

# Catalytic Oxidative Demercaptanization of Fuel Oil Produced from Gas Condensate

Yu. A. Gavrillov, I. V. Pletneva\*, and E. N. Silkina

*Semenov Institute of Chemical Physics, Russian Academy of Sciences, Moscow, Russia*

\*e-mail: [inna.pletneva@yandex.ru](mailto:inna.pletneva@yandex.ru)

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**Abstract**—The results of studying the nonextractive oxidative demercaptanization of fuel oil derived from gas-condensate in the presence of metal complex catalysts are presented in this work. The effect of the ratio of gaseous (air) and liquid (fuel oil) phases, catalyst concentration, time of contact, process temperature, and amount of total mercaptans on the degree of conversion of low molecular weight sulfur admixtures and quality of commercial products was evaluated.

**Keywords:** gas-condensate fuel oil, demercaptanization, oxidation, metal complex catalyst, hydrogen sulfide, mercaptans

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## INTRODUCTION

In order to comply with ecological requirements, it is necessary to remove hydrogen sulfide and light mercaptans ( $\text{CH}_3\text{SH}$ ,  $\text{C}_2\text{H}_5\text{SH}$ ) from hydrocarbons. According to the existing European standards, the hydrogen sulfide content must not exceed 2 ppm (0.0002 wt %) and, according to technical regulation accepted in the Russian Federation, the content of hydrogen sulfide in the fuel oil must be below or equal to 20 ppm (0.002 wt %) before December 31, 2014 and below or equal to 10 ppm (0.001 wt %) starting on January 1, 2015 [1].

The existing chemical methods of demercaptanization can be divided into the reagent and oxidative. The former are based on the stoichiometric interaction of an active component (formaldehyde, amines, triazine, etc.) with sulfhydryl groups [2, 3]. The main drawbacks include high toxicity [4], high reagent consumption, and an excessive amount of water-soluble acids and bases (WSABs) in commercial fuel oil relative to the required amount.

In oxidative methods, purification is achieved via the conversion of  $\text{H}_2\text{S}$  and  $\text{RSH}$  into nontoxic forms by the action of strong oxidizers ( $\text{H}_2\text{O}_2$ ,  $\text{O}_3$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ , and others) [5, 6]. The drawbacks of these methods include corrosiveness, high level of hazard, the high consumption of oxidizers related to side reactions, and low compatibility with hydrocarbons.

The mild oxidation of indicated admixtures to di- and polysulfides can be carried out with air oxygen in the presence of metal complex catalysts based on Cu, Fe, Co, and Ni [7–10] directly in a hydrocarbon environment. It was shown in [11] that the developed cat-

alysts were effective for purifying fuel oil obtained from crude oil. Considering that the oil-derived fuel oil and the one obtained from gas condensate differ in fraction and group composition [6], it seems reasonable to study the demercaptanization of fuel oil obtained from gas condensate in the presence of catalysts of different natures.

## EXPERIMENTAL

Catalytic systems based on copper (I) chloride were prepared using heterogeneous heterophase oxidation by oxygen in a hydrocarbon medium. Copper (I) chloride ( $\text{CuCl}$  purified, 99+%, Sigma-Aldrich) was further purified from copper oxychlorides by washing on a Schott glass filter with a 0.2 M  $\text{HCl}$  solution, followed by removing traces of  $\text{HCl}$  by washing with absolute ethanol. An *o*-xylene solution ( $\text{C}_8\text{H}_{10}$ , pure for analysis, Laverna) in petroleum from Chinarevskoye oilfield (TOO Zhaikmunai) was used as a dispersion medium. The following nitrogen-containing ligands were used to synthesize catalysts based on  $\text{CuCl}$ : *N,N*-dibutylethanolamine ( $\text{C}_{10}\text{H}_{23}\text{NO}$ , 99%, Sigma-Aldrich) and diethanolamide of fatty acids from coconut oils (Cocamide DEA,  $\text{RCON}(\text{C}_2\text{H}_4\text{OH})_2$ , where  $n = 14\dots 16$ , 95+%, trade name Awaxan F, OOO Veresk Company). The catalyst was synthesized in a hermetically sealed glass reactor of known volume in an oxygen atmosphere at 70°C with continuous stirring. The degree of  $\text{CuCl}$  conversion was controlled by measuring the pressure in the reactor using a portable manometer of model 8230 made by AZ Instruments. The moment when oxygen consumption stopped was taken as an the of the reac-

