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Dedicated to the Full Member of the Russian Academy of Sciences
V. A.Tartakovsky on occasion of his 75th birthday

# Synthesis and Molecular Structure of 2-Methoxy-4-Amino-6-dinitromethyl-1,3,5-triazines Zwitterionic Salts

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**Abstract**—Zwitterionic salts of 2-methoxy-4-amino-6-dinitromethyl-1,3,5-triazines were synthesized. The structure of the compounds and the site of nitrogen protonation in the 1,3,5-triazine ring were established based on IR and $^1$H NMR spectra and on the data of X-ray diffraction analysis.

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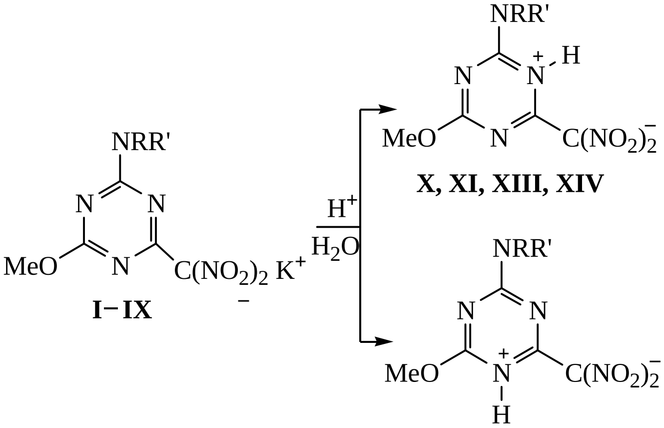
Zwitterionic heterocyclic compounds with space-separated positive and negative charges are interesting from the structural viewpoint subjects of organic chemistry. Up till now no zwitterionic compounds have been obtained in the series of 1,3,5-triazine derivatives. In the case of aminodinitromethyl 1,3,5-triazine derivatives a formation of a number of versatile structures is presumable distinguished by the protonation site on the nitrogen atoms in the 1,3,5-triazine ring and by localization or delocalization of the positive and negative charges (three endocylic nitrogens of 1,3,5-triazine, amino group nitrogen, and dinitromethyl group).

The preparation of dinitromethyl-1,3,5-triazine zwitterionic derivatives was a fairly simple procedure: A water solution or dispersion of 2-methoxy-4-amino-6-dinitromethyl-1,3,5-triazine potassium salts **I–IX** was treated with an equimolar amount of acid, and therewith the sparingly soluble in water 2-methoxy-4-amino-6-dinitromethyl-1,3,5-triazine zwitterionic salts **X–XVIII** precipitated. Salts **X–XVIII** synthesized are crystalline compounds, of color from pale yellow to bright yellow presumably due to the presence in the molecule of the ionized dinitromethyl group. In the IR spectra of all zwitterionic salts absorption bands appear characteristic of the symmetric and asymmetric stretching vibrations of the nitro group in the region 1620–1580 and 1390–1320 cm$^{-1}$ [1]. However the spectra lack a set of absorption bands characteristic of a dinitromethyl anion where two nitro groups are involved in conjugation [2]. The IR spectra of salts **X**–**XVIII** contain the absorption band of the NH group at 3120–3220 cm$^{-1}$, in the $^1$H NMR spectra the signal of this proton is observed as a singlet at 7.80–

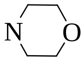


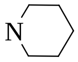
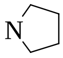
**I−IX**

**X, XI, XIII, XIV**

**XII, XV−XVIII**

NRR' = NH$_2$ (**I, X**), NHMe (**II, XI**), NMe$_2$ (**III, XII**), NHEt (**IV, XIII**),

NHPr (**V, XIV**),  (**VI, XV**), NEt$_2$ (**VII, XVI**),

 (**VIII, XVII**),  (**IX, XVIII**).

