X-ray Crystallographic Data for *N-tert*-Butoxycarbonyl-L-prolyl-L-leucyl-diethylglycinamide (8aA and 8aB)

1)	Unit cell parameters	
	a (Å)	9.670 (3)
	b (Å)	25.114 (3)
	c (Å)	11.731 (2)
	V $(Å)^3$	2832.1 (9)
	ß (°)	96.26 (2)
2)	Formula	$C_{22}H_{40}O_5N_4$
	Formula weight	440.58
	Z	4
3)	Density, calculated (g/cc)	1.033
4)	Space group	$P2_1$ (#4)
5)	Wavelength used	CuKα (λ = 1.54178 Å)
	Observed reflections	4769
	Unobserved reflections	284
6)	Data collection	Rigaku AFC6S
	Structure solution	Sheldrick, G. M. (1985). SHELZS86. *Program for the Solution of Crystal Structures.* Universitat Gottingen, Deutschland.
	Structure refinement	Molecular Strucutre Corporation. (1995). teXsan *Single Crystal Structure Analysis Software.* MCS, 3200 Research Forest Drive, The Woodlands, TX 77381, USA
7)	Final R value	0.119
8)	Final difference map	featureless [0.80 $e^-/Å^3$(near S, -0.39 $e^-/Å^3$)]

Positional parameters and B (eq) for **8aA** and **8aB**.

atom	x	y	z	B_{eq}
O1A	0.887(2)	0.2125	0.224(2)	3.3(4)
O1	0.701(2)	0.625(1)	0.243(2)	3.0(4)
O2	0.820(2)	0.701(1)	0.298(2)	4.0(5)
O2A	1.058(3)	0.152(1)	0.184(2)	6.7(7)
O3A	0.958(2)	0.2825(10)	0.018(2)	3.3(4)
O3	0.863(2)	0.555(1)	0.460(2)	3.0(4)
O4A	0.813(2)	0.4602(10)	0.107(2)	2.8(4)
O4	0.920(2)	0.3782(10)	0.369(1)	2.5(4)
O5	0.903(2)	0.365(1)	0.769(2)	3.3(4)
O5A	0.783(2)	0.4684(10)	-0.304(1)	2.4(4)
N1A	1.102(3)	0.240(1)	0.210(2)	4.0(6)
N1	0.935(2)	0.620(1)	0.293(2)	2.2(4)
N2A	0.877(2)	0.3524(10)	0.118(2)	1.1(4)
N2	0.824(2)	0.483(1)	0.344(2)	2.6(5)
N3A	0.804(2)	0.454(1)	-0.087(2)	1.4(4)
N3	0.897(2)	0.3813(10)	0.556(2)	1.5(4)
N4	1.024(3)	0.291(1)	0.777(2)	3.3(5)
N4A	0.850(3)	0.555(1)	-0.296(2)	3.8(6)
C1	0.585(4)	0.687(2)	0.113(4)	7(1)
C1A	0.784(5)	0.133(2)	0.313(4)	10(1)
C2	0.477(4)	0.605(2)	0.173(3)	5(1)
C2A	0.766(4)	0.152(2)	0.080(4)	7(1)
C3	0.515(5)	0.681(2)	0.319(4)	9(1)
C3A	0.654(4)	0.206(2)	0.228(3)	6(1)

atom	x	y	z	B_{eq}
C4	0.566(4)	0.653(2)	0.219(3)	4.9(8)
C4A	0.771(4)	0.174(2)	0.210(3)	5.6(9)
C5	0.821(3)	0.650(1)	0.283(2)	1.8(5)
C5A	1.013(4)	0.199(2)	0.207(3)	5.0(8)
C6	1.076(3)	0.641(1)	0.342(3)	3.4(7)
C6A	1.245(5)	0.233(2)	0.187(4)	7(1)
C7	1.155(4)	0.591(2)	0.360(3)	5.1(9)
C7A	1.297(5)	0.290(2)	0.249(4)	9(1)
C8	1.077(3)	0.550(2)	0.274(3)	3.9(7)
C8A	1.179(4)	0.328(1)	0.217(3)	4.5(8)
C9	0.925(3)	0.561(1)	0.270(2)	1.0(4)
C9A	1.045(3)	0.294(1)	0.214(2)	1.7(5)
C10	0.865(3)	0.537(1)	0.365(2)	2.0(5)
C10A	0.959(3)	0.308(1)	0.108(2)	2.2(6)
C11	0.786(3)	0.451(1)	0.431(2)	1.5(5)
C11A	0.788(4)	0.372(1)	0.023(3)	3.8(7)
C12	0.644(4)	0.429(1)	0.389(3)	4.6(8)
C12A	0.634(3)	0.358(1)	0.035(3)	3.9(7)
C13A	0.524(3)	0.370(1)	-0.070(3)	3.9(7)
C13	0.524(4)	0.472(2)	0.367(3)	5.9(10)
C14	0.497(5)	0.501(2)	0.477(4)	6(1)
C14A	0.558(4)	0.340(2)	-0.182(3)	7(1)
C15A	0.383(5)	0.362(2)	-0.046(4)	7(1)
C15	0.388(5)	0.449(2)	0.308(4)	10(1)

atom	x	y	z	B_{eq}
C16	0.869(3)	0.399(1)	0.447(2)	1.6(5)
C16A	0.802(3)	0.436(1)	0.021(3)	3.6(7)
C17	0.970(3)	0.336(1)	0.591(2)	2.0(5)
C17A	0.837(3)	0.509(1)	-0.111(2)	2.1(6)
C18	1.126(3)	0.338(1)	0.567(2)	3.0(6)
C18A	0.971(3)	0.526(1)	-0.062(3)	3.4(7)
C19	1.204(4)	0.381(2)	0.607(3)	6(1)
C19A	1.094(4)	0.496(2)	-0.098(3)	5.4(9)
C20A	0.722(3)	0.547(2)	-0.075(3)	4.4(8)
C20	0.909(3)	0.284(1)	0.545(3)	3.5(7)
C21	0.761(3)	0.276(1)	0.567(3)	3.9(7)
C21A	0.568(4)	0.535(2)	-0.130(3)	5.5(9)
C22A	0.818(3)	0.510(1)	-0.246(2)	2.5(6)
C22	0.967(3)	0.332(1)	0.724(2)	1.4(5)
H1'	0.6799	0.6964	0.1140	9.1854
H1"	0.5302	0.7186	0.1149	9.1854
H1A	0.7446	0.1481	0.3765	12.0011
H1A'	0.7367	0.1013	0.2897	12.0011
H1A"	0.8798	0.1255	0.3351	12.0011
H1	0.5554	0.6674	0.0460	9.1854
H2'	0.5166	0.5902	0.1097	6.1745
H2"	0.4762	0.5794	0.2320	6.1745
H2A	0.6978	0.1252	0.0683	8.6923
H2A'	0.7426	0.1808	0.0281	8.6923

atom	x	y	z	B_{eq}
H2A"	0.8544	0.1385	0.0677	8.6923
H2N	0.8222	0.4683	0.2688	3.0874
H2	0.3852	0.6167	0.1494	6.1745
H2NA	0.8798	0.3702	0.1895	1.2854
H3	0.5820	0.7067	0.3485	11.0728
H3'	0.4293	0.6981	0.2962	11.0728
H3"	0.5029	0.6557	0.3777	11.0728
H3A	0.6698	0.2236	0.3001	8.3797
H3A'	0.6391	0.2319	0.1687	8.3797
H3A"	0.5738	0.1840	0.2271	8.3797
H3NA	0.7830	0.4300	-0.1489	1.7031
H3N	0.8628	0.4022	0.6141	1.7749
H4NA	0.8824	0.5848	-0.2494	4.5251
H4NA'	0.8404	0.5584	-0.3768	4.5251
H4N	1.0237	0.2879	0.8574	3.9695
H4N'	1.0670	0.2644	0.7353	3.9695
H6A'	1.2558	0.2320	0.1074	8.6571
H6	1.1157	0.6633	0.2889	4.0498
H6'	1.0714	0.6590	0.4118	4.0498
H6A	1.2879	0.2027	0.2236	8.6571
H7A'	1.3802	0.3016	0.2205	10.9031
H7	1.2480	0.5964	0.3429	6.0860
H7'	1.1550	0.5795	0.4364	6.0860
H7A	1.3129	0.2854	0.3295	10.9031

