

Table 4. Distances of atoms from mean planes (Å)
(e.s.d.'s 0.0006–0.0011 Å)

The planes are defined by the starred atoms.

Plane (1)	C1	C2*	C3*	C4*	C5*	C10	C16
	0.455	-0.006	0.011	-0.011	0.006	-0.086	-0.110
Plane (2)	C3	C10*	C11*	C12*	C13*	C14*	C15*
	-0.034	-0.006	0.006	0	-0.006	0.007	-0.001
Plane (3)	C4	C16*	C17*	C18*	C19*	C20*	C21*
	-0.036	0.003	-0.003	0	0.003	-0.003	0

Dihedral angles (°) between normals to planes (e.s.d.'s 0.5–0.7°)

Plane (1)/plane (2)	107.6
Plane (1)/plane (3)	123.2
Plane (2)/plane (3)	66.6

consistent with that determined by electron diffraction in cyclopentene [$29.0 \pm 2.5^\circ$] (Davis & Muecke, 1970).

The dihedral angles between the normals to the planes, listed in Table 4, show that both phenyl rings are out of the plane of the C=C bond. Fig. 1 shows the molecule and gives the atomic numbering scheme. Fig. 2 shows the molecular packing.

Acta Cryst. (1989). **C45**, 780–782

Structure of 2-Imidazolidinone Hemihydrate

BY MOSHE KAPON AND GEORGE M. REISNER

Department of Chemistry, Technion-Israel Institute of Technology, Haifa 32000, Israel

(Received 14 July 1988; accepted 14 November 1988)

Abstract. C₃H₆N₂O.½H₂O, $M_r = 95.103$, tetragonal, $P4_2/c$ (No. 114), $a = 9.686$ (3), $c = 10.034$ (3) Å, $V = 941.4$ Å³, $Z = 8$, $D_x = 1.342$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.100$ mm⁻¹, $F(000) = 408$, room temperature, $R = 0.049$, $wR = 0.053$ for 419 observed reflections [$F_o \geq 3\sigma(F_o)$]. The 2-imidazolidinone (ethyleneurea) molecules are located on general positions with the water molecules situated on twofold axes along c at $(0, \frac{1}{2}, z)$. The five-membered imidazolidine ring assumes an envelope conformation. The structure is characterized by extensive hydrogen bonding. Each C₃H₆N₂O molecule is hydrogen bonded to two adjacent molecules and this arrangement leads to the formation of tetramers. Each water of crystallization is hydrogen bonded to four ethyleneurea molecules each belonging to a different tetramer.

Introduction. The crystal structures of several ethyleneurea complexes with cadmium, mercury and copper have been described (Brown, Pierce & Trefonas, 1972; Majeste & Trefonas, 1972, 1974). A careful search of the Cambridge Structural Database revealed that the

We gratefully acknowledge the award of an SERC research grant to JAM.

References

- ANTEUNIS, M. & DANNEEL, D. (1975). *Org. Magn. Reson.* **7**, 345–348.
- AVRAM, M., CONSTANTINESCU, D., DINULESCU, G. & NENITZESCU, C. D. (1969). *Tetrahedron Lett.* **59**, 5215–5218.
- BERNSTEIN, J. (1975). *Acta Cryst.* **B31**, 418–422.
- CARGILL, R. L., JACKSON, T. E., PECT, N. P. & POND, D. M. (1974). *Acc. Chem. Res.* pp. 106–113.
- DAVIS, M. I. & MUECKE, T. W. (1970). *J. Phys. Chem.* **74**, 1104–1108.
- HUNTER, G., MILLER, J. A., MOORE, M. & ULLAH, G. M. (1983). *Org. Magn. Reson.* **21**, 275–278.
- KOPTYUG, V. A., SHELDER, I. A., ISNEV, I. S., VASIL, L. V. & REZVUKHIN, A. I. (1971). *Zh. Org. Khim.* **6**, 1089–1096.
- MATSUI, T., THOMAS, J. A. & WHITE, J. D. (1981). *J. Org. Chem.* **46**, 3376–3378.
- MILLER, J. A. & ULLAH, G. M. (1982). *J. Chem. Soc. Chem. Commun.* pp. 874–875.
- MOLANDER, G. A. & TROST, B. M. (1981). *J. Am. Chem. Soc.* **103**, 5969–5972.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

structure of the uncomplexed ethyleneurea molecule was not known. We now report the structure of ethyleneurea hemihydrate.

