

Annulated benzotetrazine 1,3-dioxides

2.* [1,2,5]Oxadiazolo[3,4-*f*][1,2,3,4]benzotetrazine 1,3,7-trioxide

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Thermolysis of 8-azido-7-nitrobenzotetrazine 1,3-dioxide led to benzotetrazine 1,3-dioxide annulated with the furoxan ring at the C(7)–C(8) bond. Complete assignment of the signals in the ^{13}C NMR spectrum of the compound obtained was performed. Attempted syntheses of benzotetrazine 1,3-dioxides annulated with a furoxan ring at the C(6)–C(7) bond or two furoxan rings at the C(5)–C(6) and C(7)–C(8) bonds were unsuccessful.

Key words: benzo-1,2,3,4-tetrazines, *N*-oxides, 1,2,5-oxadiazoles, ^{13}C NMR spectroscopy.

Benzo-1,2,3,4-tetrazine 1,3-dioxides (BTDO) with electron-withdrawing substituents are donors of nitrogen oxide.^{2–4} The high activity could be expected from BTDO annulated with electron-withdrawing heterocycles. Recently,¹ we have synthesized BTDO annulated with the furoxan ring at the C(5)–C(6) bond. The goal of the present work was to obtain benzotetrazine 1,3-dioxides annulated with one (at the C(6)–C(7) or C(7)–C(8) bond) or two furoxan rings.

Results and Discussion

For annulation of benzotetrazine 1,3-dioxides with the furoxan ring, we used a conventional approach, namely, an intramolecular reaction between azido and nitro groups that are *ortho* to each other.⁵

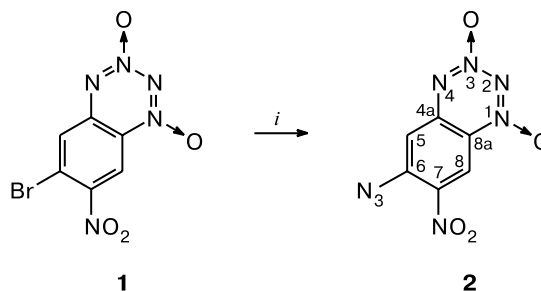
Synthesis of the starting compounds. *o*-Azido-nitro-BTDO were prepared by electrophilic and/or nucleophilic substitution reactions. The mechanisms of these reactions for the BTDO series have been described earlier.^{6,7}

Replacement of the Br atom in BTDO **1** by an azide ion in acetone gave BTDO **2** in 50% yield (Scheme 1).

Treatment of 8-bromo-7-nitro-BTDO **3** with sodium azide in acetone–water as a solvent afforded 8-azido-7-nitro-BTDO **4** in high yield (Scheme 2). Attempted synthesis of BTDO **4** by nitration of 8-azido-BTDO **5** failed because of the instability of the latter in conc. H_2SO_4 . Apparently, this is due to protonation of the azido group followed by an intramolecular reaction of the resulting N-centered cation with the *N*-oxide fragment of the tetrazine 1,3-dioxide ring, which breaks down the tetrazine ring.

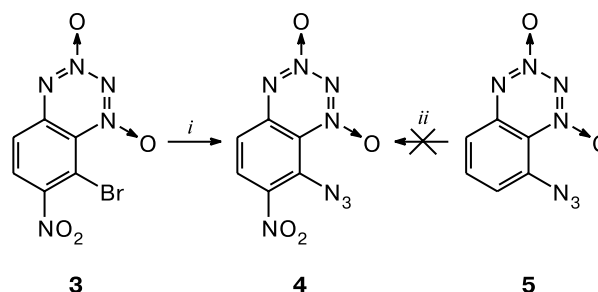
* For Part 1, see Ref. 1.

Scheme 1



Reagents, conditions, and yields: *i.* NaN_3 , acetone, 20 °C, 10 min (50%).

Scheme 2



Reagents, conditions, and yields: *i.* NaN_3 , acetone/ H_2O , 20 °C, 1.3 h (90%). *ii.* $\text{HNO}_3/\text{H}_2\text{SO}_4$, 20 °C.

The structures of BTDO **2** and **4** were unambiguously confirmed by ^1H , ^{13}C , and ^{14}N NMR spectra (Tables 1, 2).

