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Supporting Information

For

A Synthetic Strategy for the Preparation of Cyclic Peptide Mimetics Based on SET-Promoted Photocyclization Processes

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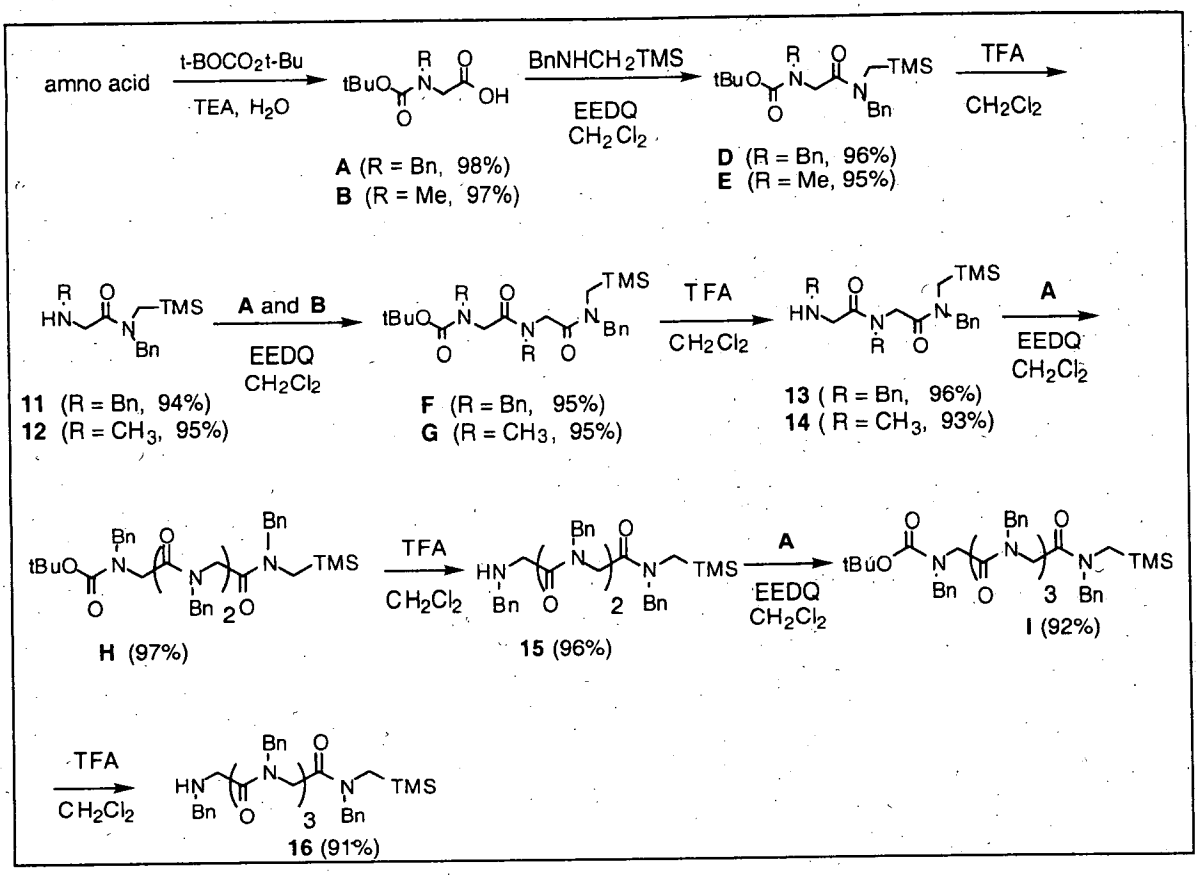

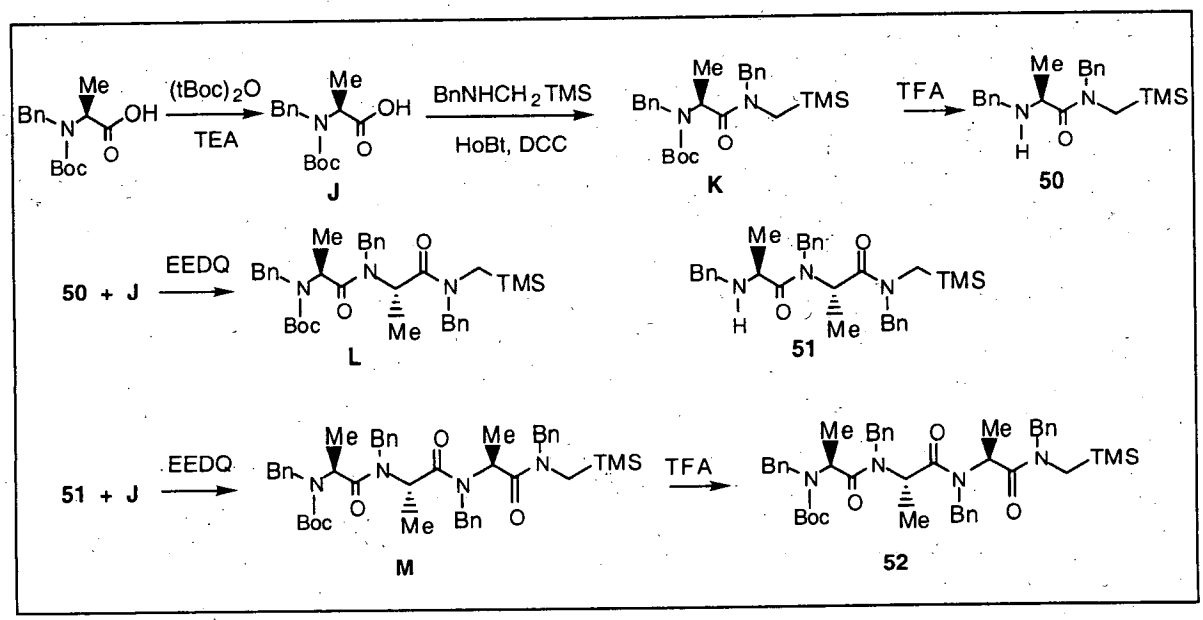

General. ^{1}H-NMR and ^{13}C-NMR spectra were recorded using 200 MHz, 300 MHz and 500 MHz spectrometers. All new compounds, characterized in this study, are oils (unless denoted as crystalline solids by their melting points) and were judged to be >90% pure by using ^{1}H-NMR and ^{13}C-NMR analyses. Low and high resolution mass spectral analyses were performed by using either EI, CI or FAB ionization techniques. All preparative irradiations were conducted by using and immersion apparatus (Hanovia 450 watt, medium pressure lamp) at a solution temperature of *ca.* 20 °C and using a Pyrex glass cut-off filter ($\lambda > 290$ nm).

N-(t-BOC)glycines A and B. Independent solutions of N-benzylglycine (1.65 g, 10.0 mmol) and sarcosine (0.89 g. 10.0 mmol) in 100 mL of water containing triethylamine (4.2 mL, 30 mmol) and di-tert-butyldicarbonate (2.18 g, 10.0 mmol) was stirred for 3 h at 25 ^{0}C, poured into aq. HCl and extracted with ethyl acetate. The ethyl acetate extracts were washed with water, dried and concentrated *in vacuo* to yield 2.60 g (98%) of **A** and 1.83 g (97%) of **B**.

A (10:9 mixture of two rotamers): ^{1}H-NMR ($CDCl_3$) of rotamer A 1.48 (s, 9H, CH_3), 3.83 (s, 2H, $C\underline{H}_2COOH$), 4.54 (s, 2H, $C\underline{H}_2Ph$), 7.25-7.35 (m, 5H, aromatic), 8.80 (br, 1H, COOH), rotamer B 1.49 (s, 9H, CH_3), 3.96 (s, 2H, $C\underline{H}_2COOH$), 4.52 (s, 2H, $C\underline{H}_2Ph$), 7.25-7.35 (m, 5H, aromatic), 8.80 (br, 1H, COOH); ^{13}C-NMR($CDCl_3$) of rotamer A 28.2 ($C(\underline{C}H_3)_3$), 47.5 ($\underline{C}H_2COOH$), 51.5 ($\underline{C}H_2Ph$), 81.1 ($(CH_3)_3\underline{C}$), 127.5, 128.1, 128.5 and 137.0 (aromatic), 156.0 and 175.9 (C=O), rotamer B 28.3 ($C(\underline{C}H_3)_3$), 47.6 ($\underline{C}H_2COOH$), 50.8 ($\underline{C}H_2Ph$), 80.9 ($(CH_3)_3\underline{C}$), 127.6, 128.2, 128.7 and 137.2 (aromatic), 155.6 ($(CH_3)_3CO\underline{C}$=O) 175.7 (COOH); MS(FAB) m/z (relative intensity) 266 (M^++1, 6), 220 (3), 210 (39), 166 (34), 91 (100), 57 (46); HRMS (FAB) m/z 266.1397 ($C_{14}H_{18}NO_4$ requires 266.1392).

B (10:9.5 mixture of two rotamers based on ^{1}H-NMR integration): ^{1}H-NMR ($CDCl_3$) of rotamer A 1.42 (s, 9H, $C(CH_3)_3$), 2.92 (s, 3H, CH_3), 3.94 (s, 2H, $C\underline{H}_2COOH$), 9.70 (s, 1H, COOH), rotamer B 1.46 (s, 9H, $C(CH_3)_3$), 2.92 (s, 3H, CH_3), 4.02 (s, 2H, $C\underline{H}_2COOH$); 9.70 (s, 1H, COOH); ^{13}C-NMR($CDCl_3$) of rotamer A 28.3 ($(\underline{C}H_3)_3C$), 35.5 ($\underline{C}H_3N$), 50.2 ($\underline{C}H_2COOH$), 80.5 ($(CH_3)_3\underline{C}$), 155.5 ($(CH_3)_3CO\underline{C}$=O), 175.1 (COOH), rotamer B 28.4 ($(\underline{C}H_3)_3C$), 35.6 ($\underline{C}H_3N$), 50.7 ($\underline{C}H_2COOH$), 80.6 ($(CH_3)_3\underline{C}$), 156.4 ($(CH_3)_3CO\underline{C}$=O), 175.5 (COOH), MS(EI), m/z(relative intensity) 189(M^+, 3), 145(16), 130(5), 88(81), 57(100), HRMS(EI), 189.0999 ($C_8H_{15}O_4N$ requires 189.1001).

N-(t-BOC)-Trimetylsilylmethylglycinamides D and E. Independent solutions of the blocked glycines **A** (2.65 g, 10.0 mmol), and **B** (1.89 g, 10.0 mmol) in CH_2Cl_2 (30 mL) containing EEDQ (3.72 g, 15.0 mmol) and N-trimethylsilylmethyl-N-benzylamine (2.20 mL, 10.0 mmol) was stirred for 3h at 25 ^{0}C, poured into 2N aq HCl and extracted with CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, dried and concentrated *in vacuo* to yield 4.22 g (96%) of the glycinamides **D** and 3.42g (95%) of **E**.

D (64:36:1 mixture of 3 rotamers): ^{1}H-NMR ($CDCl_3$) 0.02, 0.05 and 0.15 (s, 9H, $Si(C\underline{H}_3)_3$, 3 peaks for 3 rotamers), 1.44, 1.46 and 1.47 (s, 9H, $(C\underline{H}_3)_3C$), 2.60, 2.70and 2.89 and (s, 2H, $C\underline{H}_2TMS$), 3.85, 3.76 and 3.90 (s, 2H, CH_2CO), 4.34, 4.46, 4.50, 4.56, 4.59 and 4.62 (s, 4H, $C\underline{H}_2Ph$), 7.08-7.36 (m, 5H, aromatic); ^{13}C-NMR($CDCl_3$) -1.5, -1.3 and –1.0 ($Si(CH_3)_3$), 28.2, 28.3 and 28.4 ($(\underline{C}H_3)_3C$), 37.2, 38.6 and 38.0 ($\underline{C}H_2TMS$), 46.5, 46.8 and 47.2 ($\underline{C}H_2CO$), 50.3, 50.6, 50.8, 51.1, 52.2 and 52.6 ($\underline{C}H_2Ph$), 80.0 80.1 and 80.2 ($(CH_3)_3\underline{C}$), 126.2, 126.5, 127.2, 127.4, 127.5, 127.7, 127.9, 128.1, 128.3, 128.4, 128.5, 128.6, 128.8, 128.9, 136.1, 136.4, 136.9, 138.0 and 138.1 (aromatic), 155.6, 155.7 and 156.0 ($(CH_3)_3CO\underline{C}$=O), 167.2, 167.9 and 168.0 (C=O); MS(FAB) m/z(relative intensity) 441(M^++1, 17), 425(10), 369(21), 341(100), 91(85), HRMS(FAB) m/z 441.2582 ($C_{25}H_{37}N_2O_3Si$ requires 441.2573).

E (8.3:4:1 mixture of 3 rotamers): ^{1}H-NMR($CDCl_3$) 0.04, 0.11 and 0.13 (s, 9H, $Si(CH_3)_3$, 3 peaks for 3 rotamers), 1.40,1.41 and 1.45 (s, 9H, $(C\underline{H}_3)_3C$), 2.90, 2.92 and 2.96 (m, 2H, $C\underline{H}_2TMS$), 2.94, 2.95 and 2.97(s, 3H, CH_3), 3.95, 3.99 and 4.05 (s, 2H, CH_2CO),

4.44, 4.51 and 4.59 (s, 2H, $C\underline{H}_2Ph$), 7.10-7.36 (m, 5H, aromatic), ^{13}C-NMR($CDCl_3$) -1.3, -1.4 and 1.5 ($Si(CH_3)_3$), 28.2 (($\underline{C}H_3)_3C$), 35.3, 35.4 and 35.5 (NCH_3), 37.2, 38.7 and 38.9 ($\underline{C}H_2TMS$), 49.9, 50.3 and 50.5 ($N\underline{C}H_2CO$), 51.0, 52.3 and 52.7 ($\underline{C}H_2Ph$), 79.7, 79.8 and 80.0 (($CH_3)_3\underline{C}$), 126.2, 126.4, 127.2, 127.5, 127.7, 127.8, 128.4, 128.5, 128.8, 136.2, 136.5 and 136.9, (aromatic), 155.6, 155.7 and 155.9 (($CH_3)_3C\underline{C}$=O), 167.3, 168.0 and 169.8 (C=O); MS(FAB) m/z(relative intensity) 365(M^++1, 40), 425(10), 349(21), 265(100), 192(12), 173(5), 91(62), HRMS(FAB) m/z 365.2257 ($C_{19}H_{33}N_2O_3Si$ requires 365.2260).

Trimetylsilylmethylglycinamides 11 and 12. Independent solutions of the blocked glycinamides **D** (2.20 g, 5.0mmol) and **E** (1.82 g, 5.0mmol) in CH_2Cl_2 (20 mL) containing 5 mL of 98%TFA was stirred for 4 h at 25 ^{0}C, poured into saturated aq K_2CO_3 and extracted with CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, dried and concentrated *in vacuo* to afford 1.59 g (94%) of **11** and 1.25 g (95%) of **12**.

11 (1.4:1 mixture of 2 rotamers) ^{1}H-NMR ($CDCl_3$) of rotamer A 0.07 (s, 9H, $Si(CH_3)_3$), 2.93 (s, 2H, $C\underline{H}_2TMS$), 3.49 (s, 2H, CH_2CO), 3.81 (s, 2H, $PhC\underline{H}_2N(H)$), 3.91 (br, 1H, NH), 4.41 (s, 2H, $PhC\underline{H}_2N(CH_2TMS)$), 7.09-7.37 (m, 10H, aromatic), rotamer B 0.06 (s, 9H, $Si(CH_3)_3$), 2.69 (s, 2H, $C\underline{H}_2TMS$), 3.45 (s, 2H, $C\underline{H}_2CO$), 3.89 (s, 2H, $PhC\underline{H}_2N(H)$), 3.91 (br, 1H, NH), 4.62 (s, 2H, $PhC\underline{H}_2NCH_2TMS$), 7.09-7.37 (m, 10H, aromatic); ^{13}C-NMR($CDCl_3$) of rotamer A -1.3 ($Si(CH_3)_3$), 38.8 ($\underline{C}H_2TMS$), 49.1 ($NH\underline{C}H_2CO$), 52.3 ($PhCH_2N(H)$), 53.4 ($PhCH_2NCH_2TMS$), 126.4, 127.7, 128.4, 128.5, 128.9 and 136.2 (aromatic), 169.8 (C=O), rotamer B -1.5 ($Si(CH_3)_3$), 37.0 ($\underline{C}H_2TMS$), 49.5 ($NH\underline{C}H_2CO$), 50.3 ($PhCH_2N(H)$), 53.5 ($PhCH_2NCH_2TMS$), 127.1, 127.4, 127.9, 128.4, 128.6 and 139.1 (aromatic), 169.9 (C=O), MS(FAB) m/z(relative intensity) 341(M^++1, 21) 217(4), 191(10), 120(17), 91(100), HRMS(FAB) m/z 341.2037 ($C_{20}H_{29}N_2OSi$ requires 341.2049).

12 (2:1 mixture of 2 rotamers) ^{1}H-NMR ($CDCl_3$) of rotamer A 0.03 (s, 9H, $Si(CH_3)_3$), 2.39(s, 3H, CH_3), 2.88 (s, 2H, $C\underline{H}_2TMS$), 3.10 (s, 1H, NH), 3.41 (s, 2H, CH_2CO), 4.43 (s, 2H, $C\underline{H}_2Ph$), 7.10-7.37 (m, 5H, aromatic). rotamer B 0.09 (s, 9H, $Si(CH_3)_3$), 2.46 (s, 3H, CH_3), 2.67 (s, 2H, $C\underline{H}_2TMS$), 3.10 (s, 1H, NH), 3.11 (s, 2H, CH_2CO), 4.59 (s, 2H, $C\underline{H}_2Ph$), 7.10-7.37 (m, 5H, aromatic), ^{13}C-NMR ($CDCl_3$) of rotamer A -1.4 ($Si(CH_3)_3$), 36.1 (CH_3), 38.6 ($\underline{C}H_2TMS$), 51.7 ($NH\underline{C}H_2CO$), 52.2 ($\underline{C}H_2Ph$), 126.2, 127.6, 128.4 and 136.1 (aromatic), 169.6 (C=O), rotamer B -1.5 ($Si(CH_3)_3$), 36.2 (CH_3), 36.9 ($\underline{C}H_2TMS$), 50.1 ($NH\underline{C}H_2CO$), 52.2 ($\underline{C}H_2Ph$), 127.3, 127.8, 128.5 and 136.8 (aromatic), 169.6 (C=O), MS(FAB) m/z(relative intensity) 287(M^++Na, 100), 265(M^++1, 55) 249(7), 191(20), 91(62), HRMS(FAB) m/z 265.1726 ($C_{14}H_{25}N_2O_3Si$ requires 265.1736).

