APPENDIX 4

X-Ray Crystallography Reports Relevant to Chapter 3

A4.1 CRYSTAL STRUCTURE OF COMPOUND 86

Figure A4.1 ORTEP drawing of ***86*** *(shown with 50% probability ellipsoids)*

NOTE: Crystallographic data have been deposited in the Cambridge Database (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK, and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 730732.

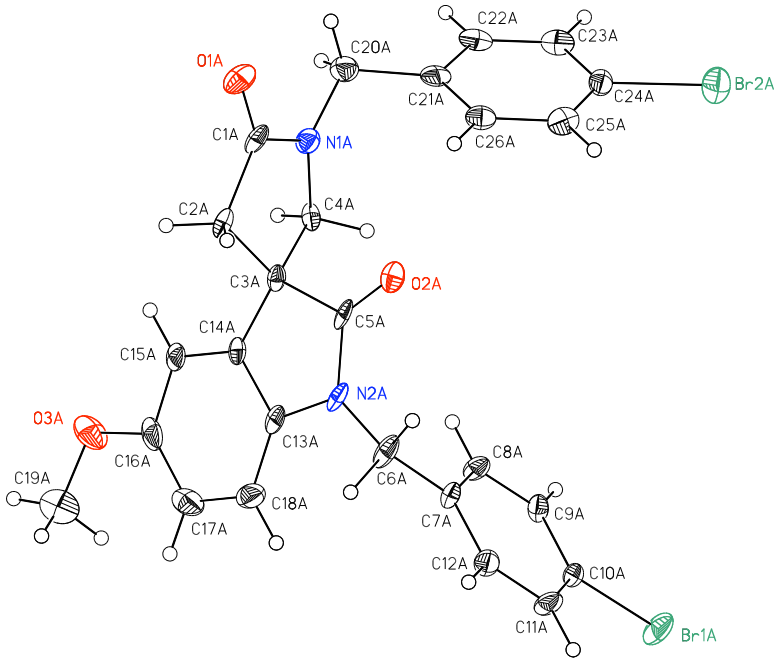

Table A4.1 Crystal data and structure refinement for compound ***86*** *(CCDC 730732)*

Empirical formula	$C_{26}H_{22}N_2O_3Br_2$
Formula weight	570.28
Crystallization Solvent	Dichloromethane/hexanes
Crystal Habit	Blade
Crystal size	0.31 x 0.15 x 0.04 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker KAPPA APEX II
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 9806 reflections used in lattice determination	2.31 to 28.58°
Unit cell dimensions	a = 17.9356(10) Å b = 5.7243(3) Å β= 101.021(3)° c = 23.3899(14) Å
Volume	2357.1(2) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1$
Density (calculated)	1.607 Mg/m^3
F(000)	1144
Data collection program	Bruker APEX2 v2.1-0
θ range for data collection	1.59 to 28.84°
Completeness to θ = 28.84°	90.2 %
Index ranges	$-23 \le h \le 23, -7 \le k \le 7, -31 \le l \le 29$
Data collection scan type	ω scans; 9 settings
Data reduction program	Bruker SAINT-Plus v7.34A
Reflections collected	34011
Independent reflections	10814 [R_{int}= 0.0407]
Absorption coefficient	3.470 mm^{-1}
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7458 and 0.5386

Table A4.1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	10814 / 1 / 597
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	2.111
Final R indices [I>2σ(I), 9564 reflections]	R1 = 0.0496, *w*R2 = 0.0966
R indices (all data)	R1 = 0.0583, *w*R2 = 0.0978
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Absolute structure determination	Anomalous differences
Absolute structure parameter	0.009(8)
Largest diff. peak and hole	2.847 and -0.755 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table A4.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for compound ***86*** *(CCDC 730732). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor*