Nucleophilic replacement of the Br atoms by the azide ion in 5,7-dibromo-BTDO **6**, as well as in 7-bromo-BTDO, occurred not so smoothly as with BTDO **3**: the

Table 1. Experimental (δ) and calculated ($[\delta]$) ^{13}C NMR spectra of compounds **2**, **4**, **7**, **9**, **11**, and **12** in acetone- d_6 ^a

Compound	δ (J/Hz)					
	C(4a)	C(5)	C(6)	C(7)	C(8)	C(8a) (br.s)
2 ^b	146.5 [153.1]	118.4 ^c [117.3]	144.5 [146.4]	142.9 (br.s) [143.3]	115.8 ^c [116.8]	125.3 [125.9]
4	148.0 (³ <i>J</i> = 10.8)	122.8	133.3	— ^d	129.2 (³ <i>J</i> = 7.1)	— ^d
7 ^e	140.9 (³ <i>J</i> _{C(4a),H(6)} = 7.8, ³ <i>J</i> _{C(4a),H(8)} = 5.0)	120.0 (² <i>J</i> = 5.0, ⁴ <i>J</i> = 1.5)	134.6 (¹ <i>J</i> = 171.1, ³ <i>J</i> = 5.9)	144.9 (² <i>J</i> _{C(7),H(6)} = ² <i>J</i> _{C(7),H(8)} = 2.9)	107.8 (¹ <i>J</i> = 174.4, ³ <i>J</i> = 5.1)	130.6 (² <i>J</i> = 3.8, ⁴ <i>J</i> = 0.9)
9	[145.5] 141.2 (³ <i>J</i> = 8.5)	[121.0] 121.2 (² <i>J</i> = 5.8)	[134.9] 135.0 (¹ <i>J</i> = 174.6)	[146.9] 137.3 (² <i>J</i> = 1.2)	[106.7] 129.4 (br.s)	[131.5] 122.2 (⁴ <i>J</i> = 1.5)
11 ^f	140.3 [144.8]	144.2 (br.s) [144.6]	116.6 [123.8]	152.8 (br.s) [153.1]	111.3 [115.8]	129.1 [131.6]
12 ^g	141.1 [147.7]	131.1 (br.s) [128.7]	134.8 [140.3]	134.4 (br.s) [127.8]	132.0 [130.1]	122.1 [118.2]

^a Signal assignment was accomplished with selective suppression of ^1H — ^{13}C couplings (compound **2**), selective cross polarization from protons (**2** and **7**), selective suppression of ^{13}C — ^{14}N couplings (**5**, **7**, and **9**), signal accumulation without proton decoupling (**5**, **7**, and **9**), and additive calculation (**11** and **12**); the broadening of the signal for the C atom bound to the nitro group or to the N(1) atom was also taken into account.

^b Calculated from the δ values for benzotetrazine 1,3-dioxide⁶ with correction for the substituent effects.

^c The assignments of these signals may be interchanged.

^d Under these conditions, no accumulation is observed because of considerable broadening.

^e Calculated from the δ values for 7-azidobenzotetrazine 1,3-dioxide⁷ with correction for the substituent effects.

^f Calculated from the δ values for 5,7-dinitrobenzotetrazine 1,3-dioxide⁶ with correction for the substituent effects.

^g Calculated from the δ values for 6-azido-5-nitrobenzotetrazine 1,3-dioxide¹ with correction for the substituent effects.

Table 2. ^1H and ^{14}N NMR spectra of compounds **2**, **4**, **7**—**9**, **11**, **12**, and **14** in acetone- d_6

Compound	δ_{H} (J/Hz)	$\delta_{^{14}\text{N}}$ ($\Delta\nu_{1/2}$ /Hz)		
		N(1), N(3)	NO_2	N_3
2	8.04 (s, 1 H, H(5)); 8.89 (s, 1 H, H(8))	−41 (50), −45 (70)	−19 (60)	−147 (60)
4	7.93 (d, 1 H, H(5), <i>J</i> = 9.1); 8.54 (d, 1 H, H(6), <i>J</i> = 9.1)	−39 (20), −45 (70)	−15 (70)	−147 (40)
7	7.94, 8.14 (both d, 1 H each, <i>J</i> = 2.3)	−41 (35), −48 (70)	—	−141 (70)
8	8.00, 8.25 (both d, 1 H each, <i>J</i> = 1.9)	−41 (60), −49 (80)	—	−142 (80)
9	8.75 (s, 1 H, H(6))	−48 (40)*	−24 (35)	−147 (70)
11	—	−42 (40), −47 (110)	−19 (110), −25 (100)	—
12	—	−42 (35), −47 (70)	−26 (80), −28 (80)	−152 (100), −155 (100)
14	7.61 (d, 1 H, H(5), <i>J</i> = 9.7); 8.18 (d, 1 H, H(6), <i>J</i> = 9.7)	−37 (60), −46 (60)	—	—

