

**Molecular Complexes Exhibiting Polarization Bonding.**  
**VI. The Crystal Structure of the 2,4,6-Tri(dimethylamino)-1,3,5-triazine**  
**– *s*-trinitrobenzene Complex**

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The 1:1 complex formed between 2,4,6-tri(dimethylamino)-1,3,5-triazine and *s*-trinitrobenzene crystallizes as yellow monoclinic needles, space group  $P2_1/m$ , with two molecules of complex in the unit cell of dimensions  $a = 6.72 \pm 0.02$ ,  $b = 16.06 \pm 0.03$ ,  $c = 9.59 \pm 0.02$  Å,  $\beta = 105.2^\circ \pm 0.5^\circ$ . The structure was solved by Patterson methods and refined by three-dimensional Fourier and least-square methods. The component molecules are stacked alternately in infinite columns along the *a* (needle) axis, the mean perpendicular separation of the molecules being 3.36 Å. Both molecules are planar to within experimental error and are similarly orientated with respect to the crystal axes. The line joining the molecular centres is perpendicular to the planes of the molecules.

### Introduction

The 1:1 molecular complex formed between 1,3,5-trinitrobenzene and 2,4,6-tri-(dimethylamino)-1,3,5-triazine was initially described by Das, Shaw, Smith, Last & Wells (1963). The molecules have similar shapes and dimensions but the nitro-groups in one are electron withdrawing whereas the dimethylamino groups in the other are electron donating. It was of interest to discover whether the combination of the charge distribution within the molecules, resulting from the electron affinities of the substituent groups, and the very favourable conditions existing for molecular  $\pi$ -orbital overlap (e.g. Wallwork, 1961) would be sufficient to cause the molecules to lie directly over one another in an infinite stack.

### Experimental

Yellow needles of the complex, large enough for X-ray analysis, were grown by recrystallization from chloroform of a sample of the complex kindly supplied to us by Dr B. C. Smith of Birkbeck College, London. In plane polarized light the crystals exhibited dichroism (with colours ranging from yellow-green to ochre) with the maximum absorption of light when the electric vector was parallel to the needle axis.

X-ray photographs were taken by the multiple-film Weissenberg technique for the  $0kl$ ,  $1kl \dots 4kl$  and  $h0l$  zones of reflexions. The intensities of the  $0kl$ ,  $1kl$  and  $2kl$  reflexions were estimated by a microphotometer method (Wallwork & Standley, 1954) and the  $3kl$  and  $4kl$  reflexions were estimated visually. The usual Lorentz and polarization factors were applied to the intensities but no corrections for absorption were applied. 593 independent non-zero reflexions were measured corresponding to about a quarter of those in the copper sphere.

### Crystal data

$C_3N_3(NMe_2)_3 \cdot C_6H_3(NO_2)_3$ ,  $M = 465.4$ . Monoclinic,  $a = 6.72 \pm 0.02$ ,  $b = 16.06 \pm 0.03$ ,  $c = 9.59 \pm 0.02$  Å,  $\beta = 105.2^\circ \pm 0.5^\circ$ ,  $U = 998.9$  Å<sup>3</sup>,  $D_m = 1.40$  g.cm<sup>-3</sup>,  $Z = 2$ ,  $D_c = 1.41$  g.cm<sup>-3</sup>.  $F(000) = 444$ . Cu  $K\alpha$ ,  $\lambda = 1.542$  Å,  $\mu = 10.7$  cm<sup>-1</sup>. Absent spectra,  $0k0$  when  $k$  odd. Space group  $P2_1$ (No.4) or  $P2_1/m$ (No.11). Statistical tests (Howells, Phillips & Rogers, 1950) indicated  $P2_1/m$  and this is confirmed by refinement of the structure.

### Determination and refinement of the structure

Examination of the X-ray data revealed that the  $1kl$  and  $3kl$  zones of reflexions were very much weaker than the  $0kl$ ,  $2kl$  or  $4kl$  zones. This fact, together with the dichroism, the  $a$  cell dimension of 6.72 Å and the symmetry requirements of the probable space group, suggested a structure in which the component molecules were both approximately perpendicular to the  $a$  axis and stacked alternately, with a separation of  $a/2$ , in columns parallel to this axis.