tion. A solution of K-1 catalyst was produced that contained the following: 8 wt % CuCl, 14 wt % dibutylethanolamine, 10 wt % diethanolamide of fatty acids from coconut oils, 16 wt % *o*-xylene, and 52 wt % petroleum.

Water-soluble catalysts were prepared via the interaction of the prescribed amounts of copper (II) sulfate (pentahydrate,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , 99+%, Sigma-Aldrich) and copper (II) acetate (monohydrate,  $(\text{CH}_3\text{COO})_2\text{Cu} \cdot \text{H}_2\text{O}$ , 98+%, Sigma-Aldrich) with monoethanolamine (MEA, 2-ethanolamine, extra pure, Laverna) or an MEA/water solution at 40°C and continuous stirring in air atmosphere. The mentioned reagents were not subjected to further purification. A nonionic surfactant, diethanolamide of fatty acids from coconut oils, was used to improve compatibility with hydrocarbons. The produced K-2 catalyst contained the following: 10 wt %  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , 8 wt %  $(\text{CH}_3\text{COO})_2\text{Cu} \cdot \text{H}_2\text{O}$ , 20 wt %  $\text{H}_2\text{O}$ , and 62 wt % MEA. The surfactant was introduced at a rate of 0.75 g per 100 g of the catalyst. Catalysts with lower contents of metal salts (10, 5, and 2.5 wt %) were prepared by diluting the K-2 catalyst with the prescribed amount of MEA/water solution with a monoethanolamine concentration of 80 wt %, which allowed the water concentration to be maintained in the final product (20 wt %).

The investigation of the activity of catalysts in the reaction of  $\text{H}_2\text{S}$ , methyl-, ethyl-, and total mercaptans RSH conversion was conducted using fuel oil produced from gas condensate (GPZ OOO Gazprom Dobycha Astrakhan) and model mixtures on its basis. The mixtures were prepared by introducing solutions with prescribed concentrations of hydrogen sulfide in diesel fuel (with oxygen removed from it) to the initial fuel oil. Hydrogen sulfide from a standard  $\text{H}_2\text{S} + \text{N}_2$  mixture with a hydrogen sulfide content of 24.9 vol % (OOO Monitoring) was used. The purification process was conducted at 90°C (typical temperature of fuel oil transport to storage vessels) and 110°C in hermetically sealed glass reactors equipped with water jacket (for processes up to 90°C) or flexible heating elements (for processes above 90°C). The ratio of gaseous and liquid phase volumes in the reaction zone was  $V_g/V_l = 0.2/3.5$ .

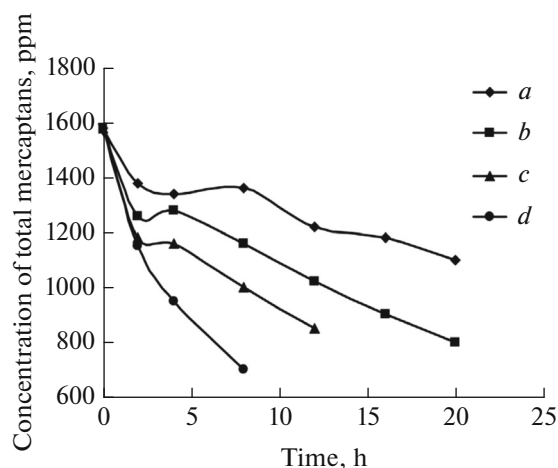
The content of hydrogen sulfide and mercaptan sulfur was determined using a potentiometric technique according to the GOST State Standard P 52030-2003. An EVL-1M3.1 silver chloride electrode was used as a reference electrode and an IONIKS 111.050 sulfide-selective electrode was used as the sensor electrode. During a selective analysis of the liquid phase, hydrogen sulfide and ethyl mercaptan were separated from fuel oil by the helium flow, followed by freezing out in a trap at a temperature of liquid nitrogen of -196°C with the prescribed amount of heptane used as an absorber. The selective analysis of  $\text{H}_2\text{S}$  and  $\text{C}_2\text{H}_5\text{SH}$  content in liquid and vapor phases of the initial fuel oil and model mixtures was carried out using

