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# 2-(2,4,6-Trichlorophenoxy)ethyl bromide

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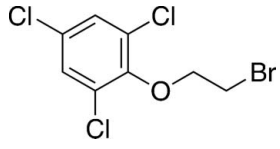
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.008 Å; $R$ factor = 0.045; $wR$ factor = 0.123; data-to-parameter ratio = 16.3.

In the title compound, $C_8H_6BrCl_3O$, there is a weak intramolecular C—H···Cl hydrogen bond involving the O bound methylene group. Intermolecular Cl···Cl contacts [3.482 (2) Å] are present in the crystal structure.

## Related literature

The title compound is used as an intermediate in the production of Prochloraz, a broad-spectrum imidazole fungicide widely used in gardening and agriculture. For the fungicidal properties of Prochloraz, see: Copping *et al.* (1984). For the preparation, see: Howard & Alfred (1982). For bond-length data, see: Allen *et al.* (1987).




## Experimental

### Crystal data

$C_8H_6BrCl_3O$
$M_r$ = 304.39
Triclinic, $P\overline{1}$
$a$ = 4.0550 (8) Å
$b$ = 8.6270 (17) Å
$c$ = 15.183 (3) Å
$\alpha$ = 90.73 (3)°
$\beta$ = 94.81 (3)°
$\gamma$ = 90.42 (3)°
$V$ = 529.21 (18) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 4.60 mm$^{-1}$
$T$ = 293 K
0.20 × 0.10 × 0.10 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.460, $T_{max}$ = 0.656
2215 measured reflections
1919 independent reflections
1280 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041
3 standard reflections
every 200 reflections
intensity decay: 1%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
$S$ = 1.01
1919 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.42 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C2—H2$A$···Cl3 | 0.97 | 2.81 | 3.276 (6) | 110 |

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2217).

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# supporting information

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## 2-(2,4,6-Trichlorophenoxy)ethyl bromide

**Jin-feng Yao, Wen-ge Yang, Xiao-lei Zhao, Lei Shen and Yong-hong Hu**

### S1. Comment

Prochloraz, *N*-propyl-*N*-[2-(2,4,6-trichlorophenoxy)-ethyl] -1*H*-imidazole-1-carboxamide, is a broad-spectrum imidazole fungicide (Copping *et al.*, 1984). As part of our studies in the synthesis of Prochloraz, the title compound (I), which is used as the key intermediate, has been synthesized. We report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). In the crystal structure, intramolecular C—H···Cl interactions (Table 1) may be effective in the stabilization of the structure.

### S2. Experimental

The title compound was prepared by following a reported procedure (Howard & Alfred, 1982). 2,4,6-Trichlorophenol (15.8 g ) and sodium hydroxide (4.8 g) were dissolved in 28 ml water and added dropwise to an excess of ethylene dibromide (75.6 g). The reaction mixture was heated under reflux for ten hours. The residue was extracted with 3 *x* 20 ml dichlormethane, and then methylene chloride phase was washed with water, dried and evaporated to dryness under reduced pressure. Fractionation under reduced pressure yielded the title compound as a colorless oil whaich was then cooled to give 18.1 g white solid (75.2%). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution of (I).

### S3. Refinement

H atoms were positioned geometrically with C—H = 0.93 and 0.97 Å for aromatic and methylene H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}$(H) = 1.2 times $U_{eq}$(C).

