Table 4. *Hydrogen bonding in* (1)

D—H···A	Symmetry of A relative to D	$d(D···A)$ (Å)	$d(H···A)$ (Å)	$(D—H···A)$ (°)
N5 H5 O8'	x, 1+y, 1+z	2·643 (3)	1·78 (4)	164· (4)
N10 H10A OW	1+x, y, 1+z	2·836 (5)	1·99 (4)	168· (4)
N10 H10B O3'	x, y, 1+z	2·937 (3)	2·23 (4)	137· (4)
N10 H10B O8'	x, 1+y, 1+z	3·120 (4)	2·48 (4)	130· (3)
O2' HO2' O6'	1+x, 1+y, z	2·703 (3)	2·09 (4)	168· (5)
O3' HO3' O6'	x, 1+y, z	2·667 (3)	1·97 (4)	145· (4)
O7' HO7' O8'	x−1, y, z	2·573 (3)	1·67 (5)	175· (5)
OW HOWB O7'	x, y, z	2·874 (4)	2·040 (2)	150·1 (2)
OW HOWC O3'	x, y, z	2·782 (4)	1·816 (2)	155·1 (2)

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Structure of 1,5,6-Trimethyl-1H-pyrazolo[3,4-d]pyridazine-4,7(5H,6H)-dione

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Abstract. $C_8H_{10}N_4O_2$, $M_r = 194\cdot2$, monoclinic, $P2_1$, $a = 3\cdot997$ (1), $b = 8\cdot474$ (2), $c = 13\cdot092$ (3) Å, $\beta = 96\cdot96$ (2)°, $V = 440\cdot2$ (3) Å³, $Z = 2$, $D_m = 1\cdot45$ (5), $D_x = 1\cdot465$ g cm⁻³, $\lambda(Mo\ K\alpha) = 0\cdot71069$ Å, $\mu = 1\cdot19$ cm⁻¹, $F(000) = 204$, $T = 293$ (1) K, final $R = 0\cdot069$ for 776 observed reflexions. The two fused rings are coplanar with a dihedral angle between the ring planes of 0·8 (6)°; the bond lengths and angles agree with expected values.

Experimental. It has been reported that cycloaddition of diazomethane to 6-hydroxy-3(2H)-pyridazinone yields not only 6-methoxy-2-methyl-3(2H)-pyridazinone and 6-methoxy-3(2H)-pyridazinone, but also a third product which has been suggested to be 1,5,6-

Table 1. *Fractional coordinates* (× 10⁴) *and equivalent isotropic temperature factors* (Å² × 10³) (*Hamilton,* 1959)

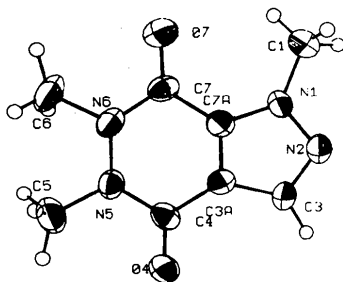|	x	y	z	U_{eq}
O(4)	8756 (13)	2667 (9)	4642 (3)	53 (2)
O(7)	1291 (13)	2670 (9)	941 (3)	57 (2)
N(1)	5159 (13)	5527 (9)	1776 (4)	38 (2)
N(2)	7102 (13)	6493 (10)	2410 (4)	45 (2)
N(5)	5388 (13)	1426 (9)	3386 (4)	43 (2)
N(6)	3396 (12)	1449 (9)	2422 (4)	43 (2)
C(1)	3620 (20)	6107 (11)	776 (6)	57 (3)
C(3)	8163 (16)	5633 (10)	3238 (5)	42 (3)
C(3a)	6821 (14)	4099 (9)	3127 (4)	34 (3)
C(4)	7145 (15)	2716 (10)	3794 (4)	39 (3)
C(5)	4894 (21)	93 (11)	4059 (6)	61 (4)
C(6)	2008 (20)	−60 (11)	2021 (6)	61 (3)
C(7)	3034 (15)	2719 (10)	1775 (4)	40 (3)
C(7a)	4966 (15)	4077	2203 (4)	37 (3)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(4)—C(4)	1·215 (7)	N(5)—C(5)	1·461 (11)
O(7)—C(7)	1·223 (7)	N(6)—C(6)	1·466 (11)
N(1)—N(2)	1·344 (9)	N(6)—C(7)	1·366 (10)
N(1)—C(1)	1·463 (9)	C(3)—C(3a)	1·407 (11)
N(1)—C(7a)	1·356 (8)	C(3a)—C(4)	1·458 (10)
N(2)—C(3)	1·332 (9)	C(3a)—C(7a)	1·340 (7)
N(5)—N(6)	1·408 (7)	C(7)—C(7a)	1·459 (8)
N(5)—C(4)	1·372 (10)		