gas chromatography on a Varian 3800 chromatograph with an HP-5 50 m  $\times$  0.32 mm capillary column (Agilent Technologies). Helium was used as a carrier gas. Detection was conducted using a pulsed flame photometer detector (*S* mode). The Galaxie 1.9 program was used to control the chromatograph, as well as to collect and process the experimental data. The determination of hydrogen sulfide, methyl-, and ethyl mercaptan contents using the Industrial Standard MI-8-TsZL-OTK-2003 "Determination of Hydrogen Sulfide and Mercaptan Sulfur in a Stable Condensate, Petroleum, and Fuel Oil" was conducted at the laboratory of the GPZ OOO Gazprom Dobycha Astrakhan.

## RESULTS AND DISCUSSION

The initial raw materials (crude oil, gas condensate) contain significant amounts of sulfur-containing compounds of various nature (sulfides, thiophenes, mercaptans, elemental sulfur, etc.), the concentration and distribution of which are defined by the nature of petroleum in each particular oilfield. Hydrogen sulfide, methyl-, and ethyl mercaptans are mainly removed during oil conditioning [12, 13]. The second emergence of hydrogen sulfide in heavy fractions during primary rectification is related to the partial thermal destruction (temperature above 200°C) of sulfur-containing compounds. In addition, low-volatile high-molecular-weight mercaptans from the initial raw material can accumulate in heavy fractions. In particular, it follows from the data of the chromatographic analysis of straight run fuel oil from the gas concentrate of the Astrakhan GPZ that the content of methyl- and ethyl mercaptans, which are the most toxic and corrosive sulfur-containing admixtures, varies in the range of 70–150 ppm and, according to the data of potentiometric analysis, the total amount of mercaptans reaches 1500–1700 ppm or possibly even higher. In connection with this, it was important to consider the effect of a high concentration of total mercaptans on the degree of conversion of low-molecular-weight mercaptans in addition to the investigation of the effect of the component, fractional, and group hydrocarbon composition on the process of catalytic demercaptanization.

Time dependence of the total mercaptan content during oxidative demercaptanization of fuel oil from gas condensate at different concentrations (75, 133, and 233 ppm) of hydrocarbon-soluble catalyst K-1 is presented in Fig. 1. One can clearly see that the kinetic dependences demonstrate similar trends and differ in the rapid reduction of mercaptan content in the early stages of the process (2–2.5 h). The total mercaptan content decreases to 1380 ppm already at the catalyst concentration of 75 ppm (Fig. 1a). The increase in the catalyst concentration to 133 ppm results in an almost proportional decrease in the amount of oxidized mercaptans. The further increase in the catalyst concentration only slightly affects conversion of thiols during the initial stage (Fig. 1c). Following the sharp initial



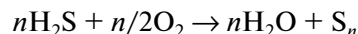
**Fig. 1.** Time dependence of total mercaptan content during oxidative demercaptanization of fuel oil obtained from gas condensate in the presence of K-1 catalyst.  $[S]_0^1 = 14$  ppm ( $\text{CH}_3\text{SH}$ );  $[S]_0 = 82$  ppm ( $\text{C}_2\text{H}_5\text{SH}$ ). Catalyst concentration: (a) 75, (b) 133, (c) 233, (d) 128 ppm. Gas phase is air ( $P = 1$  atm);  $V_g/V_l = 3.5$ ;  $T = 90^\circ\text{C}$  (curves a–c);  $T = 110^\circ\text{C}$  (curve d).

