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.

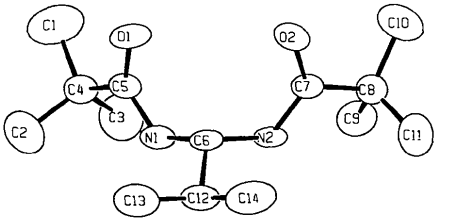

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the 'BuCO-Valψ[NH-CO]NH'Bu molecule and interatomic distances (Å) associated with intermolecular hydrogen bond N—H···O [N(1)···O(2^i) = 2.92, N(2)···O(1^i) = 2.90. Symmetry code: (i) $-x, \frac{1}{2}+y, \frac{1}{2}-z$].

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-

0108-2701/92/010176-03$03.00

Table 1. *Fractional coordinates with standard deviations and equivalent isotropic thermal parameters for* MeCO-Glyψ[NH-CO]NHMe

	x	y	z	B_{eq} (Å^2)
Molecule *A*				
O(1)	0.3097 (2)	0.2676 (5)	0.3640	4.74 (5)
O(2)	0.4676 (2)	0.1352 (6)	0.1287 (2)	5.88 (6)
N(1)	0.3278 (1)	0.2319 (5)	0.2415 (3)	3.59 (5)
N(2)	0.4673 (1)	0.2705 (5)	0.2464 (1)	3.45 (5)
C(1)	0.2248 (2)	−0.0576 (7)	0.2970 (3)	4.60 (6)
C(2)	0.2906 (1)	0.1609 (6)	0.3040 (3)	3.51 (4)
C(3)	0.3943 (1)	0.4282 (6)	0.2416 (3)	3.93 (5)
C(4)	0.4985 (1)	0.1327 (5)	0.1896 (2)	3.86 (5)
C(5)	0.5733 (2)	0.0291 (8)	0.2038 (3)	5.60 (7)
H(N1)	0.3175	0.1130	0.1938	
H(N2)	0.4981	0.2503	0.2948	
Molecule *B*				
O(3)	0.0578 (2)	0.2255 (6)	0.3796 (2)	4.97 (6)
O(4)	0.2178 (1)	0.3714 (6)	0.6151 (2)	5.96 (6)
N(3)	0.0769 (2)	0.2748 (6)	0.5017 (3)	3.92 (6)
N(4)	0.2162 (2)	0.2297 (6)	0.4958 (3)	4.01 (6)
C(6)	−0.0261 (2)	0.5576 (7)	0.4448 (3)	4.90 (6)
C(7)	0.0402 (1)	0.3413 (7)	0.4392 (3)	3.59 (5)
C(8)	0.1430 (2)	0.0757 (6)	0.5044 (3)	3.86 (5)
C(9)	0.2478 (1)	0.3734 (6)	0.5537 (3)	3.94 (5)
C(10)	0.3225 (2)	0.5338 (8)	0.5378 (3)	5.22 (7)
H(N3)	0.0612	0.3948	0.5478	
H(N4)	0.2403	0.2305	0.4438	

Table 2. *Bond lengths* (Å), *bond angles* (°) *and torsional angles for* MeCO-Glyψ[NH-CO]NHMe

