

C7—C5—C4	118.6 (3)	C24—C23—C31	120.9 (3)
C5—C6—C1	124.6 (3)	O7A—C27—C11	109.4 (5)
C8—C7—C5	120.1 (3)	O7B—C27—C11	105.3 (4)
C12—C7—C5	120.1 (3)	O8—C28—C16	112.2 (3)
O5—C8—C7	121.3 (3)	O11—C31—C23	108.4 (3)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H...A	H...A	D...A	D—H...A
O5—H5...O4	2.25	2.974 (4)	143
O7A—H7A...O8 ⁱ	1.95	2.793 (8)	174
O7B—H7B...O8 ^j	2.00	2.833 (7)	166
O8—H8...O7A ⁱⁱ	1.98	2.793 (8)	159
O8—H8...O7B ⁱⁱ	1.99	2.833 (7)	176
O11—H11...O5 ⁱⁱⁱ	1.92	2.772 (3)	175

Symmetry codes: (i) $\frac{1}{2} + x, \frac{3}{2} - y, -z$; (ii) $x - \frac{1}{2}, \frac{3}{2} - y, -z$; (iii) $x - \frac{1}{2}, y, \frac{1}{2} - z$.

The structure was determined by direct methods. H atoms were added at calculated positions and refined isotropically using a riding model. H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached. All other non-H atoms were refined anisotropically, except for the disordered O7. The hydroxyl group O7—H7 has two orientations with occupancies of 0.48 (1) and 0.52 (1).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993a). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993b). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1229). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,2-Diacetyl-4-phenyl-1,2,4-triazolidine-3,5-dione

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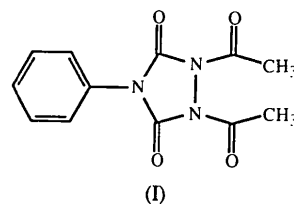
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Abstract

The title crystal, C₁₂H₁₁N₃O₄, contains three essentially identical molecules in the asymmetric unit. The triazole rings in the three independent molecules are planar within 0.014 Å. The N atom containing the phenyl substituent is trigonal, the sum of the three bond angles being 359.5°. The other two N atoms are slightly pyramidal, the sum of the three bond angles being 346°. The two acetyl groups are twisted out of the plane of the triazole ring by an average of 33° and are *trans* to each other.

Comment

The structure determination of the title compound, (I) (Simlot, Izydore, Wong & Hall, 1994), was undertaken to establish its conformation and compare it with the previously determined conformation of 1,2-diacetyl-4,4-diethyl-3,5-pyrazolidinedione (Izydore, Bernal-Ramirez & Singh, 1990). As with the pyrazolidinedione, the acetylation in the present triazole takes place at the N atoms.



The crystal structure has a curious feature in that it contains six molecules in a triclinic unit cell and, therefore, three independent molecules in the asymmetric unit of space group *P* $\bar{1}$. Although the cell-reduction routine provided with the diffractometer is quite reliable, we have further confirmed the absence of any symmetry higher than triclinic using the NRC cell-reduction program *CREDUC* (Le Page, 1982). No other efforts were made to look for higher symmetry.

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The three independent molecules in the asymmetric unit have very similar structures. The largest difference lies in one of the dihedral angles, *i.e.* that between the planar triazole ring and the benzene ring, which is 68.4° in molecule *A* but 79.0 and 82.1° in molecules *B* and *C*, respectively. Atom N(3), carrying the phenyl substituent, is trigonal in all three molecules, the sum of the three bond angles around it being 359.6 , 359.5 and 359.4° for molecules *A*, *B* and *C*, respectively. The other two N atoms, N(1) and N(2), have a slightly pyramidal hybridization, however, the sum of the three angles around N(1) being 347.0 , 346.4 and 345.8° , and around N(2) 348.3 , 350.1 and 349.1° for molecules *A*, *B* and *C*, respectively. The two acetyl groups are *trans* to each other and twisted out of the triazole plane by an average of 33° , which is comparable to the twist of 38 and 23° , respectively, of the pyrazolidine ring of the *trans* acetyl groups in 1,2-diacetyl-4,4-diethyl-3,5-pyrazolidinedione (Izdyore *et al.*, 1990). The molecule has an approximate C_2 symmetry, the twofold axis passing through atoms C(10), C(7), N(3) and the middle of the N(1)—N(2) bond.

