

Table 1 (continued)

Title	Reference	Retracted by	DOI	Refcode
{ μ -6,6'-Dimethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}- μ -nitrate-dinitratoeuropium(III)zinc(II)	Hu <i>et al.</i> (2008)	Author	10.1107/S160053680706151X	MIRPAF
Bis(4-chloro-2-formylphenolato)nickel(II)	Li <i>et al.</i> (2008)	Author	10.1107/S1600536807056309	RISTET
{ μ -6,6'-Dimethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}- μ -nitrate-dinitratoerbium(III)zinc(II)	Chen <i>et al.</i> (2008)	Author	10.1107/S1600536808006958	QIXHIP
Bis(2-ethoxy-6-formylphenolato- $\kappa^2 O^1, O^6$)nickel(II)	Han (2008)	Journal	10.1107/S160053680800809X	QIXLIT
{ μ -6,6'-Dimethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}- μ -nitrate-dinitratoholmium(III)zinc(II)	Xiao, Sui <i>et al.</i> (2008)	Author	10.1107/S1600536808013743	BIZTUA
{ μ -6,6'-Diethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratoholmium(III)nickel(II)	Xiao, Fu <i>et al.</i> (2008)	Author	10.1107/S1600536808013755	BIZVAI
Hydrogen-bonding patterns in the cocrystal terephthalic acid-4,4'-bipyridine (2I)	Wang <i>et al.</i> (2009)	Journal	10.1107/S160053680903236X	DUCZEH
{6,6'-Dimethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato- $1\kappa^4 O^1, O^1, O^6, O^6:2\kappa^4 O^1, N, N', O^1$ } (ethanol- $1\kappa O$)- μ -nitrate- $1:2\kappa^2 O:O'$ -dinitrato- $1\kappa^2 O, O'$ -samarium(III)zinc(II)	Huang <i>et al.</i> (2009)	Journal	10.1107/S1600536809033558	YUCWAV

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2-(2,4-Dichlorophenylsulfanyl)aceto- hydrazide

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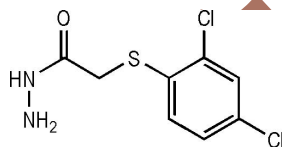
Received 4 May 2007; accepted 9 May 2007

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.044; wR factor = 0.136; data-to-parameter ratio = 12.9.

The title compound, $\text{C}_8\text{H}_8\text{Cl}_2\text{N}_2\text{OS}$, is an important intermediate for the synthesis of biologically active heterocyclic compounds. The planar hydrazide group is oriented with respect to the benzene ring at a dihedral angle of $88.93(3)^\circ$.

Related literature

For related literature, see: Ahmad *et al.* (2001); Al-Soud *et al.* (2004); Al-Talib *et al.* (1990); Allen *et al.* (1987); El-Emam *et al.* (2004); Yousif *et al.* (1986); Zheng *et al.* (2003); Furniss *et al.* (1978).



Experimental

Crystal data

$\text{C}_8\text{H}_8\text{Cl}_2\text{N}_2\text{OS}$
 $M_r = 251.13$
 Triclinic, $P\bar{1}$
 $a = 7.350(5)$ Å
 $b = 8.133(6)$ Å
 $c = 8.545(6)$ Å

$\alpha = 94.802(10)^\circ$
 $\beta = 90.140(9)^\circ$
 $\gamma = 98.492(10)^\circ$
 $V = 503.4(6)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.82$ mm⁻¹
 $T = 293(2)$ K

$0.15 \times 0.14 \times 0.14$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.882$, $T_{\max} = 0.892$

3032 measured reflections
 1644 independent reflections
 1160 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.136$
 $S = 1.14$
 1644 reflections

127 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.22$ e Å⁻³
 $\Delta\rho_{\min} = -0.12$ e Å⁻³

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); 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, 1999); software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2105).