drop in the mercaptan concentration, the period of the almost complete absence of catalyst activity is observed with a duration determined by the concentration of the catalysts (2–6 h). Next, the decrease in the mercaptan content in the fuel oil is observed, which is the most pronounced at high catalyst concentrations (curves 2, 3). The partial restoration of activity can be related to either the reactivation of the catalyst or to side reactions of the conversion of the fuel oil component, which can be readily oxidized (isoparaffins, alkyl-substituted benzene derivatives, and sulfur-containing compounds), for which the availability of induction periods is characteristic [14]. At the same time, it is known that mercaptans are inhibitors of oxidation processes and the observed consumption of mercaptans may be related to the interaction with the formed peroxy radicals. This conclusion is indirectly supported by the emergence of WSABs after 10 or more hours of the process. For example, the pH of an aqueous extract of fuel oil purified at a catalyst concentration of 233 ppm and  $V_g/V_l = 3.5$  is in the range of 5–5.5, and it decreases with an increase in the duration of the process. The similar acidity was observed after 20 h of contact when the catalyst was used at a concentration of 133 ppm. If the concentration of the catalyst was 75 ppm, the emergence of water-soluble acids was not observed under the same conditions of demercaptanization process. An increase in the reaction temperature to  $110^\circ\text{C}$  results in a sharp increase in mercaptan consumption (Fig. 1d) with a simultaneous increase in the acidity of the aqueous extract (pH 4–4.5) and the visual darkening of the fuel oil.

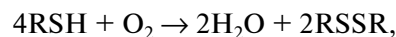
<sup>1</sup> Initial content of mercaptan sulfur.

The objective of fuel-oil conditioning (deodorization) includes the removal of hydrogen sulfide and methyl- and ethyl mercaptans; hence, in addition to evaluating the total mercaptan content, the contents of light sulfur-containing admixtures were specifically analyzed using chromatography. It can be seen from the obtained data that the complete removal of light mercaptans is even observed after 2 h at catalyst concentrations of 133 and 233 ppm (initial content of total mercaptans was 1580 ppm). It is known that, among thiols, low-molecular-weight derivatives are the most active. At the same time, the decrease in the observed rate of methyl- and ethyl mercaptan consumption might be possible at a high concentration of total mercaptans due to the competitive nature of their interactions. When establishing the effect of the total mercaptan concentration on the rate of oxidation of low-molecular-weight mercaptans, it was shown that the introduction of dodecylmercaptan in the system up to a concentration 3300 and 4800 ppm (according to mercaptan sulfur) did not affect the degree of conversion of light sulfur-containing admixtures at a high concentration of the catalyst. For example, the complete removal of ethyl mercaptan was observed after 2 h at a catalyst concentration of 133 ppm. When the catalyst concentration was decreased to 75 ppm, at a total mercaptan content of 4800 ppm, a sharp drop in the degree of ethyl mercaptan conversion to 0.5 was observed with its initial concentration 82 ppm and contact time of 2 h. The complete removal was only observed after 10–12 h.

As was mentioned above, the characteristics of fuel oil changed at a high concentration of the catalyst (133 ppm and above) depending on the contact time and were related to the oxidation of its components. It must be noted that the increase in acidity was observed at high ratio  $V_g/V_l = 3.5$ . Considering this, it seemed reasonable to investigate the effect of the gas-to-liquid phase ratio, which defined the amount of air in the reaction zone on the degree of removal of light mercaptans and the emergence of WSABs. The selected range of the  $V_g/V_l$  ratio = 0.2–0.4 was determined by the stoichiometry of the reaction of hydrogen sulfide oxidation to sulfones and elemental sulfur



and by the oxidation of thiols to disulfides



as well as by the technological limitation of the process conducted under industrial conditions.