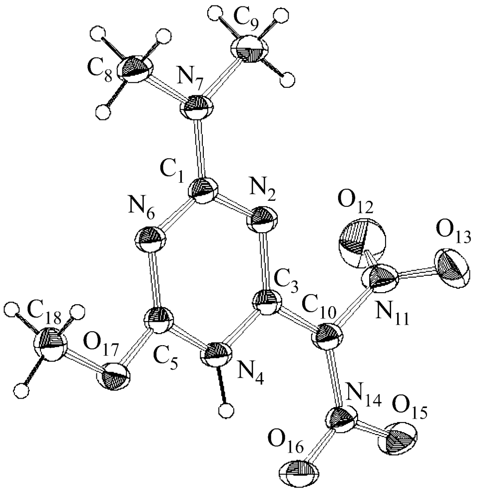


**Fig. 1.** Molecular structure of (4-dimethylamino-6-methoxy-1,3,5-triazin-1-io-2-yl)dinitromethanide (**XII**).

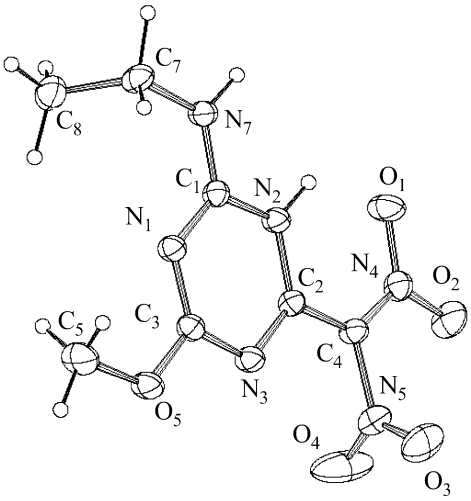
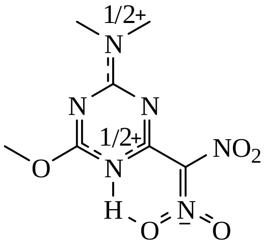


**Fig. 2.** Molecular structure of (4-methoxy-6-ethylamino-1,3,5-triazin-1-io-2-yl)dinitromethanide (**XIII**).

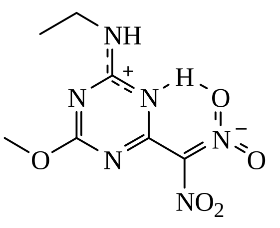
8.00 ppm. No absorption in the region 2500–3000 cm$^{-1}$ of the IR spectra of compounds **X–XVIII** shows that the structure of the molecule does not contain NH$^+$ fragment and ammonium group N$^+$HRR'. Thus a preliminary conclusion follows that protonation occurs at one of the nitrogens of the 1,3,5-triazine ring. However the data of IR and $^1$H NMR spectroscopy are not sufficient for establishing the precise point of the protonation and the distribution of the positive charge in the molecule.

The detailed structure of synthesized zwitterionic salts was investigated by X-ray diffraction analysis by an example of compounds **XII** and **XIII** (Figs. 1 and 2). It was established that the structure of the compounds obtained, in particular, which of the nitrogens in the 1,3,5-triazine ring was protonated, was governed by the structure of the exocyclic amino group. In reaction with acid of amino and monoalkylamino derivatives **I, II, IV**, and **V** the protonation occurred at the nitrogen situated between the dinitromethyl and amino groups whereas in dialkylamino derivatives **III, VI–IX** the protonated endocyclic nitrogen was located between the dinitromethyl and methoxy groups.