N-(t-BOC)-N-Trimethylsilylmethyl peptides F and G. Independent solutions of mixtures of glycine **A** (1.33 g, 5.0 mmol) and glycinamide **11** (1.70 g, 5.0 mmol) and of glycine **B** (0.95 g, 5.0 mmol) and glycinamide **12** (1.32 g, 5.0 mmol) in CH_2Cl_2 (45 mL), each containing EEDQ (1.86 g, 7.5 mmol), were stirred for 3 h at 25 ^{0}C, poured into 2N aq HCl, and extracted with CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, dried and concentrated *in vacuo* to yield 2.79 g (100%) of **F** and 2.07 g (95%) of **G**.

F (17.1:3.0:2.9:2.5:1 mixture of 5 rotamers): ^{1}H-NMR ($CDCl_3$) -0.01, 0.0, 0.03, 0.05 and 0.06 (s, 9H, $Si(CH_3)_3$ 5 peaks for 5 rotamers), 1.38, 1.40, 1.41, 1.43 and 1.45 (s, 9H, $(CH_3)_3CO$), 2.69, 2.71, 2.86, 2.89 and 2.92 (s, 2H, CH_2TMS), 3.70, 3.84, 3.92, 4.00, 4.12,

4.15, 4.25, 4.45, 4.50, 4.54 and 4.59 (s, 2H, CH_2), 6.90-7.77 (m, 15H, aromatic); ^{13}C-NMR($CDCl_3$) -1.5, -1.4, -1.3, -1.2 and –1.0 ($Si(CH_3)_3$), 28.3 ($(CH_3)_3\underline{C}$), 37.4 and 38.9 ($\underline{C}H_2TMS$), 46.0, 46.8, 47.2, 50.1, 50.4, 51.0, 51.2, 52.2 and 52.8 (CH_2), 80.2 ($(CH_3)\underline{C}$),125.9, 126.4, 126.7, 127.2, 127.3, 127.7, 127.8, 128.2, 128.3, 128.4, 128.5, 128.6, 128.9, 129.1, 135.7, 136.2, 136.7 and 137.9 (aromatic), 155.9 ($(CH_3)_3C\underline{C}$=O), 167.0 and 169.3 (C=O), MS(FAB) m/z(relative intensity) 588(M^++1, 7), 488(50), 367(6), 339(8), 295(7), 192(17), 120(46), 91(100), HRMS(FAB) m/z 588.3242 ($C_{34}H_{45}N_3O_3Si$ requires 588.3258).

G (5.5:1.3:1 mixture of 3 rotamers): ^{1}H-NMR ($CDCl_3$) -0.03, 0.00 and 0.06 (s, 9H, $Si(CH_3)_3$, 3 peakes for 3 rotamers), 1.21, 1.34 and 1.37, (s, 9H, $C(CH_3)_3$), 2.71, 2.72 and 2.78 (s, 2H, $C\underline{H}_2TMS$), 2.83 ,2.87 and 2.90 (s, 3H, $OCN(C\underline{H}_3)CH_2CO$), 2.94, 2.96 and 3.02 (s, 3H, $C\underline{H}_3NCOO$), 3.90, 3.91 and 3.95 (s, 2H, $C\underline{H}_2CONCH_2TMS$), 4.05, 4.08 and 4.15 (s, 2H, $C\underline{H}_2Ph$,), 4.40, 4.50 and 4.46, (s, 2H, $OOCN(CH_3)C\underline{H}_2$), 7.08-7.32 (m, 5H, aromatic); ^{13}C-NMR($CDCl_3$) -1.4, -1.1 and 0.0 ($Si(CH_3)_3$), 28.2, 28.3 and 28.4 ($(\underline{C}H_3)_3C$), 35.3, 35.4 and 35.5 ($\underline{C}H_3NCOO$), 35.6, 35,7 and 35.8 ($OCN(\underline{C}H_3)CH_2CO$), 38.7, 38.8 and 38.9 ($\underline{C}H_2TMS$), 49.1, 49.2, 49.6, 50.4, 51.2, 51.6, 51.7, 51.8 and 52.7(CH_2), 79.7 ($(CH_3)_3\underline{C}$), 126.2, 126.5, 126.7, 127.3, 127.6, 127.7, 128.5, 128.9, 129.1, 129.6, 136.3 and 139.2 (aromatic), 155.5 ($(CH_3)_3CO\underline{C}$=O), 167.2 and 168.8 (C=O); MS(FAB) m/z(relative intensity) 436(M^++1, 20), 420(11), 336(100), 265(51), 192(10), 91(52), HRMS(FAB) m/z 436.2624 ($C_{22}H_{118}O_4N_3Si$ requires 436.2632)

N-Trimethylsilylmethyl peptides 13 and 14. Independent solutions of the N-(t-BOC)-N-trimetylsilylmethylpeptides **F** (2.93 g, 5.0 mmol) and **G** (2.18 g, 5.0 mmol) in CH_2Cl_2 (30 mL) containing 5 mL of 98%TFA was stirred for 4 h at 25 ^{0}C, pored into aq K_2CO_3, and extracted with CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, dried and concentrated *in vacuo* to afford 2.34 g (96%) of **13** and 1.56 g (93%) of **14.**

13 (14.1:11:1.2:1 mixture of 4 rotamers) ^{1}H-NMR ($CDCl_3$) -0.04, -0.02, 0.04 and 0.05 (s, 9H, $Si(CH_3)_3$ 4 peaks for 4 rotamers), 2.55, 2.69, 2.88 and 2.96 (s, 2H, $C\underline{H}_2TMS$), 3.31, 3.41, 3.54 and 3.61 (s, 2H, $PhCH_2N(H)C\underline{H}_2$), 3.79, 3.81, 3.86 and 3.92 (s, 2H, $C\underline{H}_2CONCH_2TMS$), 4.14, 4.17, 4.18 and 4.19 (s, 2H, $PhC\underline{H}_2NH$), 4.28 (s, 1H, NH), 4.44, 4.45, 4.58 and 4.61 (s, 2H, $PhC\underline{H}_2NCH_2TMS$), 4.56, 4.60, 4.71 and 4.72 (s, 2H, $PhC\underline{H}_2N(CO)CH_2$), 7.00-7.35 (m, 15H, aromatic); ^{13}C-NMR($CDCl_3$) -1.5, -1.4 and -1.3 ($Si(CH_3)_3$), 30.9, 33.1, 37.4, 38.5, 38.9, 39.9, 46.2, 46.9, 49.2, 49.9, 51.1, 52.5, 52.7 and 53.3 (CH_2), 125.8, 126.4, 127.0, 127.1, 127.2, 127.4, 127.5, 127.7, 127.8, 127.9, 128.4, 128.5, 128.6, 128.9, 129.2, 135.7, 136.0, 136.7 and 139.4 (aromatic), 166.2, 167.0, 167.2, 171.8 and 172.0 (C=O); MS(FAB) m/z (relative intensity) 488(M^++1, 55), 425(10), 339(6), 295(6), 192(10), 120(20), 91 (100), HRMS (FAB) m/z 488.2745 ($C_{29}H_{38}N_3O_2Si$ requires 488.2733).

14 (17.3:5.9:5.9:3.1:2.2:1.8:1.7:1 mixture of 8 rotamers): ^{1}H-NMR($CDCl_3$) -0.03, 0.01, 0.02, 0.04, 0.05, 0.10, 0.12, and 0.14 (s, 9H, $Si(CH_3)_3$, 8 peaks for 8 rotomers), 2.63, 2.67, 2.68, 2.72 and 2.75 (s, 2H, $C\underline{H}_2TMS$), 2.84, 2.90, 2.94, 2.97, 3.00 and 3.05 (s, 6H, $2CH_3$), 3.75, 3.85, 3.91 and 3.97 (s, 2H, $C\underline{H}_2CONCH_2TMS$), 4.02, 4.05, 4.10, 4.15, 4.20, and 4.29 (s, 2H, $CH_3N(H)C\underline{H}_2$), 4.28 (s, 1H, NH), 4.41, 4.46, 4.50 and 4.56 (s, 2H, $C\underline{H}_2Ph$), 7.12-7.35 (m, 5H, aromatic); ^{13}C-NMR($CDCl_3$); -1.5 and -1.4 ($Si(CH_3)_3$), 33.6 ($\underline{C}H_2TMS$), 35.2, 35.5 and 35.7 ($\underline{C}H_3N(H)CH_2$), 37.6, 38.0, 39.4 and 41.8 ($OCN(CH_3)\underline{C}H_2CO$), 48.7,

49.2, 49.3, 49.4, 50.4 and 50.8 ($CH_3N(H)\underline{C}H_2$), 52.4, 52.6 and 52.8 ($\underline{C}H_2Ph$), 125.9, 126.3, 127.7, 127.9, 128.1, 128.7, 129.1, 129.3, 135.8 and 136.3 (aromatic), 165.7 and 166.7 (C=O); MS(FAB) m/z(relative intensity) 358 (M^++Na, 100), 336(M^++1, 58), 320(7), 265(30), 192(5), 143(16), 91(60); HRMS(FAB) m/z 336.2112 ($C_{17}H_{30}N_3O_2Si$ requires 336.2107).

N-(t-BOC)-N-Trimethylsilylmethylpeptides H and I. Solutions of blocked glycine **A** (1.33 g, 5.0 mmol) in CH_2Cl_2 (55 mL) containing EEDQ (1.86 g, 7.5 mmol) and either amines **13** (2.44 g, 5.0 mmol) or **15** (3.16 g, 5.0 mmol) were stirred for 3 h at 25 ^{0}C, poured into 2N aq HCl and extracted with CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, dried and concentrated *in vacuo* to yield 3.56 g (97%) of **H** and 4.05 g (92%) of **I**.

H (3.5:2.4:2.3:1.3:1.2:1.1:1.1:1 mixture of 8). ^{1}H-NMR($CDCl_3$) -0.04, -0.03, 0.00, 0.02, 0.05, 0.06, 0.08 and 0.12 (s, 9H, $Si(CH_3)_3$, 8 peaks for 8 rotamers), 1.44 (s, 9H, $(CH_3)_3CO$), 2.72, 2.98 and 3.00 (s, 2H, $C\underline{H}_2TMS$), 3.65, 3.70, 3.78, 3.85, 3.88, 3.91, 4.01, 4.02, 4.03, 4.12, 4.18, 4.22, 4.24, 4.27, 4.30, 4.31, 4.32, 4.34, 4.44, 4.49, 4.51, 4.56 and 4.59 (s, 2H, CH_2), 6.94-7.55 (m, 20H, aromatic); ^{13}C-NMR($CDCl_3$) -1.5, -1.4 and -1.3 ($Si(CH_3)_3$), 28.3 ($(\underline{C}H_3)_3C$), 36.7, 37.3, 38.7, 38.9, 39.0, 39.2 and 40.1($\underline{C}H_2TMS$), 45.8, 46.1, 46.4, 46.6, 47.1, 47.3, 50.1, 50.3, 50.4, 50.6, 51.0, 51.1, 51.3, 52.2 and 52.7 (CH_2), 80.2 ($(CH_3)_3\underline{C}$), 125.5, 125.9, 126.1, 126.3, 126.5, 126.7, 126.8, 127.0, 127.1, 127.3, 127.4, 127.6, 127.7, 127.8, 127.9, 128.3, 128.5, 128.6, 128.7, 128.8, 128.9, 129.0, 129.1, 135.7, 135.9, 136.5, 136.7 and 137.9 (aromatic), 155.9 ($(CH_3)_3C(O)\underline{C}$=O), 166.9, 168.3, 169.1, 169.6 and 169.8 (C=O).

I (3:2.0:2.1:1.5:1.04:1.01:1.01:1 mixture of 8 rotamers): ^{1}H-NMR($CDCl_3$) -0.06, -0.04, -0.02, -0.01, 0.01, 0.04, 0.05 and 0.06 (s, 9H, $Si(CH_3)_3$, 8 peakes for 8 rotamers), 1.43 (s, 9H, $(C\underline{H}_3)_3C$), 2.67, 2.82, 2.86 and 2.93 (s, 2H, $C\underline{H}_2TMS$), 3.68, 3.72, 3.86, 3.89, 3.93, 4.01, 4.06, 4.10, 4.11, 4.13, 4.17, 4.20, 4.22, 4.23, 4.25, 4.29, 4.33, 4.43, 4.45, 4.56, 4.58 and 4.68 (s, 2H, CH_2), 6.81-7.01 (m, 25H, aromatic); ^{13}C-NMR($CDCl_3$) -1.6, -1.5, -1.4, -1.3 and -1.2 ($Si(CH_3)_3$), 28.0 ($(\underline{C}H_3)_3C$), 37.3, 38.6, 38.9 and 39.6 ($\underline{C}H_2TMS$), 45.8, 46.4, 47.3, 50.2, 50.4, 50.9, 51.3, 52.2 and 52.7 (CH_2), 80.2 ($(CH_3)_3\underline{C}$), 125.6, 125.9, 126.3,126.4, 126.5, 127.1, 127.3, 127.7, 127.8, 128.4, 128.5, 128.5, 128.6, 128.7, 128.9, 130.8, 135.5 and 137.9 (aromatic), 155.9 ($(CH_3)_3C(O)\underline{C}$=O), 165.7, 165.7, 168.5 and169.4 (C=O).; MS (FAB) m/z (relative intensity) 1014 (M^++Cs, 22), 882 (M^++H, 50); HRMS (FAB) m/z 1014.3629 ($C_{52}H_{63}N_5O_6Cs$ requires 1014.3602).

N-Trimethylsilylmethyl Peptides 15 and 16. Solutions of the blocked peptides **H** (3.67 g, 5.0 mmol) and **I** (4.42 g, 5.0 mmol) in CH_2Cl_2 (30 mL) containing 5 mL of 98% TFA was stirred for 4 h at 25 ^{0}C. poured into aq K_2CO_3 and extracted with CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, dried and concentrated *in vacuo* to afford 3.04 g (96%) of **15** and 3.64 g (91%) of **16.**

15 (13.6:7.8:7.3:4.8:4.6:1 mixture of 6 rotamers). ^{1}H-NMR($CDCl_3$) -0.03, 0.0, 0.04, 0.07, 0.08 and 0.09 (s, 9H, $Si(CH_3)_3$ 6 peaks for 6 rotamer), 2.71, 2.90, 2.91, 2.92 and 2.98 (s, 2H, $C\underline{H}_2TMS$), 3.30, 3.34, 3.40, 3.54, 3.55, 3.56, 3.64, 3.67, 3.78, 3.83, 3.91, 3.92, 3.99, 4.04, 4.11, 4.16, 4.22, 4.29, 4.33, 4.42, 4.48, 4.57, 4.61 and 4.66 (s, 2H, $C\underline{H}_2$), 4.28 (1H, NH), 7.01-7.32 (m, 20H, aromatic); ^{13}C-NMR($CDCl_3$) -1.4 and -1.3 ($Si(CH_3)_3$), 38.7, 39.2, 39.9, 46.0, 47.3, 49.2, 49.8, 50.5, 51.4, 52.3, 52.8 and 53.3 (CH_2), 125.9, 126.4, 127.1, 127.5, 127.7, 128.2, 128.3, 128.5, 129.1, 134.9, 135.6, 136.0, 136.4 and 139.6 (aromatic), 163.3, 166.3, 167.0, 168.6, 169.5 and 172.1 (C=O); MS (FAB) m/z (relative intensity) 635 (M^++1,

20), 582 (15), HRMS (FAB) m/z 635.3391 ($C_{38}H_{47}N_4O_3Si$ requires 635.3417).

16 (4.3:4.3:4.2:2.7:1.9:1.5:1.4:1.4:1 1 mixture of 9 rotamers) ^{1}H-NMR ($CDCl_3$) -0.06, -0.05, -0.03, 0.01, 0.04, 0.04, 0.05, 0.08 and 0.09 (s, 9H, $Si(CH_3)_3$, 9 peakes for 9 rotamers), 2.66 and 2.87 (s, 2H, $C\underline{H}_2TMS$), 3.44, 3.56, 3.69, 3.79, 3.86, 3.91, 4.04, 4.07, 4.10, 4.11, 4.14, 4.29, 4.41, 4.46, 4.55 and 4.58 (s, 2H, $C\underline{H}_2Ph$), 4.28 (s, 1H, NH), 6.80-7.35 (m, 25H, aromatic); ^{13}C-NMR($CDCl_3$) -2.7,-1.7, and -1.4 ($Si(CH_3)_3$), 36.5, 37.8 and 39.5 ($C\underline{H}_2TMS$), 46.7, 47.2, 50.0, 50.3, 51.1, 51.2, 52.1, 52.5, 53.0 and 54.8 (CH_2), 125.5, 125.7, 126.3, 126.9, 127.2, 127.6, 128.2, 128.4, 128.8, 128.9, 129.9, 135.5, 136.1, 136.6, 137.3 and 139.2 (aromatic), 163.1, 165.5, 166.1, 166.7, 168.2 and 171.7 (C=O); MS (FAB) m/z (relative intensity) 914 (M^++Cs, 5), 781 (15), HRMS (FAB) m/z 914.3116 ($C_{47}H_{55}N_5O_4SiCs$ requires 914.3078).

N-Benzyl-(t-Boc)-L-alanine (J). To a solution of 1.79 g (10.0 mmol) N-benzyl-L-alanine (*J. Am. Chem. Soc.*, **1989**, *111*, 6301) in 35 mL of 1:1 H_2O/dioxane was added triethylamine (3.04 g, 30.0 mmol) and stirred for 20 min at 25 °C. To this solution was added di-tert-butylcarbonate (2.18 g, 10.0 mmol). The resulting solution was stirred for 4 h at 25 °C and concentrated in vacuo. The residue was treated with ice-cold aqueous HCl (ph~2) to ph~4 and extracted with ethyl acetate. The extract was dried over $MgSO_4$ and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, hexane:ethyl acetate = 1:1), yielding 1.98 g (71%) of **J**. ^{1}H NMR (mixture of 2 rotamers, 1.1:1) 1.32 (3H, $CHC\underline{H}_3$) 1.40 (s, 9H, $(C\underline{H}_3)_3C$), 4.33 (q, 1H, $NC\underline{H}CO$), 4.59 (m, 2H, $NC\underline{H}_2Ph$), 7.24 (m, 2H, aromatic), 7.30 (m, 3H, aromatic); ^{13}C NMR 15.5 ($CH\underline{C}H_3$), 28.2 ($(\underline{C}H_3)_3CO$), 50.1 ($N\underline{C}H_2Ph$), 55.1 ($N\underline{C}HCO$), 81.2 ($(CH_3)_3\underline{C}O$), 126.9, 127.3, 127.9, 128.4 and 138.7 (aromatic), 155.3 ($N\underline{C}O$), 178.0 ($\underline{C}O_2H$).

