phous with that of Th_2Ni_{17} (Florio, Baenziger & Rundle, 1956) and is a derivative structure af $CaZn_5$ (Haucke, 1940). However, we found Ba_2Mg_{17} to dissolve Mg substitutionally in its lattice. The substitution occurs only on the superlattice (here the $CaZn_5$ type lattice is considered to be the sublattice) and moreover only in a short range order. As partial evidence, we submit the 20° oscillation photographs of the single crystals obtained from samples corresponding to the atomic composition ratios of $BaMg_9$ and Ba_2Mg_{17}, rotated about the c^* axis, as shown in Fig. 1. The $3n(n=0, 1, 2, 3, \ldots)$ layers are diffractions due mostly to the sublattice whereas the layers 1, 2, 4, 5, ... *etc* are solely due to the superlattice. It is seen that the diffraction due to the superlattice in $BaMg_9$ lacks a mirror plane of symmetry across the zero layer. Furthermore, when the equi-inclination zero-layer Weissenberg photographs for both $BaMg_9$ and Ba_2Mg_{17} were made (rotated about the orthohexagonal axis) as shown in Fig. 2, the following conclusions were reached. The superlattice of $BaMg_9$ does not have $6/mmm$ diffraction symmetry whereas Ba_2Mg_{17} shows $6/mmm$ diffraction symmetry for both the sub- and the super-lattice. The latter has been confirmed to be a derivative of a $CaZn_5$ type structure as suggested by Goldish & Marsh (1955) but it is not disordered and the c_0 axis dimension is 15·52 Å instead of 10·53 Å (a_0 being 10·61 Å). The substitutional solution of Mg atoms in the superlattice of Ba_2Mg_{17} thus creates a disorder which cannot be detected by powder patterns as in the case of the investigation by Kripyakevich & Evdokimenko. Our complete investigation of all the intermediate phases of R–X systems (where $R=$ Sr, Ba and $X=$ Li, Mg) is in its final stage and will be reported in the near future.

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Bond lengths and angles in pentaerythritol tetranitrate.

By James Trotter, *Department of Chemistry, University of British Columbia, Vancouver 8, B.C., Canada*

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Very little information is available about the detailed structures of nitrate esters. Electron-diffraction investigations of fluorine nitrate and methyl nitrate (Pauling & Brockway, 1937) indicate non-planar molecules, while a more recent microwave study of methyl nitrate (Dixon and Wilson, 1961) suggests that the molecule is planar. The only X-ray analysis is that on pentaerythritol tetranitrate $C(CH_2O.NO_2)_4$ (Booth and Llewellyn, 1947); the authors noted three remarkable features of the molecular structure (Fig. 1): (i) the C(0)–C(1) bond (1·50 Å) is shorter than 1·54 Å; (ii) the C(1)–O(1) bond (1·37 Å) is considerably shorter than a normal single bond; (iii) the O(1)–N bond is of normal length. It might also be added that the N–O(2) and N–O(3) bonds seem to be abnormally long. No satisfactory explanation of these features can be given, and since no such abnormalities were noted in a recent analysis of *cis*-1,2-acenaphthenediol dinitrate (Mak & Trotter, 1963), the structure of pentaerythritol tetranitate has been reinvestigated to determine whether the abnormal distances are real.

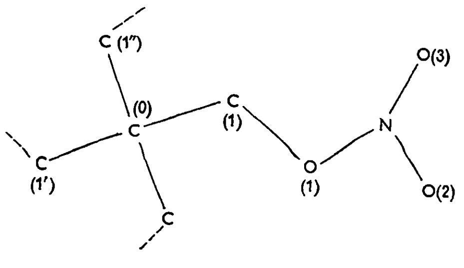

Fig. 1. Numbering of atoms in pentaerythritol tetranitrate.

Table 1. *Progress of refinement*

	R	$\Sigma w.\Delta F^2$
Initial coordinates	0·259	1129
1st least-squares cycle	0·180	654
2nd	0·160	529
3rd	0·153	525
4th	0·152	496
5th	—	—

Using Booth and Llewellyn's structure factor data (with the omission of the reflexions 021, 012, 121, 220, 222, 232, which might be affected by extinction), the scattering factors for C, N, O of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) and an overall $B=4{\cdot}5$ Å^2, the positional and isotropic thermal parameters and a scale factor were refined with a least-squares program for the IBM 1620 computer, written in FORTRAN, and with the general strategy suggested by Cruickshank (1961). The function minimized was $\Sigma w(|F_o|-|F_c|)^2$, with $w=|F_o|/10$ if $F_o<10$ and $w=10/|F_o|$ if $F_o \geqslant 10$. Refinement was complete in five cycles (Table 1), the final scale factor on Booth and Llewellyn's data being 1·079.

