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Synthesis and Study of Reducing the Corrosive Activity of Motor Fuels Using Additives of the MEE Series

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Abstract: This scientific work proposes a method for the effective synthesis of bis-carbamate MEE-2. The physicochemical parameters and vibrational spectra of the molecule were studied and the anti-corrosion properties for motor fuels were investigated. It was revealed that the authors' synthesis method is resource-saving, energy-saving and environmentally friendly. IR spectra data prove the structure and composition of the MEE-2 molecule. Studies on the corrosion activity of fuels showed that when bis-carbamate MEE-2 was added to the fuel, the copper plate was clean and corresponded to class 1, which allows it to be used as an anti-corrosion additive for motor fuels.

Keywords: Petrochemical, bis-carbamate, synthesis, IR spectra, corrosion, gasoline, isopropyl alcohol, indicator, plate, concentration.

Introduction. Oil contains phenols (especially a lot of phenols) in oxygen-containing compounds. And also cresols (o-, m-, p-), xylenols, o-ethylphenol, diethylphenols, p-naphthol and other phenols [1]. In the Surkhandarya region of the Republic of Uzbekistan there is the Khaudag field, which contains heavy oil with the following parameters [2]: density at 20 ºC - 979.5 kg/m3, viscosity (kinematic) at 80 ºC - 405.3 mm2/s, viscosity (conditional VU-80) - 53, pour point -23 ºC, coking ability - 12.1% wt, asphaltenes content - 9.5% wt, resins - 50.5% wt, paraffins - 3.85% wt, water - 3, 1% by mass, mechanical impurities - 0.01% by mass, flash point - 165 ºC. Cresols at the Haudag deposit: o-cresol 10.70%, m-cresol 8.32%, p-cresol 3.26%.

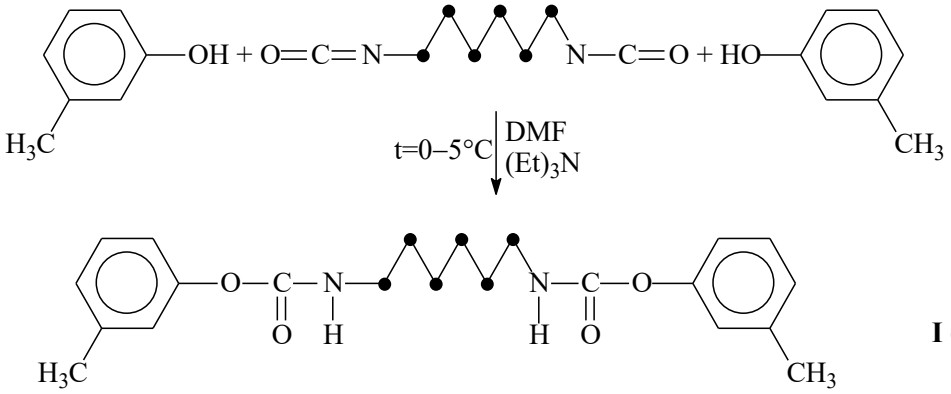
Cresols are used in a wide variety of industries: oil and gas, petrochemical, polymers, medicine, pharmaceuticals, agriculture, etc. Examples include ionol, butylated hydroxytoluene, dibunol, agidol-1, BHT - 2,6-di-tert-butyl-4-methylphenol, lipophilic organic substance, representatives of the class of phenols, which are widely used as an antioxidant in the chemical industry. The development of the chemistry of carbamates and bis-carbamates is largely determined by the wide range of beneficial properties of these substances. They are promising as means of protecting and stimulating plant growth, components in corrosion inhibitors, additives for lubricating oils, medicines; bi- and polyfunctional carbamates are used as polymers [3].

Taking into account the high biological activity of cresol and carbamates and the wide range of applications, it was necessary to determine optimal methods for introducing cresol-carbamate

groups into these types of compounds (1) and to study the dependence of the reactions used due to the mobile proton at the N-H reaction center of substituting functional groups [4 -5].