TMS-Terminated N-Benzyl-L-alanine amide (50). To a solution of N-benzyl-(t-Boc)-L-alanine **J** (1.50 g, 5.38 mmol), 1-hydroxy-benzotriazole (0.714 g, 5-38 mmol) and N-(trimethylsilylmethyl)benzylamine (1.04 g, 5.38 mmol) in 35 ml of CH_2Cl_2 at ice-salt bath was added a solution of 1,3-dicyclohexylcarbodiimide (1.43 g, 5.38 mmol) in 15 ml of CH_2Cl_2. After stirred and warmed up to room temperature for 6 h, the mixture was filtered, washed with CH_2Cl_2 and concentrated in vacuo. The residue was added 45 ml of ethyl acetate, washed with sat. Na_2CO_3, aqueous HCl and brine, dried over $MgSO_4$ and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, hexane:ethyl acetate = 2:1), yielding 1.78 g (72%) of TMS-terminated N-benzyl-(t-Boc)-L-alanine amide **K**.

To a solution of N-benzyl-(t-Boc)-L-alanine amide **K** (1.72 g, 3.77 mmol) in 15 ml of CH_2Cl_2 was added 4 ml of trifluoroacetic acid and stirred for 4 h at 25 °C. To the mixture was added sat. K_2CO_3 up to neutral and extracted with CH_2Cl_2. The extract was washed with water, dried over $MgSO_4$ and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, ethyl acetate:methanol = 9:1), yielding 1.28 g (96%) of **50**. ^{1}H NMR 0.07 (s, 9H, $(C\underline{H}_3)_3Si$), 1.22 (d, 3H, $CHC\underline{H}_3$), 2.68 and 3.20 (ABq, J=15 Hz, 2H, $C\underline{H}_2TMS$), 3.40 and 3.77 (ABq, J=12.5 Hz, 2H, $PhC\underline{H}_2NH$), 3.57 (q, 1H, $NC\underline{H}CO$), 4.30 and 4.57 (ABq, J=16.5 Hz, 2H, $PhC\underline{H}_2NCO$), 7.10 (m, 2H, aromatic), 7.19 (m, 1H, aromatic), 7.24-7.33 (m, 7H, aromatic); ^{13}C NMR -1.0 ($(\underline{C}H_3)_3Si$), 19.7 ($CH\underline{C}H_3$), 40.6 ($\underline{C}H_2TMS$), 52.9 ($Ph\underline{C}H_2NH$), 54.0 ($N\underline{C}HCO$), 54.1 ($Ph\underline{C}H_2NCO$), 128.0, 128.5, 129.0, 129.6, 129.7, 130.1, 138.2 and 140.3 (aromatic), 176.1 (C=O); IR 3315, 3060, 3027, 2950,

2897, 2848, 1634, 1491, 1450, 1434, 1397, 1246, 854, 731, 698; MS(FAB) 355 (M+H, 72), 194 (17), 134 (83), 91 (100); HRMS calcd for $C_{21}H_{31}N_2OSi$ (M+H) 355.2206, found 355.2194.

TMS-Terminated N-Benzyl-L-alanine amide (51). To a solution of N-benzyl-(t-Boc)-L-alanine **J** (279 mg, 1.00 mmol) in 6 ml of CH_2Cl_2 was added EEDQ (371 mg, 1.50 mmol) and stirred for 30 min at 25 °C. To the mixture was added N-benzyl-L-alanine amide **50** (354 mg, 1.00 mmol) in 3 ml of CH_2Cl_2 and stirred for 4 h at 25 °C. The resulting mixture was poured into 2N aqueous HCl and extracted with CH_2Cl_2. The extract was washed with water, dried over $MgSO_4$ and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, hexane:ethyl acetate = 2:1), yielding 486 mg (79%) of TMS-terminated N-benzyl-(t-Boc)-L-alanine amide **L**.

To a solution of N-benzyl-(t-Boc)-L-alanine amide **L** (486 mg, 0.79 mmol) in 5 ml of CH_2Cl_2 was added 2 ml of trifluoroacetic acid and stirred for 4 h at 25 °C. To the mixture was added sat. K_2CO_3 up to neutral and extracted with CH_2Cl_2. The extract was washed with water, dried over $MgSO_4$ and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, ethyl acetate:methanol = 9:1), yielding 396 mg (97%) of **51**. ^{1}H NMR 0.03 (s, 9H, $(C\underline{H}_3)_3Si$), 1.16 (d, 3H, $CHC\underline{H}_3$), 1.17 (d,3H, $CHC\underline{H}_3$), 2.05 (s, 1H, NH), 2.51 and 3.30 (ABq, J=15 Hz, 2H, $C\underline{H}_2TMS$), 3.26 and 3.67 (ABq, J=12.5 Hz, 2H, $PhC\underline{H}_2NH$), 4.17 (q, 1H, $NC\underline{H}CO$), 4.37 and 5.24 (ABq, J=16.5 Hz, 2H, $PhC\underline{H}_2NCO$), 4.58 and 5.05 (ABq, J=18.5 Hz, 2H, $PhC\underline{H}_2NCO$), 5.78 (q, 1H, $NC\underline{H}CO$), 7.16-7.37 (m, 15H, aromatic); ^{13}C NMR -1.3 ($(\underline{C}H_3)_3Si$), 16.1 ($CH\underline{C}H_3$), 19.6 ($CH\underline{C}H_3$), 38.9 ($\underline{C}H_2TMS$), 46.8 ($PhCH_2NH$), 48.0 ($N\underline{C}HCO$), 52.0 ($PhCH_2NCO$), 53.1 ($PhCH_2NCO$), 54.3 ($N\underline{C}HCO$), 125.6, 125.8, 126.6, 126.8, 127.0, 127.5, 127.8, 128.0, 128.2, 128.4, 128.5, 128.6, 128.8, 137.1, 138.9 and 140.0 (aromatic), 171.6 (C=O), 177.1 (C=O); IR 3310, 3084, 3063, 3026, 2979, 2897, 2833, 1736, 1631, 1444, 1415, 1188, 1164, 855, 727, 698; MS(FAB) 516 (M+H, 24), 355 (36), 190 (35), 134 (90), 91 (100); HRMS calcd for $C_{31}H_{42}N_3O_2Si$ (M+H) 516.3047, found 516.3067.

TMS-Terminated N-Benzyl-L-alanine amide (52). To a solution of N-benzyl-(t-Boc)-L-alanine **J** (130 mg, 0.46 mmol) in 5 ml of CH_2Cl_2 was added EEDQ (175 mg, 0.70 mmol) and stirred for 2 h at 25 °C. To the mixture was added N-benzyl-L-alanine amide **51** (239 mg, 0.46 mmol) in 2 ml of CH_2Cl_2 and stirred for 24 h at 25 °C. The resulting mixture was poured into 2N aqueous HCl and extracted with CH_2Cl_2. The extract was washed with water, dried over $MgSO_4$ and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, hexane:ethyl acetate = 2:1), yielding 159 mg (44%) of crude TMS-terminated N-benzyl-(t-Boc)-L-alanine amide **M**.

To a solution of N-benzyl-(t-Boc)-L-alanine amide **M** (155 mg, 0.20 mmol) in 2 ml of CH_2Cl_2 was added 0.8 ml of trifluoroacetic acid and stirred for 6 h at 25 °C. To the mixture was added sat. K_2CO_3 up to neutral and extracted with CH_2Cl_2. The extract was washed with water, dried over $MgSO_4$ and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, ethyl acetate), yielding 107 mg (79%) of **52**. ^{1}H NMR-0.10 (s, 9H, $(C\underline{H}_3)_3Si$), 1.04 (d, 3H, $CHC\underline{H}_3$), 1.17 (d, 3H, $CHC\underline{H}_3$), 1.19 (d,3H, $CHC\underline{H}_3$), 2.52 and 2.87 (ABq, J=15 Hz, 2H, $C\underline{H}_2TMS$), 3.26 and 3.63 (ABq, J=12.5 Hz, 2H, $PhC\underline{H}_2NH$), 3.36 (q, 1H, $NC\underline{H}CO$), 4.22 and 4.87 (ABq, J=16 Hz, 2H, $PhC\underline{H}_2NCO$), 4.60 and 5.11

(ABq, J=18 Hz, 2H, PhC$\underline{H}_2$NCO), 4.85 and 5.02 (ABq, J=16 Hz, 2H, PhC$\underline{H}_2$NCO), 5.49 (q, 1H, NC$\underline{H}$CO), 5.69 (q, 1H, NC$\underline{H}$CO), 7.10-7.33 (m, 20H, aromatic); ^{13}C NMR -1.2 (($\underline{C}H_3)_3$Si), 15.3 (CH$\underline{C}H_3$), 16.4 (CH$\underline{C}H_3$), 19.3 (CH$\underline{C}H_3$), 39.1 ($\underline{C}H_2$TMS), 46.9 (Ph$\underline{C}H_2$NH), 48.5 (N$\underline{C}$HCO), 49.8 (N$\underline{C}$HCO), 51.8 (Ph$\underline{C}H_2$NCO), 53.4 (Ph$\underline{C}H_2$NCO), 54.1 (N$\underline{C}$HCO), 125.5, 126.4, 126.6, 126.9, 127.1, 127.4, 127.6, 128.0, 128.2, 128.6, 128.7, 128.8, 136.8, 138.6, 138.9 and 139.8 (aromatic), 170.4 (C=O), 173.9 (C=O), 177.3 (C=O); IR 3317, 3072, 3031, 2979, 2903, 1637, 1450, 1415, 1247, 1178, 996, 855, 733, 703. MS(FAB) 677 (M+H, 35), 481 (32); HRMS calcd for $C_{41}H_{53}N_4O_3Si$ (M+H) 377.3887, found 677.3901.

TMS Terminated N-Phthalimidoglycinamides 17 and 18. To independent solutions of 5 mmol of N-benzyl-N-trimethylsilylmethylamine (**9**) and N-trimethylsilylmethylamine (**10**) and triethylamine in 20 mL of CH_2Cl_2 were added solutions of acid chloride **8** (1.1 g, 5.0 mmol) in 20 mL of 1.4-dioxane. The mixtures were stirred for 3 h at 25 ^{0}C and extracted with CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, dried and concentrated *in vacuo* to afford residues which were subjected to column chromatography (silica gel, 2:1 ethyl acetate-hexane) yielding the respective glycinamide **18** (1.7 g, 91%) or **17** (0.5 g, 60%).

17: mp 127-128^{0}C (chloroform-hexane) ^{1}H-NMR ($CDCl_3$) 0.00 (s, 9H), 2.72 (d, 2H, J=5.8Hz), 4.30 (s, 2H), 5.78 (brs, 1H), 7.24-7.88 (m, 4H); ^{13}C-NMR ($CDCl_3$) -2.7, 30.1, 40.9, 123.6, 131.9, 134.2, 166.0 and 167.8; MS (FAB) m/z (relative intensity) 291(M^++1, 100), 275(38), 153(36); HRMS (FAB) m/z 291.1153 ($C_{14}H_{14}N_2O_3Si$ requires 291.1165).

18: (2.1:1 mixture of two rotamers) ^{1}H-NMR ($CDCl_3$) of rotamer A 0.03 (s, 9H), 2.90 (s, 2H), 4.52 (s, 2H), 5.83 (s, 2H), 7.27-7.86 (m, 9H), rotamer B 0.22 (s, 9H), 2.83 (s, 2H), 4.50 (s, 2H), 4.58 (s, 2H), 7.27-7.86 (m, 9H); ^{13}C-NMR ($CDCl_3$) of rotamer A -1.52, 38.9, 39.1, 52.5, 123.6, 126.4, 127.8, 129.0, 132.2, 133.8, 135.5, 165.0 and 168.2, rotamer B -1.4, 37.2, 39.5, 50.6, 127.4, 127.8, 128.2, 128.3, 132.2, 133.8, 136.4, 164.9 and 167.9; MS (FAB) m/z (relative intensity) 381(M^++1, 100), 365(18), 289(3), 192(5), 168(18), 91(68); HRMS(FAB) m/z 381.1649 ($C_{21}H_{25}N_2O_3Si$ requires 381.1634).

TMS Terminated N-Phthalimidopeptides 19-24. To solutions of the appropriate silylamide (**11**, 0.9 g; **12**, 0.7 g, **13**, 1.2 g; **14**, 0.8 g; **15**, 1.6 g; **16**, 2.0 g) (see Supporting Information) and triethylamine (1.3 mL, 7.5 mmol) in 30 mL of CH_2Cl_2 were added a solution of acid chloride **8** (0.56g, 2.5mmol) in 20mL of 1,4-dioxane. The solutions were stirred for 3 h at 25 ^{0}C and, diluted with water and extracted with CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, dried and concentrated *in vacuo* to afford residues which were subjected to column chromatography (silica gel, 2:1 ethyl acetate-hexane) yielding the respective phthalimidopeptides **19** (1.1 g, 88%), **20** (1.0 g, 85%), **21** (1.5 g, 86%), **22** (1.1 g, 82%), **23** (1.7 g, 83%), **24** (1.9 g, 81%).

19: (4:1.1:1 mixture of three rotamers): mp 136-138^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) of rotamer A -0.01 (s, 9H), 2.86 (s, 2H), 4.17 (s, 2H), 4.43 (s, 2H), 4.61 (s, 2H), 4.81 (s, 2H), 7.04-7.86 (m, 14), rotamer B -0.02 (s, 9H), 2.86 (s, 2H), 4.03 (s, 2H), 4.36 (s, 2H), 4.66 (s, 2H), 4.84 (s, 2H), 7.04-7.86 (m, 14H), rotamer C 0.1 (s, 9H), 3.04 (s, 2H), 4.15 (s, 2H), 4.37 (s, 2H), 4.57 (s, 2H), 4.80 (s, 2H), 7.04-7.86 (m, 14H); ^{13}C-NMR ($CDCl_3$) -1.6, -1.5 and -1.3, 38.6, 39.1 and 39.2, 46.2, 46.3 and 47.1, 50.5, 51.3, 52.9, 123.5, 123.6, 126.4, 126.9, 127.6, 128.0, 128.7, 128.9, 129.1, 132.1, 132.3, 133.9, 134.2, 135.3, 136.0, 166.0, 166.7, 167.0, 167.8, 170.3 and 176.5; MS (FAB) m/z (relative intensity) 512 (M^+-CH_3, 7), 335 (26), 307(2), 335 , 160(28), 91(100); HRMS (FAB) m/z 528.2321

($C_{30}H_{34}N_3O_4Si$ requires 528.2319).

20: (2.4:1 mixture of two rotamers): ^{1}H-NMR ($CDCl_3$) of rotamer A 0.00 (s, 9H), 2.87 (s, 2H), 3.17 (s, 3H), 4.19 (s, 2H), 4.47 (s, 2H,), 4.50 (s, 2H), 7.10-7.84 (m, 9H), rotamer B 0.07 (s, 9H), 2.74 (s, 2H), 3.22 (s, 3H), 4.22 (s, 2H), 4.47 (s, 2H), 4.56 (s, 2H), 7.10-7.84 (m, 9H); ^{13}C-NMR ($CDCl_3$) of rotamer A -1.4, 35.7, 39.0, 49.1, 50.3, 52.7, 123.4, 126.3, 127.6, 128.9, 132.1, 133.9, 136.2, 166.0 166.7 and 167.7, rotamer B -1.5, 35.5, 37.4, 49.4, 50.5, 52.5, 125.8, 127.3, 127.7, 128.0, 128.5, 129.2, 136.5, 165.8, 166.1 and 167.0; MS (FAB) m/z (relative intensity) 452(M^++1, 70), 436(25), 360(2), 259(75), 160(83), 91(100); HRMS (FAB) m/z 452.2008 ($C_{24}H_{30}N_3O_4Si$ requires 452.2006).

21: (5.6:2.9:2.0:1.8:1.1:1 mixture of 6 rotamers): ^{1}H-NMR ($CDCl_3$) -0.07, -0.02, 0.02, 0.03, 0.04 and 0.11 (s, 9H), 2.66, 2.82, 2.86 and 2.92 (s, 2H), 3.60, 3.68, 3.83, 3.91, 3.96, 4.03, 4.11, 4.13, 4.18, 4.23, 4.26, 4.32, 4.35, 4.38, 4.43, 4.47, 4.53, 4.57, 4.60, 4.63, 4.64, 4.76 and 4.83 (s, 2H), 7.08-7.87 (m, 19H); ^{13}C-NMR ($CDCl_3$) -1.6, -1.5, -1.4 and -1.3, 37.3, 38.3, 38.8, 39.1, 39.6, 40.0, 45.8, 46.1, 46.2, 46.4, 46.7, 46.8, 47.2, 47.5, 48.0, 50.0, 50.3, 50.5, 51.2, 52.1, 52.7, 123.4, 126.0, 126.5, 126.8, 127.2, 127.4, 127.6, 127.7, 127.9, 128.6, 128.9, 129.0, 129.1, 132.2, 133.9, 135.3, 135.4, 135.6, 136.1, 136.3, 166.2, 166.3, 166.5, 166.7, 166.9, 167.0, 167.1, 167.5, 167.8, 168.1, 168.2, 169.2 and 169.3; MS (FAB) m/z (relative intensity) 675 (M^++1, 2), 659 (2), 482(5), 335(22), 159(35), 91(100); HRMS (FAB) m/z 675.2997 ($C_{39}H_{43}N_4O_5Si$ requires 675.3003).

22: (22.8:7.9:7.3:6.7:24:2.3:1.7:1 mixture of 8 rotamers) ^{1}H-NMR ($CDCl_3$) 0.01, 0.02, 0.04, 0.07, 0.09, 0.11, 0.12 and 0.15 (s, 9H), 2.72, 2.79 and 2.88 (s, 2H), 2.90, 2.94, 2.95, 3.00, 3.07, 3.10, 3.13, 3.14, 3.21 and 3.23 (s, 3H), 4.04, 4.10, 4.14, 4.17, 4.19, 4.20, 4.22, 4.28, 4.33, 4.35, 4.43, 4.45, 4.53, 4.56 4.57, 4.60, and 4.61 (s, 2H), 7.14-7.84 (m, 9H); ^{13}C-NMR ($CDCl_3$); -1.5, -1.4. -1.3, 35.2, 35.3, 35.4, 35.5, 35.7, 35.8, 37.2, 38.6, 38.8, 38.9, 39.5, 48.9, 49.1, 49.3 and 49.5, 50.2, 50.4, 50.6, 51.1, 52.3, 52.6, 53.4, 123.2, 125.8, 126.0, 126.2, 127.2, 127.5, 127.6, 127.7, 128.4, 128.8, 128.9, 129.1, 132.0, 133.7, 133.8, 135.7, 136.1, 136.5, 166.0, 166.3, 166.9 and 167.6; MS (FAB) m/z (relative intensity) 523 (M^++1, 50), 507 (10), 433(1), 330(30), 265(100), 160(71); HRMS (FAB) m/z 523.2386 ($C_{27}H_{35}N_4O_5Si$ requires 523.2377).