atom	x	y	z	B_{eq}
H8A'	1.1870	0.3429	0.1432	5.3953
H8	1.1061	0.5545	0.1994	4.7381
H8'	1.0975	0.5148	0.2998	4.7381
H8A	1.1797	0.3555	0.2719	5.3953
H9A	0.9974	0.2995	0.2803	1.9950
H9	0.8755	0.5523	0.1982	1.2152
H11A	0.8147	0.3569	-0.0464	4.6071
H11	0.7851	0.4700	0.5014	1.8512
H12A'	0.6082	0.3770	0.0987	4.7185
H12	0.6509	0.4106	0.3196	5.5360
H12'	0.6173	0.4048	0.4453	5.5360
H12A	0.6303	0.3205	0.0503	4.7185
H13	0.5549	0.4982	0.3162	7.0970
H13A	0.5328	0.4067	-0.0859	4.6587
H14'	0.4279	0.5276	0.4603	8.3398
H14"	0.4650	0.4760	0.5295	8.3398
H14A	0.5547	0.3022	-0.1693	8.5496
H14A'	0.4907	0.3489	-0.2440	8.5496
H14A"	0.6478	0.3493	-0.1995	8.5496
H14	0.5803	0.5170	0.5109	8.3398
H15'	0.4065	0.4309	0.2393	12.4329
H15"	0.3519	0.4237	0.3579	12.4329
H15A	0.3640	0.3843	0.0160	9.2550
H15A'	0.3211	0.3711	-0.1122	9.2550

atom	x	y	z	B_{eq}
H15A"	0.3691	0.3259	-0.0268	9.2550
H15	0.3227	0.4762	0.2898	12.4329
H18'	1.1268	0.3378	0.4857	3.5726
H18A	0.9809	0.5623	-0.0817	4.1079
H18A'	0.9737	0.5228	0.0191	4.1079
H18	1.1706	0.3072	0.5987	3.5726
H19A'	1.0973	0.5005	-0.1784	6.5387
H19A"	1.1774	0.5096	-0.0580	6.5387
H19	1.2981	0.3756	0.5939	7.9884
H19'	1.1694	0.4123	0.5691	7.9884
H19"	1.1987	0.3845	0.6874	7.9884
H19A	1.0855	0.4593	-0.0813	6.5387
H20	0.9125	0.2837	0.4643	4.2215
H20'	0.9632	0.2557	0.5792	4.2215
H20A	0.7228	0.5443	0.0059	5.2233
H20A'	0.7453	0.5819	-0.0954	5.2233
H21"	0.7312	0.2411	0.5426	4.6407
H21A	0.5067	0.5599	-0.1000	6.6391
H21A'	0.5631	0.5395	-0.2109	6.6391
H21A"	0.5427	0.5001	-0.1122	6.6391
H21	0.7536	0.2791	0.6473	4.6407
H21'	0.7030	0.3016	0.5268	4.6407

$$B_{eq} = \frac{8}{3}\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$$

Anisotropic displacement parameters for **8aA** and **8aB**.

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C1	0.07(3)	0.05(2)	0.17(5)	0.00(2)	0.01(3)	0.05(3)
C1A	0.16(5)	0.05(3)	0.16(5)	-0.04(3)	-0.05(4)	0.06(3)
C2	0.06(3)	0.07(3)	0.06(2)	0.00(2)	0.01(2)	0.01(2)
C3	0.16(5)	0.04(2)	0.18(5)	-0.03(3)	0.12(4)	-0.05(3)

The general temperature factor expression:

$$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

Intramolecular distances (Å) for **8aA** and **8aB**.

atom	atom	distance	atom	atom	distance
O1A	C4A	1.47(4)	O1A	C5A	1.30(4)
O1	C4	1.49(4)	O1	C5	1.36(3)
O2	C5	1.29(3)	O2A	C5A	1.29(4)
O3A	C10A	1.23(3)	O3	C10	1.22(3)
O4A	C16A	1.17(3)	O4	C16	1.21(3)
O5	C22	1.20(3)	O5A	C22A	1.27(3)
N1A	C5A	1.36(4)	N1A	C6A	1.45(5)
N1A	C9A	1.46(3)	N1	C5	1.33(3)
N1	C6	1.51(4)	N1	C9	1.52(3)
N2A	C10A	1.37(3)	N2A	C11A	1.42(3)
N2A	H2NA	0.95	N2	C10	1.43(3)
N2	C11	1.38(3)	N2	H2N	0.95
N3A	C16A	1.34(3)	N3A	C17A	1.45(3)
N3A	H3NA	0.95	N3	C16	1.35(3)
N3	C17	1.38(3)	N3	H3N	0.95
N4	C22	1.29(3)	N4	H4N	0.95
N4	H4N'	0.95	N4A	C22A	1.33(3)
N4A	H4NA	0.96	N4A	H4NA'	0.95
C1	C4	1.53(5)	C1	H1'	0.96
C1	H1"	0.96	C1	H1	0.95
C1A	C4A	1.58(5)	C1A	H1A	0.94
C1A	H1A'	0.94	C1A	H1A"	0.95
C2	C4	1.54(5)	C2	H2'	0.93
C2	H2"	0.95	C2	H2	0.96

atom	atom	distance	atom	atom	distance
C2A	C4A	1.62(5)	C2A	H2A	0.95
C2A	H2A'	0.95	C2A	H2A"	0.94
C3	C4	1.50(5)	C3	H3	0.94
C3	H3'	0.96	C3	H3"	0.94
C3A	C4A	1.42(5)	C3A	H3A	0.95
C3A	H3A'	0.95	C3A	H3A"	0.95
C6	C7	1.45(4)	C6	H6	0.95
C6	H6'	0.96	C6A	C7A	1.64(6)
C6A	H6A'	0.96	C6A	H6A	0.94
C7	C8	1.58(4)	C7	H7	0.96
C7	H7'	0.95	C7A	C8A	1.51(5)
C7A	H7A'	0.94	C7A	H7A	0.95
C8	C9	1.49(4)	C8	H8	0.94
C8	H8'	0.95	C8A	C9A	1.56(4)
C8A	H8A'	0.94	C8A	H8A	0.96
C9	C10	1.44(3)	C9	H9	0.95
C9A	C10A	1.46(4)	C9A	H9A	0.95
C11	C12	1.51(4)	C11	C16	1.53(3)
C11	H11	0.95	C11A	C12A	1.55(4)
C11A	C16A	1.62(4)	C11A	H11A	0.95
C12	C13	1.59(5)	C12	H12	0.95
C12	H12'	0.94	C12A	C13A	1.57(4)
C12A	H12A'	0.95	C12A	H12A	0.94
C13A	C14A	1.58(5)	C13A	C15A	1.44(5)

atom	atom	distance
C13A	H13A	0.95
C13	C15	1.53(6)
C14	H14'	0.95
C14	H14	0.96
C14A	H14A'	0.96
C15A	H15A	0.95
C15A	H15A"	0.94
C15	H15"	0.91
C17	C18	1.57(4)
C17	C22	1.56(3)
C17A	C20A	1.56(4)
C18	C19	1.37(4)
C18	H18	0.95
C18A	H18A	0.96
C19	H19	0.96
C19	H19"	0.95
C19A	H19A"	0.95
C20A	C21A	1.58(5)
C20A	H20A'	0.96
C20	H20	0.95
C21	H21"	0.95
C21	H21'	0.95
C21A	H21A'	0.96

atom	atom	distance
C13	C14	1.53(5)
C13	H13	0.94
C14	H14"	0.95
C14A	H14A	0.93
C14A	H14A"	0.94
C15A	H15A'	0.96
C15	H15'	1.00
C15	H15	0.95
C17	C20	1.50(4)
C17A	C18A	1.42(4)
C17A	C22A	1.58(4)
C18	H18'	0.95
C18A	C19A	1.51(4)
C18A	H18A'	0.96
C19	H19'	0.96
C19A	H19A'	0.96
C19A	H19A	0.95
C20A	H20A	0.95
C20	C21	1.50(4)
C20	H20'	0.94
C21	H21	0.95
C21A	H21A	0.94
C21A	H21A"	0.95

Intramolecular bond angles (°) for **8aA** and **8aB**.