	x	y	z	U_{eq}
Br(1A)	6411(1)	2780(1)	8308(1)	29(1)
Br(2A)	8097(1)	4182(1)	4916(1)	33(1)
O(1A)	12421(2)	1951(6)	6948(1)	25(1)
O(2A)	10376(2)	281(6)	7431(1)	20(1)
O(3A)	11743(2)	8771(7)	9625(1)	31(1)
N(1A)	11400(2)	4312(8)	7006(1)	18(1)
N(2A)	10271(2)	1382(7)	8361(2)	17(1)
C(1A)	11958(2)	2785(9)	7218(2)	18(1)
C(2A)	11927(2)	2345(8)	7851(2)	16(1)
C(3A)	11155(2)	3428(7)	7935(2)	14(1)
C(4A)	10986(2)	5220(8)	7430(2)	16(1)
C(5A)	10557(2)	1508(9)	7858(2)	17(1)
C(6A)	9744(2)	-396(9)	8467(2)	23(1)
C(7A)	8938(2)	465(8)	8439(2)	16(1)
C(8A)	8681(2)	2519(9)	8168(2)	18(1)
C(9A)	7926(2)	3243(9)	8131(2)	17(1)
C(10A)	7439(2)	1833(9)	8367(2)	18(1)
C(11A)	7684(2)	-265(9)	8642(2)	21(1)
C(12A)	8437(2)	-903(10)	8676(2)	20(1)
C(13A)	10593(2)	3132(9)	8760(2)	17(1)
C(14A)	11120(2)	4401(9)	8526(2)	15(1)
C(15A)	11498(2)	6247(9)	8824(2)	18(1)
C(16A)	11341(2)	6784(10)	9370(2)	24(1)
C(17A)	10833(3)	5510(10)	9610(2)	27(1)
C(18A)	10446(2)	3638(9)	9304(2)	26(1)
C(19A)	11662(3)	9297(13)	10187(2)	45(2)
C(20A)	11306(3)	5406(9)	6431(2)	22(1)
C(21A)	10504(3)	5108(8)	6076(2)	19(1)
C(22A)	10186(3)	6841(9)	5691(2)	21(1)
C(23A)	9481(3)	6587(9)	5352(2)	23(1)
C(24A)	9080(2)	4582(9)	5399(2)	19(1)
C(25A)	9368(2)	2831(10)	5787(2)	22(1)
C(26A)	10081(2)	3084(10)	6124(2)	20(1)
Br(1B)	1104(1)	2958(1)	3114(1)	28(1)
Br(2B)	3076(1)	4135(1)	-196(1)	39(1)
O(1B)	7247(2)	2063(5)	2000(1)	20(1)
O(2B)	5178(2)	151(5)	2326(1)	18(1)
O(3B)	6124(2)	7947(8)	4777(1)	34(1)
N(1B)	6222(2)	4391(7)	2035(1)	15(1)
N(2B)	4931(2)	1065(7)	3223(2)	17(1)
C(1B)	6767(2)	2850(9)	2256(2)	15(1)
C(2B)	6684(2)	2272(8)	2874(2)	15(1)
C(3B)	5900(2)	3250(8)	2920(2)	13(1)

C(4B)	5767(2)	5166(8)	2442(2)	13(1)
C(5B)	5306(2)	1295(8)	2767(2)	16(1)
C(6B)	4364(2)	-719(9)	3256(2)	22(1)
C(7B)	3573(2)	228(9)	3239(2)	19(1)
C(8B)	3342(2)	2320(8)	2951(2)	22(1)
C(9B)	2611(2)	3124(10)	2926(2)	22(1)
C(10B)	2115(2)	1891(8)	3185(2)	18(1)
C(11B)	2330(3)	-221(9)	3469(2)	24(1)
C(12B)	3067(2)	-977(10)	3500(2)	22(1)
C(13B)	5183(2)	2750(10)	3654(2)	20(1)
C(14B)	5778(2)	4049(9)	3506(2)	17(1)
C(15B)	6123(2)	5828(9)	3860(2)	19(1)
C(16B)	5850(3)	6237(9)	4380(2)	25(1)
C(17B)	5248(3)	4923(9)	4512(2)	28(1)
C(18B)	4905(2)	3141(10)	4155(2)	25(1)
C(19B)	6686(3)	9372(10)	4644(2)	28(1)
C(20B)	6167(2)	5522(9)	1472(2)	18(1)
C(21B)	5395(2)	5191(8)	1084(2)	17(1)
C(22B)	5106(3)	6938(9)	686(2)	20(1)
C(23B)	4409(3)	6641(9)	300(2)	23(1)
C(24B)	4011(2)	4584(9)	335(2)	23(1)
C(25B)	4277(2)	2834(11)	726(2)	22(1)
C(26B)	4978(2)	3164(9)	1105(2)	19(1)

