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Structure of 'BuCO-Valψ[NH-CO]NH'Bu

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Abstract. *N*-Isobutylidenedipivalamide, $C_{14}H_{28}N_2O_2$, $M_r = 256.39$, orthorhombic, $P2_12_12_1$, $a = 16.656$ (2), $b = 9.751$ (2), $c = 10.583$ (2) Å, $V = 1718.8$ Å^3, $Z = 4$, $D_x = 0.99$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 4.56$ cm^{-1}, $\mu R_{max} \ll 1$, $F(000) = 568$, $T = 293$ K, $R = 0.081$ for 887 observed reflections. The geometrical parameters of this retro-peptide molecule are quite similar to the standard values for peptides. Conformational angles are $\varphi = -101$ (1), $\varphi' = 99$ (1)°.

Experimental. Crystal size 0.20 × 0.10 × 0.08 mm, X-ray data were collected at room temperature on Enraf–Nonius CAD-4 automatic diffractometer, with Cu $K\alpha$ radiation, in the $\theta/2\theta$-scanning mode ($\theta < 70°$). Cell parameters refined by least squares on the basis of 25 independent θ values in the range 20–30°. 1861 reflections measured ($h = 0$ to 19, $k = 0$ to 11, $l = 0$ to 12), 887 with $F_o > 3\sigma(F_o)$ were used for all calculations. Two standards (203, 040) measured every 2 h showed no deviations greater than 3% in intensity. Lorentz and polarization corrections were applied to the data, but no absorption correction.

Structure solved by direct methods, using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refined by full-matrix least-squares procedure on F (*SHELX*; Sheldrick, 1976). E maps revealed all non-H atoms and atoms appeared in difference maps. Refined parameters were calculated using anisotropic temperature factors for non-H atoms and fixed isotropic temperature factors for H atoms. Final agreement factors were $R = 0.081$ and $wR = 0.087$ $\{w = 2.007/[\sigma^2(F_o) + 0.0046F_o^2]\}$. Goodness of fit = 1.30, $-0.24 < \Delta\rho < 0.21$ e Å^{-3}, shift/e.s.d.'s < 0.12. Following recommendations by Taylor & Kennard (1983), the NH H atoms were placed at 1.03 Å from N in the direction obtained by refinement. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 1. *Fractional coordinates with standard deviations and equivalent isotropic thermal parameters for* 'BuCO-Valψ[NH-CO]NH'Bu

	x	y	z	B_{eq} (Å^2)
O(1)	0.0805 (4)	−0.1233 (4)	0.3587 (6)	8.3 (2)
O(2)	−0.0250 (5)	−0.1233 (4)	0.0838 (6)	9.8 (2)
N(1)	0.0298 (4)	0.0856 (5)	0.3513 (6)	6.2 (2)
N(2)	−0.0429 (8)	0.0869 (8)	0.1614 (9)	6.5 (2)
C(1)	0.226 (1)	−0.046 (2)	0.424 (3)	17.2 (6)
C(2)	0.1602 (9)	0.120 (2)	0.547 (1)	13.0 (4)
C(3)	0.1892 (9)	0.180 (2)	0.333 (2)	13.2 (4)
C(4)	0.1679 (5)	0.0572 (8)	0.4218 (8)	6.9 (2)
C(5)	0.0904 (5)	−0.0005 (6)	0.3772 (7)	5.9 (2)
C(6)	−0.0458 (4)	0.0492 (6)	0.2929 (7)	5.5 (2)
C(7)	−0.0275 (5)	0.0010 (6)	0.0657 (8)	5.8 (2)
C(8)	−0.0204 (5)	0.0560 (8)	−0.0690 (7)	6.6 (2)
C(9)	0.0413 (8)	0.176 (1)	−0.066 (1)	9.4 (3)
C(10)	0.000 (2)	−0.053 (2)	−0.151 (1)	15.6 (5)
C(11)	−0.0996 (9)	0.117 (2)	−0.110 (1)	12.1 (4)
C(12)	−0.1181 (5)	0.1136 (7)	0.3570 (8)	6.8 (2)
C(13)	−0.118 (1)	0.076 (1)	0.491 (1)	12.4 (4)
C(14)	−0.1907 (8)	0.071 (2)	0.294 (2)	11.7 (4)
H(N1)	0.005	0.087	0.440	
H(N2)	−0.061	0.188	0.153	

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Table 2. *Bond lengths* (Å), *bond angles* (°) *and torsional angles* (°) *for* 'BuCO-Valψ[NH-CO]NH'Bu

