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ABSORPTION OIL FOR COKE OVEN GAS PURIFICATION

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ABSTRACT

Coke gas is a valuable fuel for metallurgical plants and a promising energy technology raw material, particularly for hydrogen energy. Absorption oil is used for gas purification, its prevention from thickening is an important task, so it is necessary to clarify the factors and mechanism of oil density increase in the process of its operation. For this purpose, the similarity and difference of products of oil polymerization in factory conditions with artificial polymerization of a separate fraction were established. Petrographic studies have shown that oil deposits in industrial equipment are represented by polymer-like material (94 %) with inclusions of coal particles (4 %). The results of infrared (IR) spectroscopy indicate certain similarities in the structure of substances and the presence of functional groups, which gives reason to believe that oligomerization products of absorbing oil components are present in the polymers of the benzene compartment, in the solid sediments and in the artificially obtained indene coumarone resin. It is shown that ammonia promotes the separation of ferrocyanide precipitates from the absorbing oil.

Keywords: coal, coke oven gas, benzene recovery, wash oil, polymerization, indene-coumarone resin, IR spectroscopy, deposit formation.

INTRODUCTION

To obtain environmentally friendly energy carriers, coke oven gas is considered as a feedstock for steam reforming, which is the main method for large-scale hydrogen production. It is noted that steam reforming of coke oven gas shows higher energy efficiency and lower CO_2 emissions compared to natural gas, coal and oil [1]. Purification of multi-component coke oven gas from impurities is an obligatory stage of its preparation for hydrogen power engineering and energy technological utilization. By-product recovery of valuable products during hydrogen production from coke oven gas increases the efficiency of pig iron and steel production [2 - 5].

The benzene department of coke-chemical production occupies an important place in the technological chain of coke oven gas preparation for its further utilization and is a place for obtaining a valuable product - crude benzene. For extraction of benzene hydrocarbons, absorbing oil - a fraction of coal tar distillation - is used, which should have several specific properties. Therefore, saving of valuable absorbing oil and prevention of its deterioration (thickening) are important tasks. Part of the thickened oil (mainly distillation above 270°C) in the benzene hydrocarbon recovery technology is withdrawn by distillation process as “benzene compartment polymers”.

Some technologies refer to this residue as "pitch" or "slag" emphasizing its composition characterized by the presence of high-boiling components [6]. Despite the name "polymers", this non-boiling residue is not considered by all studies to be a product of "polymerization" of oil components, although analogies with indene-coumarone resins have been traced [7]. These substances are removed from the operating cycle and require replenishment with fresh oil, which worsens the economics of the process. High-boiling oil components and ash-like residues can crystallize from the absorption oil and combine with coal dust and coke oven gas contaminants to form deposits. These hard-to-remove substances increase the hydraulic resistance of the benzene scrubber, reduce the mass transfer surface, form "plugs" in pipelines and equipment and can lead to process stoppage. For this reason, maintaining the oil characteristics in the right condition is a necessary condition to ensure the performance of the benzene department equipment. According to studies [8], solid phase is formed when the absorbing oil is exposed to ammonia.

The reasons for polymerization, compaction and deposit formation are not fully understood. Coal absorbing oil is a multicomponent mixture of aromatic compounds including unsaturated hydrocarbons, nitrogen-sulfur and oxygen-containing substances. The presence of reaction-active components, alternate heating and cooling of oil, its contact with coke oven gas containing many impurities, insufficient identification of oil components makes it difficult to establish the mechanism of oil thickening and deposit formation.

It can be noted that the variability of component and fractional composition of oil from different manufacturers, different degree of coke oven gas purification, different condition of equipment, different degree of watering of working oil, presence of corrosion products of equipment (potential catalysts of thickening) in the oil, different degree of contact of oil with the atmosphere, different impact of temperatures and the amount of steam in the distillation column prevent the unity of interpretations of oil degradation and deposit formation.

The task of the present work was to clarify the reasons and mechanism of the oil density increase in the process of its operation. For this purpose, it was planned to establish the similarity and difference of products of oil polymerization in factory conditions with artificial polymerization of a separate fraction. In addition, it was of interest to compare these products with the resulting deposits of their working oil in the production equipment. This may help to shed light on the mechanism of oil degradation and to develop recommendations to improve oil life and reduce cleaning operations in the benzene department equipment.