The X-ray diffraction analysis revealed a number of interesting and unexpected features in the structure of zwitterionic salts **XII** and **XIII**. In both molecules the 1,3,5-triazine ring, exocyclic bonds C–O, C–N, C–C, and one of the nitro groups of the dinitromethyl moiety are located in the same plane. The second nitro group in the dinitromethyl moiety is turned from the common plane of the molecule by 79.92 (**XII**) and 81.55 deg (**XIII**). Electron-acceptor character of the 1,3,5-triazine ring causes a strong conjugation between the ring and the unshared electron pair of the exocyclic nitrogen of the dimethylamino and ethylamino groups. This interaction results in a considerable shortening of the $C^1$–$N^7$ bond in compounds **XII** and **XIII** to the length value of a sesquialteral bond and in increasing the bond angles at the $C^1$ and $N^7$ to 117–120 deg. This trend is conserved also for the more electronegative oxygen of the methoxy group resulting in shortening of the C–O bond to 1.31–1.32 Å. The protonation of the 1,3,5-triazine ring led unexpectedly to the lengthening of the ring bonds: In compound **XII** $C^5$–$N^4$ and $N^4$–$C^3$, and in compound **XIII** $C^1$–$N^2$ and $N^2$–$C^2$ bonds were 1.36–1.37 Å (comparable with the length of C–N bond in cyanuric acid [3] and longer than in 2,4,6-triamino-1,3,5-triazine [4] and covalent polynitromethyl-1,3,5-triazines [5, 6]). Finally, the protonation of the 1,3,5-triazine ring and the formation of zwitterionic salts resulted in alteration of the conformation of the dinitromethyl fragment of the molecule. In the alkaline salts of the 1,3,5-triazine dinitromethyl derivatives the dinitromethyl group is planar and is completely turned from the plane of the rest of molecule [7]. In zwitterionic salts **XII** and **XIII** one nitro group as already mentioned is deviated from the plane of the molecule and does not take part in the delocalization of the negative charge. The second nitro group is located in the plane of the molecule and is involved in conjugation with the 1,3,5-triazine ring as shown by the considerable shortening of bonds $N^{14}$–$C^{10}$–$C^3$ (**XII**) and $N^4$–$C^4$–$C^2$ (**XIII**). This structure is obviously stabilized by an internal hydrogen bond between the



**XIIA**



**XIIIA**

oxygen of the nitro group and the hydrogen of the NH group of the ring: $N^{4}$–$H^{4}\cdots O^{16}$ (**XII**), parameters of the hydrogen bond: $N^{4}$–$H^{4}$ 0.925 Å, $H^{4}\cdots O^{16}$ 1.874 Å, $N^{4}\cdots O^{16}$ 2.629 Å, $\angle N^{4}$–$H^{4}\cdots O^{16}$ 137.20 deg); $N^{2}$–$H^{2}\cdots O^{1}$ (**XIII**), parameters of the hydrogen bond: $N^{2}$–$H^{2}$ 0.917 Å, $H^{2}\cdots O^{1}$ 1.988 Å, $N^{2}\cdots O^{1}$ 2.634 Å, $\angle$ $N^{2}$–$H^{2}\cdots O^{1}$ 126.07 deg). Parameters of the nitro group conjugated with the ring (bond lengths C–N, N–O, angles CNO and ONO) indicate that on this nitro group the negative charge is delocalized. Similar deviation from coplanarity of dinitromethyl group was formerly described in the aliphatic series for dinitroacetamide potassium salt [8].

Thus from all the probable structures depicting the distribution of positive and negative charges in compounds **XII** and **XIII** the largest contribution belongs to the structures **XIIA** and **XIIIA**.

## EXPERIMENTAL

Potassium salts **I–IX** were synthesized by procedure [9].

IR spectra were recorded on a spectrophotometer Avatar 360 ESP from KBr pellets. $^{1}H$ NMR spectra were registered on spectrometers Bruker WP 80 DS (80 MHz) and Bruker AM-300 (300 MHz) in acetone-$d_6$ (compound **XI** in DMSO-$d_6$), internal reference HMDS.

