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STUDIES IN THE FIELD OF CHEMISTRY OF NITRO COMPOUNDS (TO 100TH BIRTHDAY ANNIVERSARY OF S. S. NOVIKOV)

## Synthesis of Energetic Polynuclear and Polymeric Nitroazole Systems

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Abstract—Interaction of cyanuric chloride and its mono and dichloro derivatives with ammonium or sodium 4-nitro-1,2,3-triazolates and polymer-analogous transformations of tetrazole-containing polymers were used to synthesize polynuclear systems and macromolecular compounds with heterocyclic structures bearing explosophoric groups.

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In development of high-efficiency formulations of solid-propellant, explosive, and gas-generating formulations, particular attention is to be given to polynitrous heterocyclic compounds, whose application promise is determined by their high enthalpies of formation, thermal stability, resistance to mechanical shocks, and comparatively large content of nitrogen. The increase in the energetic characteristics of these compounds is favored by introduction of nitro groups and other explosophoric substituents into heterocyclic fragments [1–3]. Fairly promising energetic products are polynuclear heterocyclic systems containing in their structure nitro groups in addition to triazole, tetrazole, and 1,3,5-triazine rings. The results of syntheses of substances of this kind are reflected in our communication.

## EXPERIMENTAL

Commercial cyanuric chloride of 99.5% purity was used in the study. Its derivatives, 2,4-dichloro-6-methoxy, 2-chloro-4,6-dimethoxy-, 2,4-dichloro-6-diethylamino-, 2-chloro-4,6-diaethylamino-, and 2-chloro-4,6dimorpholino-1,3,5-triazines, were synthesized using the procedures reported in [4, 5]; 4(5)-nitro-1,2,3-triazole, 3-nitro- and 3-azido-1,2,4-triazoles, their N-chloromethyl derivatives, and chloromethyl methylnitramine were synthesized by the methods described in [6–8]. Poly-5-vinyltetrazole (PVT) ( $M = 5 \times 10^5$ ) was produced by radical polymerization of the corresponding monomer as it was done in [9].

We synthesized 2,4,6-tri(4-nitro-1,2,3-triazol-1-yl)-1,3,5-triazine as follows. To a solution of a triethylammonium salt produced from 1.14 g (10 mmol) of 4-nitro-1,2,3-triaziole and 1.02 g (10mmol) of triethylamine in 10 ml of acetone was added dropwise at room temperature 0.61 g (3mmol) of cyanuric chloride in 15 ml of acetone. The reaction mixture was agitated for 1 h and poured into 100 ml of cold water. The precipitate was filtered off and dried in air. Yield 1.5 g, mp > 350°C. IR spectrum, v, cm<sup>-1</sup>: 1560 (NO<sub>2</sub>). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm, DMSO: 160.5 (3C-2,4,6 of triazine), 153.6 (3C-4 of triazole), 124.7 (3C-5 of triazole).

Similarly, we obtained 2-(4-nitro-1,2,3-triazol-1-yl)-4,6-dimethoxytriazine from 1.4 g (8 mmol) of 2-chloro-4,6-dimethoxytriazine, 1 g (8.8 mmol) of triethylamine in 10 ml of acetone. Yield 0.6 g (30%), mp 122–124°C. IR spectrum, v, cm<sup>-1</sup>: 1219 (–O–), 1565 (NO<sub>2</sub>), 1603 (C=N of heteroring). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm, DMSO: 173.2 (C-6 of triazine), 160.5 (2C-2,4 of triazine), 153.6 (2C-4 of triazole), 124.7 (2C-5 of triazole), 56.6 (C, CH<sub>3</sub>). Found: C 28.95, H 1.03, N 46.24. C<sub>8</sub>H<sub>5</sub>N<sub>11</sub>O<sub>5</sub>. Calculated: C 28.66, H 1.49, N 45.97.