The data on the demercaptanization of model mixtures with concentrations of hydrogen sulfide and ethyl mercaptan typical of the fuel oil obtained from gas condensate in the presence of two catalytic systems of different nature are presented in Tables 1 and 2. It can be seen that the complete removal of hydrogen sulfide occurs 2 h after the beginning of the process and does not depend on the concentration of catalysts

**Table 1.** Oxidative demercaptanization of model mixtures in the presence of K-1 catalyst ( $T = 90^\circ\text{C}$ )

<i>Ckt</i> , ppm	<i>t</i> , h	$M_1$		$M_2$		$V_g/V_l$	WSAB
		H <sub>2</sub> S [ <i>S</i> ] <sub>0</sub> = 36 ppm	C <sub>2</sub> H <sub>5</sub> SH [ <i>S</i> ] <sub>0</sub> = 64 ppm	H <sub>2</sub> S [ <i>S</i> ] <sub>0</sub> = 39 ppm	C <sub>2</sub> H <sub>5</sub> SH [ <i>S</i> ] <sub>0</sub> = 110 ppm		
227	2	0	0	0	16	0.4	—
	4	—	—	—	0		Absent
	20	—	—	—	—		Absent
234	2	0	0	0	25	0.2	—
	4	—	—	—	0		Absent
119	2	0	12	0	28	0.4	—
	4	—	0	—	9		—
	8	—	—	—	0		Absent
126	2	0	14	0	38	0.2	—
	4	—	0	—	17		—
	8	—	—	—	9		Absent

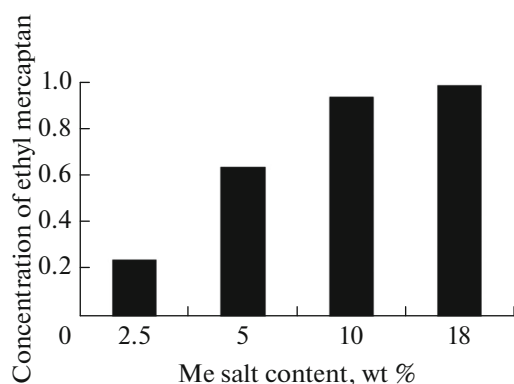
**Table 2.** Oxidative demercaptanization of model mixtures in the presence of K-2 catalyst ( $T = 90^\circ\text{C}$ )

<i>Ckt</i> , ppm	<i>t</i> , h	$M_1$		$M_2$		$V_g/V_l$	WSAB
		H <sub>2</sub> S [ <i>S</i> ] <sub>0</sub> = 42 ppm	C <sub>2</sub> H <sub>5</sub> SH [ <i>S</i> ] <sub>0</sub> = 72 ppm	H <sub>2</sub> S [ <i>S</i> ] <sub>0</sub> = 37 ppm	C <sub>2</sub> H <sub>5</sub> SH [ <i>S</i> ] <sub>0</sub> = 105 ppm		
115	2	0	23	0	34	0.4	—
	4	—	—	—	0		Absent
	8	—	—	—	—		Absent
104	2	14	24	12	56	0.2	—
	4	0	0	0	18		—
	8	—	—	—	11		Absent
62	2	15	34	13	58	0.4	—
	4	0	14	0	12		—
	8	—	0	—	0		Absent

and the volume ratio (Table 1). With a catalyst content of 227–234 ppm and initial concentration of mercaptan sulfur ( $[S]_0$ ) 64 ppm, the removal of the latter was observed after 2 h and did not depend on the  $V_g/V_l$  ratio. The increase in the  $[S]_0$  concentration to 110 ppm causes an increase in the time required for complete removal (4 h). As can be seen from Table 1, the decrease in the catalyst concentration to 119–126 ppm resulted in a significant increase in the contact time (to 8 h or more) for the complete removal of ethyl mercaptan depending on the ratio of gaseous and liquid phases. The concentration of WSABs did not exceed the prescribed threshold in all of the presented experiments [15].