N(2)—N(1)—C(1)	119·8 (7)	C(3)—C(3a)—C(7a)	105·6 (5)
N(2)—N(1)—C(7a)	110·8 (5)	C(4)—C(3a)—C(7a)	121·9 (6)
C(1)—N(1)—C(7a)	129·4 (6)	O(4)—C(4)—N(5)	121·2 (7)
N(1)—N(2)—C(3)	105·8 (7)	O(4)—C(4)—C(3a)	125·0 (7)
N(6)—N(5)—C(4)	123·0 (6)	N(5)—C(4)—C(3a)	113·8 (5)
N(6)—N(5)—C(5)	116·6 (6)	O(7)—C(7)—N(6)	122·2 (7)
C(4)—N(5)—C(5)	118·8 (5)	O(7)—C(7)—C(7a)	126·0 (7)
N(5)—N(6)—C(6)	117·2 (6)	N(6)—C(7)—C(7a)	111·8 (5)
N(5)—N(6)—C(7)	124·9 (6)	N(1)—C(7a)—C(3a)	107·9 (5)
C(6)—N(6)—C(7)	117·3 (5)	N(1)—C(7a)—C(7)	127·6 (5)
N(2)—C(3)—C(3a)	110·0 (6)	C(3a)—C(7a)—C(7)	124·5 (5)
C(3)—C(3a)—C(4)	132·5 (5)		

trimethyl-1H-pyrazolo[3,4-d]pyridazine-4,-7(5H,6H-dione without any firm structural proof. This same compound has been isolated by cycloaddition of diazomethane to 1,2-dimethyl-1,2-dihydro-3,6(1H,2H)-pyridazinedione (King, 1982). During our systematic studies of the cycloaddition of diazoalkanes to pyridazine derivatives we isolated, by the cycloaddition of diazomethane to 1,2-dimethyl-1,2-dihydro-3,6(1H,-2H)-pyridazinedione, two isomeric products, the major component of which was identical with the compound reported earlier (Stanovnik, Kermavnar & Tišler, 1986).
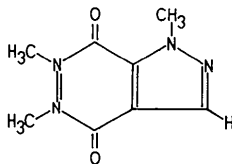
Density measured by flotation in chlorobenzene/ CCl_4; thin needles, $0·18 \times 0·22 \times 0·53$ mm; Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation (graphite monochromator), lattice parameters by least-squares fit of 45 reflexions ($8 < \theta < 13°$); $\omega/2\theta$ scans, variable scan rate (min. 1·8, max. 20·1° min^{-1}), max. scan time 90 s, scan width (2θ) $(0·9 + 0·3\tan\theta)°$, aperture $(2·5 + 0·9\tan\theta)$ mm, background measured for $\frac{1}{4}$ of scan time at each scan limit, $(\sin\theta/\lambda)_{max}$ $= 0·64$ Å^{-1}. Three reflexions monitored at intervals of

140 reflexions, intensity decline 6·0%; data corrected for intensity variation and Lp effects, but absorption ignored; 3387 ($\pm h$, $\pm k$, $\pm l$) measured reflexions, 979 unique ($R_{int} = 0·058$), 776 observed (h $-5/5$, k 0/10, l 0/16) with $I > 2\sigma(I)$, $\sigma(I)$ based on counting statistics. Structure solution by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), full-matrix least-squares refinement minimizing $\sum w(\Delta F)^2$ with *SHELX*76 (Sheldrick, 1976); final R and wR values: 0·069 and 0·083 with $w = 1·53/[\sigma^2(F_o) + 0·0017F_o^2]$; H-atom positions from a difference synthesis and included in the refinement; common isotropic temperature factor U for H atoms refined to 0·111 (5) Å^2. $\Delta\rho$ within $-0·16$ and 0·18 e Å^{-3} in final difference map. Max. value of Δ/σ at convergence 0·35 [U_{22}, C(6)]. The y coordinate of C(7a) was kept fixed. Scattering factors for O, N and C from *International Tables for X-ray Crystallography* (1974), for H from Stewart, Davidson & Simpson (1965). All calculations performed on the DEC 1091 computer at University Computer Centre, Ljubljana. *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) was used for data reduction and interpretation. The final atomic parameters are in Table 1.* Bond lengths and angles are given in Table 2. Views of the molecule with the atomic numbering and of the molecular packing are presented in Figs. 1 and 2.

