APPENDIX 4

X-Ray Crystallography Reports Relevant to Appendix 3

A4.1 CRYSTAL STRUCTURE OF PHENOXY IMINOISOBENZOFURAN 548p

Figure A7.1. ORTEP drawing of phenoxy iminoisobenzofuran ***548p*** *(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 739396.

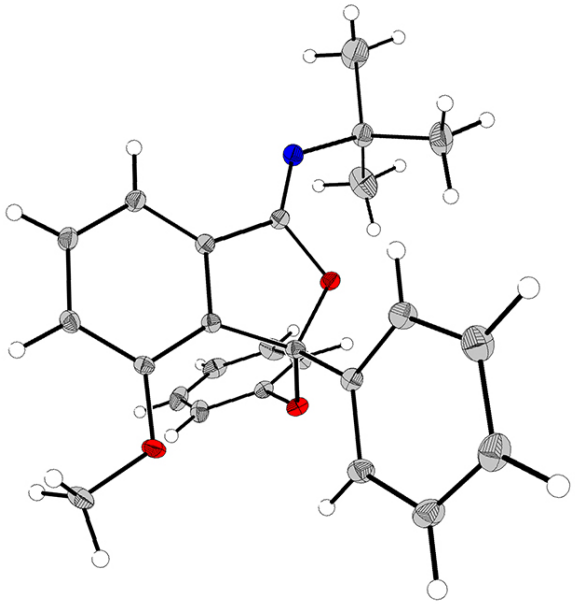
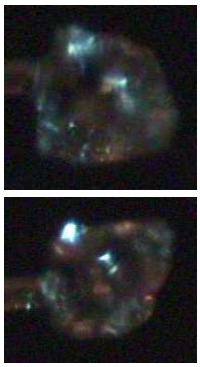


*Table A7.1. Crystal data and structure refinement for phenoxy iminoisobenzofuran **548p** (CCDC 739396)*

Empirical formula	$C_{25}H_{25}NO_3$
Formula weight	387.46
Crystallization Solvent	$CDCl_3$
Crystal Habit	Block
Crystal size	0.31 x 0.27 x 0.25 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 9578 reflections used in lattice determination	2.48 to 38.91°	
Unit cell dimensions	a = 8.4862(3) Å b = 9.8342(3) Å c = 13.1013(4) Å	α= 77.791(2)° β= 77.424(2)° γ = 80.138(2)°
Volume	1033.97(6) $Å^3$	
Z	2	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.245 Mg/m^3	
F(000)	412	
Data collection program	Bruker APEX2 v2.1-0	
θ range for data collection	2.14 to 39.22°	
Completeness to θ = 39.22°	95.3 %	
Index ranges	$-14 \le h \le 14, -15 \le k \le 17, -22 \le l \le 23$	
Data collection scan type	ω scans; 18 settings	
Data reduction program	Bruker SAINT-Plus v7.34A	
Reflections collected	51220	
Independent reflections	11606 [R_{int}= 0.0801]	
Absorption coefficient	0.081 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9800 and 0.9752	

Table A7.1. (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	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	11606 / 0 / 362
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.891
Final R indices [I>2σ(I), 8945 reflections]	R1 = 0.0431, *w*R2 = 0.1014
R indices (all data)	R1 = 0.0581, *w*R2 = 0.1036
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
Largest diff. peak and hole	0.648 and -0.292 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.