O(1)—C(2)	1.233 (5)	O(3)—C(7)	1.236 (6)
O(2)—C(4)	1.226 (6)	O(4)—C(9)	1.226 (6)
N(1)—C(2)	1.339 (6)	N(3)—C(7)	1.332 (6)
N(1)—C(3)	1.445 (3)	N(3)—C(8)	1.448 (4)
N(2)—C(3)	1.445 (3)	N(4)—C(8)	1.446 (4)
N(2)—C(4)	1.314 (4)	N(4)—C(9)	1.347 (4)
C(1)—C(2)	1.504 (4)	C(6)—C(7)	1.502 (4)
C(4)—C(5)	1.501 (4)	C(9)—C(10)	1.503 (4)
C(2)—N(1)—C(3)	121.5 (4)	C(7)—N(3)—C(8)	122.7 (4)
C(3)—N(2)—C(4)	122.6 (3)	C(8)—N(4)—C(9)	119.9 (2)
O(1)—C(2)—N(1)	121.7 (3)	O(3)—C(7)—N(3)	122.1 (3)
O(1)—C(2)—C(1)	121.9 (4)	O(3)—C(7)—C(6)	121.2 (4)
N(1)—C(2)—C(1)	116.4 (4)	N(3)—C(7)—C(6)	116.7 (4)
N(1)—C(3)—N(2)	113.0 (2)	N(3)—C(8)—N(4)	112.7 (2)
O(2)—C(4)—N(2)	121.6 (3)	O(4)—C(9)—N(4)	122.2 (3)
O(2)—C(4)—C(5)	122.0 (4)	O(4)—C(9)—C(10)	122.6 (4)
N(2)—C(4)—C(5)	116.4 (4)	N(4)—C(9)—C(10)	115.2 (4)
	Molecule *A*		Molecule *B*
	φ 93.2 (2)		φ 90.6 (2)
	φ' 77.0 (2)		φ' 79.6 (2)
C(1)—C(2)—N(1)—C(3)	$\omega 1$ −178.0 (2)	C(6)—C(7)—N(3)—C(8)	$\omega 3$ −178.4 (3)
C(3)—N(2)—C(4)—C(5)	$\omega 2$ −178.2 (3)	C(8)—N(4)—C(9)—C(10)	$\omega 4$ −178.1 (2)
O(1)—C(2)—N(1)—C(3)	2.5 (4)	O(3)—C(7)—N(3)—C(8)	−3.6 (4)
C(3)—N(2)—C(4)—O(2)	1.3 (4)	C(8)—N(4)—C(9)—O(4)	2.3 (4)

ecule assumes two nearly identical conformational states (A $\varphi = 93.2$, $\varphi' = 77.0$; B $\varphi = 90.6$, $\varphi' = 79.6°$) with planar *trans* amide functions. The bond lengths and bond angles are very close to the standard dimensions of the peptide group.

Experimental. Crystal size 0.18 × 0.12 × 0.08 mm, X-ray data were collected at room temperature on an Enraf–Nonius CAD-4 automatic diffractometer, with monochromated Cu $K\alpha$ radiation up to a θ value of 70° ($\omega/2\theta$-scanning mode). Cell parameters refined by least squares on the basis of 25 independent θ values in the range 20–30°. 1401 reflections measured ($h = -21$ to 21, $k = 0$ to 5, $l = 0$ to 22), 1318 with $F_o > 3\sigma(F_o)$ were used for all calculations. Three standards (221, 541, 801) measured every 2 h showed no deviations greater than 3%. Intensity data were corrected for Lorentz and polarization effects but not for absorption.

The diffraction patterns showed that both *Pnma* and $Pna2_1$ space groups (the first being centrosymmetrical and the second presenting two independent molecules in the asymmetric unit) were equally possible. Resolution of structure by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) failed for *Pnma*, but led to a rapid convergence for space group $Pna2_1$ with a pseudo-symmetrical centre ($x = 0.625$, $y = 0$, $z = 0.622$ not compatible with space group *Pnma*) between the two independent molecules.

Full-matrix least-squares refinement procedure on F (*SHELX*; Sheldrick, 1976) was applied using anisotropic temperature factors for non-H atoms, and fixed isotropic thermal factors for H atoms, which were located on difference maps. Final agreement factors were $R = 0.044$ and $wR = 0.051$ [$1/w = \sigma^2(F) + 0.006F^2$]. Goodness of fit = 0.8, $-0.25 < \Delta\rho < 0.30$ 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. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