The molecular orientations round the  $a$  axis were deduced by comparison of the  $0kl$  weighted reciprocal lattice with the optical transform of a pattern which approximately represented either molecule. The  $a$  and  $c$  axis Patterson projections were interpreted in terms of a trial structure based on all these indications. The  $y$  and  $z$  coordinates of the patterns were partially refined by successive structure factor calculations and Fourier  $0kl$  electron density projections to a stage where  $R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.24$ .

The structure was further refined from the  $hkl$  data, by the block-diagonal structure factor least-squares method using a program written by Dr R. D. Diamand. The weighting scheme used throughout was  $\sqrt{w} = 1$  if  $|F_o| < F^*$  otherwise  $\sqrt{w} = F^*/|F_o|$ . Hydrogen atoms were not included in the calculations and the scattering factors of Berghuis, Haanappel, Potters, Loopstra, Mac Gillavry & Veenendaal (1955) for carbon, nitrogen and

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Table 1. Comparison of observed and final calculated structure factors

The asterisk line gives  $h, k$ ; subsequent lines give  $l, 50F_o, 50F_c$ .

0	0										
8	-906	-939	6	-1052	-1017	5	-1284	-1382	4	3098	3524
3	1349	934	2	-4656	-9234	1	-1902	-2345			
0	1										
9	930	482	7	-393	-353	6	-377	-271	5	-1700	-1751
4	1477	1600	3	-1365	-931	2	-1461	-1335	1	-3810	-3241
0	2										
6	-281	-236	5	297	216	4	-947	-950	3	728	391
1	3532	3941	0	-3693	-4302						
0	3										
6	-626	-420	5	-56	-334	4	-1421	-1977	3	-217	-196
2	-811	-718	1	-3612	-3959						
0	4										
8	946	950	7	-265	-253	6	971	924	5	-482	-528
3	321	292	2	-1140	-876	1	-2512	-2642	0	-763	-624
0	5										
7	570	459	4	-474	-406	3	2216	2464	2	-1236	-1168
1	-1437	-1213									
0	6										
8	-297	-252	7	1750	1847	6	-674	-630	5	-827	-722
4	602	379	3	1634	1328	2	-1019	-593	1	-2175	-2122
0	-490	-205									
0	7										
7	-321	-354	6	1421	1481	5	610	518	4	-522	-450
3	-979	-697	2	3074	3374						
0	8										
7	-1324	-1327	6	795	693	5	658	687	4	-257	-270
3	-739	-700	2	610	567	1	1044	1048			
0	9										
6	-321	-212	3	1156	990	2	-337	-223			
0	10										
6	-554	-574	1	1180	1089	0	273	303			
0	11										
4	-730	-717	3	345	324	1	-1011	-852			
0	12										
3	-265	-270	1	-730	-584	0	1164	1144			
0	13										
5	-514	-541	1	-1204	-924						
0	14										
4	-578	-578	2	610	653	1	466	372	0	-2047	-2073
0	15										
5	321	324	1	466	445						
0	16										
10	88	19	9	169	268	7	-161	-265	6	-112	-62
5	498	116	4	-313	-538	3	-450	-335	2	546	344
1	104	168	-2	602	997	-4	289	276	-5	490	539
-7	-257	-177	-9	-209	-252	-10	-193	-184			
0	17										
9	120	101	7	-104	-76	6	128	174	5	-169	-25
4	-169	-282	3	-136	-166	-2	273	361	1	-289	-653
-2	120	292	-3	-72	-327	-4	273	140	-5	506	258
-6	297	214	-10	-144	-139						
0	18										
1	2										
9	-96	-89	6	136	82	5	-329	-287	4	-321	-201
3	329	412	2	329	212	-1	458	691	-2	-153	-267
-3	-233	-208	-4	88	356	-5	169	86	-6	-225	-163
-7	128	90									
0	19										
7	80	74	6	161	36	5	169	88	4	-289	-225
3	-193	-334	1	193	213	0	-393	-688	-1	-209	-420
-2	257	219	-3	586	379	-4	-177	-152	-5	153	87
-8	128	69									
0	20										
1	4										
8	-104	-75	6	104	206	4	-88	-62	3	-96	-57
2	321	275	1	217	92	0	-153	-282	-1	-289	-279
-2	-257	-295	-4	-273	-163	-5	-225	-283	-6	-353	-188
-7	185	115									
0	21										
1	5										
7	209	81	6	-233	-249	5	88	64	4	249	150
3	-289	-139	2	273	238	1	233	235	0	-522	-490
-2	530	987	-4	-265	-194	-6	-193	-72	-7	-289	-98
-8	209	56									
0	22										
1	6										
7	249	78	6	345	194	5	104	111	4	-193	-161
3	-249	-159	2	177	295	1	321	309	-1	-337	-388
-2	72	13	-3	-458	-312	-4	104	56	-5	257	185
-6	-193	-198	-8	233	384						
0	23										
1	7										
8	-153	-102	7	-361	-358	5	217	294	4	-329	-256
3	-104	-198	2	730	623	1	128	162	0	-225	-256
-2	425	466	-4	-345	-320	-5	273	281	-7	-530	-443
-8	-257	-134									
0	24										
1	8										
7	-160	-28	6	-305	-128	4	169	152	3	-329	-118
-2	-257	-283	1	305	258	-1	-361	-318	-2	-417	-228
-5	602	610	-4	120	119	-5	-225	-225	-6	209	106
-8	-128	-228									
0	25										
1	9										
7	88	40	2	-249	-343	1	225	285	0	241	277
0	26										
1	10										
5	136	168	4	-88	-91	3	-249	-245	2	104	132
1	345	322	5	-104	-129	-1	-249	-291	-2	233	368
-5	-104	-44	-3	193	145	-4	-169	-127			
0	27										
1	11										
3	-144	-162	2	-104	-191	-1	177	35	-2	-201	-206
0	28										
1	12										
2	-88	-202	0	169	87						
0	29										
1	13										
4	-161	-16	2	72	77	0	-321	-59	-1	-265	-60
-3	112	78	-4	104	54						
0	30										
1	14										
5	112	39	1	161	147	0	209	66	-1	-289	-197
-5	-153	-156									
0	31										
1	15										
4	72	116	0	281	286						
0	32										
1	16										
-1	153	158									



