene) (PIB) additives in mechanical stability and superior to PMA-D and the same as PIB in thermal stability.

The effect of copolymer composition on viscosity–temperature properties was also investigated. Propylene content in the copolymer was varied within the 27–53% range. The M-4 oil from the Baku crude oil was used as the base oil in examining thickening ability of the EPA-6 additives with different propylene contents.

The base oil was thickened to the viscosity level of the M-8 base oil (viscosity = 7.5–7.7 mm^2/s at 100°C). Table IV shows that the EPAs containing different amounts of propylene exhibit satisfactory thickening ability without a deteriorating flash point or pour point of the thickened oil. Optimum propylene content was found to be 27–30%, which yields a polymeric additive with excellent stability.

Higher propylene contents in the EPA-6 additive result in certain impairment of its thermal and mechanical stability and slight deterioration of viscosity–temperature properties of the oil.

Table V reports the properties of the M-4$_3$/8B$_1$ motor oil, obtained by thickening the M-4 base oil from the Baku crude oil, as well as the M-8B$_1$ and M-10B$_2$ oils obtained from the sulfur-bearing and the Baku sulfur-free crude oils by adding the EPA-6 additive.

The M-4$_3$/8B$_1$, M-8B$_2$, and M-10B$_2$ motor oils with a complex of additives were found to meet all the requirements of the corresponding USSR State

Table III Thermal and Mechanical Stability of EPA of Different Molecular Mass

				Thermal Destruction		Mechanical Destruction	
Additive	M_v	Consumption (wt %)	Initial Kinematic Viscosity of Thickened Oils at 100°C (mm^2/s)	Kinematic Viscosity at 100°C after 12 h of Testing (mm^2/s)	Oil Destruction (%)	Kinematic Viscosity at 100°C after 60 m Exposure to Sonic Vibration	Oil Destruction (%)
EPA	2000	15.5	9.91	9.70	2.12	9.90	0.01
EPA	4000	8.0	9.52	9.20	3.40	9.40	1.06
EPA	6000	6.5	10.40	9.96	3.20	10.07	3.17
EPA	7000	4.5	9.68	9.09	6.21	8.90	8.06
EPA	9200	3.3	10.00	8.45	15.50	8.14	18.60
PIB	10000	7.0	9.50	9.20	3.16	7.50	21.05
PMA-D	20000	2.8	9.60	6.00	37.50	4.91	48.25

Table IV The Effect of Propylene Content in EPA upon Thermal and Mechanical Stability of Thickened Oil

Oil or Additive	Propylene Content (Wt %)	Consumption (wt %)	Viscosity (mm^2/s)		VI	Flash Point (°C)	Pour Point (°C)	Destruction (%)	
			100°C	−18°C				Thermal	Mechanical
M-4	—	—	3.8	1200	75	184	−42	—	—
EPA	27	4.0	7.54	2560	130	180	−42	1.86	1.72
EPA	30	4.0	7.82	3258	130	180	−42	4.00	3.13
EPA	37	4.0	7.68	3300	130	180	−42	6.60	4.00
EPA	48	4.0	7.68	3350	130	180	−42	7.60	4.70
EPA	53	4.0	7.5	3500	125	180	−42	10.00	4.87

Table V The Quality of Thickened Motor Oil

Motor Oils	4-4$_3$/8B$_1$	M-8B$_1$	M-10B$_2$
Percent of polymeric additive	4	0.6	1.5
Specific gravity at 20°C (kg/m^3)	871	887	898.9
Kinematic viscosity			
at 100°C	8.4	8.5	10.27
at 0°C	620	959	1704
Viscosity index	130	108	95
Flash point in open crucible (°C)	180	206	213
Pour point (°C)	−42	−25	−17
Stability from induction period of sedimentation (IPS)	Test passed	Test passed	Test passed

Table VI The Quality of High-Viscosity Oils Prepared at EPC Moderate Destruction

T (°C)	Destruction Time (min)	Yield (Wt %)	Kinematic Viscosity at 100°C (mm^2/s)	VI	Pour Point (°C)	Flash Point (°C)
400	60	82	19	130	−26	224
400	50	83	24	134	−22	226
400	40	84	34	135	−20	230
400	50	85	44	140	−20	235
400	20	86	59	142	−19	240
390	30	88	61	143	−18	245
380	30	90	92	—	−15	255

Standards and to pass the qualification tests to satisfaction.