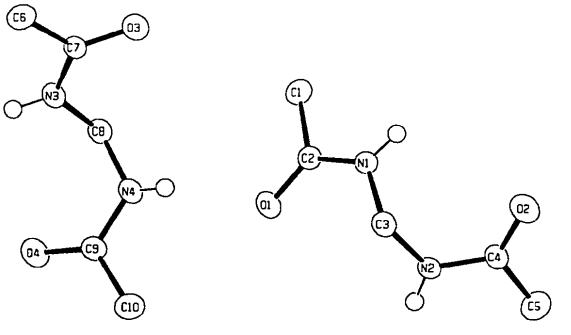

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the MeCO-Glyψ[NH-CO]NHMe molecule and interatomic distances (Å) associated with N—H···O hydrogen bond [O(1)···N(4^i) = 2.887 (4), O(2)···N(3^{ii}) = 2.916 (6), N(1)···O(4^{ii}) = 2.912 (5), N(2)···O(3^{iii}) = 2.874 (4). Symmetry code (i) x, y, z; (ii) $\frac{1}{2} - x, -\frac{1}{2} + y, -\frac{1}{2} + z$; (iii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} + z$].

Final atomic parameters are given in Table 1.* Bond distances, bond angles and torsional angles are given in Table 2 (IUPAC–IUB Commission on Biochemical Nomenclature, 1970; IUPAC–IUB Joint Commission on Biochemical Nomenclature, 1974). Fig. 1 shows a thermal ellipsoid plot with the atomic numbering scheme. The two pseudo-centrosymmetrical molecules *A* and *B* form cyclic dimers by means of two hydrogen bonds [N(1)···O(4) and N(3)···O(2)]. The dimers are also connected in such a way that each *A* (or *B*) molecule is hydrogen bonded to two other *B* (or *A*) molecules [N(2)···O(3) and N(4)···O(1)].

* 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 54436 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(*Received* 6 *June* 1991; *accepted* 3 *July* 1991)

Abstract. *N,N'*-Dimethylisopropylmalonamide, $C_8H_{16}N_2O_2$, $M_r = 172.23$, orthorhombic, *Pbcm*, $a = 4.859$ (1), $b = 13.523$ (2), $c = 15.469$ (2) Å, $V = 1016.4$ Å^3, $Z = 4$, $D_x = 1.12$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu R_{max} \ll 1$, $\mu = 5.88$ cm^{-1}, $F(000) = 376$, $T = 293$ K, $R = 0.060$ for 665 observed reflections. Dimensions of this retropeptide molecule are quite similar to the standard values for peptides. The C^α and C^β atoms are in a mirror ($z = \frac{1}{4}$), so conformational angles are $\psi' = -\psi = -110.4°$ (2).

Experimental. Crystal size $0.24 \times 0.8 \times 0.04$ mm, X-ray data were collected at room temperature on an Enraf–Nonius CAD-4 automatic diffractometer, with Cu $K\alpha$ radiation up to a θ value of 70° ($\theta/2\theta$-scanning mode). Cell parameters refined by least squares on the basis of 25 independent θ values in the range 20–30°. 864 reflections measured ($h = 0$ to 4, $k = 0$ to 16, $l = 0$ to 18), 665 with $F_o > 3\sigma(F_o)$ were used for all calculations. Three standards (400, 2$\bar{1}$0, 200) measured every 2 h showed no deviations greater than 2% in intensity. Intensity data were corrected for Lorentz and polarization effects but not for absorption.

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 H atoms appeared in difference maps. Refined parameters were calculated by using anisotropic temperature factors for non-H atoms and fixed isotropic temperature factors for H atoms. Final agreement factors were $R = 0.060$ and $wR = 0.070$ $\{w = 4.450/[\sigma^2(F_o) + 0.0009F_o^2]\}$. Goodness of fit = 2.25, $-0.31 < \Delta\rho < 0.19$ e Å^{-3}, shift/e.s.d.'s < 0.09. Following recommendations by Taylor & Kennard (1983), the NH H atoms were placed at 1.03 Å from N in the direction obtained by refinement. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

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