A comparison of the catalyst based on water-soluble copper salts and MEA (K-2) with the one soluble in hydrocarbons (K-1) showed that they demonstrated

similar efficiencies in the process of fuel oil demercaptanization (Tables 1, 2). For example, with a catalyst concentration of 115 ppm, the initial concentration of hydrogen sulfide and ethyl mercaptan 37 and 105 ppm, respectively, and the ratio  $V_g/V_l = 0.4$ , the complete removal of hydrogen sulfide was observed after 2 h and the complete removal of ethyl mercaptan was observed after 4 h. The decrease in the  $V_g/V_l$  ratio to 0.2 or decrease in the catalyst concentration to 62 ppm ( $V_g/V_l = 0.4$ ) resulted in an increase in the time of contact (Table 2). As was noted above, the significant increase in acidity of the final product was observed at the  $V_g/V_l$  ratios above 3.0. As can be seen in Table 2, the decrease in the  $V_g/V_l$  ratio to 0.2...0.4 allows one to eliminate the formation of WSABs in the entire investigated range.



**Fig. 2.** Effect of copper salt concentration in the K-2 catalyst on degree of conversion of ethyl mercaptan.  $T = 90^{\circ}\text{C}$ ,  $V_g/V_l = 0.4$ ;  $t = 4$  h, 37 ppm of hydrogen sulfide sulfur; 105 ppm of mercaptan sulfur. Catalyst concentration ( $110 \pm 10$ ) ppm.

The developed catalysts comprise solutions of metal complexes of copper salt with nitrogen-containing ligands in the water–MEA solution or in hydrocarbon medium. Furthermore, the degree of conversion of hydrogen sulfide and light mercaptans is predominantly determined by the concentration of metal complexes in the reaction mixture. Therefore, it seems reasonable to consider the effect of the transitional metal content in the catalyst solutions on the oxidation of sulfur-containing admixtures in the fuel oil (Fig. 2). It can clearly be seen that the almost twofold reduction in the total metal salt content in the K-2 catalyst is not reflected in the degree of conversion of sulfhydryl derivatives during the oxidation process conducted under the same conditions ( $t = 4$  h,  $T = 90^{\circ}\text{C}$ ,  $V_g/V_l = 0.4$ ). The further reduction in the metal concentration in the initial catalyst resulted in a noticeable decrease in the degree of conversion, and the increase in contact time to 20 h allowed one to increase the degree of purification with a degree of conversion of the ethyl mercaptan that reached 0.84. The decrease in the copper-salt concentration to 2.5 wt % causes a sharp drop in the catalyst activity. By way of illustration, the degree of conversion of ethyl mercaptan is 0.25 at a contact time of 4 h and does not change over time, which can be related to the fact that the threshold concentration of the metal complex introduced into the hydrocarbon medium is reached [16].

## CONCLUSIONS

The conducted investigations showed that the developed catalytic systems based on water- or hydrocarbon-soluble copper metal complexes are effective catalysts in the process of purifying gas-condensate-derived fuel oil from hydrogen sulfide and light mercaptans. The conditions for producing a commercial product that is in compliance with the Russian and

European standards were identified. It was shown that the ratio  $gkt/gS_{\text{tot}}$  can reach a value of 0.4–0.8, which is significantly lower than in the cases where the widely employed neutralizers–absorbers are used and is related to catalytic nature of the process. The close values of the  $gkt/gS_{\text{tot}}$  ratio were obtained during the demercaptanization of petroleum-derived fuel oil and crude oil from different oil fields, which indicated only a slight effect of the nature of hydrocarbon medium on the purification depth and high versatility of the developed catalysts [10, 11]. The secondary emergence of light sulfur-containing products was also not observed during prolonged storage (up to 1 month at  $70^{\circ}\text{C}$  and 1 year at room temperature), which is often observed when neutralizers are applied. The process of purification can be implemented in industry via the constant dispensing of the liquid-phase catalyst and the prescribed amount of air into the fuel oil flow transported to the storage vessel, where the oxidation of sulfhydryl derivatives continues. The intensification of the process can be achieved via the better distribution of the catalysts and air in the fuel oil using additional dispersing units such as, e.g., multisectional diffuser–confuser mixers.

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