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(*RS*)-Phenylsuccinic acid

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(*RS*)-Phenylsuccinic acid, $C_{10}H_{10}O_4$, crystallizes with two molecules of the acid in the asymmetric unit. In the crystal structure, the carboxyl groups of each acid molecule are connected to those of adjacent molecules *via* hydrogen bonds; each molecule is connected to two other molecules, forming chains. Crystals of (*RS*)-phenylsuccinic acid are twinned. Data for one twin domain could be obtained and the structure could be solved with satisfactory results.

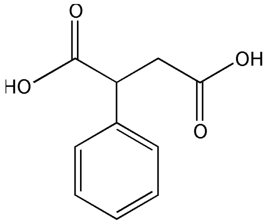
Key indicators

Single-crystal X-ray study
$T = 100$ K
Mean σ(C–C) = 0.005 Å
R factor = 0.056
wR factor = 0.133
Data-to-parameter ratio = 13.6

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

Comment

Phenylsuccinic acid (PSA) is used as a resolving agent in the classical resolution of pharmaceuticals (Bayley & Vaidya, 1992; Kozma, 2002). In order to study and understand the solid-state properties of PSA and its interactions with solvents or with other common reagents, it is essential to know the cystal structures of both the pure enantiomer and the racemate. Generally, there are three classes of racemates in the solid state (Jacques *et al.*, 1994). Two classes contain both enantiomers mixed in the crystal lattice: the racemic compounds that have an ordered, alternating (*R*)- and (*S*)-enantiomer distribution in the structure and the solid solutions which have both enantiomers randomly mixed in the lattice. The third class of racemates, the racemic conglomerate, has an identical crystal lattice to that of the pure enantiomer. This means that, although the whole crystal mass is racemic, each crystal itself is enantiomerically pure. Most chiral substances form a racemic compound, while approximately 5–10% form racemic conglomerates. The racemic conglomerate can be separated by direct seeding crystallization methods.



(I)

The racemic compound, on the other hand, must be separated by other methods such as diastereomeric salt formation, chromatography or kinetic resolution. The stability of racemic compounds *versus* conglomerates has been extensively discussed in the literature. Often, hydrogen bonds within the structures are identified as the key factor influencing the stability of the structures (Böcksei *et al.*, 1996; Brock, 1996; Kinbara *et al.*, 1996; Li *et al.*, 1999). Since the structures of neither (*S*)- nor (*RS*)-PSA were known, we grew crystals of both compounds in order to determine to which class of

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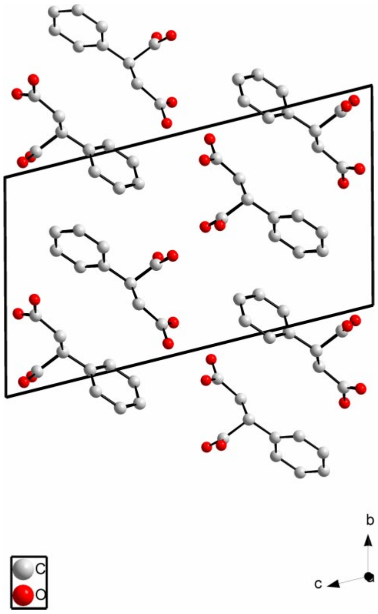

Figure 1
The unit cell contents of (*RS*)-PSA, viewed along *a*. H atoms have been omitted.

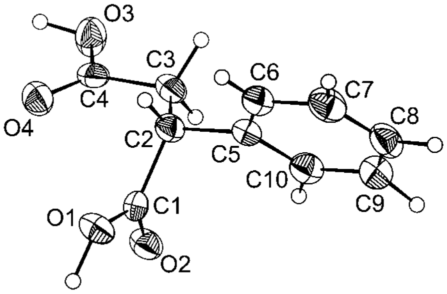

Figure 2
One of the two crystallographically independent molecules in the structure of (*RS*)-PSA. Displacement ellipsoids are drawn at the 70% probability level.

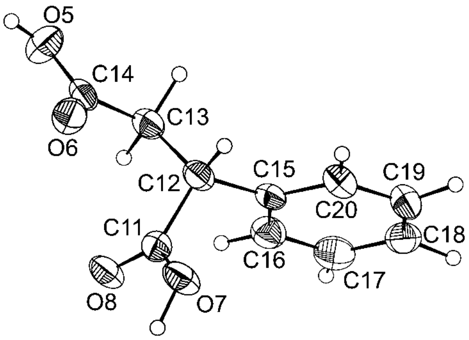

Figure 3
The second of the two crystallographically independent molecules in the structure of (*RS*)-PSA. Displacement ellipsoids are drawn at the 70% probability level.