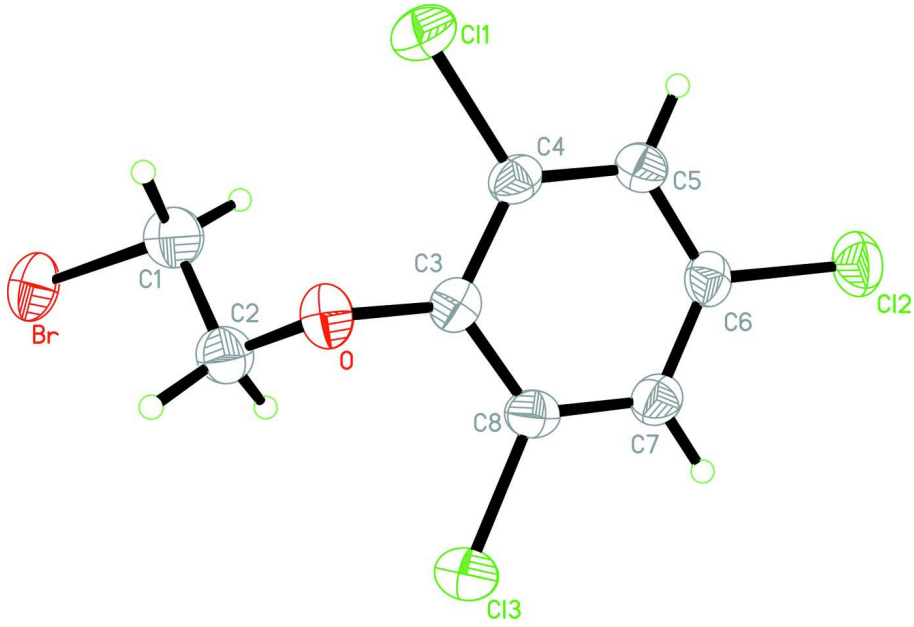


**Figure 1**

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at 30% probability levels.

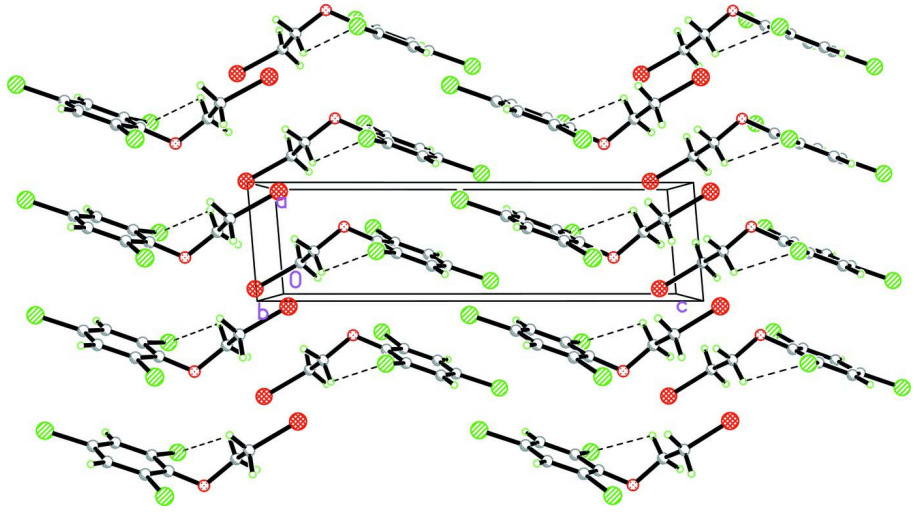


**Figure 2**

A packing diagram of the title compound. Intramolecular hydron bonds are shown as dashed lines.

**2-(2,4,6-Trichlorophenoxy)ethyl bromide**

*Crystal data*

$C_8H_6BrCl_3O$
$M_r = 304.39$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.0550\ (8)$ Å
$b = 8.6270\ (17)$ Å
$c = 15.183\ (3)$ Å
$\alpha = 90.73\ (3)°$
$\beta = 94.81\ (3)°$
$\gamma = 90.42\ (3)°$
$V = 529.21\ (18)$ Å$^3$
$Z = 2$
$F(000) = 296$
$D_x = 1.910$ Mg m$^{-3}$

Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 25 reflections
$\theta$ = 10–14°
$\mu$ = 4.60 mm$^{-1}$
$T$ = 293 K
Block, colorless
0.20 × 0.10 × 0.10 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.460, $T_{max}$ = 0.656
2215 measured reflections
1919 independent reflections
1280 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041
$\theta_{max}$ = 25.3°, $\theta_{min}$ = 1.4°
$h$ = 0→4
$k$ = −10→10
$l$ = −18→18
3 standard reflections every 200 reflections
intensity decay: 1%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
$S$ = 1.01
1919 reflections
118 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.42 e Å$^{-3}$