The presented research can be used in the production and use of coal oils, in coke oven gas washing technologies for benzene and naphthalene extraction, gas treatment at transportation plants, and gas purification plants.

EXPERIMENTAL

Samples of benzene scrubber sediments, working absorption oil and polymers of benzene compartment from the operating coke-chemical plant of Ukraine were obtained for research. Indene-coumarone resin with molecular weight 900, obtained in laboratory conditions from indene-coumarone fraction of crude benzene by catalytic polymerization and further recrystallization, was taken as a reference standard [9].

Samples were analysed according to the methods of determining the indicators of TU U 20.1-00190443-117:2017 "Coal tar wash oil" and standardized methods of analysing the properties of coal tar according to TU U 19.1-00190443-100:2016.

To obtain samples for IR spectroscopy, oil and polymer samples were distilled in a glass flask to obtain a solid non-boiling residue. The IR spectra of the samples were measured from 4000 cm^{-1} to 400 cm^{-1} (at 1 cm^{-1} steps) using the Shimadzu FTIR-8400S IR spectrometer and spectrum processing was performed using IR Solutions software.

Mass concentrations of wash oil components were determined by the chromatographic method using a chromatograph "Crystal 2000 M" with the software "Chromatec Analytic". The measurement method consisted in direct chromatography of the sample with subsequent calculation of chromatograms and calculation of the mass fraction of benzene hydrocarbons relative to the mass of the "internal standard" added to the wash solution - tetradecane, and higher boiling oil components relative to the mass of the "internal standard" - acetophenone. The chromatography conditions were as follows: column thermostat temperature, 90 - 200°C; programming rate of temperature 6°C min^{-1}; evaporator temperature 200°C; detector temperature 250°C; analysis duration 55 min. Solid substances insoluble in toluene were obtained from the sediments, which were studied on the automated petrographic complex "Lucia" of the company "LECO", equipped with a microscope Olympus BX51M.

The chemical composition of ash sediments was determined according to the national standard of Ukraine DSTU 9045:2020: Solid fuel oil. Methods of determination of chemical composition of ash.

RESULTS AND DISCUSSION

Characteristics of the operational absorbing oil, polymers and resulting deposits of the benzene scrubber are given in Tables 1 and 2.

The operational oil is a liquid with increased density in relation to the requirements of the regulations of the benzene plant, relatively high content of oil fractions above 285°C is balanced by a rather high content of naphthalene for the working oil. At losses of the latter due to evaporation with coke gas and distillation with benzene hydrocarbons, the oil is prone to precipitation.

Polymers of benzene unit were obtained in production conditions by semi-periodic distillation with maximum possible distillation of the fraction up to 270°C within the limits providing fluidity and emptying by gravity of the batch of the regeneration unit. The presence in polymers of a high content of distillation up to 270°C (on good oil in polymers it is possible to receive 0 - 5 % of distillation up to 270°C) testifies to danger of crystallization of the given oil and loss of fluidity at the further distillation.

The benzene scrubber deposits were a dark coloured lumpy solid with a distinctive odour of absorbent oil, the lumps were well crushed and crumbled when pressed. The sediment was heterogeneous, most likely a solid powder in a liquid, and the presence of hard fine abrasive particles was felt during averaging and analyses. When the sample was heated in a spoon on a gas torch, no melting or fluidity was observed, volatile vapours were emitted and the material thickened. The presence of a significant amount of absorbed water and the presence of an up to 270°C distillation was noticed. When attempting to determine the softening temperature of the deposits, using the ring-rod method, the material released oil, and the residue polymerized to such an extent that the softening temperature raised above 100°C.

Table 1. Fractional and chromatographic composition of samples of working oil and polymers of the benzene unit.

Properties	Working oil	Polymers
Density, g cm^{-3}	1.101	1.158
Water content, % vol.	5.3	0.3
Phenol content, % vol.	0.7	1.0
Chromatographically identified components, % weight		
naphthalene	10.8	3.8
2-methylnaphthalene	5.5	2.6
1-methylnaphthalene	3.4	1.6
biphenyl	13.1	7.2
acenaphthene	23.0	15.6
diphenylene oxide	13.0	14.6
fluorene + indole	8.1	9.2
phenanthrene + anthracene	1.7	3.1
Fractional composition		
Distillation content, % vol.: up to 240°C	227	256
up to 250°C	14.6	
up to 260 °C	29.0	
up to 270°C	45.9	12.3
up to 280°C	63.5	26.5
up to 285°C	75.1	
up to 290°C	80.6	
up to 300°C	83.4	

Table 2. Characteristics of sediments.