It was also found that the EPA-6 polymeric additive containing 27–30% of propylene can be used for producing the M-4$_3$/8 all-weather oil and, in an amount of 0.5–1.5%, as an viscosity index improver for lubricating oils. The EPA-6 polymeric additive is also recommended for replacing the MC-20 residual oil as a component of the ISP-65 industrial oil and satisfies the qualification test.

The ISP-65 oil was obtained from the VI-40 base oil by adding hydrogenated concentrate of the EPA-6 additive plus ISO additive composition (DF-11 [wear-resistant dialkyl dithiophosphate additive]—2%, ABES [wear-resistant alkyl dibenzyl sulfide additive]—6%, and B-15/41—0.1%).

Addition of EPA-6 in place of the residual component to the oil composition yielded a lube oil surpassing the specifications for coke formation, pour point, and color and featured excellent viscosity-

Table VII The Quality of Semi-Synthetic Base Motor Oils

	Composition (wt %)			Properties								
					Viscosity							
Oil	Distillate Oil	Residual Oil	TDO	Specific Gravity (kg/m^3)	100°C	0°C	VI	Coke Formation (%)	Acid Number (mg KOH/g)	Flash Point (°C)	Pour Point (°C)	Colorimeter Test (Unit)
TDO	—	—	100	854	28.4	—	135	0.2	0.01	230	−20	2
Hydrogenerated concentrate	50	—	50	867	17.0	2150	130	0.10	0.01	222	−19	2
M-8	80	13	7	870	8.0	720	100	0.08	0.01	205	−18	2
M-10	60	30	10	878	10.2	1570	102	0.12	0.01	215	−17	3
M-12	50	35	15	882	12.1	1950	104	0.15	0.01	225	−16	4

temperature characteristics. It was also found that a high viscosity and high VI oil could be obtained by subjecting EPR to thermal destruction at different temperatures, atmospheric pressure, and a destruction time of 15–20 min (moderate destruction).

Table VI shows the yield and properties of high-viscosity oils from EPR thermal destruction containing 35–40 wt % of propylene. This oil is an atmospheric/vacuum distillation residium boiling over 400°C. It is evident from the table that thermal destruction of EPR at different temperatures (380–400°C) results in oils with a viscosity of 19.0–92.0 at 100°C and yields of 82–90 wt %. These oils are characterized by a high flash point and low pour point and a VI range from 130 to 143.

The EPR thermal destruction oils can be employed in their own right or as a high-viscosity component for semisynthetic high VI oils blended with petroleum oils. Production of high-viscosity oils such as MC-20, P-28, P-40, Octol-600, etc., is also useful and economically attractive. The oils obtained by copolymer thermal destruction must be stabilized by hydrogenation before use.

In the production of semisynthetic motor, industrial, turbine, and other oils, it is advisable to employ thermodestruction oil (TDO) with a viscosity in the range of 25–35 mm^2/s.

Physicochemical properties of the M-8, M-10, and M-12 semisynthetic base oils with TDO having a viscosity of 28.4 mm^2/s at 100°C are summarized in Table VII. TDO as a 50% concentrate in the M-6 oil obtained from the Baku crude oil was hydrogenated before use over an Al—Co—Mo catalyst

Table VIII The Quality of Low-Viscosity Oils Prepared at EPC Deep Destruction

	Boiling Range (°C)	
Fractions	290–350	< 350
Yield based on distillate product (wt %)	16.7	67.3
Kinematic viscosity (mm^2/s)		
at 100°C	—	5.31
at 50°C	2.2	18.9
at 0°C	—	540
Viscosity index	—	148
Flash point (°C)	120	212
Pour point (°C)	−68	−42
Acid number (mg KOH/g)	0.015	0.02
Colorimeter test (units)	1	2

Table IX The Quality of Arctic Transformer Oil

	Transformer Oil (290–350°C bp Fraction)	ATM-65 Arctic Oil Specifications
Yield based on EPR (wt %)	13.8	—
Specific gravity (ρ_4^{20}) (kg/m^3)	784	875 max.
Kinematic viscosity (mm^2/s)		
at 50°C	2.8	3.2 max.
at −50°C	135	2000 max.
Pour point (°C)	−68	−65 or lower
Flash point (°C)	120	100 or higher
Acid number (mg KOH/g)	0.01	0.015
Oxidation stability	No sediment	No sediment
Loss tangent of dielectric	0.04	0.5 max.
Sodium hydroxide test (units)	0.2	0.4 max.
Copper strip test	Positive	Positive

