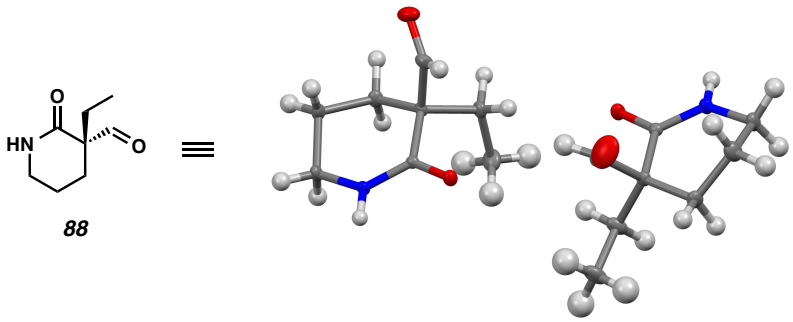
APPENDIX 3

X-Ray Crystallographic Data Relevant to Chapter 3

X-Ray Structure Determination

Low-temperature diffraction data (ϕ-and ω-scans) were collected on a Bruker Kappa diffractometer coupled to a Apex II CCD detector with graphite monochromated Mo K_α radiation (λ = 0.71073 Å) for the structure of compound **88** and (±)-**84**. The structure was solved by direct methods using SHELXS[1] and refined against F^2 on all data by full-matrix least squares with SHELXL-2013[2] using established refinement techniques.[3] All non-hydrogen atoms were refined anisotropically. Unless otherwise noted, all hydrogen atoms were included into the model at geometrically calculated positions and refined using a riding model. The isotropic displacement parameters of all hydrogen atoms were fixed to 1.2 times the U value of the atoms they are linked to (1.5 times for methyl groups).



88

Compound **88** crystallizes in the monoclinic space group $P2_1$ with two molecules in the asymmetric unit. The coordinates for the hydrogen atoms bound to N1 and N2 were located in the difference Fourier synthesis and refined semi-freely with the help of a distance restraint. The N-H distances were restrained to be 0.88(4) Å. The absolute configuration was determined during the synthetic procedure and is consistent with the

[1] Sheldrick, G. M. *Acta Cryst*. **1990**, A46, 467–473.
[2] Sheldrick, G. M. *Acta Cryst*. **2008**, A64, 112–122.
[3] Müller, P. *Crystallography Reviews* **2009**, *15*, 57–83.

diffraction data. Bayesian statistics P2(true) 0.977, P3(true).... 0.680, P3(rac-twin) 0.304, P3(false) .. 0.016.[4]

*Table A3.1 Crystal data and structure refinement for **88**.*

Identification code	A14103	
CCDC Deposition Number	1000826	
Empirical formula	C8 H13 N O2	
Formula weight	155.19	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21	
Unit cell dimensions	a = 8.1906(13) Å	α = 90°.
	b = 10.4809(17) Å	β = 110.678(7)°.
	c = 10.3279(16) Å	Y = 90°.
Volume	829.5(2) $Å^3$	
Z	4	
Density (calculated)	1.243 Mg/m^3	
Absorption coefficient	0.089 mm^{-1}	
F(000)	336	
Crystal size	0.700 x 0.070 x 0.050 mm^3	
Theta range for data collection	2.108 to 36.313°.	
Index ranges	-13<=h<=13, -17<=k<=17, -17<=l<=17	
Reflections collected	39923	
Independent reflections	8043 [R(int) = 0.0504]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7474 and 0.6748	
Refinement method	Full-matrix least-squares on F^2	

[4] Hoft, R.; Straver, L.; Spek, A. *J. Appl. Cryst.* **2008**, *41*, 96–103.

