| | | | |
|---|---|---|---|
| O21—C2—N3 | 128.4 (1) | C412—C411—C4 | 119.3 (2) |
| O21—C2—N1 | 124.8 (1) | C422—C421—C4 | 124.6 (2) |
| N3—C2—N1 | 106.9 (1) | C426—C421—C4 | 117.3 (2) |
| C2—N3—C4 | 114.0 (1) | N51—C5—N1 | 124.7 (1) |
| C2—N3—H3 | 122.0 (13) | N51—C5—C4 | 128.4 (1) |
| C4—N3—H3 | 123.7 (13) | N1—C5—C4 | 106.9 (1) |
| N3—C4—C411 | 111.9 (1) | C5—N51—H51 | 110.3 (15) |
| N3—C4—C5 | 100.7 (1) | | |

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3···N51$^{i}$ | 0.84 (2) | 2.07 (2) | 2.873 (2) | 158 (2) |

Symmetry code: (i) $x - \frac{1}{2}, 1 - y, z$.

From the systematic absences ($0kl$: $k = 2n$ and $h0l$: $l = 2n$) the possible space groups *Pbcm* (No. 57) and $Pca2_1$ (No. 29) were deduced. With Z = 4, space group *Pbcm* is only possible if the molecule lies on the mirror plane. For steric reasons, this is very unlikely. Together with $|E^*E-1| = 0.825$, the structure could be solved easily in $Pca2_1$ (No. 29), while several computer runs in *Pbcm* did not produce any reasonable solutions (Sheldrick, 1990).

The positions of H3 and H51 were taken from a difference Fourier map and refined without any restrictions; all other H atoms were refined with distance restraints for the C—H distances (Sheldrick, 1993). All distances and angles are generally as expected. Some Friedel opposites have been collected. These Friedel pairs are regarded as symmetry independent and have not been merged.

Since the title compound crystallizes in a non-centrosymmetric space group, polar axis restraints were applied by the method of Flack & Schwarzenbach (1988).

Data collection: Enraf–Nonius CAD-4 diffractometer software. Cell refinement: Enraf–Nonius CAD-4 diffractometer software. Data reduction: *CADSHEL* (Kopf, 1987). Program(s) used to solve structure: *SHELXS*90 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *SHELXTL-Plus* (Sheldrick, 1992); *PLATON*92 (Spek, 1992). Software used to prepare material for publication: *CIF2TEX* (Kopf, 1992*a*); *FCF2FOC* (Kopf, 1992*b*).

This work was supported by the Fond der Chemischen Industrie.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1252). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# A Derivative of 1,3,4-Oxadiazole

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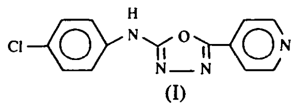
## Abstract

The molecules of title compound, 2-(4-chloroanilino)-5-(4-pyridyl)-1,3,4-oxadiazole, $C_{13}H_9ClN_4O$, are nearly planar with a maximum deviation of 0.094 (2) Å from the plane. Intermolecular N(1)—H···N(4′) hydrogen bonds link the molecules into chains [N(1)···N(4′) = 2.902 (3) Å, N(1)—H···N(4′) = 178 (2)°].

## Comment

It has been reported that compounds containing the heterocyclic 1,3,4-oxadiazole ring may have biological activity (Vansdadia, Vikani & Hansa Rarekh, 1988). In order to determine the structure of such a ring system we have synthesized the title compound, (I), following the method of Zhang, Yang & Zeng (1988).



(I)

The molecule is composed of three coplanar rings. The torsion angles C(8)—O—C(7)—N(1), C(2)—C(3)—

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C(4)—N(1) and C(9)—C(10)—C(11)—N(4) are −179.9 (2), 179.2 (2) and −0.4 (4)°, respectively. The maximum deviation of the atoms from the plane is 0.094 (2) Å.

Bond distances and angles are within the expected ranges (Rodier, Robert & Le Baut, 1993; Giordano, 1992). The asymmetric phenyl and pyridyl rings may cause some differences between the N=C and C—O bond lengths. The molecules are linked together to form chains by N(1)—H···N(4′) hydrogen bonds with N(1)···N(4′) = 2.902 (3) Å and N(1)—H···N(4′) = 178 (2)°. The angles C(4)—N(1)—H and C(7)—N(1)—H are 118 (3) and 116 (3)°, respectively.

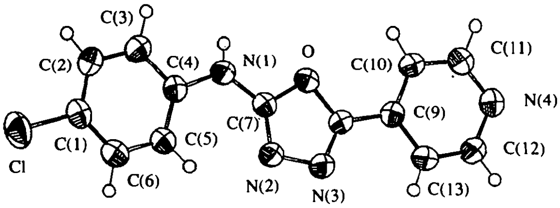

Fig. 1. An *ORTEP* drawing of the molecular structure. Displacement ellipsoids are shown at the 50% probability level.