23: (1.6:1.4:1.4:1.3:1 mixture of 5 rotamers): ^{1}H-NMR ($CDCl_3$) -0.07, -0.02, 0.02, 0.03 and 0.04 (s, 9H), 2.66, 2.82, 2.86, 2.92 and 2.93 (s, 2H), 3.60, 3.68, 3.83, 3.91, 3.96, 4.03, 4.11, 4.12, 4.22, 4.26, 4.33, 4.35, 4.38, 4.43, 4.47, 4.53, 4.57, 4.60, 4.63, 4.64, 4.76 and 4.83 (s, 2H), 6.85-7.83 (m, 24H), ^{13}C-NMR ($CDCl_3$) -1.7, -1.6, -1.5, -1.4, -1.3, 37.3, 38.3, 38.8, 39.1, 39.6, 40.0, 45.8, 46.1, 46.2, 46.4, 46.7, 46.8, 47.2, 47.5, 48.0, 50.0, 50.3, 50.5, 51.2, 52.1, 52.6 (CH_2), 123.3, 125.5, 125.8, 126.2, 126.4, 126.6, 126.7, 126.8, 127.0, 127.1, 127.3, 127.4, 127.7, 127.8, 127.9, 128.1, 128.5,128.6, 128.8, 129.0, 132.1, 132.3, 133.5, 133.7, 133.8, 134.1, 135.1, 135.3, 135.4, 135.5, 135.6, 135.9, 136.0, 136.2, 136.3, 136.5, 136.6, 165.7, 166.1, 166.3, 166.5, 166.6, 166.7, 166.8, 167.1, 167.2, 167.7, 167.8, 168.0, 168.2, 168.3, 168.5, 168.9, 169.2, 169.4 and 169.6); MS (FAB) m/z (relative intensity) 822 (M^++1, 2), 806 (2), 482 (25), 335 (19), 159(35); HRMS (FAB) m/z 954.2706 ($C_{48}H_{51}N_5O_6SiCs$ requires 954.2663).

24: (6.1:3.1:3.1:2.7:1 mixtures of 5 rotamers): ^{1}H-NMR ($CDCl_3$) -0.06, -0.01, 0.00, 0.35, 0.59 (s, 9H), 2.60, 2.62, 2.82, 2.86 and 2.96 (s, 2H), 3.58, 3.70, 3.76, 3.83, 3.89, 3.90, 3.99, 4.09, 4.11, 4.14, 4.25, 4.30, 4.33, 4.41, 4.52, 4.56, 4.60, 4.66, 4.70, 4.75 and 4.83 (s, 2H), 6.88-7.93 (m. 29H); ^{13}C-NMR ($CDCl_3$) -1.7, -1.6, -1.4, 37.3, 38.8, 39.1, 39.6, 40.2,

45.8, 46.3, 46.7; 47.0, 47.3, 47.7, 48.5, 50.2, 50.3, 50.5, 51.1, 52.2, 52.6 (CH_2), 123.2, 125.5, 125.7,125.8, 126.4, 126.5, 126.7, 126.8, 127.0, 127.3, 127.4, 127.7, 127.8, 128.5, 128.8, 128.9, 132.1, 133.5, 133.8, 135.2, 135.4, 135.5, 136.0, 136.2, 136.3, 136.6, 136.7, 165.6, 166.1, 166.4, 166.5, 167.2, 167.6, 167.9, 168.0 168.3, 168.8, 169.1 and 169.0; MS (FAB) m/z (relative intensity) 1101 (M^++Cs, 22); HRMS (FAB) m/z 1101.3307 ($C_{57}H_{60}N_6O_7Cs$ requires 1101.3347).

Carboxylic Acid Terminated N-Phthalimidopeptides 25-30. A solution of 10 mmol of the appropriate amino acid or peptide (glycine, 0.8 g; glycine-glycine, 1.3 g; glycine-glycine-glycine, 1.8 g; sarcosine, 0.9 g, N-benzylglycine, 1.7 g; proline, 1.2 g)) in 75 mL of water containing magnesium oxide (0.6 g, 15 mmol) was stirred for 4 h at 25 ^{0}C. To this solution at 0 ^{0}C was added The phthalimidoglycine acid chloride **8** (2.2 g, 10 mmol) in 25 mL of 1.4-dioxane. The resulting solution was stirred for 2 h at 25 ^{0}C, diluted with aq. HCl and concentrated *in vacuo* to yield the corresponding N-phthalimidopeptide (2.4 g (91%) of **25**,[19] 2.7 g (87%) of **28**, 3.2 g (85%) of **29**, 2.8 g (95%) of **26**,[20] 1.7 g (95%) of **27** and 2.3 g (77%) of **30**, respectively.

27: (1.3:1 of two rotamers); mp 140-143 ^{0}C (CH_3OH); ^{1}H-NMR (DMSO) of rotamer A 3.88 (s, 2H), 4.03 (s, 2H), 4.53 (s, 2H), 7.20-7.39 (m, 5H), 7.83-7.89 (m, 4H), rotamer B 4.11 (s, 2H), 4.46 (s, 2), 4.74 (s, 2H), 7.20-7.39 (m, 5H), 7.83-7.89 (m, 4H); ^{13}C-NMR (DMSO) rotamer A 38.3, 47.0, 48.9, 122.2, 125.9, 126.8, 127.4, 127.7, 130.6, 133.7, 135.9, 165.7, 166.5 and 169.2, rotamer B 38.3, 47.5, 49.6, 122.3, 126.2, 126.5, 127.5, 130.5, 133.6, 135.5, 165.5, 166.5 and 169.7; MS (FAB) m/z (relative intensity) 353(M^++1, 33), 331(9), 165(5), 160(36), 91(100); HRMS(FAB) m/z 353.1131 ($C_{19}H_{17}N_2O_5$ requires 353.1137).

28: mp 238-241^{0}C (CH_3OH); ^{1}H-NMR (DMSO) 3.77 (d, 2H, J=6.0Hz), 3.78 (d, 2H, J=5.5Hz), 4.30 (s, 2H), 7.85-7.89 (m, 4H), 8.23 (t, 1H, J=6.0Hz), 8.57 (t, 1H, J=5.5Hz); ^{13}C-NMR (DMSO) 40.7, 41.3, 42.5, 123.9, 132.3, 135.3, 168.3 169.7 167.8, and 171.8; MS (FAB) m/z (relative intensity) 320 (M^++1, 55), 245 (18), 188(6), 160 (27), 119 (100); HRMS (FAB) m/z 320.0901 ($C_{14}H_{14}O_6N_3$ requires 320.0883).

29: mp 239-241^{0}C (CH_3OH); ^{1}H-NMR (DMSO) 3.70(s, 2H), 3.73(s,2H), 3.77 (s, 2H), 4.25 (s, 2H), 7.83-7.90 (m, 4H), 8.15 (t, 1H, J=5.5Hz), 8.21 (t, 1H, J=5.0Hz), 8.52 (t, 1H, J=5.0Hz), ^{13}C-NMR (DMSO) 40.7, 41.3, 42.1, 42.8, 123.9, 132.3, 135.3, 167.3, 168.2, 169.6, 169.9 and 171.8. MS (FAB) m/z (relative intensity) 377 (M^++1,12), 359 (5), 302 (10), 245 (11), 160 (25), 91 (100); HRMS (FAB) m/z 377.0888 ($C_{16}H_{17}O_7N_4$ requires 377.0971).

30: mp 198-200^{0}C (CH_3OH); ^{1}H-NMR (DMSO) 1.67-2.18 (m, 4H), 3.10-3.13 (m, 1H), 3.32-3.35 (m, 1H), 4.08-4.21 (m, 2H), 4.41-4.51 (m, 2H), 7.77-7.91 (m, 4H); ^{13}C-NMR (two rotamers, DMSO) 27.0 and 29.5, 33.8 and 35.9, 50.9 and 51.6, 53.7, 63.3 and 64.0, 128.4, 136.7, 139.8, 169.3, 169.7, 172.6, 178.2 and 178.4; MS (FAB) m/z (relative intensity) 303 (M^++1, 90), 160 (75); HRMS (FAB) m/z 303.0967 ($C_{15}H_{15}O_5N_2$ requires 303.0981).

α-Bromo-N-(trimethylsilylmethyl)-N-benzylacetamide (31). To a solution of α-bromoacetyl chloride (0.84 mL, 10.0 mmol) in 50 mL of CH_3CN was added N-(trimethylsilylmethyl)benzylamine (1.34 mL, 10.0 mmol) at 0 ^{0}C and stirred for 2 h at 25 ^{0}C. Concentration gave a residue, which was subjected to column chromatography (silica gel, ethyl 1:10 acetate-hexane) yielding 2.5 g (80%) of **31** (4:1 mixture of two rotamers). ^{1}H-NMR ($CDCl_3$) of rotamer A 0.06 (s, 9H), 2.87 (s, 2H), 3.78 (s, 2H), 4.55 (s, 2H), 7.12-7.34 (m, 5H); rotamer B 0.10 (s, 9H), 2.83 (s, 2H), 3.83 (s, 2H), 4.55 (s, 2H), 7.12-7.34 (m, 5H); ^{13}C-NMR ($CDCl_3$) rotomer A -1.46, 26.0, 38.8, 53.9, 126.1, 127.7, 128.8, 135.8, 166.2, rotamer

B -1.66, 26.5, 38.9, 50.0, 127.3, 127.5, 128.5, 136.2, 166.2; MS (FAB) m/z (relative intensity) 314 (M^++1, 28), 299 (12), 278 (2), 234 (15), 91 (100); HRMS (FAB) m/z 314.0568 ($C_{13}H_{21}Br$ requires. 314.0576)

TMS Terminated N-(3-Nitrophthalimido-glycinamide 32. A solution of potassium 4-nitrophthalimide (1.13 g, 5.0 mmol) and bromoacetamide **31** (1.57 g, 5.0 mmol) in 50 mL of DMF and 20 mL of CH_2Cl_2 was stirred for 24 h at 65 ^{0}C and extracted with CH_2Cl_2. The CH_2Cl_2 extracts were washed with water, dried and concentrated *in vacuo* to afford a residue which was subjected to column chromatography (silica gel, 1:1 ethyl acetate-hexane) yielding 2.0 g (93%) of **32** (3:1 mixture of two rotamers). ^{1}H-NMR ($CDCl_3$) 0.00 and 0.20 (s, 9H), 2.80 and 2.88 (s, 2H), 4.53 and 4.54 (s, 2H), 4.55 and 4.56 (s, 2H), 7.14-7.38 (m, 5H), 7.98-8.02 (m, 1H), 8.52-8.59 (m, 2H), ^{13}C-NMR ($CDCl_3$) -1.8 and –1.7, 37.2 and 39.2, 39.5 and 40.0, 50.1 and 53.0, 119.2, 124.3, 126.1, 127.5, 127.7, 128.5, 129.0, 129.1, 129.3, 133.5, 135.4, 136.1, 136.8, 151.5, 163.8, 164.2, 165.4, 165.5, 165.8 and 165.9; MS (FAB) m/z (relative intensity) 426 (M^++1, 30), 410 (43), 394(17), 334(15), 220(7), 91(100); HRMS (FAB) m/z 426.1473 ($C_{21}H_{24}N_3O_5Si$ requires 426.1485).

Photocyclization Reactions. Preparative photochemical reactions of the phthalimide substrates were conducted by using an apparatus consisting of a 450W medium pressure mercury lamp surrounded by a Pyrex glass filter in a quartz immersion well. The irradiated solutions (see Tables 4 and 5 for substrate concentrations, solvents and irradiation times) were maintained at 17 ^{0}C under a N_2 atmosphere. In each case, concentration of the photolysates was followed by column chromatography (silica gel, ethyl acetate-hexane) giving the photoproducts (**33-49**) in the yields listed in Tables 4 and 5.

33: mp 252-254 ^{0}C (CH_3OH); ^{1}H-NMR (CD_3OD) 3.76 (s, 2H), 4.37 and 4.50 (two d, 2H, J=16.5Hz); ^{13}C-NMR (CD_3OD) 40.4, 43.8, 87.6, 121.3, 123.3, 129.7, 132.0, 144.9, 165.9 and 166.3.

35: mp 92-93 ^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) 3.20 and 3.74 (two d, 2H, J=12.5Hz), 3.82 and 4.50 (two d, 2H, J=18.0Hz), 4.55 and 4.75 (two d, 2H, J=14.5Hz), 4.99 (s, 1H), 7.24-7.35 (m, 5H), 7.46-7.64 (m, 4H); ^{13}C-NMR ($CDCl_3$) 41.0, 50.6, 54.3, 83.3, 122.0, 123.7, 127.8, 128.0, 128.8, 130.4, 132.8, 135.5, 144.8, 164.0 and 164.8; MS (FAB) m/z (relative intensity) 309 (M^++1, 100), 291(10), 250(3), 188(3), 119(18), 91(36); HRMS (FAB) m/z 309.1254 ($C_{18}H_{17}N_2O_3$ requires 309.1239).

36: mp 270-272 ^{0}C (CH_3OH); ^{1}H-NMR (D_2O) 3.62 (s, 2H), 3.99 and 4.30 (two d, 2H, J=16.5Hz), 4.62 (s, 2H); ^{13}C-NMR (CD_3OD), 42.4, 46.3, 64.1, 79.9, 120.3, 121.0, 127.0, 128.6, 130.2, 142.7, 169.3, 169.8 and 171.5.

37: ^{1}H-NMR (DMSO) 3.16-3.84 (m, 6H), 3.42 (br, 1H), 4.05 and 4.41 (two d, 2H, J=16.8Hz), 6.81 (br, 1H), 6.94 (br, 1H), 7.83-7.91 (m, 4H), 8.34 (br, 1H); ^{13}C-NMR (DMSO) 40.5, 42.6, 44.0, 46.1, 87.3, 121.5, 121.8, 128.9, 129.2, 131.7, 145.7, 166.3, 168.7, 169.5 and 170.0.

38: (1:1 mixture of two rotamers) ^{1}H-NMR (D_2O) 1.21-3.69 (m, 7H), 3.98 and 4.69 (two d, 2H, J=7.2Hz), 4.06 and 4.58 (two, d, 2H, J=6.7Hz), 4.80 (s, 1H), 7.68-7.88 (m, 4H); ^{13}C-NMR (D_2O) 21.5, 21.8, 26.8, 29.4, 40.8, 41.0, 44.5, 46.5, 49.1, 64.1, 65.0, 85.7, 88.1, 123.2, 123.8, 123.9, 124.3, 130.4, 130.8, 133.8, 134.1, 144.0, 144.6, 165.8, 167.2, 169.3 and 169.6; MS (FAB) m/z (relative intensity) 259 (M^++1, 100), 241(28), 160(3); HRMS (FAB) m/z 259.10890($C_{14}H_{15}N_2O_3$ requires 259.10890).

39: mp 143-145 ^{0}C (chloroform-hexane); ^{1}H-NMR (DMSO) 2.54 and 4.42 (two d,

2H, J=5.8Hz), 3.22 and 4.82 (two d, 2H, J=6.2Hz), 3.68 and 4.78 (two d, 2H, J=6.2Hz), 3.80 and 4.42 (two d, 2H, J=6.3Hz), 4.31 and 5.15 (two d, 2H, J=6.0Hz), 4.79 (s, 1H), 6.96(d, 2H, J=2.9Hz), 7.04(s, 1H), 7.20-7.34 (m, 7H), 7.62-7.83 (m, 4H); ^{13}C-NMR(DMSO), 44.6, 48.7, 50.2, 90.0, 122.8, 123.2, 127.1, 127.9, 128.4, 130.3, 130.7, 137.2, 137.2, 147.3, 168.8 169.7 and 170.9; MS (FAB) m/z (relative intensity) 456 (M^++1, 18), 438(3), 366(2), 93(100); HRMS (FAB) 456.1944 ($C_{27}H_{36}N_3O_4$ requires 456.1923).

40: mp 146-148 ^{0}C (chloroform-hexane); MS (FAB) m/z (relative intensity) 380 (M^++1, 61), 361(32), 337(15), 291(22), 160(18), 118(11), 91(100); HRMS (FAB) ($C_{21}H_{22}N_3O_4$ requires 380.1736).

41a: mp 155-158 ^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) 3.04 and 3.13 (two d, 2H, J=17.6Hz), 3.12 and 4.05 (two d, 2H, J= 16.5 Hz), 3.37 and 3.45 (two d, 2H, J=15.7 Hz), 3.68 and 5.55 (two d, 2H, J=14.3Hz), 3. 98 and 4.75 (two d, 2H, J=18.0Hz), 4.37 (d, 2H, J=17.6Hz), 4.76 (s, 1H), 5.20 and 5.28 (two d, 2H, J=20.0Hz), 6.40 (d, 2H, J=5.4Hz), 6.96-7.10 (m, 3H), 7.13-7.25 (m, 10H), 7.33-7.63 (m, 4H); ^{13}C-NMR ($CDCl_3$) 38.6, 90.1, 46.3, 47.9, 48.6, 48.7, 49.8, 50.6, 122.1, 123.6, 125.9, 126.6, 127.7, 128.6, 128.7, 128.9, 129.1, 129.3, 129.4, 133.1, 134.5, 135.5, 136.0, 147.2, 166.1, 167.0 169.1 and 169.2; MS (FAB) m/z(relative intensity) 603 (M^++1, 19), 585(20), 495(1), 310(10), 154(75), 119(100); HRMS (FAB) m/z 603.2626 ($C_{36}H_{35}N_4O_5$ requires 603.2607).

41b: mp 163-166 ^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) 3.32 and 4.53 (two d, 2H, J=17.6Hz), 3.39 and 4.17 (two d, 2H, J=7.8Hz), 3.46 and 3.53 (two d, 2H, J=5.4Hz), 3.72 and 5.42 (two d 2H, J=14.2Hz), 4.10 and 4.15 (two d, 2H, J=15.0Hz), 4.45 (s, 1H), 4.74 and 4.72 (two d, 2H, J=16.8Hz), 5.20 (s, 1H), 5.23 and 5.59 (two d, 2H, J=14.4Hz), 6.44 (m, 2H, J=7.6Hz), 7.05-7.27 (m, 10H), 7.31-7.40 (m, 3H), 7.40-7.59 (m, 4H); ^{13}C-NMR ($CDCl_3$) 38.8, 47.1, 48.0, 48.6, 49.3, 50.2, 50.6, 93.8, 122.1, 124.3, 126.1, 127.2, 127.2, 127.9, 128.6, 128.8, 128.9, 129.1, 130.4, 131.7, 132.7, 134.8, 135.4, 136.2, 143.5, 166.2 168.6 and 169.3; MS (FAB) m/z (relative intensity) 623 (M^++CH_2Li 100), 585(M^+-OH, 20), 522(5), 291(8), 161(47), 91(100); HRMS (FAB) m/z 585.2491 (M^+-OH, $C_{36}H_{33}N_4O_4$ requires 585.2502).