Table A4.3 Bond lengths [Å] and angles [°] for ***86*** *(CCDC 730732)*

Br(1A)-C(10A)	1.901(4)	C(13A)-C(14A)	1.384(6)
Br(2A)-C(24A)	1.917(4)	C(14A)-C(15A)	1.372(6)
O(1A)-C(1A)	1.233(5)	C(15A)-C(16A)	1.393(6)
O(2A)-C(5A)	1.213(5)	C(16A)-C(17A)	1.369(7)
O(3A)-C(19A)	1.383(6)	C(17A)-C(18A)	1.397(7)
O(3A)-C(16A)	1.416(6)	C(20A)-C(21A)	1.527(6)
N(1A)-C(1A)	1.350(6)	C(21A)-C(22A)	1.387(6)
N(1A)-C(4A)	1.443(5)	C(21A)-C(26A)	1.401(7)
N(1A)-C(20A)	1.464(6)	C(22A)-C(23A)	1.365(7)
N(2A)-C(5A)	1.373(5)	C(23A)-C(24A)	1.370(7)
N(2A)-C(13A)	1.414(6)	C(24A)-C(25A)	1.384(7)
N(2A)-C(6A)	1.443(6)	C(25A)-C(26A)	1.377(6)
C(1A)-C(2A)	1.511(6)	Br(1B)-C(10B)	1.890(4)
C(2A)-C(3A)	1.563(5)	Br(2B)-C(24B)	1.905(4)
C(3A)-C(14A)	1.502(6)	O(1B)-C(1B)	1.226(5)
C(3A)-C(5A)	1.522(6)	O(2B)-C(5B)	1.207(5)
C(3A)-C(4A)	1.550(6)	O(3B)-C(16B)	1.373(6)
C(6A)-C(7A)	1.517(6)	O(3B)-C(19B)	1.378(6)
C(7A)-C(8A)	1.373(6)	N(1B)-C(1B)	1.344(6)
C(7A)-C(12A)	1.384(6)	N(1B)-C(4B)	1.437(5)
C(8A)-C(9A)	1.402(6)	N(1B)-C(20B)	1.453(5)
C(9A)-C(10A)	1.379(6)	N(2B)-C(5B)	1.371(5)
C(10A)-C(11A)	1.393(6)	N(2B)-C(13B)	1.406(6)
C(11A)-C(12A)	1.385(6)	N(2B)-C(6B)	1.454(6)
C(13A)-C(18A)	1.381(6)	C(1B)-C(2B)	1.518(6)

C(2B)-C(3B)	1.537(5)
C(3B)-C(14B)	1.500(5)
C(3B)-C(5B)	1.539(6)
C(3B)-C(4B)	1.552(6)
C(6B)-C(7B)	1.513(6)
C(7B)-C(12B)	1.371(6)
C(7B)-C(8B)	1.397(6)
C(8B)-C(9B)	1.381(6)
C(9B)-C(10B)	1.364(6)
C(10B)-C(11B)	1.399(7)
C(11B)-C(12B)	1.380(6)
C(13B)-C(18B)	1.376(6)
C(13B)-C(14B)	1.397(6)
C(14B)-C(15B)	1.382(6)
C(15B)-C(16B)	1.415(6)
C(16B)-C(17B)	1.397(7)
C(17B)-C(18B)	1.386(7)
C(20B)-C(21B)	1.516(6)
C(21B)-C(26B)	1.386(7)
C(21B)-C(22B)	1.397(6)
C(22B)-C(23B)	1.406(7)
C(23B)-C(24B)	1.387(7)
C(24B)-C(25B)	1.379(7)
C(25B)-C(26B)	1.406(6)