Data / restraints / parameters	8043 / 3 / 207
Goodness-of-fit on F^2	1.034
Final R indices [I>2sigma(I)]	R1 = 0.0436, wR2 = 0.1030
R indices (all data)	R1 = 0.0578, wR2 = 0.1104
Absolute structure parameter	0.0(4)
Extinction coefficient	n/a
Largest diff. peak and hole	0.439 and -0.239 $e \cdot Å^{-3}$

Table A3.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for ***88****. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.*

	x	y	z	U(eq)
C(1)	5129(2)	2251(1)	3426(1)	11(1)
O(1)	4972(1)	2819(1)	2328(1)	15(1)
C(2)	6748(2)	1423(1)	4116(1)	12(1)
C(6)	6594(2)	318(1)	3110(2)	17(1)
O(2)	7134(2)	-744(1)	3455(1)	25(1)
C(7)	8414(2)	2137(1)	4138(1)	16(1)
C(8)	8695(2)	3426(2)	4864(2)	25(1)
C(3)	6914(2)	931(1)	5548(1)	17(1)
C(4)	5150(2)	517(1)	5581(2)	19(1)
C(5)	3940(2)	1663(1)	5263(2)	18(1)
N(1)	3887(1)	2283(1)	3982(1)	13(1)
C(11)	10512(2)	4123(1)	1308(1)	12(1)
O(3)	10666(1)	3510(1)	2374(1)	16(1)
C(12)	8793(2)	4826(1)	554(1)	14(1)
C(16)	7434(2)	3755(2)	91(2)	24(1)
O(4)	6589(2)	3526(2)	-1097(2)	47(1)
C(17)	8355(2)	5646(2)	1638(2)	23(1)
C(18)	6550(2)	6260(2)	1077(2)	30(1)
C(13)	8826(2)	5624(2)	-676(2)	20(1)
C(14)	9870(2)	4978(2)	-1451(2)	24(1)
C(15)	11737(2)	4807(2)	-482(2)	21(1)
N(2)	11786(1)	4161(1)	787(1)	16(1)

Table A3.3 Bond lengths [Å] and angles [°] for ***88***

C(1)-O(1)	1.2460(15)
C(1)-N(1)	1.3336(16)
C(1)-C(2)	1.5334(17)
C(2)-C(3)	1.5262(19)
C(2)-C(6)	1.5311(19)
C(2)-C(7)	1.5497(18)
C(6)-O(2)	1.2037(18)
C(6)-H(6)	0.9500
C(7)-C(8)	1.523(2)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(3)-C(4)	1.5208(19)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-C(5)	1.5176(19)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-N(1)	1.4617(17)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
N(1)-H(1N)	0.901(17)
C(11)-O(3)	1.2420(16)
C(11)-N(2)	1.3334(17)
C(11)-C(12)	1.5352(17)
C(12)-C(13)	1.5286(19)
C(12)-C(16)	1.533(2)
C(12)-C(17)	1.552(2)
C(16)-O(4)	1.202(2)
C(16)-H(16)	0.9500
C(17)-C(18)	1.526(2)
C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800

C(18)-H(18C)	0.9800
C(13)-C(14)	1.521(2)
C(13)-H(13A)	0.9900
C(13)-H(13B)	0.9900
C(14)-C(15)	1.514(2)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-N(2)	1.4625(18)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
N(2)-H(2N)	0.851(17)

O(1)-C(1)-N(1)	122.04(11)
O(1)-C(1)-C(2)	119.17(11)
N(1)-C(1)-C(2)	118.69(11)
C(3)-C(2)-C(6)	111.08(11)
C(3)-C(2)-C(1)	114.09(10)
C(6)-C(2)-C(1)	105.21(10)
C(3)-C(2)-C(7)	111.85(10)
C(6)-C(2)-C(7)	103.66(11)
C(1)-C(2)-C(7)	110.25(10)
O(2)-C(6)-C(2)	124.42(13)
O(2)-C(6)-H(6)	117.8
C(2)-C(6)-H(6)	117.8
C(8)-C(7)-C(2)	114.49(12)
C(8)-C(7)-H(7A)	108.6
C(2)-C(7)-H(7A)	108.6
C(8)-C(7)-H(7B)	108.6
C(2)-C(7)-H(7B)	108.6
H(7A)-C(7)-H(7B)	107.6
C(7)-C(8)-H(8A)	109.5
C(7)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(4)-C(3)-C(2)	110.99(10)
C(4)-C(3)-H(3A)	109.4
C(2)-C(3)-H(3A)	109.4
C(4)-C(3)-H(3B)	109.4

