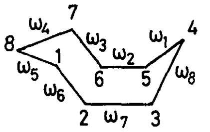
Table 3. *Torsion angles (°) for eight-membered rings*



	ω_4 ω_5	ω_3 ω_6	ω_2 ω_7	ω_1 ω_8	ΔBC (°)*
(I)	65·2	−112·3	64·2	50·4	18·7
	−70·2	98·8	−30·3	−72·7	
(II)	68·0	−69·9	0·7	86·6	26·1
	−80·5	−107·3	−47·4	−54·0	
(IV)	69·2	107·5	47·5	62·8	2·6
	−67·3	105·0	−50·1	−59·6	

* $\Delta BC = (|\omega_1 + \omega_8| + |\omega_2 + \omega_7| + |\omega_3 + \omega_6| + |\omega_4 + \omega_5|)/4$.

rings take envelope conformations, out-of-plane atoms being C(1) in (I) and C(8) in (II). The furan ring in (II) is essentially planar with deviations less than 0·005 Å for each C atom from the least-squares plane.

References

BEGLEY, M. J., MELLOR, M. & PATTENDEN, G. (1983). *J. Chem. Soc. Perkin Trans.* 1, pp. 1905–1912.

ENGLER, E. M., ANDOSE, J. D. & SCHLEYER, P. VON R. (1973). *J. Am. Chem. Soc.* **95**, 8005–8025.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

HENDRICKSON, J. B. (1967). *J. Am. Chem. Soc.* **89**, 7036–7043.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

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

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MILLER, W. R. & MCPHAIL, A. T. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 1527–1531.

SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–77.

UMEHARA, M., TAKAYANAGI, H., OGURA, H. & HISHIDA, S. (1978). *Bull. Chem. Soc. Jpn*, **51**, 3277–3281.

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Structure of 3,4-Diphenyl-1,2,4-oxadiazol-5-one

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Abstract. $C_{14}H_{10}N_2O_2$, $M_r = 238{\cdot}24$, monoclinic, $P2_1/a$, $a = 13{\cdot}064(3)$, $b = 5{\cdot}903(2)$, $c = 15{\cdot}341(3)$ Å, $\beta = 102{\cdot}22(2)°$, $V = 1156{\cdot}2(8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}37$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}0876$ mm^{-1}, $F(000) = 496$, $T = 296$ K, final $R(F) = 0{\cdot}032$ for 1020 observed unique reflections. The structure contains a planar oxadiazole heterocycle showing aromatic character and two phenyl rings twisted 35 and 66° from the heterocycle plane.

Introduction. The 1,2,4-oxadiazol-5-one derivatives have many biological and pharmaceutical properties (Breuer, 1976). The title compound was synthesized from the action of isocyanates on hydroximates (Bel Hadj Amor & Baccar, 1986). It was characterized by IR and ^{1}H NMR spectroscopy and elemental analysis. Many conformations are possible for the heterocycle, so we have undertaken a study by X-ray diffraction to determine the crystal structure and the exact geometry of the molecule.

Experimental. Prismatic transparent crystals were obtained by slow evaporation from a solution of the material in methanol. A sample *ca* 0·60 × 0·40 × 0·20 mm was chosen for X-ray diffraction. Data were collected on a CAD-4 diffractometer, with graphite-monochromated Mo $K\alpha$ radiation and $\omega/2\theta$ scan. Cell parameters from setting angles of 23 reflections with $9{\cdot}5 < \theta < 12°$. Hemisphere up to $2\theta = 46°$, $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}55$ Å^{-1}, range of hkl: h:0→14, k: ±6, l: ±16. Two standard reflections measured every 100 reflections remained stable. Total number of reflections 3260. Averaging of equivalent reflections ($R_{\rm int} = 0{\cdot}039$) resulted in 1615 independent reflections for structure determination. Only 1020 with $I > 3\sigma(I)$ were used for structure refinement. The intensities were corrected for Lorentz–polarization effects but for neither absorption nor extinction. Structure determination by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and anisotropic refinement on F by *SHELX*76 (Sheldrick, 1976). All H atoms were found

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Table 1. *Atomic fractional coordinates and thermal parameters* (Å² × 10⁴)

