

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SE1050). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 277–279

(*E,E*)-4-[3-(3,5-Dichloro-2-hydroxyphenylazo)-2-morpholino-2-butenoyl]morpholine

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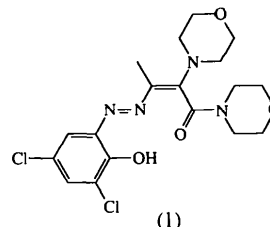
(Received 29 November 1993; accepted 11 August 1994)

Abstract

The title morpholide, $C_{18}H_{22}Cl_2N_4O_4$, shows an (*E,E*) configuration. An intramolecular hydrogen bond is formed between O4 and N3.

Comment

We synthesized (*E,E*)-4-[3-(3,5-dichloro-2-hydroxyphenylazo)-2-morpholino-2-butenoyl]morpholine (1) to investigate its catalytic and complexing properties. A satisfactory determination of the configuration of the C=C double bond was not possible by usual NMR techniques. Therefore, the compound was purified by column chromatography and an X-ray structure analysis performed.



A drawing of the molecule with the atomic numbering scheme is shown in Fig. 1. The bond lengths indicate that N3—N4 and C1—C11 are double bonds. The molecule shows an (*E,E*) configuration. An intramolecular hydrogen bond is formed between O4 and N3 [O4···N3 2.593(2), H4O···N3 1.810(3) Å, O4—H4O···N3 150(3)°]. The packing of the molecules in the unit cell is shown in Fig. 2. The molecular arrangement is determined by van der Waals interactions.

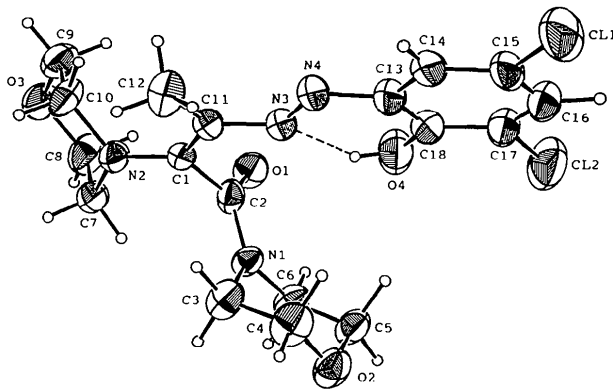


Fig. 1. *ORTEP* (Johnson, 1971) drawing of the molecular structure showing the crystallographic numbering scheme (50% probability displacement ellipsoids, hydrogen bond shown by dashed line).

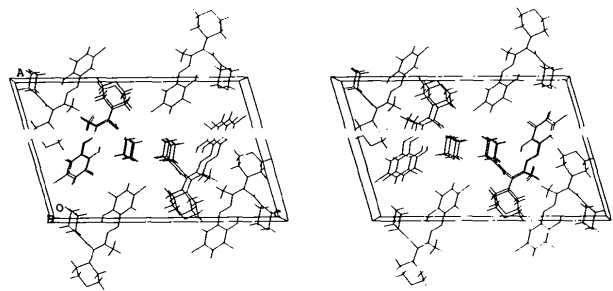


Fig. 2. *PLUTO* (Motherwell & Clegg, 1976) drawing of the molecular packing.

Experimental*Crystal data*C₁₈H₂₂Cl₂N₄O₄ $M_r = 429.30$

Monoclinic

 $P2_1/n$ $a = 14.9821 (9) \text{ \AA}$ $b = 5.9131 (7) \text{ \AA}$ $c = 23.502 (2) \text{ \AA}$ $\beta = 105.99 (1)^\circ$ $V = 2001.6 (3) \text{ \AA}^3$ $Z = 4$ $D_x = 1.424 \text{ Mg m}^{-3}$ Cu K α radiation $\lambda = 1.54178 \text{ \AA}$

Cell parameters from 25 reflections

 $\theta = 36\text{--}40^\circ$ $\mu = 3.24 \text{ mm}^{-1}$ $T = 298 \text{ K}$

Prism

 $0.21 \times 0.20 \times 0.14 \text{ mm}$

Orange

Crystal source: crystallization from diethyl ether

C13	0.4655 (1)	0.8544 (4)	0.20287 (9)	0.0484 (6)
C14	0.4127 (2)	0.9342 (5)	0.1482 (1)	0.0557 (7)
C15	0.3297 (1)	0.8308 (5)	0.1204 (1)	0.0602 (7)
C16	0.2982 (2)	0.6459 (5)	0.1449 (1)	0.0653 (8)
C17	0.3507 (2)	0.5656 (5)	0.1986 (1)	0.0599 (7)
C18	0.4350 (1)	0.6655 (4)	0.2290 (1)	0.0522 (6)

Table 2. Selected geometric parameters (\AA , $^\circ$)

