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TRISUBSTITUTED 1,3,5-TRIAZINES. 5*. REACTION OF 2,4,6-TRIS[DI(*tert*-BUTOXYCARBONYL)NITROMETHYL]-1,3,5-TRIAZINE WITH NUCLEOPHILES

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The reaction of 2,4,6-tris[di(tert-butoxycarbonyl)nitromethyl]-1,3,5-triazine with ammonia, amines, and hydrazine has been studied. It was possible to substitute one of the di(tert-butoxycarbonyl)nitromethyl groups in this compound in the presence of ammonia, primary aliphatic amines, dimethylamine, and morpholine. The reaction with hydrazine leads to both mono- and disubstituted products. A double dealkoxylation occurs in the presence of diethylamine to give the bis(dimethylammonium) salt of 2,4-bis(tert-butoxycarbonylnitromethyl)-6-[di(tert-butoxycarbonyl)nitromethyl]-1,3,5-triazine.

Keywords: di(*tert*-butoxycarbonyl)nitromethyl group, 2,4,6-tris[di(*tert*-butoxycarbonyl)nitromethyl]-1,3,5-triazine, nucleophilic substitution, nucleophilic reagents.

We have previously reported the synthesis of 2,4,6-tris[di(*tert*-butoxycarbonyl)nitromethyl]-1,3,5-triazine (**1**) and its conversion to 2,4,6-tris(nitromethyl)-1,3,5-triazine which is the first example of a novel group of nitromethyl substituted triazines [2]. With the potential importance of the latter as physiologically active compounds, pesticides, and herbicides it was of interest to develop methods for their synthesis via compound **1**. The nucleophilic substitution of a di(*tert*-butoxycarbonyl)nitromethyl group has been proposed as the most convenient method for preparing precursors of substituted nitromethyl-1,3,5-triazines. It is known that an S_NAr addition-elimination mechanism has been proposed for such processes in halo- and alkoxy-1,3,5-triazine derivatives with the formation of a Meisenheimer type tetrahedral intermediate at the reaction center [3, 4]. Whereas rather a large amount of both synthetic and theoretical work [3, 4] has been dedicated to the nucleophilic substitution of halide, ammonium, and alkoxy groups in 1,3,5-triazines the evidence for a similar process in 1,3,5-triazine CH-acids is restricted to data for trichloromethyl [5, 6], perfluoroalkyl [7], and trinitromethyl groups [8, 9]. The formation of a malonate through substitution of the dicarbethoxymethylene fragment of 2,4,6-tris(dicarbethoxymethylene)-1,3,5-hexahydrotriazine in the presence of various nucleophilic reagents is accompanied by the elimination of the two carbethoxy groups on the one carbon atom to form the corresponding methyl 1,3,5-triazine derivatives [10].

* For Communication 4 see [1].