Related literature. Crystal structures of similar heterocyclic systems have been reported by Stanovnik, Kupper, Tišler, Leban & Golič (1984) and Golič, Leban, Stanovnik & Tišler (1977, 1978).

Fig. 1. *ORTEP* (Johnson, 1965) view of the molecule with the atomic numbering scheme. Thermal ellipsoids are drawn at 50% probability level.

Fig. 2. View of the molecular packing.

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Structure of 1,2,4-Oxadiazolidine-3,5-dione Monohydrate

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Abstract. $C_2H_2N_2O_3.H_2O$, $M_r = 120.07$, monoclinic, $P2_1/c$, $a = 5.011$ (1), $b = 11.796$ (2), $c = 7.689$ (2) Å, $\beta = 95.22$ (2)°, $V = 452.61$ Å^3, $Z = 4$, $D_x = 1.76$, $D_m = 1.75$ g cm^{-3}, $\lambda(Cu\ K\alpha) = 1.5418$ Å, $\mu = 14.0$ cm^{-1}, $F(000) = 248$, $T = 293$ K, crystal quality was poor and the final $R = 0.107$, $wR = 0.090$ for 881 observed reflections. The compound is derived from a novel form of the monopropellant oxalohydroxamic acid. The two exocyclic C—O bond lengths of 1.240 (3) and 1.228 (4) Å indicate double bonds. The C—N bond lengths of 1.334 (4), 1.390 (4) and 1.359 (4) Å are characteristic of the amide bond. The N atom covalently bonded to the two carbonyl C atoms acts as a proton donor in an intermolecular hydrogen bond to the ring O atom: N1···O3^i = 2.854 Å (i = x − 1, y, z), H···O = 2.15 Å, N—H···O = 159°.

Experimental. During the investigation of thermal decomposition of oxalohydroxamic acid, a product was obtained as shiny crystals through sublimation. The crystal quality was poor and X-ray diffraction spots were of elongated shape in the Weissenberg photographs. It was important to establish its molecular structure by the method of single-crystal X-ray diffraction, in order to understand the nature of the thermal decomposition. The molecular weight of the compound was determined to be 102 using a mass spectrometer.

Experimental conditions for the data collection using an Enraf CAD-4 diffractometer are given in Table 1.

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Table 1. *Experimental conditions*

Crystal size	0·2 × 0·2 × 0·5 mm
Number and θ range of reflections used for lattice parameters	25, 27–54°
Scan method	$\omega/2\theta$
Data collection range	0–75° (θ)
Range of *hkl* values	h −6 to 6, k 0 to 14, l 0 to 9
Number of measured reflections	918
Number of observed reflections used in refinement $||F_o| > 3\sigma(F_o)|$	881
Number of parameters refined	77
Final R, wR	0·107, 0·090

Crystal density measured by flotation using a mixture of bromoform and chloroform. Three check reflections 112, 1$\bar{2}$1, 11$\bar{1}$ were monitored every 30 min. No decay was observed. Absorption corrections were not applied. Lorentz and polarization corrections were applied. Systematic absences $0k0$, $k = 2n + 1$ and $h0l$, $l = 2n + 1$. Structure was solved using the program *SHELX*76 (Sheldrick, 1976). The C, N and O atoms were all refined anisotropically. The H atom covalently bonded to N1 (HN1 in Fig. 1) could be located in the difference electron density map and was refined without constraints. Its isotropic temperature factor was varied. The H atom HN2 (Fig. 1) covalently bonded to N2 was fixed from stereochemical considerations and was not included either in the structure-factor calculations or in the least-squares refinement. Full-matrix least squares with *SHELX*76. $w = 1/\sigma^2(F)$. 77 parameters varied during the final cycle. Ten reflections with either asymmetric backgrounds or with $(|F_c| − |F_o|) > 3\sigma(F_o)$ and hence suspected to be affected by secondary extinction were not included in the final refinement calculations. Maximum Δ/σ was 0·04 in the