X-ray diffraction study of compounds **XII** (1124 reflections measured) and **XIII** (2175 reflections measured) was performed on a diffractometer Enraf-Nonius Kappa CCD (298 K, λ 0.71073 Å Mo$K_{\alpha}$, graphite monochromator, ω/2Θ-scanning, $\Theta_{max}$ 26.33 deg). The primary treatment of the diffraction patterns was carried out using a program Denzo, the calculations were done along MaXus software. The structure was solved by the direct method using SIR92 software and refined by the least squares procedure in an anisotropic approximation for nonhydrogen atoms.

**Crystallographic data of (4-dimethylamino-6-methoxy-1,3,5-triazin-1-io-2-yl)dinitromethanide (XII)**. Crystals were grown from a mixture MeOH–water, 1:1. $C_7H_{10}N_6O_5$, *M* 258.194, space group P21/*m*, crystals monoclinic: *a* 9.6510(10), *b* 6.6030(10), *c* 9.8770(10) Å , β 117.430(10) deg, *V* 558.70(10) $Å^3$, $d_{calc}$ 1.535 g/$cm^3$, *Z* 2. Final values of divergence factors *R* 0.047 , $R_W$ 0.084 for 983 independent reflections with $I > 3\sigma(I)$. Main bond distances, Å: $N^{6}$–$C^{5}$ 1.2976(6), $N^{6}$–$C^{1}$1.3598(6), $N^{2}$–$C^{1}$ 1.3556(6), $N^{2}$–$C^{3}$ 1.3187(6), $N^{4}$–$C^{5}$ 1.3551(6), $N^{4}$–$C^{3}$ 1.3630(6), $N^{14}$–$C^{10}$ 1.3550(6), $N^{14}$–$O^{16}$ 1.2562(5), $N^{14}$–$O^{15}$ 1.2453(5), $C^{10}$–$N^{11}$ 1.4497(6), $C^{10}$–$C^{3}$ 1.4074(6), $C^{5}$–$O^{17}$ 1.3093(5), $N^{7}$–$C^{1}$ 1.3309(5), $N^{7}$–$C^{9}$ 1.4544(7), $N^{7}$–$C^{8}$ 1.4673(6), $N^{11}$–$O^{13}$ 1.300(3), $N^{11}$–$O^{12}$ 1.120(3), $O^{17}$–$C^{18}$ 1.4509(6); bond angles, deg: $C^{5}N^{6}C^{1}$ 115.63(4), $C^{1}N^{2}C^{3}$ 116.33(4), $C^{5}N^{4}C^{3}$ 118.42(3), $C^{10}N^{14}O^{16}$ 119.35(4), $C^{10}N^{14}O^{15}$ 119.39(4), $O^{16}N^{14}O^{15}$ 121.26(4), $N^{14}C^{10}N^{11}$ 114.67(4), $N^{14}C^{10}C^{3}$ 127.64(4), $N^{11}C^{10}C^{3}$ 117.69(4), $N^{6}C^{5}N^{4}$ 123.32(4), $N^{6}C^{5}O^{17}$ 123.71(4), $N^{4}C^{5}O^{17}$ 112.97(4), $C^{1}N^{7}C^{9}$ 122.22(4), $C^{1}N^{7}C^{8}$ 12218(4), $C^{9}N^{7}C^{8}$ 115.60(4), $N^{6}C^{1}N^{2}$ 124.87(4), $N^{6}C^{1}N^{7}$ 117.43(4), $N^{2}C^{1}N^{7}$ 117.71(4), $C^{10}N^{11}O^{13}$ 111.41(12), $C^{10}N^{11}O^{12}$ 126.2(2), $O^{13}N^{11}O^{12}$ 122.33(10), $C^{5}O^{17}C^{18}$ 116.41(3), $N^{2}C^{3}N^{4}$ 121.4(4), $N^{2}C^{3}C^{10}$ 119.11(4), $N^{4}C^{3}C^{10}$ 119.45(4).

