References

Buckleton, J. S. & Waters, T. N. (1984). *Acta Cryst.* **C40**, 1587–1589.

Enraf–Nonius (1981). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Karle, J. M., Cysyk, R. D. & Karle, I. L. (1980). *Acta Cryst.* **B36**, 3012–3016.

Sheldrick, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS86.* Univ. of Göttingen, Germany.

Wilson, W. R., Baguley, B. C., Wakelin, L. P. G. & Waring, M. J. (1981). *Mol. Pharmacol.* **20**, 404–414.

Acta Cryst. (1992). **C48**, 1088–1090

Structure of 1,2,4-Triazolidine-3,5-dione (Urazole) at 105 K

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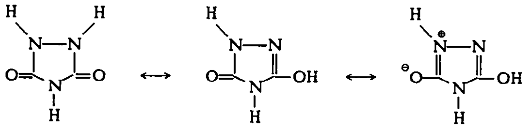
(*Received* 20 *May* 1991; *accepted* 12 *November* 1991)

Abstract. $C_2H_3N_3O_2$, $M_r = 101.06$, monoclinic, $P2_1/n$, $a = 3.462\,(1)$, $b = 9.513\,(1)$, $c = 10.995\,(2)$, $\beta = 95.06\,(2)°$, $V = 360.7\,(1)\,\text{Å}^3$, $Z = 4$, $D_x = 1.861\ \text{g cm}^{-3}$, $\lambda(\text{Mo}\ K\alpha) = 0.71069\ \text{Å}$, $\mu = 1.52\ \text{cm}^{-1}$, $F(000) = 208$, $T = 105$ K, $R = 0.0458$, $wR = 0.0359$ for 1326 unique observed reflections and 81 parameters. In the crystal the molecules exist in the diketo form. The two adjacent H atoms are in *trans* position; the N atom between the two carbonyl groups has a nearly planar geometry. The H atoms form one bifurcated and two almost linear hydrogen bonds resulting in a three-dimensional hydrogen-bonding network.

Introduction. 1,2,4-Triazolidine-3,5-dione (1) is a long-known parent compound of a large number of derivatives. Whereas the crystal structures of 11 non-fused and 95 fused 1,2,4-triazolidine-3,5-diones have already been reported in the literature, the structure of (1) is unknown, possibly because of its poor crystallization. Recently, the crystal structure of the new compound [1,2,4]triazolo[1,2-*a*][1,2,4]triazole-1,3,5,7(2*H*,6*H*)-tetrone (urazourazole) (2) was determined (Nachbaur, Faleschini, Belaj & Janoschek, 1988). The determination of the structure of the parent compound (1), for which three tautomers (1*a*–*c*) are discussed in the literature (Bourdais, Cugniet, Prin & Chabrier, 1964), was therefore desirable.



(1*a*) (1*b*) (1*c*)

Experimental. A colourless single crystal (0.16 × 0.24 × 0.44 mm), obtained by repeated recrystallization in H_2O, was used to collect data at 105 K on a modified Stoe four-circle diffractometer using graphite-monochromated Mo *Kα* radiation with Nonius low-temperature equipment. Cell parameters were determined from a least-squares fit of 40 well-refined reflections (θ range 9–15°). 2611 reflections were measured using ω scans (scan range 1.6°) for 2θ from 3 to 80° (h 0 to 6, k 0 to 17, l −19 to 19), 1589 reflections had $I > 2.5\sigma(I)$, 1334 were unique. Three standard reflections measured every 100 reflections showed ± 1.3% variation in intensity. Data were corrected for Lp effects, an empirical absorption correction (maximum/minimum transmission factors 1.05/0.89) was applied with *DIFABS* (Walker & Stuart, 1983), and eight low-order reflections were omitted, probably suffering from extinction. The structure solution was by direct methods using *SHELXS86* (Sheldrick, 1986). Refinement (on F) with anisotropic thermal parameters for all non-H atoms, with individual isotropic temperature factors for the H atoms, N—H bond lengths constrained to 1.02 Å [the mean value obtained from neutron data (Olovsson & Jönsson, 1976)], and refinement of five interlayer scale factors, defined by the index h, until no parameter shifts occurred (maximum $\Delta/\sigma = 0.003$), gave $R = 0.046$, $wR = 0.036$ [$w = 1/\sigma^2(F)$, 1326 reflections, 81 parameters]. The final difference Fourier map maximum and minimum peaks were 0.47 and $-0.38\ \text{e Å}^{-3}$, respectively. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (Ibers & Hamilton, 1974). VAX 6000 computer, *SHELX76* (Sheldrick, 1976), *PLATON* (Spek, 1982) and *ORTEP* (Johnson, 1965) programs.