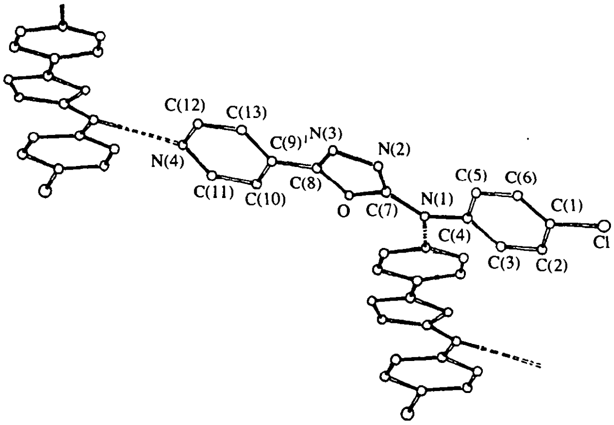

Fig. 2. Part of the polymeric chain structure. Hydrogen bonds are shown by dashed lines.

## Experimental

The title compound was prepared as described by Zhang, Yang & Zeng (1988) and was recrystallized from methanol. The density $D_m$ was measured by flotation.

### *Crystal data*

$C_{13}H_9ClN_4O$
$M_r = 272.70$
Monoclinic
$P2_1/n$
$a = 7.080$ (2) Å
$b = 14.245$ (1) Å
$c = 12.072$ (3) Å
$\beta = 95.86$ (2)°
$V = 1211.1$ (5) Å$^3$
$Z = 4$
$D_x = 1.50$ Mg m$^{-3}$
$D_m = 1.55$ Mg m$^{-3}$

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 12$–24°
$\mu = 2.80$ mm$^{-1}$
$T = 294$ K
Cubic
0.20 × 0.20 × 0.20 mm
Colourless

### *Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans [width $(0.6 + 0.140\tan\theta)$°]
Absorption correction: $\psi$ scans
$T_{\min} = 0.73$, $T_{\max} = 1.00$
1687 measured reflections
1610 independent reflections
1416 observed reflections [$F > 3\sigma(F)$]

$R_{\rm int} = 0.036$
$\theta_{\max} = 55°$
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 12$
3 standard reflections monitored every 150 reflections
intensity decay: 2%

### *Refinement*

Refinement on $F$
$R = 0.052$
$wR = 0.068$
$S = 1.88$
1416 reflections
208 parameters
All H-atom parameters refined with $B_{\rm iso} = 4$ Å$^2$
$w = 4(F_o^2)/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{\max} = 0.240$
$\Delta\rho_{\max} = 0.44$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.31$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$B_{\rm eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

| | $x$ | $y$ | $z$ | $B_{\rm eq}$ |
|---|---|---|---|---|
| Cl | 0.21670 (9) | 0.37971 (5) | 0.62385 (6) | 5.78 (2) |
| O | 1.0275 (2) | 0.0828 (1) | 0.3501 (1) | 3.58 (3) |
| N(1) | 0.8476 (3) | 0.1960 (1) | 0.4234 (2) | 3.92 (4) |
| N(2) | 0.7629 (3) | 0.0333 (1) | 0.4153 (2) | 4.12 (5) |
| N(3) | 0.8566 (3) | −0.0432 (1) | 0.3736 (2) | 4.21 (5) |
| N(4) | 1.4120 (3) | −0.1760 (1) | 0.1859 (2) | 3.98 (5) |
| C(1) | 0.4045 (3) | 0.3235 (2) | 0.5676 (2) | 3.94 (5) |
| C(2) | 0.5592 (4) | 0.3746 (2) | 0.5434 (2) | 4.18 (5) |
| C(3) | 0.7037 (3) | 0.3308 (2) | 0.4962 (2) | 3.87 (5) |
| C(4) | 0.6947 (3) | 0.2353 (2) | 0.4732 (2) | 3.42 (5) |
| C(5) | 0.5398 (3) | 0.1849 (2) | 0.5010 (2) | 3.58 (3) |
| C(6) | 0.3941 (3) | 0.2287 (2) | 0.5480 (2) | 4.05 (5) |
| C(7) | 0.8685 (3) | 0.1048 (2) | 0.3992 (2) | 3.43 (5) |
| C(8) | 1.0081 (3) | −0.0126 (2) | 0.3362 (2) | 3.40 (5) |
| C(9) | 1.1499 (3) | −0.0664 (2) | 0.2851 (2) | 3.33 (5) |
| C(10) | 1.3163 (3) | −0.0291 (2) | 0.2568 (2) | 4.13 (6) |
| C(11) | 1.4418 (3) | −0.0852 (2) | 0.2078 (2) | 4.42 (6) |
| C(12) | 1.2507 (3) | −0.2122 (2) | 0.2161 (2) | 3.79 (5) |
| C(13) | 1.1173 (3) | −0.1614 (2) | 0.2648 (2) | 3.87 (5) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Cl—C(1) | 1.747 (3) | C(1)—C(6) | 1.372 (4) |
| O—C(7) | 1.362 (3) | C(2)—C(3) | 1.371 (4) |
| O—C(8) | 1.374 (3) | C(3)—C(4) | 1.389 (3) |
| N(1)—C(4) | 1.408 (4) | C(4)—C(5) | 1.380 (3) |
| N(1)—C(7) | 1.343 (3) | C(5)—C(6) | 1.377 (3) |
| N(2)—N(3) | 1.397 (3) | C(8)—C(9) | 1.450 (3) |
| N(2)—C(7) | 1.290 (4) | C(9)—C(10) | 1.367 (3) |
| N(3)—C(8) | 1.282 (3) | C(9)—C(13) | 1.390 (4) |
| N(4)—C(11) | 1.334 (3) | C(10)—C(11) | 1.373 (4) |
| N(4)—C(12) | 1.337 (3) | C(12)—C(13) | 1.370 (4) |
| C(1)—C(2) | 1.372 (4) | | |