The benzene and carbonyl bond lengths are normal. Among the N—C bond lengths, however, N—COCH₃ with a mean of $1.429(5)$ Å and N—C(endocyclic) with a

mean length of $1.413(5)$ Å are slightly but significantly shorter than N—C(Ph) [$1.448(5)$ Å]. The C—H bond distances vary from $0.78(8)$ to $1.02(6)$ Å.

A drawing of molecule *A* is shown in Fig. 1. Molecules *B* and *C* have similar structures. A packing diagram viewed down the *a* axis is shown in Fig. 2. Selected bond distances and bond angles for the three molecules are given in Table 2.

Experimental

A mixture containing 0.030 mol of 4-phenyl-1,2,4-triazolidine-3,5-dione (purchased from Aldrich Chemical Company), 0.060 mol of powdered lead diacetate trihydrate and 0.45 mol of acetic acid anhydride in 200 ml CH_2Cl_2 , was prepared. The mixture was stirred at room temperature for 48 h. The reaction mixture was filtered and the filtrate washed three times with 100 ml of water and three times with 75 ml of 10% Na_2CO_3 , dried ($MgSO_4$) and evaporated under reduced pressure to give a white solid. The solid was recrystallized from absolute EtOH to yield the title compound, m.p. 442 – 444 K.

A quantity of the title compound weighing 1.0 g was dissolved in 50 ml boiling absolute EtOH. The hot solution was filtered and allowed to stand at room temperature for three days. The precipitate was filtered to give 0.7 g of small white crystals.

Crystal data

$C_{12}H_{11}N_3O_4$

$M_r = 261.24$

Triclinic

$P\bar{1}$

$a = 11.002(4)$ Å

$b = 12.962(5)$ Å

$c = 13.643(6)$ Å

$\alpha = 103.94(3)^\circ$

$\beta = 92.10(3)^\circ$

$\gamma = 97.31(3)^\circ$

$V = 1868(1)$ Å³

$Z = 6$

$D_x = 1.39$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 10$ – 13.5°

$\mu = 0.08$ mm⁻¹

$T = 298$ K

Thin plate

$0.50 \times 0.28 \times 0.07$ mm

Colorless

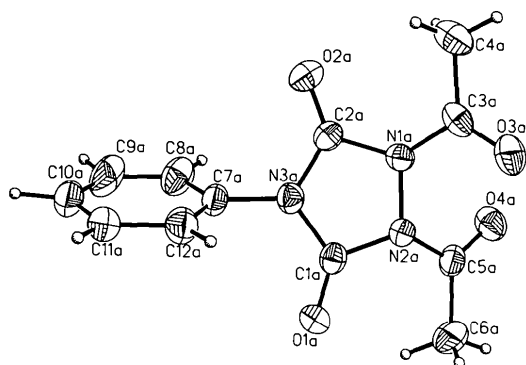


Fig. 1. Molecule *A* viewed normal to the five-membered triazole ring. Displacement ellipsoids for the non-H atoms are drawn at the 50% probability level. H atoms are shown as small circles.

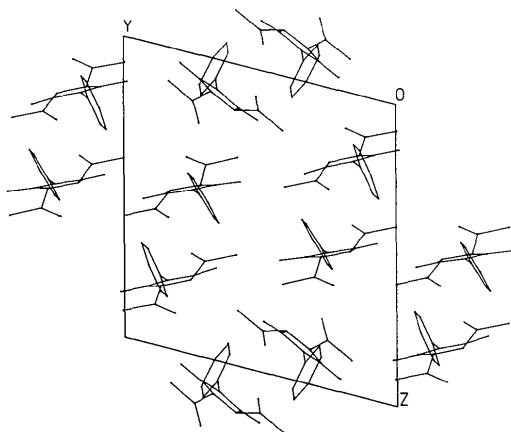


Fig. 2. Packing viewed down the *a* axis.