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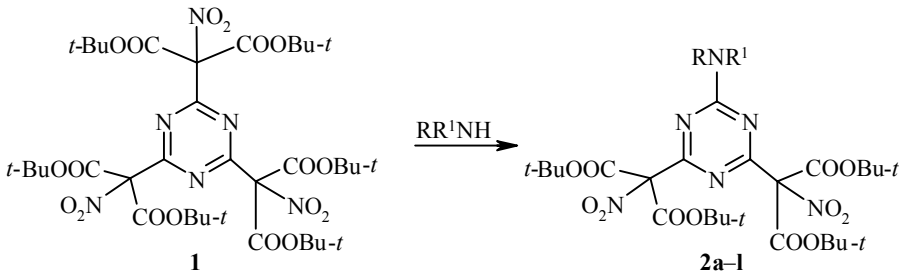
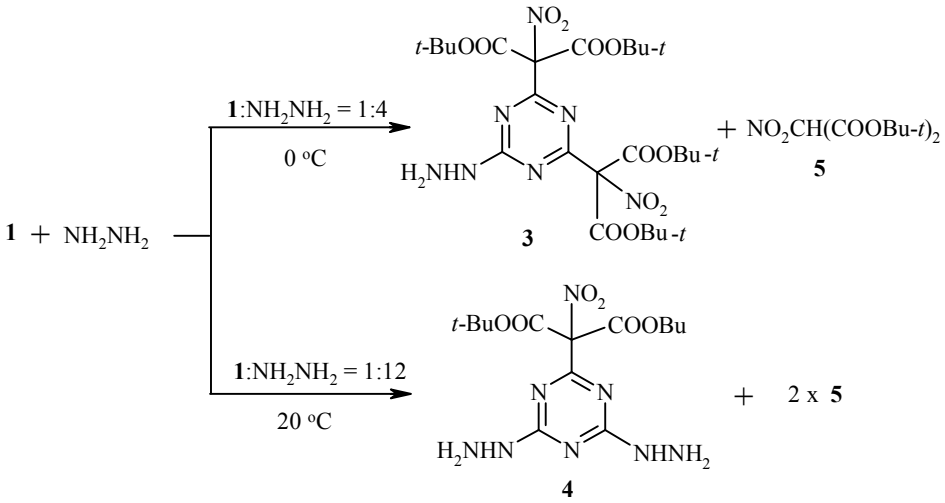
Bearing in mind that in compound **1** there are at least two more reaction centers (the carbalkoxy and nitro groups) beside the triazine ring and the rather large bulk of the substituted fragment, a mixture of nucleophilic substitution products for the di(*tert*-butoxycarbonyl)nitromethyl group might have been expected. This would be analogous to the substitution or reduction of the nitro group [11] and the transformation of a 1,3,5-triazine ring to a 1,2,4-triazine or 1,2,4-triazole derivative as reported for 1,3,5-triazines containing ester groups in reaction with certain substituted hydrazines [12-14].

The results obtained by us show that the reaction of compound **1** with ammonia, primary aliphatic amines, dimethylamine, and morpholine leads to the substitution of only one di(*tert*-butoxycarbonyl)nitromethyl group to form the corresponding substituted aminotriazines **2a-l**.



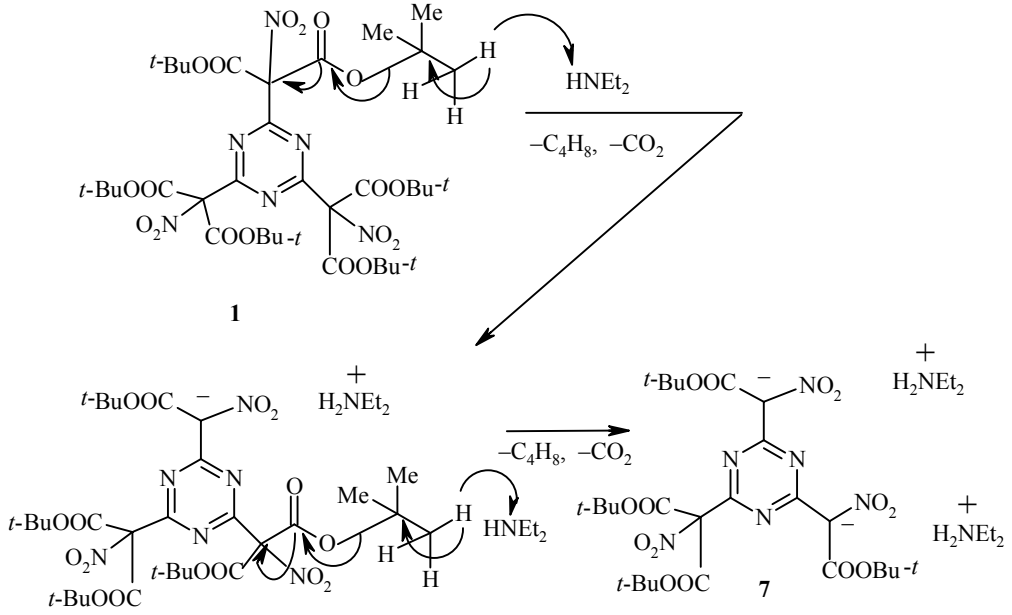
2 a–j R = H, R^1 = H; **k** R = Me; **a, b** R^1 = Me; **c** R^1 = Et; **d** R^1 = Pr; **e** R^1 = Pr-*i*; **f** R^1 = All; **g** R^1 = Bu-*t*; **h** R^1 = CH_2CH_2OH; **i** R^1 = $CH_2CH_2NH_2$; **j** R^1 = Bn; R^1 = Me; **l** R + R^1 = $(CH_2)_2O(CH_2)_2$

When the starting triazine **1** is treated with hydrazine hydrate there occur both mono- and disubstitution to give the corresponding mono- and dihydrazines **3** and **4**, depending of the amount of hydrazine and the reaction temperature.