*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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br | 0.06298 (16) | 0.24181 (8) | −0.05453 (4) | 0.0696 (3) |
| O | 0.6382 (9) | 0.3089 (4) | 0.1806 (2) | 0.0537 (9) |
| Cl1 | 0.6363 (4) | −0.01264 (17) | 0.24134 (11) | 0.0689 (5) |
| Cl2 | 0.1372 (4) | 0.22953 (19) | 0.52835 (10) | 0.0707 (5) |
| Cl3 | 0.4167 (5) | 0.59982 (17) | 0.26392 (11) | 0.0777 (5) |
| C1 | 0.3028 (15) | 0.1966 (7) | 0.0606 (4) | 0.0651 (16) |
| H1A | 0.1585 | 0.1417 | 0.0977 | 0.078* |
| H1B | 0.4922 | 0.1318 | 0.0521 | 0.078* |
| C2 | 0.4125 (15) | 0.3430 (7) | 0.1030 (4) | 0.0609 (15) |
| H2A | 0.2235 | 0.4002 | 0.1208 | 0.073* |
| H2B | 0.5249 | 0.4058 | 0.0620 | 0.073* |
| C3 | 0.5043 (12) | 0.2915 (6) | 0.2586 (3) | 0.0436 (12) |

| | | | | |
|---|---|---|---|---|
| C4 | 0.4945 (12) | 0.1458 (6) | 0.2977 (4) | 0.0467 (13) |
| C5 | 0.3811 (13) | 0.1253 (6) | 0.3795 (3) | 0.0486 (13) |
| H5A | 0.3760 | 0.0273 | 0.4042 | 0.058* |
| C6 | 0.2752 (13) | 0.2530 (6) | 0.4241 (3) | 0.0476 (13) |
| C7 | 0.2808 (13) | 0.3971 (6) | 0.3887 (4) | 0.0506 (14) |
| H7A | 0.2071 | 0.4822 | 0.4195 | 0.061* |
| C8 | 0.3966 (13) | 0.4152 (6) | 0.3068 (4) | 0.0482 (13) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br | 0.0586 (4) | 0.0971 (5) | 0.0522 (4) | 0.0021 (3) | 0.0018 (3) | −0.0099 (3) |
| O | 0.045 (2) | 0.070 (2) | 0.046 (2) | 0.0014 (18) | 0.0040 (19) | 0.0043 (18) |
| Cl1 | 0.0775 (11) | 0.0580 (9) | 0.0722 (10) | 0.0176 (8) | 0.0128 (9) | −0.0101 (7) |
| Cl2 | 0.0801 (11) | 0.0855 (11) | 0.0482 (9) | 0.0115 (9) | 0.0131 (8) | 0.0051 (8) |
| Cl3 | 0.1140 (14) | 0.0486 (8) | 0.0695 (11) | −0.0029 (9) | 0.0020 (10) | 0.0059 (7) |
| C1 | 0.053 (4) | 0.069 (4) | 0.075 (4) | −0.002 (3) | 0.018 (3) | 0.002 (3) |
| C2 | 0.062 (4) | 0.058 (3) | 0.064 (4) | −0.001 (3) | 0.016 (3) | 0.004 (3) |
| C3 | 0.033 (3) | 0.052 (3) | 0.045 (3) | 0.001 (2) | −0.001 (2) | −0.003 (2) |
| C4 | 0.044 (3) | 0.045 (3) | 0.051 (3) | 0.008 (2) | −0.001 (3) | −0.007 (2) |
| C5 | 0.049 (3) | 0.046 (3) | 0.051 (3) | 0.004 (2) | 0.003 (3) | 0.003 (3) |
| C6 | 0.041 (3) | 0.059 (3) | 0.042 (3) | 0.005 (3) | −0.005 (2) | −0.001 (3) |
| C7 | 0.052 (3) | 0.050 (3) | 0.050 (3) | 0.009 (3) | −0.002 (3) | −0.004 (3) |
| C8 | 0.048 (3) | 0.041 (3) | 0.054 (3) | 0.001 (2) | −0.003 (3) | 0.001 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br—C1 | 1.973 (6) | C2—H2B | 0.9700 |
| O—C3 | 1.353 (6) | C3—C8 | 1.380 (7) |
| O—C2 | 1.464 (7) | C3—C4 | 1.398 (7) |
| Cl1—C4 | 1.731 (5) | C4—C5 | 1.373 (7) |
| Cl2—C6 | 1.737 (5) | C5—C6 | 1.376 (7) |
| Cl3—C8 | 1.733 (5) | C5—H5A | 0.9300 |
| C1—C2 | 1.459 (8) | C6—C7 | 1.361 (7) |
| C1—H1A | 0.9700 | C7—C8 | 1.375 (8) |
| C1—H1B | 0.9700 | C7—H7A | 0.9300 |
| C2—H2A | 0.9700 | | |
| | | | |
| C3—O—C2 | 117.4 (4) | C5—C4—C3 | 122.1 (5) |
| C2—C1—Br | 108.5 (4) | C5—C4—Cl1 | 119.2 (4) |
| C2—C1—H1A | 110.0 | C3—C4—Cl1 | 118.6 (4) |
| Br—C1—H1A | 110.0 | C4—C5—C6 | 118.5 (5) |
| C2—C1—H1B | 110.0 | C4—C5—H5A | 120.8 |
| Br—C1—H1B | 110.0 | C6—C5—H5A | 120.8 |
| H1A—C1—H1B | 108.4 | C7—C6—C5 | 121.4 (5) |
| C1—C2—O | 108.5 (5) | C7—C6—Cl2 | 119.5 (4) |
| C1—C2—H2A | 110.0 | C5—C6—Cl2 | 119.1 (4) |
| O—C2—H2A | 110.0 | C6—C7—C8 | 119.2 (5) |