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supplementary materials

Article retracted

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2-(2,4-Dichlorophenylsulfanyl)acetohydrazide

G. Qadeer, N. H. Rama and W.-T. Chen

Comment

Aromatic hydrazides are important intermediates in heterocyclic chemistry and have been used for the synthesis of various biologically active five-membered heterocycles such as 2,5-disubstituted-1,3,4-oxadiazoles (Zheng *et al.*, 2003; Al-Talib *et al.*, 1990) and 5-substituted-2-mercapto-1,3,4-oxadiazoles (Yousif *et al.*, 1986; Ahmad *et al.*, 2001; Al-Soud *et al.*, 2004; El-Emam *et al.*, 2004). In view of the versatility of these compounds, we have synthesized the title compound, (I), and reported its crystal structure (Fig. 1). Bond distances and angles are within expected ranges (Allen *et al.*, 1987). The dihedral angle between the planar hydrazidic group (C8/O1/N1/N2) and benzene ring (C1—C6) is 91.07 (3)°. The two centrosymmetrically related N1—H1A···O1 (N1···O1, 3.078 Å, H1A···O1, 2.666 Å, N1—H1A···O1, 110.8 °) hydrogen bonds form a dimer (Fig. 3).

Experimental

A mixture of methyl-2-(2,4-dichlorophenylthio)acetate (2.51 g, 10 mmol) and hydrazine hydrate (15 ml, 80%) in absolute ethanol (50 ml) was refluxed for 5 h at 413–423 K. The excess solvent was removed by distillation. The solid residue was filtered off, washed with water and recrystallized from ethanol (30%) to give the title compound (yield, 2.28 g; 91%, m.p. 333–335 K). Colourless single crystals of (I) were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH and NH₂) and C—H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C}, \text{N})$, where $x = 1.2$ for all other H atoms.

Figures

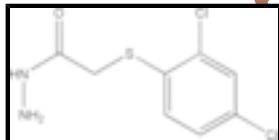


Fig. 1. Chemical diagram of (I).

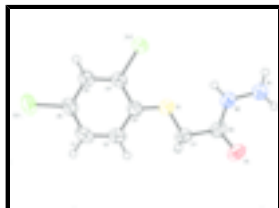
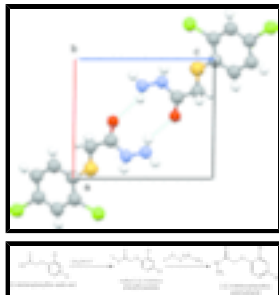


Fig. 2. The molecular structure of (I) with the 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

Fig. 3. The packing diagram of (I), viewed down the *b* axis.



2-(2,4-Dichlorophenylsulfanyl)acetohydrazide

Crystal data

$C_8H_8Cl_2N_2O_1S_1$

$M_r = 251.13$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.350$ (5) Å

$b = 8.133$ (6) Å

$c = 8.545$ (6) Å

$\alpha = 94.802$ (10)°

$\beta = 90.140$ (9)°

$\gamma = 98.492$ (10)°

$V = 503.4$ (6) Å³

$Z = 2$

$F_{000} = 240$

$D_x = 1.657$ Mg m⁻³

Melting point: 333(2) K

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1520 reflections

$\theta = 2.7\text{--}24.9^\circ$

$\mu = 0.82$ mm⁻¹

$T = 293$ (2) K

Block, colourless

$0.15 \times 0.14 \times 0.14$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: rotating-anode generator

Monochromator: graphite

$T = 293$ (2) K

ϕ - ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.882$, $T_{\max} = 0.892$