* The integral intensity of the signal corresponds to two N atoms.

total yield of products **7** and **8** was only 41% (Scheme 3). Bromine displacement from positions 5 and 7 by the azide

ion (**7** : **8** = 10 : 4) was somewhat less selective than that by the methoxide anion (the ratio of 7-bromo-5-methoxy- and 5-bromo-7-methoxybenzotetrazine 1,3-dioxides was 9 : 1).⁷ However, the substitution in position 7 was dominant in both cases.

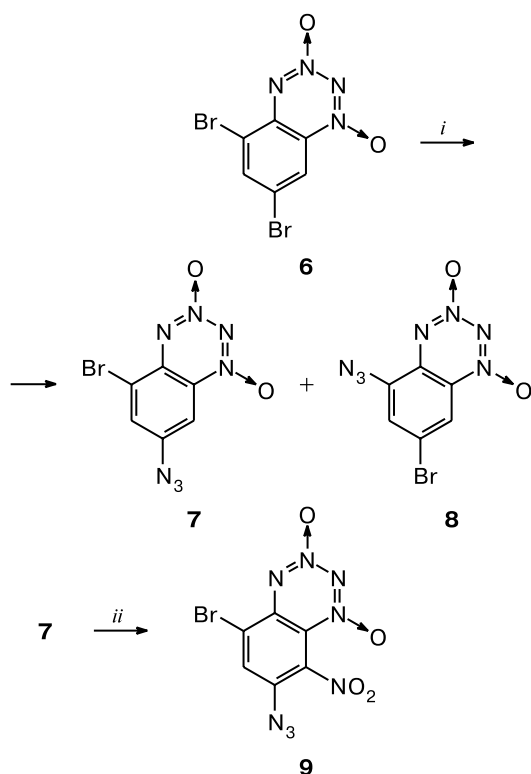
Structures **7** or **8** were assigned to the isomers obtained because of a good agreement between experimental (unambiguously assigned with the use of appropriate procedures) and additively calculated chemical shifts (see Table 1 and Experimental).

Nitration of compound **7** with HNO_3 —oleum gave BTDO **9** in 86% yield. This is a very high yield, because azidobenzenes often decompose in mixtures of sulfuric and nitric acids.⁸

Nitration of BTDO **10** with HNO_3 —oleum afforded BTDO **11** in good yield (Scheme 4). Slow addition of a solution of NaN_3 in water—acetone to a solution of BTDO **11** in acetone gave 6,8-diazido-5,7-dinitro-BTDO **12**.

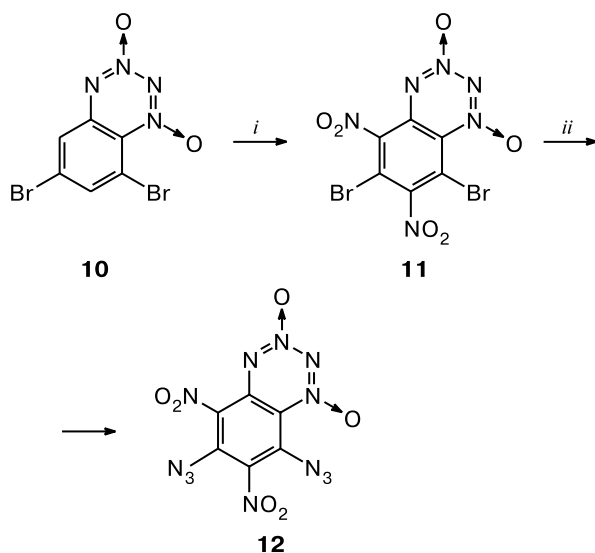
The structure of BTDO **11** was unambiguously confirmed by IR and ^{13}C and ^{14}N NMR spectroscopy (see Tables 1, 2). We failed to carry out elemental analysis of BTDO **12** since this compound exploded on heating. However, its structure was convincingly proved by ^{13}C and ^{14}N NMR spectroscopy. The ^{13}C NMR spectrum shows six signals, three of them being broadened. This indicates that the respective C atoms are bound to the

Scheme 3



Reagents, conditions, and yields: *i.* NaN₃, DMF, 20 °C, 2 h (29% (7) and 12% (8)). *ii.* HNO₃/oleum, 20 °C, 30 min (86%).

