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

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**Abstract.**  $C_2H_2N_2O_3 \cdot H_2O$ ,  $M_r = 120.07$ , 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<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.76$ ,  $D_m = 1.75$  g cm<sup>-3</sup>,  $\lambda(\text{Cu K}\alpha) = 1.5418$  Å,  $\mu = 14.0$  cm<sup>-1</sup>,  $F(000) = 248$ ,  $T = 293$  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:  $N1 \cdots O3^i = 2.854$  Å ( $i = x - 1, y, z$ ),  $H \cdots O = 2.15$  Å,  $N-H \cdots O = 159^\circ$ .

**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.

Table 1. *Experimental conditions*

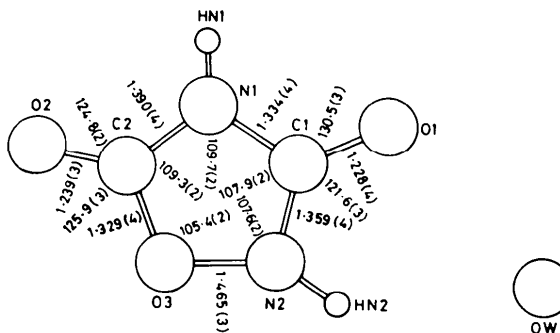
Crystal size	0.2 × 0.2 × 0.5 mm
Number and $\theta$ range of reflections used for lattice parameters	25, 27–54°
Scan method	$\omega/2\theta$
Data collection range	0–75° ( $\theta$ )
Range of $hkl$ values	$h = -6$ to 6, $k = 0$ to 14, $l = 0$ to 9
Number of measured reflections	918
Number of observed 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,  $1\bar{2}\bar{1}$ ,  $11\bar{1}$  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 *SHELX76* (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 *SHELX76*.  $w = 1/\sigma^2(F)$ . 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  $\Delta/\sigma$  was 0.04 in the

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Table 2. Atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )
$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$U_{eq}/U$
C1	0.7763 (6)	0.1259 (2)	0.6200 (4)	0.021 (1)
C2	0.8367 (6)	0.1197 (2)	0.3366 (3)	0.018 (1)
N2	1.0421 (5)	0.1397 (2)	0.6035 (3)	0.017 (1)
O3	1.0804 (5)	0.1343 (2)	0.4171 (3)	0.028 (1)
O1	0.6883 (5)	0.1225 (3)	0.7638 (3)	0.036 (1)
O2	0.7818 (4)	0.1079 (2)	0.1772 (3)	0.026 (1)
N1	0.6507 (5)	0.1165 (2)	0.4601 (3)	0.021 (1)
OW	1.2555 (5)	0.1186 (2)	-0.0115 (3)	0.032 (1)
HN1	0.507 (10)	0.108 (3)	0.433 (5)	0.010 (8)
HN2	1.186	0.151	0.701	

Fig. 1. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) in 1,2,4-oxadiazolidine-3,5-dione monohydrate.

final cycle.  $\Delta\rho$  in the final difference map was within  $+0.84$  to  $-1.43 e \text{\AA}^{-3}$ . Atomic scattering factors as supplied in *SHELX76*.

Atomic coordinates and thermal parameters are given in Table 2.\* A plot of the molecule with bond lengths and angles is shown in Fig. 1.

**Related literature.** The crystal structure of the title compound was studied as part of our program of investigations on propellant formulations (Sameena Begum, Jain, Khetrpal & Shiva Prakash, 1987). Characteristic amide bond lengths are given in Venkatesan & Ramakumar (1981) and Sutton (1965).

\* Lists of structure factors, anisotropic thermal parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44342 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of a Mycophenolic Acid Derivative (CAM)

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**Abstract.** 4-{1,3-Dihydro-5-[(*E*)-5-ethoxycarbonyl-3-methyl-2-pentenyl]-6-methoxy-7-methyl-3-oxo-4-iso-benzofuranyloxycarbonylamino}benzoic acid (CAM),  $C_{27}H_{29}NO_9$ ,  $M_r = 511.531$ , orthorhombic, *Pbca*,  $a = 19.010$  (2),  $b = 20.604$  (1),  $c = 12.917$  (1)  $\text{\AA}$ ,  $V = 5059.16$   $\text{\AA}^3$ ,  $Z = 8$ ,  $D_x = 1.343$   $\text{g cm}^{-3}$ ,  $\lambda(\text{Cu } K\alpha) = 1.5418$   $\text{\AA}$ ,  $\mu = 8.04$   $\text{cm}^{-1}$ ,  $F(000) = 2160$ ,  $T = 298$  K, final  $R = 0.036$  for 3673 unique reflections [ $F_o^2 >$

$3\sigma(F_o^2)$ ]. The *trans* zigzag chain of the ethoxycarbonyl-pentyl moiety and the benzene ring approach each other to maintain intramolecular van der Waals contacts and form a long molecular shape. The least-squares planes of these moieties are approximately perpendicular to the isobenzofuranyl ring.

**Experimental.** Colorless prisms of CAM were grown from acetone. Crystal size  $0.23 \times 0.18 \times 0.15$  mm, Enraf-Nonius CAD-4  $\kappa$ -cradle diffractometer,  $\text{Cu } K\alpha$

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