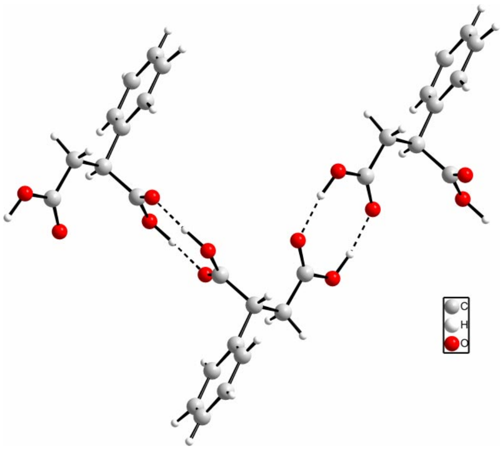

Figure 4
The chains formed by the molecules of (*RS*)-PSA. Only the carboxy H atoms are drawn. Hydrogen bonds are indicated by dashed lines.

racemates PSA belongs, and how the acid molecules are arranged in each structure. The structure of (*S*)-PSA has been published earlier (Fischer & Profir, 2003). The structure of (*RS*)-PSA, (I), is presented here. Fig. 1 shows the unit cell of (*RS*)-PSA in a view along the crystallographic *a* axis. The structure contains two molecules of (*R*)-PSA in the asymmetric unit; these are shown in Figs. 2 and 3. Since the structure is centrosymmetric, the opposite enantiomer of each molecule is generated. Thus, (*RS*)-PSA belongs to the first class of racemates mentioned above, the racemic compounds with an ordered distribution of the molecules. In (*RS*)-PSA, each (*R*)-molecule is connected to two adjacent (*S*)-molecules *via* hydrogen bonds and *vice versa*. These hydrogen bonds are built in such a way that both carboxyl groups bind to one carboxyl group of the other molecule. In this way, chains of molecules are formed as shown in Fig. 4. There are two crystallographically different chains in this structure, formed by the two different molecules in the asymmetric unit. The geometry of the molecules is unexceptional, although one quite close H···H contact of 2.03 Å is observed. This is, however, still slightly larger than the sum of the van der Waals radii (2.0 Å) given by Baur (1972).

Experimental

Crystals were grown from aqueous solutions by dissolving the purchased material (Fluka, >99%) in pure, distilled and deionized water at room temperature. The clear solutions were evaporated to dryness under low-pressure conditions at room temperature, yielding twinned crystals of (*RS*)-PSA.

Crystal data

$C_{10}H_{10}O_4$
$M_r = 194.19$
Triclinic, $P\overline{1}$
$a = 5.428\ (12)$ Å
$b = 10.368\ (6)$ Å
$c = 17.625\ (9)$ Å
$\alpha = 102.82\ (5)^\circ$
$\beta = 94.10\ (14)^\circ$
$\gamma = 100.50\ (9)^\circ$
$V = 944.\ (2)$ Å^3

$Z = 4$
$D_x = 1.366$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 154 reflections
$\theta = 4.2$–21.4°
$\mu = 0.11$ mm^{-1}
$T = 100$ K
Needle, colourless
0.30 × 0.15 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
14 054 measured reflections
3445 independent reflections
2223 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.075$
$\theta_{\rm max} = 27.5°$
$h = -6 \rightarrow 6$
$k = -12 \rightarrow 13$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.133$
$S = 1.03$
3445 reflections
253 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.7275P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.29$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.29$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1O$\cdots$O2$^{\rm i}$	1.03	1.63	2.651 (3)	171
O3—H3O$\cdots$O4$^{\rm ii}$	0.98	1.65	2.620 (4)	171
O5—H5O$\cdots$O6$^{\rm iii}$	0.87	1.75	2.612 (3)	170
O7—H7O$\cdots$O8$^{\rm iv}$	0.99	1.64	2.625 (3)	172

Symmetry codes: (i) $2 - x, 1 - y, 1 - z$; (ii) $2 - x, 2 - y, 1 - z$; (iii) $1 - x, 1 - y, -z$; (iv) $2 - x, 2 - y, -z$.

Reflection data for one twin domain were obtained using *EvalCCD* (Duisenberg, 1998). Orientation matrices for both twin domains were determined. During integration, all overlapping reflections were removed. One fifth of the observable reflections were lost due to overlap. All H atoms were located from a difference Fourier map. They were refined with a riding model, with $U_{\rm iso}$ equal to $1.2U_{\rm eq}$ of the non-H atom to which they are attached.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *EvalCCD* (Duisenberg, 1998); data reduction: *EvalCCD*; 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.*, 1998).

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