**Crystallographic data of (4-methoxy-6-ethylamino-1,3,5-triazin-1-io-2-yl)dinitromethanide (XIII)**. Crystals were grown from a mixture MeOH–water, 1:1. $C_7H_{10}N_6O_5$, *M* 258.194, space group P21/*n*, crystals monoclinic: *a* 7.306(1), *b* 13.267(1), *c* 11.780(1) Å, β 104.36(10) deg, *V* 1106.0(3) $Å^3$, $d_{calc}$ 1.562 g/$cm^3$, *Z* 4. Final values of divergence factors *R* 0.043, $R_W$ 0.055 for 1843 independent reflections with $I > 3\sigma(I)$. Main bond distances, Å: $C^{1}$–$N^{1}$ 1.3345(5), $N^{1}$–$C^{3}$ 1.3194(5), $C^{3}$–$N^{3}$ 1.3432(5), $N^{3}$–$C^{2}$ 1.3209(5), $C^{2}$–$N^{2}$ 1.3567(5), $N^{2}$–$C^{1}$ 1.3699(5), $C^{3}$–$O^{5}$ 1.3187(5), $O^{5}$–$C^{5}$ 1.4495(6), $C^{1}$–$N^{7}$ 1.3124(5), $N^{7}$–$C^{7}$ 1.4746(5), $C^{7}$–$C^{8}$ 1.5060(7), $C^{2}$–$C^{4}$ 1.4167(5), $C^{4}$–$N^{5}$ 1.4608(5), $C^{4}$–$N^{4}$ 1.3489(5), $N^{4}$–$O^{1}$ 1.2666(4), $N^{4}$–$O^{2}$ 1.2504(4), $N^{5}$–$O^{3}$ 1.2007(5), $N^{5}$–$O^{4}$ 1.1899(5); bond angles, deg: $C^{3}O^{5}C^{5}$ 117.7(1), $C^{3}N^{1}C^{1}$ 115.1(1), $C^{2}N^{2}C^{1}$ 119.4(1), $C^{1}N^{7}C^{7}$ 122.7(1), $O^{3}N^{5}C^{4}$ 117.2(1), $O^{3}N^{5}O^{4}$ 123.3(1), $C^{4}N^{5}O^{4}$ 119.5(1), $N^{2}C^{2}N^{3}$ 121.9(1), $N^{2}C^{2}C^{4}$ 120.2(1), $N^{3}C^{2}C^{4}$ 117.9(1), $C^{2}N^{3}C^{3}$ 114.4(1), $N^{5}C^{4}C^{2}$ 117.3(1), $N^{5}C^{4}N^{4}$ 115.0(1), $C^{2}C^{4}N^{4}$ 127.7(1), $O^{5}C^{3}N^{1}$ 118.6(1), $O^{5}C^{3}N^{3}$ 112.8(1), $N^{1}C^{3}N^{3}$ 128.6(1), $N^{1}C^{1}N^{2}$ 120.7(1), $N^{1}C^{1}N^{7}$ 120.8(1), $N^{2}C^{1}N^{7}$ 118.5(1), $O^{1}N^{4}O^{2}$ 120.3(1), $O^{1}N^{4}C^{4}$ 118.4(1), $O^{2}N^{4}C^{4}$ 121.3(1), $N^{7}C^{7}C^{8}$ 113.3(1).

**Zwitterionic salts of 2-methoxy-4-amino-6-dinitromethyl-1,3,5-triazines X–XVIII.** To a dispersion of 0.01 mol of potassium salt **I–IX** in 40 ml of water was added dropwise while stirring at 20–25°C 0.98 ml

(0.011 mol) of concn. HCl. The reaction mixture was stirred for 1 h, the crystalline precipitate was filtered off, washed with water (2×10 ml), and dried in air.