atom	atom	atom	angle	atom	atom	atom	angle
C4A	O1A	C5A	121(2)	C4	O1	C5	123(2)
C5A	N1A	C6A	121(3)	C5A	N1A	C9A	117(2)
C6A	N1A	C9A	119(2)	C5	N1	C6	123(2)
C5	N1	C9	120(2)	C6	N1	C9	115(2)
C10A	N2A	C11A	120(2)	C10A	N2A	H2NA	120.0
C11A	N2A	H2NA	119.1	C10	N2	C11	120(2)
C10	N2	H2N	119.2	C11	N2	H2N	120.0
C16A	N3A	C17A	122(2)	C16A	N3A	H3NA	118.9
C17A	N3A	H3NA	119.1	C16	N3	C17	127(2)
C16	N3	H3N	116.4	C17	N3	H3N	116.4
C22	N4	H4N	119.8	C22	N4	H4N'	119.9
H4N	N4	H4N'	120.3	C22A	N4A	H4NA	120.5
C22A	N4A	H4NA'	120.7	H4NA	N4A	H4NA'	118.8
C4	C1	H1'	110.4	C4	C1	H1"	109.9
C4	C1	H1	110.9	H1'	C1	H1"	108.1
H1'	C1	H1	108.8	H1"	C1	H1	108.8
C4A	C1A	H1A	109.2	C4A	C1A	H1A'	108.5
C4A	C1A	H1A"	108.5	H1A	C1A	H1A'	110.8
H1A	C1A	H1A"	110.0	H1A'	C1A	H1A"	109.9
C4	C2	H2'	110.4	C4	C2	H2"	109.2
C4	C2	H2	108.2	H2'	C2	H2"	110.7
H2'	C2	H2	110.0	H2"	C2	H2	108.2
C4A	C2A	H2A	108.5	C4A	C2A	H2A'	108.9
C4A	C2A	H2A"	109.8	H2A	C2A	H2A'	108.9

atom	atom	atom	angle	atom	atom	atom	angle
H2A	C2A	H2A"	110.5	H2A'	C2A	H2A"	110.3
C4	C3	H3	109.4	C4	C3	H3'	108.2
C4	C3	H3"	109.6	H3	C3	H3'	109.6
H3	C3	H3"	110.7	H3'	C3	H3"	109.4
C4A	C3A	H3A	109.8	C4A	C3A	H3A'	109.6
C4A	C3A	H3A"	109.2	H3A	C3A	H3A'	109.3
H3A	C3A	H3A"	109.5	H3A'	C3A	H3A"	109.4
O1	C4	C1	103(2)	O1	C4	C2	97(2)
O1	C4	C3	115(3)	C1	C4	C2	105(2)
C1	C4	C3	117(3)	C2	C4	C3	115(3)
O1A	C4A	C1A	110(2)	O1A	C4A	C2A	105(2)
O1A	C4A	C3A	103(3)	C1A	C4A	C2A	119(3)
C1A	C4A	C3A	104(3)	C2A	C4A	C3A	112(3)
O1	C5	O2	118(2)	O1	C5	N1	116(2)
O2	C5	N1	124(2)	O1A	C5A	O2A	128(3)
O1A	C5A	N1A	113(3)	O2A	C5A	N1A	118(3)
N1	C6	C7	101(2)	N1	C6	H6	111.7
N1	C6	H6'	110.6	C7	C6	H6	112.4
C7	C6	H6'	111.4	H6	C6	H6'	109.0
N1A	C6A	C7A	93(3)	N1A	C6A	H6A'	111.9
N1A	C6A	H6A	113.4	C7A	C6A	H6A'	112.9
C7A	C6A	H6A	114.5	H6A'	C6A	H6A	109.8
C6	C7	C8	105(2)	C6	C7	H7	109.6
C6	C7	H7'	110.9	C8	C7	H7	110.6

atom	atom	atom	angle	atom	atom	atom	angle
C8	C7	H7'	112.0	H7	C7	H7'	108.5
C6A	C7A	C8A	104(3)	C6A	C7A	H7A'	110.5
C6A	C7A	H7A	109.6	C8A	C7A	H7A'	111.2
C8A	C7A	H7A	110.8	H7A'	C7A	H7A	110.0
C7	C8	C9	107(2)	C7	C8	H8	110.8
C7	C8	H8'	109.9	C9	C8	H8	109.9
C9	C8	H8'	108.8	H8	C8	H8'	109.9
C7A	C8A	C9A	105(2)	C7A	C8A	H8A'	110.1
C7A	C8A	H8A	109.3	C9A	C8A	H8A'	111.8
C9A	C8A	H8A	110.9	H8A'	C8A	H8A	109.3
N1	C9	C8	97(2)	N1	C9	C10	107(2)
N1	C9	H9	112.3	C8	C9	C10	112(2)
C8	C9	H9	112.9	C10	C9	H9	113.0
N1A	C9A	C8A	100(2)	N1A	C9A	C10A	112(2)
N1A	C9A	H9A	113.3	C8A	C9A	C10A	105(2)
C8A	C9A	H9A	112.4	C10A	C9A	H9A	112.0
O3	C10	N2	119(2)	O3	C10	C9	126(2)
N2	C10	C9	113(2)	O3A	C10A	N2A	122(2)
O3A	C10A	C9A	123(2)	N2A	C10A	C9A	113(2)
N2	C11	C12	105(2)	N2	C11	C16	113(2)
N2	C11	H11	111.7	C12	C11	C16	100(2)
C12	C11	H11	112.2	C16	C11	H11	112.6
N2A	C11A	C12A	110(2)	N2A	C11A	C16A	107(2)
N2A	C11A	H11A	110.0	C12A	C11A	C16A	107(2)

atom	atom	atom	angle
C12A	C11A	H11A	110.0
C11	C12	C13	115(2)
C11	C12	H12'	108.2
C13	C12	H12'	108.2
C11A	C12A	C13A	117(2)
C11A	C12A	H12A	107.5
C13A	C12A	H12A	107.4
C12A	C13A	C14A	111(2)
C12A	C13A	H13A	105.2
C14A	C13A	H13A	106.3
C12	C13	C14	111(3)
C12	C13	H13	107.6
C14	C13	H13	106.6
C13	C14	H14'	110.5
C13	C14	H14	109.3
H14'	C14	H14	108.7
C13A	C14A	H14A	108.9
C13A	C14A	H14A"	109.0
H14A	C14A	H14A"	111.6
C13A	C15A	H15A	109.6
C13A	C15A	H15A"	109.9
H15A	C15A	H15A"	110.6
C13	C15	H15'	106.5
C13	C15	H15	109.9

atom	atom	atom	angle
C16A	C11A	H11A	110.4
C11	C12	H12	108.0
C13	C12	H12	106.9
H12	C12	H12'	110.2
C11A	C12A	H12A'	106.9
C13A	C12A	H12A'	107.1
H12A'	C12A	H12A	110.7
C12A	C13A	C15A	113(2)
C14A	C13A	C15A	112(3)
C15A	C13A	H13A	106.6
C12	C13	C15	112(3)
C14	C13	C15	110(3)
C15	C13	H13	108.1
C13	C14	H14"	110.5
H14'	C14	H14"	109.5
H14"	C14	H14	108.3
C13A	C14A	H14A'	107.4
H14A	C14A	H14A'	110.2
H14A'	C14A	H14A"	109.6
C13A	C15A	H15A'	108.3
H15A	C15A	H15A'	108.7
H15A'	C15A	H15A"	109.7
C13	C15	H15"	112.3
H15'	C15	H15"	108.6

atom	atom	atom	angle	atom	atom	atom	angle
H15'	C15	H15	106.0	H15"	C15	H15	113.2
O4	C16	N3	120(2)	O4	C16	C11	122(2)
N3	C16	C11	116(2)	O4A	C16A	N3A	128(2)
O4A	C16A	C11A	120(2)	N3A	C16A	C11A	110(2)
N3	C17	C18	112(2)	N3	C17	C20	115(2)
N3	C17	C22	106(2)	C18	C17	C20	108(2)
C18	C17	C22	108(2)	C20	C17	C22	104(2)
N3A	C17A	C18A	114(2)	N3A	C17A	C20A	110(2)
N3A	C17A	C22A	102(2)	C18A	C17A	C20A	110(2)
C18A	C17A	C22A	113(2)	C20A	C17A	C22A	104(2)
C17	C18	C19	118(2)	C17	C18	H18'	107.7
C17	C18	H18	107.0	C19	C18	H18'	107.6
C19	C18	H18	106.9	H18'	C18	H18	109.3
C17A	C18A	C19A	116(2)	C17A	C18A	H18A	109.1
C17A	C18A	H18A'	108.7	C19A	C18A	H18A	107.4
C19A	C18A	H18A'	106.8	H18A	C18A	H18A'	108.2
C18	C19	H19	109.8	C18	C19	H19'	109.6
C18	C19	H19"	110.5	H19	C19	H19'	108.3
H19	C19	H19"	109.3	H19'	C19	H19"	109.2
C18A	C19A	H19A'	108.7	C18A	C19A	H19A"	110.4
C18A	C19A	H19A	109.2	H19A'	C19A	H19A"	109.4
H19A'	C19A	H19A	109.1	H19A"	C19A	H19A	110.0
C17A	C20A	C21A	116(2)	C17A	C20A	H20A	107.4
C17A	C20A	H20A'	107.2	C21A	C20A	H20A	108.3

atom	atom	atom	angle	atom	atom	atom	angle
C21A	C20A	H20A'	108.3	H20A	C20A	H20A'	108.6
C17	C20	C21	113(2)	C17	C20	H20	107.6
C17	C20	H20'	107.8	C21	C20	H20	108.4
C21	C20	H20'	109.0	H20	C20	H20'	110.3
C20	C21	H21"	109.4	C20	C21	H21	110.0
C20	C21	H21'	110.2	H21"	C21	H21	109.2
H21"	C21	H21'	108.9	H21	C21	H21'	109.1
C20A	C21A	H21A	109.8	C20A	C21A	H21A'	109.1
C20A	C21A	H21A"	109.4	H21A	C21A	H21A'	109.3
H21A	C21A	H21A"	110.1	H21A'	C21A	H21A"	109.1
O5A	C22A	N4A	122(2)	O5A	C22A	C17A	121(2)
N4A	C22A	C17A	116(2)	O5	C22	N4	123(2)
O5	C22	C17	117(2)	N4	C22	C17	118(2)