C(19A)-O(3A)-C(16A)	116.0(4)
C(1A)-N(1A)-C(4A)	115.0(3)
C(1A)-N(1A)-C(20A)	124.2(4)
C(4A)-N(1A)-C(20A)	119.5(4)
C(5A)-N(2A)-C(13A)	110.8(4)
C(5A)-N(2A)-C(6A)	123.3(4)
C(13A)-N(2A)-C(6A)	125.8(4)
O(1A)-C(1A)-N(1A)	125.9(4)
O(1A)-C(1A)-C(2A)	126.3(4)
N(1A)-C(1A)-C(2A)	107.8(3)
C(1A)-C(2A)-C(3A)	104.8(3)
C(14A)-C(3A)-C(5A)	103.0(3)
C(14A)-C(3A)-C(4A)	114.8(4)
C(5A)-C(3A)-C(4A)	110.9(3)
C(14A)-C(3A)-C(2A)	117.2(3)
C(5A)-C(3A)-C(2A)	108.6(3)
C(4A)-C(3A)-C(2A)	102.4(3)
N(1A)-C(4A)-C(3A)	103.5(3)
O(2A)-C(5A)-N(2A)	126.1(4)
O(2A)-C(5A)-C(3A)	126.1(4)
N(2A)-C(5A)-C(3A)	107.8(4)
N(2A)-C(6A)-C(7A)	114.4(4)
C(8A)-C(7A)-C(12A)	118.8(4)
C(8A)-C(7A)-C(6A)	122.1(4)
C(12A)-C(7A)-C(6A)	119.0(4)
C(7A)-C(8A)-C(9A)	121.1(4)
C(10A)-C(9A)-C(8A)	118.6(4)
C(9A)-C(10A)-C(11A)	121.4(4)
C(9A)-C(10A)-Br(1A)	118.9(3)
C(11A)-C(10A)-Br(1A)	119.7(3)
C(12A)-C(11A)-C(10A)	118.1(4)
C(11A)-C(12A)-C(7A)	121.9(5)
C(18A)-C(13A)-C(14A)	121.3(5)
C(18A)-C(13A)-N(2A)	129.1(4)
C(14A)-C(13A)-N(2A)	109.6(4)
C(15A)-C(14A)-C(13A)	120.7(4)
C(15A)-C(14A)-C(3A)	130.5(4)
C(13A)-C(14A)-C(3A)	108.7(4)
C(14A)-C(15A)-C(16A)	118.0(4)
C(17A)-C(16A)-C(15A)	121.7(5)
C(17A)-C(16A)-O(3A)	125.4(4)
C(15A)-C(16A)-O(3A)	112.9(4)
C(16A)-C(17A)-C(18A)	120.2(4)
C(13A)-C(18A)-C(17A)	118.0(4)
N(1A)-C(20A)-C(21A)	112.5(4)
C(22A)-C(21A)-C(26A)	118.8(4)
C(22A)-C(21A)-C(20A)	120.1(4)
C(26A)-C(21A)-C(20A)	121.1(4)
C(23A)-C(22A)-C(21A)	121.4(5)
C(22A)-C(23A)-C(24A)	119.0(5)
C(23A)-C(24A)-C(25A)	121.6(4)
C(23A)-C(24A)-Br(2A)	119.4(4)
C(25A)-C(24A)-Br(2A)	119.0(4)
C(26A)-C(25A)-C(24A)	119.2(5)
C(25A)-C(26A)-C(21A)	119.9(5)
C(16B)-O(3B)-C(19B)	116.8(4)
C(1B)-N(1B)-C(4B)	114.3(3)
C(1B)-N(1B)-C(20B)	124.0(3)
C(4B)-N(1B)-C(20B)	120.9(4)
C(5B)-N(2B)-C(13B)	110.7(4)
C(5B)-N(2B)-C(6B)	124.1(4)
C(13B)-N(2B)-C(6B)	125.2(4)
O(1B)-C(1B)-N(1B)	125.5(4)
O(1B)-C(1B)-C(2B)	126.2(4)
N(1B)-C(1B)-C(2B)	108.3(3)
C(1B)-C(2B)-C(3B)	104.1(3)
C(14B)-C(3B)-C(2B)	118.0(3)
C(14B)-C(3B)-C(5B)	102.6(3)
C(2B)-C(3B)-C(5B)	108.7(4)
C(14B)-C(3B)-C(4B)	114.4(4)
C(2B)-C(3B)-C(4B)	102.9(3)
C(5B)-C(3B)-C(4B)	110.1(3)
N(1B)-C(4B)-C(3B)	103.3(3)
O(2B)-C(5B)-N(2B)	124.9(4)
O(2B)-C(5B)-C(3B)	127.0(4)
N(2B)-C(5B)-C(3B)	108.0(4)
N(2B)-C(6B)-C(7B)	114.2(4)
C(12B)-C(7B)-C(8B)	119.0(4)
C(12B)-C(7B)-C(6B)	120.3(4)
C(8B)-C(7B)-C(6B)	120.6(4)
C(9B)-C(8B)-C(7B)	119.8(4)

C(10B)-C(9B)-C(8B)	120.2(5)	C(17B)-C(16B)-C(15B)	120.5(5)
C(9B)-C(10B)-C(11B)	120.9(4)	C(18B)-C(17B)-C(16B)	122.5(4)
C(9B)-C(10B)-Br(1B)	119.3(4)	C(13B)-C(18B)-C(17B)	116.1(4)
C(11B)-C(10B)-Br(1B)	119.6(3)	N(1B)-C(20B)-C(21B)	112.5(4)
C(12B)-C(11B)-C(10B)	118.1(4)	C(26B)-C(21B)-C(22B)	119.2(4)
C(7B)-C(12B)-C(11B)	121.9(5)	C(26B)-C(21B)-C(20B)	121.4(4)
C(18B)-C(13B)-C(14B)	122.9(5)	C(22B)-C(21B)-C(20B)	119.4(4)
C(18B)-C(13B)-N(2B)	127.0(4)	C(21B)-C(22B)-C(23B)	120.9(5)
C(14B)-C(13B)-N(2B)	110.1(4)	C(24B)-C(23B)-C(22B)	117.9(4)
C(15B)-C(14B)-C(13B)	121.3(4)	C(25B)-C(24B)-C(23B)	122.7(4)
C(15B)-C(14B)-C(3B)	130.2(4)	C(25B)-C(24B)-Br(2B)	119.0(4)
C(13B)-C(14B)-C(3B)	108.5(4)	C(23B)-C(24B)-Br(2B)	118.3(3)
C(14B)-C(15B)-C(16B)	116.7(4)	C(24B)-C(25B)-C(26B)	118.3(5)
O(3B)-C(16B)-C(17B)	115.3(4)	C(21B)-C(26B)-C(25B)	120.9(5)
O(3B)-C(16B)-C(15B)	124.2(4)		