The reaction of a series of carboxy substituted triazines with hydrazine derivatives has been reported before [13, 14]. The hydrazine initially reacts with the triazine and this is followed by opening of the triazine ring and then recyclization to either 1,2,4-triazine derivatives or to substituted 1,2,4-triazoles. In our case the carboxyl groups are not affected and novel heterocycles are not formed. The di(*tert*-butoxycarbonyl)nitromethyl group behaves as a pseudohalogen and leaves in the presence of nucleophiles as di(*tert*-butoxycarbonyl)-nitromethane (**5**). The structure of the latter was confirmed by separation and a full characterization of its methylammonium salt **6**.

The reaction of diethylamine with the triazine **1** gave an almost quantitative yield of the 2,4-bis(diethylammonium) salt of 2,4-bis(*tert*-butoxycarbonylnitromethyl)-6-[di(*tert*-butoxycarbonyl)nitromethyl]-1,3,5-triazine (**7**).



In the case of the reaction with diethylamine (as the most powerful and the bulkiest amine) we propose that, in place of a nucleophilic substitution, there occurs a successive or simultaneous deprotonation of the two *tert*-butyl groups in the triazine **1** with the subsequent loss of molecules of isobutylene and carbon dioxide to give the salt **7** which is stable under these reaction conditions. Although it is known that *tert*-butoxycarbonyl groups are usually stable under basic hydrolysis conditions [15], the presence of two acceptor groups (nitro and triazinyl), the steric inaccessibility of the reaction centers of the triazine, and the efficient formation of nitrotriazinylacetic acid anions stabilized by conjugation makes the deprotonation and subsequent elimination the dominant reaction route. Decarboxylation using amines has been reported in the literature for the case of hexahydrotriazine derivatives but it occurs under much more rigid conditions and produces methyl substituted triazines [10].

The structure of the compounds obtained was proved by a combination of physico-chemical methods and elemental analytical data. Thanks to hindered rotation about the C–N bond the ^{13}C NMR spectra of the monosubstituted aminotriazines show an increased number of signals with a $\Delta\delta$ ^{13}C = 0.01-0.80 ppm (an increased number of signals in the ^{13}C, ^{14}N, and ^{19}F spectra of substituted amino-1,3,5-triazines associated with the presence of a barrier to internal rotation has been reported in the literature [8, 15]). No clear dependence on the nature of the substituent at the exocyclic nitrogen atom was observed (see Table 2). The effect of the bulk of the substituent was clearly shown in the case of compounds **2g** (R^1 = Bu-*t*), **2i** (R^1 = $(CH_2)_2NH_2$), and **2j** (R^1 = Bn) for which a $\Delta\delta$ ^{13}C of 0.02-0.03 ppm for the *tert*-butyl groups was observed in contrast to the remaining compounds with less bulky substituents for which $\Delta\delta$ ^{13}C = 0. The increase in doubling of the number of signals in the ^{13}C NMR spectrum of compound **7** is due to the difference in the chemical shifts of the groups bonded to the negatively charged molecular fragments and the groups not so bonded. In this case hindered rotation does not play a part since the molecule **7** has an axis of symmetry, as in the example of compounds **2k,l** and **4** which have either a single substituent at the exocyclic nitrogen atom (**2k,l**) or two equivalent substituents for which hindered rotation is not possible (**4**). It was interesting to find that compound **4**, which is symmetrical in its ^{13}C NMR spectrum, has a complex ^{1}H NMR spectrum in which there are present two signals for the two nonequivalent NH_2 groups and four signals for the two NH groups (see Table 1).