C(2)-C(3)-H(3B)	109.4
H(3A)-C(3)-H(3B)	108.0
C(5)-C(4)-C(3)	108.79(11)
C(5)-C(4)-H(4A)	109.9
C(3)-C(4)-H(4A)	109.9
C(5)-C(4)-H(4B)	109.9
C(3)-C(4)-H(4B)	109.9
H(4A)-C(4)-H(4B)	108.3
N(1)-C(5)-C(4)	110.86(11)
N(1)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5A)	109.5
N(1)-C(5)-H(5B)	109.5
C(4)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	108.1
C(1)-N(1)-C(5)	126.55(11)
C(1)-N(1)-H(1N)	115.4(13)
C(5)-N(1)-H(1N)	118.0(13)
O(3)-C(11)-N(2)	121.64(11)
O(3)-C(11)-C(12)	119.05(11)
N(2)-C(11)-C(12)	119.30(11)
C(13)-C(12)-C(16)	111.43(11)
C(13)-C(12)-C(11)	113.70(11)
C(16)-C(12)-C(11)	103.98(11)
C(13)-C(12)-C(17)	111.89(12)
C(16)-C(12)-C(17)	107.65(12)
C(11)-C(12)-C(17)	107.73(10)
O(4)-C(16)-C(12)	123.93(15)
O(4)-C(16)-H(16)	118.0
C(12)-C(16)-H(16)	118.0
C(18)-C(17)-C(12)	113.55(12)
C(18)-C(17)-H(17A)	108.9
C(12)-C(17)-H(17A)	108.9
C(18)-C(17)-H(17B)	108.9
C(12)-C(17)-H(17B)	108.9
H(17A)-C(17)-H(17B)	107.7
C(17)-C(18)-H(18A)	109.5
C(17)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(17)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5

H(18B)-C(18)-H(18C)	109.5
C(14)-C(13)-C(12)	111.85(12)
C(14)-C(13)-H(13A)	109.2
C(12)-C(13)-H(13A)	109.2
C(14)-C(13)-H(13B)	109.2
C(12)-C(13)-H(13B)	109.2
H(13A)-C(13)-H(13B)	107.9
C(15)-C(14)-C(13)	109.24(13)
C(15)-C(14)-H(14A)	109.8
C(13)-C(14)-H(14A)	109.8
C(15)-C(14)-H(14B)	109.8
C(13)-C(14)-H(14B)	109.8
H(14A)-C(14)-H(14B)	108.3
N(2)-C(15)-C(14)	110.44(12)
N(2)-C(15)-H(15A)	109.6
C(14)-C(15)-H(15A)	109.6
N(2)-C(15)-H(15B)	109.6
C(14)-C(15)-H(15B)	109.6
H(15A)-C(15)-H(15B)	108.1
C(11)-N(2)-C(15)	126.69(11)
C(11)-N(2)-H(2N)	115.7(14)
C(15)-N(2)-H(2N)	117.6(14)

*Table A3.4 Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **88**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$*

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	9(1)	11(1)	11(1)	0(1)	2(1)	0(1)
O(1)	14(1)	18(1)	13(1)	4(1)	5(1)	2(1)
C(2)	9(1)	14(1)	12(1)	1(1)	4(1)	1(1)
C(6)	13(1)	19(1)	19(1)	-4(1)	5(1)	2(1)
O(2)	21(1)	17(1)	36(1)	-3(1)	7(1)	3(1)
C(7)	9(1)	20(1)	18(1)	2(1)	5(1)	-1(1)
C(8)	23(1)	26(1)	26(1)	-7(1)	8(1)	-11(1)
C(3)	13(1)	22(1)	14(1)	7(1)	3(1)	4(1)
C(4)	18(1)	18(1)	23(1)	9(1)	11(1)	4(1)
C(5)	18(1)	20(1)	21(1)	8(1)	12(1)	5(1)

N(1)	11(1)	15(1)	15(1)	4(1)	6(1)	4(1)
C(11)	11(1)	12(1)	12(1)	0(1)	3(1)	0(1)
O(3)	14(1)	20(1)	14(1)	6(1)	5(1)	4(1)
C(12)	11(1)	16(1)	13(1)	4(1)	3(1)	3(1)
C(16)	14(1)	24(1)	30(1)	7(1)	1(1)	-2(1)
O(4)	34(1)	52(1)	37(1)	-1(1)	-7(1)	-20(1)
C(17)	20(1)	30(1)	17(1)	2(1)	5(1)	11(1)
C(18)	25(1)	39(1)	28(1)	7(1)	11(1)	18(1)
C(13)	18(1)	22(1)	18(1)	9(1)	7(1)	6(1)
C(14)	25(1)	31(1)	16(1)	8(1)	8(1)	7(1)
C(15)	21(1)	26(1)	21(1)	11(1)	12(1)	6(1)
N(2)	12(1)	19(1)	16(1)	6(1)	6(1)	4(1)