Table A4.4 Anisotropic displacement parameters ($Å^2$ x 10^4) for ***86*** *(CCDC 730732). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$*

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1A)	128(2)	360(3)	409(3)	30(3)	94(2)	53(3)
Br(2A)	238(2)	399(3)	346(3)	-7(3)	2(2)	-5(3)
O(1A)	196(16)	220(20)	360(20)	-52(16)	134(15)	40(14)
O(2A)	160(15)	167(19)	267(18)	-23(15)	13(14)	-6(14)
O(3A)	390(19)	320(20)	210(17)	-104(16)	27(15)	72(18)
N(1A)	144(16)	200(20)	195(18)	7(19)	36(14)	11(18)
N(2A)	60(16)	150(20)	290(20)	72(18)	21(15)	-8(15)
C(1A)	102(17)	110(20)	310(20)	60(20)	44(17)	-20(20)
C(2A)	84(17)	120(30)	270(20)	-28(19)	18(17)	0(17)
C(3A)	114(18)	110(30)	180(20)	25(17)	-18(17)	-26(16)
C(4A)	120(20)	160(30)	160(20)	-49(19)	-25(17)	24(18)
C(5A)	40(18)	170(30)	300(30)	50(20)	-2(18)	12(18)
C(6A)	90(19)	200(30)	400(30)	90(20)	39(19)	37(19)
C(7A)	101(19)	160(30)	210(20)	0(20)	16(18)	-31(18)
C(8A)	159(19)	160(30)	230(20)	60(20)	69(17)	-10(20)
C(9A)	147(18)	160(30)	190(20)	23(19)	-15(17)	-27(19)
C(10A)	91(19)	280(30)	170(20)	0(20)	19(17)	30(19)
C(11A)	180(20)	220(30)	240(20)	100(20)	83(19)	-20(20)
C(12A)	183(19)	210(30)	210(20)	50(20)	24(17)	-10(20)
C(13A)	64(16)	180(30)	250(20)	30(20)	-3(16)	49(19)
C(14A)	89(17)	160(30)	180(20)	0(20)	-27(16)	29(19)
C(15A)	90(19)	220(30)	200(20)	0(20)	-19(17)	47(19)
C(16A)	150(20)	330(30)	200(30)	-50(20)	-50(20)	100(20)
C(17A)	260(20)	360(30)	180(20)	0(20)	60(20)	160(20)
C(18A)	170(20)	380(40)	230(20)	100(20)	67(19)	60(20)
C(19A)	480(30)	560(40)	320(30)	0(30)	100(30)	-30(40)
C(20A)	240(20)	230(30)	200(20)	10(20)	60(20)	-30(20)
C(21A)	260(20)	180(30)	160(20)	-58(19)	112(19)	10(20)