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Discussion. The final atomic positional and thermal parameters are listed in Table 1,* and the bond lengths and angles are given in Fig. 1. The packing of the molecules in the unit cell, with the atoms represented by thermal ellipsoids, is shown in Fig. 2.

The structure analysis showed undoubtedly that, in the crystal, compound (1) exists in the neutral diketo form (1*a*). Whereas N(1) and N(2) are pyramidal, N(4) has an almost planar geometry caused by the two adjacent carbonyl groups. The two H atoms H(1) and H(2) are in *trans* position with respect to the 'least-squares' ring plane. Therefore C_s symmetry must be excluded for (1) and C_2 symmetry is to be expected. In the crystal, (1) does not show C_2 symmetry: the puckering amplitude Q and the phase angle φ of the five-membered ring as defined by Cremer & Pople (1975) are 0.084 (1) Å and 8.0 (8)°, respectively. Thus, the conformation of the five-membered ring is neither an envelope (C_s symmetry, $\varphi = 0°$) nor a twist (C_2 symmetry, $\varphi = 18°$) conformation.

A search in the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) yielded 12 urazole fragments in 11 non-fused urazole derivatives: two are almost planar with $Q < 0.015$ Å, six show a twist conformation ($15.3 < \varphi < 17.4°$) and four fragments have a conformation between envelope and twist ($7.6 < \varphi < 10.7°$) like (1); the maximum puckering amplitude Q was 0.104 Å ($\overline{Q} = 0.058$ Å). In the 95 fused urazole derivatives (113 fragments) found in the database, the φ values are evenly distributed, and the puckering amplitudes somewhat larger ($\overline{Q} = 0.086$ Å). In urazourazole (2), the two five-membered rings show completely different conformations: $\varphi = 1.4$ (8) and 17.0 (8)°, $Q = 0.053$ (1) and 0.069 (1) Å, respectively. Therefore, the ring puckering in urazole derivatives seems to be strongly affected by intermolecular interactions.

Each molecule has eight hydrogen bonds to six neighbouring molecules giving a three-dimensional hydrogen-bonding network (Fig. 2). About the two doubly connected neighbouring molecules, the hydrogen bonds are arranged around symmetry centres. Each O atom forms two hydrogen bonds, directed to the 'lone pairs', so that they have a nearly planar environment. Because of the donor–acceptor ratio of 3:4, there is one bifurcated hydrogen bond [from H(2)] with evidently longer H···O distances compared to the two other hydrogen bonds [from H(1) and H(4)] showing almost linear N—H···O geometries (Fig. 1). The strength of the hydrogen

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N(1)	4076 (3)	7733 (1)	1977 (1)	97 (4)
N(2)	5150 (3)	6683 (1)	1167 (1)	93 (4)
C(3)	3630 (3)	7043 (1)	19 (1)	86 (4)
N(4)	2173 (3)	8384 (1)	107 (1)	90 (4)
C(5)	2496 (4)	8818 (1)	1309 (1)	87 (4)
O(3)	3607 (3)	6342 (1)	−924 (1)	128 (4)
O(5)	1499 (3)	9973 (1)	1694 (1)	125 (4)
H(1)	5825 (38)	7965 (14)	2746 (8)	313 (47)
H(2)	5273 (51)	5682 (6)	1499 (13)	396 (49)
H(4)	897 (43)	8937 (14)	−615 (9)	408 (51)

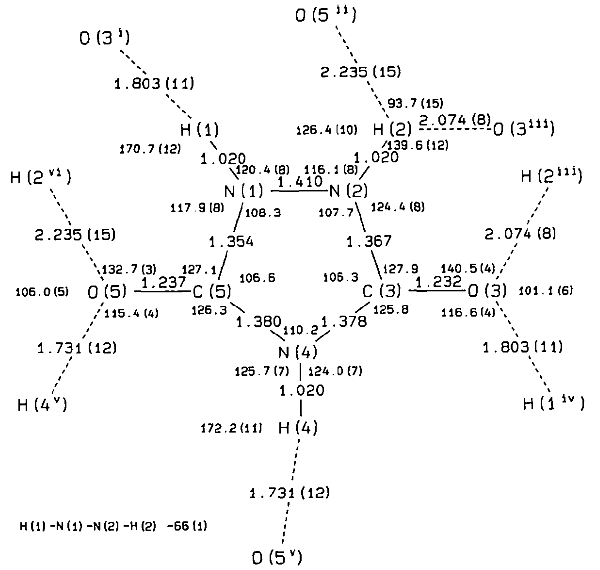

Fig. 1. Bond distances (Å) and bond angles (°). The standard deviations are 0.001 Å and 0.1°, if not given. Symmetry code: (i) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$; (ii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $1-x, 1-y, -z$; (iv) $x-\frac{1}{2}, \frac{3}{2}-y, z-\frac{1}{2}$; (v) $-x, 2-y, -z$; (vi) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