We synthesized 2-(4-nitro-5-phenyl-1,2,3-triazol-1yl)-4,6-dimethoxy-1,3,5-triazine from 0.87 g (5 mmol) of 2-chloro-4,6-dimethoxytriazine, 1 g (5.3 mmol) of 4-nitro-5-phenyl-1,2,3-triazole, and 0.59 g (5.3 mmol) of triethylamine in 10 ml of acetone. Yield 0.75 g (47%), mp 142–144°C. IR spectrum, v, cm<sup>-1</sup>: 1600 (Ph), 1565 (NO<sub>2</sub>). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm, DMSO: 173.2 (2C-4,6 of triazine), 163.1 (C-2 of triazine), 151.5 (C-4 of triazole), 130.3 (C-*p* of Ph), 129.1 (C-*o* of Ph), 126.3 (C-*i* of Ph). Found: C 46.72, H 3.02, N 31.11. C<sub>13</sub>H<sub>11</sub>N<sub>7</sub>O<sub>4</sub>. Calculated: C 47.42, H 3.34, N 29.79.

We synthesized 2,4-di(4-nitro-1,2,3-triazol-1-yl)-6methoxy-1,3,5-triazine as follows. To a solution of 0.5 g (2.5 mmol) of 2,4-dichloro-6-methoxytriazine and 0.7 g (6.1 mmol) of 4(5)-nitro-1,2,3-triazole in 10 ml of acetone was added in portions, at 25°C under agitation, 0.51 g (0.61 mmol) NaHCO<sub>3</sub> in 1 ml of water. The mixture was agitated for 2 h, allowed to stay overnight, and poured into 50 ml of cold water. The precipitate was filtered of and dried in air. Yield 0.62 g (69%), mp 131–133°C. IR spectrum, v, cm<sup>-1</sup>: 1219 (–O–), 1565 (NO<sub>2</sub>), 1603 (C=N of heteroring). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm, DMSO: 173.2 (C-6 of triazine), 160.5 (2 C-2,4 of triazine), 153.6 (2 C-4 of triazole), 124.7 (2 C-5 of triazole), 56.6 s (CH<sub>3</sub>). Found: C 28.95, H 1.03, N 46.24. C<sub>8</sub>H<sub>5</sub>N<sub>11</sub>O<sub>5</sub>. Calculated: C 28.66, H 1.49, N 45.97.

In similar way we obtained 2,4-di(4-nitro-1,2,3-triazol-1-yl) from 0.5 g (2.3 mmol) of 2,4-dichloro-6diethylaminotriazine, 0.52 g (4.5 mmol) of 4(5)-nitro-1,2,3-triazole, and 0.38 g (4.5 mmol) of NaHCO<sub>3</sub> in 10 ml of acetone and 1 ml of water. Yield 0.63 g (75%), mp 206–208°C (from DMFA–ethanol). IR spectrum, v, cm<sup>-1</sup>: 1560 (NO<sub>2</sub>). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm, DMSO: 163.2 (C-6 of triazine), 159.2 (2C-2,4 of triazine), 152.3 (2C-4 of triazole), 123.8 (2C-5 of triazole), 41.5 (2C, CH<sub>2</sub>), 12.5 (2C, CH<sub>3</sub>). Found: C 35.11, H 3.44, N 44.21. C<sub>11</sub>H<sub>12</sub>N<sub>12</sub>O<sub>4</sub>. Calculated: C 35.11, H 3.19, N 44.68.

PVT was alkylated with chloromethyl derivatives of triazoles and methylnitramine with an equimolar amount of the corresponding alkylating agent gradually added to a 1.5 M solution of a triethylammonium salt of PVT in DMFA, with the reaction mixture then agitated at a temperature of 60°C for 6 h. The resulting polymeric products were precipitated into acidified water, washed with distilled water, and dried in air at room temperature. The samples obtained were washed with diethyl ether in a Soxhlet apparatus and dried in a vacuum to constant mass.