TABLE 1. Characteristics of Compounds **2a-l**, **3**, **4**, **6**, **7**

Compound	Empirical formula	Found, % / Calculated, %			mp, °C	IR spectrum, ν, cm^{-1}	^1H NMR spectrum* δ, ppm (*J*, Hz)*[2]	Reaction time, h	Yield, %
		C	H	N					
1	2	3	4	5	6	7	8	9	10
2a	$C_{25}H_{38}N_6O_{12}$	49.00 / 48.85	6.29 / 6.23	14.00 / 13.76	172-173	3450, 3350, 3230, 2970, 1830, 1710, 1630, 1570, 1530, 1400, 1370, 1335, 1285, 1255, 1150, 1070, 1030, 980, 840, 830	1.45 (36H, s, $12CH_3$); 8.45 (2H, s, NH_2)	2.00	93.3
2b	$C_{26}H_{40}N_6O_{12}$	49.81 / 49.52	6.39 / 6.39	13.27 / 13.32	145-146	3400, 2990, 2930, 1765, 1615, 1580, 1535, 1435, 1400, 1370, 1330, 1285, 1255, 1150, 1100, 985, 835, 810	1.49 (18H, s, $6CH_3$); 1.51 (18H, s, $6CH_3$); 3.00 (3H, d , ^3J = 5.0, NCH_3); 5.94 (1H, br. d, NH); [8.94, br. s]	0.25	93.3
2c	$C_{27}H_{42}N_6O_{12}$	50.44 / 50.30	6.50 / 6.57	13.10 / 13.03	118-119	3370, 2980, 2930, 1755, 1620, 1570, 1550, 1400, 1370, 1340, 1330, 1285, 1250, 1150, 1100, 985, 840	1.21 (3H, t, ^3J = 7.23, CH_3 in Et); 1.51 (36H, s, $12CH_3$); 3.47 (2H, m, ^3J = 7.23, ^3J = 5.91, NCH_2); 5.92 (1H, t, ^3J = 5.91, NH); [9.04, br. s]	1.00	86.8
2d	$C_{28}H_{44}N_6O_{12}$	51.53 / 51.06	6.71 / 6.73	12.36 / 12.76	101-102	3375, 2975, 2930, 1750, 1600, 1570, 1450, 1400, 1375, 1280, 1250, 1150, 1100, 1060, 835	0.95 (3H, t, ^3J = 7.03, CH_3 in Pr); 1.52 (36H, s, $12CH_3$); 1.61 (2H, m, ^3J = 7.03, ^3J = 7.28, CH_2); 3.51 (2H, m, ^3J = 7.28, ^3J = 6.32, NCH_2); 5.87 (1H, t, ^3J = 6.32, NH); [9.05, br. s]	2.00	94.8
2e	$C_{28}H_{44}N_6O_{12}$	51.25 / 51.06	6.85 / 6.73	12.50 / 12.76	156-157	3350, 2975, 2930, 1760, 1595, 1575, 1565, 1530, 1460, 1420, 1370, 1340, 1280, 1250, 1150, 1140, 1110, 980, 835	1.25 (6H, d, ^3J = 6.65, $2CH_3$ in *i*-Pr); 1.51 (36H, s, $12CH_3$); 4.14 (1H, m, ^3J = 8.35, ^3J = 6.65, NCH); 5.63 (1H, t, ^3J = 8.35, NH); [8.80, br. d]	0.50	94.7
2f	$C_{28}H_{42}N_6O_{12}$	—	—	12.30 / 12.79	109-110	3370, 2980, 2930, 1715, 1680, 1600, 1575, 1530, 1450, 1400, 1370, 1330, 1280, 1250, 1145, 1100, 1070, 980, 810, 800	1.52 (36H, s, $12CH_3$); 4.07 (2H, t, ^3J = 6.03, CH_2); 5.21 (2H, m, CH_2=); 5.85 (1H, m, CH=); 5.96 (1H, t, ^3J = 6.03, NH); [9.22, br. s]	1.30	94.8
2g	$C_{29}H_{46}N_6O_{12}$	52.19 / 51.93	6.81 / 6.91	12.50 / 12.52	142-143	3400, 2970, 2925, 1760, 1590, 1570, 1560, 1475, 1455, 1395, 1365, 1345, 1275, 1250, 1145, 1100, 1050, 835	1.44 (9H, s, $3CH_3$); 1.51 (18H, s, $6CH_3$); 1.53 (18H, s, $6CH_3$); 5.77 (1H, s, NH); [7.85, br. s]	24.00	61.0