Table A3.5 Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for ***88****.*

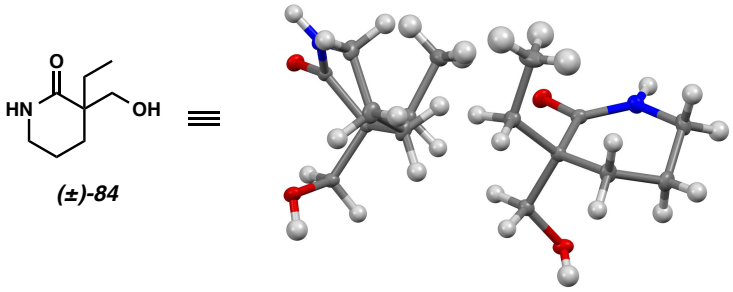
	x	y	z	U(eq)
H(6)	6035	481	2149	21
H(7A)	9440	1590	4603	19
H(7B)	8351	2267	3172	19
H(8A)	7744	4005	4359	38
H(8B)	9810	3787	4891	38
H(8C)	8712	3315	5811	38
H(3A)	7729	199	5795	20
H(3B)	7401	1612	6241	20
H(4A)	4651	-162	4887	22
H(4B)	5288	172	6506	22
H(5A)	2750	1385	5174	22
H(5B)	4350	2280	6037	22
H(1N)	2940(20)	2750(20)	3510(20)	16
H(16)	7250	3243	786	29
H(17A)	8425	5102	2439	27
H(17B)	9243	6327	1974	27
H(18A)	6455	6781	265	45
H(18B)	6381	6801	1793	45
H(18C)	5656	5591	812	45

H(13A)	7615	5763	-1318	23
H(13B)	9345	6468	-340	23
H(14A)	9351	4138	-1802	28
H(14B)	9842	5508	-2252	28
H(15A)	12388	4297	-946	25
H(15B)	12309	5651	-249	25
H(2N)	12730(20)	3790(20)	1260(20)	19

*Table A3.6 Hydrogen bonds for **88** [Å and °].*

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(5)-H(5B)...O(2)#1	0.99	2.55	3.277(2)	130.3
N(1)-H(1N)...O(3)#2	0.901(17)	1.984(17)	2.8771(15)	171(2)
N(2)-H(2N)...O(1)#3	0.851(17)	2.047(17)	2.8963(15)	177(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,y+1/2,-z+1 #2 x-1,y,z #3 x+1,y,z



(±)-84

Compound (±)-**84** crystallizes in the triclinic space group $P\bar{1}$ with two molecules in the asymmetric unit. The structure was refined with two twin domains related by a 2-fold axis, twin law (-100/0-10/001). The twin ratio refined to 0.1275(16):0.8725(16). The highest electron density maxima are located on the bonds between atoms.

*Table A3.7 Crystal data and structure refinement for (±)-**84**.*

Identification code	A14199	
CCDC Deposition Number	1002339	
Empirical formula	C8 H15 N O2	
Formula weight	157.21	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 6.0486(5) Å	α = 89.349(5)°.
	b = 11.6947(9) Å	β = 88.952(5)°.
	c = 11.8938(9) Å	γ = 84.996(5)°.
Volume	837.94(11) $Å^3$	

Z	4
Density (calculated)	1.246 Mg/m^3
Absorption coefficient	0.089 mm^{-1}
F(000)	344
Crystal size	0.250 x 0.200 x 0.150 mm^3
Theta range for data collection	1.713 to 35.630°.
Index ranges	-9<=h<=9, -19<=k<=19, -19<=l<=19
Reflections collected	48695
Independent reflections	7714 [R(int) = 0.0550]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7471 and 0.6469
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7714 / 4 / 214
Goodness-of-fit on F^2	1.078
Final R indices [I>2sigma(I)]	R1 = 0.0793, wR2 = 0.2239
R indices (all data)	R1 = 0.1022, wR2 = 0.2511
Extinction coefficient	n/a
Largest diff. peak and hole	0.709 and -0.413 $e.Å^{-3}$