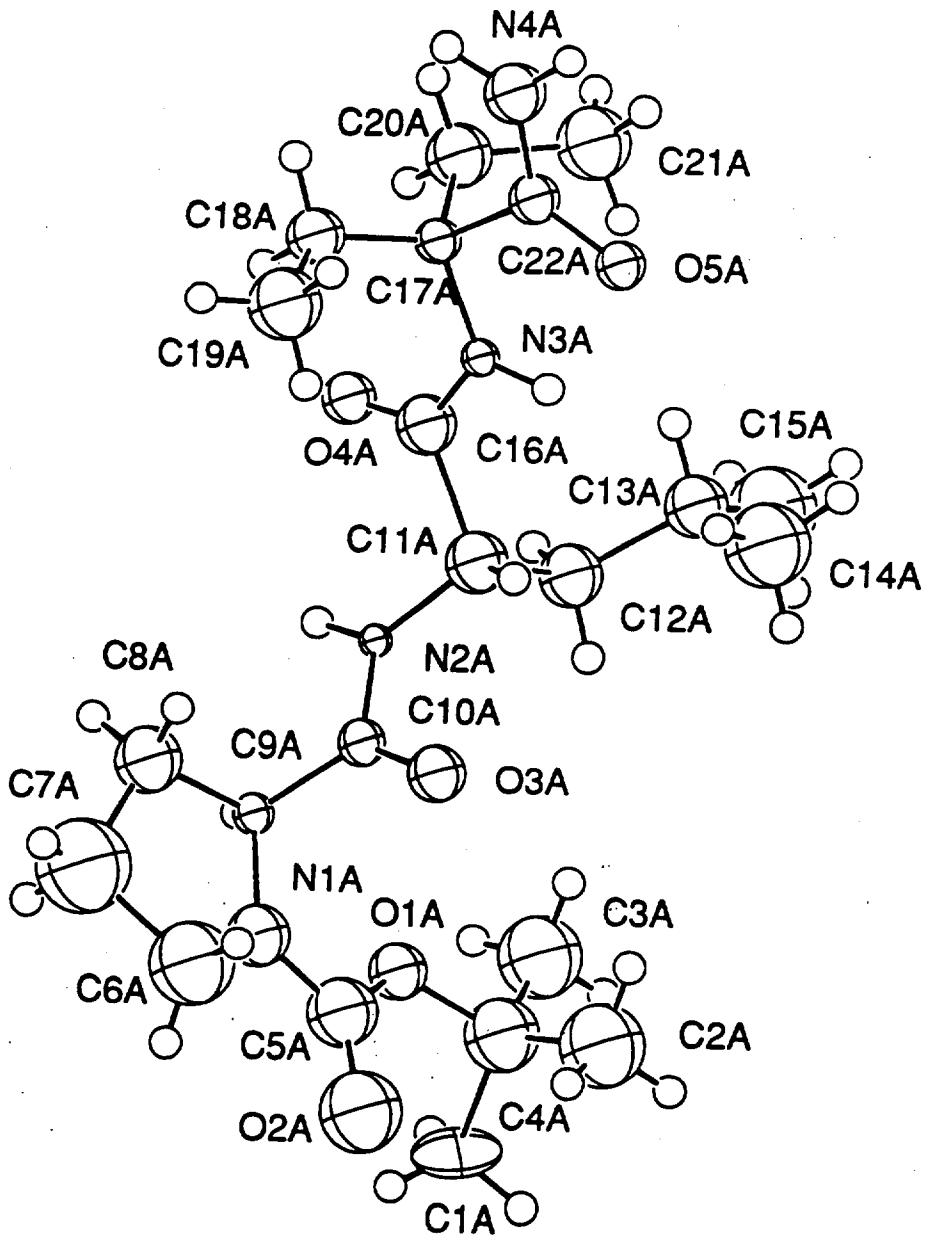

Figure 1. ORTEP representation of *tert*-butoxycarbonyl-L-prolyl-L-leucyl-diethylglycinamide (8aB) with crystallographic numbering system. Hydrogen atoms are drawn as spheres of arbitrary radius.

X-ray Crystallographic Data for 2-[3(*R*)-[[2(*S*)-*N*-Benzyloxycarbonyl-pyrrolidinylcarbonyl]amino]-2-oxo-pyrrolidin-1-yl]-cyclopropane-1-carboxamide (14b)

1)	Unit cell parameters	
	a (Å)	9.1704 (4)
	b (Å)	9.8152 (5)
	c (Å)	23.1792 (11)
	V $(Å)^3$	2086.3 (2)
	α, ß, γ (°)	90, 90, 90
2)	Formula	$C_{21}H_{26}N_4O_5$
	Formula weight	414.46
	Z	4
3)	Density, calculated (g/cc)	1.319
4)	Space group	$P2_12_12_1$
5)	Wavelength used (Å)	0.71073
	Observed reflections	3654
	Unobserved reflections	7060
6)	Data collection	Siemens SMART Paltform CCD
	Structure solution and refinement	SHELXTL-Plus V5.0, Siemens Industrial Automation, Inc., Madison, WI
7)	Final R value	0.0794
8)	Final difference map	featureless [0.184 $e^-/Å^3$(near S, -0.190 $e^-/Å^3$)]

Positional parameters (X 10^4) and U (eq) for 14b.
U (eq) is defined as one third of the trace orthoganalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
O(1)	-1405(2)	5387(2)	1923(1)	35(1)	1
N(1)	592(3)	5633(2)	1364(1)	34(1)	1
C(1)	-72(3)	5225(3)	1840(1)	27(1)	1
O(2)	3270(2)	6362(2)	2444(1)	41(1)	1
C(2)	828(3)	4545(3)	2292(1)	29(1)	1
N(2)	2331(2)	4296(2)	2159(1)	27(1)	1
N(3)	5622(2)	5566(2)	1653(1)	33(1)	1
O(3)	3662(2)	6080(2)	1107(1)	33(1)	1
C(3)	81(4)	3522(4)	2677(1)	49(1)	1
O(4)	4279(2)	7537(2)	-505(1)	42(1)	1
N(4)	5167(3)	7805(3)	400(1)	33(1)	1
C(4)	434(3)	4886(4)	2905(1)	44(1)	1
O(5)	5635(2)	5886(2)	-79(1)	32(1)	1
C(5)	3406(3)	5240(3)	2235(1)	30(1)	1
C(6)	2805(3)	3030(3)	1887(2)	42(1)	1
C(7)	4381(3)	3281(3)	1712(1)	40(1)	1
C(8)	4833(3)	4610(3)	2014(1)	32(1)	1
C(9)	4961(3)	6242(3)	1229(1)	30(1)	1
C(10)	5897(3)	7288(3)	914(1)	34(1)	1
C(11)	6067(4)	8588(4)	1270(1)	54(1)	1
C(12)	4678(4)	9371(4)	1135(2)	68(1)	1
C(13)	4416(4)	9098(3)	499(2)	53(1)	1
C(14)	4969(3)	7123(3)	-89(1)	30(1)	1
C(15)	5432(3)	5046(3)	-582(1)	40(1)	1
C(16)	4169(3)	4069(3)	-517(1)	30(1)	1
C(17)	3151(3)	4189(3)	-80(1)	37(1)	1
C(18)	2013(3)	3265(3)	-42(1)	41(1)	1
C(19)	1876(3)	2229(3)	-436(1)	43(1)	1
C(20)	2883(3)	2099(4)	-873(1)	45(1)	1
C(21)	4030(3)	3015(3)	-910(1)	38(1)	1