C(22A)	330(30)	170(30)	170(20)	0(20)	110(20)	-10(20)
C(23A)	290(30)	200(30)	220(30)	40(20)	100(20)	60(20)
C(24A)	160(20)	230(30)	190(20)	-70(20)	34(18)	0(20)
C(25A)	280(20)	150(30)	270(20)	-70(30)	105(19)	-30(30)
C(26A)	250(20)	180(30)	190(20)	-20(20)	87(18)	20(20)
Br(1B)	132(2)	337(3)	363(3)	-22(3)	46(2)	44(3)
Br(2B)	193(2)	704(4)	240(3)	-31(3)	-56(2)	86(3)
O(1B)	190(15)	144(19)	299(18)	-31(14)	129(14)	-3(13)
O(2B)	174(15)	139(18)	228(17)	-23(14)	1(13)	24(13)
O(3B)	318(17)	380(20)	309(18)	-60(20)	78(15)	-90(20)
N(1B)	138(15)	150(20)	157(17)	37(18)	38(14)	34(17)
N(2B)	140(17)	190(20)	176(19)	2(17)	45(15)	-22(16)
C(1B)	129(17)	120(20)	210(20)	-50(20)	48(16)	-60(20)
C(2B)	83(18)	140(30)	210(20)	22(18)	8(17)	20(17)
C(3B)	135(18)	110(30)	150(20)	1(18)	42(16)	18(17)
C(4B)	120(20)	110(20)	140(20)	-8(18)	21(17)	25(17)
C(5B)	85(19)	120(30)	260(30)	30(20)	23(18)	24(18)
C(6B)	160(20)	150(30)	370(30)	120(20)	62(19)	0(20)
C(7B)	150(20)	230(30)	200(20)	30(20)	32(18)	20(20)
C(8B)	160(20)	190(30)	310(30)	70(20)	62(19)	8(19)
C(9B)	210(20)	160(30)	270(20)	20(20)	35(19)	50(20)
C(10B)	98(19)	240(30)	190(20)	-20(20)	18(18)	32(18)
C(11B)	190(20)	290(30)	270(30)	50(20)	80(20)	-30(20)
C(12B)	200(20)	190(30)	300(20)	90(20)	72(19)	-20(20)
C(13B)	172(19)	220(30)	210(20)	80(20)	49(17)	80(20)
C(14B)	116(18)	200(30)	190(20)	0(20)	-4(16)	80(20)
C(15B)	170(20)	200(30)	200(20)	10(20)	-6(19)	40(20)
C(16B)	210(20)	270(30)	250(30)	-30(20)	-20(20)	20(20)
C(17B)	280(30)	390(40)	160(20)	20(20)	50(20)	-10(20)
C(18B)	210(20)	310(30)	250(20)	40(20)	88(19)	20(20)
C(19B)	290(20)	270(30)	270(30)	-30(30)	60(20)	-90(30)
C(20B)	170(20)	230(30)	140(20)	10(20)	18(18)	-30(20)
C(21B)	210(20)	190(30)	140(20)	-24(19)	76(19)	30(20)
C(22B)	320(20)	190(30)	130(20)	54(19)	130(20)	90(20)
C(23B)	280(20)	280(30)	140(20)	50(20)	60(20)	140(20)
C(24B)	220(20)	320(30)	150(20)	-20(20)	27(18)	110(20)
C(25B)	220(20)	250(30)	190(20)	-50(20)	48(18)	10(30)
C(26B)	200(20)	170(30)	170(20)	-10(20)	3(18)	40(20)

A4.2 CRYSTAL STRUCTURE OF SPIROCYCLIC LACTAM 138

Figure A4.2. ORTEP drawing of spirocyclic lactam ***138*** *(shown with 50% probability ellipsoids)*

NOTE: Crystallographic data have been deposited in the Cambridge Database (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK, and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 725334.

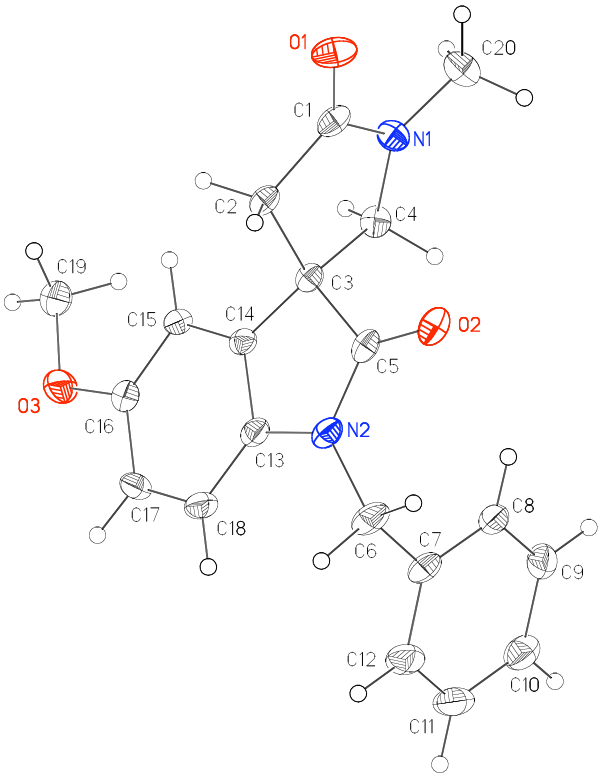

*Table A4.5 Crystal data and structure refinement for spirocyclic lactam **138** (CCDC 725334)*

