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(*R*)-1-Phenylethylammonium *N*-tetradecanoyl-L-phenylalaninate monohydrate

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.005 Å
R factor = 0.068
wR factor = 0.151
Data-to-parameter ratio = 10.7

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

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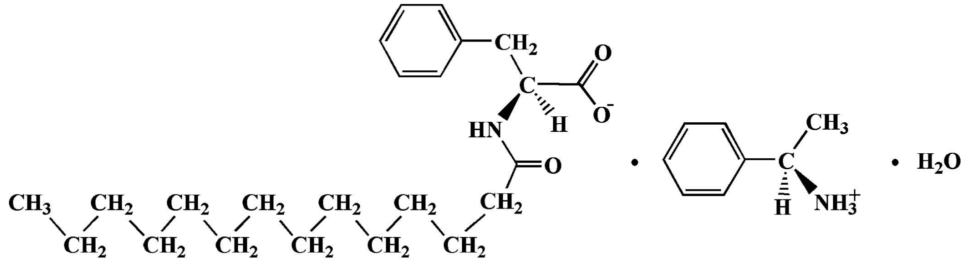
The title compound, $C_8H_{12}N^+\cdot C_{23}H_{36}NO_3^-\cdot H_2O$, has one molecule of *N*-tetradecanoyl-L-phenylalanine and one molecule of (*R*)-1-phenylethylamine as a diastereoisomeric salt, and a water molecule in the asymmetric unit. In the crystal structure, the packing of the molecules is stabilized by N—H···O and O—H···O hydrogen bonds involving the amide, ammonium and carboxylate groups, and the solvent water molecule.

Comment

N-Acyl amino acids are generally formed by condensation of amino acids with fatty acids and produce water-soluble anionic surfactants when their acid groups are neutralized with a range of bases. These compounds are widely used as additives, dispersants and detergents in a variety of industrial and commercial applications due to their biodegradability and low toxicity (Takehara, 1984). There are also growing concerns over the molecular associations of chiral surfactants (Shinitzky & Haimovitz, 1993; Parazak *et al.*, 1994; Ohta *et al.*, 2003) and their applications to chiral analytical techniques such as electrophoresis (Taurus *et al.*, 2003). When *N*-acyl amino acids are neutralized with optically active bases, diastereoisomeric surfactant salts are formed. Structural studies on these diastereomeric salts may provide the basis for understanding the mechanism of chiral discrimination of *N*-acyl amino acid surfactants. In this work, we have prepared a diastereomeric salt, (I), from *N*-tetradecanoyl-L-phenylalanine and (*R*)-1-phenylethylamine as a model of chiral discrimination of *N*-acyl amino acids and determined its structure.



(I)

The conformation of the tetradecanoyl chain is as most often found for larger alkanes, *i.e.* staggered with the largest substituents at any C—C bond antiperiplanar with respect to each other (Fig. 1). The C—C bond lengths within the chain range from 1.518 (6) to 1.533 (5) Å and the angles from 112.8 (3) to 114.1 (3)°. These values are similar to those found in a related fatty acid (Amai *et al.*, 1998).

The molecular packing is such that the hydrocarbon chains lie side by side, with neighboring hydrophilic heads pointing in opposite directions. The hydrophilic heads are connected by a network of hydrogen bonds involving the carboxylate group of *N*-tetradecanoyl-L-phenylalanine, the ammonium group of (*R*)-1-phenylethylamine and the water molecule (Table 1 and Fig. 2). The amide linkages of *N*-tetradecanoyl-L-phenylalanine molecules form an N—H···O hydrogen-bonded chain running along the *b* axis.

Experimental

N-Tetradecanoyl-L-phenylalanine was prepared by the reaction of l-phenylalanine with tetradecanoyl chloride as described previously (Miyagishi & Nishida, 1978) and was recrystallized from an acetone/methanol solution three times. The purity was checked by HPLC and DSC and by observing no minimum on the surface tension *vs* concentration curves at 298.15 K. The title compound was obtained by neutralization of *N*-tetradecanoyl-L-phenylalanine with equimolar of (*R*)-1-phenylethylamine in methanol. After evaporation of the solvent, the residue was recrystallized from a methanol/water solution to obtain suitable crystals for X-ray diffraction (m.p. 374–376 K).