Metal complexes of 4(5)-nitro-1,2,3-triazole and mixed triazole-tetrazole polymeric complexes with

copper (2+) and nickel (2+) ions were produced by pouring together, under agitation, aqueous-acetonitrile solutions of nitrotriazole or its equimolar mixture of PVT and a metal chloride in a solvent at room temperature. The precipitates formed were washed with water to remove unreacted salts and nitrotriazole. The precipitates obtained in systems containing a polymer were additionally treated with hot DMFA to remove homo complexes of nitrotriazole with metal ions. The share of the fraction soluble in DMFA did not exceed 3–4 wt % relative to the total yield of metal complexes. The precipitates were filtered off and dried in a vacuum to constant mass. The composition of the products obtained was calculated from elemental analysis data.

The density of the polymers was found pycnometrically [10]. Potentiometric measurements were made with an EV-74 ion meter. <sup>13</sup>C NMR spectra were recorded with a Varian VXR-500S spectrometer (working frequency 126 MHz) in acetone and DMSO, with signals of their methyl groups serving as the internal standard (29.5 and 39.5 ppm, respectively). IR spectra were recorded on Infralum FT-801 and Specord M-80 instruments in Vaseline oil on KBr glasses. The elemental analysis was made on a FLASH BA 1112 Series CHN-analyzer. The reactions were monitored by TLC on plates with a fixed Silufol UF 254 layer, hexane–ethyl acetate as eluent, development with iodine vapor.

As a starting compound for formation of polynuclear systems serves 4-nitro-1,2,3-triazole (**I**), whose synthesis and properties are reflected in a recently published review [1]. Exhibiting properties of an N–H acid, nitrotriazole (**I**) interacts with bases to give salts and, in the form of a triazolate anion, reacts as a nucleophile with various electrophilic reagents to give N-substitution products. This property was used in synthesis of 4-nitro-1,2,3-triazole-containing 1,3,5-triazines by reacting chlorosubstituted triazines with nitrotriazole (**I**) in a basic medium (Scheme 1).

As demonstrated by experiments, cyanuric chloride (II) actively reacts with three moles of nitrotriazole (I) in an aqueous–acetone solution at 20–25°C in the presence of sodium hydrocarbonate or triethanolamine to give tris(4-nitro-1,2,3-triazolyl)-1,3,5-triazine (III). Substitution of chlorine in 2,4-dichloro-6-methoxy- (IVa) and 2-chloro-4,6-dimethoxy-1,3,5-triazines (Va), and also in 2,4-dichloro-6-diethylamino-1,3,5-triazine (IVb), requires a prolonged reaction. As a result, mono- (VIa,





**IV, VII**: R = OM (a),  $R = N(Et)_2$  (b); V: R = OM (a),  $R = N(Et)_2$  (b),  $R = N(CH_2)_2O$  (c); VI: R = OM,  $R^1 = H$  (a), R = OM,  $R^1 = Ph$  (b).

**VIb**), and di(nitrotriazolyl)triazines (**VIIa**, **VIIb**) are formed. 2-Chloro-4,6-diethylamino-1,3,5-triazine (**Vb**) and 2-chloro-4,6-dimorpholino-1,3,5-triazine (**Vc**) could not at all be introduced into a reaction with nitrotriazole (**I**), even with sodium hydroxide used as a base. Probably, the presence of two amino groups in compounds **Vb**, **Vc** substantially diminishes the mobility of the chlorine atom in position 6 of the triazine ring.

Reactions of substitution of the chlorine atom in the triazine ring with nitrotriazole moieties can yield isomeric compounds via formation of a C–N bond involving various nitrogen atoms of the triazole ring. However, the reaction, as a rule, predominantly yields one of isomers, which is isolated by fractional crystallization of the reaction products. This is also confirmed by spectral studies of the polynuclear compounds obtained.