Parameters	Value
Ash content, %	11.1
Insoluble in toluene, %	68.2
Water content, %	21.0
Distillation content up to 270°C, %	22.0
Up to 285°C, %	5.1
Non-boiling residue %	49.6
Losses, %	2.3

Fig. 1 shows a typical micrograph of toluene insoluble substances obtained from benzene scrubber deposits.

In the image, a coal particle is marked with a circle in the center, around which there is a certain pitch-like mass with mineral inclusions on the surface. Relative to the surface of the coal particle, this mass has a softer texture with mineral components of sediments pressed into it. Also, in small quantities there are separate fragments of semi-coke, as well as particles of mesophase. Proceeding from the fact that mesophase in coal pitch is formed at thermal exposure in the absence of air, the presence of mesophase in sediments can be explained by possible operations of steaming of scrubber packing with superheated steam. The results of counting the content of particles identified by appearance are presented in Table 3.

Thus, the sediments are represented primarily by a polymer-like or pitch-like material, in which the content of the fluid fraction (distillation up to 270°C) is only a small amount lower than that of the benzene unit polymers. Fig. 2 shows the characterization of the identified charcoal particles according to the arbitrary reflectance of vitrinite.

As follows from Fig. 2, coal particles in deposits of benzene scrubber are present mainly in the form of relatively “soft” coal concentrates “GZhO”+”GZh” (39 %) and “Zh” (39 %), which indicates that mainly easily crushed coal grades of the coking charge are carried away from the coke chamber with coke gas.

The component composition of ash, obtained at the determination of ash content of deposits, is given in Table 4.

The mineral part of the sediments mainly contains products of equipment corrosion (iron compounds), while the amount of sulphur determined by the analysis is not adequate to form a stoichiometric amount of FeS (iron sulphide). In the composition of sediments, the SiO_2/Al_2O_3 ratio, which is typical for hard coals, is approximately observed. Thus, the presence of coal charge components in the sediments is confirmed. The presence of calcium oxides in the sediments looks uncharacteristic, which indicates a probable

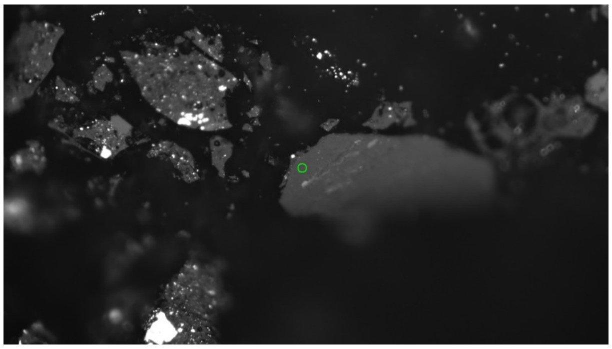

Fig. 1. Micrograph of toluene insoluble substances obtained from benzene scrubber sediments.

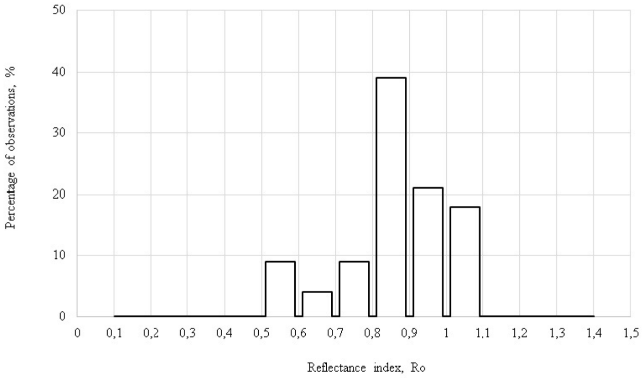

Fig. 2. Reflectogram of vitrinite reflectance obtained during the study of a coal particle from a sediment sample.

Table 3. Results of identification of sediment substances under the petrographic microscope.