*Table A3.8 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for (±)-**84**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.*

	x	y	z	U(eq)
N(1)	5794(3)	6168(2)	5728(2)	17(1)
C(1)	5529(3)	5411(2)	6563(2)	14(1)
O(1)	6927(2)	4579(1)	6721(1)	19(1)
C(2)	3485(3)	5561(2)	7352(2)	13(1)
C(3)	1921(3)	6634(2)	7084(2)	18(1)
C(6)	2270(3)	4459(2)	7288(2)	16(1)
O(2)	1294(2)	4312(1)	6233(1)	18(1)
C(7)	4304(3)	5600(2)	8571(2)	17(1)
C(8)	5917(4)	6501(2)	8764(2)	27(1)
C(4)	1908(3)	6950(2)	5834(2)	19(1)
C(5)	4248(4)	7153(2)	5440(2)	20(1)
N(101)	11065(3)	1082(1)	9237(1)	16(1)
C(101)	10787(3)	325(2)	8443(2)	13(1)
O(101)	12055(2)	-576(1)	8353(1)	17(1)
C(102)	8897(3)	540(2)	7617(2)	13(1)
C(103)	7567(3)	1713(2)	7772(2)	18(1)
C(106)	7405(3)	-457(2)	7767(2)	15(1)
O(102)	6312(2)	-464(1)	8840(1)	17(1)
C(107)	9890(3)	448(2)	6410(2)	16(1)
C(108)	11805(3)	1187(2)	6147(2)	21(1)
C(104)	7387(4)	2097(2)	8991(2)	20(1)
C(105)	9683(4)	2151(2)	9448(2)	19(1)

*Table A3.9 Bond lengths [Å] and angles [°] for (±)-**84**.*

N(1)-C(1)	1.340(3)
N(1)-C(5)	1.461(3)
N(1)-H(1N)	0.869(17)
C(1)-O(1)	1.247(2)
C(1)-C(2)	1.537(2)
C(2)-C(3)	1.538(2)
C(2)-C(6)	1.542(3)

C(2)-C(7)	1.542(3)
C(3)-C(4)	1.528(3)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(6)-O(2)	1.415(2)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
O(2)-H(2O)	0.820(18)
C(7)-C(8)	1.520(3)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(4)-C(5)	1.520(3)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
N(101)-C(101)	1.324(2)
N(101)-C(105)	1.464(2)
N(101)-H(01N)	0.880(17)
C(101)-O(101)	1.252(2)
C(101)-C(102)	1.524(2)
C(102)-C(103)	1.539(2)
C(102)-C(106)	1.543(3)
C(102)-C(107)	1.547(3)
C(103)-C(104)	1.522(3)
C(103)-H(10A)	0.9900
C(103)-H(10B)	0.9900
C(106)-O(102)	1.426(2)
C(106)-H(10C)	0.9900
C(106)-H(10D)	0.9900
O(102)-H(02O)	0.822(18)
C(107)-C(108)	1.531(3)
C(107)-H(10E)	0.9900
C(107)-H(10F)	0.9900
C(108)-H(10G)	0.9800
C(108)-H(10H)	0.9800
C(108)-H(10I)	0.9800

C(104)-C(105)	1.507(3)
C(104)-H(10J)	0.9900
C(104)-H(10K)	0.9900
C(105)-H(10L)	0.9900
C(105)-H(10M)	0.9900

C(1)-N(1)-C(5)	126.10(16)
C(1)-N(1)-H(1N)	121(2)
C(5)-N(1)-H(1N)	113(2)
O(1)-C(1)-N(1)	121.07(17)
O(1)-C(1)-C(2)	118.75(16)
N(1)-C(1)-C(2)	120.18(15)
C(1)-C(2)-C(3)	113.54(15)
C(1)-C(2)-C(6)	107.54(14)
C(3)-C(2)-C(6)	111.31(15)
C(1)-C(2)-C(7)	108.13(14)
C(3)-C(2)-C(7)	110.52(15)
C(6)-C(2)-C(7)	105.42(15)
C(4)-C(3)-C(2)	112.97(15)
C(4)-C(3)-H(3A)	109.0
C(2)-C(3)-H(3A)	109.0
C(4)-C(3)-H(3B)	109.0
C(2)-C(3)-H(3B)	109.0
H(3A)-C(3)-H(3B)	107.8
O(2)-C(6)-C(2)	113.33(15)
O(2)-C(6)-H(6A)	108.9
C(2)-C(6)-H(6A)	108.9
O(2)-C(6)-H(6B)	108.9
C(2)-C(6)-H(6B)	108.9
H(6A)-C(6)-H(6B)	107.7
C(6)-O(2)-H(2O)	109(3)
C(8)-C(7)-C(2)	114.14(17)
C(8)-C(7)-H(7A)	108.7
C(2)-C(7)-H(7A)	108.7
C(8)-C(7)-H(7B)	108.7
C(2)-C(7)-H(7B)	108.7
H(7A)-C(7)-H(7B)	107.6
C(7)-C(8)-H(8A)	109.5
C(7)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5