42: mp 258-260 ^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) 2.85 (s, 3H), 2.83 (s, 3H), 3.15 and 3.35 (two d, 2H, J=14.3Hz), 3.20 and 3.88 (two d, 2H, J=18.0Hz), 3.29 and 4.10 (two d, 2H, J=18.0Hz), 3.33 and 4.48 (two d, 2H, J=17.6Hz), 3.45 and 5.11(two d, 2H, J=15.0Hz), 5.34 (s, 1H), 6.48 (t, 1H, J=3.3Hz), 7.09-7.74 (m, 8H); ^{13}C-NMR($CDCl_3$) 33.7, 36.3, 39.5, 46.2, 48.6, 50.4, 52.0, 122.1, 123.8, 125.9, 127.7, 128.8, 129.1, 129.5, 129.6, 133.1,, 147.5, 165.4, 167.9, 168.8 and 169.2; MS (FAB) m/z (relative intensity) 451 (M^++1, 40), 433 (100), 149 (75); HRMS (FAB) m/z 451.1984 ($C_{24}H_{27}N_4O_5$ requires 451.1981).

43: mp 140-142 ^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) 3.32 and 3.45 (two d, 2H, J=5.4Hz), 3.63 and 3.67 (two d 2H, J=5.8Hz), 3.71 and 3.80 (two d, 2H, J=5.8Hz), 4.02 and 4.27 (two d, 2H, J=7.2Hz), 4.27 and 4.53 (two d, 2H, J=6.0Hz), 4.74 and 5.36 (two d, 2H, J=6.5Hz), 4.77 and 4.81 (two d, 2H, J=5.0Hz), 5.31 and 5.52 (two d, 2H, J=5.8Hz), 5.34 and 5.37 (two d, 2H, J=5.6Hz), 5.27 (s, 1H, OH), 6.98-7.75 (m, 25H); ^{13}C-NMR ($CDCl_3$), 42.0, 47.5, 47.8, 49.1, 49.6, 50.2, 51.4, 52.1, 52.6, 90.8, 121.8, 122.1, 123.4, 123.8, 123.9, 125.8, 127.6, 127.7, 128.8, 129.1, 129.5, 129.6, 129.9, 133.1, 133.9, 134.5, 136.5,147.0, 147.5, 167.5, 167.6, 168.4,169.9 and 172.7; MS (FAB) m/z (relative intensity) 882 (M^++Cs, 1), 732 (10); HRMS (FAB) m/z 882.2242 ($C_{45}H_{43}N_5O_6Cs$ requires 882.2268).

44: mp 126-138^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) 2.90-5.80 (m, 22H), 6.81-7.85 (m, 29H); ^{13}C-NMR ($CDCl_3$) 45.3, 47.0, 48.3, 48.4, 48.9, 49.1, 50.0, 50.4, 51.5,

52.1, 52.6, 89.2, 121.7, 121.9, 123.8, 125.8, 126.8, 127.1, 127.3, 127.6, 127.8, 128.2, 128.7, 128.9, 129.0, 129.1, 129.2, 129.8, 130.6, 131.0, 132.3, 134.9, 135.5, 135.8, 136.9, 147.5, 148.3, 167.5, 168.7, 169.0, 170.8, 171.2 and 182.2; MS (FAB) m/z (relative intensity) 1029 (M^++Cs, 26); HRMS (FAB) m/z 1029.2928 ($C_{54}H_{52}N_6O_7Cs$ requires 1029.2952).

45: (2:1 of two rotamers) mp 135-136 ^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) of rotamer A 3.0 (s, 3H), 4.54 (s, 2H), 4.58 (s, 2H), 7.21-7.90 (m, 9H), rotamer B 2.96 (s, 3H), 4.55 (s, 2H), 4.60 (s, 2H), 7.21-7.90 (m, 9H); ^{13}C-NMR ($CDCl_3$) rotamer A 33.7, 39.3, 51.5, 123.5, 126.4, 128.2, 128.7, 134.0, 136.4, 165.6 and 168.1, rotamer B 34.2, 39.2, 52.6, 123.4, 127.6, 128.0, 128.3, 128.8, 132.2, 135.4, 165.9 and 168.1; MS (FAB) m/z (relative intensity) 309 (M^++1, 40); HRMS (FAB) m/z 309.1236 ($C_{18}H_{17}N_2O_3$ requires 309.1239).

46: mp 135-136 ^{0}C (chloroform-hexane, lit[21] 250 ^{0}C); spectroscopic data matched those reported previously;[21] ^{1}H-NMR ($CDCl_3$) 2.82 (d, 3H, J=4.8Hz), 4.33 (s, 2H), 5.81 (br, 1H), 7.26-7.90 (m, 4H); ^{13}C-NMR (CD_3OD) 22.7, 37.3,120.6, 129.8, 131.8, 165.6 and 166.1.

47: ^{1}H-NMR ($CDCl_3$) 3.25 and 3.79 (two d, 2H, J=12.4Hz), 4.00 and 4.63 (two d, 2H, J=18.4Hz), 4.56 and 4.69 (two d, 2H, J=16.0Hz), 4.99 (s, 1H), 7.19-7.35 (m, 5H), 7.69-8.45 (m, 3H); ^{13}C-NMR ($CDCl_3$) 41.3, 50.7, 53.9, 83.1, 119.3, 123.4, 127.9, 128.0, 128.9, 132.6, 135.0, 149.6, 150.0, 162.3 and 163.9; MS (FAB) m/z (relative intensity) 354 (M^++1, 35), 336 (8), 309 (9), 261 (5), 91 (100); HRMS (FAB) m/z 354.1075 ($C_{18}H_{16}N_3O_5$ requires 354.1090).

48: mp 158-159 ^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) 4.83 (s, 2H), 4.92 (s, 2H), 7.22-7.40 (m, 5H), 8.05-8.62 (m, 3H), 9.22 (br, 1H); ^{13}C-NMR ($CDCl_3$) 41.8, 45.0, 119.1, 125.0, 127.8, 128.2, 129.0, 129.6, 133.3, 135.3, 136.2, 151.9, 165.0, 165.3 and 167.4; MS

49: mp 118-120 ^{0}C (chloroform-hexane); ^{1}H-NMR ($CDCl_3$) 4.40 (s, 2H), 4.45 (d, 2H, J=7.5Hz), 6.01 (s, 1H), 7.25-7.38 (m, 5H), 8.10-8.68 (m, 3H); ^{13}C-NMR ($CDCl_3$) 41.0, 44.0, 119.5, 125.3, 128.0, 129.1, 129.7, 136.3, 137.5, 165.2, 165.4 and 165.9; MS (FAB) m/z (relative intensity) 340 (M^++1, 34), 107 (10); HRMS (FAB) m/z 340.0936 ($C_{17}H_{14}N_3O_5$ requires 340.0933).

TMS-Terminated N-Phthaloyl-(N-benzyl-L-alanine) Polypeptide 53. To a solution of N-benzyl-L-alanine amide **50** (395 mg, 1.12 mmol) and triethylamine (341 mg, 3.36 mmol) in 6 mL of CH_2Cl_2 at 0 °C was added N-phthaloylglyclychloride **8** (280 mg, 1.24 mmol). After stirring for 30 min at 0 °C and for 3h at 25 °C, the mixture was washed with water and satd. $NaHCO_3$, dried and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, 1:1 hexane:EtOAc), yielding 522 mg (87%) of **53**. ^{1}H NMR 0.01 (s, 9H), 1.17 (d, 3H), 2.56 and 3.13 (ABq, J=15 Hz, 2H), 4.19 and 4.38 (ABq, J=16.5 Hz, 2H), 4.28 and 5.08 (ABq, J=16.5 Hz, 2H), 4.71 and 5.10 (ABq, J=18.5 Hz, 2H), 5.70 (q, 1H), 7.13 (m, 2H), 7.23-7.39 (m, 8H), 7.68 (m, 2H), 7.82 (m, 2H); ^{13}C NMR -1.3, 15.9, 39.2, 39.9, 47.0, 48.3, 53.3, 123.4, 125.7, 126.7, 127.4, 127.5, 128.6, 128.8, 129.0, 132.3, 134.0, 137.5 and 137.7, 167.2, 167.7, 170.7; IR 3061, 3031, 2950, 2897, 1719, 1666, 1631, 1433, 1392, 1246, 960, 855, 727, 698; MS (FAB) 542 (M+H, 21), 349 (86), 160 (59), 91 (100); HRMS 542.2479 ($C_{31}H_{36}N_3O_4Si$ requires 542.2476).

TMS-Terminated N-Phthaloyl-(N-benzyl-L-alanine) Polypeptide 54. To a solution of N-benzyl-L-alanine amide **51** (80.0 mg, 0.16 mmol) and triethylamine (49 mg, 0.48 mmol) in 1 mL of CH_2Cl_2 at 0 °C was added N-phthaloylglyclychloride **8** (35.0 mg, 0.16

mmol). After stirring for 30 min at 0°C and for 3h at 25 °C, the mixture was washed with water and satd. $NaHCO_3$, dried and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, 1:1 hexane:EtOAc), yielding 89 mg (79%) of **54**. ^{1}H NMR -0.10 (s, 9H); 1,00 (d, 3H), 1.11 (d, 3H), 2.51 and 2.85 (ABq, J=15 Hz, 2H), 4.18 and 4.31 (ABq, J=16.5 Hz, 2H), 4.21 and 4.96 (ABq, J=16.5 Hz, 2H), 4.77 and 5.01 (ABq, J=16.5 Hz, 2H), 4.78 and 4.94 (ABq, J=18.5 Hz, 2H), 5.44 (q, 1H), 5.56 (q, 1H), 7.08 (m, 2H), 7.17-7.42 (m, 13H), 7.68 (m, 2H), 7.82 (m, 2H), 13C NMR –1.2, 15.4, 16.2, 39.2, 39.6, 46.9, 47.1, 48.6, 49.9, 53.4, 123.4, 125.6, 126.5, 127.0, 127.2, 127.4, 128.5, 128.8, 128.9, 132.2, 134.0, 137.0, 137.4 and 138.4, 166.0, 167.7, 171.0, 173.2; IR 3066, 3031, 2979, 2956, 2897, 1730, 1637, 1435, 1398, 1252, 1170, 1111, 960, 861, 738, 698; MS (FAB) 709 (M+Li, 64), 510 (20), 349 (47), 160 (76), 134 (74), 91 (100); HRMS 709.3420 ($C_{41}H_{46}LiN_4O_5Si$ requires 709.2647).

TMS-Terminated N-Phthaloyl-(N-benzyl-L-alanine) Polypeptide 55. To a solution of N-benzyl-L-alanine amide **13** (107 mg, 0.16 mmol) and triethylamine (49 mg, 0.48 mmol) in 2 ml of CH_2Cl_2 at 0 °C was added N-phthaloylglyclychloride **8** (35.5 mg, 0.16 mmol). After stirring for 30 min at 0 °C and for 3h at 25 °C, the mixture was washed with water, sat. $NaHCO_3$ and brine, dried over $MgSO_4$ and concentrated in vacuo giving a residue which was subjected to column chromatography (silica, hexane:ethyl acetate = 1:1), yielding 112 mg (85%) of **55**. ^{1}H NMR -0.17 (s, 9H), 1.00-1.02 (3d, 9H), 2.38 and 2.58 (ABq, J=15 Hz, 2H), 4.04 (d, J=16.5 Hz, 1H), 4.19 and 4.29 (ABq, J=16.5 Hz, 2H), 4.69 (d, J=18.5 Hz, 1H), 4.70 (d, J=16.5 Hz, 1H), 4.76 (d, J=16.5 Hz, 1H), 4.86-4.95 (m, 4H), 5.27 (q, 1H), 5.38 (q, 1H), 5.45 (q, 1H), 7.07-7.33 (m, 20H), 7.67 (m, 2H), 7.80 (m, 2H); ^{13}C NMR –1.2, 15.4, 15.7, 15.8, 39.2, 39.8, 46.5, 46.8, 48.1, 49.5, 49.6, 53.4, 60.3, 123.4, 125.6, 126.4, 126.6, 127.1, 127.3, 127.5, 128.4, 128.7, 128.9, 132.2, 133.9, 138.1, 138.4, 139.2 and 139.4, 167.0, 167.7, 170.0, 172.3, 173.0; IR 3061, 3043, 2979, 2950, 2886, 1777, 1724, 1643, 1433, 1398, 1246, 1170, 1112, 960, 855, 733, 703. MS (FAB) 631 (M+H, 30), 613 (27), 349 (6), 291 (8), 190 (4), 134 (35), 91 (100); HRMS 631.2916 ($C_{38}H_{39}N_4O_5$ requires 631.2921).

Irradiation of TMS-Terminated N-Phthaloyl-(N-benzyl-L-alanine) Polypeptides 53-55. Solutions of TMS-terminated N-phthaloyl-(N-benzyl-L-alanine) polypeptide **53** (240 mg, 0.44 mmol), **54** (80 mg, 0.114 mmol) and **55** (105 mg, 0.122 mmol) each in 145 ml of CH_3CN/H_2O (35%) were irradiated with pyrex glass filtered light for 1.5 h (for **53** and **54**) and 1 h (for **55**). The photolysates were concentrated in vacuo giving residues that were subjected to column chromatography on silica gel (9:1 EtOAc-MeOH, for residues from **53** and **54**, and 1:1 hexane acetone for residue derived from **55**) yielding 112 mg (55%) of **57** (mp 235.3-235.7 °C, $CHCl_3$-THF), 53 mg (74%) of **58** (mp 221.4-222.3 °C, CH_2Cl_2-hexane), and 37 mg (38%) of **59** (mp 203.4-204.2 °C, CH_2Cl_2-hexane).

57: ^{1}H NMR 1.15 (d, 3H), 2.21 and 4.79 (ABq, J=15 Hz, 2H), 3.67 and 4.25 (ABq, J=16 Hz, 2H), 3.83 and 4.80 (ABq, J=16 Hz, 2H), 4.76 and 4.96 (ABq, J=16 Hz, 2H), 5.06 (q, 1H), 6.51 (s, 1H), 6.81 (m, 2H), 7.12 (m, 1H), 7.15-7.22 (m, 7H), 7.36 (d, 1H), 7.41 (t, 1H), 7.54 (d, 1H), 7.59 (t, 1H); ^{13}C NMR 18.1, 45.5, 47.1, 48.3, 49.9, 52.3, 90.6, 122.4, 123.6, 126.3, 126.8, 127.4, 128.2, 128.7, 130.2, 130.5, 132.9, 136.6, 139.7 and 147.0, 170.1, 170.5, 174.1; IR 3301, 3085, 3056, 3027, 2978, 2925, 1701, 1650, 1470, 1450, 1415, 1320, 1217, 1172, 1074, 1037, 915, 768, 731, 695, 666; MS (FAB) 470 (M+1, 7), 452 (5), 378 (2), 349 (4), 160 (12), 134 (25), 91 (100); HRMS 470.2070 ($C_{28}H_{28}N_3O_4$ requires 470.2080).

58: ^{1}H NMR 1.13 (d, 3H), 1.31 (d, 3H), 3.32 and 5.01 (ABq, J=15 Hz, 2H), 3.71 and 4.33 (ABq, J=18.5 Hz, 2H), 3.74 and 3.88 (ABq, J=17 Hz, 2H), 4.26 and 5.41 (ABq, J=16.5 Hz, 2H), 4.41 (m, 2H), 4.92 (q, 1H), 5.90 (q, 1H), 6.45(d, 2H), 6.85 (d, 1H), 7.09-7.13 (m, 4H), 7.14-7.21 (m, 5H), 7.23 (m, 2H), 7.26 (d, 1H), 7.33-7.39 (m, 3H), 7.61 (d, 1H); ^{13}C NMR 15.9, 17.6, 40.1, 46.7, 46.9, 49.0, 50.0, 50.4, 54.3, 89.4, 121.4, 124.0, 125.9, 126.0, 126.5, 126.6, 127.3, 127.4, 128.3, 128.5, 129.0, 129.6, 129.7, 132.6, 134.0, 137.4, 139.9 and 146.6, 167.9, 168.7, 171.7, 173.2; IR 3358, 3078, 3026, 2985, 1713, 1654, 1433, 1394, 1176, 913, 727, 698, 654; MS (FAB) 631 (M+1, 30), 613 (26), 452 (4), 349 (7), 291 (9), 263 (5), 190 (4), 134 (35), 91 (100); HRMS 631.2916 ($C_{38}H_{39}N_4O_5$ requires 631.2921).

59: ^{1}H NMR (DMSO) 0.26 (d, 3H), 1.03 (d, 3H), 1.37 (d, 3H), 2.11 (q, 1H), 3.20 and 3.43 (ABq, J=16 Hz, 2H), 3.30 and 4.48 (ABq, J=15 Hz, 2H), 3.79 and 4.29 (ABq, J=16 Hz, 2H), 3.99 (q, 1H), 4.46 (m, 2H), 4.69 and 4.76 (ABq, J=15 Hz, 2H), 5.16 (q, 1H), 4.75 and 5.33 (ABq, J=15.5 Hz, 2H), 6.33 (d, 2H), 6.73 (d, 1H), 7.06-7.33 (m, 12H), 7.36 (d, 2H), 7.46 (d, 2H), 7.52 (d, 2H), 7.61(t, 1H), 7.68 (t, 1H), 7.94 (d, 1H); ^{13}C NMR 13.7, 14.5, 15.5, 38.1, 47.0, 49.1, 52.0, 52.4 (), 52.5 (NCHCO), 53.5 (NCH$_2$COH), 56.7 (NCHCO), 56.8 (NCHCO), 89.2 (COH), 123.2, 125.7, 125.9, 126.2, 126.5, 126.8, 127.1, 127.5, 127.8, 128.1, 128.4, 128.5, 128.6, 128.7, 129.5, 129.9, 131.1, 132.2, 135.4, 136.5, 137.5, 139.1 and 143.1, 165.4, 167.5, 169.3, 170.1, 172.2; IR 3347, 3084, 3055, 3026, 2979, 2938, 1713, 1660, 1497, 1450, 1433, 1287, 1170, 1077, 740, 709, 674; MS (FAB) 792 (M+1, 11), 774 (11), 671 (6), 510 (23), 349 (12), 321 (12), 291 (6), 217 (9), 190 (8), 134 (35), 91 (100); HRMS 792.3762 ($C_{48}H_{50}N_5O_6$ requires 792.3762).