Composition	Mass fraction, %
Pitch and polymeric materials	94.4
Coal	4.0
Semicoke and minerals	1.6

Table 4. Characterization of ash part of benzene scrubber deposits (mass fraction).

Compound	Middle zone sediments	Upper zone sediments
SiO_2	12.16	10.74
Al_2O_3	5.18	5.10
Fe_2O_3	59.35	60.85
CaO	3.85	3.15
SO_3	11.38	11.50

undesirable flow of cooling water into the working oil in the process of its indirect cooling in the heat exchanger.

It was mentioned above that the formation of solid suspensions of absorption oil is most intensive in the presence of ammonia [8]. For this reason, 10 cm^3 of concentrated ammonia water was added to the absorbent oil (100 cm^3), the sample was kept at room temperature for a day and then the non-boiling residue was obtained from the mixture by distillation. A graphical representation of the IR-spectroscopy results of the obtained and analyzed samples is shown in Fig. 3.

In the spectra of sediments, slightly larger peak areas are observed in comparison with polymers in the ranges 3760-2170 cm^{-1}, 1760-1510 cm^{-1}, 1505-1290 cm^{-1} and 1210-1050 cm^{-1}, which indicates a higher concentration of functional groups containing oxygen and nitrogen atoms, carbonyl groups, as well as a higher concentration of aromatic rings, functional groups of phenols and esters. Thus, the deposits on the scrubber packing clearly differ from polymers by the higher intensity of functional oxygen- and nitrogen-containing groups, their higher concentration, and the higher intensity of peaks of out-of-plane aromatic structures.

In the region of 3150-3000 cm^{-1} in all spectra it is possible to distinguish bands 3065 and 3019 cm^{-1}, which are most clearly marked in the spectra of indene-coumarone resin, are visible in the spectra of polymers, are slightly noticeable in the spectrum of working oil and are not marked at all in scrubber deposits. The bands in this range usually correspond to stretching vibrations of aromatic C–H bonds in monosubstituted aromatic hydrocarbons (PAHs). The exact position of the bands may vary depending on the substitution scheme and the presence of other functional groups.

The peak corresponding to 3019 cm^{-1} is attributed to stretching vibrations of hydrogen bonding with aromatic compounds. This band is accompanied by a peak at 1598 cm^{-1}, which is assigned to vibrations of the C–C bond in the aromatic ring and is clearly expressed in all samples except for the deposits. In addition, the band 3019 cm^{-1} in the fingerprint region is accompanied by peaks of medium intensity, inherent to out-of-plane vibrations of aromatic molecules. The peaks are most strongly evident and are maximal for their spectra at 746 cm^{-1} for indene-coumarone resin, 739 cm^{-1} for polymers and operating oil. For sediments, the 739 cm^{-1} band is only 14 % smaller from the main peak height of 2067 cm^{-1}. It should be noted that the obtained peak coincides well with the bright peak of indene-coumarone resin in the 747 cm^{-1} band available in the spectral database [10].