Anisotropic displacement parameters (Å2 X 10^3) for **14b**.
The anisotropic displacement factor exponent takes the form:
$-2\pi^2[(ha^*)^2 U_{11} + \ldots + 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
O(1)	16(1)	48(1)	40(1)	2(1)	0(1)	2(1)
N(1)	19(1)	52(2)	32(1)	10(1)	-2(1)	6(1)
C(1)	21(2)	29(2)	32(2)	-5(1)	-3(1)	0(1)
O(2)	32(1)	42(1)	48(1)	-15(1)	0(1)	-3(1)
C(2)	20(2)	34(2)	34(2)	4(1)	1(1)	3(1)
N(2)	18(1)	30(1)	33(1)	0(1)	0(1)	1(1)
N(3)	16(1)	47(2)	37(1)	1(1)	-5(1)	-1(1)
O(3)	16(1)	50(1)	32(1)	-1(1)	-2(1)	-2(1)
C(3)	29(2)	55(2)	64(2)	29(2)	8(2)	3(2)
O(4)	28(1)	57(2)	41(1)	17(1)	-7(1)	-7(1)
N(4)	24(1)	37(2)	37(1)	-2(1)	-1(1)	-1(1)
C(4)	30(2)	70(2)	31(2)	7(2)	5(1)	12(2)
O(5)	28(1)	35(1)	33(1)	-2(1)	-2(1)	-3(1)
C(5)	24(2)	40(2)	26(2)	2(1)	-5(1)	3(2)
C(6)	28(2)	39(2)	60(2)	-10(2)	5(2)	3(2)
C(7)	28(2)	40(2)	52(2)	1(2)	4(2)	10(2)
C(8)	20(2)	43(2)	34(2)	4(1)	-2(1)	3(1)
C(9)	20(2)	43(2)	28(2)	-9(1)	3(1)	-2(1)
C(10)	22(2)	46(2)	33(2)	-3(1)	2(1)	-6(1)
C(11)	54(2)	61(2)	47(2)	-15(2)	4(2)	-24(2)
C(12)	73(3)	46(2)	84(3)	-26(2)	16(2)	-6(2)
C(13)	40(2)	38(2)	80(3)	-6(2)	5(2)	2(2)
C(14)	14(1)	38(2)	39(2)	9(2)	4(1)	-8(2)
C(15)	35(2)	50(2)	34(2)	-10(2)	4(1)	-10(2)
C(16)	24(2)	37(2)	28(1)	1(1)	-4(1)	-1(1)
C(17)	32(2)	43(2)	35(2)	-6(2)	0(1)	-6(2)
C(18)	31(2)	58(2)	36(2)	4(2)	4(2)	-7(2)
C(19)	33(2)	49(2)	47(2)	-1(2)	-3(2)	-16(2)
C(20)	43(2)	45(2)	46(2)	-11(2)	1(2)	-6(2)
C(21)	27(2)	48(2)	40(2)	-6(2)	6(1)	1(2)

Intramolecular distances (Å) and bond angles (°) for 14b.

O(1)-C(1)	1.248(3)	N(1)-C(1)	1.322(3)
C(1)-C(2)	1.492(4)	O(2)-C(5)	1.209(3)
C(2)-N(2)	1.434(3)	C(2)-C(4)	1.503(4)
C(2)-C(3)	1.507(4)	N(2)-C(5)	1.364(4)
N(2)-C(6)	1.459(4)	N(3)-C(9)	1.332(3)
N(3)-C(8)	1.450(3)	O(3)-C(9)	1.235(3)
C(3)-C(4)	1.475(5)	O(4)-C(14)	1.224(3)
N(4)-C(14)	1.328(4)	N(4)-C(10)	1.458(4)
N(4)-C(13)	1.462(4)	O(5)-C(14)	1.359(3)
O(5)-C(15)	1.440(3)	C(5)-C(8)	1.536(4)
C(6)-C(7)	1.521(4)	C(7)-C(8)	1.538(4)
C(9)-C(10)	1.525(4)	C(10)-C(11)	1.527(4)
C(11)-C(12)	1.520(5)	C(12)-C(13)	1.516(5)
C(15)-C(16)	1.511(4)	C(16)-C(17)	1.382(4)
C(16)-C(21)	1.384(4)	C(17)-C(18)	1.385(4)
C(18)-C(19)	1.373(4)	C(19)-C(20)	1.377(4)
C(20)-C(21)	1.387(4)		

O(1)-C(1)-N(1)	122.8(3)	O(1)-C(1)-C(2)	119.3(3)
N(1)-C(1)-C(2)	117.9(2)	N(2)-C(2)-C(1)	117.1(2)
N(2)-C(2)-C(4)	118.2(2)	C(1)-C(2)-C(4)	115.6(2)
N(2)-C(2)-C(3)	116.8(2)	C(1)-C(2)-C(3)	117.5(3)
C(4)-C(2)-C(3)	58.7(2)	C(5)-N(2)-C(2)	123.4(2)
C(5)-N(2)-C(6)	114.8(2)	C(2)-N(2)-C(6)	121.6(2)
C(9)-N(3)-C(8)	121.4(2)	C(4)-C(3)-C(2)	60.5(2)
C(14)-N(4)-C(10)	125.8(3)	C(14)-N(4)-C(13)	120.5(3)
C(10)-N(4)-C(13)	112.9(2)	C(3)-C(4)-C(2)	60.8(2)
C(14)-O(5)-C(15)	116.1(2)	O(2)-C(5)-N(2)	126.6(3)
O(2)-C(5)-C(8)	126.0(3)	N(2)-C(5)-C(8)	107.4(2)
N(2)-C(6)-C(7)	105.1(2)	C(6)-C(7)-C(8)	105.8(2)
N(3)-C(8)-C(5)	110.9(2)	N(3)-C(8)-C(7)	114.9(2)
C(5)-C(8)-C(7)	105.3(2)	O(3)-C(9)-N(3)	123.0(3)
O(3)-C(9)-C(10)	121.3(3)	N(3)-C(9)-C(10)	115.7(2)
N(4)-C(10)-C(9)	111.6(2)	N(4)-C(10)-C(11)	101.4(2)
C(9)-C(10)-C(11)	111.2(2)	C(12)-C(11)-C(10)	103.0(3)
C(13)-C(12)-C(11)	104.1(3)	N(4)-C(13)-C(12)	103.4(3)
O(4)-C(14)-N(4)	125.2(3)	O(4)-C(14)-O(5)	122.8(3)
N(4)-C(14)-O(5)	112.0(2)	O(5)-C(15)-C(16)	112.4(2)
C(17)-C(16)-C(21)	118.9(3)	C(17)-C(16)-C(15)	122.4(3)
C(21)-C(16)-C(15)	118.7(2)	C(16)-C(17)-C(18)	120.0(3)
C(19)-C(18)-C(17)	120.7(3)	C(18)-C(19)-C(20)	119.8(3)
C(19)-C(20)-C(21)	119.6(3)	C(16)-C(21)-C(20)	121.0(3)

Symmetry transformations used to generate equivalent atoms:

NMR Data for the Synthetic Intermediates of 3a-3d and 4a-4d.

***N-tert*-Butoxycarbonyl-diethylglycinamide (6a).** ^{1}H NMR ($CDCl_3$) δ 6.19 (br s, 2 H, $CONH_2$), 5.63 (br s, 1 H, Boc-NH), 2.23-2.30 (m, 2 H, Deg ß-CH_2), 1.56-1.69 (m, 2 H, Deg ß-CH_2), 1.40 (s, 9 H, $C(CH_3)_3$), 0.77-0.82 (t, J = 7.21, 6 H, Deg γ-CH_3); ^{13}C NMR ($CDCl_3$) δ 8.63 (Deg γ-C), 29.00 ($C(CH_3)_3$), 29.11 (Deg ß-C), 64.41 (Deg α-C), 79.86 ($C(CH_3)_3$), 154.83 (Boc-CO), 176.87 ($CONH_2$).

***N-tert*-Butoxycarbonyl-L-leucyl-diethylglycinamide (7a).** ^{1}H NMR (DMSO) δ 7.59 (s, 1 H, CONH), 7.41-7.46 (m, 2 H, CONH, Boc-NH), 7.34 (s, 1 H, CONH), 3.68-3.70 (m, 1 H, Leu α-CH), 2.26-2.35 (m, 2 H, Deg ß-CH_2), 1.57-1.66 (m, 4 H, Leu ß-CH_2, Deg ß-CH_2), 1.38-1.46 (m, 1 H, Leu γ-H), 1.36 (s, 9 H, $C(CH_3)_3$), 0.83, 0.79 (dd, J = 12.9 Hz, 6 H, Leu δ-CH_3), 0.59 (t, J = 7.2 Hz, 6 H, Deg γ-CH_3); ^{13}C NMR (DMSO) δ 8.45, 8.22 (Deg γ-C), 23.48, 21.66 (Leu δ-C), 24.84 (Deg ß-C), 27.74 (Leu γ-C), 27.95 (Leu ß-C) 28.62 ($C(CH_3)_3$), 54.74 (Leu α-C), 64.16 (Deg α-C), 78.6 ($C(CH_3)_3$), 155.94 (Boc-CO), 171.44 (CONH), 174.96 ($CONH_2$).