Scheme 4

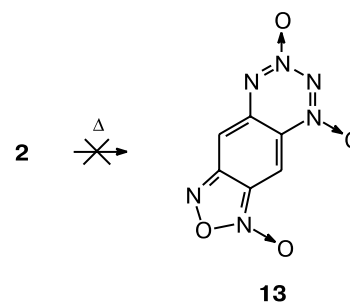


Reagents, conditions, and yields: *i.* HNO₃/oleum, 1 h at 20 °C, 1 h at 90 °C (58%). *ii.* Addition of NaN₃ for 1 h, acetone, 20 °C (69%).

nitro N atoms and the N(1) atom of the tetrazine 1,3-dioxide ring (see Ref. 4). Signal assignments in the ¹³C NMR spectra were performed according to the additive scheme (see Table 1). The ¹⁴N NMR spectrum contains two sharp signals for each of the two nitro groups and for each of the two azido groups and two signals for the N(1) and N(3) atoms of the tetrazine 1,3-dioxide ring in their typical ranges (see Table 2).

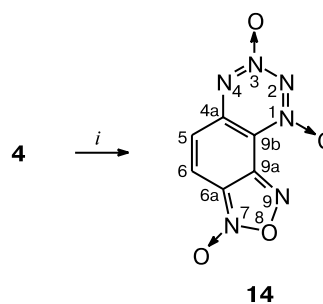
Synthesis of furoxan-annulated BTDO. Attempted synthesis of linear annulated compound **13*** failed (Scheme 5). Heating of 6-azido-7-nitro-BTDO **2** under various conditions (in AcOH, in toluene, or without any solvent) caused only tarring of the reaction mixture.

Scheme 5



Cyclization of BTDO **4** in AcOH at 118 °C gave angular annulated compound **14** (Scheme 6). These conditions are somewhat more drastic than those required for cyclization of 6-azido-5-nitrotetrazine 1,3-dioxide (100 °C, AcOH).¹

Scheme 6



Reagents, conditions, and yields: *i.* AcOH, 118 °C, 30 min (30%).

Compound **14** is thermally not so stable as BTDO annulated with the furoxan ring at the C(5)—C(6) bond.¹ The latter melts without decomposition at 204 °C,¹ while compound **14** melts at 74–76 °C, decomposing

* The IUPAC name is [1,2,5]oxadiazolo[3,4-*g*][1,2,3,4]benzotetrazine 1,3,8-trioxide (**13**).

above 90 °C. Apparently, this is responsible for its comparatively low yield.

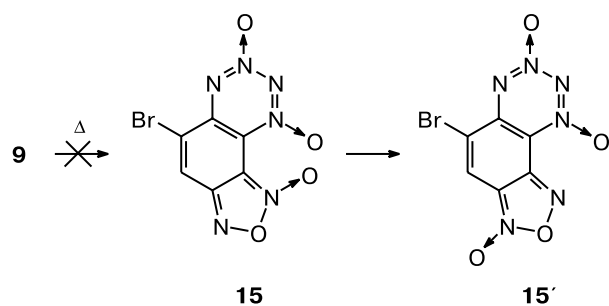
According to its ^1H (see Table 2) and ^{13}C NMR spectra (the latter was recorded in acetone- d_6), compound **14** exists as one isomer. Signal assignments were performed with selective ^1H — ^{13}C decoupling and selective cross polarization from protons; the broadening of the signal for the C atom bound to the N(1) atom was also taken into account. An analysis of the ^{13}C chemical shifts provides unambiguous evidence that this isomer has the outward *N*-oxide oxygen atom in the furoxan ring. Note that the experimental (δ_{exp}) and calculated chemical shifts (δ_{calc}) obtained with consideration of those for BTDO⁹ and benzofuroxan⁵ agree well.