The IR spectra of the nitro compounds obtained, **VIa**, **VIb** and **VIIa**, **VIIb**, show absorption bands related to stretching vibrations of the nitro group (1565 cm<sup>-1</sup>) and stretching-deformation vibrations of C=N bonds of the heterorings (1603 cm<sup>-1</sup>). The <sup>13</sup>C NMR spectra show signals characteristic of nuclei of the triazine rings bound to the methoxy group at 171–173 ppm and signals of carbons having nitrotriazole rings as substituents at 159–

163 ppm. Signals characteristic of the  $C_5$  carbon atom are observed at 122–124 ppm, and signals of the  $C_4$  carbon atom of the triazole ring bonded to a nitro group, at around 153.5 ppm. Thus, <sup>13</sup>C NMR spectroscopic data demonstrate that nitrotriazole moieties exist in the polynuclear systems we isolated, supposedly in the (4-nitro-1,2,3-triazol-1-yl)-isomeric form.

Nitrotriazole (I) in the form of a triazolate anion can react as a nucleophile with macromolecular electrophilic reagents having at their backbone substituents capable of substitution under the action of nucleophilic reagents. For example, the reaction of halogen substitution in vinyl chloride moieties has been used to synthesize highly energetic nitrotriazole-tetrazole–containing polymeric products, which cannot be obtained by polymerization. These copolymers **VIII** have been obtained by reacting sodium 4-nitro-1,2,3-triazolate with a copolymer of vinyl chloride with 2-methyl-5-vinyltetrazole in DMFA at 120–150°C [11] (Scheme 2).

In contrast to the low-molecular-weight polynuclear compounds considered above, the copolymers obtained contain two isomeric vinylnitrotriazole monomer units. The ratio between the isomeric fragments characterized in <sup>1</sup>H NMR spectra by signals at 9.1 ( $H_4$ ) and 8.59 ppm





Scheme 3.



 $(H_5)$  (5-nitro-1,2,3-triazol-1-yl isomer and 4-nitro-1,2,3-triazol-1-yl isomer, respectively) is 1 : 3.

Polymeric products combining nitrotriazole and tetrazole moieties in their structure can synthesized using another approach based on the ability of compounds with N–H unsubstituted tetrazole rings to form salts in interaction with bases and to react in the form of a tetrazolate anion as a nucleophile with various electrophilic reagents. The role of such nucleophilic substrates can be played in the presence of bases, e.g., triethylamine, by monomer units of poly-5-vinyltetrazole (**IX**). In this study, we used chloromethyl derivatives of nitro-1,2,3- and 1,2,4-triazoles, azido-

1,2,4-triazole, and methylnitramine as alkylating electrophilic agents. We used the reaction on Scheme 3 to synthesize N-substituted tetrazole-containing polymeric products bearing nitroazole and other energetic moieties as substituents from polymer **IX**. The conditions and results of the reaction are listed in Table 1. Under the chosen conditions, the degree of substitution did not exceed 85%, i.e., the resulting polymeric products (**Xa–Xd**) are copolymers containing monomer units of substituted and unsubstituted 5-vinyl-terazoe. Raising the concentration of chloromethyl reagents and elevating the process temperature resulted in that, already in the early stages of the reaction, gelation leading to a decrease in the

Table 1.	. C	conditions and	results of	f mod	ificati	ion of	pol	ly-5-viny	ltetrazo	le. I	Poymer : al	lky	lating	g agent =	1:1	I, DMFA	60°C	, 6 h
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Sample			Solubility						
	Conversion, %	Density g cm <sup>-3</sup>	DMFA, DMSO	0.1 M NaOH solution in water	conc. H <sub>2</sub> SO <sub>4</sub>				
IXa	84	1.38	swells						
IXb	80	1.30	dissolves	swells	decomposes				
IXc	82	1.52	the same	insoluble	dissolves				
IXd	76	1.44	swells	insoluble	swells				

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degree of substitution of tetrazole units in the modification product was observed in the system.

