order of S > N > O, as measured by Rh—P distances, was thus established (Botha, Basson & Leipoldt, 1987). For bidentate ligands having the same donor atoms, like β-diketones, it was also found that the donor atom nearest to the most electronegative substituent has the largest *trans* influence. Exceptions to this phenomenon have been found in structure determinations, such as (1,1,1-trifluoro-5,5,5-trimethylpentanedionato)(carbonyl)(triphenylphosphine)-rhodium(I) (Leipoldt, Basson & Potgieter, 1986), and could be ascribed to steric intervention of bulky groups on the bidentate ligand preventing the formation of the expected isomer. The structure of [Rh(cupf)(CO)(PPh$_3$)] (Basson, Leipoldt, Roodt & Venter, 1986) also showed the normal expected isomer with P *trans* to the nitroso O atom. The present complex with a much less bulky but stronger π-bonding P ligand [cone angle = 101° compared to 145° of PPh$_3$ (Tolman, 1977)] unexpectedly gave the other isomer compared to [Rh(cupf)(CO)(PPh$_3$)]. This result is still under investigation.

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Structure of ^{t}BuCO-Gly-Glyψ[CH$_2$—N$^+$H$_2$]NHEt.BPh$_4^-$

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Abstract. *N*-(*tert*-Butylcarbonylglycylaminoethyl)-*N*-(ethyl)ammonium tetraphenylborate, $C_{11}H_{24}N_3O_2^+.C_{24}H_{20}B^-$, $M_r = 549.57$, triclinic, $P\bar{1}$, $a = 11.567\,(2)$, $b = 11.922\,(2)$, $c = 14.484\,(3)$ Å, $\alpha = 70.99\,(2)$, $\beta = 74.83\,(2)$, $\gamma = 59.33\,(1)°$, $V = 1613.1$ Å^3, $Z = 2$, $D_x = 1.13$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1.5418$ Å, $\mu = 4.69$ cm^{-1}, $\mu R_{\max} \ll 1$, $F(000) = 592$, $T = 293$ K, $R = 0.058$ for 3491 observed reflections. This pseudopeptide is folded by a short N$^+$—H···O═C hydrogen bond (N3···O1 = 2.81 Å) which closes a ten-membered ring. This results in a β-turn structure that can be classified as type II on the basis of the conformational angles for the N-terminal glycine. The conformational angles φ_1, ψ_1, φ_2 and ψ_2 are $-53.4\,(6)$, $139.7\,(4)$, $91.5\,(5)$ and $-62.6\,(6)°$ respectively.

Experimental. Crystal size 0.20 × 0.10 × 0.18 mm, X-ray data were collected at room temperature on Enraf–Nonius CAD-4 automatic diffractometer, with Cu $K\alpha$ radiation up to 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°. 6108 reflections measured ($h = -14$ to 14, $k = -14$ to 14, $l = 0$ to 17), 3491 with $F_o > 3\sigma(F_o)$ were used for all calculations. Three standards (212, 022, 002) 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 the non-H atoms, and the H atoms appeared in difference maps. Refined parameters were calculated using

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Table 1. *Fractional coordinates with standard deviations and equivalent isotropic thermal parameters*

