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The crystal structure of N-methylurea nitrate.*

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Since a resumption of the structure analysis of methylurea nitrate may be delayed for a considerable time, this note summarizing the results of the Fourier analysis is being published. Eventually a least-squares refinement of anisotropic temperature parameters as well as positional parameters will be made to try to clearly resolve the positions of the hydrogen atoms.

Crystals of methylurea nitrate suitable for X-ray work were grown by evaporation of an aqueous solution at room temperature. The crystals are orthorhombic with the following unit cell dimensions (λ of Cu$K\alpha$ = 1·5418 Å):

$a_0 = 11{\cdot}22 \pm 0{\cdot}04$, $b_0 = 8{\cdot}12 \pm 0{\cdot}03$, $c_0 = 6{\cdot}36 \pm 0{\cdot}02$ Å.

There are four molecules per unit cell (calculated density = 1·571 g.cm.$^{-3}$). The space-group extinctions observed are: $hk0$ present in all orders; $h0l$ present only with $h+l = 2n$; and $0kl$ present only with $k = 2n$. The presence of molecular mirror planes perpendicular to the c axis was shown from a rotation photograph about this axis, on which the intensities of reflections on the even layer lines (l = 0, 2, ...) showed the same sequence and magnitude, and similarly for the odd layer lines (l = 1, 3, ...). The space group is therefore *Pbnm* with the molecules lying in the mirrors.

The intensities of the $hk0$ and $hk1$ reflections were estimated visually from multiple-film equi-inclination Weissenberg photographs. The structure was determined from these data by generalized Patterson and electron-density projections. The method used was essentially the same as that described by Curtis & Pasternak (1955). The final generalized electron-density projection is shown in Fig. 1, and the final Fourier parameters are listed in Table 1. The percentage discrepancy between the observed and calculated structure amplitudes was 12·9 for the $hk0$ reflections and 10·9 for the $hk1$ reflections. An isotropic temperature correction, $\exp[-B(\sin\theta/\lambda)^2]$ with B = 2·08 Å^2, was used with the calculated structure amplitudes.

Table 1. *Final parameters from the Fourier analysis*

Atom	x	y	z
O_1	0·1235	0·5248	0·2500
O_2	−0·0200	0·6971	0·2500
O_3	0·1587	0·7814	0·2500
O_4	−0·0492	0·3129	0·2500
N_1	0·0874	0·6668	0·2500
N_2	0·1044	0·1350	0·2500
N_3	−0·0866	0·0473	0·2500
C_1	−0·0085	0·1658	0·2500
C_2	−0·2133	0·0749	0·2500

Table 2 lists important interatomic distances and bond angles. There are three strong N–O hydrogen bonds and one strong O–O hydrogen bond in this structure. An interesting feature suggested by the Fourier projection as well as by the essentially equal dimensions of the nitrate ion is the apparent transfer of the nitric acid proton to the methylurea oxygen. It is hoped that this point will be unambiguously resolved by the least squares refinement.

Table 2. *Bond lengths and bond angles*

N_1–O_1	1·22 Å	O_1–N_1–O_2	120·9°
N_1–O_2	1·23	O_2–N_1–O_3	119·2
N_1–O_3	1·23	O_1–N_1–O_3	119·9
C_1–O_4	1·28	N_2–C_1–O_4	122·1
C_1–N_2	1·29	N_3–C_1–O_4	116·8
C_1–N_3	1·30	N_2–C_1–N_3	121·2
N_3–C_2	1·44	C_1–N_3–C_2	123·4
$O_1 \cdots O_4$	2·59	C_1–O_4–O_1	110·7
$N_2 \cdots O_3$	2·94	N_1–O_1–O_4	112·3
$N_2 \cdots O_3''$	2·91	C_1–N_2–O_3'	113·2
$N_3 \cdots O_2'$	2·94	C_1–N_2–O_3''	144·7
		C_1–N_3–O_2'	123·0

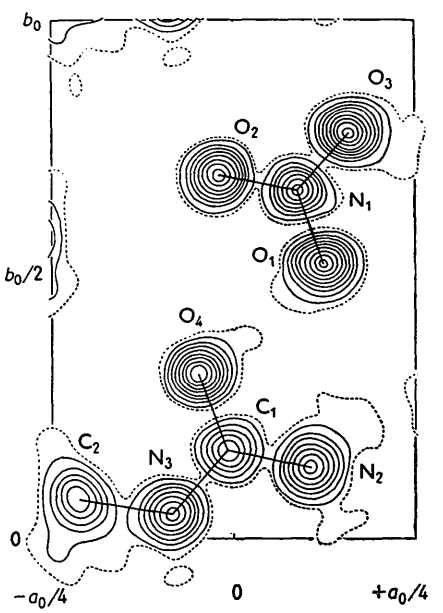

Fig. 1. Generalized electron-density projection.

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Reference

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