Data collection

Siemens $R3m/\mu$ diffractometer

ω scans

Absorption correction:

none

10540 measured reflections

9947 independent reflections

4346 observed reflections

$[I \geq 2.5\sigma(I)]$

$R_{int} = 0.023$

$\theta_{max} = 29^\circ$

$h = -14 \rightarrow 14$

$k = -17 \rightarrow 17$

$l = 0 \rightarrow 18$

2 standard reflections

frequency: 48 min

intensity decay: 2.3%

Refinement

Refinement on F

$R = 0.067$

$wR = 0.078$

$S = 1.3$

4346 reflections

647 parameters

$\Delta\rho_{max} = 0.34$ e Å⁻³

$\Delta\rho_{min} = -0.29$ e Å⁻³

Extinction correction:

Zachariasen (1968)

Extinction coefficient:

$1.3(4) \times 10^{-6}$

All H-atom parameters

refined

$$w = 1/[\sigma^2(F_o) + 0.001F_o^2]$$

$$(\Delta/\sigma)_{\max} = 0.49$$

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)*

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
N(1A)	0.2870 (3)	0.8311 (2)	0.4740 (2)	0.029 (1)
N(2A)	0.2896 (3)	0.7204 (2)	0.4309 (2)	0.029 (1)
C(1A)	0.1690 (3)	0.6668 (3)	0.4188 (3)	0.031 (1)
O(1A)	0.1364 (3)	0.5727 (2)	0.3863 (2)	0.042 (1)
N(3A)	0.0954 (3)	0.7457 (3)	0.4519 (2)	0.033 (1)
C(2A)	0.1625 (4)	0.8380 (3)	0.4871 (3)	0.033 (1)
O(2A)	0.1252 (3)	0.9287 (3)	0.5230 (3)	0.050 (1)
C(3A)	0.3837 (4)	0.8852 (3)	0.5470 (3)	0.033 (1)
O(3A)	0.4655 (3)	0.8380 (3)	0.5642 (2)	0.043 (1)
C(4A)	0.3776 (5)	1.0003 (4)	0.5955 (4)	0.054 (2)
C(5A)	0.3811 (3)	0.6979 (3)	0.3603 (3)	0.035 (1)
O(4A)	0.4548 (3)	0.7684 (3)	0.3496 (2)	0.046 (1)
C(6A)	0.3775 (5)	0.5817 (4)	0.3091 (4)	0.060 (2)
C(7A)	-0.0354 (3)	0.7247 (3)	0.4599 (3)	0.035 (1)
C(8A)	-0.1142 (4)	0.7633 (4)	0.4024 (4)	0.050 (2)
C(9A)	-0.2390 (4)	0.7455 (5)	0.4148 (4)	0.062 (2)
C(10A)	-0.2818 (4)	0.6913 (4)	0.4840 (4)	0.058 (2)
C(11A)	-0.2024 (4)	0.6529 (4)	0.5414 (4)	0.055 (2)
C(12A)	-0.0771 (4)	0.6687 (4)	0.5293 (4)	0.043 (2)
N(1B)	0.5961 (3)	0.2434 (3)	0.2475 (2)	0.031 (1)