Epimerization of Photoproduct 58. A solution of photoproduct **58** (15 mg, 0.024 mmol) in 2 mL of MeCN-H_2O (35%) containing 5 drops of 37% aqueous HCl was stirred at room temperature for 12 h. The mixture was concentrated in vacuo giving a residue that was subjected to column chromatography (silica, hexane:acetone = 1:1) yielding a mixture of compound **58** and its ß-OH epimer in a ratio of 2.4 : 1 (based on ^{1}H NMR), and 6 mg of the dehydration product **60**. ^{1}H NMR 1.33 (d, 3H), 1.39 (d, 3H), 4.01 and 5.54 (ABq, J=13.7 Hz, 2H), 4.09 and 4.45 (ABq, J=16.5 Hz, 2H), 4.34 and 4.51 (ABq, J=18 Hz, 2H), 4.47 and 5.55 (ABq, J=16.5 Hz, 2H), 4.71 (q, 1H), 5.68 (q, 1H), 6.14 (s, 1H), 7.15-7.30 (m, 12H), 7.32 (d, 1H), 7.38 (t, 2H), 7.54 (m, 3H), 7,89 (d, 1H); ^{13}C NMR 15.7, 43.5, 48.1, 49.1, 51.9, 54.1, 54.5, 103.5, 119.7, 124.4, 126.2, 126.3, 126.5, 128.0, 128.1, 128.4, 128.5, 128.8, 129.2, 130.5, 132.9, 135.7, 136.5, 136.6, 138.7, 139.9, 168.1, 169.5, 170.6, 172.6; IR 1730, 1660.

X-Ray Crystallographic Structure Determinations. The structures of compounds **40**, **41a**, **57**, **58** and **59** were determined by using x-ray crystallographic methods. Suitable crystals of these substances were obtained by recrystallization from the solvents specified in the experimental section above. The crystals were mounted on glass fibers and placed on a Siemens (Bruker) P4/F diffractometer, equipped with a graphite monochromator. Mo Kα [λ=0.71073 A] was used to collect omega scan data at 293 °K. Several standard reflections were monitored every 97 reflections to check for crystal decay. There were no signs of crystal decay in each case. The data were reduced and corrected for Lorentz and polarization effects by using XSCANS (Siemen, Bruker, 1995). Absorption corrections, direct methods structure solutions, tabulation of results, and CIF generation were accomplished by using the SHELXTL-NT crystallographic package (Siemens, Bruker, 1997). Direct methods showed all non-hydrogen atom positions. Final refinements included all hydrogen atoms. Hydrogen atoms on carbon were positioned in idealized geometries and had fixed isotropic U's set to 1.25 U (equiv) of their parent atoms. Hydrogen atoms on oxygen

were refined in position and isotropic U. Crystallographic data for these compounds are given in Table 6 and additional experimental details, crystallographic results, bond lengths and bond angles are given in the Supporting Information.

SUPPORTING INFORMATION

Xray Data for 40

A colorless plate of compound with approximate dimensions 0.062 x 0.34 x 0.02mm was mounted on a glass fiber and used for the X-ray crystallographic analysis. The X-ray data were measured at 298K on a Bruker/Siemens P4 diffractometer using a scintillation detector, graphite monochromator, and Mo Ka fine-focus sealed tube [wavelength=0.71073 Å] operated at 1.5kW power [50kV, 30mA], using ω scans for all data 2_ between 3° and 45° at 298 K.

The final cell constants were determined by a least squares fit to the settings of 20 reflections with 2_ between 5.16° and 15.07°; the crystals were small, reflections quite broad; crystal used was best of 2 examined; scan ranges ω scans, 1.7° above and below reflection, variable speeds 4-20°/min, 3 check reflections were monitored every 97 measurements; A total of 10956 reflections [2614 independent reflections] were collected in 239 hours with 3 standard reflections monitored every 97 reflections. Crystal decay was negligible during data collection.

The structure was solved and refined using the Bruker SHELXTL-NT (V.5.10) Software package in space group P2(1)2(1)2(1) with Z=4 for the formula Unit $C_{21}H_{23}N_3O_5$ with Z=4. All nonhydrogen atoms refined anisotropically; H's on C's in idealized positions [riding model], H on O allowed to refine In position. All H's with fixed isotropic U's set to 1.25Uequiv of parent atom. Final full matrix least-squares refinement on F^2 with 272 variables converged at R1=6.24% for the observed data and wR2=15.81% for all data. The largest peak and hole on the final difference electron density synthesis was 0.278 and –0.295 $e^-/Å^3$ with an RMS deviation from mean of 0.06 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.314 g/cm^3 and F(000) was 840 e^-. Data could not distinguish absolute structure.

Table 1A: Crystal data and data collection

Identification code	PM021
Empirical formula	$C_{21}H_{23}N_3O_5$
Formula weight	397.42
Crystal size	0.62 x 0.34 x 0.02 mm
Crystal color and habit	colorless plate
Crystal system	Orthorhombic
Space group	$P2_12_12_1$

Unit cell dimensions	a = 7.469(3) Å	α = 90°
	b = 8.452(4) Å	β = 90 °
	c = 31.817(12) Å	γ = 90°
Volume	2008.5(15) $Å^3$	
Z	4	
Density (calculated)	1.314 Mg/m^3	
Absorption coefficient	0.095 mm^{-1}	
F(000)	840	
Diffractometer used	Siemens P4	
Radiation and wavelength	MoKα with λ=0.71073 Å	
Scan type	ω	
Temperature	298 K	
2θ range for data collection	5.00 to 45.00°	
Index ranges	$-8 \le h \le 8$ $-9 \le k \le 9$	$-34 \le l \le 34$
Reflections collected	10394	
Independent reflections	2614 (R_{int} = 0.1225)	
Observed reflections	1750 (F>4σ(F))	
Absorption correction	Face-indexed gaussian	
Max. and min. transmission	0.99809 and 0.97451	

Table 1B: Solution and refinement

Structure solution program	SHELTX-NT (Bruker AXS,1997)
Solution	direct methods
Refinement method	Full-matrix Least-Squares on F^2
Extinction correction	SHELXTL-NT (Bruker AXS,1997)
Extinction coefficients	0.014(2)
Hydrogen atoms	mixed
Weighting scheme	$w^{-1}=\sigma^2Fo^2+(0.0724P)^2+0.11P$ where $P=(Fo^2+2Fc^2)/3$
Data / restraints / parameters	2614 / 0 / 272
Data-to-parameter-ratio	9.6 : 1 (6.4 : 1 [F>4σ(F)])
Final R indices [F>4σ(F)]	$R_1 = 0.0624$, $wR_2 = 0.1338$
R indices (all data)	$R_1 = 0.1054$, $wR_2 = 0.1581$
Goodness-of-Fit on F^2	1.059
Largest and mean Δ/σ	0.000 0.000
Largest difference peak	0.278 eÅ^{-3}
Largest difference hole	-0.295 eÅ^{-3}
Refinement details :	
Program used	'SHELXTL-NT (Bruker AXS,1997)'
Cif by XCIF	'SHELXTL-NT (Bruker AXS,1997)'
Function minimized	$\Sigma w(F_o^2-F_c^2)^2$
WR_2	$\{\Sigma w(F_o^2-F_c^2)^2/\Sigma w(F_o^2)^2\}^{1/2}$
R_1	$\Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$
The solution is a racemic twin	

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [Å x 10^3] for pm021. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U(eq)
O(1)	6101(6)	6068(6)	945(1)	61(1)
O(2)	11359(6)	6639(5)	322(1)	48(1)
O(3)	8894(6)	2840(6)	1522(1)	58(1)
O(4)	5499(6)	3208(5)	215(1)	49(1)
O(5)	4280(6)	5916(6)	-159(1)	52(1)
N(1)	8862(6)	5490(5)	663(1)	36(1)
N(2)	11342(6)	3837(5)	1207(2)	42(1)
N(3)	7448(6)	2220(5)	684(1)	39(1)
C(1)	7721(9)	6241(7)	937(2)	44(2)
C(2)	8841(8)	7275(6)	1201(2)	37(1)
C(3)	8428(9)	8212(7)	1536(2)	49(2)
C(4)	9753(11)	9122(8)	1720(2)	59(2)
C(5)	11465(10)	9111(7)	1558(2)	54(2)
C(6)	11908(9)	8145(7)	1224(2)	53(2)
C(7)	10573(8)	7225(6)	1048(2)	40(2)
C(8)	10738(7)	6030(7)	705(2)	39(2)
C(9)	11951(9)	4655(7)	826(2)	45(2)
C(10)	9796(8)	2995(7)	1206(2)	43(2)
C(11)	9316(8)	2055(7)	822(2)	41(2)
C(12)	6971(9)	3288(7)	402(2)	39(2)
C(13)	8230(8)	4635(7)	296(2)	41(2)
C(14)	6196(9)	984(7)	812(2)	57(2)
C(15)	11933(9)	4501(7)	1619(2)	51(2)
C(16)	12797(9)	3239(7)	1892(2)	41(2)
C(17)	11937(10)	2569(8)	2223(2)	56(2)
C(18)	12734(12)	1458(9)	2477(2)	67(2)
C(19)	14507(12)	1050(9)	2390(2)	74(2)
C(20)	15386(11)	1713(9)	2068(2)	65(2)
C(21)	14558(9)	2801(8)	1819(2)	52(2)

Table 3. Bond lengths [Å] and angles [°] for pm021.

O(1)-C(1)	1.219(7)
O(2)-C(8)	1.399(7)
O(2)-H(2O)	0.97(7)
O(3)-C(10)	1.217(6)
O(4)-C(12)	1.253(7)
O(5)-H(5W)	0.97(7)
O(5)-H(5Y)	0.94(7)
N(1)-C(1)	1.373(7)
N(1)-C(13)	1.452(7)
N(1)-C(8)	1.480(7)
N(2)-C(10)	1.356(7)
N(2)-C(9)	1.467(7)
N(2)-C(15)	1.494(7)

N(3)-C(12)	1.322(7)
N(3)-C(14)	1.460(7)
N(3)-C(11)	1.469(7)
C(1)-C(2)	1.474(8)
C(2)-C(3)	1.363(8)
C(2)-C(7)	1.383(8)
C(3)-C(4)	1.384(9)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.379(9)
C(4)-H(4A)	0.9300
C(5)-C(6)	1.380(9)
C(5)-H(5A)	0.9300
C(6)-C(7)	1.384(8)
C(6)-H(6A)	0.9300
C(7)-C(8)	1.493(8)
C(8)-C(9)	1.523(8)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-C(11)	1.502(8)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(12)-C(13)	1.515(8)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-H(14A)	0.9600
C(14)-H(14B)	0.9600
C(14)-H(14C)	0.9600
C(15)-C(16)	1.520(8)
C(15)-H(15A)	0.9700
C(15)-H(15B)	0.9700
C(16)-C(17)	1.358(8)
C(16)-C(21)	1.386(8)
C(17)-C(18)	1.373(9)
C(17)-H(17A)	0.9300
C(18)-C(19)	1.396(10)
C(18)-H(18A)	0.9300
C(19)-C(20)	1.338(10)
C(19)-H(19A)	0.9300
C(20)-C(21)	1.363(9)
C(20)-H(20A)	0.9300
C(21)-H(21A)	0.9300

C(8)-O(2)-H(2O)	121(4)
H(5W)-O(5)-H(5Y)	100(5)
C(1)-N(1)-C(13)	122.5(5)
C(1)-N(1)-C(8)	112.9(5)
C(13)-N(1)-C(8)	122.2(5)
C(10)-N(2)-C(9)	120.7(5)
C(10)-N(2)-C(15)	116.8(5)
C(9)-N(2)-C(15)	117.2(5)
C(12)-N(3)-C(14)	120.3(5)
C(12)-N(3)-C(11)	121.5(5)
C(14)-N(3)-C(11)	117.2(5)
O(1)-C(1)-N(1)	125.1(6)
O(1)-C(1)-C(2)	128.4(6)
N(1)-C(1)-C(2)	106.5(6)

C(3)-C(2)-C(7)	120.3(6)
C(3)-C(2)-C(1)	131.5(6)
C(7)-C(2)-C(1)	108.1(5)
C(2)-C(3)-C(4)	119.5(6)
C(2)-C(3)-H(3A)	120.3
C(4)-C(3)-H(3A)	120.3
C(5)-C(4)-C(3)	120.1(6)
C(5)-C(4)-H(4A)	120.0
C(3)-C(4)-H(4A)	120.0
C(4)-C(5)-C(6)	121.0(6)
C(4)-C(5)-H(5A)	119.5
C(6)-C(5)-H(5A)	119.5
C(5)-C(6)-C(7)	118.2(6)
C(5)-C(6)-H(6A)	120.9
C(7)-C(6)-H(6A)	120.9
C(2)-C(7)-C(6)	120.9(5)
C(2)-C(7)-C(8)	110.8(5)
C(6)-C(7)-C(8)	128.2(6)
O(2)-C(8)-N(1)	110.5(4)
O(2)-C(8)-C(7)	114.5(5)
N(1)-C(8)-C(7)	101.3(5)
O(2)-C(8)-C(9)	107.7(5)
N(1)-C(8)-C(9)	110.5(5)
C(7)-C(8)-C(9)	112.3(5)
N(2)-C(9)-C(8)	112.6(5)
N(2)-C(9)-H(9A)	109.1
C(8)-C(9)-H(9A)	109.1
N(2)-C(9)-H(9B)	109.1
C(8)-C(9)-H(9B)	109.1
H(9A)-C(9)-H(9B)	107.8
O(3)-C(10)-N(2)	121.8(5)
O(3)-C(10)-C(11)	118.9(6)
N(2)-C(10)-C(11)	118.8(5)
N(3)-C(11)-C(10)	114.7(5)
N(3)-C(11)-H(11A)	108.6
C(10)-C(11)-H(11A)	108.6
N(3)-C(11)-H(11B)	108.6
C(10)-C(11)-H(11B)	108.6
H(11A)-C(11)-H(11B)	107.6
O(4)-C(12)-N(3)	121.5(5)
O(4)-C(12)-C(13)	118.6(5)
N(3)-C(12)-C(13)	119.8(5)
N(1)-C(13)-C(12)	113.4(4)
N(1)-C(13)-H(13A)	108.9
C(12)-C(13)-H(13A)	108.9
N(1)-C(13)-H(13B)	108.9
C(12)-C(13)-H(13B)	108.9
H(13A)-C(13)-H(13B)	107.7
N(3)-C(14)-H(14A)	109.5
N(3)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
N(3)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
N(2)-C(15)-C(16)	111.4(5)
N(2)-C(15)-H(15A)	109.4
C(16)-C(15)-H(15A)	109.4

N(2)-C(15)-H(15B)	109.4
C(16)-C(15)-H(15B)	109.4
H(15A)-C(15)-H(15B)	108.0
C(17)-C(16)-C(21)	117.9(6)
C(17)-C(16)-C(15)	122.4(6)
C(21)-C(16)-C(15)	119.6(6)
C(16)-C(17)-C(18)	122.4(7)
C(16)-C(17)-H(17A)	118.8
C(18)-C(17)-H(17A)	118.8
C(17)-C(18)-C(19)	117.6(7)
C(17)-C(18)-H(18A)	121.2
C(19)-C(18)-H(18A)	121.2
C(20)-C(19)-C(18)	120.9(7)
C(20)-C(19)-H(19A)	119.5
C(18)-C(19)-H(19A)	119.5
C(19)-C(20)-C(21)	120.3(8)
C(19)-C(20)-H(20A)	119.8
C(21)-C(20)-H(20A)	119.8
C(20)-C(21)-C(16)	120.9(7)
C(20)-C(21)-H(21A)	119.6
C(16)-C(21)-H(21A)	119.6

Table 4. Anisotropic displacement parameters [Å x 10^3] for pm021.The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$(ha^*)^2U_{11}$ + ... + 2hka*b*U_{12}]

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	33(3)	75(3)	73(3)	-19(3)	5(2)	1(2)
O(2)	52(3)	49(3)	43(2)	11(2)	12(2)	-2(2)
O(3)	54(3)	78(3)	43(3)	-4(2)	0(2)	-9(3)
O(4)	54(3)	45(3)	48(3)	-4(2)	-20(2)	1(2)
O(5)	54(3)	53(3)	48(3)	11(2)	9(2)	4(2)
N(1)	37(3)	39(3)	31(3)	0(2)	-3(2)	1(2)
N(2)	47(3)	38(3)	41(3)	1(2)	-7(3)	-3(3)
N(3)	55(3)	30(3)	34(3)	4(2)	-8(2)	-7(2)
C(1)	56(5)	38(4)	37(4)	6(3)	3(3)	12(3)
C(2)	50(4)	26(3)	35(3)	0(3)	1(3)	3(3)
C(3)	60(4)	45(4)	42(4)	1(3)	-1(4)	8(4)
C(4)	93(6)	44(4)	40(4)	-13(3)	-3(4)	14(4)
C(5)	67(5)	35(4)	60(4)	-2(3)	-15(4)	-5(4)
C(6)	59(4)	41(4)	57(4)	-1(4)	-14(4)	-2(3)
C(7)	60(4)	28(3)	31(3)	2(3)	-7(3)	-2(3)
C(8)	39(4)	38(3)	40(4)	6(3)	-1(3)	-6(3)
C(9)	48(4)	41(4)	45(4)	6(3)	4(3)	1(3)
C(10)	46(4)	51(4)	32(3)	6(3)	1(3)	0(3)
C(11)	54(4)	27(3)	41(3)	6(3)	-8(3)	-7(3)
C(12)	56(4)	32(3)	28(3)	-4(3)	0(3)	2(3)
C(13)	56(4)	33(3)	35(3)	7(3)	6(3)	0(3)
C(14)	70(5)	41(4)	60(4)	16(3)	-8(4)	-6(4)
C(15)	57(4)	50(4)	45(4)	-6(3)	-16(3)	1(4)
C(16)	57(4)	36(4)	30(3)	-9(3)	-2(3)	-8(3)

C(17)	64(5)	64(5)	40(4)	-8(4)	-2(4)	-1(4)
C(18)	102(6)	66(5)	34(4)	9(4)	3(4)	-8(5)
C(19)	97(7)	71(5)	53(5)	12(4)	-19(5)	19(5)
C(20)	69(5)	76(5)	51(4)	2(4)	-8(4)	9(4)
C(21)	57(5)	56(4)	42(4)	2(4)	3(3)	-5(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for pm021.

Atom	x	y	z	U(eq)
H(2O)	10850(80)	7590(80)	204(18)	60
H(5W)	3350(90)	6220(80)	40(20)	65
H(5Y)	4700(90)	4980(80)	-31(19)	65
H(3A)	7264	8239	1640	61
H(4A)	9489	9743	1954	74
H(5A)	12333	9764	1675	68
H(6A)	13072	8113	1121	66
H(9A)	11994	3905	595	56
H(9B)	13155	5049	871	56
H(11A)	10093	2378	593	51
H(11B)	9552	946	878	51
H(13A)	9253	4215	145	51
H(13B)	7618	5369	111	51
H(14A)	6266	116	618	71
H(14B)	5001	1401	813	71
H(14C)	6499	620	1089	71
H(15A)	10909	4944	1765	64
H(15B)	12785	5349	1570	64
H(17A)	10766	2874	2281	70
H(18A)	12116	995	2699	84
H(19A)	15084	306	2557	92
H(20A)	16567	1431	2015	82
H(21A)	15182	3255	1597	65

Table 6. Torsion angles [°] for pm021.