TABLE 1 (continued)

1	2	3	4	5	6	7	8	9	10
2h	$C_{27}H_{42}N_6O_{13}$	49.21 / 49.24	6.37 / 6.42	12.71 / 12.75	137-138	3100, 2970, 2920, 1755, 1600, 1575, 1450, 1395, 1370, 1345, 1330, 1285, 1250, 1150, 1100, 980, 835	1.50 (36H, s, 12CH_3); 2.92 (1H, br. s, OH); 3.59 (2H, m, ^3J = 5.63 , ^3J = 4.88, NCH_2); 3.79 (2H, t, ^3J = 4.88, OCH_2); 6.97 (1H, t, ^3J = 5.63, NH); [8.83, br. m]	0.60	94.2
2i	$C_{27}H_{43}N_7O_{12}$	49.35 / 49.30	6.42 / 6.59	—	145-146	3400, 3200, 2970, 2930, 1760, 1700, 1670, 1570, 1550, 1450, 1390, 1360, 1320, 1270, 1250, 1150, 1080, 980, 830	1.507 (18H, s, 6CH_3); 1.514 (18H, s, 6CH_3); 2.91 (2H, t, ^3J = 5.63, CH_2NH_2); 3.46 (2H, t, ^3J = 5.63, CH_2NH); [3.09, br. s, NH_2] and [8.60, br. s, NH]	0.60	96.9
2j	$C_{32}H_{44}N_6O_{12}$	54.13 / 54.54	6.27 / 6.29	11.91 / 11.92	146-147	3375, 2980, 2925, 1760, 1710, 1600, 1570, 1530, 1450, 1400, 1370, 1330, 1280, 1250, 1140, 1100, 1060, 830	1.50 (18H, s, 6CH_3); 1.52 (18H, s, 6CH_3); 4.64 (2H, d, ^3J = 6.00, CH_2Ph); 6.13 (1H, t, ^3J = 6.00, NH); 7.33 (5H, m, Ph); [9.50, br. s]	0.50	88.1
2k	$C_{27}H_{42}N_6O_{12}$	50.48 / 50.30	6.69 / 6.57	13.02 / 13.03	159-160	2970, 2930, 1760, 1750, 1600, 1560, 1530, 1500, 1410, 1390, 1385, 1320, 1275, 1240, 1140, 1085, 845, 800	1.52 (36H, s, 12CH_3); 3.21 (6H, s, 2CH_3)	0.16	93.5
2l	$C_{29}H_{44}N_6O_{13}$	—	—	12.02 / 12.27	149-150	2980, 2930, 1760, 1600, 1570, 1510, 1450, 1370, 1280, 1250, 1150, 840	1.51 (36H, s, 12CH_3); 3.75 (4H, m, 2NCH_2); 3.85 (4H, m, 2OCH_2)	24.00	33.3
3	$C_{25}H_{39}N_7O_{12}$	47.05 / 47.69	6.46 / 6.24	16.19 / 15.57	78-79	3330, 3270, 2975, 2925, 1750, 1730, 1565, 1550, 1530, 1470, 1450, 1390, 1370, 1330, 1275, 1250, 1150, 1140, 1115, 1090, 985, 835	1.53 (36H, s, 12CH_3); 4.07 (2H, br. s, NH_2); 7.34 (1H, br. s, NH); 4.54 (br. s, NH_2); 10.12 (br. s, NH)	1.50	91.1
4	$C_{14}H_{24}N_8O_6$	—	—	27.84 / 27.98	109-110	3400-3100 broad band, 2975, 2925, 1760, 1585, 1570, 1540, 1510, 1435, 1400, 1360, 1230, 1210, 1150, 1090, 1040, 970	1.52 (18H, s, 6CH_3); 4.05 and 4.20 (4H, two br. s, 2NH_2); 7.40; 7.54; 8.08; 8.54 (2H, four br. s, NH); 4.15 (br. s, NH_2); 8.90 (br. s, NH)	1.50	75.0
6	$C_{12}H_{24}N_2O_4$	49.05 / 49.30	8.60 / 8.27	9.98 / 9.58	129-130 (dec.)	—	1.42 (18H, s, 6CH_3); 2.42 (3H, s, CH_3); 7.67 (3H, br. s, 3H, $CH_3N\underline{H_3^+}$)	—	83.0
7	$C_{34}H_{60}N_8O_{14}$	51.66 / 50.73	7.79 / 7.51	14.37 / 13.92	160 (dec.)	3400, 2920, 2925, 1760, 1590, 1570, 1560, 1530, 1475, 1455 1395, 1365, 1345, 1275, 1250, 1145, 1100, 940, 835, 825	1.16 (12H, t, ^3J = 7.3, 4CH_3); 1.45 (36H, s, 12CH_3); 2.99 (8H, q, ^3J = 7.2, 4CH_2); 9.20 (4H, narrow m, 2NH_2^+)	42.00	98.0