C(7)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(5)-C(4)-C(3)	109.48(17)
C(5)-C(4)-H(4A)	109.8
C(3)-C(4)-H(4A)	109.8
C(5)-C(4)-H(4B)	109.8
C(3)-C(4)-H(4B)	109.8
H(4A)-C(4)-H(4B)	108.2
N(1)-C(5)-C(4)	110.39(16)
N(1)-C(5)-H(5A)	109.6
C(4)-C(5)-H(5A)	109.6
N(1)-C(5)-H(5B)	109.6
C(4)-C(5)-H(5B)	109.6
H(5A)-C(5)-H(5B)	108.1
C(101)-N(101)-C(105)	126.71(16)
C(101)-N(101)-H(01N)	117(2)
C(105)-N(101)-H(01N)	116(2)
O(101)-C(101)-N(101)	121.24(16)
O(101)-C(101)-C(102)	118.64(16)
N(101)-C(101)-C(102)	120.12(15)
C(101)-C(102)-C(103)	113.21(15)
C(101)-C(102)-C(106)	106.96(14)
C(103)-C(102)-C(106)	111.53(15)
C(101)-C(102)-C(107)	108.21(14)
C(103)-C(102)-C(107)	110.07(15)
C(106)-C(102)-C(107)	106.57(14)
C(104)-C(103)-C(102)	113.37(15)
C(104)-C(103)-H(10A)	108.9
C(102)-C(103)-H(10A)	108.9
C(104)-C(103)-H(10B)	108.9
C(102)-C(103)-H(10B)	108.9
H(10A)-C(103)-H(10B)	107.7
O(102)-C(106)-C(102)	113.08(15)
O(102)-C(106)-H(10C)	109.0
C(102)-C(106)-H(10C)	109.0
O(102)-C(106)-H(10D)	109.0
C(102)-C(106)-H(10D)	109.0
H(10C)-C(106)-H(10D)	107.8
C(106)-O(102)-H(02O)	104(3)

C(108)-C(107)-C(102)	115.97(16)
C(108)-C(107)-H(10E)	108.3
C(102)-C(107)-H(10E)	108.3
C(108)-C(107)-H(10F)	108.3
C(102)-C(107)-H(10F)	108.3
H(10E)-C(107)-H(10F)	107.4
C(107)-C(108)-H(10G)	109.5
C(107)-C(108)-H(10H)	109.5
H(10G)-C(108)-H(10H)	109.5
C(107)-C(108)-H(10I)	109.5
H(10G)-C(108)-H(10I)	109.5
H(10H)-C(108)-H(10I)	109.5
C(105)-C(104)-C(103)	109.30(17)
C(105)-C(104)-H(10J)	109.8
C(103)-C(104)-H(10J)	109.8
C(105)-C(104)-H(10K)	109.8
C(103)-C(104)-H(10K)	109.8
H(10J)-C(104)-H(10K)	108.3
N(101)-C(105)-C(104)	111.07(16)
N(101)-C(105)-H(10L)	109.4
C(104)-C(105)-H(10L)	109.4
N(101)-C(105)-H(10M)	109.4
C(104)-C(105)-H(10M)	109.4
H(10L)-C(105)-H(10M)	108.0

Symmetry transformations used to generate equivalent atoms:

*Table A3.10 Anisotropic displacement parameters ($Å^2 x\ 10^3$) for (±)-**84**. The anisotropic displacement factor exponent takes the form: $-2p^2[\, h^2 a^{*2} U^{11} + \ldots + 2\, h\, k\, a^*\, b^*\, U^{12}\,]$*