	x	y	z	B_{eq} (Å^2)
^{t}BuCO-Gly-Glyψ(CH$_2$N$^+$H$_2$)NHEt				
N(1)	0.2702 (3)	0.4088 (3)	0.2668 (2)	5.5 (1)
N(2)	0.3282 (3)	0.3723 (3)	0.0247 (2)	5.7 (1)
N(3)	0.6204 (3)	0.2603 (3)	0.0325 (2)	4.6 (1)
O(1)	0.4807 (2)	0.2653 (2)	0.2226 (1)	4.8 (1)
O(2)	0.3429 (2)	0.5239 (2)	0.0739 (2)	6.1 (1)
C(1)	0.3354 (6)	0.4227 (8)	0.4337 (5)	12.1 (2)
C(2)	0.4828 (9)	0.1904 (6)	0.4367 (4)	12.9 (3)
C(3)	0.5604 (9)	0.352 (1)	0.3479 (5)	17.6 (4)
C(4)	0.4426 (3)	0.3269 (4)	0.3740 (2)	5.7 (1)
C(5)	0.3997 (3)	0.3322 (3)	0.2822 (2)	4.5 (1)
C(6)	0.2203 (4)	0.4117 (5)	0.1859 (3)	5.9 (1)
C(7)	0.3021 (3)	0.4414 (3)	0.0897 (2)	4.7 (1)
C(8)	0.4256 (4)	0.3666 (4)	−0.0621 (3)	5.6 (1)
C(9)	0.5618 (4)	0.2506 (4)	−0.0425 (3)	6.0 (1)
C(10)	0.7575 (4)	0.1499 (4)	0.0524 (3)	9.0 (2)
C(11)	0.8165 (5)	0.1724 (6)	0.1175 (4)	9.0 (2)
H(N1)	0.205	0.461	0.319	
H(N2)	0.294	0.302	0.046	
H1(N3)	0.635	0.344	0.004	
H2(N3)	0.563	0.266	0.100	
BPh$_4^-$				
B	0.2245 (3)	0.2668 (3)	−0.2276 (2)	3.9 (1)
C(12)	0.3872 (3)	0.2012 (3)	−0.2689 (2)	3.9 (1)
C(13)	0.4582 (3)	0.2744 (3)	−0.3050 (2)	4.8 (1)
C(14)	0.5959 (4)	0.2169 (4)	−0.3354 (3)	6.2 (1)
C(15)	0.6689 (3)	0.0826 (4)	−0.3329 (3)	5.8 (1)
C(16)	0.6036 (3)	0.0059 (4)	−0.2981 (2)	4.9 (1)
C(17)	0.4660 (3)	0.0639 (3)	−0.2662 (2)	4.5 (1)
C(18)	0.2071 (3)	0.2409 (3)	−0.1071 (2)	4.0 (1)
C(19)	0.3020 (3)	0.1298 (3)	−0.0476 (2)	4.3 (1)
C(20)	0.2806 (3)	0.1008 (3)	0.0540 (2)	4.9 (1)
C(21)	0.1619 (3)	0.1820 (3)	0.1020 (2)	5.1 (1)
C(22)	0.0683 (3)	0.2941 (4)	0.0476 (2)	5.2 (1)
C(23)	0.0919 (3)	0.3231 (4)	−0.0536 (2)	4.9 (1)
C(24)	0.1492 (3)	0.4287 (3)	−0.2797 (2)	4.1 (1)
C(25)	0.0788 (3)	0.4804 (3)	−0.3616 (2)	4.9 (1)
C(26)	0.0224 (4)	0.6142 (4)	−0.4088 (3)	5.9 (1)
C(27)	0.0329 (4)	0.7041 (4)	−0.3770 (3)	5.8 (1)
C(28)	0.0997 (4)	0.6590 (4)	−0.2963 (3)	5.7 (1)
C(29)	0.1547 (3)	0.5250 (3)	−0.2495 (3)	4.8 (1)
C(30)	0.1476 (3)	0.1997 (3)	−0.2560 (2)	4.4 (1)
C(31)	0.0241 (4)	0.2087 (4)	−0.2079 (3)	6.5 (1)
C(32)	−0.0499 (4)	0.1712 (5)	−0.2403 (4)	8.9 (2)
C(33)	−0.0023 (5)	0.1203 (5)	−0.3207 (4)	9.2 (2)
C(34)	0.1210 (4)	0.1046 (4)	−0.3697 (3)	6.8 (1)
C(35)	0.1931 (4)	0.1448 (3)	−0.3379 (2)	5.3 (1)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and torsional angles* (°) *for* ^{t}BuCO-Gly-Gly-ψ(CH$_2$N$^+$H$_2$)NHEt

N(1)—C(5)	1.333 (4)	C(1)—C(4)	1.492 (7)
N(1)—C(6)	1.419 (6)	C(2)—C(4)	1.475 (7)
N(2)—C(7)	1.327 (6)	C(3)—C(4)	1.469 (13)
N(2)—C(8)	1.446 (4)	C(4)—C(5)	1.510 (6)
N(3)—C(9)	1.483 (7)	C(6)—C(7)	1.514 (5)
N(3)—C(10)	1.480 (4)	C(8)—C(9)	1.493 (4)
O(1)—C(5)	1.233 (3)	C(10)—C(11)	1.454 (10)
O(2)—C(7)	1.229 (5)		