δ	C(4a)	C(5)	C(6)	C(6a)	C(9a)	C(9b)
δ_{exp}	154.6	125.4	126.3	113.8	143.9	119.2
(J/Hz)	$(^3J = 9.6)$			$(^3J = 11.4)$		$(^3J = 4.5)$ (br.s)
δ_{calc}	148.4	124.6	122.7	117.4	143.5	117.7

In the ^{14}N NMR spectra of compound **14** (see Table 2), the signal for the N(1) atom is slightly shifted downfield compared to its usual position for BTDO, while the signal for the N(3) atom remains immobile. It should be emphasized that the signals for the N(1) and N(2) atoms have equal half-widths, as in 5,6-furoxan-annulated BTDO,¹ although the signal for the N(3) atom in BTDO is usually somewhat broader than the signal for the N(1) atom.

We attempted thermal cyclization of compound **9**, in which the mutual arrangement of the azido and nitro groups is inverted with respect to that in compound **4**: the nitro group occupies position 8, while the azido group is in position 7. However, neither compound **15** nor its thermodynamically more stable isomer **15'** was obtained: only tarring of the reaction mixture was observed (Scheme 7).

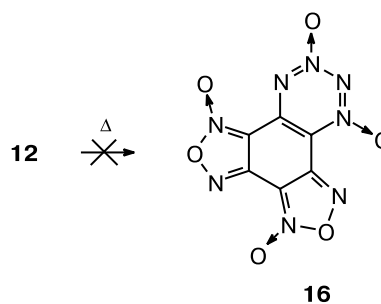
Scheme 7



Attempted synthesis of BTDO annulated with two furoxan rings (compound **16**)* also failed. Heating of BTDO **12** under various conditions always resulted in tarring (Scheme 8).

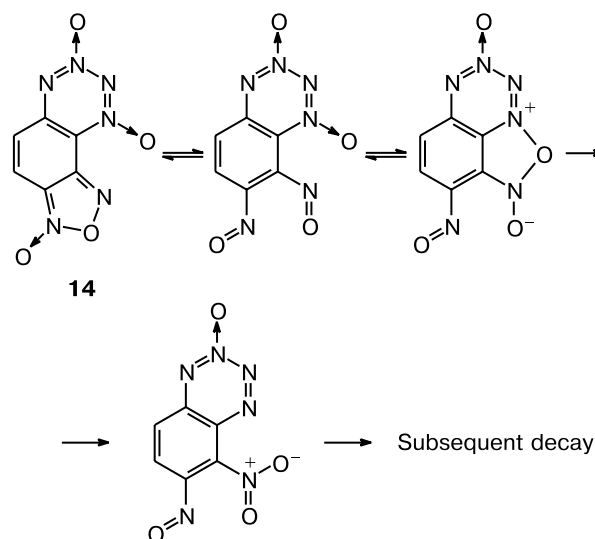
* The IUPAC name is bis[1,2,5]oxadiazolo[3,4-*f*:3',4'-*h*][1,2,3,4]benzotetrazine 1,4,7,9-trioxide (**16**).

Scheme 8



One can assume that compound **16** is unstable under conditions of thermal ring closure. A possible reason for its instability, as well as for the instability of compound **14**, is an intramolecular reaction of the *N*-oxide oxygen atom of the tetrazine 1,3-dioxide ring with the neighboring nitroso group, which breaks down the tetrazine ring (Scheme 9)*.

Scheme 9



Thus, we obtained for the first time benzotetrazine 1,3-dioxide annulated with the furoxan ring at the C(7)—C(8) bond. This compound is thermally unstable, decomposing above 90 °C.

Experimental

^1H , ^{13}C , and ^{14}N NMR spectra were recorded on a Bruker AM-300 spectrometer (300.13, 75.5, and 21.5 MHz, respectively). Chemical shifts were measured relative to Me_4Si (^1H and ^{13}C) or MeNO_2 (^{14}N , the external standard; high-field

* The instabilities of benzotetrazine 2-oxides, possible reaction intermediates in the decomposition of compounds **14** and **16**, have been discussed earlier.²

δ values are negative). IR spectra were recorded on a UR-20 instrument. Mass spectra were recorded on a Varian MAT-311A instrument (EI, 70 eV). The course of the reactions was monitored by TLC (Silufol UV-254).

All benzotetrazine 1,3-dioxides obtained here are colored yellow.

