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# 2-Trichloromethylthio-1*H*-isoindole-1,3(2*H*)-dione (Folpet)

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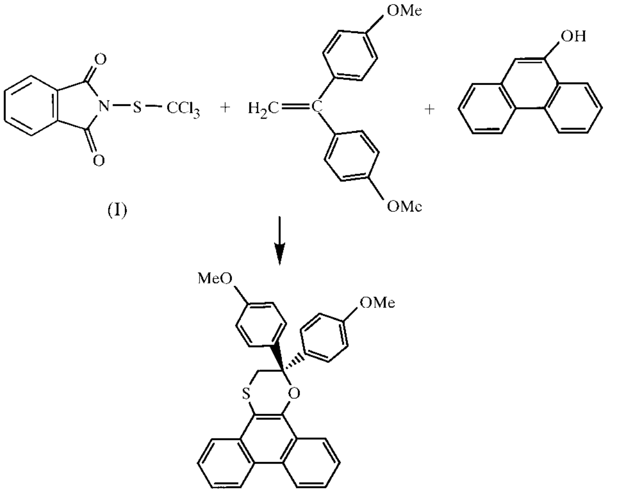
The title compound ($C_9H_4Cl_3NO_2S$), commonly known as Folpet, belongs to a group of phthalimides which function as fungicides or can be used in the laboratory as sulfurizing agents. The phthalimide moiety is slightly folded with a dihedral angle of 3.5 (4)°. The molecule participates in C—H···O and Cl···Cl intermolecular interactions.

## Comment

The title compound, (I), is a member of a class of *N*-(trichloromethylthio)phthalimides which function as useful fungicidal, insecticidal and germicidal agents (Hargreaves *et al.*, 1970). We were particularly interested in the utility of (I) as a convenient sulfurizing agent in the synthesis of dihydro-oxathiin complexes. For example, the reaction shown below gives the synthetic route for the preparation of 2,2-bis-(4-methoxyphenyl)-2,3-dihydrophenanthro[9,10-*b*]-1,4-dioxathiine using (I) (Carle, 1997).



The structure of (I) (Fig. 1) resembles other functionalized phthalimides, namely *N*-trichloromethylthio-3a,4,7,7a-tetrahydrophthalimide (Captan; Moss & Jacobson, 1981), *N*-(2-nitrophenylthio)phthalimide (Iwasaki & Masuko, 1986) and *N*-*tert*-butyldithiophthalimide (Mazhar-ul-Haque & Behforouz, 1979). The thiophthalimido group is slightly folded, with a dihedral angle of 3.5 (4)° between the N1/C2/C3/C8/C9 and C3–C8 planes, whereas nitrophenyl- and butyldithiophthalimide are more planar with corresponding angles of 1.4 and 1.5°, respectively. The S—N bond length [1.668 (5) Å] is shorter than similar bonds in the above related compounds [1.689 (3)–1.702 (2) Å], but is consistent with previously observed S—N single bonds (1.63–1.68 Å) exhibiting $\pi$ character (Iwasaki & Masuko, 1986).

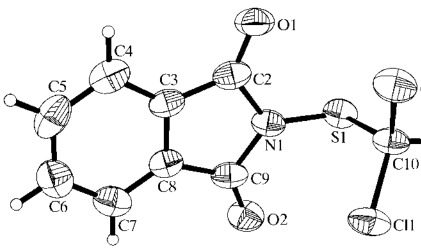


**Figure 1**
The structure of (I) showing 50% probability displacement ellipsoids and the atomic numbering scheme.

An intermolecular interaction of the C—H···O=C type (Fig. 2) is observed between the aromatic C atoms of the phthalimide moiety and the symmetry-related carbonyls [C4—H4···O1$^{i}$ 3.297 (7) Å and 153°; C7—H7···O2$^{ii}$ 3.619 (8) Å and 167°; symmetry codes: (i) $-x$, $1-y$, $1-z$; (ii) $-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$]. The H···O distances of 2.44 and 2.71 Å, respectively, are in the range that some consider to be moderate to weak non-conventional hydrogen bonding (Taylor & Kennard, 1982; Steiner, 1996; Jeffrey, 1997). Similar interactions are observed for Captan (C···O 3.400–3.445 Å and 133–145°; Moss & Jacobson, 1981), *N*-(2-nitrophenylthio)phthalimide (C···O 3.32–3.33 Å and 152–155°; Iwasaki & Masuko, 1986)