$U_{eq} = \frac{1}{3}$ Trace **U** for C, N and O atoms; anisotropic thermal parameters U_{ij} from the expression

$$\exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)\right].$$

H atoms are refined with isotropic U values.

	x	y	z	U_{eq}/U_{iso}
N(1)	0·9224 (2)	0·2259 (4)	0·6853 (1)	571 (12)
O(1)	0·9608 (1)	0·0403 (3)	0·7434 (1)	612 (11)
C(2)	0·8949 (2)	0·0018 (4)	0·7979 (2)	524 (15)
N(2)	0·8168 (1)	0·1582 (3)	0·7776 (1)	471 (11)
C(1)	0·8375 (2)	0·2884 (3)	0·7096 (1)	445 (12)
O(2)	0·9066 (1)	−0·1463 (3)	0·8527 (1)	753 (13)
C(21)	0·7704 (2)	0·4682 (4)	0·6624 (1)	447 (13)
C(22)	0·8175 (2)	0·6508 (4)	0·6288 (2)	560 (15)
C(23)	0·7552 (3)	0·8135 (4)	0·5791 (2)	668 (18)
C(24)	0·6490 (3)	0·7977 (5)	0·5638 (2)	688 (20)
C(25)	0·6020 (2)	0·6185 (5)	0·5972 (2)	640 (17)
C(26)	0·6632 (2)	0·4528 (4)	0·6466 (2)	534 (16)
C(31)	0·7382 (2)	0·1866 (3)	0·8294 (1)	422 (13)
C(32)	0·7394 (2)	0·3783 (4)	0·8814 (2)	496 (14)
C(33)	0·6670 (2)	0·3990 (5)	0·9336 (2)	597 (16)
C(34)	0·5944 (2)	0·2316 (5)	0·9346 (2)	642 (17)
C(35)	0·5936 (2)	0·0419 (5)	0·8825 (2)	622 (17)
C(36)	0·6654 (2)	0·0188 (4)	0·8293 (2)	528 (15)
H(22)	0·898 (2)	0·657 (4)	0·644 (2)	705 (25)
H(23)	0·789 (2)	0·927 (5)	0·553 (3)	705 (25)
H(24)	0·605 (2)	0·900 (5)	0·527 (2)	705 (25)
H(25)	0·524 (2)	0·606 (4)	0·585 (2)	705 (25)
H(26)	0·631 (2)	0·339 (4)	0·669 (2)	705 (25)
H(32)	0·794 (2)	0·492 (5)	0·883 (2)	705 (25)
H(33)	0·665 (2)	0·539 (5)	0·974 (2)	705 (25)
H(34)	0·544 (2)	0·253 (4)	0·972 (2)	705 (25)
H(35)	0·544 (2)	−0·068 (5)	0·881 (2)	705 (25)
H(36)	0·663 (2)	−0·109 (5)	0·790 (2)	705 (25)

in the difference Fourier map and refined with the same overall isotropic thermal parameter. Weighting scheme $w(F) = 1{\cdot}43/\sigma^2(F)$. Final difference map peaks $<0{\cdot}3$ e Å^{-3}, $(\Delta/\sigma)_{max} = 0{\cdot}1$, $R(F) = 0{\cdot}032$, $wR = 0{\cdot}042$ and $S = 0{\cdot}76$. Scattering factors, f' and f'' from *International Tables for X-ray Crystallography* (1974). Calculations on a VAX 11/780 of the Khawarezmi Computer Centre of Tunis.

Discussion. The atomic coordinates are reported in Table 1; bond lengths and angles in Table 2.* A projection of the molecule with thermal ellipsoids of vibration is shown in Fig. 1 and the packing of the molecules in the unit cell is presented in Fig. 2.