N(2B)	0.6002 (3)	0.1342 (3)	0.2015 (3)	0.033 (1)
C(1B)	0.4787 (4)	0.0794 (3)	0.1872 (3)	0.037 (1)
O(1B)	0.4479 (3)	-0.0132 (3)	0.1487 (3)	0.061 (1)
N(3B)	0.4062 (3)	0.1560 (3)	0.2258 (3)	0.032 (1)
C(2B)	0.4724 (4)	0.2562 (3)	0.2653 (3)	0.034 (1)
O(2B)	0.4345 (3)	0.3362 (2)	0.3062 (3)	0.048 (1)
C(3B)	0.6965 (4)	0.2958 (3)	0.3193 (3)	0.035 (1)
O(3B)	0.7768 (3)	0.2469 (3)	0.3354 (2)	0.045 (1)
C(4B)	0.6921 (5)	0.4111 (4)	0.3673 (4)	0.051 (2)
C(5B)	0.6954 (4)	0.1122 (4)	0.1340 (3)	0.039 (1)
O(4B)	0.7661 (3)	0.1842 (3)	0.1214 (2)	0.052 (1)
C(6B)	0.6999 (5)	-0.0039 (4)	0.0876 (5)	0.070 (2)
C(7B)	0.2759 (3)	0.1310 (3)	0.2366 (3)	0.031 (1)
C(8B)	0.2395 (4)	0.0862 (4)	0.3137 (3)	0.039 (1)
C(9B)	0.1143 (4)	0.0644 (4)	0.3263 (4)	0.049 (2)
C(10B)	0.0304 (4)	0.0894 (4)	0.2611 (4)	0.051 (2)
C(11B)	0.0694 (4)	0.1339 (4)	0.1842 (4)	0.054 (2)
C(12B)	0.1927 (4)	0.1554 (4)	0.1706 (3)	0.043 (2)
N(1C)	-0.0427 (3)	0.5928 (3)	0.1549 (2)	0.031 (1)
N(2C)	-0.0010 (3)	0.6789 (3)	0.1129 (2)	0.031 (1)
C(1C)	-0.1038 (3)	0.7194 (3)	0.0790 (3)	0.031 (1)
O(1C)	-0.1036 (3)	0.7929 (3)	0.0414 (3)	0.052 (1)
N(3C)	-0.2040 (3)	0.6557 (3)	0.1009 (2)	0.031 (1)
C(2C)	-0.1726 (3)	0.5762 (3)	0.1447 (3)	0.034 (1)
O(2C)	-0.2396 (3)	0.5089 (3)	0.1686 (3)	0.052 (1)
C(3C)	0.0282 (4)	0.5058 (3)	0.1361 (3)	0.038 (1)
O(3C)	0.1177 (3)	0.5129 (3)	0.0901 (3)	0.056 (1)
C(4C)	-0.0151 (6)	0.4157 (4)	0.1823 (6)	0.061 (2)
C(5C)	0.1110 (3)	0.7451 (3)	0.1589 (3)	0.031 (1)
O(4C)	0.1629 (3)	0.7239 (3)	0.2272 (2)	0.050 (1)
C(6C)	0.1538 (4)	0.8344 (4)	0.1120 (4)	0.042 (2)
C(7C)	-0.3289 (3)	0.6615 (3)	0.0668 (3)	0.031 (1)
C(8C)	-0.4039 (4)	0.7145 (4)	0.1334 (4)	0.051 (2)
C(9C)	-0.5222 (4)	0.7203 (4)	0.0984 (4)	0.058 (2)
C(10C)	-0.5632 (4)	0.6719 (4)	0.0003 (4)	0.049 (2)
C(11C)	-0.4873 (4)	0.6197 (4)	-0.0647 (4)	0.052 (2)
C(12C)	-0.3681 (4)	0.6145 (4)	-0.0316 (3)	0.047 (2)