* ^{1}H NMR spectra of compounds **2a** and **6** were taken in DMSO-d_6, compounds **2b-l 3**, **4**, and **7** in $CDCl_3$.

*^{2} The chemical shifts of the NH and NH_2 groups in DMSO-d_6 are given in square brackets.

TABLE 2. ^{13}C NMR Spectra for the Compounds Synthesized

Com-pound	Chemical shifts, δ, ppm (Δδ)						
	$C_{(2)}$	$C_{(4)}$ and $C_{(6)}$	COO	C–NO_2	$(CH_3)_3\underline{C}$	CH_3	Other groups
2a*	166.30	169.02	158.19	100.50	86.27	27.50	
2b	165.59	168.80 168.00 (0.80)	158.30	101.20 100.77 (0.43)	86.08 85.97 (0.11)	27.50	28.00 (CH_3N)
2c	164.94	168.76 168.10 (0.66)	158.29	101.41 100.69 (0.72)	86.01 85.88 (0.13)	27.48	36.27 (CH_2N) 14.13 (CH_3)
2d	165.21	168.79 168.13 (0.66)	158.33	101.18 100.75 (0.43)	86.03 85.91 (0.12)	27.52	43.03 (CH_2N) 22.23 (CH_2) 11.12 (CH_3)
2e	164.27	168.75 168.14 (0.61)	158.33	101.15 100.72 (0.43)	86.03 85.90 (0.13)	27.52	43.59 (CH) 22.13 (CH_3)
2f	165.07	168.82 168.29 (0.53)	158.236	101.0 100.7 (0.30)	86.07 85.95 (0.12)	27.48	132.26 (CH=) 117.80 (CH_2=) 43.56 (CH_2N)
2g	164.28	168.03 167.85 (0.18)	158.33	101.03 100.64 (0.39)	86.00 85.96 (0.04)	27.49 27.47 (0.02)	46.33 (C_{quat}) 28.22 (CH_3)
2h	165.13	168.63 167.79 (0.84)	158.45 158.30 (0.15)	101.12 100.66 (0.46)	86.35 86.10 (0.25)	27.47	60.53 (CH_2O) 43.13 (CH_2N)
2i	165.18	168.58 167.95 (0.63)	158.31	101.2*[2] 100.6 (0.6)	85.98 85.61 (0.12)	27.49 27.47 (0.02)	43.38 (CH_2NH) 40.37 (CH_2NH_2)
2j	165.14	168.94 168.40 (0.54)	158.27 158.26 (0.01)	101.10 100.72 (0.38)	86.12 86.03 (0.08)	27.50 27.47 (0.03)	136.45, 128.86, 128.02, 127.82 (C_6H_5) 45.20 (CH_2N)
2k	163.99	167.86	158.41	101.36	85.76	27.49	36.52 (CH_3N)
2l	163.43	168.28	158.31	101.19	85.94	27.53	66.16 (CH_2O) 43.89 (CH_2N)
3	166.76	169.07 168.14 (0.93)	158.16	100.98 100.64 (0.34)	86.25	27.50	
4	167.41 (2) and (4)	166.93	158.97	101.09	85.629	27.634	
7*[3]	168.99 [170.41]		158.70 [163.13]	100.98 [112.77]	85.05 [80.06]	27.39 [28.12]	42.30 (CH_2N) 11.29 (CH_3)

* ^{14}C NMR spectrum = 15.25 ppm.