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	16(1)	17(1)	17(1)	0(1)	4(1)	0(1)
C(1)	12(1)	13(1)	16(1)	-3(1)	1(1)	1(1)
O(1)	13(1)	19(1)	23(1)	-1(1)	1(1)	6(1)
C(2)	11(1)	12(1)	15(1)	-1(1)	2(1)	2(1)
C(3)	17(1)	15(1)	21(1)	0(1)	3(1)	6(1)
C(6)	15(1)	14(1)	18(1)	0(1)	1(1)	-1(1)

O(2)	12(1)	22(1)	19(1)	-5(1)	1(1)	-1(1)
C(7)	18(1)	19(1)	15(1)	-1(1)	0(1)	-1(1)
C(8)	26(1)	36(1)	21(1)	-10(1)	3(1)	-14(1)
C(4)	20(1)	15(1)	22(1)	2(1)	-3(1)	5(1)
C(5)	25(1)	15(1)	20(1)	3(1)	2(1)	0(1)
N(101)	16(1)	14(1)	16(1)	-1(1)	-4(1)	0(1)
C(101)	12(1)	11(1)	15(1)	0(1)	0(1)	1(1)
O(101)	12(1)	15(1)	22(1)	0(1)	-1(1)	4(1)
C(102)	13(1)	12(1)	14(1)	0(1)	-2(1)	2(1)
C(103)	20(1)	15(1)	19(1)	-2(1)	-5(1)	7(1)
C(106)	12(1)	15(1)	17(1)	-1(1)	-1(1)	-2(1)
O(102)	12(1)	24(1)	17(1)	2(1)	0(1)	-1(1)
C(107)	18(1)	17(1)	14(1)	-1(1)	1(1)	-2(1)
C(108)	20(1)	23(1)	20(1)	2(1)	-1(1)	-6(1)
C(104)	22(1)	15(1)	21(1)	-2(1)	0(1)	7(1)
C(105)	26(1)	12(1)	18(1)	-2(1)	-3(1)	1(1)

*Table A3.11 Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for (±)-**84**.*

	x	y	z	U(eq)
H(1N)	6870(40)	6050(30)	5240(20)	20
H(3A)	395	6497	7333	21
H(3B)	2382	7289	7516	21
H(6A)	3338	3788	7444	19
H(6B)	1096	4481	7881	19
H(2O)	-50(30)	4450(30)	6300(30)	27
H(7A)	3002	5752	9081	20
H(7B)	5034	4837	8773	20
H(8A)	7266	6324	8307	41
H(8B)	6301	6502	9561	41
H(8C)	5225	7259	8549	41
H(4A)	1357	6320	5398	23
H(4B)	903	7652	5709	23
H(5A)	4271	7279	4615	24
H(5B)	4714	7851	5800	24

H(01N)	12130(40)	910(30)	9720(20)	19
H(10A)	8287	2297	7320	22
H(10B)	6055	1670	7480	22
H(10C)	6274	-400	7173	18
H(10D)	8323	-1193	7668	18
H(02O)	5030(30)	-580(30)	8690(30)	26
H(10E)	10420	-365	6274	19
H(10F)	8687	659	5873	19
H(10G)	11282	2000	6225	31
H(10H)	12348	1044	5376	31
H(10I)	13012	991	6673	31
H(10J)	6554	2863	9038	24
H(10K)	6574	1549	9444	24
H(10L)	10384	2800	9088	23
H(10M)	9577	2290	10268	23

*Table A3.12 Hydrogen bonds for (±)-**84** [Å and °].*

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1N)...O(2)#1	0.869(17)	2.08(2)	2.926(2)	163(3)
O(2)-H(2O)...O(1)#2	0.820(18)	1.880(19)	2.686(2)	168(4)
C(4)-H(4A)...O(2)	0.99	2.54	3.168(3)	121.0
N(101)-H(01N)...O(102)#3	0.880(17)	2.01(2)	2.858(2)	161(3)
O(102)-H(02O)...O(101)#2	0.822(18)	1.849(19)	2.664(2)	171(4)
C(104)-H(10K)...O(102)	0.99	2.49	3.128(3)	122.0

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1 #2 x-1,y,z #3 -x+2,-y,-z+2