C(1A)—N(3A)	1.386 (5)	N(3A)—C(2A)	1.378 (5)
N(3A)—C(7A)	1.444 (5)	C(2A)—O(2A)	1.195 (5)
C(3A)—O(3A)	1.197 (5)	C(3A)—C(4A)	1.490 (6)
C(5A)—O(4A)	1.182 (5)	C(5A)—C(6A)	1.494 (6)
C(7A)—C(8A)	1.366 (7)	C(7A)—C(12A)	1.383 (7)
C(8A)—C(9A)	1.387 (7)	C(9A)—C(10A)	1.369 (9)
C(10A)—C(11A)	1.369 (8)	C(11A)—C(12A)	1.389 (6)
N(1B)—N(2B)	1.411 (4)	N(1B)—C(2B)	1.414 (5)
N(1B)—C(3B)	1.432 (5)	N(2B)—C(1B)	1.415 (5)
N(2B)—C(5B)	1.430 (5)	C(1B)—O(1B)	1.188 (5)
C(1B)—N(3B)	1.371 (5)	N(3B)—C(2B)	1.378 (5)
N(3B)—C(7B)	1.450 (5)	C(2B)—O(2B)	1.187 (5)
C(3B)—O(3B)	1.193 (6)	C(3B)—C(4B)	1.487 (6)
C(5B)—O(4B)	1.187 (6)	C(5B)—C(6B)	1.494 (7)
C(7B)—C(8B)	1.368 (6)	C(7B)—C(12B)	1.377 (6)
C(8B)—C(9B)	1.395 (6)	C(9B)—C(10B)	1.380 (7)
C(10B)—C(11B)	1.372 (8)	C(11B)—C(12B)	1.378 (6)
N(1C)—N(2C)	1.407 (5)	N(1C)—C(1C)	1.414 (5)
N(1C)—C(3C)	1.430 (5)	N(2C)—C(1C)	1.414 (5)
N(2C)—C(5C)	1.437 (4)	C(1C)—O(1C)	1.185 (6)
C(1C)—N(3C)	1.378 (5)	N(3C)—C(2C)	1.383 (6)
N(3C)—C(7C)	1.451 (5)	C(2C)—O(2C)	1.185 (5)
C(3C)—O(3C)	1.192 (5)	C(3C)—C(4C)	1.491 (9)
C(5C)—O(4C)	1.180 (5)	C(5C)—C(6C)	1.487 (7)
C(7C)—C(8C)	1.369 (6)	C(7C)—C(12C)	1.362 (6)
C(8C)—C(9C)	1.387 (7)	C(9C)—C(10C)	1.367 (7)
C(10C)—C(11C)	1.359 (7)	C(11C)—C(12C)	1.387 (7)
N(2A)—N(1A)—C(2A)	107.8 (3)	N(2A)—N(1A)—C(3A)	116.7 (3)
C(2A)—N(1A)—C(3A)	122.5 (3)	N(1A)—N(2A)—C(1A)	108.6 (3)
N(1A)—N(2A)—C(5A)	114.7 (3)	C(1A)—N(2A)—C(5A)	125.0 (3)
N(2A)—C(1A)—O(1A)	127.2 (4)	N(2A)—C(1A)—N(3A)	105.6 (3)
O(1A)—C(1A)—N(3A)	127.2 (3)	C(1A)—N(3A)—C(2A)	112.5 (3)
C(1A)—N(3A)—C(7A)	124.2 (3)	C(2A)—N(3A)—C(7A)	122.9 (3)
N(1A)—C(2A)—N(3A)	105.5 (3)	N(1A)—C(2A)—O(2A)	126.7 (3)
N(3A)—C(2A)—O(2A)	127.8 (4)	N(1A)—C(3A)—O(3A)	119.3 (3)
N(1A)—C(3A)—C(4A)	116.6 (4)	O(3A)—C(3A)—C(4A)	124.2 (4)
N(2A)—C(5A)—O(4A)	119.8 (4)	N(2A)—C(5A)—C(6A)	114.6 (4)
O(4A)—C(5A)—C(6A)	125.5 (4)	N(3A)—C(7A)—C(8A)	119.9 (4)