***N-tert*-Butoxycarabonyl-L-proline-L-leucine-diethylglycinamide (8a).** ^{1}H NMR ($CDCl_3$) δ 7.16-7.10 (m, 2 H, CONH), 6.12 (br s, 1 H, CONH), 5.67 (br s, 1 H, CONH), 4.49-4.24 (m, 2 H, Pro α-CH, Leu α-CH), 3.44-3.12 (m, 2 H, Pro δ-CH_2), 2.22-2.22 (m, 3H, Leu ß-CH_2, Pro ß-CH), 2.08-2.01 (m, 1 H, Pro ß-CH), 1.95-1.83 (m, 2H, Pro γ-CH_2), 1.81-1.52 (m, 5 H, Leu γ-CH, Deg ß-CH_2), 1.45 (s, 9 H, $C(CH_3)_3$), 0.94 (dd, J= 6.8, 6 H, Leu-CH_3), 0.83 & 0.79 (tt, J= 3.6, 6 H, Deg-CH_3); ^{13}C NMR ($CDCl_3$) δ 8.37 & 8.83 (Deg γ-C), 22.26 & 22.30 (Leu δ–C), 23.68 (Pro γ-C), 25.33 (Leu γ-C), 25.36 (Pro ß-C), 25.57 (Leu ß-C), 28.62 & 29.28 (Deg ß-C), 28.93 ($C(CH_3)_3$), 47.88 (Pro δ-C), 54.08 (Leu α-C), 60.79 (Pro α-C), 64.92 (Deg α-C), 81.41 ($C(CH_3)_3$), 156.56 (Boc-CO), 171.88 (CONH), 173.59 (CONH), 176.56 ($CONH_2$).

***N*-Benzyloxycarbonyl-L-prolyl-L-leucyl-1-aminocyclopropanecarboxamide (8b).** ^{1}H NMR ($CDCl_3$, rotamers) δ 7.28-7.34 (m, 5 H, C_6H_5), 7.05-7.08 (m, 1 H, CONH), 6.83-6.86 (m, 1 H, CONH), 4.99-5.22 (m, 2 H, $CONH_2$), 4.69-4.75 (m, 1 H, Pro α-CH), 4.66 (s, 2 H, Cbz-CH_2), 4.25-4.31 (m, 1 H, Leu α-CH), 3.33-3.66 (m, 4 H, Pro δ-CH_2, Leu ß-CH_2), 2.01-2.13 (m, 1 H, Pro ß-CH), 1.76-1.95 (m, 4 H, Leu γ-CH, Pro ß-CH and γ-CH_2), 0.95-1.69 (m, 4 H, Ac_3c ß-CH_2), 0.79-0.92 (m, 6H, Leu δ-CH_3); ^{13}C NMR ($CDCl_3$, rotamers) δ 22.42, 22.65 (Ac_3c γ-C), 24.02, 24.75 (Leu δ-C), 25.34, 25.44, (Ac_3c γ-C), 26.64 (Pro γ-C), 29.88 (Leu γ-C), 31.93 (Leu ß-C), 42.51 (Ac_3c α-C), 46.96, 46.37 (Pro ß-C), 47.71, 48.20 (Pro δ-C), 50.01, 50.04 (Leu α-C), 61.22, 61.66 (Pro α-C), 65.55, 67.75 (Cbz-CH_2), 127.56, 127.96, 129.04, 129.17, 136.25, 141.96 (C_6H_5), 155.48, 156.21 (Cbz-CO), 171.39, 172.32 (CONH), 172.85 ($CONH_2$).

***N*-Benzyloxycarbonyl-L-prolyl-L-leucyl-1-aminocyclopentanecarboxamide (8c).** ^{1}H NMR ($CDCl_3$) δ 7.29-7.41 (m, 5 H, C_6H_5), 7.02 (br s, 1 H, CONH), 6.91 (br s, 1 H, CONH), 6.78 (br s, 1 H, CONH), 5.53 (br s, 1 H, CONH), 5.14 (s, 2 H, Cbz-CH_2), 4.30, 4.28 (dd, J = 5.1 & 6.1 Hz, 1 H, Pro α-CH), 4.09-4.14 (m, 1 H, Leu α-CH), 3.51-3.56 (m, 2 H, Pro δ-CH_2), 1.92-2.33 (m, 9 H, Ac_5c γ-CH_2, Pro ß-CH_2, Leu ß-CH_2 and γ-CH), 1.49-1.75 (m, 6 H, Ac_5c ß-CH_2, Pro γ-CH_2), 0.92, 0.86 (2d, J = 5.4 Hz, 6 H, Leu δ- CH_3); ^{13}C NMR ($CDCl_3$) δ 22.39, 23.58 (Ac_5c γ-C), 25.01 (Leu δ-C), 25.42, 25.78 (Ac_5c ß-C), 30.22 (Pro γ-C), 37.46 (Leu γ-C), 37.68 (Leu ß-C), 40.07 (Pro ß-C), 47.91 (Pro δ-C), 54.01 (Leu α-C), 61.94 (Pro α-C), 67.75 (Cbz-CH_2), 68.30 (Ac_5c α-C), 128.57, 129.06, 129.30, 136.77 (C_6H_5), 156.80 (Cbz-CO), 172.41, 173.62 (CONH), 177.62 ($CONH_2$).

***N*-Benzyloxycarbonyl-L-prolyl-L-leucyl-1-aminocyclohexanecarboxamide (8d).** ^{1}H NMR ($CDCl_3$) δ 7.51 (br s, 1 H, CONH), 7.32-7.34 (m, 5 H, C_6H_5), 6.84-6.90 (m, 2 H, $CONH_2$), 5.44 (br s, 1 H, CONH), 5.13 (s, 2 H, Cbz-CH_2), 4.30-4.34 (m, 1 H, Pro α-CH), 4.16-4.19 (m, 1 H, Leu α-CH), 3.47-3.58 (m, 2 H, Pro δ-CH_2), 2.04-2.20 (m, 4 H, Pro ß-CH_2, Leu ß-CH_2), 1.70-1.97 (m, 5 H, Leu γ-CH, Ac_6c δ-CH_2,

Pro γ-CH_2), 1.58-1.65 (m, 4 H, Ac_6c γ-CH_2), 1.26-1.39 (m, 4 H, Ac_6c ß-H), 0.92, 0.87 (dd, J = 6.0 Hz, 6 H, Leu δ-CH_3); ^{13}C NMR ($CDCl_3$) δ 22.11 (Ac_6c δ-C), 23.51, 25.41 (Ac_6c γ-C), 25.75, 25.86 (Ac_6c ß-C), 22.03, 22.45 (Leu δ-C), 30.07 (Leu γ-C), 32.22 (Pro γ-C), 32.72 (Leu ß-C), 40.11 (Pro ß-C), 47.82 (Pro δ-C), 54.46 (Leu α-C), 60.82 (Ac_6c α-C), 61.76 (Pro α-C), 68.18 (Cbz-CH_2), 128.49, 128.98, 129.27, 136.83 (C_6H_5), 156.76 (Cbz-CO), 172.38, 173.82 (CONH), 178.10 ($CONH_2$).

***N-tert*-Butoxycarbonyl-D-methionyl-1-aminocyclopropane-1-carboxylic Acid Methyl Ester (9b).** ^1H NMR ($CDCl_3$) δ 7.14 (br s, 1 H, CONH), 5.35 (br s, 1 H, CONH), 4.25-4.28 (m, 1 H, Met α-CH), 3.64 (s, 3 H, OCH_3), 2.54 (t, J = 5.8 Hz, 2 H, Met γ-CH_2), 1.86-2.08 (m, 2 H, Met ß-CH_2), 2.03 (s, 3 H, Met-CH_3), 1.55 (t, J = 6.6 Hz, 2 H, Ac_3c ß-CH_2), 1.40 (s, 9 H, $C(CH_3)_3$), 1.09 (t, J = 6.6 Hz, 2 H, Ac_3c ß-CH_2); ^{13}C NMR ($CDCl_3$) δ 15.83, 18.04, (Ac_3c ß-C), 18.35 (Met-CH_3), 28.93 ($C(CH_3)_3$), 30.43 (Ac_3c α-C), 32.39 (Met γ-C), 33.95 (Met ß-C), 53.13 (OCH_3), 53.68 (Met α-C), 80.72 ($C(CH_3)_3$), 156.31 (Boc-CO); 173.15 ($COOCH_3$), 173.42 (CONH).