Table 1 (cont.)

• 4	0											
8	-281	-283	6	225	268	4	618	256	0	1124	1253	
-1	883	1023	-2	-2087	-2657	-3	-875	-747	-4	450	368	
-5	1108	890	-6	-1710	-1362	-10	329	447				
• 4	1											
4	-257	-238	3	1060	987	2	-265	-249	1	-755	-804	
-2	-957	-1189	-3	-1268	-925	-4	185	302	-5	827	648	
-6	-329	-299	-7	-337	-287							
• 4	2											
3	456	450	2	-257	-264	0	706	705	-1	-843	-647	
-2	-112	-187	-3	153	98	-4	337	318	-5	-1076	-892	
-6	506	400										
• 4	3											
5	-255	-230	4	177	192	3	-169	-153	2	209	134	
3	939	775	-2	1597	1127	-3	-578	-532	-4	-120	-102	
-7	-433	-436										
• 4	4											
6	-265	-238	4	-185	-115	2	-490	-464	0	-530	-407	
-1	313	347	-2	458	311	-3	88	66	-4	787	686	
-7	353	415										
• 4	5											
6	-321	-367	5	265	315	1	-450	-377	0	-169	-121	
-1	151	210	-2	127	172	-3	-169	-179	-4	-586	-583	
-5	233	244	-7	353	393	-8	-682	-801	-9	361	387	
• 4	6											
6	257	331	5	-626	-661	2	554	555	1	-1300	-1150	
-1	225	283	-3	1292	1054	-4	-506	-538	-7	361	389	
-9	-313	-358										
• 4	7											
6	578	678	5	-233	-255	4	-385	-390	2	450	356	
2	-939	-811	-1	153	127	-2	-642	-689	-3	-578	-471	
-4	1084	1005	-6	-546	-624	-7	-385	-463	-8	1132	1378	
-9	-209	-161										
• 4	8											
6	-201	-237	5	562	559	3	-185	-250	2	-361	-347	
1	875	845	-3	-730	-644	-4	297	292	-7	-377	-433	
-9	201	259										
• 4	9											
5	209	272	-4	-225	-57	-6	265	101	-8	-281	-442	
• 4	10											
3	136	117	0	257	300	-2	-120	-212				
• 4	11											
2	136	152	0	345	290	-2	450	510	-3	-233	-243	
• 4	12											
-2	-265	-245	-5	265	384	-6	-120	-131				
• 4	13											
0	-136	-174	-1	947	1026	-2	-233	-316	-3	-265	-296	
-5	177	165										
• 4	14											
0	-401	-418	-1	-120	-172	-2	634	677	-6	209	388	
• 4	15											
-1	-498	-588										
• 6	0											
2	361	342	0	-393	-468	-1	-859	-610	-2	1846	1754	
-4	-497	-596	-6	658	676							
• 8	0											
-2	-578	-594										