Experimental. Crystals in the form of transparent spheres, ~0.3 mm in diameter. Intensity data collected on a Philips PW 1100 four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed $2^\circ \omega \text{ min}^{-1}$, scan width $1.20^\circ \omega$, $5 \leq 2\theta \leq 50^\circ$, 994 unique reflections ($h0 \rightarrow 11$, $k0 \rightarrow 11$, $l0 \rightarrow 11$) of which 424 with $F_o \geq 3\sigma(F_o)$. Cell parameters by least squares from 25 reflections with $5.8 \leq 2\theta \leq 25.3^\circ$. No significant intensity variation for 3 standards (002, 112, 040) measured every 2 h. Corrections for Lorentz and polarization applied. Structure solved by *SHELXS86* (Sheldrick, 1986) and refinement performed using the *SHELX77* system of programs (Sheldrick, 1977). Full-matrix least squares, 87 parameters refined (scale factor, atomic coordinates, anisotropic temperature factors for non-hydrogen atoms and isotropic temperature factors for hydrogens). Function minimized $\sum w(|F_o| - |F_c|)^2$.

Final $R = 0.049$, $wR = 0.053$, weighting scheme $a/[\sigma^2(F_o) + b(F_o)^2]$ with $a = 1.8944$ and $b = 0.001496$, $(\Delta/\sigma)_{\max} = 0.12$ for non-H parameters, 0.26 for H. Max. and min. height in final $\Delta\rho$ map -0.35 and $+0.164 e \text{ \AA}^{-3}$.

Discussion. Final atomic coordinates are listed in Table 1.* Fig. 1 shows the atomic nomenclature and a packing diagram is shown in Fig. 2. The ethyleneurea

* Anisotropic temperature factors for the non-hydrogen atoms, details of the least-squares plane and a list of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51615 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic coordinates ($\times 10^4$ for the non-hydrogen atoms and $\times 10^3$ for the hydrogens)

U_{eq} values are ($\text{\AA}^2 \times 10^3$) for the non-hydrogen atoms, U_{iso} values are ($\text{\AA}^2 \times 10^3$).

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* (\mathbf{a}_i \cdot \mathbf{a}_j).$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}/U_{iso}
C(1)	7999 (4)	828 (4)	1828	42 (1)
C(2)	8443 (6)	1768 (5)	3844 (4)	59 (1)
C(3)	9537 (5)	2249 (5)	2896 (5)	54 (1)
O(1)	7422 (3)	187 (4)	885 (3)	63 (1)
N(1)	7690 (4)	754 (4)	3081 (3)	54 (1)
N(2)	9046 (4)	1706 (5)	1646 (3)	61 (1)
OW(1)	10000	5000	535 (4)	55 (1)
H(21)	778 (7)	257 (6)	405 (7)	11 (2)
H(22)	874 (6)	137 (6)	464 (5)	9 (2)
H(31)	1037 (6)	181 (5)	312 (6)	8 (2)
H(32)	957 (4)	332 (4)	293 (4)	6 (1)
HN(1)	693 (5)	40 (5)	330 (5)	5 (2)
HN(2)	948 (4)	175 (5)	104 (5)	4 (1)
HW(1)	1008 (6)	429 (6)	4 (4)	8 (2)

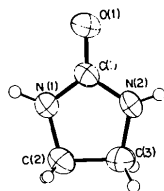


Fig. 1. Perspective view of the ethyleneurea molecule showing atomic nomenclature and 50% probability ellipsoids (ORTEP; Johnson, 1965).

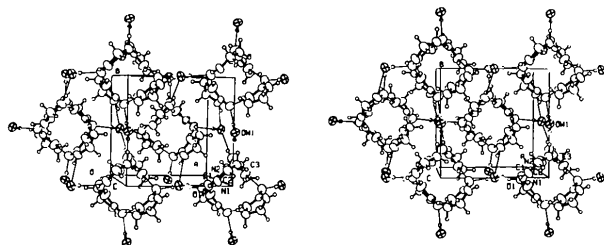


Fig. 2. ORTEP (Johnson, 1965) stereodiagram of the crystal structure. The thermal-motion ellipsoids represent 50% probability distributions. Numbering is shown for the 55601 unit, the reference (55501) unit coordinates being those listed in Table 1. The hydrogen bonds are shown by thin bonds.

