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L-Phenylalanine–nitric acid (2/1)

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In the title compound, $2\,C_9H_{11}NO_2\cdot H^+\cdot NO_3^-$, both phenylalanine residues have a *gauche* I conformation. The aggregation of the hydrophilic zone is along $z = 0$ and the hydrophobic zone is sandwiched between two such layers at $z = 0$ and $z = 1$.

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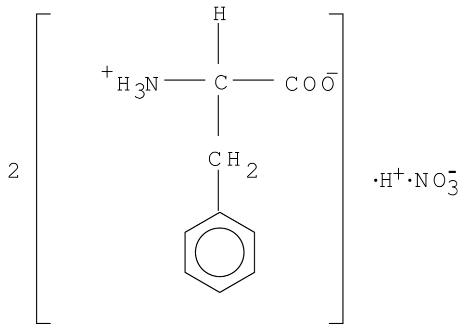
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.012 Å
H-atom completeness 70%
R factor = 0.069
wR factor = 0.220
Data-to-parameter ratio = 7.6

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

Comment

The crystal structures of L-phenylalanine hydrochloride (Gurskaya & Vainshtein, 1963; Al-Karaghouli & Koetzle, 1975), L-phenylalanine L-phenylalaninium formate (Gorbitz & Etter, 1992), bis(L-phenylalanine) sulfate monohydrate (Nagashima *et al.*, 1992) and L-phenylalanine L-phenylalaninium perchlorate (Srinivasan & Rajaram, 1997) have been reported. Even though the title compound, (I), has been studied (Srikrishnan *et al.*, 1984), no structural data are available as the above publication does not contain the atomic coordinates and has a misprint in one of the cell dimensions. Therefore, in the present work, an independent X-ray diffraction study of the title compound was undertaken.



(I)

Both phenylalanine residues (1 and 2) have similar geometries (Fig. 1). In both residues, the bond distances C—O [1.313 (6) and 1.311 (7) Å] and C═O [1.202 (8) and 1.220 (7) Å] are nearly equal and the single-bonded O atoms of 1 and 2 (O12···O22) are at a distance of 2.426 (8) Å. This may refer to a short hydrogen bond between the singly bonded carboxyl O atoms (O12···O22), as found in L-phenylalanine L-phenylalaninium perchlorate (Srinivasan & Rajaram, 1997). However, this H atom could not be located unequivocally. Hence, taking into account the equality of the C—O distances (see above), it can be surmised that the two residues may be connected by either a symmetric hydrogen bond or an asymmetric hydrogen bond with the H atom disordered over two positions.

The conformation angles ψ^1 for residues 1 and 2 are −9.9 (7) and 2.5 (8)°, respectively (see Table 1). This tendency

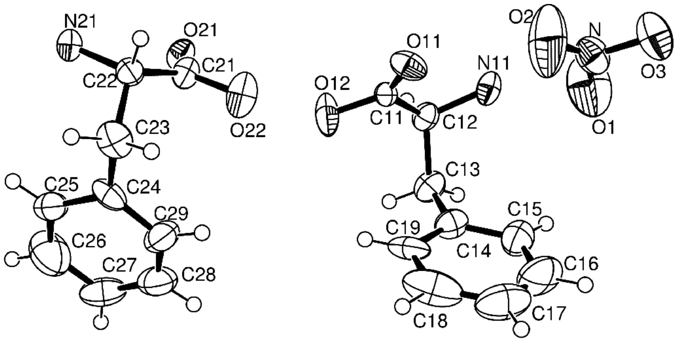


Figure 1
The structures of phenylalanine residues showing the atomic numbering scheme and 50% probability displacement ellipsoids (Johnson, 1976).