3032 measured reflections

1644 independent reflections

1160 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.8^\circ$

$h = -8 \rightarrow 8$

$k = -9 \rightarrow 9$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.136$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0784P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.14$	$(\Delta/\sigma)_{\max} < 0.001$
1644 reflections	$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$
127 parameters	$\Delta\rho_{\min} = -0.12 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.26701 (14)	1.01419 (12)	-0.39917 (10)	0.0743 (4)
C12	0.29479 (11)	0.81304 (11)	0.18225 (9)	0.0635 (3)
S1	-0.0786 (2)	0.6652 (3)	0.1237 (2)	0.0532 (6)
O1	-0.4782 (3)	0.4371 (3)	0.2779 (3)	0.0636 (7)
N1	-0.2216 (3)	0.4350 (3)	0.5150 (3)	0.0590 (7)
H1A	-0.3282	0.3804	0.5317	0.071*
H1B	-0.1347	0.4414	0.5838	0.071*
N2	-0.1897 (3)	0.5134 (3)	0.3760 (3)	0.0493 (6)
H2A	-0.0822	0.5674	0.3615	0.059*
C1	-0.0239 (4)	0.8374 (4)	-0.2621 (4)	0.0553 (8)
H1C	-0.0901	0.8442	-0.3536	0.066*
C2	-0.1059 (4)	0.7541 (4)	-0.1410 (4)	0.0530 (8)
H2B	-0.2280	0.7042	-0.1521	0.064*
C3	-0.0111 (4)	0.7427 (3)	-0.0025 (3)	0.0420 (7)
C4	0.1726 (4)	0.8207 (4)	0.0105 (3)	0.0440 (7)
C5	0.2569 (4)	0.9021 (4)	-0.1106 (3)	0.0500 (8)
H5A	0.3796	0.9509	-0.1015	0.060*
C6	0.1565 (4)	0.9103 (4)	-0.2466 (4)	0.0489 (7)
C7	-0.2690 (3)	0.5928 (4)	0.1181 (4)	0.0480 (8)
H7A	-0.2920	0.5122	0.0274	0.058*
H7B	-0.3450	0.6792	0.1076	0.058*
C8	-0.3188 (4)	0.5081 (4)	0.2650 (4)	0.0465 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0896 (7)	0.0862 (7)	0.0453 (5)	-0.0039 (5)	0.0005 (4)	0.0252 (5)

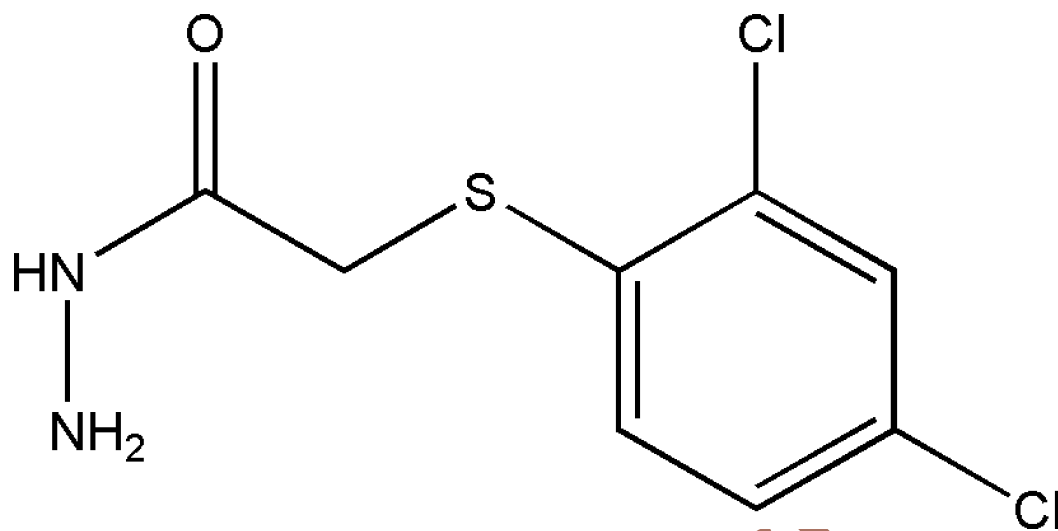
supplementary materials

C12	0.0477 (5)	0.0938 (7)	0.0471 (5)	-0.0065 (4)	-0.0114 (3)	0.0262 (4)
S1	0.0355 (11)	0.0735 (14)	0.0494 (13)	-0.0026 (9)	-0.0025 (9)	0.0172 (11)
O1	0.0348 (11)	0.0873 (17)	0.0636 (15)	-0.0072 (11)	0.0015 (10)	0.0057 (12)
N1	0.0428 (14)	0.085 (2)	0.0482 (16)	-0.0029 (13)	-0.0014 (11)	0.0211 (14)
N2	0.0376 (13)	0.0612 (16)	0.0485 (15)	-0.0014 (11)	0.0028 (11)	0.0158 (12)
C1	0.061 (2)	0.067 (2)	0.0393 (18)	0.0140 (16)	-0.0130 (15)	0.0063 (16)
C2	0.0393 (16)	0.069 (2)	0.050 (2)	0.0043 (14)	-0.0081 (14)	0.0072 (16)
C3	0.0410 (15)	0.0484 (17)	0.0376 (16)	0.0080 (13)	0.0005 (12)	0.0075 (13)
C4	0.0432 (15)	0.0502 (17)	0.0382 (16)	0.0057 (13)	-0.0063 (12)	0.0033 (14)
C5	0.0521 (17)	0.0557 (19)	0.0398 (17)	-0.0019 (14)	-0.0020 (14)	0.0080 (14)
C6	0.0595 (18)	0.0514 (18)	0.0356 (16)	0.0073 (14)	-0.0010 (13)	0.0043 (13)
C7	0.0322 (14)	0.0607 (19)	0.0501 (19)	0.0027 (13)	-0.0025 (13)	0.0057 (15)
C8	0.0359 (15)	0.0531 (18)	0.0493 (18)	0.0052 (13)	0.0026 (13)	-0.0006 (14)