| | | | |
|---|---|---|---|
| C1—C2—H2B | 110.0 | C6—C7—H7A | 120.4 |
| O—C2—H2B | 110.0 | C8—C7—H7A | 120.4 |
| H2A—C2—H2B | 108.4 | C7—C8—C3 | 122.1 (5) |
| O—C3—C8 | 122.7 (5) | C7—C8—Cl3 | 118.9 (4) |
| O—C3—C4 | 120.4 (4) | C3—C8—Cl3 | 118.9 (4) |
| C8—C3—C4 | 116.7 (5) | | |

| | | | |
|---|---|---|---|
| Br—C1—C2—O | −170.3 (3) | C4—C5—C6—C7 | 0.1 (8) |
| C3—O—C2—C1 | −90.6 (6) | C4—C5—C6—Cl2 | −179.0 (4) |
| C2—O—C3—C8 | −75.1 (6) | C5—C6—C7—C8 | −0.4 (8) |
| C2—O—C3—C4 | 110.3 (5) | Cl2—C6—C7—C8 | 178.7 (4) |
| O—C3—C4—C5 | 175.5 (5) | C6—C7—C8—C3 | 0.8 (8) |
| C8—C3—C4—C5 | 0.6 (7) | C6—C7—C8—Cl3 | −177.7 (4) |
| O—C3—C4—Cl1 | −3.4 (6) | O—C3—C8—C7 | −175.7 (5) |
| C8—C3—C4—Cl1 | −178.4 (4) | C4—C3—C8—C7 | −0.9 (8) |
| C3—C4—C5—C6 | −0.2 (8) | O—C3—C8—Cl3 | 2.8 (7) |
| Cl1—C4—C5—C6 | 178.8 (4) | C4—C3—C8—Cl3 | 177.7 (4) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2*A*···Cl3 | 0.97 | 2.81 | 3.276 (6) | 110 |