Empirical formula	$C_{20}H_{20}N_2O_3$
Formula weight	336.38
Crystallization Solvent	Dichloromethane/ethylacetate/hexanes
Crystal Habit	Blade
Crystal size	0.33 x 0.14 x 0.07 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	1.54178 Å CuKα
Data Collection Temperature	100(2) K
θ range for 11578 reflections used in lattice determination	3.58 to 69.41°
Unit cell dimensions	a = 5.50060(10) Å b = 15.6733(3) Å c = 19.9921(3) Å
Volume	1723.57(5) $Å^3$
Z	4
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Density (calculated)	1.296 Mg/m^3
F(000)	712
Data collection program	Bruker SMART v5.630
θ range for data collection	3.58 to 69.47°
Completeness to θ = 69.47°	98.8 %
Index ranges	$-5 \le h \le 6, -19 \le k \le 18, -23 \le l \le 24$
Data collection scan type	ω scans at 16 ϕ settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	23652
Independent reflections	3194 [R_{int}= 0.0979]
Absorption coefficient	0.712 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9518 and 0.7989

Table A4.5 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	3194 / 0 / 229
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.599
Final R indices [I>2σ(I), 2906 reflections]	R1 = 0.0349, *w*R2 = 0.0684
R indices (all data)	R1 = 0.0394, *w*R2 = 0.0697
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Absolute structure determination	Anomalous differences
Absolute structure parameter	-0.1(2)
Largest diff. peak and hole	0.148 and -0.215 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table A4.6 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for spirocyclic lactam ***138*** *(CCDC 725334). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor*

	x	y	z	U_{eq}
O(1)	4099(2)	3561(1)	4909(1)	29(1)
O(2)	5712(2)	3042(1)	2912(1)	23(1)
O(3)	-1517(2)	-702(1)	3577(1)	28(1)
N(1)	1457(2)	3395(1)	4033(1)	20(1)
N(2)	5206(2)	1626(1)	2626(1)	20(1)
C(1)	3263(3)	3142(1)	4442(1)	20(1)
C(2)	3996(3)	2250(1)	4248(1)	18(1)
C(3)	2911(3)	2120(1)	3541(1)	16(1)
C(4)	798(3)	2766(1)	3531(1)	18(1)
C(5)	4779(3)	2342(1)	2999(1)	18(1)
C(6)	6585(3)	1623(1)	2000(1)	26(1)
C(7)	4892(3)	1665(1)	1403(1)	22(1)
C(8)	3225(3)	2328(1)	1358(1)	23(1)
C(9)	1587(3)	2363(1)	830(1)	26(1)
C(10)	1612(3)	1739(1)	340(1)	28(1)
C(11)	3264(4)	1079(1)	377(1)	30(1)
C(12)	4896(3)	1042(1)	907(1)	28(1)
C(13)	3623(3)	954(1)	2822(1)	19(1)
C(14)	2210(3)	1223(1)	3358(1)	16(1)
C(15)	459(3)	691(1)	3638(1)	18(1)
C(16)	171(3)	-122(1)	3359(1)	21(1)
C(17)	1664(3)	-394(1)	2837(1)	24(1)
C(18)	3413(3)	142(1)	2559(1)	24(1)
C(19)	-3252(3)	-409(1)	4053(1)	26(1)
C(20)	61(3)	4170(1)	4118(1)	27(1)

*Table A4.7 Bond lengths [Å] and angles [°] for spirocyclic lactam **138** (CCDC 725334)*

