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A 2-propanol solvate of (*RS*)-phenylsuccinic acid

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Key indicators

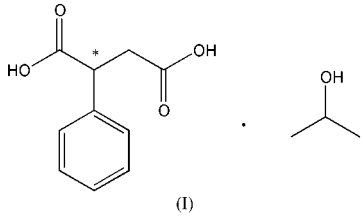
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.131
Data-to-parameter ratio = 17.8

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

A solvate of (*RS*)-phenylsuccinic acid (*RS*-PSA) has been obtained from 2-propanol (IPA). The compound, $C_{10}H_{10}O_4{\cdot}C_3H_8O$, crystallizes in the monoclinic crystal system (space group *C*2/*c*). It features molecules of the *S* form of the acid, one of whose carboxy groups is connected to one carboxy group of a molecule of the opposite chirality *via* two hydrogen bonds. The second carboxy group of the *S* molecule is connected to two other *S* molecules *via* the OH groups of two 2-propanol solvent molecules.

Comment

In addition to polymorphs, which are commonly encountered for many organic compounds, pseudo-polymorphs, such as hydrates and solvates, can also be crystallized from solution. Unexpected hydrates or solvates can complicate the manufacturing of the desired crystal modification, and therefore as many metastable crystal modifications as possible should be identified for a given compound. To the best of our knowledge, no hydrates or other solvates of either racemic or enantiomerically pure phenylsuccinic acid have been previously reported. This paper reports the crystal structure of a racemic 2-propanol (IPA) solvate of phenylsuccinic acid (PSA), (I), which is stable only as long as it is in contact with the saturated solution in the temperature range 258–289 K.



(I)

The structure of the title compound contains one independent molecule of (*S*)–PSA, whose geometry is unexceptional (see Fig. 1). One carboxy group of this molecule is connected to one carboxy group of a crystallographically equivalent molecule of (*R*)–PSA, *via* two hydrogen bonds, as can be seen in Fig. 2. These two acid molecules are related by an inversion centre between the two carboxy groups.

The second carboxy group of the (*S*)-PSA molecule is connected to two molecules of IPA, one of which functions as a donor, the other as an acceptor. These two IPA molecules are, in turn, connected to one molecule of (*S*)-PSA. The lengths and angles of the hydrogen bonds are listed in Table 1. They are in the same range as those found in the structures of the unsolvated (*S*)- and (*RS*)-PSA [(*S*)–PSA: Fischer & Profir (2003*a*); (*RS*)-PSA: Fischer & Profir (2003*b*)].

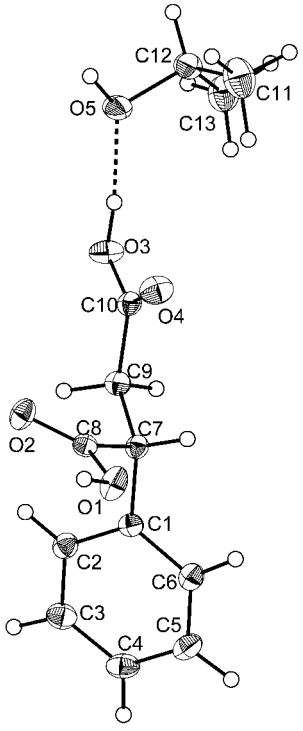


Figure 1
The molecules of (*S*)-PSA and IPA in the asymmetric unit of the structure of (I). Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

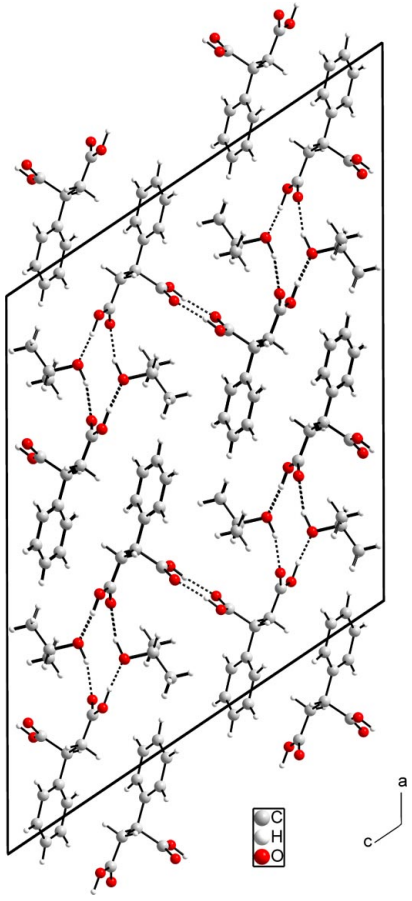


Figure 2
The unit cell contents of (I), viewed along *b*. Hydrogen bonds are shown as dashed lines.

Experimental

Large single crystals of the IPA solvate were obtained by preparing solutions of initial concentrations of 160–212 g/kg IPA, that are slightly supersaturated at 273 K. All solutions were completely dissolved, filtered by a 0.2 μm PTFE membrane filter and kept at 343 K for at least 10 min, with continuous stirring. The solutions were cooled to 273 K and seeded with racemic PSA crystals. To prevent any large nucleation events, the stirring was immediately stopped upon seeding. The temperature in the crystallizer was then lowered stepwise by 1 K approximately every 4 h. Despite the use of different crystal modifications of PSA as seeds, the IPA solvate reported in this paper was always obtained as the crystallization product. After 1 d, suitable crystals were removed from the solution and transferred immediately to the cold N_2 flow (150 K) of the diffractometer. This procedure was necessary because crystals of the solvate decompose quickly, with loss of IPA, on removal from the solution.

Crystal data

$C_{10}H_{10}O_4{\cdot}C_3H_8O$
$M_r = 254.28$
Monoclinic, $C2/c$
$a = 26.46\ (1)$ Å
$b = 5.467\ (2)$ Å
$c = 21.477\ (6)$ Å
$\beta = 123.93\ (3)^\circ$
$V = 2577.8\ (18)$ Å^3
$Z = 8$
$D_x = 1.310$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 87 reflections
$\theta = 4.6$–27.5°
$\mu = 0.10$ mm^{-1}
$T = 150$ K
Block, colourless
0.40 × 0.20 × 0.15 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: none
9953 measured reflections
2904 independent reflections
1868 reflections with $I > 2\sigma(I)$
$R_{int} = 0.083$
$\theta_{max} = 27.5^\circ$
$h = -32 \rightarrow 34$
$k = -5 \rightarrow 7$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.131$
$S = 1.08$
2904 reflections
163 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0296P)^2 + 5.2191P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O2^{i}	0.89	1.78	2.668 (2)	173
O3—H3O···O5	0.95	1.67	2.587 (2)	163
O5—H5O···O4^{ii}	0.95	1.82	2.743 (2)	163

Symmetry codes: (i) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

All H atom were located in a difference Fourier map and were refined using a riding model, with U_{iso} values of $1.2U_{eq}$ of the non-H atom to which they were attached. C—H = 0.91–1.08 Å and O—H = 0.89–0.95 Å.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX/LSQ* (Duisenberg, 1992); data reduction: *EvalCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *maXus* (Mackay *et al.*, 1999).

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