C(13)-N(1)-C(1)-O(1)	12.6(9)
C(8)-N(1)-C(1)-O(1)	175.4(6)
C(13)-N(1)-C(1)-C(2)	-166.6(5)
C(8)-N(1)-C(1)-C(2)	-3.8(6)
O(1)-C(1)-C(2)-C(3)	5.1(11)
N(1)-C(1)-C(2)-C(3)	-175.8(6)
O(1)-C(1)-C(2)-C(7)	-173.1(6)
N(1)-C(1)-C(2)-C(7)	6.0(6)
C(7)-C(2)-C(3)-C(4)	0.5(9)
C(1)-C(2)-C(3)-C(4)	-177.6(6)
C(2)-C(3)-C(4)-C(5)	1.7(9)
C(3)-C(4)-C(5)-C(6)	-3.0(10)
C(4)-C(5)-C(6)-C(7)	2.1(9)
C(3)-C(2)-C(7)-C(6)	-1.5(9)
C(1)-C(2)-C(7)-C(6)	177.0(5)
C(3)-C(2)-C(7)-C(8)	175.5(5)
C(1)-C(2)-C(7)-C(8)	-6.0(6)
C(5)-C(6)-C(7)-C(2)	0.2(9)

C(5)-C(6)-C(7)-C(8)	-176.2(6)
C(1)-N(1)-C(8)-O(2)	-121.5(5)
C(13)-N(1)-C(8)-O(2)	41.4(7)
C(1)-N(1)-C(8)-C(7)	0.3(6)
C(13)-N(1)-C(8)-C(7)	163.1(5)
C(1)-N(1)-C(8)-C(9)	119.5(5)
C(13)-N(1)-C(8)-C(9)	-77.6(6)
C(2)-C(7)-C(8)-O(2)	122.5(5)
C(6)-C(7)-C(8)-O(2)	-60.8(8)
C(2)-C(7)-C(8)-N(1)	3.6(6)
C(6)-C(7)-C(8)-N(1)	-179.7(6)
C(2)-C(7)-C(8)-C(9)	-114.3(5)
C(6)-C(7)-C(8)-C(9)	62.4(8)
C(10)-N(2)-C(9)-C(8)	67.7(7)
C(15)-N(2)-C(9)-C(8)	-85.6(6)
O(2)-C(8)-C(9)-N(2)	-175.4(5)
N(1)-C(8)-C(9)-N(2)	-54.7(6)
C(7)-C(8)-C(9)-N(2)	57.6(7)
C(9)-N(2)-C(10)-O(3)	-150.4(6)
C(15)-N(2)-C(10)-O(3)	2.9(8)
C(9)-N(2)-C(10)-C(11)	37.9(8)
C(15)-N(2)-C(10)-C(11)	-168.8(5)
C(12)-N(3)-C(11)-C(10)	92.8(6)
C(14)-N(3)-C(11)-C(10)	-98.1(6)
O(3)-C(10)-C(11)-N(3)	53.5(8)
N(2)-C(10)-C(11)-N(3)	-134.5(5)
C(14)-N(3)-C(12)-O(4)	-5.2(8)
C(11)-N(3)-C(12)-O(4)	163.6(5)
C(14)-N(3)-C(12)-C(13)	175.5(5)
C(11)-N(3)-C(12)-C(13)	-15.7(8)
C(1)-N(1)-C(13)-C(12)	-59.6(7)
C(8)-N(1)-C(13)-C(12)	139.2(5)
O(4)-C(12)-C(13)-N(1)	128.8(5)
N(3)-C(12)-C(13)-N(1)	-51.9(7)
C(10)-N(2)-C(15)-C(16)	78.1(7)
C(9)-N(2)-C(15)-C(16)	-127.6(6)
N(2)-C(15)-C(16)-C(17)	-104.0(7)
N(2)-C(15)-C(16)-C(21)	79.5(7)
C(21)-C(16)-C(17)-C(18)	-1.7(9)
C(15)-C(16)-C(17)-C(18)	-178.3(6)
C(16)-C(17)-C(18)-C(19)	1.5(10)
C(17)-C(18)-C(19)-C(20)	-0.4(11)
C(18)-C(19)-C(20)-C(21)	-0.2(12)
C(19)-C(20)-C(21)-C(16)	-0.1(11)
C(17)-C(16)-C(21)-C(20)	1.0(9)
C(15)-C(16)-C(21)-C(20)	177.7(6)

Table 7. Hydrogen bonds for pm021 [Å].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)-H(5Y)...O(4)	0.94(7)	1.79(7)	2.735(6)	179(6)
O(5)-H(5W)...O(2)#1	0.97(7)	1.77(7)	2.734(6)	169(6)
O(2)-H(2O)...O(5)#2	0.97(7)	1.73(6)	2.637(6)	155(5)

Symmetry transformations used to generate equivalent atoms:

#1 x-1,y,z #2 x+1,-y+3/2,-z

Xray Data for 41a

A colorless prism of compound with approximate dimensions 0.46 x 0.27 x 0.23mm was mounted on a glass fiber and used for the X-ray crystallographic analysis. The X-ray data were measured at 298K on a Bruker/Siemens P4 diffractometer using a scintillation detector, graphite monochromator, and Mo Ka fine-focus sealed tube [wavelength=0.71073 Å] operated at 1.5kW power [50kV, 30mA], using ω scans for all data 2_ between 3° and 48° at 293 K.

The final cell constants were determined by a least squares fit to the settings of 49 reflections with 2_ between 5.71° and 24.96°; scan ranges for ω scans : 1.0° above and below reflection, variable speeds 7-36°/min, 3 check reflections were monitored every 97 measurements. A total of 10560 reflections [4713 independent reflections] were collected in 95 hours with 3 standard reflections monitored every 97 reflections. Crystal decay was negligible during data collection.

The structure was solved and refined using the Bruker SHELXTL-NT (V.5.10) Software package in space group P-1 with Z=2 for the formula Unit $C_{36}H_{36}N_4O_6$ with Z=4. All nonhydrogen atoms refined anisotropically; H's on C's in idealized positions [riding model], H on O allowed to refine In position. All H's with fixed isotropic U's set to 1.25Uequiv of parent atom. Final full matrix least-squares refinement on F^2 with 424 variables converged at R1=3.87% for the observed data and wR2= 11.54% for all data. The largest peak and hole on the final difference electron density synthesis was 0.249 and –0.233 $e^-/Å^3$ with an RMS deviation from mean of 0.05 $e^-/Å^3$. On the basis of the final model, the calculated densitywas 1.327 g/cm^3 and F(000) was 656 e^-.

Table 1A: Crystal data and data collection

Identification code	PM022	
Empirical formula	$C_{36}H_{36}N_4O_6$	
Formula weight	620.69	
Crystal size	0.46 x 0.27 x 0.23 mm	
Crystal color and habit	colorless prism	
Crystal system	Triclinic	
Space group	P-1(No.2)	
Unit cell dimensions	a = 6.6763(14) Å	α = 111.772(6)°
	b =15.329(3) Å	β = 97.092(13) °

	c = 16.572(4) Å γ = 93.043(14)°
Volume	1553.9(6) $Å^3$
Z	2
Density (calculated)	1.327 Mg/m^3
Absorption coefficient	0.091 mm^{-1}
F(000)	656
Diffractometer used	Siemens P4
Radiation and wavelength	MoKα with λ=0.71073 Å
Scan type	ω
Temperature	298 K
2θ range for data collection	3.00 to 48.00°
Index ranges	$-7 \le h \le 7$ $-16 \le k \le 16$ $-18 \le l \le 18$
Reflections collected	9986
Independent reflections	4713 (R_{int} = 0.0303)
Observed reflections	3509 (F>4σ(F))
Absorption correction	Face-indexed gaussian
Max. and min. transmission	0.9868 and 0.9795

Experimental details :

Cell determined using 49 reflections with 2θ between 3-48°;
ω scans, 1° above and below reflection, variable speeds 7-36°/min,
3 check reflections monitored every 97 measurements;
Crystal mounted on glass fiber

Table 1B: Solution and refinement

Structure solution program	SHELTX-NT (Bruker AXS,1997)
Solution	direct methods
Refinement method	Full-matrix Least-Squares on F^2
Extinction correction	SHELXTL-NT (Bruker AXS,1997)
Extinction coefficients	none
Hydrogen atoms	mixed
Weighting scheme	$w^{-1}=\sigma^2Fo^2+(0.0626P)^2+0.08P$ where $P=(Fo^2+2Fc^2)/3$
Data / restraints / parameters	4713 / 0 / 424
Data-to-parameter-ratio	11.11: 1 (8.28: 1 [F>4σ(F)])
Final R indices [F>4σ(F)]	$R_1 = 0.0387$, $wR_2 = 0.1025$
R indices (all data)	$R_1 = 0.0576$, $wR_2 = 0.1154$
Goodness-of-Fit on F^2	1.064
Largest and mean Δ/σ	0.000 0.000
Largest difference peak	0.249 eÅ^{-3}
Largest difference hole	-0.233 eÅ^{-3}
Refinement details :	
Program used	'SHELXTL-NT (Bruker AXS,1997)'
Cif by XCIF	'SHELXTL-NT (Bruker AXS,1997)'
Function minimized	$\Sigma w(F_o^2-F_c^2)^2$
wR_2	$\{\Sigma w(F_o^2-F_c^2)^2/\Sigma w(F_o^2)^2\}^{1/2}$
R_1	$\Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for pm022. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	2294(2)	4558(1)	4107(1)	49(1)
O(2)	7617(2)	3471(1)	4943(1)	44(1)
O(3)	5853(2)	821(1)	2876(1)	42(1)
O(4)	2721(2)	-350(1)	178(1)	55(1)
O(5)	1303(2)	2397(1)	2774(1)	42(1)
O(6)	7894(3)	-24(1)	1385(1)	64(1)
N(1)	4374(2)	3421(1)	4127(1)	36(1)
N(2)	3839(2)	1463(1)	3901(1)	34(1)
N(3)	2801(2)	-215(1)	1583(1)	38(1)
N(4)	3492(2)	1845(1)	1806(1)	36(1)
C(1)	3160(3)	4098(1)	4487(1)	38(1)
C(2)	3171(3)	4185(1)	5407(1)	39(1)
C(3)	2118(3)	4738(2)	6043(1)	51(1)
C(4)	2461(4)	4679(2)	6862(2)	63(1)
C(5)	3821(4)	4103(2)	7033(2)	63(1)
C(6)	4893(4)	3562(2)	6399(1)	53(1)
C(7)	4529(3)	3606(1)	5577(1)	38(1)
C(8)	5538(3)	3129(1)	4784(1)	35(1)
C(9)	5590(3)	2060(1)	4502(1)	37(1)
C(10)	4164(3)	876(1)	3093(1)	34(1)
C(11)	2319(3)	277(1)	2454(1)	36(1)
C(12)	2506(3)	126(1)	937(1)	39(1)
C(13)	1939(3)	1131(1)	1165(1)	41(1)
C(14)	2999(3)	2433(1)	2586(1)	35(1)
C(15)	4734(3)	3137(1)	3230(1)	39(1)
C(16)	1858(3)	1549(1)	4202(1)	38(1)
C(17)	1790(3)	1344(1)	5021(1)	36(1)
C(18)	817(3)	1906(2)	5680(1)	47(1)
C(19)	722(4)	1707(2)	6426(2)	64(1)
C(20)	1601(4)	954(2)	6518(2)	62(1)
C(21)	2543(4)	391(2)	5863(2)	61(1)
C(22)	2633(3)	578(2)	5112(1)	51(1)
C(23)	3625(3)	-1118(1)	1421(1)	44(1)
C(24)	2163(3)	-1897(1)	1430(1)	41(1)
C(25)	2917(4)	-2591(2)	1692(2)	56(1)
C(26)	1641(5)	-3350(2)	1648(2)	71(1)
C(27)	-377(5)	-3416(2)	1361(2)	75(1)
C(28)	-1167(4)	-2722(2)	1117(2)	68(1)
C(29)	110(3)	-1962(2)	1150(1)	52(1)
C(30)	5430(3)	1953(1)	1513(1)	41(1)
C(31)	5687(3)	2769(1)	1216(1)	43(1)
C(32)	4163(4)	3304(2)	1132(2)	56(1)
C(33)	4484(5)	4025(2)	825(2)	71(1)
C(34)	6329(6)	4193(2)	604(2)	77(1)
C(35)	7855(5)	3675(2)	692(2)	84(1)
C(36)	7542(4)	2964(2)	997(2)	69(1)

Table 3. Bond lengths [Å] and angles [°] for pm022.

O(1)-C(1)	1.228(2)
O(2)-C(8)	1.414(2)
O(2)-H(2)	0.84(2)
O(3)-C(10)	1.223(2)
O(4)-C(12)	1.229(2)
O(5)-C(14)	1.215(2)
O(6)-H(1W)	1.11(3)
O(6)-H(2W)	0.95(3)
N(1)-C(1)	1.356(2)
N(1)-C(15)	1.441(2)
N(1)-C(8)	1.476(2)
N(2)-C(10)	1.359(2)
N(2)-C(9)	1.456(2)
N(2)-C(16)	1.466(2)
N(3)-C(12)	1.352(2)
N(3)-C(11)	1.445(2)
N(3)-C(23)	1.461(3)
N(4)-C(14)	1.365(2)
N(4)-C(30)	1.459(2)
N(4)-C(13)	1.465(2)
C(1)-C(2)	1.480(3)
C(2)-C(7)	1.377(3)
C(2)-C(3)	1.381(3)
C(3)-C(4)	1.385(3)
C(3)-H(3)	0.9300
C(4)-C(5)	1.376(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.380(3)
C(5)-H(5)	0.9300
C(6)-C(7)	1.381(3)
C(6)-H(6)	0.9300
C(7)-C(8)	1.508(3)
C(8)-C(9)	1.532(3)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-C(11)	1.522(3)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(12)-C(13)	1.525(3)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-C(15)	1.534(3)
C(15)-H(15A)	0.9700
C(15)-H(15B)	0.9700
C(16)-C(17)	1.508(3)
C(16)-H(16A)	0.9700
C(16)-H(16B)	0.9700
C(17)-C(18)	1.376(3)
C(17)-C(22)	1.377(3)
C(18)-C(19)	1.387(3)
C(18)-H(18)	0.9300
C(19)-C(20)	1.371(3)
C(19)-H(19)	0.9300
C(20)-C(21)	1.361(3)
C(20)-H(20)	0.9300

C(21)-C(22)	1.384(3)
C(21)-H(21)	0.9300
C(22)-H(22)	0.9300
C(23)-C(24)	1.507(3)
C(23)-H(23A)	0.9700
C(23)-H(23B)	0.9700
C(24)-C(29)	1.377(3)
C(24)-C(25)	1.387(3)
C(25)-C(26)	1.378(3)
C(25)-H(25)	0.9300
C(26)-C(27)	1.358(4)
C(26)-H(26)	0.9300
C(27)-C(28)	1.378(4)
C(27)-H(27)	0.9300
C(28)-C(29)	1.386(3)
C(28)-H(28)	0.9300
C(29)-H(29)	0.9300
C(30)-C(31)	1.513(3)
C(30)-H(30A)	0.9700
C(30)-H(30B)	0.9700
C(31)-C(32)	1.365(3)
C(31)-C(36)	1.381(3)
C(32)-C(33)	1.395(3)
C(32)-H(32)	0.9300
C(33)-C(34)	1.367(4)
C(33)-H(33)	0.9300
C(34)-C(35)	1.352(4)
C(34)-H(34)	0.9300
C(35)-C(36)	1.376(4)
C(35)-H(35)	0.9300
C(36)-H(36)	0.9300
C(8)-O(2)-H(2)	104.6(16)
H(1W)-O(6)-H(2W)	107(2)
C(1)-N(1)-C(15)	122.91(16)
C(1)-N(1)-C(8)	112.81(15)
C(15)-N(1)-C(8)	123.52(15)
C(10)-N(2)-C(9)	117.24(15)
C(10)-N(2)-C(16)	124.85(15)
C(9)-N(2)-C(16)	117.89(15)
C(12)-N(3)-C(11)	122.75(16)
C(12)-N(3)-C(23)	120.62(16)
C(11)-N(3)-C(23)	116.63(15)
C(14)-N(4)-C(30)	123.72(15)
C(14)-N(4)-C(13)	119.55(16)
C(30)-N(4)-C(13)	116.31(15)
O(1)-C(1)-N(1)	124.90(18)
O(1)-C(1)-C(2)	128.24(18)
N(1)-C(1)-C(2)	106.82(16)
C(7)-C(2)-C(3)	121.80(19)
C(7)-C(2)-C(1)	108.01(16)
C(3)-C(2)-C(1)	130.17(19)
C(2)-C(3)-C(4)	117.3(2)
C(2)-C(3)-H(3)	121.4
C(4)-C(3)-H(3)	121.4
C(5)-C(4)-C(3)	120.9(2)
C(5)-C(4)-H(4)	119.5
C(3)-C(4)-H(4)	119.5

C(4)-C(5)-C(6)	121.6(2)
C(4)-C(5)-H(5)	119.2
C(6)-C(5)-H(5)	119.2
C(5)-C(6)-C(7)	117.6(2)
C(5)-C(6)-H(6)	121.2
C(7)-C(6)-H(6)	121.2
C(2)-C(7)-C(6)	120.78(19)
C(2)-C(7)-C(8)	110.39(16)
C(6)-C(7)-C(8)	128.71(19)
O(2)-C(8)-N(1)	111.38(15)
O(2)-C(8)-C(7)	111.23(15)
N(1)-C(8)-C(7)	100.92(14)
O(2)-C(8)-C(9)	102.90(14)
N(1)-C(8)-C(9)	114.66(14)
C(7)-C(8)-C(9)	116.07(16)
N(2)-C(9)-C(8)	116.60(15)
N(2)-C(9)-H(9A)	108.1
C(8)-C(9)-H(9A)	108.1
N(2)-C(9)-H(9B)	108.1
C(8)-C(9)-H(9B)	108.1
H(9A)-C(9)-H(9B)	107.3
O(3)-C(10)-N(2)	122.36(17)
O(3)-C(10)-C(11)	120.48(16)
N(2)-C(10)-C(11)	117.15(16)
N(3)-C(11)-C(10)	112.05(15)
N(3)-C(11)-H(11A)	109.2
C(10)-C(11)-H(11A)	109.2
N(3)-C(11)-H(11B)	109.2
C(10)-C(11)-H(11B)	109.2
H(11A)-C(11)-H(11B)	107.9
O(4)-C(12)-N(3)	121.45(18)
O(4)-C(12)-C(13)	119.55(17)
N(3)-C(12)-C(13)	118.99(16)
N(4)-C(13)-C(12)	113.03(16)
N(4)-C(13)-H(13A)	109.0
C(12)-C(13)-H(13A)	109.0
N(4)-C(13)-H(13B)	109.0
C(12)-C(13)-H(13B)	109.0
H(13A)-C(13)-H(13B)	107.8
O(5)-C(14)-N(4)	122.60(17)
O(5)-C(14)-C(15)	121.34(17)
N(4)-C(14)-C(15)	116.05(17)
N(1)-C(15)-C(14)	111.67(15)
N(1)-C(15)-H(15A)	109.3
C(14)-C(15)-H(15A)	109.3
N(1)-C(15)-H(15B)	109.3
C(14)-C(15)-H(15B)	109.3
H(15A)-C(15)-H(15B)	107.9
N(2)-C(16)-C(17)	113.73(15)
N(2)-C(16)-H(16A)	108.8
C(17)-C(16)-H(16A)	108.8
N(2)-C(16)-H(16B)	108.8
C(17)-C(16)-H(16B)	108.8
H(16A)-C(16)-H(16B)	107.7
C(18)-C(17)-C(22)	118.89(18)
C(18)-C(17)-C(16)	120.08(17)
C(22)-C(17)-C(16)	121.00(17)