The working absorbing oil and polymers are

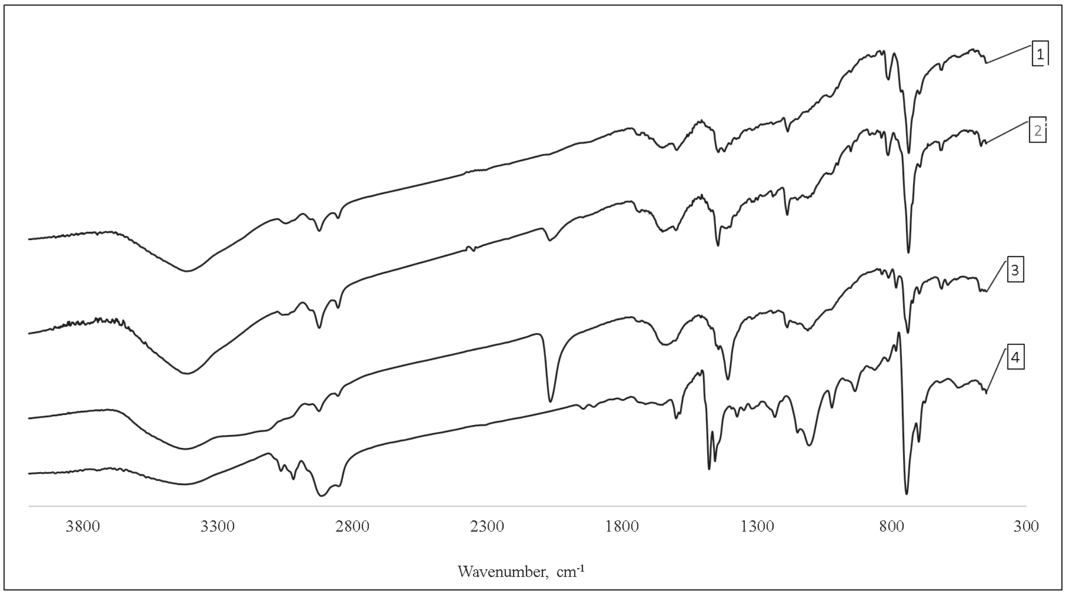

Fig. 3. The IR-spectrograms of the samples under study: 1 - benzene unit polymers; 2 - working absorbing oil distilled in the presence of ammonia; 2 - benzene scrubber deposits, 4 - indene-coumarone resin.

united by the presence of a peak at the wave number 2955 cm^{-1}, which corresponds to the frequency of stretching vibrations of the –CH$_2$– group [11].

The presence of a peak in the samples of benzene unit and recycle oil polymers shifted by 7 cm^{-1} to the high frequency region relative to the peak of indene-coumarone resin (746 cm^{-1}) indicates the "polymeric" nature of the high-boiling fractions of absorption oil. In this region, the second most significant peak for indene-coumarone resin, from the database spectrum, corresponds to a wave number of 702 cm^{-1}. In the samples we analysed, this peak is most clearly expressed for indene-coumarone resin (701 cm^{-1}) and is also present in all other samples with a shift to the low frequency region (696 - 699 cm^{-1}). The peak 718 cm^{-1}, obtained in the spectrum of indene-coumarone resin according to literature data, is present in the spectra of the studied samples in the form of peaks-"riders" of low intensity.

The peaks of strong intensity 2924 cm^{-1} are marked for the investigated samples of oil, polymers and sediments, in the sample of indene coumarone resin it is shifted to the wave number 2916 cm^{-1}. From the analysis of IR-spectra of coal tar and its derivatives, it is known that these bands refer to the valence vibrations of the C–H bond for aliphatic hydrogen [12]. This also indicates the structural similarity of the tested samples.

The presence in the spectrum of a band with a wave number 2854 cm^{-1} of approximately the same intensity related to the symmetric stretching vibrations of the –CH$_2$– group also unites all the studied samples [13]. In addition, the strong bands at 2854 cm^{-1} and 2924 (2916) cm^{-1} are characteristic frequencies of the CH$_2$ group vibrations and are found in the spectra of polyindene [14].

The spectrum bands at wave numbers 1233 and 1025 cm^{-1} in indene coumarone resin correlate with the wave numbers in benzene compartment polymers 1240 and 1025 cm^{-1} and in operating oil 1025 cm^{-1}. It is known that there is a strong 1240 cm^{-1} peak expressed in the spectra of polycoumarone resin, which is attributed to the asymmetric stretching of the =C–O–C group in the polymer, while the 1020 cm^{-1} peak represents the symmetric vibration of this bond [14].

A significant difference between the spectra is the presence of a strong band at 2067 cm^{-1} in the sediments and a medium intensity band in the spectrum of the absorbing oil treated with ammonia. Such bands are completely absent in the spectra of indene-coumarone resin and benzene compartment polymer samples. This band is absent in the spectra of coal tar and its fractions [12, 13]. According to Rennert et al. the absorption band of CN group in ferrocyanide complex compound lies in the range of 2069 - 2065 cm^{-1} [15].

The formation of ferrocyanides in the reducing environment of coke oven gas and absorbing oil requires the presence of ammonia, divalent iron and hydrogen cyanide:

$$FeSO_4 + 6NH_3 + 6HCN \rightarrow (NH_4)_4[Fe(CN)_6] + (NH_4)_2SO_4 \qquad (1)$$

Similar reactions with the formation of similar soluble insoluble salts occur in the processes of ammonia removal from coke oven gas, where the formation of ferrocyanides is favoured by high content of hydrogen cyanide, presence of equipment corrosion products and increased acidity. The benzene unit is located right after the ammonia recovery unit, and the conditions of ferrocyanide formation remain the same in case of NH$_3$ purification failures and operating oil watering.