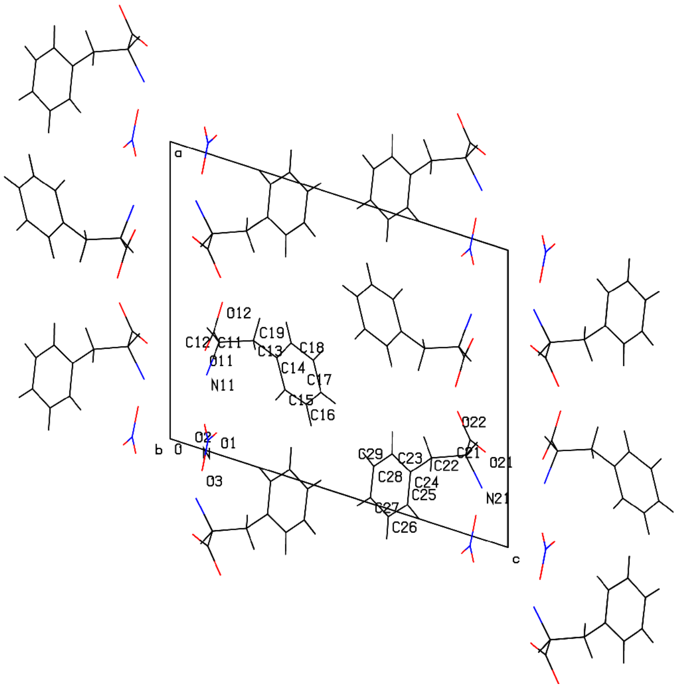


Figure 2
Packing diagram for the title compound viewed down the *b* axis

towards non-planarity is also found in various amino acids (Lakshminarayanan *et al.*, 1967). The branched side conformation angle χ^1 shows a *gauche* I conformation for both residues [68.2 (7) and 71.0 (7)°], which agrees well with formate and perchlorate complexes. The χ^{21} [89.0 (8) and 97.0 (8)°] and χ^{22} [−92.3 (8) and −84.3 (7)°] torsion angles indicate that the residues have folded conformations.

The aggregation of the hydrophilic zone is along the $z = 0$ plane. The hydrophobic zone at $z = \frac{1}{2}$ consists of the phenyl groups of both molecules and is sandwiched between two hydrophilic layers in the planes $z = 0$ and $z = 1$ (Fig. 2).

Experimental

The title compound was crystallized from an aqueous solution of a 2:1 stoichiometric ratio of L-phenylalanine and nitric acid by slow evaporation.

Crystal data

$2C_9H_{11}NO_2{\cdot}H^+{\cdot}NO_3^-$
$M_r = 393.39$
Monoclinic, $P2_1$
$a = 12.536\ (16)$ Å
$b = 5.378\ (4)$ Å
$c = 14.962\ (9)$ Å
$\beta = 107.83\ (8)°$
$V = 960.2\ (15)$ Å^3
$Z = 2$
$D_x = 1.361$ Mg m^{-3}
$D_m = 1.350$ Mg m^{-3}
D_m measured by flotation in a mixture of carbon tetrachloride and xylene
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.5$–$23.4°$
$\mu = 0.11$ mm^{-1}
$T = 293\ (2)$ K
Needle, colorless
0.45 × 0.30 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.963$, $T_{\max} = 0.984$
1895 measured reflections
1895 independent reflections
1356 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 25.0°$
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 6$
$l = 0 \rightarrow 17$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.220$
$S = 1.09$
1895 reflections
250 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1548P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0 (4)

Table 1
Selected geometric parameters (Å, °).

O11—C11	1.202 (8)	O21—C21	1.220 (7)
O12—C11	1.313 (6)	O22—C21	1.311 (7)
O11—C11—C12—N11	−9.9 (7)	O21—C21—C22—N21	2.5 (8)
N11—C12—C13—C14	68.2 (7)	N21—C22—C23—C24	71.0 (7)
C12—C13—C14—C19	89.0 (8)	C22—C23—C24—C29	97.0 (8)
C12—C13—C14—C15	−92.3 (8)	C22—C23—C24—C25	−84.3 (7)

H atoms bonded to C atoms were placed in geometrically calculated positions and allowed to ride on the attached atoms. Attempts to introduce H atoms attached to the amino N atom yielded a slight increase in all discrepancy values. Therefore, these H atoms were not included in the final refinement. The absolute configuration is known from the synthesis and is indeterminate from the X-ray diffraction experiment.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97.

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References

Al-Karaghouli, A. R. & Koetzle, T. F. (1975). *Acta Cryst.* B**31**, 2461–2465.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Flack H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Acta Cryst. (2001). E**57**, o916–o918 N. Srinivasan *et al.* • $2C_9H_{11}NO_2{\cdot}H^+{\cdot}NO_3^-$ **o917**

Gorbitz, C. H. & Etter, M. C. (1992). *Acta Cryst.* C**48**, 1317–1320.
Gurskaya, G. V. & Vainshtein, B. K. (1963). *Sov. Phys. Crystallogr.* **8**, 288–291.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Lakshminarayanan, A. V., Sashisekaran, V. & Ramachandran, G. N. (1967). In *Conformation of Biopolymers*, edited by G. N. Ramachandran. London: Academic Press.
Nagashima, N., Sano, C., Kawakita, T. & Iitaka, Y. (1992). *Anal. Sci.* **8**, 723–725.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Spek, A. L. (1999). *PLATON for Windows*. Utrecht University, The Netherlands.
Srikrishnan, T., Narasinga Rao, S. & Parthasarathy, R. (1984). *Acta Cryst.* A**40**, C-92.
Srinivasan, N. & Rajaram, R. K. (1997). *Acta Cryst.* C**53**, 1711–1713.

o918 N. Srinivasan *et al.* • $2C_9H_{11}NO_2 \cdot H^+ \cdot NO_3^-$ *Acta Cryst.* (2001). E**57**, o916–o918