O(1)-C(1)	1.2309(19)	O(1)-C(1)-N(1)	125.42(16)
O(2)-C(5)	1.2240(19)	O(1)-C(1)-C(2)	126.27(15)
O(3)-C(16)	1.3704(19)	N(1)-C(1)-C(2)	108.26(13)
O(3)-C(19)	1.424(2)	C(1)-C(2)-C(3)	104.78(13)
N(1)-C(1)	1.347(2)	C(14)-C(3)-C(5)	102.24(13)
N(1)-C(20)	1.447(2)	C(14)-C(3)-C(4)	114.71(12)
N(1)-C(4)	1.452(2)	C(5)-C(3)-C(4)	110.24(13)
N(2)-C(5)	1.367(2)	C(14)-C(3)-C(2)	116.40(13)
N(2)-C(13)	1.422(2)	C(5)-C(3)-C(2)	110.92(12)
N(2)-C(6)	1.4640(19)	C(4)-C(3)-C(2)	102.51(12)
C(1)-C(2)	1.507(2)	N(1)-C(4)-C(3)	104.39(12)
C(2)-C(3)	1.546(2)	O(2)-C(5)-N(2)	125.90(15)
C(3)-C(14)	1.503(2)	O(2)-C(5)-C(3)	125.82(15)
C(3)-C(5)	1.535(2)	N(2)-C(5)-C(3)	108.28(13)
C(3)-C(4)	1.542(2)	N(2)-C(6)-C(7)	110.80(13)
C(6)-C(7)	1.514(2)	C(8)-C(7)-C(12)	118.60(16)
C(7)-C(8)	1.389(2)	C(8)-C(7)-C(6)	119.30(15)
C(7)-C(12)	1.392(2)	C(12)-C(7)-C(6)	122.07(16)
C(8)-C(9)	1.388(2)	C(9)-C(8)-C(7)	120.54(15)
C(9)-C(10)	1.385(2)	C(8)-C(9)-C(10)	120.19(17)
C(10)-C(11)	1.378(3)	C(11)-C(10)-C(9)	119.92(17)
C(11)-C(12)	1.390(3)	C(10)-C(11)-C(12)	119.88(17)
C(13)-C(18)	1.382(2)	C(11)-C(12)-C(7)	120.87(17)
C(13)-C(14)	1.390(2)	C(18)-C(13)-C(14)	121.77(15)
C(14)-C(15)	1.391(2)	C(18)-C(13)-N(2)	128.99(14)
C(15)-C(16)	1.399(2)	C(14)-C(13)-N(2)	109.24(14)
C(16)-C(17)	1.396(2)	C(13)-C(14)-C(15)	121.01(15)
C(17)-C(18)	1.393(2)	C(13)-C(14)-C(3)	109.17(14)
		C(15)-C(14)-C(3)	129.82(14)
C(16)-O(3)-C(19)	116.90(12)	C(14)-C(15)-C(16)	117.66(15)
C(1)-N(1)-C(20)	124.58(14)	O(3)-C(16)-C(17)	115.65(15)
C(1)-N(1)-C(4)	113.85(13)	O(3)-C(16)-C(15)	123.66(14)
C(20)-N(1)-C(4)	121.18(13)	C(17)-C(16)-C(15)	120.68(15)
C(5)-N(2)-C(13)	110.70(13)	C(18)-C(17)-C(16)	121.32(15)
C(5)-N(2)-C(6)	123.89(14)	C(13)-C(18)-C(17)	117.48(15)
C(13)-N(2)-C(6)	123.36(14)		

*Table A4.8 Anisotropic displacement parameters ($Å^2$ x 10^4) for spirocyclic lactam **138** (CCDC 725334). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$*

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	269(7)	394(8)	210(6)	-109(5)	-24(5)	-55(5)
O(2)	189(6)	298(7)	204(6)	67(5)	-26(5)	-51(5)

O(3)	359(7)	181(6)	294(6)	-18(5)	77(5)	-57(5)
N(1)	182(7)	189(7)	224(7)	-25(6)	-19(5)	-7(6)
N(2)	173(7)	263(8)	153(7)	33(6)	21(5)	22(6)
C(1)	161(8)	286(10)	154(8)	17(7)	20(6)	-67(7)
C(2)	153(8)	249(9)	139(8)	22(6)	-12(6)	-4(6)
C(3)	130(8)	201(8)	144(7)	19(6)	-17(6)	4(6)
C(4)	154(8)	207(9)	194(8)	-2(6)	-48(6)	-29(6)
C(5)	137(8)	261(9)	144(8)	53(7)	-63(6)	3(6)
C(6)	185(9)	398(10)	189(8)	31(8)	67(7)	32(7)
C(7)	191(8)	300(10)	165(8)	31(7)	75(6)	-26(7)
C(8)	304(10)	209(9)	168(8)	6(6)	22(7)	-29(7)
C(9)	314(10)	228(9)	229(9)	29(7)	-41(7)	19(8)
C(10)	317(10)	319(10)	197(8)	7(7)	-20(7)	-52(9)
C(11)	365(11)	326(11)	220(9)	-77(7)	68(8)	-31(9)
C(12)	264(9)	331(10)	248(9)	-26(8)	79(7)	57(8)
C(13)	179(8)	235(9)	143(7)	28(6)	5(6)	38(7)
C(14)	165(8)	193(8)	128(7)	3(6)	-21(6)	33(6)
C(15)	203(9)	190(8)	139(7)	-4(6)	14(6)	16(6)
C(16)	246(9)	194(9)	185(8)	30(6)	12(7)	-4(7)
C(17)	330(10)	176(8)	203(8)	-49(7)	-12(7)	54(7)
C(18)	288(10)	272(10)	171(8)	-10(7)	53(7)	75(7)
C(19)	280(10)	239(9)	252(9)	29(7)	33(8)	-27(7)
C(20)	243(9)	216(9)	346(9)	-33(8)	17(7)	-2(7)