The structure of polymeric products formed in modification of polymer IX was confirmed by spectral methods and results of potentiometric titration. The IR spectra of samples Xc, Xd contain high-intensity absorption bands at 1300-1310 and 1550-1560 cm<sup>-1</sup>, associated with symmetric and antisymmetric vibrations of the nitro group, and a band at 830–840 cm<sup>-1</sup>, related to its deformation vibrations. Strong bands at 1515 and 1520 cm<sup>-1</sup> are associated with vibrations of, respectively, 1,2,4- and 1,2,3-triazole rings containing a nitro group. The IR spectrum of sample Xb shows a strong absorption band at 2140 cm<sup>-1</sup>, related to antisymmetric vibrations of the azide group, and a band at 1530 cm<sup>-1</sup>, characterizing vibrations of the triazole ring. As a rule, the absorption bands of the tetrazole ring at 1500-1540 cm<sup>-1</sup> have low intensity and are overlapped with neighboring high-intensity bands. The broad high-intensity bands in the IR spectrum of sample Xa at 1265, 1295, and 1540  $cm^{-1}$  are due to symmetric and antisymmetric vibrations of the nitramine group, and vibrations of the tetrazole ring are characterized by a low-intensity absorption band at 1540 cm<sup>-1</sup>, and vibrations of the tetrazole ring are characterized by a low-intensity absorption band at 1540 cm<sup>-1</sup>.

The reaction of alkylation of a 5-substituted tetrazole ring in a basic medium always yields two isomeric 1,5- and 2,5-disubstituted products [12]. A similar pattern was also observed in alkylation of a polymeric substrate IX with chloromethyl derivatives of triazoles and nitramines. For example, the presence of two signals from the carbon atom of the tetrazole ring, with chemical shifts of 167.8 and 163.0 ppm, and two signals from carbon atoms of the methylene bridges connecting the triazole and tetrazole rings, at 62.67 and 57.6 ppm, indicates that the macromolecule contains two isomeric 1,5- and 2,5-disubstituted tetrazole rings. The triazole ring is characterized by a singlet with a chemical shift of 157.0 ppm, related to the signal of a cyclic carbon bonded to the nitro group, and by a doublet at 148.3 ppm, associated with the cyclic CH moiety. To the hydrocarbon backbone correspond broadened signals at 37 and 30 ppm, associated with the CH and CH<sub>2</sub> groups.

The resulting N-substituted polyvinyltetrazoles (Xa–Xd) are uncolored powder-like substances with a comparatively high density and limited solubility

(Table 1). Owing to the amphoteric nature of N-Hunsubstituted tetrazole ring, the starting polyvinyltetrazole (IX) is soluble in all of the liquids mentioned above. Most of its modification products lose the ability to be dissolved and acquire a property of limited swelling. Possibly, this is due to side processes of "cross-linking" of macromolecular chains, with nitro and azido groups involved. The possibility of substitution of these groups in the triazole ring with the tetrazolate anion was found for reactions involving model compounds, N-substituted nitro and azido triazoles, in which only nitro and azido groups are subject to nucleophilic substitution. The involvement of these groups in the substitution reactions could be reliably confirmed only for systems in which NaI was used as a catalyst. However, occurrence of side reactions involving nitro and azido groups cannot be fully ruled out under the conditions of the reaction of alkylation of polyvinyltetrazole (IX) without a catalyst, because the number of intermacromolecular "cross-links," which can lead to loss of solubility by a polymeric sample is within the error of their analytical determination.

The presence of residual monomer units with N–H-unsubstituted tetrazole rings in modified polyvinyltetrazoles Xa-Xd favors manifestation of polyelectrolyte properties by these samples and, in particular, ability to swell in aqueous alkali solutions.