C11—C15	1.750 (2)	N3—C11	1.376 (3)
C12—C17	1.731 (3)	N4—C13	1.413 (3)
O1—C2	1.222 (3)	C1—C2	1.520 (3)
O2—C4	1.422 (3)	C1—C11	1.381 (3)
O2—C5	1.422 (3)	C3—C4	1.513 (3)
O3—C8	1.427 (3)	C5—C6	1.508 (3)
O3—C9	1.416 (3)	C7—C8	1.497 (4)
O4—C18	1.350 (3)	C9—C10	1.500 (4)
N1—C2	1.343 (2)	C11—C12	1.490 (3)
N1—C3	1.458 (3)	C13—C14	1.393 (3)
N1—C6	1.460 (3)	C13—C18	1.409 (3)
N2—C1	1.350 (3)	C14—C15	1.378 (3)
N2—C7	1.465 (3)	C15—C16	1.379 (4)
N2—C10	1.473 (3)	C16—C17	1.375 (4)
N3—N4	1.292 (2)	C17—C18	1.400 (3)
C4—O2—C5	109.9 (2)	O3—C8—C7	111.2 (2)
C8—O3—C9	110.4 (2)	O3—C9—C10	113.1 (2)
C2—N1—C3	126.0 (2)	N2—C10—C9	109.1 (2)
C2—N1—C6	121.2 (2)	N3—C11—C1	112.3 (2)
C3—N1—C6	112.7 (2)	N3—C11—C12	120.7 (2)
C1—N2—C7	122.6 (2)	C1—C11—C12	126.7 (2)
C1—N2—C10	123.5 (2)	N4—C13—C14	114.1 (2)
C7—N2—C10	110.4 (2)	N4—C13—C18	125.9 (2)
N4—N3—C11	115.6 (2)	C14—C13—C18	120.0 (2)
N3—N4—C13	114.2 (2)	C13—C14—C15	119.8 (2)
N2—C1—C2	115.3 (2)	C11—C15—C14	118.7 (2)
N2—C1—C11	126.7 (2)	C11—C15—C16	119.9 (2)
C2—C1—C11	118.0 (2)	C14—C15—C16	121.4 (2)
O1—C2—N1	123.8 (2)	C15—C16—C17	118.9 (2)
O1—C2—C1	119.7 (2)	C12—C17—C16	119.4 (2)
N1—C2—C1	116.6 (2)	C12—C17—C18	118.6 (2)
N1—C3—C4	108.5 (2)	C16—C17—C18	122.0 (2)
O2—C4—C3	110.7 (2)	O4—C18—C13	122.9 (2)
O2—C5—C6	110.6 (2)	O4—C18—C17	119.2 (2)
N1—C6—C5	109.6 (2)	C13—C18—C17	117.9 (2)
N2—C7—C8	110.3 (2)		

Data collection: CAD-4 diffractometer control software (Enraf-Nonius, 1988). Cell refinement: CAD-4 diffractometer control software. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to solve structure: *MULTAN11/82* (Main *et al.*, 1982). Program(s) used to refine structure: *MolEN LSFM*. Software used to prepare material for publication: *PLATON92* (Spek, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SE1047). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

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

	x	y	z	U_{eq}
C11	0.26296 (4)	0.9411 (2)	0.05295 (3)	0.0829 (2)
C12	0.31115 (5)	0.3363 (2)	0.23047 (4)	0.0866 (3)
O1	0.6863 (1)	0.5948 (3)	0.39136 (7)	0.0505 (4)
O2	0.4186 (1)	0.9824 (3)	0.41760 (7)	0.0576 (5)
O3	0.9798 (1)	0.8448 (3)	0.44071 (7)	0.0643 (6)
O4	0.4839 (1)	0.5773 (3)	0.28123 (7)	0.0635 (5)
N1	0.5999 (1)	0.8746 (3)	0.41505 (7)	0.0396 (5)
N2	0.7993 (1)	1.0310 (3)	0.39646 (7)	0.0450 (5)
N3	0.5922 (1)	0.9185 (3)	0.28033 (7)	0.0432 (5)
N4	0.5476 (1)	0.9788 (3)	0.22727 (7)	0.0490 (5)
C1	0.7147 (1)	0.9672 (4)	0.36269 (8)	0.0390 (5)
C2	0.6648 (1)	0.7943 (3)	0.39071 (8)	0.0370 (5)
C3	0.5705 (2)	1.1098 (4)	0.4148 (1)	0.0494 (6)
C4	0.4669 (2)	1.1214 (4)	0.3865 (1)	0.0603 (8)
C5	0.4453 (1)	0.7528 (4)	0.4149 (1)	0.0535 (7)
C6	0.5479 (1)	0.7239 (4)	0.4434 (1)	0.0468 (6)
C7	0.8356 (1)	0.9630 (5)	0.45868 (9)	0.0534 (7)
C8	0.9054 (2)	0.7772 (5)	0.4640 (1)	0.0623 (8)
C9	0.9454 (2)	0.9113 (5)	0.3808 (1)	0.0606 (8)
C10	0.8758 (1)	1.0997 (5)	0.3720 (1)	0.0558 (7)
C11	0.6707 (1)	1.0422 (4)	0.30629 (8)	0.0417 (6)
C12	0.6948 (2)	1.2464 (4)	0.2765 (1)	0.0508 (6)

