

2-Trichloromethylthio-1*H*-isoindole-
1,3(2*H*)-dione (Folpet)A. Björn Carle, Jeanette A. Krause Bauer* and R. Marshall
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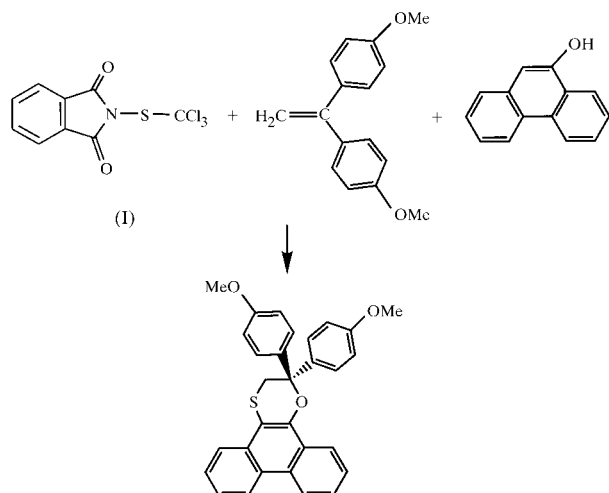
Received 2 August 1999

Accepted 13 October 1999

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 $3.5(4)^\circ$. The molecule participates in $C-H\cdots O$ and $Cl\cdots Cl$ 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-oathiin 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 &

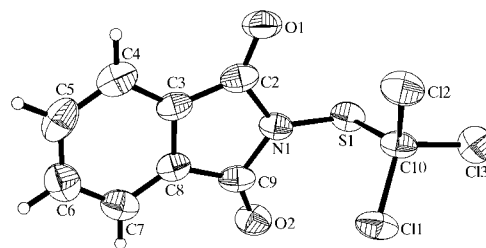


Figure 1

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

Behforouz, 1979). The thiophthalimido group is slightly folded, with a dihedral angle of $3.5(4)^\circ$ 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 π character (Iwasaki & Masuko, 1986).

An intermolecular interaction of the $C-H\cdots O=C$ type (Fig. 2) is observed between the aromatic C atoms of the phthalimide moiety and the symmetry-related carbonyls [$C4-H4\cdots O1^i$ $3.297(7)$ Å and 153° ; $C7-H7\cdots O2^{ii}$ $3.619(8)$ Å and 167° ; symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $-x, y-\frac{1}{2}, \frac{1}{2}-z$]. The $H\cdots O$ 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 ($C\cdots O$ 3.400 – 3.445 Å and 133 – 145° ; Moss & Jacobson, 1981), *N*-(2-nitrophenylthio)phthalimide ($C\cdots O$ 3.32 – 3.33 Å and 152 – 155° ; Iwasaki & Masuko, 1986)

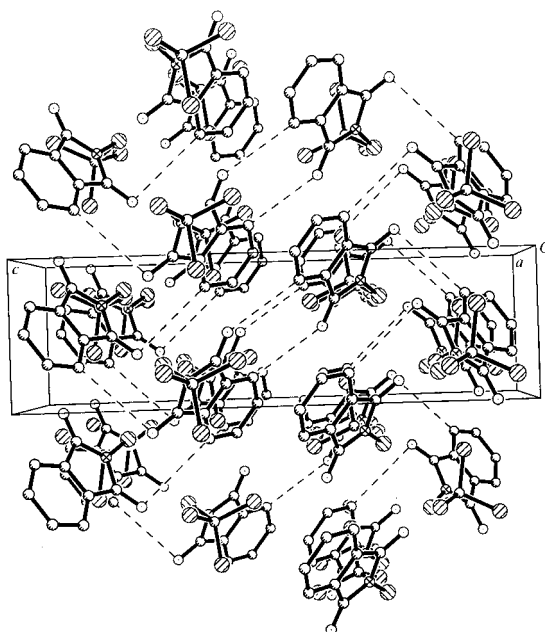


Figure 2

A packing diagram of (I) illustrating $C\cdots O$ interactions in the *bc* plane.

and *N*-tert-butylthiophthalimide ($C \cdots O$ 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 \cdots 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 \cdots S of 3.675 (2) Å.

Experimental

A heptane solution of trichloromethanesulfonyl 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 CH₂Cl₂ at 298 K and was fully characterized by spectroscopic techniques. ¹H NMR (250 MHz, CDCl₃): δ 7.88–7.93 (*m*, 2H), 8.02–8.07 (*m*, 2H) p.p.m.; ¹³C NMR (63 MHz, CDCl₃): δ 99.11, 124.65, 131.30, 135.44, 165.77 p.p.m.; IR (CH₂Cl₂): 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 (CH₂Cl₂): λ 239, 296 nm; HRMS (EI): *m/z* 295 (*M*⁺), 260, 241, 150, 130, 114, 104, 79, 70; exact mass for C₉H₄Cl₃NO₂S calculated 294.903, found 294.898; m.p. 448–449 K.

Crystal data

C₉H₄Cl₃NO₂S
M_r = 296.54
 Monoclinic, *P*₂₁/*c*
a = 9.7666 (19) Å
b = 5.7576 (12) Å
c = 19.754 (3) Å
 β = 94.905 (14)°
V = 1106.8 (4) Å³
Z = 4

D_x = 1.780 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 7.5–15.0°
 μ = 0.996 mm⁻¹
T = 298 (2) K
 Rod, colorless
 0.45 × 0.30 × 0.25 mm

Data collection

Siemens *P*3 diffractometer
 θ–2θ scans
 2702 measured reflections
 2554 independent reflections
 1281 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.035
 θ_{max} = 27.55°

h = 0 → 12
k = 0 → 7
l = –25 → 25
 3 standard reflections every 250 reflections
 intensity decay: negligible

Refinement

Refinement on *F*²
R [*F*² > 2σ(*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 $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.40 e Å⁻³
 Δρ_{min} = –0.52 e Å⁻³
 Extinction correction: *SHELXTL*
 Extinction coefficient: 0.022 (2)

Data collection: *P3–P4/PC* (Siemens, 1989); cell refinement: *P3–P4/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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