Table 2. *Final positional and thermal parameters with standard deviations*

Atom	*x*	$10^5 \times \sigma(x)$	*y*	$10^5 \times \sigma(y)$	*z*	$10^5 \times \sigma(z)$	*B*	$\sigma(B)$
C(0)	0·0000	0	0·0000	0	0·0000	0	1·56 Å^2	0·34
C(1)	0·1207	122	0·0636	117	0·1270	164	2·36	0·20
O(1)	0·1608	86	−0·0483	78	0·2681	126	2·58	0·13
N	0·2870	104	−0·0165	101	0·3716	134	2·41	0·16
O(2)	0·3158	101	−0·1050	99	0·4951	173	4·32	0·20
O(3)	0·3500	97	0·0927	98	0·3240	137	4·08	0·20

The final positional and thermal parameters with their standard deviations (calculated from the inverses of the diagonal terms in the least-squares matrix) are listed in Table 2, and the bond lengths and valency angles are compared in Table 3 with the values of Booth and Llewellyn. It is immediately apparent that none of the remarkable features previously noted is real. The C(0)–C(1) distance (1·53$_7$ Å) is equal to the normal single-bond length; the C(1)–O(1) (1·46$_2$ Å) and O(1)–N(1) (1·40$_4$ Å) bonds are both slightly longer than the distances quoted for single bonds (1·43 and 1·36 Å respectively in *Tables of Interatomic Distances*, 1958). All the dimensions of the nitrate group are very similar to those in nitric acid (Millen and Morton, 1960). The pentaerythritol tetranitrate molecule thus exhibits no unusual features.

Table. 3. *Bond lengths and valency angles (with standard deviations)*

	Booth and Llewellyn (1947)	Present analysis
C(0)–C(1)	1·498 Å	1·537 ± 0·011 Å
C(1)–O(1)	1·371	1·462 ± 0·014
O(1)–N	1·364	1·404 ± 0·012
N–O(2)	1·272	1·203 ± 0·014
N–O(3)	1·285	1·225 ± 0·013
C(1)–C(0)–C(1′)	112·6	113·0 ± 0·6°
C(1)–C(0)–C(1″)	107·9	107·7 ± 0·6
C(0)–C(1)–O(1)	108·5	105·6 ± 0·7
C(1)–O(1)–N	115·7	112·6 ± 0·7
O(1)–N–O(2)	116·1	112·5 ± 0·8
O(1)–N–O(3)	121·1	117·1 ± 0·8
O(2)–N–O(3)	122·7	130·4 ± 0·9

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Magnetically different crystal modifications of copper(II) salicylate.

By Motomichi Inoue, Michihiko Kishita and Masaji Kubo, *Chemistry Department, Nagoya University, Chikusa, Nagoya, Japan*

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Ploquin (1951; Foëx, 1957) has determined the magnetic susceptibility of copper(II) salicylate tetrahydrate, which yields a magnetic moment per copper atom equal to about 1·44 Bohr magnetons. This subnormal magnetic moment smaller than the spin-only moment 1·73 B.M. for one unpaired electron is not surprising as it stands, because a number of copper(II) carboxylates show subnormal magnetic moments (Figgis & Martin, 1956; Kondo & Kubo, 1958; Asai, Kishita & Kubo, 1959). In fact, van Niekerk & Schoening (1953) have shown that in the crystals of copper(II) acetate monohydrate, copper atoms are bridged in pairs by four Cu–O–C–O–Cu links with the Cu–Cu distance as short as 2·64 Å permitting spin interaction between the copper atoms involved in the pair. However, a complete X-ray crystal analysis carried out by Hanic & Michalov (1960) on copper(II) salicylate tetrahydrate has indicated that planar centrosymmetric molecules $Cu(C_6H_4.OH.CO_2)_2.2H_2O$ are linked in the structure by a system of hydrogen bonds in which two remaining water molecules also take part. The shortest Cu–Cu distance is equal to 3·728 Å, which would lead to a magnetic moment of about 1·9 ± 0·1 B.M., normally observed for a majority of copper(II) salts. The present investigation has been undertaken in order to clarify this apparent discrepancy.

Copper(II) salicylate tetrahydrate was prepared in accordance with a method employed by Hanic and Michalov. The solution of copper(II) sulfate pentahydrate