N(3A)—C(7A)—C(12A)	118.3 (4)	C(8A)—C(7A)—C(12A)	121.7 (4)
C(7A)—C(8A)—C(9A)	118.6 (5)	C(8A)—C(9A)—C(10A)	120.5 (5)
C(9A)—C(10A)—C(11A)	120.7 (5)	C(10A)—C(11A)—C(12A)	119.8 (5)
C(7A)—C(12A)—C(11A)	118.8 (5)	N(2B)—N(1B)—C(2B)	108.0 (3)
N(2B)—N(1B)—C(3B)	115.5 (3)	C(2B)—N(1B)—C(3B)	122.9 (3)
N(1B)—N(2B)—C(1B)	108.3 (3)	N(1B)—N(2B)—C(5B)	116.2 (3)
C(1B)—N(2B)—C(5B)	125.6 (3)	N(2B)—C(1B)—O(1B)	126.5 (4)
N(2B)—C(1B)—N(3B)	105.3 (3)	O(1B)—C(1B)—N(3B)	128.2 (4)
C(1B)—N(3B)—C(7B)	113.1 (3)	C(1B)—N(3B)—C(7B)	123.1 (3)
C(2B)—N(3B)—C(7B)	123.3 (3)	N(1B)—C(2B)—N(3B)	105.3 (3)
N(1B)—C(2B)—O(2B)	127.0 (3)	N(3B)—C(2B)—O(2B)	127.7 (4)
N(1B)—C(3B)—O(3B)	119.7 (3)	N(1B)—C(3B)—C(4B)	115.3 (4)
O(3B)—C(3B)—C(4B)	125.0 (4)	N(2B)—C(5B)—O(4B)	119.6 (4)
N(2B)—C(5B)—C(6B)	115.4 (4)	O(4B)—C(5B)—C(6B)	125.0 (4)
N(3B)—C(7B)—C(8B)	118.8 (4)	N(3B)—C(7B)—C(12B)	119.2 (4)
C(8B)—C(7B)—C(12B)	122.0 (4)	C(7B)—C(8B)—C(9B)	119.2 (4)
C(8B)—C(9B)—C(10B)	119.1 (5)	C(9B)—C(10B)—C(11B)	120.5 (4)
C(10B)—C(11B)—C(12B)	121.0 (5)	C(7B)—C(12B)—C(11B)	118.2 (5)
N(2C)—N(1C)—C(2C)	108.1 (3)	N(2C)—N(1C)—C(3C)	115.4 (3)
C(2C)—N(1C)—C(3C)	122.3 (3)	N(1C)—N(2C)—C(1C)	108.9 (3)
N(1C)—N(2C)—C(5C)	116.5 (3)	C(1C)—N(2C)—C(5C)	123.7 (3)
N(2C)—C(1C)—O(1C)	127.6 (3)	N(2C)—C(1C)—N(3C)	104.7 (3)
O(1C)—C(1C)—N(3C)	127.7 (4)	C(1C)—N(3C)—C(2C)	113.3 (3)
C(1C)—N(3C)—C(7C)	127.7 (4)	C(2C)—N(3C)—C(7C)	123.4 (3)
N(1C)—C(2C)—N(3C)	105.0 (3)	N(1C)—C(2C)—O(2C)	125.5 (4)
N(3C)—C(2C)—O(2C)	127.6 (4)	N(1C)—C(3C)—O(3C)	118.7 (4)
N(1C)—C(3C)—C(4C)	115.2 (4)	O(3C)—C(3C)—C(4C)	126.0 (5)
N(2C)—C(5C)—O(4C)	119.2 (4)	N(2C)—C(5C)—C(6C)	115.3 (4)
O(4C)—C(5C)—C(6C)	125.4 (4)	N(3C)—C(7C)—C(8C)	119.6 (3)
N(3C)—C(7C)—C(12C)	118.7 (3)	C(8C)—C(7C)—C(12C)	121.7 (4)
C(7C)—C(8C)—C(9C)	118.4 (4)	C(8C)—C(9C)—C(10C)	120.4 (5)
C(9C)—C(10C)—C(11C)	120.3 (4)	C(10C)—C(11C)—C(12C)	120.2 (4)
C(7C)—C(12C)—C(11C)	119.0 (4)		

