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2,6-Bis(phenylsulfanylmethyl)pyridinium-4-olate

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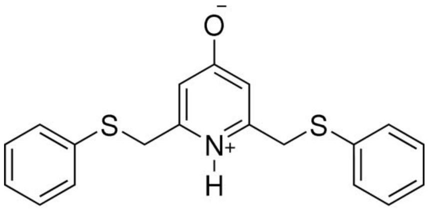
In the crystal structure of the title compound, $C_{19}H_{17}NOS_2$, hydrogen bonds link the molecules into infinite chains running along the *b* axis.



(I)

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.100
Data-to-parameter ratio = 18.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Experimental

All reagents were of analytical grade and were used without further purification. To a well stirred mixture at 268 K of sodium hydride (60% dispersion in mineral oil 50 mmol, 2g), thiophenol (40mmol, 4.4 g) and tetrahydrofuran (THF, 50 ml), a solution of 4-(tetrahydro-2-pyranoxy)-2,6-pyridinedimethyl ditosylate (10mmol, 5.5 g) in THF (20 ml) was added under a nitrogen atmosphere and the mixture was stirred for 8 h. The solvent was removed under reduced pressure. The resulting residue was dissolved in water (20 ml), extracted in diethyl ether (4 × 30 ml) and dried over Na_2SO_4. The solvent was then removed at reduced pressure. The crude product was purified by column chromatography through silica gel using ethyl acetate–hexane (1:4) as eluant, yielding 2,6-bis(phenylthiomethyl)-4-(tetrahydro-2-pyranoxy)pyridine (1.48 g, yield 35%). A mixture of 2,6-bis(phenylthiomethyl)-4-(tetrahydro-2-pyranoxy)pyridine (2 mmol, 0.85g), ethanol (10 ml) and acetic acid (0.2 ml) was refluxed for 4 h under a nitrogen atmosphere. The solvent was removed under reduced pressure and the resulting residue was crystallized from ethanol to afford the title compound (0.61 g, yield 90%). The compound was recrystallized from ethanol, yielding colourless crystals suitable for structural analysis.

Crystal data

$C_{19}H_{17}NOS_2$
$M_r = 339.46$
Monoclinic, $P2_1/c$
$a = 15.321$ (6) Å
$b = 6.654$ (2) Å
$c = 16.792$ (5) Å
$\beta = 90.361$ (15)°
$V = 1711.7$ (10) Å^3
$Z = 4$
$D_x = 1.317$ Mg m^{-3}
Mo *Kα* radiation
$\mu = 0.31$ mm^{-1}
$T = 298$ (2) K
Prism, colourless
0.31 × 0.28 × 0.25 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.905$, $T_{max} = 0.924$
16160 measured reflections
3933 independent reflections
3045 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.5°$

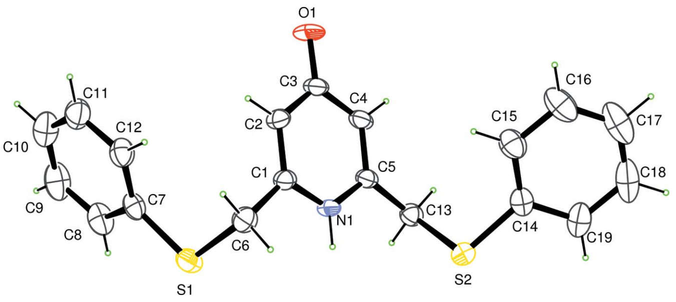


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level for non-H atoms.

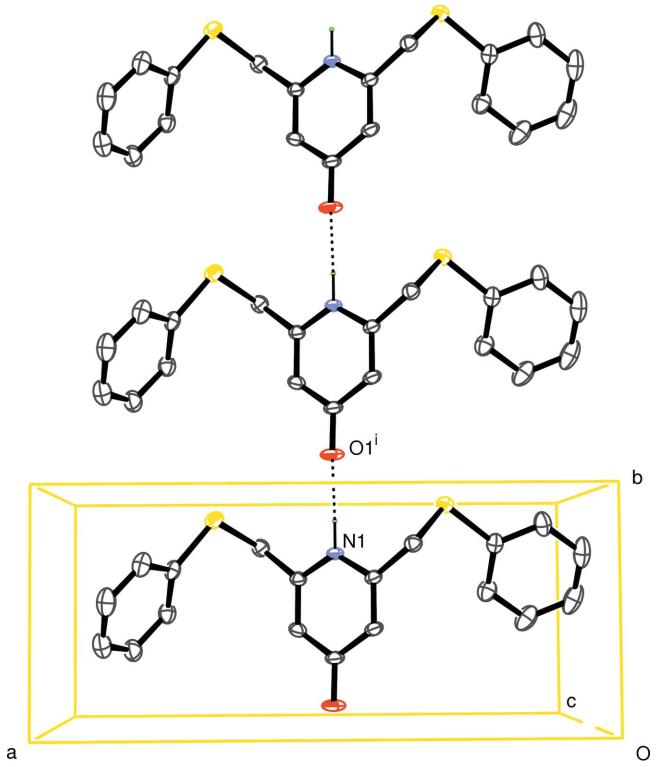


Figure 2
A view of the structure of (I), viewed down the *c* axis, showing hydrogen-bonded chains (dashed lines) running along the *b* axis. H atoms not involved in hydrogen bonding have been omitted.[Symmetry codes: (i) *x*, 1 + *y*, *z*.]

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.100$
$S = 1.06$
3933 reflections
213 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.211P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.009 (1)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.87 (2)	1.78 (2)	2.645 (2)	173.6 (15)
C13—H13A···Cg^{ii}	0.97	2.92	3.881 (2)	171

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, -y+1, -z$. *Cg* is the centroid of the C7–C12 ring.

Atom H1N was found in a difference Fourier map and refined freely. The H atoms of the methylene groups and of the aromatic ring were placed in calculated positions, with C—H distances of 0.97 and 0.93 Å, respectively, and were included in the final cycles of the least squares refinement as riding on their carrier atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *PROCESS-AUTO* (Rigaku,1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2003).

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