6-Azido-7-nitrobenzo-1,2,3,4-tetrazine 1,3-dioxide (2). Sodium azide (70 mg, 1.1 mmol) was added to a stirred solution of 6-bromo-7-nitro-BTDO **1** (280 mg, 1.0 mmol) in acetone (15 mL). The resulting suspension was stirred at 20 °C for 10 min, the solvent was removed *in vacuo*, and the residue was purified by chromatography on silica gel with C_6H_6 —AcOEt (5 : 1) as an eluent. The yield of BTDO **2** was 125 mg (50%); this compound decomposed without melting at $T > 120$ °C. Found (%): C, 28.67; H, 0.80; N, 44.45. $C_6H_2N_8O_4$. Calculated (%): C, 28.81; H, 0.81; N, 44.80. IR (KBr), ν/cm^{-1} : 1378, 1540 (NO_2), 1429, 1504 (N_4O_2), 2135 (N_3).

8-Azido-7-nitrobenzo-1,2,3,4-tetrazine 1,3-dioxide (4). A solution of NaN_3 (10 mg, 0.16 mmol) in a mixture of water (0.6 mL) and acetone (0.6 mL) was added at 20 °C to a stirred solution of 8-bromo-7-nitro-BTDO **3** (30 mg, 0.104 mmol) in acetone (3 mL). After 1.3 h, the reaction mixture was poured into water (15 mL) and the product was extracted with CH_2Cl_2 (2×10 mL). The organic layer was dried with $MgSO_4$ and concentrated *in vacuo* to give virtually pure BTDO **4** (23 mg, 90%), m.p. 120–122 °C (decomp.). Found (%): C, 28.94; H, 0.82; N, 44.57. $C_6H_2N_8O_4$. Calculated (%): C, 28.81; H, 0.81; N, 44.80. IR (KBr), ν/cm^{-1} : 1340, 1540 (NO_2), 1428, 1519 (N_4O_2), 2157 (N_3).

7-Azido-5-bromo- (7) and 5-azido-7-bromobenzo-1,2,3,4-tetrazine 1,3-dioxides (8). Pulverized NaN_3 (390 mg, 6 mmol) was added to a stirred solution of 5,7-dibromo-BTDO **6** (960 mg, 3.0 mmol) in DMF (20 mL). The reaction mixture was stirred at 20 °C for 2 h and poured into water. The product was extracted with AcOEt, dried with $MgSO_4$, and concentrated *in vacuo*. The residue was separated by chromatography on silica gel with $CHCl_3$ as an eluent to give BTDO **7** (250 mg, 29%) and BTDO **8** (100 mg, 12%).

Compound 7, m.p. 159–161 °C. Found (%): C, 25.11; H, 0.70; Br, 28.35; N, 34.20. $C_6H_2BrN_7O_2$. Calculated (%): C, 25.37; H, 0.71; Br, 28.13; N, 34.52. MS, m/z : 283, 285 (1 : 1) $[M]^+$.

Compound 8, m.p. 139–144 °C. Found (%): C, 25.50; H, 0.72; Br, 28.31; N, 34.33. $C_6H_2BrN_7O_2$. Calculated (%): C, 25.37; H, 0.71; Br, 28.13; N, 34.52. ^{13}C NMR (acetone- d_6), δ : 117.8 (C(8), $^1J = 180.4$ Hz, $^3J = 5.5$ Hz) [116.7]; 124.7 (C(7), $^2J_{C(7),H(6)} = ^2J_{C(7),H(8)} = 4.5$ Hz) [126.1]; 130.8 (C(6), $^1J = 171.3$ Hz, $^3J = 6.1$ Hz) [132.2] (the chemical shifts calculated from the δ values for 7-bromobenzotetrazine 1,3-dioxide⁶ with correction for the substituent effects are given in brackets). MS, m/z : 283, 285 (1 : 1) $[M]^+$.