**(6-Amino-4-methoxy-1,3,5-triazin-1-io-2-yl)dinitromethanide (X).** Yield 92%, mp 150–151°C (decomp.). IR spectrum, ν, $cm^{-1}$: 3416, 3328, 3288, 3232, 3128, 1668, 1620, 1580, 1536, 1438, 1352, 1304, 1252, 1144, 1080, 1016, 880, 800, 784, 768, 744, 608, 584, 552. $^1H$ NMR spectrum, δ, ppm: 3.87 s (3H, $OCH_3$), 7.41 br.s (2H, $NH_2$), 7.82 s (1H, NH). Found, %: C 26.12; H 2.58; N 36.46. $C_5H_6N_6O_5$. Calculated, %: C 26.09; H 2.63; N 36.52.

**(6-Methylamino-4-methoxy-1,3,5-triazin-1-io-2-yl)dinitromethanide (XI).** Yield 90%, mp 149–150°C (decomp.). IR spectrum, ν, $cm^{-1}$: 3356, 3304, 3168, 1648, 1608, 1580, 1536, 1416, 1376, 1296, 1264, 1144, 1080, 1016, 872, 776. $^1H$ NMR spectrum, δ, ppm: 2.76–2.92 m (3H, $NCH_3$), 3.90 s (3H, $OCH_3$), 8.52 br.s (1H, NH), 9.08 m (1H, NH). Found, %: C 29.45; H 3.36; N 34.47. $C_6H_8N_6O_5$. Calculated, %: C 29.51; H 3.30; N 34.42.

**(4-Dimethylamino-6-methoxy-1,3,5-triazin-1-io-2-yl)dinitromethanide (XII).** Yield 89%, mp 110–112°C (decomp.). IR spectrum, ν, $cm^{-1}$: 3176, 2942, 2888, 1620, 1564, 1536, 1500, 1476, 1380, 1316, 1244, 1200, 1136, 1048, 1004, 968, 904, 856, 780, 728, 712. $^1H$ NMR spectrum, δ, ppm: 3.12 and 3.19 s (6H, $NCH_3$, $J$ 4.2 Hz), 3.92 s (3H, $OCH_3$), 7.98 s (1H, NH). Found, %: C 32.51; H 3.94; N 32.62. $C_7H_{10}N_6O_5$. Calculated, %: C 32.56; H 3.90; N 32.55.

**(4-Methoxy-6-ethylamino-1,3,5-triazin-1-io-2-yl)dinitromethanide (XIII).** Yield 94%, mp 129–130°C (decomp.). IR spectrum, ν, $cm^{-1}$: 3309, 3157, 3006, 2971, 2954, 2933, 2875, 1646, 1577, 1523, 1485, 1452, 1405, 1386, 1371, 1340, 1309, 1265, 1247, 1143, 1101, 1074, 1031, 997, 865, 792, 775. $^1H$ NMR spectrum, δ, ppm: 1.19 t (3H, $CH_3$, $J$ 6.8 Hz), 3.39 q (2H, $NCH_2$, $J$ 6.8 Hz), 3.92 c (3H, $OCH_3$), 7.63 m (1H, NH), 7.87 br.s (1H, NH). Found, %: C 32.57; H 3.85; N 32.49. $C_7H_{10}N_6O_5$. Calculated, %: C 32.56; H 3.90; N 32.55.

**(4-Methoxy-6-propylamino-1,3,5-triazin-1-io-2-yl)dinitromethanide (XIV).** Yield 96%, mp 130–132°C (decomp.). IR spectrum, ν, $cm^{-1}$: 3308, 3156, 2968, 2936, 2892, 1656, 1598, 1572, 1552, 1530, 1452, 1420, 1384, 1312, 1268, 1144, 1082, 1056, 1034, 1008, 968, 872, 800, 778. $^1H$ NMR spectrum, δ, ppm: 0.89 t (3H, $CH_3$, $J$ 7.0 Hz), 1.57 m (2H, $CH_2$), 3.34 m (2H, $NCH_2$), 3.92 s (3H, $OCH_3$), 7.65 m (1H, NH), 7.85 br.s (1H, NH). Found, %: C 35.32; H 4.39; N 30.95. $C_8H_{12}N_6O_5$. Calculated, %: C 35.30; H 4.44; N 30.87.