***N-tert*-Butoxycarbonyl-D-methionyl-1-aminocyclopentanecarboxamide (9c).** ^1H NMR ($CDCl_3$) δ 7.04 (br s, 1 H, CONH), 6.98 (br s, 1 H, CONH), 5.84 (br s, 1 H, CONH), 5.73 (br s, 1 H, CONH), 4.11 (q, J = 6.9 Hz, 1 H, Met α-CH), 3.11-3.21 (m, 2 H, Met γ-CH_2) 2.52 (t, J = 6.6 Hz, 2 H, Met ß-CH_2), 1.72-2.33 (m, 8 H, Ac_5c -H), 2.07 (s, 3 H, Met-CH_3), 1.40 (s, 9 H, $C(CH_3)_3$); ^{13}C NMR ($CDCl_3$) δ 24.86 (Ac_5c γ-C), 27.17 (Ac_5c ß-C), 28.51 ($C(CH_3)_3$), 36.62 (Met-CH_3), 38.46 (Met γ-C), 40.07 (Met ß-C), 46.94 (Met α-C), 55.05 (Ac_5c α-C), 81.34 ($C(CH_3)_3$), 155.12 (Boc-CO); 173.03 (CONH), 177.64 ($CONH_2$).

***N-tert*-Butoxycarbonyl-D-methionyl-1-aminocyclohexanecarboxamide (9d).** ^1H NMR ($CDCl_3$) δ 7.00 (br s, 1 H, CONH), 6.98 (br s, 1 H, CONH), 6.40 (br s, 1 H, CONH), 5.68 (br s, 1 H, CONH), 4.11 (m, 1 H, Met α-CH), 3.11-3.24 (m, 2 H, Met γ-CH_2), 2.55-2.60 (m, 2 H, Met ß-CH_2), 1.94-2.16 (m, 5 H, Met-CH_3, Ac_6c δ-CH_2), 1.55-1.70 (m, 6 H, Ac_6c γ-CH_2 and ß-CH_2), 1.31-1.38 (m, 2 H, Ac_6c ß-CH_2), 1.42 (s, 9 H,

$C(CH_3)_3$); ^{13}C NMR ($CDCl_3$) δ 15.91 (Ac_6c δ-C), 21.96 (Ac_6c γ-C), 25.75 (Ac_6c ß-C), 28.90 ($C(CH_3)_3$), 31.03 (Met-CH_3), 31.53 (Met γ-C), 33.53 (Met ß-C), 55.12 (Met α-C), 60.86 (Ac_6c α-C), 81.34 ($C(CH_3)_3$), 157.06 (Boc-CO); 172.41 (CONH), 177.78 ($CONH_2$).

1-[3(*R*)-[*N-tert*-Butoxycarbonylamino]-2-oxo-pyrrolidin-1-yl]-2-ethyl-butanamide (10a). ^1H NMR ($CDCl_3$) δ 6.90 (s, 1 H, CONH), 6.08 (d, J = 4.8, 1 H, CONH), 6.02 (s, 1 H, CONH), 4.11-4.08 (m, 1 H, lactam 3-CH), 3.51-3.52 (m, 1 H, lactam 5-CH), 3.37-3.40 (m, 1 H, lactam 5-CH), 2.37-2.41 (m, 1 H, lactam 4-CH), 2.02-2.09 (m, 1 H, lactam 4-CH), 1.84-1.98 (m, 2 H, Deg ß-CH_2), 1.76-1.81 (m, 2 H, Deg ß-CH_2), 1.30 (s, 9 H, $C(CH_3)_3$), 0.68-0.78 (m, 6 H, Deg γ-CH_3); ^{13}C NMR ($CDCl_3$) δ 8.52 (Deg γ-C), 24.13 (Deg ß-C), 25.57 (lactam 4-C), 28.93 ($C(CH_3)_3$), 44.19 (lactam 5-C), 53.28 (lactam 3-C), 66.68 (Deg α-C), 80.02 ($C(CH_3)_3$), 156.67 (CONH), 174.04 (lactam-CO), 176.45 ($CONH_2$).

1-[3(*R*)-[*N-tert*-Butoxycarbonylamino]-2-oxo-pyrrolidin-1-yl]-cyclopropane-1-carboxylic Acid Methyl Ester (10b). ^1H NMR ($CDCl_3$) δ 5.14 (s, 1 H, CONH), 4.12-4.18 (m, 1 H, lactam 3-CH), 3.63 (s, 3 H, OCH_3), 3.33-3.42 (m, 1 H, lactam 5-CH), 3.22-3.28 (m, 1 H, lactam 5-CH), 2.51-2.60 (m, 1 H, lactam 4-CH), 1.77-1.84 (m, 1 H, lactam 4-CH), 1.84-1.98 (m, 2 H, Ac_3c ß-CH_2), 1.39 (s, 9 H, $C(CH_3)_3$), 1.09-1.32 (m, 2 H, Ac_3c ß-CH_2); ^{13}C NMR ($CDCl_3$) δ 16.70, 18.23 (Ac_3c ß-C), 28.93 ($C(CH_3)_3$), 29.08 (lactam 4-C), 36.88 (Ac_3c α-C), 45.23 (lactam 5-C), 53.26 (OCH_3), 53.44 (lactam 3-C), 80.42 ($C(CH_3)_3$), 156.44 (CONH), 172.38 ($COOCH_3$), 174.43 (lactam-CO).

1-[3(*R*)-[*N-tert*-Butoxycarbonylamino]-2-oxo-pyrrolidin-1-yl]-cyclopentane-1-carboxamide (10c). ^1H NMR ($CDCl_3$) δ 7.15 (br s, 1 H, CONH), 5.25 (br s, 2 H, $CONH_2$), 4.11 (m, 1 H, lactam 3-CH), 3.53 (t, J = 8.7, 1 H, lactam 5-CH), 3.39-3.48 (m, 1 H, lactam 5-CH), 2.45-2.55 (m, 2 H, lactam 4-CH_2), 2.26-2.33 (m, 2 H, Ac_5c γ-CH_2), 2.00-2.09 (m, 2 H, Ac_5c γ-CH_2), 1.62-1.83 (m, 4 H, Ac_5c ß-CH_2),

1.42 (s, 9 H, $C(CH_3)_3$); ^{13}C NMR ($CDCl_3$) δ 24.54, 24.70 (Ac_5c γ-C), 28.90 ($C(CH_3)_3$), 29.11 (Ac_5c ß-C), 35.95 (Ac_5c ß-C), 36.47 (lactam 4-C), 43.63 (lactam 5-C), 53.10 (lactam 3-C), 69.77 (Ac_5c α-C), 80.27 ($C(CH_3)_3$), 156.44 (Boc-CO); 174.08 (CONH), 174.49 ($CONH_2$).

1-[3(*R*)-[*N-tert*-Butoxycarbonylamino]-2-oxo-pyrrolidin-1-yl]-cyclohexane -1-carboxamide (10d). ^1H NMR ($CDCl_3$) δ 6.94 (br s, 1 H, CONH), 5.48 (br s, 1 H, CONH), 5.39 (br s, 1 H, CONH), 4.02-4.07 (m, 1 H, lactam 3-CH), 3.43-3.45 (m, 2 H, lactam 5-CH_2), 2.63-2.66 (m, 1 H, lactam 4-CH), 2.43-2.49 (m, 1 H, lactam 4-CH), 1.73-2.03 (m, 6 H, Ac_6c δ-CH_2 and γ-CH_2), 1.36-1.63 (m, 4 H, Ac_6c ß-CH_2), 1.40 (s, 9 H, $C(CH_3)_3$); ^{13}C NMR ($CDCl_3$) δ 23.05 (Ac_6c δ-C), 25.70 (Ac_6c γ-C), 28.96 ($C(CH_3)_3$), 30.33 (Ac_6c ß-C), 37.14 (lactam 4-C), 43.62 (lactam 5-C), 53.91 (lactam 3-C), 65.27 (Ac_6c α-C), 80.82 ($C(CH_3)_3$), 163.21 (Boc-CO), 174.60 (CONH), 176.26 ($CONH_2$).