Table 2. Final atomic parameters

Each positional parameter and its e.s.d. on the line below is  $\times 10^4$ 

<i>s</i> -Trinitrobenzene					<i>s</i> -Tridimethylaminotriazine				
Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>B</i>	Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>B</i>
C(1)	2382 28	2500 —	2081 16	2.1 Å <sup>2</sup>	N(3)	7550 32	2500 —	2216 16	5.5 Å <sup>2</sup>
C(2)	2739 28	1821 8	2859 13	4.0	C(5)	7586 26	1818 9	2947 14	5.1
C(3)	3110 22	1737 8	4343 13	2.8	N(4)	8074 24	1766 9	4433 14	6.1
C(4)	3405 38	2500 —	4989 20	5.3	C(6)	8224 32	2500 —	5031 18	3.1
N(1)	2415 20	1005 8	2087 12	4.1	N(5)	7337 28	1066 9	2154 17	7.3
N(2)	3672 31	2500 —	6570 19	6.0	N(6)	8727 30	2500 —	6587 19	4.8
O(1)	1916 23	1041 9	0683 12	7.8	C(7)	7012 33	1016 12	0663 21	7.8
O(2)	2774 20	0379 7	2881 13	6.8	C(8)	7475 39	0283 11	2784 27	4.4
O(3)	4010 22	1820 9	7254 12	7.8	C(9)	8884 33	1703 13	7310 18	6.4

oxygen were used. The reflexions too weak to be observed were omitted from the analysis. Eight cycles of least-squares refinement using individual isotropic temperature factors and  $F^* = 1000$  (on the scale of Table 1) were followed by six cycles using individual anisotropic temperature factors and  $F^* = 600$ . The final reliability index,  $R$ , was 0.165. The comparison of observed and final calculated structure factors is shown in Table 1.

Sections of the three-dimensional electron-density distribution were calculated prior to anisotropic refinement and a composite electron-density diagram is shown in Fig. 1.

The final atomic coordinates and their estimated standard deviations (derived from the least-squares normal equations block-diagonal matrix) are summarized in Table 2. The anisotropy of the thermal parameters is unlikely to have quantitative significance in view of the disorder effects discussed below, so only the equivalent isotropic parameters are quoted in Table 2. The bond distances and interbond angles, together with their standard deviations, are illustrated in Fig. 2.

A least-squares plane through each of the molecules was calculated by the modification (Blow, 1960) of the method of Schomaker, Waser, Marsh & Bergman (1959). All of the atoms, other than hydrogen, in each molecule were used to define each plane. The pertinent parameters of the planes and the deviations of the atoms from them are summarized in Table 3. Least-squares planes were also calculated for the central ring systems of each of the molecules and also for each group of the type C-NO<sub>2</sub> and C-NC<sub>2</sub>, but the improvement in the deviations of atoms from these separate planes was not sufficient to justify the description of the molecules in this more complicated manner.

### Description of the structure and discussion

The structure consists of infinite columns of alternate donor and acceptor molecules arranged in a plane-to-plane manner. The columns are parallel to the  $a$  axis of the unit cell. The normal to the mean plane of the trinitrobenzene molecule is inclined at 2.8° to the stacking axis ( $+a$ ), at 90° to the  $+b$  axis and at 108.0° to

the  $+c$  axis. The normal to the mean plane of the tri(dimethylamino)triazine molecule is at 3.4°, 90° and 108.6° to the  $+a$ ,  $+b$  and  $+c$  axes respectively. Fig. 3 is a diagrammatic representation of the bounded projection ( $y=0$  to  $y=0.25$ ) of the structure showing the principal close approaches between molecules in the same column. The mean separations of the trinitrobenzene molecule from the two tri(dimethylamino)triazine molecules adjacent to it in the column are 3.33 and 3.39 Å. This difference is unlikely to be significant and the mean interplanar separation may be taken as 3.36 Å. Although this is not much less than the expected van der Waals separation of the molecules, there is a large number of short contacts due to the almost complete overlapping of the two molecules when the structure is viewed along the  $a$  axis. The fairly wide variation in the intermolecular approaches within one column of molecules is due mainly to the apparent lack of planarity of the molecules. The deviations from planarity seem to be quite random and no larger for the nitro and dimethylamino groups than for the central atoms. The root mean square deviation is 0.04 Å for each molecule (about  $2\sigma$ ) so, although the molecules may be slightly non-planar, no significance can be attached to the observed deviations, especially in view of the limitation of the X-ray data in the  $a^*$  direction to  $h=4$ .

