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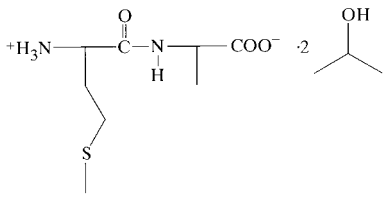
The crystal structure of the title compound, $C_8H_{16}N_2O_3S{\cdot}2C_3H_8O$, is divided into hydrophobic and hydrophilic layers. Two peptide molecules in the asymmetric unit are related by pseudo-translational symmetry along the *a* axis, as are two of the four 2-propanol molecules. The last two 2-propanol molecules in the asymmetric unit have different relative orientations and hydrogen-bond interactions.

Comment

Like several other dipeptides with two hydrophobic residues (Görbitz, 1999*a*,*b*, and references therein), the crystal structure of the title compound, (I), is divided into hydrophobic and hydrophilic layers, but the specific molecular arrangement is different from those observed previously. The two dipeptide molecules *A* and *B* in the asymmetric unit are stacked in an alternating sequence along the *a* axis with pseudo-translational symmetry. The geometries of the L-Met residues of *A* an *B* are almost identical, and both side chains are in the *trans,-trans,gauche-* conformation for χ^1, χ^2 and χ^3. The general pseudo-translational symmetry along the *a* axis includes the two 2-propanol solvent molecules *C* and *F*, but not *D* and *E* which have different orientations.



(I)

As expected, two of the three amino H atoms of each peptide molecule are engaged in hydrogen bonding in the hydrophilic layers, while 2-propanol molecules *C* and *F*, which are very similarly embedded in the hydrophobic layers, serve the important roles as acceptors for the remaining amino H atoms. The 2-propanol molecules *C*, *D* and *F* are donors in O—H···O—H interactions, but molecule E is unique in donating its hydroxylic H atom to a carboxylate group. The acceptor atom, O2*B*, is made available through a rotation of the carboxylate group compared with peptide molecule *A*, with N2—C6—C8—O2 = 11.5 (6)° compared to −13.2 (7)° for A. Unlike O2*A*, O2*B* can then no longer accept an amino H atom, which is instead accepted by O3*B*.

The unit cell for the thin needle-shaped crystals also obtained in the crystallization experiments has a = *b* = 14.2459 (12) Å, *c* = 9.7152 (11) Å, $\alpha = \beta = 90°$ and $\gamma = 120°$. An unambiguous determination of the space group was not possible, but we suspect that the structure is closely related to the hexagonal $P6_1$ structures of L-Val-L-Ala (Görbitz & Gundersen, 1996), L-Val-L-Val and L-Ala-L-Ile (Görbitz, 2000).

Experimental

Crystals of the title compound were prepared by vapour diffusion of 2-propanol into 30 µl of an aqueous solution of the dipeptide. At low initial solute concentration (0.16–0.32 mg in 30 µl of water), bundles of exceedingly thin needles were formed which were too small for collection of X-ray data (<0.01 mm), but permitted determination of the unit cell. A second polymorph in the shape of ultrathin plates appeared (together with the needles) when higher concentrations were used. The plates are soft and easily bent, and can not be cut without inflicting substantial damage. They furthermore decay within a few s when exposed to air. The specimen used for data collection was obtained from a test tube containing 0.64 mg of the peptide, and was transferred to a drop of oil before being attached to the glass rod. Thin plates were also obtained with 1-propanol as precipitating agent, but no unit cell was found for any of the crystals tested. 2-Butanol, on the other hand, gave very thin needles, while methanol and ethanol failed to give precipitates in the diffusion experiments.

Crystal data

$C_8H_{16}N_2O_3S{\cdot}2C_3H_8O$
M_r = 340.48
Monoclinic, $P2_1$
a = 9.9277 (2) Å
b = 16.4556 (3) Å
c = 12.4895 (2) Å
β = 97.594 (1)°
V = 2022.47 (6) Å^3
Z = 4
D_x = 1.118 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 4393 reflections
θ = 2–25°
μ = 0.181 mm^{-1}
T = 150 (2) K
Plate, colourless
0.80 × 0.48 × 0.01 mm

Data collection

Siemens SMART CCD diffractometer
Sets of exposures each taken over 0.3° ω rotation scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
T_{min} = 0.865, T_{max} = 0.998
14 839 measured reflections
6911 independent reflections
4437 reflections with $I > 2\sigma(I)$
R_{int} = 0.0933
θ_{max} = 25.02°
h = −11 → 11
k = −19 → 19
l = −14 → 14

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.202
S = 1.010
6911 reflections
448 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0400P)^2 + 1.5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.014
$\Delta\rho_{max}$ = 0.527 e Å^{-3}
$\Delta\rho_{min}$ = −0.301 e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0070 (16)
Absolute structure: Flack (1983), 3230 Friedel pairs
Flack parameter = −0.01 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A···O3B	0.91	1.88	2.734 (5)	155
N1A—H2A···O3A^{i}	0.91	1.80	2.706 (5)	176
N1A—H3A···O1F	0.91	1.96	2.790 (7)	151
N2A—H4A···O1B^{ii}	0.88	2.61	3.410 (5)	151
C1A—H11B···O1B^{ii}	1.00	2.18	3.176 (5)	171
N1B—H1B···O2A^{iii}	0.91	1.83	2.735 (5)	175
N1B—H2B···O3B^{iv}	0.91	1.97	2.813 (5)	153
N1B—H3B···O1C	0.91	1.87	2.772 (6)	174
N2B—H4B···O1A^{i}	0.88	2.19	3.044 (5)	165
C1B—H11A···O1A^{i}	1.00	2.40	3.307 (5)	150
O1C—H1C···O1D^{iii}	0.85	1.91	2.734 (6)	163
O1D—H1D···O1E^{ii}	0.85	1.92	2.768 (6)	176
O1E—H1E···O2B	0.85	1.82	2.666 (5)	177
O1F—H1F···O1E	0.85	2.09	2.748 (7)	133

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

The data collection nominally covered over a hemisphere of reciprocal space by a combination of three sets of exposures with the detector set at $2\theta = 29°$ and a 4.95 cm crystal-to-detector distance.

Displacement ellipsoids for the solvent molecule are quite large, and for molecules *C*, *E* and *F* the disorder has been resolved into a major and a minor position, with occupancies 0.68 (2):0.32 (2), 0.896 (13):0.104 (13) and 0.806 (7):0.194 (7), respectively. For *C* and *E*, the disorder arises from a rotation around the O1—C2 bond, while for *F*, O1 and C2 are also shifted to alternative positions.

Heavy atoms were refined anisotropically, except for the O and C atoms of the least populated positions of the disordered 2-propanol molecules *C*, *E* and *F*, which were refined isotropically (C) or assigned U_{iso} values equivalent to U_{eq} for the corresponding atom in the most populated position. A mild *SHELXTL* (Sheldrick, 1997) *SAME* constraint (s1 = 0.006 and s2 = 0.009) was used for the geometry of the two peptide molecules, and similar commands ($s1$ = 0.008 and $s2$ = 0.012) for the geometry of all 2-propanol molecules. H atoms were placed geometrically and refined with a riding model which included free rotation for amino groups and methyl groups with occupancy greater than 0.5. Hydroxylic H atoms were constrained to have tetrahedral geometry and rotated so as to minimize the distance to the closest acceptor, but with deviations from perfect staggered positions not exceeding 30°. U_{iso} values were constrained to be $1.2U_{eq}$ of the carrier atom, or $1.5U_{eq}$ for methyl, hydroxyl and amino groups.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Sheldrick, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997).

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