In the heterocycle, the C(1)–N(1) bond length [1·295 (3) Å] is typical for a C=N double bond and is very close to values observed in conjugated dienes (Ruiz-Valero, Gutiérrez-Puebla & Monge, 1985) and in aromatic heterocycles (Bois, Philoche-Levisalles & Chihaoui, 1983). C(1)–N(2) and C(2)–N(2)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–N(1)	1·295 (3)	C(23)–C(24)	1·361 (4)
N(1)–O(1)	1·434 (3)	C(24)–C(25)	1·375 (4)
O(1)–C(2)	1·340 (3)	C(25)–C(26)	1·383 (3)
C(2)–N(2)	1·363 (3)	C(26)–C(21)	1·373 (3)
N(2)–C(1)	1·369 (3)	C(31)–C(32)	1·382 (3)
C(2)–O(2)	1·199 (3)	C(32)–C(33)	1·369 (4)
N(2)–C(31)	1·435 (3)	C(33)–C(34)	1·372 (4)
C(1)–C(21)	1·466 (3)	C(34)–C(35)	1·375 (4)
C(21)–C(22)	1·394 (3)	C(35)–C(36)	1·374 (4)
C(22)–C(23)	1·380 (4)	C(36)–C(31)	1·374 (3)
C(1)–N(1)–O(1)	104·3 (2)	C(21)–C(22)–C(23)	119·2 (3)
N(1)–O(1)–C(2)	109·4 (2)	C(22)–C(23)–C(24)	120·6 (3)
O(1)–C(2)–N(2)	106·9 (2)	C(23)–C(24)–C(25)	120·5 (3)
C(2)–N(2)–C(1)	107·2 (2)	C(24)–C(25)–C(26)	119·8 (3)
N(2)–C(1)–N(1)	112·2 (2)	C(25)–C(26)–C(21)	119·9 (2)
O(1)–C(2)–O(2)	123·8 (2)	C(26)–C(21)–C(22)	120·0 (2)
O(2)–C(2)–N(2)	129·3 (2)	C(31)–C(32)–C(33)	119·1 (2)
C(2)–N(2)–C(31)	122·8 (2)	C(32)–C(33)–C(34)	120·5 (3)
N(2)–C(1)–C(21)	126·0 (2)	C(34)–C(35)–C(36)	120·3 (3)
N(1)–C(1)–C(21)	121·6 (2)	C(35)–C(36)–C(31)	119·1 (2)
N(2)–C(31)–C(36)	119·4 (2)	C(36)–C(31)–C(32)	121·0 (2)
N(2)–C(31)–C(32)	119·5 (2)	C(1)–C(21)–C(22)	118·5 (2)
C(1)–C(21)–C(26)	121·4 (2)	C(33)–C(34)–C(35)	120·0 (2)
C(1)–N(2)–C(31)	129·6 (2)		

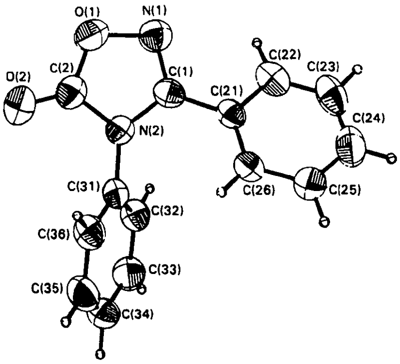

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule showing the atom numbering. Except for the H atoms thermal ellipsoids are shown with 50% probability.

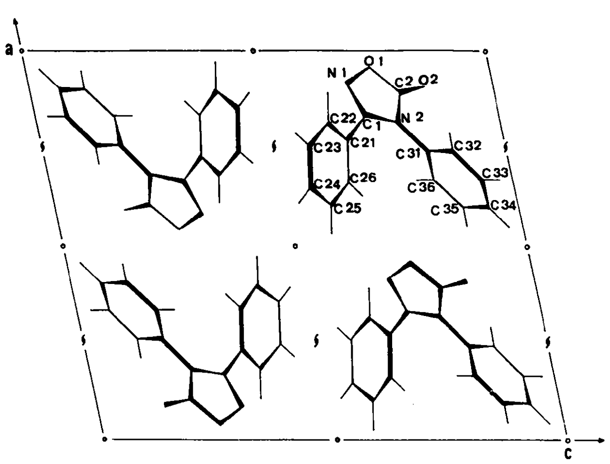

Fig. 2. Projection of $C_{14}H_{10}N_2O_2$ along the b axis.