*[2] low accuracy due to low peak intensity.

*[3] The chemical shifts of the corresponding carbon atoms assigned to the negatively charged molecular fragments are given in square brackets.

EXPERIMENTAL

IR spectra were recorded on a Specord UR-20 instrument for KBr tablets. ^1H, ^{13}C, and ^{14}N NMR spectra were obtained on a Bruker AM-300 instrument (300, 75, and 21 MHz respectively) with TMS as internal standard. Melting points were determined on a Boetius type heating block with a heating rate of 4°C/min at the melting point. The synthesis of 2,4,6-tris[di(*tert*-butoxycarbonyl)nitromethyl]-1,3,5-triazine (**1**) has been reported in [1]. The characteristics of the compounds synthesized are given in Tables 1 and 2.

Substituted 2-Amino-4,6-bis[di(*tert*-butoxycarbonyl)nitromethyl]-1,3,5-triazines (2a-f,h-l). (General Method). The corresponding amine or its aqueous solution (15 mmol) was added to a stirred solution of the nitro ester **1** (1.716 g, 2 mmol) in ethanol (20 ml) at room temperature and the stirring was continued for the time indicated in Table 1. It was then diluted with water to a volume of 40-50 ml and the precipitated solid product was filtered off, washed with water, and dried in air.

2-*tert*-Butylamino-4,6-bis[di(*tert*-butoxycarbonyl)nitromethyl]-1,3,5-triazine (2g). *tert*-Butylamine (5 ml, 45 mmol) was added to a solution of compound **1** (1.716 g, 2 mmol) in ether (20 ml) and held at this temperature for 24 h. Solvent was distilled off and the residue was chromatographed (L 40/100 grade silica gel, eluent hexane–chloroform, 4:1) to give the product **2g**.

2-Hydrazino-4,6-bis[di(*tert*-butoxycarbonyl)nitromethyl]-1,3,5-triazine (3). Hydrazine hydrate (100%, 0.4 ml, ~8 mmol) was added to a solution of the nitro ester **1** (1.716 g, 2 mmol) in ethanol (20 ml) with cooling (ice bath). The mixture was stirred at the same temperature for 1.5 h, diluted with water to a volume of 40-50 ml, and the precipitated solid product was filtered off and dried in air.

2,4-Dihydrazino-6-[di(*tert*-butoxycarbonyl)nitromethyl]-1,3,5-triazine (4). Hydrazine hydrate (100%, 1.5 ml, ~ 24 mmol) was added to a solution of the nitro ester **1** (1.716 g, 2 mmol) in ethanol (20 ml) at room temperature with stirring. The mixture was stirred for 1.5 h, diluted with water to a volume of 60-70 ml, and held until formation of a solid product which was filtered off and dried in air.

Methylammonium Salt of Di-*tert*-butyl Nitromalonate (6). An aqueous solution of methylamine (25%, 2 ml) (or passage of gaseous methylamine) was added to a solution of compound **1** (1.716 g, 2 mmol) in ether (20 ml) at room temperature and with stirring until the solid product **6** was formed. The precipitate was filtered off, washed with ether, and dried in air. The ether layer of the filtrate was dried and evaporated to give compound **2b** in 93% yield.

2,4-Bis(diethylammonium) Salt of 2,4-Bis(*tert*-butoxycarbonylnitromethyl)-6-[di(*tert*-butoxycarbonyl)nitromethyl]-1,3,5-triazine (7). Diethylamine (5 ml) was added to a solution of compound **1** (1.716 g, 2 mmol) in ether (20 ml) and the mixture was held at room temperature for 42 h. The precipitated solid product **7** was filtered, washed with ether, and dried in air.

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