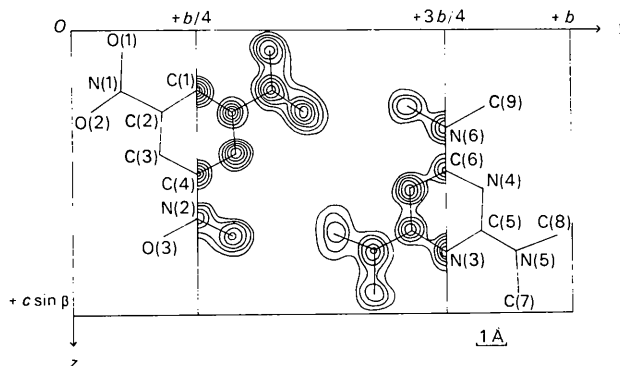


Fig. 1. Composite Fourier electron density diagram, from sections nearest to atomic centres, projected along the  $x$  axis, to show one layer of the structure. (Contours at 1, 2, 3, ... e.Å<sup>-3</sup>.)

Table 3. Molecular least squares planes

	<i>s</i> -Trinitrobenzene	<i>s</i> -Tri(dimethylamino)triazine
Perpendicular distance from origin	-1.20 Å	-4.54 Å
Direction cosines relative to orthogonal axes	$\left\{ \begin{array}{l} a \\ b \\ c^* \end{array} \right. \begin{array}{l} -0.9988 \\ 0.0000 \\ -0.0487 \end{array}$	$\left\{ \begin{array}{l} a \\ b \\ c^* \end{array} \right. \begin{array}{l} -0.9982 \\ 0.0000 \\ -0.0608 \end{array}$
Perpendicular distance of atoms from mean plane	$\left\{ \begin{array}{l} C(1) \\ C(2) \\ C(3) \\ C(4) \\ N(1) \\ N(2) \\ O(1) \\ O(2) \\ O(3) \end{array} \right. \begin{array}{l} 0.03 \text{ Å} \\ -0.05 \\ 0.01 \\ -0.06 \\ 0.01 \\ 0.09 \\ 0.06 \\ -0.07 \\ 0.00 \end{array}$	$\left\{ \begin{array}{l} N(3) \\ C(5) \\ N(4) \\ C(6) \\ N(5) \\ N(6) \\ C(7) \\ C(8) \\ C(9) \end{array} \right. \begin{array}{l} -0.1 \text{ Å} \\ 0.02 \\ -0.02 \\ 0.00 \\ 0.03 \\ -0.04 \\ -0.04 \\ 0.06 \\ 0.00 \end{array}$

The columns of molecules are arranged next to each other in an approximately close-packed array. This causes a trinitrobenzene molecule in one column, for example, to have six triazine neighbours in adjacent columns and also approximately in the same plane perpendicular to the *a* axis. Each triazine molecule similarly has six trinitrobenzene neighbours. The shortest sideways contacts are 3.50 Å, between oxygen

atoms and methyl groups, corresponding only to van der Waals interaction. This lack of strong sideways interaction between columns is consistent with the occurrence of disorder in the structure, recognized by the appearance of diffuse scattering on the X-ray photographs. This took the form of discs in reciprocal space of constant *h* and non-integral *k* and *l*. Such an effect would be caused by displacing columns of molecules