Crystal data

$C_8H_{12}N^+\cdot C_{23}H_{36}NO_3^-\cdot H_2O$
M_r = 514.73
Monoclinic, $P2_1$
a = 10.920 (4) Å
b = 4.9902 (17) Å
c = 27.926 (10) Å
β = 94.085 (8)°
V = 1517.9 (9) Å^3
Z = 2
D_x = 1.126 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5442 reflections
θ = 7.2–27.5°
μ = 0.07 mm^{-1}
T = 123.2 K
Plate, colorless
0.50 × 0.20 × 0.10 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
ω scans
Absorption correction: none
16351 measured reflections
3774 independent reflections
2920 reflections with $I > 2\sigma(I)$
R_{int} = 0.100
θ_{max} = 27.5°
$h = -13 \rightarrow 14$
$k = -6 \rightarrow 6$
$l = -35 \rightarrow 35$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.151
S = 1.06
3774 reflections
352 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H49···O4^{i}	0.88 (5)	2.01 (5)	2.857 (4)	164 (4)
O4—H50···O1^{ii}	1.05 (4)	1.64 (4)	2.683 (4)	176 (4)
N1—H1···O3^{iii}	0.94 (4)	1.98 (4)	2.913 (4)	170 (4)
N2—H37···O2^{ii}	0.88 (5)	1.97 (5)	2.845 (4)	172 (3)
N2—H38···O2^{i}	0.96 (4)	1.83 (4)	2.747 (4)	158 (4)
N2—H39···O4^{i}	0.86 (4)	1.96 (4)	2.810 (4)	172 (4)

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

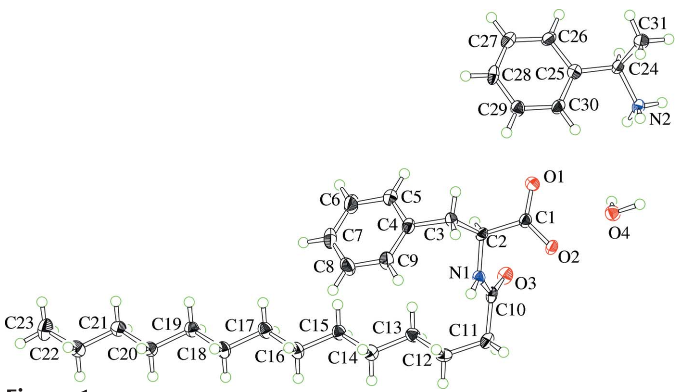


Figure 1
View of the asymmetric unit of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

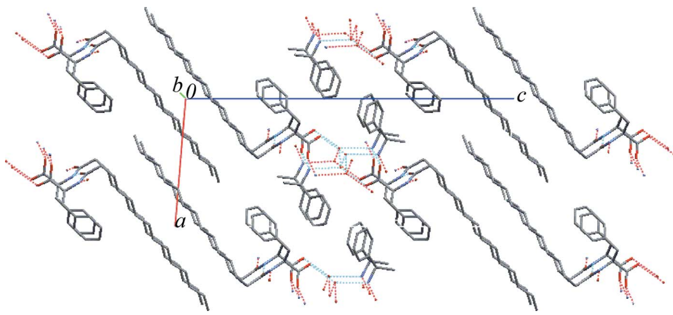


Figure 2
The molecular packing of (I), viewed along the *b* axis. Dashed lines indicate the hydrogen-bonding interactions. H atoms have been omitted for clarity.

H atoms involved in hydrogen bonds were located in a difference map, and their positional parameters were refined. The U_{iso}(H) values were set at 1.2U_{eq}(N or O). Other H atoms were included in calculated positions using a riding-model approximation, with C—H distances in the range 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C). Due to the absence of any significant anomalous scatterers, Friedel pairs were merged before the final refinement. The absolute configuration has been assigned on the basis of the known configuration of the reagents.

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2000); program(s) used to solve structure: *MITHRIL*90 (Gilmore, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*III (Johnson & Burnett, 1996) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *TEXSAN*.

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