O(1)—C(5)	1.234 (8)	C(4)—C(5)	1.485 (12)
O(2)—C(7)	1.229 (7)	C(6)—C(12)	1.52 (1)
N(1)—C(5)	1.345 (10)	C(7)—C(8)	1.507 (11)
N(1)—C(6)	1.444 (10)	C(8)—C(9)	1.558 (14)
N(2)—C(6)	1.452 (14)	C(8)—C(10)	1.42 (2)
N(2)—C(7)	1.343 (13)	C(8)—C(11)	1.51 (2)
C(1)—C(4)	1.40 (2)	C(12)—C(13)	1.46 (2)
C(2)—C(4)	1.46 (2)	C(12)—C(14)	1.44 (2)
C(3)—C(4)	1.56 (2)		

C(5)—N(1)—C(6)	126.1 (6)	N(2)—C(6)—C(12)	110.7 (8)
C(6)—N(2)—C(7)	125.3 (7)	O(2)—C(7)—N(2)	120.4 (8)
C(1)—C(4)—C(2)	110 (1)	O(2)—C(7)—C(8)	119.3 (6)
C(1)—C(4)—C(3)	114 (1)	N(2)—C(7)—C(8)	120.1 (6)
C(1)—C(4)—C(5)	110 (1)	C(7)—C(8)—C(9)	107.3 (7)
C(2)—C(4)—C(3)	104 (1)	C(7)—C(8)—C(10)	109.2 (9)
C(2)—C(4)—C(5)	111.8 (8)	C(7)—C(8)—C(11)	109.9 (8)
C(3)—C(4)—C(5)	107.2 (8)	C(9)—C(8)—C(10)	114 (1)
O(1)—C(5)—N(1)	118.6 (8)	C(9)—C(8)—C(11)	106.6 (9)
O(1)—C(5)—C(4)	122.6 (7)	C(10)—C(8)—C(11)	109 (1)
N(1)—C(5)—C(4)	118.8 (5)	C(6)—C(12)—C(13)	108.9 (9)
N(1)—C(6)—N(2)	108.7 (8)	C(6)—C(12)—C(14)	109.7 (8)
N(1)—C(6)—C(12)	113.5 (5)	C(13)—C(12)—C(14)	112 (1)

φ	−101 (1)	φ'	99 (1)
N(1)—C(6)—C(12)—C(13)	χ^1/C(13) −56 (1)	N(2)—C(6)—C(12)—C(13)	$\chi^{1'}$/C(13) −178 (1)
N(1)—C(6)—C(12)—C(14)	χ^1/C(14) −179 (1)	N(2)—C(6)—C(12)—C(14)	$\chi^{1'}$/C(14) 58 (1)
C(4)—C(5)—N(1)—C(6)	ω_1 172 (1)	C(6)—N(2)—C(7)—C(8)	ω_2 −176 (1)

Final atomic parameters are given in Table 1,* bond distances, bond angles and conformational angles in Table 2 (IUPAC–IUB Commission on Biochemical Nomenclature, 1970; IUPAC–IUB Joint Commission on Biochemical Nomenclature, 1984).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54434 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

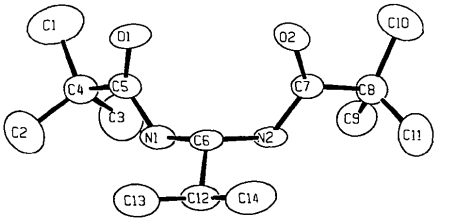

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the 'BuCO-Valψ[NH-CO]NH'Bu molecule and interatomic distances (Å) associated with intermolecular hydrogen bond N—H···O [N(1)···O(2^i) = 2.92, N(2)···O(1^i) = 2.90. Symmetry code: (i) $-x, \frac{1}{2}+y, \frac{1}{2}-z$].

Fig. 1 shows a thermal ellipsoid plot with the atomic numbering scheme. The observed conformation corresponds to a saddle point on the energy map (Stern, Chorev, Goodman & Hagler, 1983). Molecules are hydrogen bonded in such a way that they form a parallel β-sheet structure.

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Structure of MeCO-Glyψ[NH-CO]NHMe

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Abstract. *N,N'*-Methylenediacetamide, $C_5H_{10}N_2O_2$, $M_r = 130.15$, orthorhombic, $Pna2_1$, $a = 17.218$ (1), $b = 4.489$ (1), $c = 18.124$ (1) Å, $V = 1400{,}8$ Å^3, $Z = 8$, $D_x = 1.23$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu R_{max} \ll 1$, $\mu = 7.19$ cm^{-1}, $F(000) = 560$, $T = 293$ K, $R = 0.044$ for 1318 observed reflections. This retro-peptide mol-

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