C(17)-C(18)-C(19)	120.1(2)
C(17)-C(18)-H(18)	120.0
C(19)-C(18)-H(18)	120.0
C(20)-C(19)-C(18)	120.6(2)
C(20)-C(19)-H(19)	119.7
C(18)-C(19)-H(19)	119.7
C(21)-C(20)-C(19)	119.4(2)
C(21)-C(20)-H(20)	120.3
C(19)-C(20)-H(20)	120.3
C(20)-C(21)-C(22)	120.5(2)
C(20)-C(21)-H(21)	119.7
C(22)-C(21)-H(21)	119.7
C(17)-C(22)-C(21)	120.5(2)
C(17)-C(22)-H(22)	119.8
C(21)-C(22)-H(22)	119.8
N(3)-C(23)-C(24)	114.99(17)
N(3)-C(23)-H(23A)	108.5
C(24)-C(23)-H(23A)	108.5
N(3)-C(23)-H(23B)	108.5
C(24)-C(23)-H(23B)	108.5
H(23A)-C(23)-H(23B)	107.5
C(29)-C(24)-C(25)	118.9(2)
C(29)-C(24)-C(23)	122.05(19)
C(25)-C(24)-C(23)	119.0(2)
C(26)-C(25)-C(24)	120.5(2)
C(26)-C(25)-H(25)	119.8
C(24)-C(25)-H(25)	119.8
C(27)-C(26)-C(25)	120.2(3)
C(27)-C(26)-H(26)	119.9
C(25)-C(26)-H(26)	119.9
C(26)-C(27)-C(28)	120.3(2)
C(26)-C(27)-H(27)	119.9
C(28)-C(27)-H(27)	119.9
C(27)-C(28)-C(29)	119.8(3)
C(27)-C(28)-H(28)	120.1
C(29)-C(28)-H(28)	120.1
C(24)-C(29)-C(28)	120.2(2)
C(24)-C(29)-H(29)	119.9
C(28)-C(29)-H(29)	119.9
N(4)-C(30)-C(31)	114.74(16)
N(4)-C(30)-H(30A)	108.6
C(31)-C(30)-H(30A)	108.6
N(4)-C(30)-H(30B)	108.6
C(31)-C(30)-H(30B)	108.6
H(30A)-C(30)-H(30B)	107.6
C(32)-C(31)-C(36)	118.5(2)
C(32)-C(31)-C(30)	123.30(19)
C(36)-C(31)-C(30)	118.2(2)
C(31)-C(32)-C(33)	120.3(2)
C(31)-C(32)-H(32)	119.8
C(33)-C(32)-H(32)	119.8
C(34)-C(33)-C(32)	119.8(3)
C(34)-C(33)-H(33)	120.1
C(32)-C(33)-H(33)	120.1
C(35)-C(34)-C(33)	120.4(2)
C(35)-C(34)-H(34)	119.8
C(33)-C(34)-H(34)	119.8

C(34)-C(35)-C(36)	119.8(3)
C(34)-C(35)-H(35)	120.1
C(36)-C(35)-H(35)	120.1
C(35)-C(36)-C(31)	121.1(3)
C(35)-C(36)-H(36)	119.4
C(31)-C(36)-H(36)	119.4

Table 4. Anisotropic displacement parameters [Å^2 x 10^3] for pm022.The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [(ha*)$^2U_{11}$ + ... + 2hka*b*U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	52(1)	36(1)	54(1)	14(1)	-6(1)	9(1)
O(2)	41(1)	34(1)	48(1)	7(1)	0(1)	3(1)
O(3)	47(1)	39(1)	36(1)	12(1)	10(1)	4(1)
O(4)	88(1)	43(1)	31(1)	8(1)	16(1)	5(1)
O(5)	44(1)	41(1)	39(1)	12(1)	7(1)	3(1)
O(6)	60(1)	74(1)	66(1)	34(1)	16(1)	16(1)
N(1)	45(1)	29(1)	29(1)	7(1)	2(1)	8(1)
N(2)	40(1)	30(1)	28(1)	8(1)	3(1)	3(1)
N(3)	55(1)	27(1)	28(1)	8(1)	6(1)	1(1)
N(4)	47(1)	29(1)	29(1)	9(1)	6(1)	0(1)
C(1)	37(1)	29(1)	41(1)	7(1)	-3(1)	1(1)
C(2)	36(1)	31(1)	38(1)	2(1)	2(1)	-1(1)
C(3)	44(1)	40(1)	55(1)	0(1)	9(1)	2(1)
C(4)	71(2)	55(2)	47(1)	-3(1)	24(1)	-2(1)
C(5)	87(2)	57(2)	36(1)	7(1)	13(1)	-2(1)
C(6)	69(2)	47(1)	35(1)	10(1)	3(1)	5(1)
C(7)	44(1)	32(1)	31(1)	5(1)	1(1)	0(1)
C(8)	35(1)	33(1)	33(1)	9(1)	-2(1)	2(1)
C(9)	40(1)	34(1)	32(1)	9(1)	0(1)	6(1)
C(10)	46(1)	26(1)	32(1)	15(1)	8(1)	6(1)
C(11)	48(1)	31(1)	29(1)	11(1)	7(1)	-1(1)
C(12)	48(1)	33(1)	31(1)	9(1)	5(1)	-6(1)
C(13)	55(1)	35(1)	27(1)	10(1)	-1(1)	-4(1)
C(14)	49(1)	27(1)	30(1)	13(1)	6(1)	5(1)
C(15)	48(1)	32(1)	33(1)	10(1)	4(1)	-2(1)
C(16)	43(1)	37(1)	33(1)	12(1)	4(1)	5(1)
C(17)	40(1)	34(1)	31(1)	11(1)	4(1)	2(1)
C(18)	53(1)	45(1)	41(1)	11(1)	12(1)	8(1)
C(19)	71(2)	75(2)	39(1)	11(1)	22(1)	1(1)
C(20)	62(2)	87(2)	46(1)	38(1)	9(1)	-2(1)
C(21)	65(2)	70(2)	67(2)	46(1)	12(1)	12(1)
C(22)	60(1)	50(1)	49(1)	24(1)	17(1)	14(1)
C(23)	55(1)	32(1)	41(1)	11(1)	8(1)	2(1)
C(24)	56(1)	31(1)	31(1)	4(1)	9(1)	1(1)
C(25)	77(2)	40(1)	50(1)	17(1)	11(1)	7(1)
C(26)	115(3)	40(1)	61(2)	20(1)	21(2)	-2(2)
C(27)	115(3)	42(2)	56(2)	7(1)	24(2)	-25(2)
C(28)	67(2)	61(2)	54(2)	0(1)	11(1)	-19(1)
C(29)	65(2)	41(1)	40(1)	8(1)	5(1)	-2(1)
C(30)	53(1)	33(1)	37(1)	12(1)	12(1)	4(1)
C(31)	60(1)	34(1)	31(1)	7(1)	11(1)	-3(1)

C(32)	67(2)	49(1)	54(1)	29(1)	-2(1)	-6(1)
C(33)	100(2)	52(2)	58(2)	29(1)	-16(1)	-10(1)
C(34)	134(3)	49(2)	44(1)	20(1)	7(2)	-28(2)
C(35)	115(3)	60(2)	82(2)	25(2)	49(2)	-12(2)
C(36)	86(2)	50(1)	79(2)	22(1)	42(2)	3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for pm022.

	x	y	z	U(eq)
H(2)	7650(30)	4054(17)	5230(15)	55
H(1W)	6890(40)	288(18)	1878(18)	80
H(2W)	7340(40)	51(19)	863(18)	80
H(3)	1215	5133	5926	64
H(4)	1761	5035	7303	79
H(5)	4023	4077	7588	79
H(6)	5828	3182	6521	66
H(9A)	6790	1891	4227	46
H(9B)	5743	1915	5027	46
H(11A)	1792	-180	2673	46
H(11B)	1267	681	2421	46
H(13A)	1724	1258	631	51
H(13B)	671	1181	1400	51
H(15A)	5996	2850	3172	48
H(15B)	4876	3691	3083	48
H(16A)	840	1118	3735	47
H(16B)	1507	2186	4316	47
H(18)	223	2421	5625	59
H(19)	56	2087	6868	80
H(20)	1554	829	7024	77
H(21)	3132	-124	5920	77
H(22)	3266	184	4665	63
H(23A)	4800	-1021	1865	54
H(23B)	4089	-1324	854	54
H(25)	4293	-2545	1900	70
H(26)	2164	-3819	1815	89
H(27)	-1230	-3931	1329	94
H(28)	-2553	-2762	932	85
H(29)	-421	-1495	983	65
H(30A)	5577	1373	1031	52
H(30B)	6515	2038	1991	52
H(32)	2906	3187	1281	69
H(33)	3445	4390	772	89
H(34)	6536	4668	391	96
H(35)	9113	3797	547	105
H(36)	8598	2610	1056	87

Table 6. Torsion angles [°] for pm022.

C(15)-N(1)-C(1)-O(1)	-2.2(3)
C(8)-N(1)-C(1)-O(1)	168.07(17)
C(15)-N(1)-C(1)-C(2)	179.86(15)
C(8)-N(1)-C(1)-C(2)	-9.8(2)
O(1)-C(1)-C(2)-C(7)	-173.02(19)
N(1)-C(1)-C(2)-C(7)	4.8(2)

O(1)-C(1)-C(2)-C(3)	5.5(3)
N(1)-C(1)-C(2)-C(3)	-176.70(19)
C(7)-C(2)-C(3)-C(4)	-0.8(3)
C(1)-C(2)-C(3)-C(4)	-179.1(2)
C(2)-C(3)-C(4)-C(5)	1.0(3)
C(3)-C(4)-C(5)-C(6)	0.0(4)
C(4)-C(5)-C(6)-C(7)	-1.1(4)
C(3)-C(2)-C(7)-C(6)	-0.3(3)
C(1)-C(2)-C(7)-C(6)	178.34(18)
C(3)-C(2)-C(7)-C(8)	-176.84(18)
C(1)-C(2)-C(7)-C(8)	1.8(2)
C(5)-C(6)-C(7)-C(2)	1.3(3)
C(5)-C(6)-C(7)-C(8)	177.1(2)
C(1)-N(1)-C(8)-O(2)	-107.70(18)
C(15)-N(1)-C(8)-O(2)	62.5(2)
C(1)-N(1)-C(8)-C(7)	10.4(2)
C(15)-N(1)-C(8)-C(7)	-179.31(15)
C(1)-N(1)-C(8)-C(9)	135.97(17)
C(15)-N(1)-C(8)-C(9)	-53.8(2)
C(2)-C(7)-C(8)-O(2)	111.20(18)
C(6)-C(7)-C(8)-O(2)	-65.0(3)
C(2)-C(7)-C(8)-N(1)	-7.05(19)
C(6)-C(7)-C(8)-N(1)	176.8(2)
C(2)-C(7)-C(8)-C(9)	-131.62(17)
C(6)-C(7)-C(8)-C(9)	52.2(3)
C(10)-N(2)-C(9)-C(8)	116.95(18)
C(16)-N(2)-C(9)-C(8)	-61.9(2)
O(2)-C(8)-C(9)-N(2)	-150.78(15)
N(1)-C(8)-C(9)-N(2)	-29.7(2)
C(7)-C(8)-C(9)-N(2)	87.5(2)
C(9)-N(2)-C(10)-O(3)	2.1(2)
C(16)-N(2)-C(10)-O(3)	-179.10(16)
C(9)-N(2)-C(10)-C(11)	-177.86(15)
C(16)-N(2)-C(10)-C(11)	0.9(2)
C(12)-N(3)-C(11)-C(10)	-95.6(2)
C(23)-N(3)-C(11)-C(10)	84.81(19)
O(3)-C(10)-C(11)-N(3)	-8.1(2)
N(2)-C(10)-C(11)-N(3)	171.88(15)
C(11)-N(3)-C(12)-O(4)	-172.07(18)
C(23)-N(3)-C(12)-O(4)	7.5(3)
C(11)-N(3)-C(12)-C(13)	9.2(3)
C(23)-N(3)-C(12)-C(13)	-171.15(17)
C(14)-N(4)-C(13)-C(12)	-120.85(18)
C(30)-N(4)-C(13)-C(12)	66.4(2)
O(4)-C(12)-C(13)-N(4)	-117.55(19)
N(3)-C(12)-C(13)-N(4)	61.2(2)
C(30)-N(4)-C(14)-O(5)	171.45(16)
C(13)-N(4)-C(14)-O(5)	-0.7(3)
C(30)-N(4)-C(14)-C(15)	-9.7(2)
C(13)-N(4)-C(14)-C(15)	178.05(15)
C(1)-N(1)-C(15)-C(14)	-81.3(2)
C(8)-N(1)-C(15)-C(14)	109.42(19)
O(5)-C(14)-C(15)-N(1)	24.0(2)
N(4)-C(14)-C(15)-N(1)	-154.80(16)
C(10)-N(2)-C(16)-C(17)	122.09(18)
C(9)-N(2)-C(16)-C(17)	-59.1(2)
N(2)-C(16)-C(17)-C(18)	137.72(19)

N(2)-C(16)-C(17)-C(22)	-44.5(3)
C(22)-C(17)-C(18)-C(19)	1.0(3)
C(16)-C(17)-C(18)-C(19)	178.79(19)
C(17)-C(18)-C(19)-C(20)	0.4(4)
C(18)-C(19)-C(20)-C(21)	-1.1(4)
C(19)-C(20)-C(21)-C(22)	0.5(4)
C(18)-C(17)-C(22)-C(21)	-1.5(3)
C(16)-C(17)-C(22)-C(21)	-179.3(2)
C(20)-C(21)-C(22)-C(17)	0.8(4)
C(12)-N(3)-C(23)-C(24)	-112.1(2)
C(11)-N(3)-C(23)-C(24)	67.5(2)
N(3)-C(23)-C(24)-C(29)	32.6(3)
N(3)-C(23)-C(24)-C(25)	-150.16(18)
C(29)-C(24)-C(25)-C(26)	2.0(3)
C(23)-C(24)-C(25)-C(26)	-175.3(2)
C(24)-C(25)-C(26)-C(27)	-1.2(4)
C(25)-C(26)-C(27)-C(28)	-0.4(4)
C(26)-C(27)-C(28)-C(29)	1.2(4)
C(25)-C(24)-C(29)-C(28)	-1.2(3)
C(23)-C(24)-C(29)-C(28)	176.04(19)
C(27)-C(28)-C(29)-C(24)	-0.4(3)
C(14)-N(4)-C(30)-C(31)	-73.4(2)
C(13)-N(4)-C(30)-C(31)	98.99(19)
N(4)-C(30)-C(31)-C(32)	-6.9(3)
N(4)-C(30)-C(31)-C(36)	174.86(18)
C(36)-C(31)-C(32)-C(33)	0.5(3)
C(30)-C(31)-C(32)-C(33)	-177.8(2)
C(31)-C(32)-C(33)-C(34)	0.3(3)
C(32)-C(33)-C(34)-C(35)	-1.0(4)
C(33)-C(34)-C(35)-C(36)	0.8(4)
C(34)-C(35)-C(36)-C(31)	0.0(4)
C(32)-C(31)-C(36)-C(35)	-0.6(4)
C(30)-C(31)-C(36)-C(35)	177.7(2)

Table 7. Hydrogen bonds for pm022 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(2)-H(2)...O(1)#1	0.84(2)	2.00(2)	2.836(2)	179(2)
O(6)-H(1W)...O(3)	1.11(3)	1.79(3)	2.878(2)	165(2)
O(6)-H(2W)...O(4)#2	0.95(3)	1.94(3)	2.841(2)	158(2)

Symmetry transformations used to generate equivalent atoms:
"#1 -x+1,-y+1,-z+1 #2 -x+1,-y,-z "

Xray Data for 57

A colorless prism of compound with approximate dimensions 0.64 x 0.32 x 0.21 mm was mounted on a glass fiber and used for the X-ray crystallographic analysis. The X-ray data were measured at 294K on a Bruker/Siemens P4 diffractometer using a scintillation detector, graphite monochromator, and Mo Ka fine-focus sealed tube [wavelength=0.71073 Å] operated at 1.5kW power [50kV, 30mA], using ω scans for h,k,+-l and -h,-k,+-l data 2_ between 3° and 50° at 294 K.

The final cell constants were determined by a least squares fit to the settings of 58 reflections with 2_ between 5.85° and 30.334°; scan ranges for ω scans : 0.6° above and below reflection, variable speeds 6-48°/min, 3 check reflections were monitored every 97 measurements. A total of 9857 reflections [4254 independent reflections] were collected in 51 hours with 3 standard reflections monitored every 97 reflections. Crystal decay was negligible during data collection.

The structure was solved and refined using the Bruker SHELXTL-NT (V.5.10) Software package in space group P2(1)2(1)2(1) with Z=4 for the formula Unit $C_{28}H_{27}N_3O_4$ with Z=4. All nonhydrogen atoms refined anisotropically; All H's in idealized positions [riding model], H on O was fixed between O2 And its closest neighboring O4 at the appropriate distance [looked very good For H-bonding].All H's with fixed isotropic U's set to 1.25Uequiv of parent atom. Final full matrix least-squares refinement on F^2 with 317 variables convered at R1=4.12% for the observed data and wR2= 11.82% for all data. The largest peak and hole on the final difference electrondensity synthesis was 0.627 and –0.668 $e^-/Å^3$ with an RMS deviation from mean of 0.32 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.286 g/cm^3 and F(000) was 992 e^-. Data could not determine absolute structure.

Table 1. Crystal data and structure refinement for pm034.

Formula weight	469.53
Identification code	pm034
Empirical formula	C28H27N3O4
Temperature	294(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	P212121
Unit cell dimensions	a =10.8635(12) A alpha =90o
	b =13.9599(17) A beta =90o
	c =15.9849(18) A gamma =90o
Volume, Z	2424.2(5) A3, 4
Density (calculated)	1.286 Mg/m3
Absorption coefficient	0.087 mm-1
F(000)	992