Geometric parameters (\AA , $^\circ$)

C11—C6	1.749 (3)	C1—C2	1.376 (4)
C12—C4	1.729 (3)	C1—H1C	0.9300
S1—C3	1.353 (3)	C2—C3	1.388 (4)
S1—C7	1.435 (3)	C2—H2B	0.9300
O1—C8	1.237 (3)	C3—C4	1.405 (4)
N1—N2	1.399 (4)	C4—C5	1.375 (4)
N1—H1A	0.8600	C5—C6	1.387 (4)
N1—H1B	0.8600	C5—H5A	0.9300
N2—C8	1.334 (4)	C7—C8	1.502 (4)
N2—H2A	0.8600	C7—H7A	0.9700
C1—C6	1.371 (4)	C7—H7B	0.9700
C3—S1—C7	117.5 (2)	C5—C4—C12	119.6 (2)
N2—N1—H1A	120.0	C3—C4—C12	119.1 (2)
N2—N1—H1B	120.0	C4—C5—C6	119.0 (3)
H1A—N1—H1B	120.0	C4—C5—H5A	120.5
C8—N2—N1	122.8 (2)	C6—C5—H5A	120.5
C8—N2—H2A	118.6	C1—C6—C5	121.0 (3)
N1—N2—H2A	118.6	C1—C6—C11	120.8 (2)
C6—C1—C2	119.4 (3)	C5—C6—C11	118.2 (2)
C6—C1—H1C	120.3	S1—C7—C8	110.3 (2)
C2—C1—H1C	120.3	S1—C7—H7A	109.6
C1—C2—C3	121.7 (3)	C8—C7—H7A	109.6
C1—C2—H2B	119.2	S1—C7—H7B	109.6
C3—C2—H2B	119.2	C8—C7—H7B	109.6
S1—C3—C2	126.5 (3)	H7A—C7—H7B	108.1
S1—C3—C4	116.0 (2)	O1—C8—N2	122.9 (3)
C2—C3—C4	117.5 (3)	O1—C8—C7	118.5 (3)
C5—C4—C3	121.3 (3)	N2—C8—C7	118.6 (2)