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cis-5-Methyl-4-phenyl-3-(4-toluenesulfonyl)-1,3-oxazin-2-one

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Abstract

The structure of the title compound, $C_{18}H_{19}NO_4S$, has been determined. This confirms the *anti* relative stereochemistry of the product of the lithium aluminium hydride reduction of methyl 2-methylene-3-phenyl-3-(4-toluenesulfonylamino)propanoate.

Comment

In order to further exploit a method for preparing substituted α -methylene- β -aminoesters (Perlmutter & Teo, 1984) some simple synthetic transformations of these compounds have been studied (Campi, Holmes, Perlmutter & Teo, 1994). In one case it was found that the product of a reduction was obtained as a single diastereoisomer. In order to determine the relative stereochemistry of this product it was converted to the title compound, (I). As there can sometimes be ambiguities in the 1H NMR analysis of the stereochemistry of oxazin-2-ones, the crystal structure of the title compound was determined. The results of this study confirmed the relative stereochemistry as 4,5-*cis*. Thus, the oxazinone adopts a conformation in which the methyl and 4-toluenesulfonyl substituents occupy pseudo-equatorial positions and the phenyl group has a pseudo-axial orientation. The factors which govern this striking feature are currently under investigation.

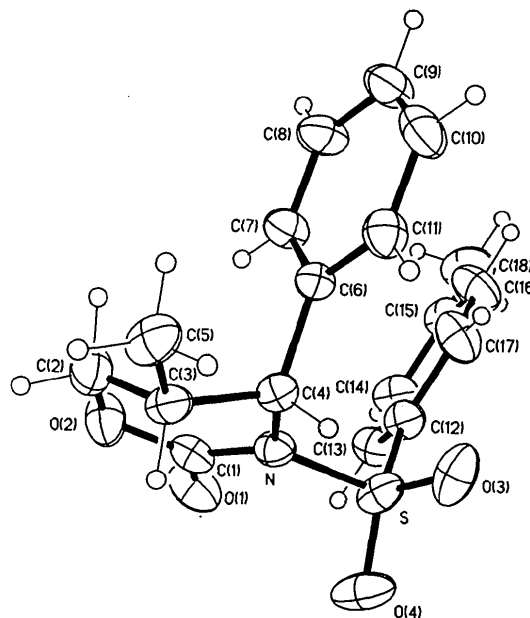
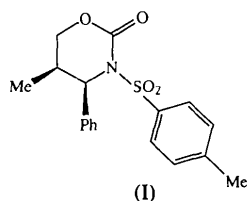


Fig. 1. A view of the title compound showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 40% probability level for non-H atoms; H atoms are drawn as circles of arbitrary radii.

Experimental

Crystal data

$C_{18}H_{19}NO_4S$

$M_r = 345.4$

Orthorhombic

Pbca

$a = 18.688$ (9) Å

$b = 11.428$ (6) Å

$c = 15.797$ (8) Å

$V = 3374$ (3) Å³

$Z = 8$

$D_x = 1.36$ Mg m⁻³

$D_m = 1.35$ (1) Mg m⁻³

D_m measured by flotation in CCl_4 /hexane

Cu $K\alpha$ radiation

$\lambda = 1.5418$ Å

Cell parameters from 24 reflections

$\theta = 14$ – 19°

$\mu = 1.85$ mm⁻¹

$T = 293$ K

Prism

$0.17 \times 0.10 \times 0.07$ mm

Colourless

Data collection

Philips PW1100 diffractometer

θ scans

Absorption correction:

Gaussian (SHELX76;

Sheldrick, 1976)

$T_{min} = 0.767$, $T_{max} =$

0.874

3259 measured reflections

2875 independent reflections

2006 observed reflections
[$I > 3\sigma(I)$]

$\theta_{max} = 65.0^\circ$

$h = 0 \rightarrow 20$

$k = 0 \rightarrow 13$

$l = 0 \rightarrow 18$

3 standard reflections

frequency: 240 min

intensity decay: none

Refinement

Refinement on F

$R = 0.048$

$wR = 0.058$

$\Delta\rho_{max} = 0.243$ e Å⁻³

$\Delta\rho_{min} = -0.476$ e Å⁻³

Extinction correction: none