2-[3(*R*)-[[2(*S*)-*N-tert*-Butoxycarbonyl-pyrrolidinyl carbonyl]amino]-2-oxo-pyrrolidin-1-yl]-2-ethyl-butanamide (11a). ^1H NMR ($CDCl_3$, 50 °C) δ 7.24 (br s, 1 H, CONH), 6.21 (br s, 2 H, $CONH_2$), 4.15-4.23 (m, 2 H, Pro α-CH, lactam 3-CH), 3.58-3.65 (m, 1 H, lactam 5-CH), 3.38-3.51 (m, 3H, lactam 5-CH, Pro δ-CH_2), 2.48-2.52 (m, 1 H, lactam 4-CH), 2.15-2.22 (m, 1 H, lactam 4-CH), 1.75-2.04 (m, 8 H, Pro γ-CH_2 and ß-CH_2, Deg ß-CH_2), 1.44 (s, 9 H, $C(CH_3)_3$), 0.85, 0.79 (2t, J = 7.2 Hz, 6H, Deg γ-CH_3); ^{13}C NMR ($CDCl_3$, 50 °C) δ 8.47, 8.62 (Deg γ-C), 24.49 (Deg ß-C), 24.72 (Pro γ-C), 25.77 (lactam 4-C), 27.17 (Pro ß-C), 29.00 ($C(CH_3)_3$), 44.43 (lactam 5-C), 47.68 (Pro δ-C), 52.94 (lactam 3-C), 60.93 (Pro α-C), 67.28 (Deg α-C), 80.82 ($C(CH_3)_3$), 155.02 (Boc-CO), 173.14 (CONH), 174.04 (lactam-CO), 175.86 ($CONH_2$).

2-[3(*R*)-[[2(*S*)-*N-tert*-Butoxycarbonyl-pyrrolidinylcarbonyl]amino]-2-oxo-pyrrolidin-1-yl]-cyclopropane-1-carboxylic Acid Methyl Ester (11b). ^1H NMR ($CDCl_3$, 50 °C) δ 6.80 (br s, 1 H, CONH), 4.36-4.47 (m, 1 H, Pro α-CH), 4.27-4.44 (m, 1H, lactam 3-CH), 3.61 (s, 3H, OCH_3), 3.40-3.48 (m, 2 H, Pro δ-CH_2), 3.28-3.35 (m, 2H, lactam 5-CH_2), 2.62-2.71 (m, 1 H, lactam 4-CH), 2.22-2.23 (m, 1 H,

lactam 4-CH), 1.77-1.99 (m, 4 H, Pro γ-CH_2 and ß-CH_2), 1.52-1.68 (m, 2H, Ac_3c ß-CH_2), 1.46 (s, 9 H, $C(CH_3)_3$), 1.15-1.38 (m, 2H, Ac_3c ß-CH_2); ^{13}C NMR ($CDCl_3$, 50 °C) δ 16.64, 17.99 (Ac_3c ß-C), 24.72 (Pro γ-C), 28.67 (Pro ß-C), 29.00 ($C(CH_3)_3$), 29.05 (lactam 4-C), 36.96 (Ac_3c α-C), 45.33 (lactam 5-C), 47.71 (Pro δ-C), 52.41 (OCH_3), 53.15 (lactam 3-C), 61.26 (Pro α-C), 81.07 ($C(CH_3)_3$), 155.67 (Boc-CO), 172.29 ($COOCH_3$), 173.61 (CONH), 174.10 (lactam-CO).

2-[3(*R*)-[[2(*S*)-*N*-Benzyloxycarbonyl-pyrrolidinylcarbonyl]amino]-2-oxo-pyrrolidin-1-yl]-cyclopentane-1-carboxamide (11c). ^1H NMR ($CDCl_3$, rotamers) δ 7.67 (br s, 1 H, CONH), 7.28-7.37 (m, 5 H, C_6H_5), 7.07 (br s, 1 H, CONH), 5.84 (br s, 1 H, CONH), 5.18, 5.02 (s, rotomers, 2 H, Cbz-CH_2), 4.20-4.28 (m, 1 H, Pro α-CH), 4.06-4.14 (m 1 H, lactam 3-CH), 3.41-3.49 (m, 4 H, Pro δ-CH_2, lactam 5-CH_2), 1.50-2.50 (m, 14 H, Ac_5c -H, Pro γ-CH_2 and ß-CH_2, lactam 4-CH_2); ^{13}C NMR ($CDCl_3$, rotamers) δ 24.33, 24.49, 24.51, 24.65 (Ac_5c γ-C), 25.18, 25.89 (Ac_5c ß-C), 29.63 (Pro γ-C), 31.87 (Pro ß-C), 36.34, 35.83 (lactam 4-C), 44.50 (Pro δ-C), 48.14, 47.73 (lactam 5-C), 53.14, 52.80 (lactam 3-C), 60.09, 61.12 (Pro α-C), 67.86 (Cbz-CH_2), 71.64 (Ac_5c α-C), 137.07, 129.12, 128.70, 128.44 (C_6H_5), 155.52, 156.44 (Cbz-CO), 173.42 (lactam-CO), 176.74, 176.61 ($CONH_2$).

2-[3(*R*)-[[2(*S*)-*N*-Benzyloxycarbonyl-pyrrolidinylcarbonyl]amino]-2-oxo-pyrrolidin-1-yl]-cyclohexane-1-carboxamide (11d). ^1H NMR ($CDCl_3$, rotamers) δ 7.67, 7.37 (br s, 1 H, CONH), 7.28 (m, 5 H, C_6H_5), 7.13, 7.07 (br s, 1 H, CONH), 5.93 (br s, 1 H, CONH), 5.15, 5.11, 5.04, 5.00 (s, 2 H, Cbz-CH_2), 4.11-4.30 (m, 1 H, Pro α-CH), 4.13 (q, J = 6.6 Hz, 1 H, lactam 3-CH), 3.36-3.45 (m, 4 H, Pro γ-CH_2, lactam 5-CH_2), 2.69-2.72 (m, 1 H, Pro ß-CH), 1.78-2.12 (m, 9 H, Pro ß-CH and γ-CH_2, lactam 4-CH_2, Ac_6c δ-CH_2 and γ-CH_2), 1.50-1.60 (m, 2 H, Ac_6c ß-CH_2), 1.20-1.29 (m, 2 H, Ac_6c ß-CH_2); ^{13}C NMR ($CDCl_3$, rotamers) δ 23.01, 24.33 (Ac_6c ß-C), 25.09, 25.71 (Ac_6c γ-C), 29.63 (Ac_6c δ-C), 31.92 (Pro γ-C), 32.96 (lactam 4-C), 43.91 (Pro ß-C), 47.74 (lactam 5-C), 48.14 (Pro δ-C), 53.00, 53.36 (lactam 3-C), 60.94, 61.17 (Pro α-C),

65.45 (Ac_6c α-C), 67.86 (Cbz-CH_2), 128.47, 128.70, 129.12, 137.04 (C_6H_5), 155.57, 156.49 (Cbz-CO); 173.34, 174.03 (CONH), 176.53, 176.57, 176.70 ($CONH_2$).

2-[3(*R*)-[[2(*S*)-*N*-Benzyloxycarbonyl-pyrrolidinylcarbonyl]amino]-2-oxo-pyrrolidin-1-yl]-cyclopropane-1-carboxamide (14b). ^1H NMR ($CDCl_3$, rotamers) δ 7.82, 7.74 (br s, 1 H, CONH), 7.64, 7.46 (br s, 1 H, CONH), 7.33 (m, 5 H, C_6H_5), 5.81, 5.75 (br s, 1 H, CONH), 5.05, 5.20 (s, 2 H, Cbz-CH_2), 4.26 (m, 1 H, Pro α-CH), 3.80-3.87 (m, 1 H, lactam 3-CH), 3.27-3.70 (m, 4 H, Pro δ-CH_2, lactam 5-CH_2), 2.08-2.25 (m, 3 H, Pro ß-CH_2, lactam 4-CH), 1.81-1.96 (m, 3 H, Pro γ-CH_2, lactam 4-CH), 1.59-1.67 (m, 1 H, Ac_3c ß-CH), 1.48-1.55 (m, 1 H, Ac_3c ß-CH), 1.23-1.29 (m, 1 H, Ac_3c -CH), 0.87-0.94 (m, 1 H, Ac_3c -CH); ^{13}C NMR ($CDCl_3$, rotamers) δ 14.75, 18.70 (Ac_3c ß-C), 25.03, 25.27 (Pro γ-C), 29.21 (lactam 4-C), 31.62 (Pro ß-C), 38.00 (Ac_3c α-C), 45.90 (lactam 5-C), 47.74, 48.12 (Pro δ-C), 52.85, 53.16 (lactam 3-C), 60.70, 60.81 (Pro α-C), 67.95 (Cbz-CH_2), 128.45, 128.76, 129.15, 137.04 (C_6H_5), 156.57, 156.60 (Cbz-CO), 173.20, 173.47 (CONH), 173.74, 173.88 ($CONH_2$).