C(5)—N(1)—C(6)	122.1 (3)	N(1)—C(5)—C(4)	118.8 (3)
C(7)—N(2)—C(8)	124.2 (4)	O(1)—C(5)—C(4)	121.7 (3)
C(9)—N(3)—C(10)	114.8 (3)	N(1)—C(6)—C(7)	111.3 (4)
C(1)—C(4)—C(2)	107.4 (4)	N(2)—C(7)—O(2)	122.4 (3)
C(1)—C(4)—C(3)	108.6 (6)	N(2)—C(7)—C(6)	116.1 (4)
C(1)—C(4)—C(5)	114.9 (4)	O(2)—C(7)—C(6)	121.5 (4)
C(2)—C(4)—C(3)	108.5 (6)	N(2)—C(8)—C(9)	111.4 (3)
C(2)—C(4)—C(5)	107.5 (5)	N(3)—C(9)—C(8)	112.6 (4)
C(3)—C(4)—C(5)	109.7 (4)	N(3)—C(10)—C(11)	112.5 (4)
N(1)—C(5)—O(1)	119.5 (3)		

C(4)—C(5)—N(1)—C(6)	−175.5 (4)	C(9)—N(3)—C(10)—C(11)	173.2 (4)
C(6)—C(7)—N(2)—C(8)	−167.3 (3)	O(1)—C(5)—C(4)—C(1)	174.8 (5)
C(8)—C(9)—N(3)—C(10)	−178.1 (3)	O(1)—C(5)—C(4)—C(2)	−65.6 (5)
O(1)—C(5)—N(1)—C(6)	2.9 (6)	O(1)—C(5)—C(4)—C(3)	52.2 (6)
O(2)—C(7)—N(2)—C(8)	11.9 (4)		

φ_1 −53.4 (6) ψ_1 139.7 (4) φ_2 91.5 (5) ψ_2 −62.6 (6)

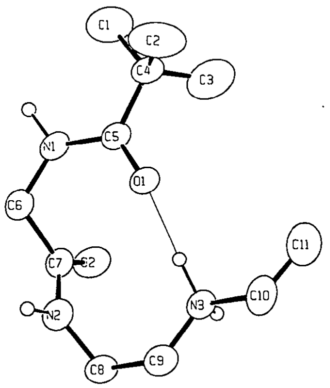

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the ^{t}BuCO-Gly-Glyψ[CH$_2$N$^+$H$_2$]NHEt molecule and interatomic distances (Å) associated with N—H···O hydrogen bonds [N(3)···O(1^i) = 2.806 (3), N(3)···O(2^{ii}) = 2.714 (4). Symmetry code: (i) x, y, z; (ii) $1-x$, $1-y$, $-z$].

anisotropic temperature factors for non-H atoms and fixed isotropic temperature factors for H atoms. Final agreement factors were $R = 0.058$ and $wR = 0.061$ $\{w = 1.960/[\sigma^2(F_o) + 0.0007F_o^2]\}$. Goodness of fit = 1.65, $-0.35 < \Delta\rho < 0.21$ e Å^{-3}, shift/e.s.d.'s < 0.10. 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).

Final atomic parameters are given in Table 1.* Bond distances, bond angles and conformational angles are given in Table 2 (IUPAC–IUB Commission on Biochemical Nomenclature, 1970; IUPAC–IUB Joint Commission on Biochemical Nomenclature, 1984). Fig. 1 shows a thermal ellipsoid plot with the atomic numbering scheme.

The conformation is very similar to those observed in ^{t}BuCO-L-Pro-Glyψ[CH$_2$N$^+$H$_2$]NHEt and in form I of ^{t}BuCO-L-Pro-Glyψ(CH$_2$N$^+$HMe)NHMe (El-Masdouri, Aubry, Sakarellos, Gomez & Marraud, 1988) and closely resembles the βII-folded conformation of the parent ^{t}BuCO-Gly-Gly-NH^{i}Pr dipeptide (Aubry, Lecomte, Boussard & Marraud, 1983).

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* Lists of structure factors, anisotropic thermal parameters, bond distances and angles for BPh$_4^-$ and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54433 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of ^{t}BuCO-Valψ[NH-CO]NH^{t}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* ^{t}BuCO-Valψ[NH-CO]NH^{t}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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