Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*,*E*,*E*)-2-(2-{[3-(2,6-Dichlorophenyl)-1-methyl-*trans*-2-propenylidene]aminooxymethyl}phenyl)-2-methoxyimino-*N*-methylacetamide

Fan-Yong Yan,[a]* Liu Dong-Qing,[b] Zeng Tao,[a] Ren-bing Zhou[c] and Duan Xue-Min[d]

[a]College of Pharmaceuticals & Biotechnology, Tianjin University, Tianjin 300072, People's Republic of China, [b]College of Chemical Engineering, Tianjin University, Tianjin 300072, People's Republic of China, [c]Shenyang Research Institute of Chemical Industry, Shenyang 110021, People's Republic of China, and [d]School of Pharmacy, Jiangxi Science & Technology, Normal University, Nanchang, Jiangxi 330013, People's Republic of China

Correspondence e-mail: yfany@163.com

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.118
Data-to-parameter ratio = 16.0

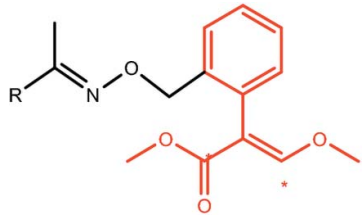
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 31 August 2005
Accepted 28 September 2005
Online 12 October 2005

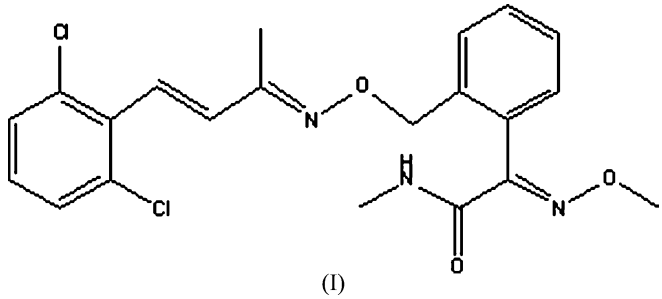
In the title compound, $C_{21}H_{21}Cl_2N_3O_3$, the dihedral angle between the benzene rings is 81.1 (5)°.

Comment

The title compound, (I), is the most active in a series of antifungal compounds (Zhang & Steven, 2001, Zhang *et al.*, 2002).



In the above compounds, the active part of the compound is shown in the diagram in red. From this it can be seen that it is a propanoic acid derivative. Recently, we found that when the C atom marked by an asterisk was replaced by N, the activity of the compound improved. Thus, this compound can be considered a propanoic acid derivative, and can be used as a broad-spectrum fungicide to control the disease caused by phytopathogenic fungi, for example, wheat leaf rust, wheat powdery mildew and wheat leaf blotch (Zhang & Steven, 2001; Zhang *et al.*, 2002). The structure of the title compound, (I), is shown in Fig. 1. The dihedral angle between the benzene rings is 81.1 (5)°.



(I)

Experimental

A solution of *trans*-1-(2,6-dichlorophenyl)-3-butene-2-one 2-oxime (2.2 g, 0.01 mol) in dimethylformamide (DMF, 20 ml) was added to methyl 2-(2-bromomethylphenyl)-2-methoxyiminoacetate (2.7 g, 0.01 mol) and DMF (10 ml). When the mixture dissolved, NaOH (0.58 g, 14.4 mmol) was added. After stirring overnight at room temperature, the mixture was poured into water (100 ml), extracted with ethyl acetate (150 ml), washed with water (50 ml), dried by anhydrous magnesium sulfate and concentrated. A mixture of two

diastereoismers was obtained, which was separated by silica gel flash chromatography (ethyl acetate/hexane, 1:8), giving 1.28 g of (*E*,*E*,*E*)-methyl 2-(2-{[1-methyl-3-(2,6-dichlorophenyl)-*trans*-2-propenylidene]aminooxymethyl}phenyl)-2-methoxyiminoacetate as a yellow oil (*A*). Isomer *A* (0.5 g, 0.0011 mol) was placed in a 10 ml round-bottomed flask, and 20 ml MeOH and 40% aqueous methyl amine (0.7 g, 0.009 mol) were added. The mixture was refluxed for 26 h and the solvent was then evaporated. The residue was extracted with ethyl acetate and water, dried by anhydrous magnesium sulfate and concentrated. The crude product, (I), was purified by silica-gel flash chromotography (ethyl acetate/hexane, 1:2) and 200 mg oil was obtained, which crystallized after standing. Suitable crystals (m.p. 404–406 K) were obtained by slow evaporation of a solution in a mixture of ethyl acetate and ethanol (1:3 *v*/*v*). ^{1}H NMR ($CDCl_3$): δ 2.03 (*s*, 3H), 3.87 (*d*, 3H), 4.97 (*s*, 2H), 6.5–6.6 (*m*, 1H), 6.71 (*s*, 2H), 6.80–7.50 (*m*, 7H).

Crystal data

$C_{21}H_{21}Cl_2N_3O_3$	D_x = 1.379 Mg m^{-3}
M_r = 434.31	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 4514 reflections
a = 8.2864 (14) Å	θ = 2.7–26.3°
b = 14.469 (2) Å	μ = 0.34 mm^{-1}
c = 17.703 (3) Å	T = 294 (2) K
β = 99.681 (3)°	Prism, colourless
V = 2092.3 (6) Å^3	0.16 × 0.12 × 0.04 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	4309 independent reflections
φ and ω scans	3090 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	R_{int} = 0.022
T_{min} = 0.940, T_{max} = 0.987	θ_{max} = 26.5°
11730 measured reflections	$h = -8 \rightarrow 10$
	$k = -18 \rightarrow 18$
	$l = -14 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.879P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.042	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.118	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
4309 reflections	
269 parameters	
H atoms treated by a mixture of independent and constrained refinement	

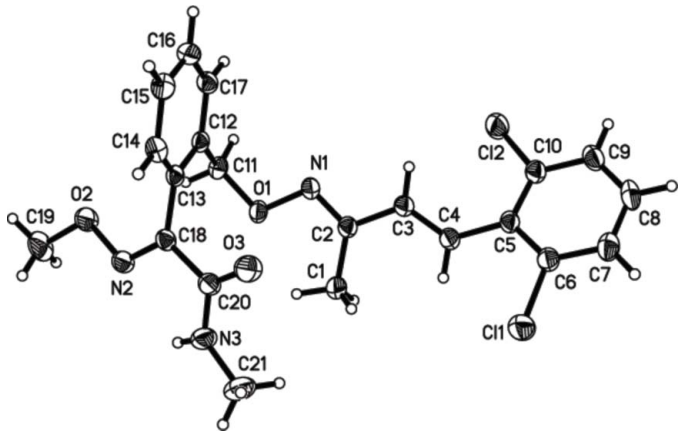


Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

The H atom attached to N was initially located in a difference Fourier map and its coordinates and isotropic displacement parameter were freely refined. All other H atoms were positioned geometrically, with C—H = 0.93 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1997). *SADABS*, *SMART*, *SAINT* and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zhang, L. X., Liu, D. Q. & Steven, H. S. (2002). CN Patent, 1 309 897.
Zhang, L. X. & Steven, H. S. (2001). US Patent, 6 303 818.