Table 2. *Selected geometric parameters (\AA , $^\circ$)*

N(1A)—N(2A)	1.418 (4)	N(1A)—C(2A)	1.419 (5)
N(1A)—C(3A)	1.418 (4)	N(2A)—C(1A)	1.401 (5)
N(2A)—C(5A)	1.428 (5)	C(1A)—O(1A)	1.195 (5)

Most of the reflections are weak, especially those at high θ values. Their precision is poor owing to unfavourable counting statistics which may explain the somewhat high *R* factor and a high proportion of data being tagged as unobserved.

The intensity data were collected at variable scan rates from 4 to 29.3° min⁻¹ depending on intensity. Stationary backgrounds were measured on both sides of a peak, each for one-half of the scan time. The structure was solved by direct methods and difference Fourier techniques, and refined by blocked-cascade least-squares refinement (Sparks, 1961). Non-H atoms were refined with anisotropic and H atoms with isotropic displacement parameters. Calculations were performed on the Data General micro-eclipse computer. Software used for structure solution, refinement and molecular graphics: *SHELXTL* (Sheldrick, 1985).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1002). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Acetyl-L-phenylalanyl-L-alaninamide

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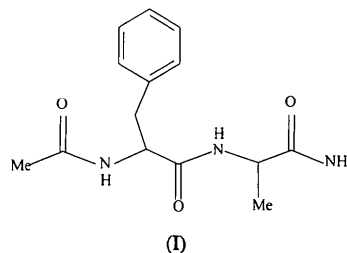
Abstract

The conformation of the peptide chain in *N*-acetyl-L-phenylalanyl-L-alaninamide (NAFAA), C₁₄H₁₉N₃O₃, is

rather extended and falls in the *E* region of the φ , ψ map, according to the classification of Zimmerman, Pottle, Némethy & Scheraga [*Macromolecules* (1977), **10**, 1–9]. The values of φ , ψ torsion angles are like those of an antiparallel β pleated sheet. Side-chain conformation of Phe residue is defined by $\chi_1 = -174.5(4)^\circ$ and $\chi_2 = 105.5(6)^\circ$ and comes within the *B*₂ class of the observed statistical distribution for the aromatic residues in peptides [Cody, Duax & Hauptman, (1973). *Int. J. Peptide Protein Res.* **5**, 297–308]. Crystal packing is ruled by four intermolecular hydrogen bonds that involve all the donor groups, the acetyl O atom acting as a double acceptor. In the crystal there are ribbons of molecules translated along the *a* axis of the *P*2₁ space group and joined through three hydrogen bonds. The fourth hydrogen bond interconnects screw-related ribbons. The phenylalanyl rings stack parallel to the *a* direction with interplanar distances of 3.334 (7) Å.

Comment

X-ray studies on protected aminoacids and oligopeptides are useful for a better understanding of the conformational preferences of amino acid residues in biopolymers. The results obtained from small structures can also be used in the interpretation of refinement data of protein structures. As part of a program concerning crystallographic determinations and thermodynamic behaviour during the phase transitions of *N*-acetyl peptidamides (Puliti, Barone, Giancola & Mattia, 1996; Puliti & Mattia, 1995, and references therein), we present here the X-ray structure of *N*-acetyl-L-phenylalanyl-L-alaninamide (NAFAA), (I).



A perspective view of the NAFAA molecule is shown in Fig. 1 together with the atomic labels used. The most significant values of the intramolecular geometry are reported in Table 2. Bond lengths and bond angles are generally in good agreement with the corresponding values reported in the literature for similar compounds (Ramachandran, Kolaskar, Ramakrishnan & Sasisekharan, 1974) and in particular for other *N*-acetylpeptidamides (Puliti & Mattia, 1995, and references therein).

The molecule adopts a rather extended conformation. The peptide linkage is in a slightly distorted *trans* form [$\omega = 173.1(4)^\circ$] and φ , ψ torsion angles for the two residues fall in the *E* region of the conventional Zimmerman map for peptides (Zimmerman, Pottle,