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* Lists of structure factors, anisotropic thermal parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43898 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

[1·369 (3) and 1·363 (3) Å respectively] are also intermediate between a single and a double bond. C(2)—O(1) [1·340 (3) Å] also has partial double-bond character and is comparable to the carbon–oxygen single bond in carboxylic acids [1·36 (1) Å]. The C(2)—O(2) distance [1·199 (3) Å] is short and is similar to the carbon–oxygen double-bond length [1·207 (6) Å] observed in conjugated systems. (*International Tables for X-ray Crystallography*, 1962). Finally the N(1)—O(1) bond [1·434 (2) Å] is close to the N—O bond in furazan *N*-oxide (Sillitoe & Harding, 1978). We can conclude from these results that the heterocycle is strongly conjugated.

The torsional angles C(2)—N(2)—C(1)—N(1) = 0·01 and N(2)—C(1)—N(1)—O(1) = −0·56° show that the heterocycle is planar. On the other hand, N(2)—C(2)—C(1)—C(31) is very small (3°), which may suggest that N(2) is sp^2 hybridized and its unshared pair would be orthogonal to the heterocycle. This assumption is supported by the very short distance (0·05 Å) of the N(2) atom from the plane of its ligands defined by C(1), C(2) and C(31).

The phenyl ring, linked to N(2), makes a dihedral angle of 66° with the heterocycle plane and cannot be conjuguated with N(2); this is suggested by the N(2)—C(31) bond [1·435 (3) Å] which is comparable to the C—N bond [1·47 Å] in the amino compounds. The other phenyl ring linked to C(1) may be partially conjugated with the heterocycle, as suggested by the dihedral angle of 35° between the two rings and the C(1)—C(21) bond length [1·466 (3) Å].

Finally, the steric repulsion between the two phenyl rings prevents planarity; their dihedral angle is 72°.

References

BEL HADJ AMOR, A. & BACCAR, B. (1986). *J. Soc. Chim. Tunisie*, **11**, 9–13.

BOIS, C., PHILOCHE-LEVISALLES, M. & CHIHAOUI, M. (1983). *J. Heterocycl. Chem.* **20**, 97.

BREUER, H. (1976). *Chem. Abstr.* **85**, 123953a.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press (Present distributor D. Reidel, Dordrecht.)

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

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

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

RUIZ-VALERO, C., GUTIÉRREZ-PUEBLA, E. & MONGE, A. (1985). *Acta Cryst.* C**41**, 1791–1792.

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

SILLITOE, A. K. & HARDING, M. M. (1978). *Acta Cryst.* B**34**, 2021–2022.

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Structure of Acetamidinium Nitrate at 293 and 116 K

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Abstract. $C_2H_7N_2^+.NO_3^-$, $M_r = 121{\cdot}10$, monoclinic, $P2_1/m$, $a = 6{\cdot}457\,(2)$, $b = 6{\cdot}442\,(2)$, $c = 6{\cdot}884\,(1)$ Å, $\beta = 97{\cdot}56\,(2)°$, $V = 283{\cdot}9\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}4167\,(5)$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}2$ cm^{-1}, $F(000) = 128$, $T = 293\,(2)$ K, $R = 0{\cdot}035$ for 360 unique observed reflections. The structure was also refined from data at 116 K [cell dimensions: $a = 6{\cdot}415\,(3)$, $b = 6{\cdot}221\,(3)$, $c = 6{\cdot}832\,(3)$ Å, $\beta = 97{\cdot}27\,(4)°$] to $R = 0{\cdot}052$ for 467 unique observed reflections. The crystals were obtained by evaporation of a methanolic solution. The crystal structure contains layers of hydrogen-bonded ions with crystallographic mirror symmetry. Two hydrogen bonds, both with an N···O distance of 2·934 (4) Å, are formed between one acetamidinium ion and one nitrate ion to form a hydrogen-bonded ion pair.

Introduction. The present study is part of a research project on the structures of acetamidines and their metal complexes [see *e.g.* Norrestam, Mertz & Crossland (1983) and Norrestam (1984)]. When attempting to prepare a chromium(III) complex with acetamidine by evaporating a methanolic solution of chromium(III) nitrate and acetamidine (molar ratio 1:4) a crystalline specimen was formed. The lack of color of the crystals and the size of the unit cell (*cf.* Table 1) suggested that the crystals formed were not a chromium complex. Furthermore, the unit-cell parameters were close to

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