Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.027$
$\omega/2\theta$ scans	$\theta_{max} = 25°$
Absorption correction: none	$h = 0 \rightarrow 5$
1064 measured reflections	$k = 0 \rightarrow 19$
944 independent reflections	$l = -8 \rightarrow 8$
791 reflections with $I > 2\sigma(I)$	3 standard reflections every 200 reflections intensity decay: 1.8%

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.270$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.044$	$\Delta\rho_{min} = -0.186$ e Å^{-3}
$wR(F^2) = 0.133$	Extinction correction: *SHELXL*97 (Sheldrick, 1997)
$S = 1.084$	Extinction coefficient: 0.14 (2)
944 reflections	Scattering factors from *International Tables for Crystallography* (Vol. C)
99 parameters	
All H atoms refined	
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$	
$(\Delta/\sigma)_{max} < 0.001$	

Table 1. *Selected geometric parameters (Å, °)*

O—C1	1.345 (2)	C3—C4	1.379 (3)
O—C2	1.381 (2)	C7—C6	1.390 (3)
N—C1	1.289 (2)	C5—C6	1.367 (3)
N—C7	1.393 (2)	C5—C4	1.384 (3)
C2—C3	1.368 (2)	C1—C1^{i}	1.447 (3)
C2—C7	1.377 (2)		
C1—O—C2	103.40 (13)	C6—C7—N	130.94 (16)
C1—N—C7	103.21 (14)	C6—C5—C4	122.51 (19)
C3—C2—C7	124.47 (17)	C5—C6—C7	116.38 (18)
C3—C2—O	128.21 (16)	N—C1—O	116.92 (15)
C7—C2—O	107.32 (15)	N—C1—C1^{i}	126.1 (2)
C2—C3—C4	114.88 (17)	O—C1—C1^{i}	117.01 (19)
C2—C7—C6	119.92 (16)	C3—C4—C5	121.83 (17)
C2—C7—N	109.14 (15)		

Symmetry code: (i) $-x, -y, 1 - z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1990). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Enraf–Nonius, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1995). Software used to prepare material for publication: *SHELXL*97.

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(2,4-Dichlorophenoxy)acetohydrazide

N. K. LOKANATH,[a] M. A. SRIDHAR,[a] J. SHASHIDHARA PRASAD,[a] H. S. NAGARAJA[b] AND P. MOHAN RAO[b]

[a]*Department of Studies in Physics, University of Mysore, Manasagangothri, Mysore 570 006, India, and* [b]*Department of Studies in Physics, Mangalore University, Mangalagangotri, Mangalore 574 199, India. E-mail: mysxrd@giasbg01.vsnl.net.in*

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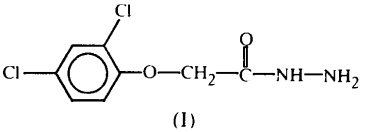
Abstract

The title compound, $C_8H_8Cl_2N_2O_2$, is an intermediate compound in the synthesis of one of the important 1,2,4-triazoles that possess diverse pharmacological activities.

Comment

Various 1,2,4-triazoles are found to possess varied pharmacological activities. Many of them are conveniently prepared starting from hydrazides. They are important due to their analgesic, antibacterial, antifungal, antiviral, herbicidal, insecticidal and antitubercular (Rudnicka & Osmialowska, 1979) properties. They also find application in the preparation of photographic plates (Martin *et al.*, 1978), in polymers and as analytical reagents for the estimation of metals such as silver, copper and lead. Various condensed nitrogen heterocycles derived from hydrazides are important due to their antibacterial and anticancer properties. The title hydrazide, (I), is an intermediate compound in the synthesis of 4-amino-3-(2,4-dichlorophenoxymethyl)-5-mercapto-1,2,4-triazole.



(I)

The bond distances and angles of (I) do not show any large deviations from expected values.

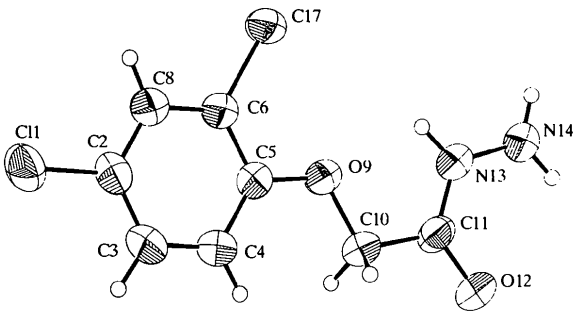


Fig. 1. Plot of the title molecule with 50% probability displacement ellipsoids for non-H atoms.

Experimental

Phenol on reacting with ethyl chloroacetate in dry acetone gave ethyl (2,4-dichlorophenoxy)acetate. Hydrolysis of this ester with hydrazine hydrate in absolute ethanol yielded compound (I). Single crystals were grown by slow evaporation of a solution of (I) in dimethylformamide.

Crystal data

$C_8H_8Cl_2N_2O_2$
$M_r = 235.06$
Triclinic
$P\bar{1}$
$a = 8.107\ (3)$ Å
$b = 8.530\ (3)$ Å
$c = 7.339\ (3)$ Å
$\alpha = 90.11\ (3)°$
$\beta = 98.44\ (3)°$
$\gamma = 94.859\ (3)°$
$V = 500.2\ (3)$ Å^3
$Z = 2$
$D_x = 1.561$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 16 reflections
$\theta = 6.8–9.2°$
$\mu = 0.623$ mm^{-1}
$T = 293\ (2)$ K
Prismatic
0.2 × 0.2 × 0.2 mm
Transparent

Data collection

Rigaku AFC-7*S* diffractometer
ω–2θ scans
Absorption correction: none
1436 measured reflections
1351 independent reflections
1046 reflections with $I > 2\sigma(I)$

$R_{int} = 0.041$
$\theta_{max} = 25°$
$h = 0 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -8 \rightarrow 8$
3 standard reflections
every 150 reflections
intensity decay: −1.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.154$
$S = 1.166$
1350 reflections
140 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.170$ e Å^{-3}
$\Delta\rho_{min} = -0.198$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.10 (13)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1248). Services for accessing these data are described at the back of the journal.

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4-Methylspiro[4-azahomoadamantane-5,3′-[3′*H*]naphth[2,1-*b*][1,4]oxazine], a New Photochromic Spirooxazine

KARINE CHAMONTIN,[a] VLADIMIR LOKSHIN,[a] ROBERT GUGLIELMETTI,[a] ANDRÉ SAMAT[a] AND GÉRARD PÈPE[b]

[a]*LCMOM, Université d'Aix-Marseille II, Campus de Luminy, Case 901, 13288 Marseille CEDEX 9, France, and* [b]*Centre de Recherche sur les Mécanismes de la Croissance Cristalline, Universités d'Aix-Marseille II et III, Campus de Luminy, Case 913, 13288 Marseille CEDEX 9, France. E-mail: genmol@crmc2.univ-mrs.fr*

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Abstract

Comparison of the molecular geometry of the title compound, $C_{22}H_{24}N_2O$, with that of other spirooxazines indicates that there is no correlation between the degree