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C_{60} thiophene disolvate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.010 Å
Disorder in solvent or counterion
R factor = 0.077
wR factor = 0.227
Data-to-parameter ratio = 7.4

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

The crystal structure of fullerene thiophene disolvate, $C_{60}\cdot 2C_4H_4S$, at 120 K consists of C_{60} molecules in an arrangement close to primitive hexagonal, with disordered thiophene molecules occupying all of the trigonal-prismatic interstitial sites. The C_{60} molecules lie on inversion centres.

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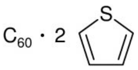
Comment

When fullerenes are crystallized from organic solvents, the resulting crystal structures commonly incorporate solvent molecules [see, for example, Collins *et al.* (1999) and Bürgi *et al.* (1994)]. Crystallization of C_{60} from thiophene gives the title compound, (I) (Fig. 1). At 120 K, the crystal structure consists of close-packed layers of C_{60} molecules, lying on inversion centres, in an arrangement close to primitive hexagonal (Fig. 2). The deviation from a true hexagonal arrangement lies in a displacement of approximately 1.54 Å parallel to [001] between adjacent layers of C_{60} molecules lying in (100), giving rise to a monoclinic lattice with $\beta = 98.817\ (4)^\circ$ (Fig. 3). The thiophene molecules occupy all of the trigonal-prismatic interstitial sites, with twofold orientational disorder. The structure is comparable to that of the bromobenzene solvate $C_{60}\cdot 2C_6H_5Br$ (Korobov *et al.*, 1998).



(I)

Experimental

Crystals of the title compound were obtained by slow evaporation of a saturated solution of C_{60} in thiophene.

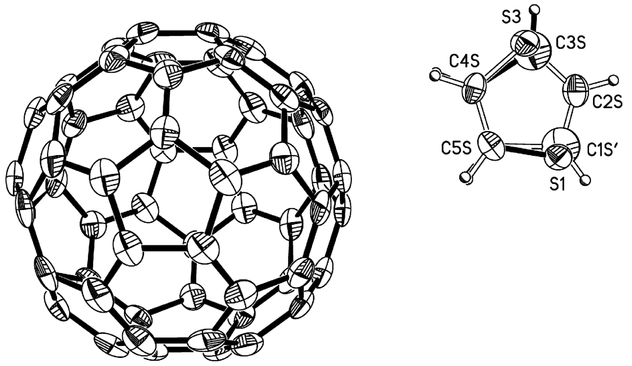


Figure 1
Structure of the molecules of $C_{60}\cdot 2C_4H_4S$, showing displacement ellipsoids at the 50% probability level. Atom labels have been omitted from the C_{60} molecule for clarity.

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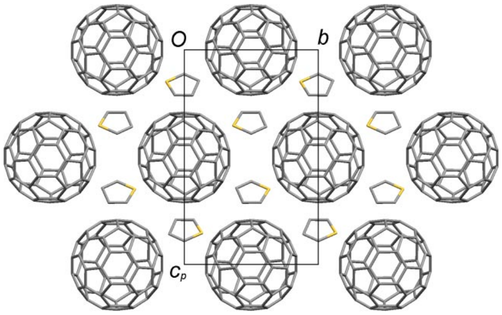

Figure 2
Projection along [100], showing the pseudo-hexagonal packing arrangement. Only one disorder component of the thiophene molecules is shown.

Crystal data

$C_{60}\cdot 2C_4H_4S$
$M_r = 888.86$
Monoclinic, $P2_1/c$
$a = 10.0300$ (9) Å
$b = 10.3536$ (6) Å
$c = 16.7649$ (13) Å
$\beta = 98.817$ (4)°
$V = 1720.4$ (2) Å^3
$Z = 2$

$D_x = 1.716$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6658 reflections
$\theta = 1.0$–23.0°
$\mu = 0.22$ mm^{-1}
$T = 120$ (2) K
Block, red
0.10 × 0.05 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.822$, $T_{\max} = 0.993$
12 159 measured reflections
2372 independent reflections

1513 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.118$
$\theta_{\max} = 23.0°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.077$
$wR(F^2) = 0.227$
$S = 1.24$
2372 reflections
322 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 5.4P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}

The C_{60} molecules are apparently well ordered and were treated with anisotropic displacement parameters, without restraints of any kind. The thiophene molecules are disordered and were modelled in two orientations related by a 180° rotation approximately about the C2—H2 bond. The molecules in the two orientations were restrained to be flat, with similar geometries, and their site-occupancy factors were refined to 0.61 (1) and 0.39 (1); these were constrained to 0.60 and 0.40 in the final cycles of refinement. Atoms C3S and C1S′, each overlapping the S atom of the other orientation of the thiophene molecule, were treated with isotropic displacement parameters fixed at 0.08 Å^2. H atoms bound to C atoms were positioned geometrically and allowed to ride during subsequent refinement [C—H = 0.95 Å and $U_{\rm iso}$(H) = 1.2 $U_{\rm eq}$(C)].

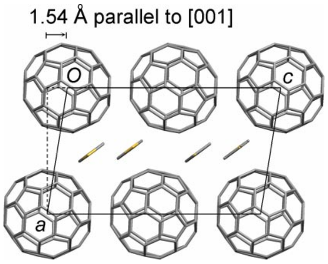

Figure 3
Projection along [010], showing the offset parallel to [001] of adjacent C_{60} layers in (100).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2000); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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