| | | | |
|---|---|---|---|
| C(7)—O—C(8) | 101.8 (2) | C(1)—C(6)—C(5) | 119.2 (2) |
| C(4)—N(1)—C(7) | 125.7 (3) | O—C(7)—N(1) | 115.7 (2) |
| N(3)—N(2)—C(7) | 105.0 (2) | O—C(7)—N(2) | 113.6 (3) |
| N(2)—N(3)—C(8) | 107.9 (2) | N(1)—C(7)—N(2) | 130.8 (2) |
| C(11)—N(4)—C(12) | 116.3 (3) | O—C(8)—N(3) | 111.8 (3) |
| Cl—C(1)—C(2) | 119.7 (2) | O—C(8)—C(9) | 120.6 (2) |
| Cl—C(1)—C(6) | 119.3 (2) | N(3)—C(8)—C(9) | 127.6 (2) |
| C(2)—C(1)—C(6) | 121.0 (2) | C(8)—C(9)—C(10) | 123.6 (2) |
| C(1)—C(2)—C(3) | 119.6 (2) | C(8)—C(9)—C(13) | 118.7 (3) |
| C(2)—C(3)—C(4) | 120.5 (2) | C(10)—C(9)—C(13) | 117.8 (2) |
| N(1)—C(4)—C(3) | 116.9 (3) | C(9)—C(10)—C(11) | 119.6 (2) |
| N(1)—C(4)—C(5) | 124.3 (2) | N(4)—C(11)—C(10) | 123.6 (2) |
| C(3)—C(4)—C(5) | 118.9 (2) | N(4)—C(12)—C(13) | 124.0 (2) |
| C(4)—C(5)—C(6) | 120.8 (2) | C(9)—C(13)—C(12) | 118.7 (2) |

Data collection was performed using *CAD*-4 diffractometer software (Enraf–Nonius, 1977). The structure was solved by direct methods with the *MULTAN*11/82 programs (Main *et al.*, 1982). The structure was refined by full-matrix least-squares methods using the *SDP* programs (Frenz, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving H atoms, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: AB1133). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Depogen, a Haemorheological Agent

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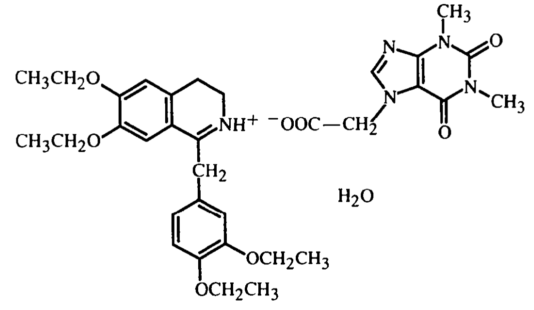
## Abstract

The crystal structure of the haemorheological agent depogen {1-[(3,4-diethoxyphenyl)methyl]-6,7-diethoxy-3,4-dihydro-2*H*$^+$-isoquinolinium 1,2,3,6-tetrahydro-7*H*-1,3-dimethyl-2,6-dioxopurine-7-acetate monohydrate, $C_{24}H_{32}NO_4^+.C_9H_9N_4O_4^-.H_2O$} reveals the role of a water molecule in building up a network of hydrogen bonds in its crystal lattice. Knowledge of the structure may help the elucidation of the differences in the pharmacological properties of the hydrated and the dehydrated compound.

## Comment

The haemorheological agent depogen, (I) (Kapui *et al.*, 1992), has been produced in two crystal forms. While the cotton-like anhydrous form resisted attempts to produce single crystals of good quality (and indeed a substance suitable for drug formulation purposes), the monohydrate crystals were of sufficient quality to be subjected to X-ray diffractometry.



(I)