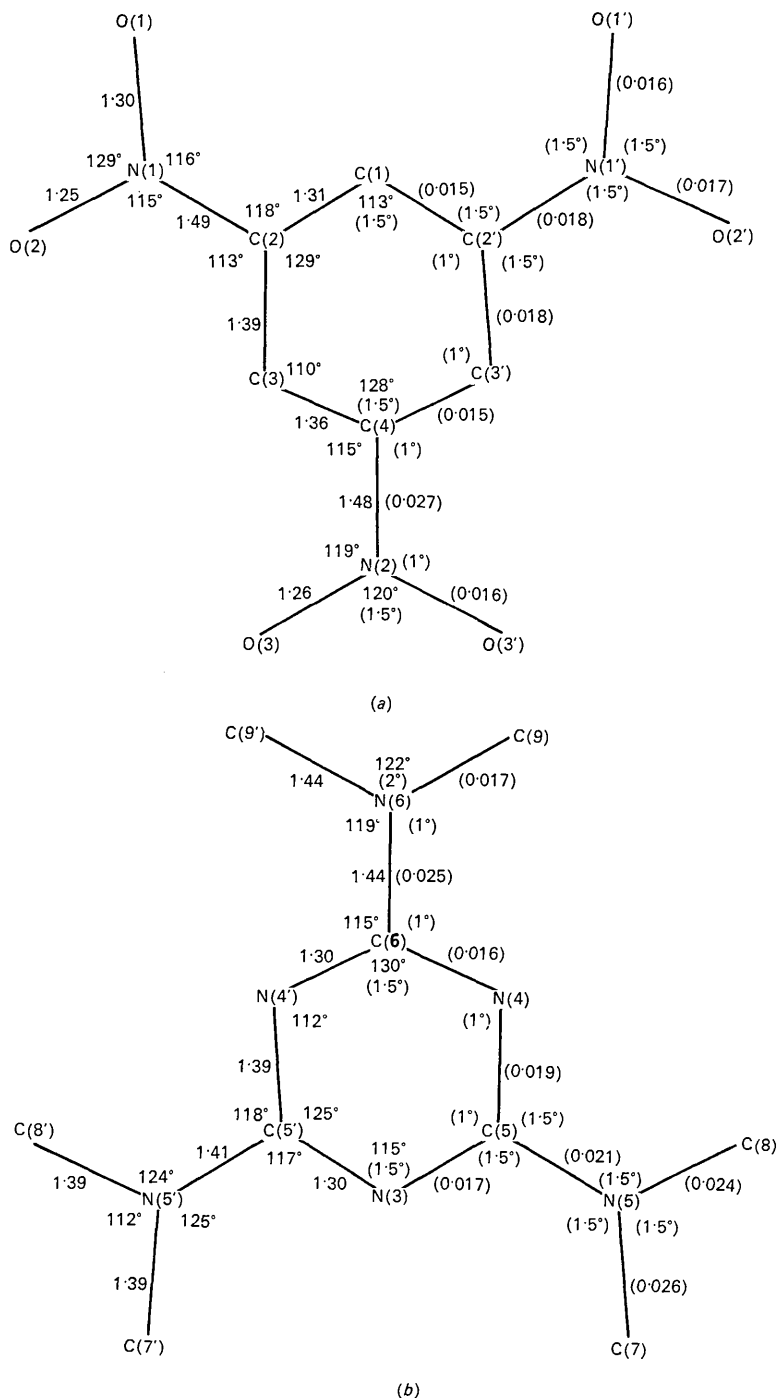


Fig. 2. Bond lengths (Å) and bond angles with (in parentheses) their standard deviations, for (a) *s*-trinitrobenzene, (b) 2,4,6-tri(dimethylamino)-1,3,5-triazine.

relative to each other in the  $x$  direction. In view of the almost identical size and shape of the two component molecules it might be expected that such a displacement by  $a/2$  would occur fairly easily, resulting in the occupation of some of the trinitrobenzene molecule sites by tri(dimethylamino)triazine molecules, and *vice versa*. Such disorder readily accounts for the final value of 0.165 for the reliability index.