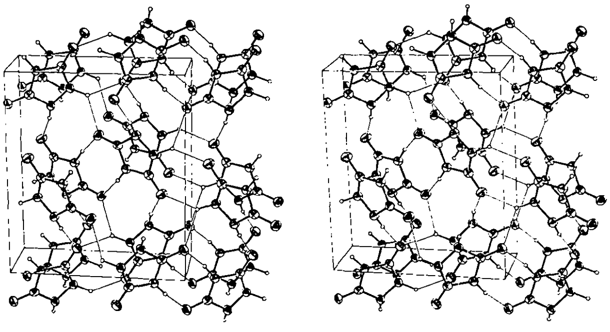

Fig. 2. *ORTEP* (Johnson, 1965) plot showing the hydrogen-bonding network. The thermal ellipsoids are drawn at the 90% probability level.

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* Lists of anisotropic thermal parameters, structure factors and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54855 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

bonds corresponds with the $H\cdots O$ distances as well as with the $N\cdots O$ distances [$N(4)\cdots O(5^{v})$ 2.745 (1) > $N(1)\cdots O(3^{i})$ 2.815 (1) > $N(2)\cdots O(3^{iii})$ 2.926 (1) > $N(2)\cdots O(5^{ii})$ 2.957 (1) Å].

The author thanks Dr Ch. Kratky, Institute of Physical Chemistry, University of Graz, for the use of the diffractometer.

References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
Bourdais, J., Cugniet, F., Prin, J.-C. & Chabrier, P. (1964). *Bull. Soc. Chim. Fr.* pp. 500–507.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Ibers, J. A. & Hamilton, W. C. (1974). Editors. *International Tables for X-ray Crystallography*, Vol. IV, pp. 99, 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Nachbaur, E., Faleschini, G., Belaj, F. & Janoschek, R. (1988). *Angew. Chem.* **100**, 703–704; *Angew. Chem. Int. Ed. Engl.* **27**, 701–703.
Olovsson, I. & Jönsson, P.-G. (1976). *The Hydrogen Bond*, Vol. 2, edited by P. Schuster, G. Zundel & C. Sandorfy, pp. 440–452. Amsterdam, New York, Oxford: North-Holland.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Sheldrick, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
Spek, A. L. (1982). *Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1992). C**48**, 1090–1092

Structure of Prudomestin

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(*Received* 2 *September* 1991; *accepted* 20 *November* 1991)

Abstract. 3,5,7-Trihydroxy-8-methoxy-2-(4-methoxyphenyl)-4*H*-1-benzopyran-4-one, $C_{17}H_{14}O_7$, M_r = 330.3, triclinic, $P\bar{1}$, a = 6.579 (5), b = 8.441 (7), c = 13.893 (14) Å, α = 101.92 (9), β = 103.38 (6), γ = 97.44 (6)°, V = 721 Å^3, Z = 2, D_x = 1.52 g cm^{-3}, Mo $K\alpha$ radiation, λ = 0.71069 Å, μ = 1.2 cm^{-1}, $F(000)$ = 344, T = 200 K, R = 0.076 for 1534 unique observed [$I/\sigma(I) \geq 2.0$] reflections. The definitive structure of prudomestin is established by this investigation. The molecule is essentially planar with the methyl groups projecting above and below the plane. The major intermolecular force within a layer of molecules in the solid state results from hydrogen bonding.

Introduction. *Prunus domestica Linn.* is the tree of the well-known fruit plum and grows ideally in the western temperate Himalayas and is cultivated for its fruit in India. Its fruits are used as a mild laxative and demulcent and, in combination with other drugs, they are used in leucorrhoea, irregular menstruation and debility following miscarriage (Chopra, Nayar & Chopra, 1956). The prudomestin used in this study was extracted from the wood of *Prunus domestica Linn.* The objective of this investigation was to provide an unambiguous identification of the structure of prudomestin.

Experimental. The prudomestin was isolated from the hot-water soluble portion of the benzene solubles of an alcoholic extract from the heartwood of *Prunus domestica Linn.* collected from Baramulla, Kashmir, India. The compound was recrystallized from chloroform–petrol and separated as yellow needles.

Data were collected with a Siemens P3R3 four-circle diffractometer in ω–2θ mode. The crystal was held at 200 K with an Oxford Cryosystems Cryostream Cooler (version 2.4); this temperature was chosen because the crystal diffracted rather poorly under ambient conditions. Maximum 2θ was 50° with scan range ± 0.7° (2θ) around the $K\alpha_1 - K\alpha_2$ angles, scan speed 3–29° min^{-1}, depending on the intensity of the 2 s prescan; backgrounds were measured at each end of the scan for 0.25 of the scan time. *hkl* ranges were 0/7, −9/10, −15/15 (unique reflections only). Three standard reflections were

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