Table 2. Molecular geometry of 2-imidazolidinone hemihydrate

(a) Bond lengths (\AA)

C(1)—O(1)	1.262 (4)	C(2)—H(21)	1.03 (8)
C(1)—N(1)	1.295 (4)	C(2)—H(22)	0.93 (5)
N(1)—C(2)	1.442 (5)	C(3)—H(31)	0.94 (6)
C(2)—C(3)	1.498 (7)	C(3)—H(32)	1.04 (4)
C(3)—N(2)	1.441 (6)	N(2)—HN(2)	0.74 (5)
C(1)—N(2)	1.336 (6)	OW(1)—HW(1)	0.85 (5)
N(1)—HN(1)	0.84 (5)		

(b) Valence angles ($^\circ$)

N(1)—C(1)—O(1)	126.8 (4)	N(1)—C(2)—C(3)	103.4 (3)
N(2)—C(1)—O(1)	123.2 (2)	C(2)—C(3)—N(2)	101.9 (3)
N(1)—C(1)—N(2)	110.1 (3)	C(3)—N(2)—C(1)	111.4 (3)
C(1)—N(1)—C(2)	111.2 (4)		

(c) Hydrogen bonding scheme. The target atoms are denoted by ORTEP notation (Johnson, 1965), i.e. the reference moiety (coordinates listed in Table 1) is denoted 55501, translations along the crystal axes being specified by addition or subtraction of integers from the reference code. The sequence of symmetry elements (denoted by suffixes 01, 02 etc.) is that of *International Tables for X-ray Crystallography* (1952). Distances are in \AA and angles in deg.

Donor atom (D)	Acceptor atom (A)	D—H	D...A	H...A	D...H...A
OW(1)—HW(1)...	O(1)(65504)	0.85 (5)	2.750 (4)	1.90 (5)	171 (5)
N(1)—HN(1)...	OW(1)(64505)	0.84 (5)	3.042 (4)	2.24 (5)	158 (5)
N(2)—HN(2)...	O(1)(66503)	0.74 (5)	2.896 (5)	2.20 (5)	156 (4)

molecules are located on general positions with the waters of crystallization situated on twofold axes along c at $0, \frac{1}{2}, z$.

The five-membered imidazolidine ring assumes an envelope conformation. Atoms C(1), C(3), N(1) and N(2) are in the same plane (max. deviation 0.01 \AA) with C(2) situated 0.22 \AA from this plane. The O atom deviates by only 0.04 \AA from this plane.*

The structure is characterized by extensive hydrogen bonding (Table 2c). Each ethyleneurea molecule is hydrogen bonded to two adjacent molecules *via* the carbonyl O and N(2) [O(1)...N(2) = $2.896 (5) \text{ \AA}$]. This arrangement leads to the formation of tetrameric units of mutually hydrogen bonded ethyleneurea molecules around the $\bar{4}$ axes. Adjacent tetramers, translated along c , interact only by van der Waals contacts between the methylene groups, but the tetramers translated along a and b are linked *via* the water molecules. Thus, each water molecule is involved in four hydrogen bonds (two of which are independent) to four ethyleneurea molecules of four different tetramers. The coordination sphere around the water molecule is a distorted tetrahedron with four independent angles of $117.6 (3)$, $119.9 (3)$, $88.6 (3)$ and $125.7 (3)^\circ$.

The geometry of the ethyleneurea molecule is given in Table 2. The value of the carbonyl bond length [$1.262 (4) \text{ \AA}$] is close to that of $1.270 (7) \text{ \AA}$ reported for urea where the O atom is similarly involved in two acceptor hydrogen bonds of 2.985 \AA (Caron & Donohue, 1969).

* See deposition footnote.

The two C(carbonyl)—N bonds are significantly different [1.295 (4) and 1.336 (6) Å]. We ascribe this to the different environments around the two N atoms. Also, there is an asymmetric bend of the carbonyl O towards one of the nitrogens resulting in an N—C(1)—O angle of 126.8 (4)° for one side and 123.2 (2)° for the other. A similar asymmetry has also been reported for the pentakis(2-imidazolidinone)copper(II) perchlorate complex where the corresponding angles are 127.6 (6) and 124.7 (7)° (Majeste & Trefonas, 1974). The remaining C—N distances [1.442 (5) and 1.441 (6) Å] and C—C distance [1.498 (7) Å] are also similar to those reported for the copper(II) complex, where average distances of 1.43 and 1.50 Å, respectively, were reported.