Fig. 1



Article retraced

Fig. 2

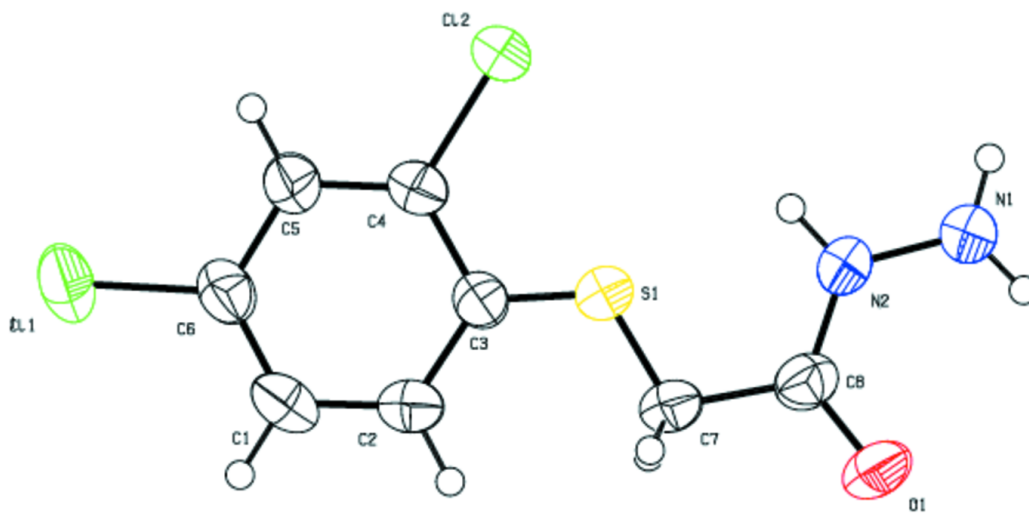
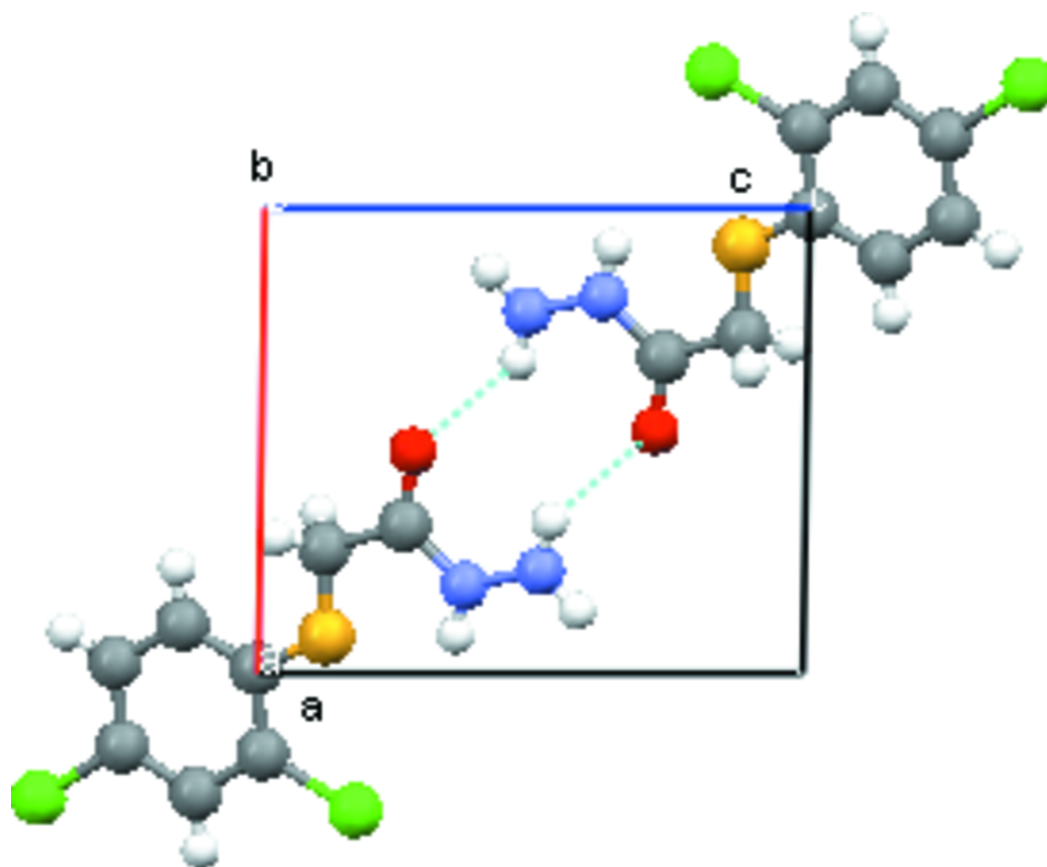
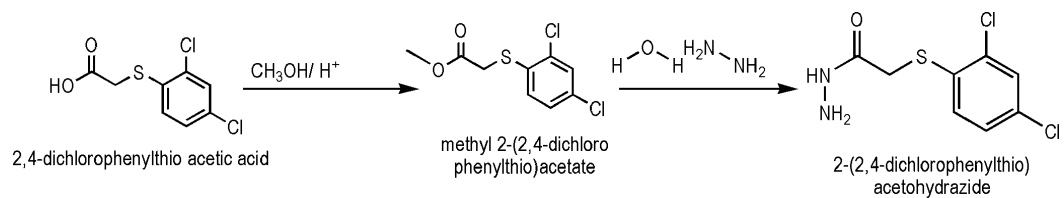


Fig. 3



Article

Fig. 4



Article retracted