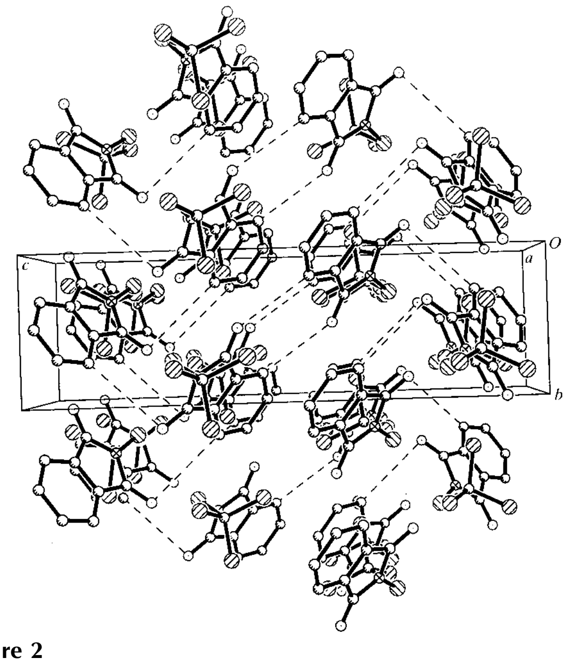


**Figure 2**
A packing diagram of (I) illustrating C···O interactions in the *bc* plane.

and *N-tert*-butyldithiophthalimide (C···O 3.53 Å and 153°; Mazhar-ul-Haque & Behforouz, 1979).

There is one short intermolecular interaction of 3.350 (3) Å between neighboring Cl atoms which is less than the van der Waals distance (Cl radius = 1.75 Å; Bondi, 1964)). Additionally, one borderline Cl···Cl interaction is observed with a distance of 3.529 (2) Å, which is slightly longer than the van der Waals distance. The corresponding closest contact distance between Cl atoms in Captan is 3.556 Å (Moss & Jacobson, 1981). In Folpet, the nearest-neighbor contact between Cl and S atoms is Cl3···S of 3.675 (2) Å.

## Experimental

A heptane solution of trichloromethanesulfenyl chloride was added dropwise to a stirred dimethylformamide solution of phthalimide and triethylamine producing (I) (Wunderly, 1972). Crystals of (I) were obtained by slow evaporation from $CH_2Cl_2$ at 298 K and was fully characterized by spectroscopic techniques. $^1$H NMR (250 MHz, $CDCl_3$): $\delta$ 7.88–7.93 (*m*, 2H), 8.02–8.07 (*m*, 2H) p.p.m.; $^{13}$C NMR (63 MHz, $CDCl_3$): $\delta$ 99.11, 124.65, 131.30, 135.44, 165.77 p.p.m.; IR ($CH_2Cl_2$): 3948 (*m*), 3692–3750 (*w*), 3054 (*vs*), 2987 (*s*), 2681 (*m*), 2410 (*m*), 2306 (*s*), 1798 (*w*), 1752 (*s*), 1721 (*w*), 1422 (*s*), 1259 (*vs*), 1150 (*m*), 1026 (*w*), 896 (*s*), 763 (*vs*), 711 (*vs*) cm$^{-1}$; UV/vis ($CH_2Cl_2$): $\lambda$ 239, 296 nm; HRMS (EI): *m*/*z* 295 ($M^+$), 260, 241, 150, 130, 114, 104, 79, 70; exact mass for $C_9H_4Cl_3NO_2S$ calculated 294.903, found 294.898; m.p. 448–449 K.

*Crystal data*

$C_9H_4Cl_3NO_2S$
$M_r = 296.54$
Monoclinic, $P2_1/c$
$a = 9.7666$ (19) Å
$b = 5.7576$ (12) Å
$c = 19.754$ (3) Å
$\beta = 94.905$ (14)°
$V = 1106.8$ (4) Å$^3$
$Z = 4$
$D_x = 1.780$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 7.5$–15.0°
$\mu = 0.996$ mm$^{-1}$
$T = 298$ (2) K
Rod, colorless
0.45 × 0.30 × 0.25 mm

*Data collection*

Siemens *P*3 diffractometer
$\theta$–$2\theta$ scans
2702 measured reflections
2554 independent reflections
1281 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\rm max} = 27.55°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 7$
$l = -25 \rightarrow 25$
3 standard reflections every 250 reflections intensity decay: negligible

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.157$
$S = 1.100$
2552 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 1.2199P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.40$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.52$ e Å$^{-3}$
Extinction correction: *SHELXTL*
Extinction coefficient: 0.022 (2)

Data collection: *P*3–*P*4/*PC* (Siemens, 1989); cell refinement: *P*3–*P*4/*PC*; data reduction: *XDISK* (Siemens, 1989); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1096). Services for accessing these data are described at the back of the journal.

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