The agreement in bond lengths and bond angles for chemically similar bonds and environments within each molecule is only moderate but it is consistent with the estimated standard deviations. The average values in *s*-trinitrobenzene,  $d(\text{C}-\text{C})=1.35$ ,  $d(\text{C}-\text{N})=1.49$  Å,  $\text{C}\hat{\text{C}}\text{C}=129^\circ$  at  $\text{C}(\text{NO}_2)$  and  $111^\circ$  at  $\text{CH}$ ,  $\text{C}\hat{\text{C}}\text{N}=115^\circ$ ,  $\text{C}\hat{\text{N}}\text{O}=117^\circ$  and  $\text{O}\hat{\text{N}}\text{O}=125^\circ$ , are in reasonable agreement with the corresponding values for the same molecule of 1.38, 1.49 Å, and  $125$ ,  $115$ ,  $117$ ,  $118$ ,  $126^\circ$  found in the complex with anthracene (Brown, Wallwork & Wilson, 1964) and 1.38<sub>5</sub>, 1.48 Å, and  $123$ ,  $117$ ,  $118$ ,  $118$ ,  $124^\circ$  in the two complexes with skatole and azulene (Hanson, 1964, 1965) but the average  $d(\text{N}-\text{O})$  of 1.27 is longer than expected and this is almost certainly due to the previously mentioned disorder effects. In the tri(dimethylamino)triazine molecule the average ring C-N bond length of 1.33 Å agrees with the average distance  $1.339 \pm 0.005$  Å quoted for conjugated heterocyclic C-N bonds (Sutton, 1965). In *s*-triazine itself the C-N distance is 1.314 Å and  $\text{C}\hat{\text{N}}\text{C}$  is  $115.1^\circ$  (Wheatley, 1957) compared with an average of  $113^\circ$  in the present structure. The average dimethylamino group dimensions,  $d(\text{N}-\text{ring C})=1.42$ ,  $d(\text{N}-\text{Me})=1.41$  Å,  $\text{C}\hat{\text{N}}\text{Me}=123^\circ$  and  $\text{Me}\hat{\text{N}}\text{Me}=117^\circ$ , may be compared with the corresponding values 1.35<sub>8</sub> Å, 1.49<sub>8</sub> Å and  $121.0^\circ$ ,  $118.2^\circ$  in *N,N*-dimethyl-*p*-nitroaniline (Mak & Trotter, 1965). These differences in the dimethylamino group dimensions are on the border of significance but are probably to be attributed as much to the disorder effects as to differences in the electronic structure between the two molecules. However the fact that the bond between the ring and the nitrogen atom is shorter in the tri(dimethylamino)triazine molecule than in the trinitrobenzene molecule is in agreement with the expected increased contribution to the resonance states

of structures involving  $\text{C}=\text{N}$  in the case of the dimethylamino group.

The interesting conclusion which may be drawn from this work is that, in spite of the direct overlap and identical orientations round the stacking axis of both component molecules, so that the conditions for overlap of molecular  $\pi$  orbitals are most favourable, a 'strong' complex is not formed. That is to say, the plane-to-plane separation (3.36 Å) is only slightly less than the expected van der Waals separation, and even the solid complex is not highly coloured but yellow (the maximum of the charge transfer band occurs at approximately  $24,800 \text{ cm}^{-1}$ ). In this sense the complex resembles, and is intermediate between, the complexes of *s*-trinitrobenzene with naphthalene (pale yellow and

perpendicular separation 3.45 Å) and with anthracene (orange and perpendicular separation 3.28 Å) although these two structures both have less favourable overlap of the component molecules (Wallwork, 1961). The spectroscopic properties of this type of complex clearly depend much more on the electronic properties of the components than on the efficiency with which the molecules can overlap in the structure of the complex.

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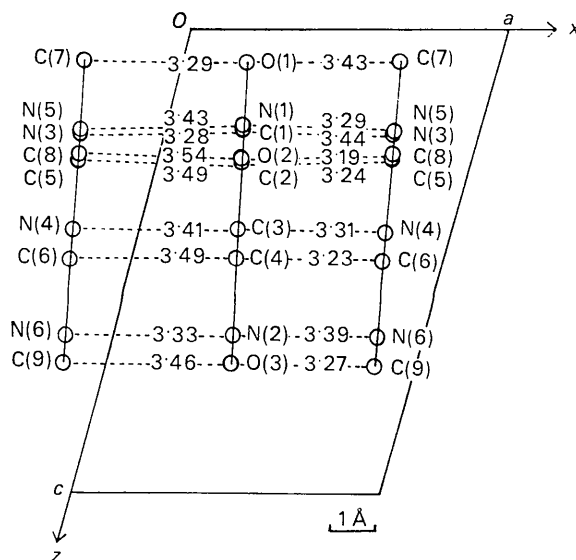


Fig. 3. Edge-on view of one column of molecules showing the close approaches (Å) between atoms of adjacent molecules.