**(6-Methoxy-4-morpholino-1,3,5-triazine-1-io-2-yl)dinitromethanide (XV).** Yield 90%, mp 135–136°C (decomp.). IR spectrum, ν, $cm^{-1}$: 3212, 2956, 2928, 2890, 1634, 1602, 1566, 1546, 1536, 1500, 1448, 1438, 1392, 1312, 1280, 1268, 1246, 1184, 1156, 1116, 1076, 1038, 1004, 958, 888, 856, 828, 772, 746. $^1H$ NMR spectrum, δ, ppm: 3.69–3.80 m (8H, $NCH_2CH_2O$), 3.92 c (3H, $OCH_3$), 7.95 s (1H, NH). Found, %: C 36.07; H 4.10; N 27.90. $C_9H_{12}N_6O_6$. Calculated, %: C 36.00; H 4.03; N 27.99.

**(4-Diethylamino-6-methoxy-1,3,5-triazin-1-io-2-yl)dinitromethanide (XVI).** Yield 88%, mp 80–82°C (decomp.). IR spectrum, ν, $cm^{-1}$: 3202, 2966, 2944, 2928, 2894, 1596, 1568, 1520, 1480, 1448, 1384, 1328, 1272, 1240, 1216, 1192, 1128, 1096, 976, 920, 816, 776. $^1H$ NMR spectrum, δ, ppm: 1.10 t and 1.21 t (6H, $CH_3$, $J$ 7.2 Hz), 3.52 q and 3.65 q (4H, $NCH_2$, $J$ 7.2 Hz), 3.92 s (3H, $OCH_3$), 7.95 s (1H, NH). Found, %: C 37.81; H 4.89; N 29.45. $C_9H_{14}N_6O_5$. Calculated, %: C 37.76; H 4.93; N 29.36.

**(6-Methoxy-4-piperidino-1,3,5-triazin-1-io-2-yl)dinitromethanide (XVII).** Yield 92%, mp 115–116°C (decomp.). IR spectrum, ν, $cm^{-1}$: 3174, 3014, 2946, 2927, 2858, 1629, 1592, 1564, 1535, 1488, 1421, 1382, 1311, 1272, 1251, 1201, 1162, 1132, 1047, 1024, 993, 854, 821, 777, 736. $^1H$ NMR spectrum, δ, ppm: 1.62–1.80 m (6H, $CH_2CH_2CH_2$), 3.79 t and 3.88 t (4H, $NCH_2$, $J$ 4.4 Hz), 3.98 s (3H, $OCH_3$), 7.96 s (1H, NH). Found, %: C 40.36; H 4.78; N 28.28. $C_{10}H_{14}N_6O_5$. Calculated, %: C 40.27; H 4.73; N 28.18.

**[6-Methoxy-4-(pyrrolidin-1-yl)-1,3,5-triazin-1-io-2-yl]dinitromethanide (XVIII).** Yield 95%, mp 111–112$^{O}$C (decomp.). IR spectrum, ν, $cm^{-1}$: 3103, 2987, 2958, 2883, 1635, 1608, 1567, 1540, 1483, 1432, 1392, 1340, 1301, 1257, 1232, 1211, 1182, 1141, 1033, 1002, 966, 894, 856, 833, 777, 752, 729. $^1H$ NMR spectrum, δ, ppm: 1.96–2.04 m (4H, $CH_2CH_2$), 3.54 t 3.66 t (4H, $NCH_2$, $J$ 4.6 Hz), 3.94 s (3H, $OCH_3$), 7.95 s (1H, NH). Found, %: C 38.00; H 4.35; N 29.54. $C_9H_{12}N_6O_5$. Calculated, %: C 38.03; H 4.26; N 29.57.

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