In all samples, except for sediments, the presence of a 1025 cm^{-1} band is noted, which can be attributed to the C–O–C bond and confirms the presence of coumarone fragment in the resin composition, a similar band (1056 cm^{-1}) is found in indene-coumarone resin obtained from light coal tar oil [16].

The peak at 1150 cm^{-1} belongs to the vibrations of the C–O–C group and is found in the spectra of coal tar [17]. The peak at 1187 cm^{-1} is also attributed to the vibrations of the same group [18]. These bands confirm the presence of common functional groups in the analysed samples.

The band corresponding to the vibrations of the C–O bond (1320 cm^{-1}) is present in the spectra of polymers and indene-coumarone resin, which also indicates the structural similarities of these samples [8]. The absence of the 880 cm^{-1} band indicates the absence of penta-substituted aromatic rings (which are present in coal tar pitches) [19].

Thus, the structural similarities of the benzene unit polymer samples and indene-coumarone resin can be traced, which are summarized and presented in Table 5.

The results of IR-spectroscopy indicate certain similarities in the structure of substances and presence of functional groups, which gives grounds to believe that

Table 5. Similar and matching band spectra of indene-coumarone resin and benzene unit polymers.

Polymers of benzene unit		Indene-coumarone resin	
Wave number, cm^{-1}	Relative peak intensity, %	Wave number, cm^{-1}	Relative peak intensity, %
3419	54	3419	17
3048	18	3040	14
2924	31	2916	32
2854	22	2854	27
1598	28	1598	16
1443	32	1456	45
1374	19	1374	8
1340	11	1349	5
1320	11	1320	6
1240	6	1233	14
1025	7	1025	18
739	100	746	100
698	42	698	62

oligomerization products of absorbing oil components are present in polymers of the benzene compartment and solid deposits. The latter was previously assumed to be high-boiling crystallizing components of the absorption oil (anthracene, fluorene, etc.) deposited on the nozzle at the lowering of the ambient temperature.

The developed technology is an alternative to other technologies when using motor fuels as an energy source [20 - 22].

CONCLUSIONS

IR-spectroscopy confirms the similarity of structures of indene-coumarone resin and residues from distillation of absorbing oil and polymers of benzene unit, which gives grounds to assert that unsaturated hydrocarbons of absorbing oil are one of the significant sources of formation of oligomeric compounds, oil thickening, increased oil consumption and formation of deposits on the equipment surface. The closest resemblance to the structure of indene-coumarone resins was shown by benzene compartment polymers.

The artificial addition of concentrated ammonia water to the absorbing oil resulted in the same appearance of a peak on the IR-spectrum of the residue from the distillation of the mixture, which corresponds to the absorption band of the CN group in the ferrocyanide complex compound and was detected only in the deposits. This is explained by the susceptibility of deposits in the scrubber to the complex influence of ammonia and hydrogen cyanide of coke gas and corrosion products of equipment with the formation of ferrocyanide complexes.

Sediments are represented primarily by polymer-like or pitch-like material. According to the presence of functional groups (phenolic, carbonyl, nitrogen-containing, ether, and oxygen-containing), the most intense peaks of the spectra belong to sediments. This indicates their more diverse structure than the more ordered polymers of the benzene compartment, the structure of sediments is closer to the diverse components of coal tar. The results of petrography have shown that coal particles in deposits of benzene scrubber are present predominantly in the form of relatively non-hard coal concentrates, which indicates that mainly easily over-crushed grades of coke charge coals are carried away from the coke oven chamber with coke gas. The analysis of the mineral part showed that the SiO_2/Al_2O_3 ratio in the sediments is approximately the same as for coking coals.

To remove the dust introduced with coking gas (it is necessary to continuously operate the regenerator, which allows to remove solid sediments from the working cycle. Steam scrubbing of the scrubber can remove the easily flowing part of the deposits, but the solid oil deposits will be compacted.

Authors' contributions: D.M.: conceptualization, L.B.: writing; A.B.:investigation, writing; S.N.: review; F.C.: editing; S.K.: data analysis.

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