7-Azido-5-bromo-8-nitrobenzo-1,2,3,4-tetrazine 1,3-dioxide (9). Concentrated HNO_3 (2 mL, $d = 1.5$ g cm^{-3}) and 20% oleum (1 mL) in H_2SO_4 (5 mL) were added at 0 °C to a stirred solution of 7-azido-5-bromo-BTDO **7** (180 mg, 0.63 mmol) in conc. H_2SO_4 (10 mL, $d = 1.8$ g cm^{-3}). The reaction mixture was stirred at 20 °C for 30 min and poured onto finely crushed ice (50 g). The precipitate that formed was filtered off, washed with water, and dried *in vacuo* to give virtually pure BTDO **9** (100 mg, 48%), m.p. 154–158 °C (decomp.). Extraction with CH_2Cl_2 and column chromatography on silica gel with CH_2Cl_2 as an

eluent gave additional crop of BTDO **9** (80 mg). The total yield was 86%. Found (%): C, 22.05; H, 0.31; Br, 24.44; N, 33.79. $C_6HBrN_8O_4$. Calculated (%): C, 21.90; H, 0.31; Br, 24.28; N, 34.06.

6,8-Dibromo-5,7-dinitrobenzo-1,2,3,4-tetrazine 1,3-dioxide (11). Concentrated HNO_3 (4 mL, $d = 1.5$ g cm^{-3}) and 20% oleum (3 mL) were added at 0 °C to a stirred solution of 6,8-dibromo-BTDO **10** (1.0 g, 3.14 mmol) in conc. H_2SO_4 (30 mL, $d = 1.8$ g cm^{-3}). The resulting solution was stirred at 20 °C for 1 h and then at 90 °C for 1 h. On cooling to 20 °C, the reaction mixture was poured onto finely crushed ice (120 g). The precipitate that formed was filtered off, washed with cold water, and dried *in vacuo* over P_4O_{10} to give BTDO **11** (250 mg). The product from the filtrate was extracted with CH_2Cl_2 (4×100 mL), the organic layer was dried with $MgSO_4$, and the solvent was removed *in vacuo*. The additional crop of BTDO **11** was 500 mg. The total yield was 58%, m.p. >220 °C (decomp.). Found (%): C, 17.69; Br, 38.58; N, 20.21. $C_6Br_2N_6O_6$. Calculated (%): C, 17.50; Br, 38.80; N, 20.40. IR (KBr), ν/cm^{-1} : 1321, 1555 (NO_2), 1455, 1515 (N_4O_2).

6,8-Diazido-5,7-dinitrobenzo-1,2,3,4-tetrazine 1,3-dioxide (12). *Caution! This compound is sensitive to friction and should be handled with as carefully as with an explosive.* A solution of NaN_3 in a mixture of acetone (2 mL) and water (2 mL) was added dropwise at 20 °C for 1 h to a stirred solution of BTDO **11** (230 mg, 0.558 mmol) in acetone (5 mL). After the addition was completed, the solution was kept for an additional 5 min and concentrated *in vacuo*. Then water (20 mL) was added and the product was extracted with CH_2Cl_2 . The organic layer was dried with $MgSO_4$ and concentrated *in vacuo*. The residue was separated by column chromatography on silica gel with C_6H_6 as an eluent into the starting BTDO **11** (70 mg) and BTDO **12** (105 mg). Analytically pure BTDO **12** (90 mg, 69% with respect to the consumed BTDO **11**) was obtained by additional chromatography on silica gel with $CHCl_3$ as an eluent. Compound **12** decomposed without melting at $T > 65$ °C. Elemental analysis and MS (EI) data are missing since this compound explodes on heating. IR (KBr), ν/cm^{-1} : 1350, 1555 (NO_2), 1435, 1512 (N_4O_2), 2162 (N_3).

[1,2,5]Oxadiazolo[3,4-*f*][1,2,3,4]benzotetrazine 1,3,7-trioxide (14). A solution of BTDO **4** (260 mg, 1.04 mmol) in glacial AcOH (15 mL) was refluxed for 30 min. The reaction mixture was cooled and concentrated to dryness *in vacuo*. The residue was purified by column chromatography on silica gel with light petroleum ether—AcOEt (4 : 1) as an eluent. The yield of BTDO **14** was 70 mg (30%), m.p. 72–74 °C. This compound decomposed at $T > 90$ °C. Found (%): C, 32.66; H, 0.92; N, 37.60. $C_6H_2N_6O_4$. Calculated (%): C, 32.44; H, 0.91; N, 37.84. IR (KBr), ν/cm^{-1} : 1390 s, 1405 w, 1430 s, 1480 w, 1502 s. MS, m/z : 222 $[M]^+$.

This work was financially supported by the Russian Foundation for Basic Research (Project No. 04-03-32432).

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*Received August 19, 2005;
in revised form October 24, 2005*