Table X The Quality of All-Weather Thickened Oil

		Properties									
			Viscosity (mm^2/s)								
Oil	Polymeric Additive (wt %)	Specific Gravity (kg/m^3)	100°C	0°C	−18°C	VI	Coke Formation (%)	Acid Number (mg KOH/g)	Flash Point (°C)	Pour Point (°C)	Colorimeter Test (Unit)
H-5 base oil	—	851	5.31	540	800	148	0.01	0.02	212	−42	2
TDO > 350°C in:											
M-5$_3$/8	2.4	852	8.1	560	920	182	0.01	0.02	212	−45	2
M-5$_3$/10	3.2	854	10.3	575	955	189	0.01	0.02	212	−48	2
M-5$_3$/12	4.0	860	12.2	600	1050	195	0.01	0.02	212	−49	2
Alkyl aromatic oil	—	878	5.1	760	1100	123	0.1	0.03	200	−50	3
M-5$_3$/8 alkyl aromatic oil	7.1		7.5	850	2100	148	0.1	0.03	200	−47	3

at the temperature of 330°C, pressure of 4 MPa, LHSV = 1, and the hydrogen feed rate of 300 L per liter of the feed. Distillate oil (M-6) and residual oil from the Baku crude were used as a distillate and a residual petroleum component, respectively.

It is seen from Table VII that 7–15 wt % of TDO added to the base oil to obtain the M-8, M-10, and M-12 motor oils greatly improves their physicochemical and viscosity properties; VI increases from 83–86 to 100–104. Blending semisynthetic oils with TDO along with additive compositions yields motor oils of the V, G, and D group.

A method for deep EPR destruction has also been developed to produce low-pour oils with viscosity values of 3–6 mm^2/s at 100°C. It was found that a deeper thermal destruction of EPR (at 400°C) yielded low-viscous oil fractions with a high VI, low pour point, improved low-temperature properties, high flash point, and low acid number, which can be used as the base oils for thickened all-weather lubricating oils. Oils with better viscosity–temperature characteristics can be obtained by thermal destruction of EPR with a propylene content of 45–50 mol %.

The yields and properties of low-viscous TDOs obtained by thermal destruction of EPR are presented in Table VIII. It should be noted that it is possible to obtain a great variety of oil fractions with different viscosities by varying the process temperature and vacuum fractionation conditions and these fractions can be advantageously used as the base oils for low-pour lubricating oils. The starting material of high value for a high-quality arctic transformer oil (ATM-65) is an oil fraction boiling in the range from 290 to 350°C. When hydrogenated, this fraction is characterized by excellent dielectric properties, high oxidation stability, and low pour point and acid number and was found to satisfy the requirements to the ATM-65 arctic transformer oil (Table IX).

The oil fraction boiling above 350°C is a valuable base for all-weather motor oils obtained by thickening the M-5 base oil with the PMA–D additive. It is evident from Table IX that thickened all-weather oils with unique pour-point and viscosity–temperature properties can be obtained from oil fractions with V_{100} = 5.31.

Table X indicates that the low-viscosity TDO exhibits far better susceptibility to a thickening agent than does alkyl aromatic oil. About three times as much polymeric additive is required for obtaining thickened oil with a viscosity of 7.3–7.5 from alkyl aromatic oil than from TDO.

CONCLUSIONS

1. In EPR destruction, an increase in propylene content and molecular weight results in lower thermal stability and higher destruction rate.
2. Viscosity-improving ethylene–propylene additive (EPA) with a molecular weight of 4000–6000 obtained by thermal destruction of EPR features high thickening ability and high stability to both thermal and mechanical degradation. This additive can be employed in the production of all-weather lubricating oils and for improving the viscosity–temperature properties of motor oils.
3. High viscosity index lubricating oils (VI = 140–190) can be obtained by thermal de-

struction of a copolymer with 40–85 mol % of ethylene. Along with a high viscosity index, these oils are characterized by improved antioxidant and anticorrosive properties and low pour point and can be used as special purpose lubricants and as viscosity index improvers for petroleum oils.

4. On the basis of this research, a continuous process has been developed for EPR destruction to high-quality viscosity improvers and lubricating oils.
5. The process for producing lubricating oils by EPR destruction is unique and yields high viscosity index oils in a broad viscosity range as well as polymeric additives with various molecular weights.

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