Materials and Methods. Synthesis of N,N'-hexamethylene bis-[(m-cresolyl)-carbamate] i.e. MEE-2: To 10.8 g (0.1 mol) of meta-cresol add 10 ml of triethylamine, 35 ml of dimethylformamide (DMF), stir, dissolve in 20 ml of DMF at room temperature, add 8.4 g (0.05 mol) of hexamethylene diisocyanate . After the time has elapsed, the reaction mixture is stirred at a temperature of 35-48°C for 3.0 hours, the contents of the flask are poured into a glass and water is added. The resulting precipitate is washed with thin layer chromatography (TLC). After drying, a white powder is obtained, the product yield is 18.16 g (94.6% of theoretical); Melt temperature=131-132°C; Rf=0.71. Found, % C-68.62; H-7.17; N-7.24; Calculated % for $C_{22}H_{28}N_2O_4$ C-68.75; H-7.29; N-7.29 The progress of the reaction and the individuality of the compounds are monitored by TLC on aluminum oxide (II) of the degree of activity with the appearance of spots by iodine vapor. IR spectra of functional groups were recorded in a Nicolet iS50 spectrometer (Th.scientific, USA) at the Center for Advanced Research. To identify the inhibitory properties of bis-carbamate MEE-2, tests were carried out in accordance with the method described in accordance with SAUS 32329.

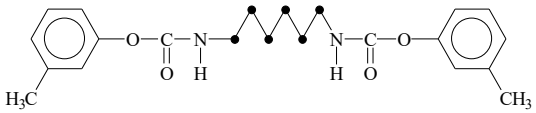
Results and Discussions. By reacting meta-cresol with hexamethylene diisocyanate, new derivatives of N,N'-hexamethylene bis-(m-cresolyl)-carbamate were obtained: A cost-effective, waste-free, energy-saving synthesis was carried out according to scheme 1:



I (1)

The reaction is carried out in a medium of dimethylformamide and the organic base triethylamine at room temperature for 4 hours. It should be noted that bis-carbamate MEE-2 derivatives were obtained in fairly high yield. The physicochemical characteristics of compounds are given in Table 1.

Table 1. Physicochemical properties of bis-carbamate MEE-2

Structural formula	Exit, %	T.me, ^{0}C	R_f	Brutto formula	Element analysis, %		M_m
					Calculated	Found	
					N	N	
	94,6	131-132	0,71	$C_{22}H_{28}N_2O_4$	7,29	7,24	384,47

In the IR spectrum of N,N'–hexamethylene bis-[(m-cresolyl)-carbamate] (Fig. 1), the ring vibration of the aromatic ring appeared in the region of 1487 cm^{-1}, the average stretching vibration characteristic of the C-N bond of the ring was at in the region of 3046 cm^{-1}, and the deformation vibration appeared in the region of 642 cm^{-1}. A highly asymmetrical stretching vibration of the methyl group (CH_3) on the aromatic ring was observed around 2962.49 cm^{-1}. The methylene groups of meta-cresolyl carbamate (-CH_2) have a high asymmetric stretching

vibration in the region of 2927 cm^{-1}, a symmetric stretching vibration in the region of 2859 cm^{-1}, a weak sharp bending stretching vibration in the region of 1461 cm^{-1}, respectively, spindle and pendulum vibrations in the region of 1339.46 and 735.89 cm^{-1}. High stretching vibrations to the amino group (NH) were observed in the region of 3289 cm^{-1}, and stretching vibrations characteristic of the -C-N bond were observed in the region of 1183 cm^{-1}. Intense absorption lines of the carbonyl group (C=O) were observed in the region of 1699 cm^{-1}. Very strong stretching vibrations of the ether bond (C-O-C) in intensity were observed in the absorption region of 1257 cm^{-1}. Based on the IR spectra of N,N'-hexamethylene bis-[(m-cresolyl)-carbamate], we can say that the structure of the substance corresponds to the above scheme 1.