One more way to combine tetrazole and nitrotriazole moieties in a polymeric structure consists in that N-Hunsubstituted derivatives of tetrazole and 1,2,3-triazole to be involved in complexation reactions with metal ions. For example, polyvinyltetrazole (IX) directly forms, upon mechanical mixing of solutions of the polymer and copper and nickel chlorides at room temperature, salt-like complexes with Cu<sup>2+</sup> and Ni<sup>2+</sup> ions of composition L :  $M^{2+} = 2$ : 1, in which tetrazolate anions acts as a ligand (L) [13]. It was found that nitrotriazole I behaved in about the same way in reactions with copper and nickel salts to give ionic complexes of similar composition exclusively with nitrotriazolate ligands (Table 2). According to elemental analysis data, the metal complexes contain no chlorine. In contrast to polymeric tetrazole-containing complexes, which are insoluble in organic solvents and water, products of reactions with nitrotriazole are soluble in DMFA. Taking into account that tetrazole- and triazolecontaining compounds similarly behave in reactions with metal salts and the acidity constants of these N-H acids are close { $pK_a$  of nitrotriazole (I) is 4.8 [14], and

Hataraavalia raagant	MCl	Viold %	Fou	nd, %	$\mathbf{L} \cdot \mathbf{M}^{2+}$ ratio mal : a at		
Heterocyclic reagent	IVIC 12	1 1010, 70	С	N	E. Wi Tatio, mor. g-at		
I	CuCl <sub>2</sub>	98	16.4	37.9	2:1		
Ι	NiCl <sub>2</sub>	93	16.5	38.8	2:1		
I + IX	CuCl <sub>2</sub>	95	24.7	42.0	2:1		
I + IX	NiCl <sub>2</sub>	97	23.9	42.9	2:1		

**Table 2.** Complexation of 4(5)-nitro-1,2,3-triazole (I) and its 1 : 1 mixture with poly-5-vinyltetrazole (IX) with metal salts (MCl<sub>2</sub>)

 $pK_0$  of polyvinyltetrazole (IX) is 4.65 [11]}, we made an attempt to form mixed metal complexes simultaneously containing tetrazolate and nitroazolate ligands. By mixing aqueous-acetonitrile solutions of 1 : 1 mixtures of the polymer and nitrotriazole with solutions of copper and nickel chlorides, we obtained, in nearly quantitative yield, insoluble colored powder-like products containing no elementary chlorine according to elemental analysis data (Table 2).

An analysis of the IR spectra of the compounds synthesized demonstrated that products formed an equimolar mixture of a tetrazole-containing polymer (IX) and nitrotriazole (I) contains both types of heterorings. This is indicated by the presence of absorption bands characteristic of model homo complexes of, respectively, polyvinyltetrazole and nitrotriazole with Cu<sup>2+</sup> and Ni<sup>2+</sup> ions. To the absorption by the nitrotriazolate moiety belong bands at 1650-1655, 1240-1255, 1290-1295, and 825-830 cm<sup>-1</sup>, associated with various kinds of vibrations of the nitro group, and the absorption band of the nitroazolate anion at 1535 cm<sup>-1</sup>. Vibrations of the tetrazolate ring give rise to the absorption band at 1060 cm<sup>-1</sup> (other bands related to vibrations of tetrazole and triazole rings in the IR spectra coincide in frequency). Taking into account the aforesaid, elemental analysis data, and absence of solubility in the products synthesized, we can suggest that a macromolecular compound containing in their structure fragments of a "mixed" metal complex of composition  $(L_1 + L_2)$ :  $M^{2+} = 2 : 1$  (Scheme 4).



Thus, one more way is found to obtain nitrotriazolecontaining polymers whose synthesis by polymerization could not be performed so far.

## CONCLUSIONS

(1) Various energetic polynuclear and polymeric compounds bearing nitro groups were synthesized on the basis of 4(5)-nitro-1,2,3-triazole.

(2) A method for "linking" of nitrotriazole rings to macromolecular compounds by introduction of anchor electrophilic groups capable of reacting with nucleophilic moieties of polymers into triazole rings was developed.

(3) It was shown that nitrotriazole and tetrazole moieties can be combined in the structure of a polymeric compound via the reaction of joint complexation of nitrotriazole and a tetrazole-containing polymer with metal ions.

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