Acta Cryst. (1989). **C45**, 782–785

Structure of 3-Benzyl-4-hydroxy-5-phenyl-1,2,4-triazole

BY ABDELGHANI BEL HADJ AMOR AND AHMED KALLEL

Faculté des Sciences de Tunis, Campus Belvédère, 1060 Tunis, Tunisia

AND HELMUT PAULUS

Abteilung für Strukturforschung, Technische Hochschule, D-6101 Darmstadt, Federal Republic of Germany

(Received 5 May 1988; accepted 1 August 1988)

Abstract. C₁₅H₁₃N₃O, *M_r* = 251.29, monoclinic, C2/*c*, *a* = 20.065 (3), *b* = 5.291 (1), *c* = 24.816 (5) Å, β = 101.83 (1)°, *V* = 2578.6 (1.5) Å³, *Z* = 8, *D_x* = 1.29 Mg m⁻³, λ(Cu Kα) = 1.5418 Å, μ = 0.64 mm⁻¹, *F*(000) = 1056, *T* = 300 K, final *wR*(*F*) = 0.030 for 1247 observed reflections. The structure consists of an intermediate form between an *N*-hydroxytriazole and a triazole *N*-oxide. The molecules are connected into chains *via* strong hydrogen bonds between pairs of 4-hydroxy O atoms and 2-nitrogens, respectively. The chains thus formed run along [110] in *z* = 0 and along [110] in *z* = ½.

Introduction. The title compound is obtained by the reaction of hydrazides and hydroximates in methanol (Bel Hadj Amor & Baccar, 1988). As illustrated schematically in Fig. 1, a first reaction step leads to *N*³-hydroxy-*N*¹-acylamidrazones which can then react to produce either an oxatriazine *A* or an *N*-hydroxytriazole *B*.

IR and ¹H NMR spectra and elemental analyses confirm the empirical formula of these heterocycles but cannot distinguish between the two isomers *A* and *B*.

References

- BROWN, J. N., PIERCE, A. G. JR & TREFONAS, L. M. (1972). *Inorg. Chem.* **11**, 1830–1833.
 CARON, A & DONOHUE, J. (1969). *Acta Cryst.* **B25**, 404.
International Tables for X-ray Crystallography (1952). Vol. I. Birmingham: Kynoch Press.
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
 MAJESTE, R. J. & TREFONAS, L. M. (1972). *Inorg. Chem.* **11**, 1834–1836.
 MAJESTE, R. J. & TREFONAS, L. M. (1974). *Inorg. Chem.* **13**, 1062–1065.
 SHELDRICK, G. M. (1977). *SHELX77*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRICK, G. M. (1986). *SHELXS86*. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

Therefore an X-ray study was undertaken on one compound obtained from this reaction.

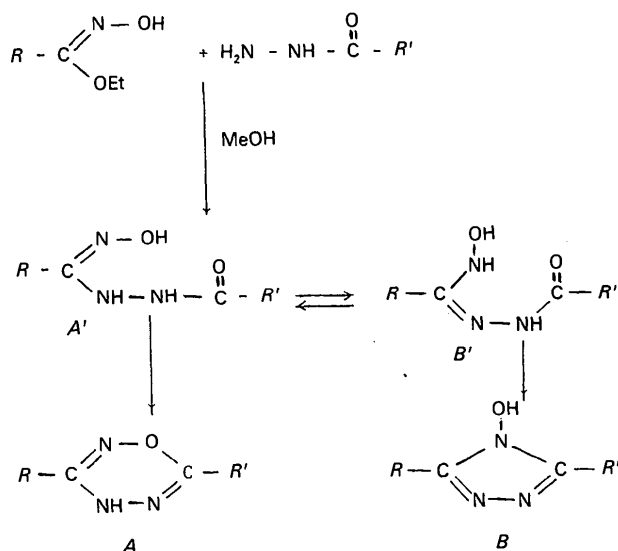


Fig. 1. Mechanism of action of hydrazides on hydroximates.