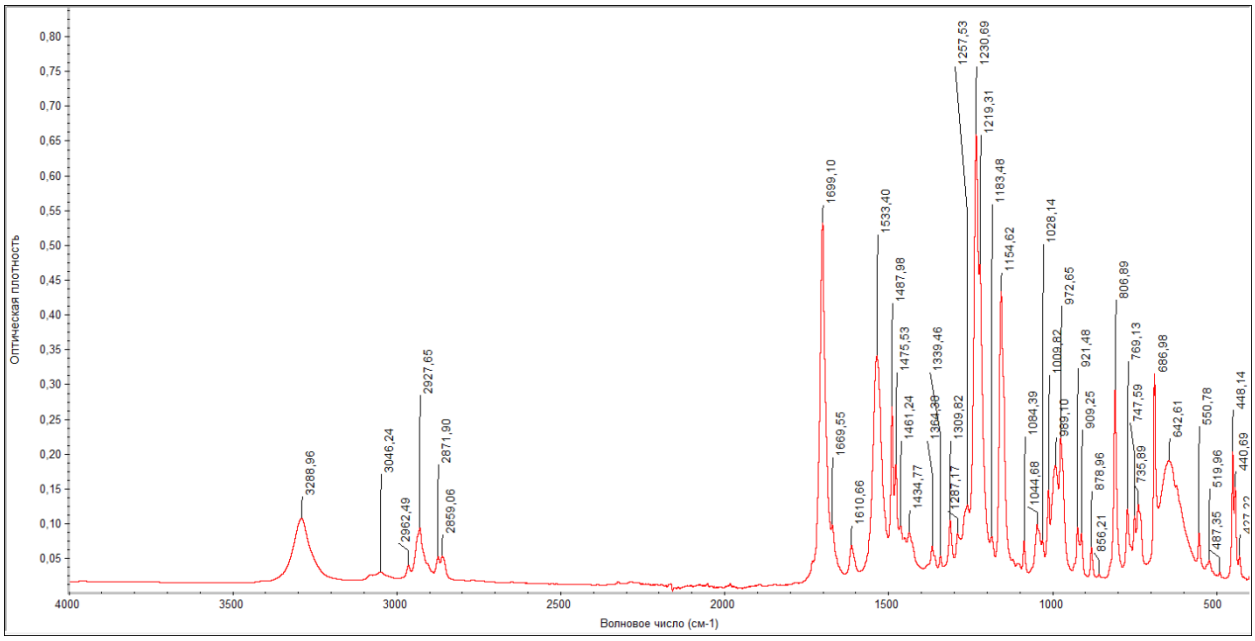


Figure 1. IR spectrum of N,N'–hexamethylene bis-[(m-cresolyl)-carbamate]

Studies have shown [7] that benzotriazole and benzimidazole derivatives are capable of inhibiting corrosion processes due to their ability to be sorbed on the metal surface. Compounds with unshared electron pairs can exhibit inhibitory properties; due to them, they are absorbed on the metal surface, protecting against corrosion. For the same reason, one can expect the manifestation of anti-corrosion activity from carbamates and their derivatives.

To identify the inhibitory properties of N,N'-hexamethylene bis-[(m-cresolyl)-carbamate], further MEE-2 tests were carried out in accordance with the method described in Gureev's work [8]. Since the substances taken for analysis are not soluble in gasoline, 1 liter of sample was prepared by dissolving in isopropyl alcohol (MEE-2 on average 12 mg) and mixing with gasoline in a ratio of 1:19 (by volume). At the same time, the isopropyl alcohol indicator given in UzDST 3031-2015 did not exceed 15%.

AI-80 gasoline was used as a model medium and analysis was carried out according to SAUS 3232. The results are presented in Table 2:

Table 2. Results of a study of the corrosion activity of fuel with the MEE-2 additive

Gasoline concentration, % (wt.)	Corrosion, g/m^2	Appearance of the plate
$1*10^{-4}$	1,48	Spots on records
$1*10^{-4}$	0,65	Spots on records
$1*10^{-3}$	0,12	Dots on plates
$1*10^{-3}$	-	The records are clean

Conclusion. The method of synthesizing bis-carbamate with hexamethylene diisocyanates made it possible to obtain a high yield of the product, and also, the IR spectra of the molecule prove the structure of the resulting bis-carbamate MEE-2. From the results of the study of the corrosive activity of gasoline, we can say that when the MEE-2 additive was added to gasoline in an amount of 0.005%, the copper plate was clean and corresponded to class 1. Final conclusions can be drawn after industrial application, but now we need to continue research work on bis-carbamate MEE-2.

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