Figure A7.2. Phenoxy iminoisobenzofuran ***548p***

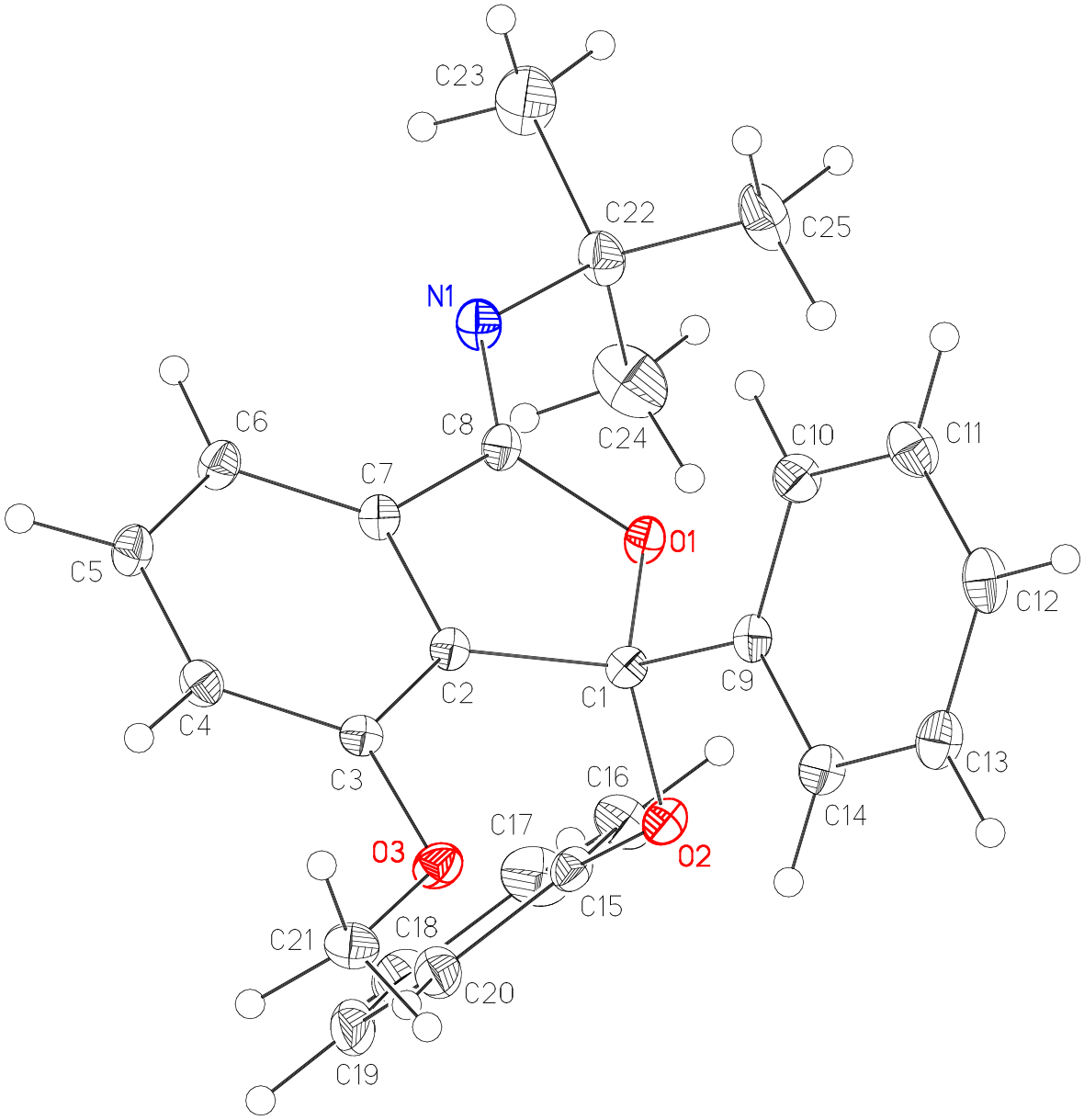


Table A7.2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for ***548p*** *(CCDC 739396). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor*

	x	y	z	U_{eq}
O(1)	3472(1)	9014(1)	1779(1)	12(1)
O(2)	5165(1)	9139(1)	2930(1)	12(1)
O(3)	2639(1)	9400(1)	5108(1)	15(1)
N(1)	1898(1)	7586(1)	1261(1)	14(1)
C(1)	3544(1)	9449(1)	2754(1)	11(1)
C(2)	2329(1)	8643(1)	3583(1)	11(1)
C(3)	1846(1)	8646(1)	4671(1)	11(1)
C(4)	591(1)	7865(1)	5232(1)	13(1)
C(5)	-124(1)	7092(1)	4714(1)	14(1)
C(6)	360(1)	7080(1)	3636(1)	14(1)
C(7)	1594(1)	7881(1)	3088(1)	11(1)
C(8)	2302(1)	8109(1)	1950(1)	11(1)
C(9)	3117(1)	11036(1)	2619(1)	11(1)
C(10)	1992(1)	11714(1)	1985(1)	15(1)
C(11)	1592(1)	13170(1)	1848(1)	18(1)
C(12)	2320(1)	13954(1)	2340(1)	18(1)
C(13)	3425(1)	13278(1)	2980(1)	17(1)
C(14)	3820(1)	11817(1)	3134(1)	14(1)
C(15)	5864(1)	7735(1)	3005(1)	12(1)
C(16)	6663(1)	7253(1)	2082(1)	19(1)
C(17)	7442(1)	5878(1)	2155(1)	24(1)
C(18)	7425(1)	5014(1)	3138(1)	23(1)
C(19)	6657(1)	5516(1)	4060(1)	22(1)
C(20)	5858(1)	6889(1)	3996(1)	17(1)
C(21)	2051(1)	9494(1)	6203(1)	16(1)
C(22)	2651(1)	7806(1)	125(1)	17(1)
C(23)	1637(1)	7108(1)	-400(1)	32(1)
C(24)	4405(1)	7088(1)	-15(1)	30(1)
C(25)	2593(1)	9360(1)	-384(1)	25(1)

Table A7.3. Bond lengths [Å] and angles [°] for ***548p*** *(CCDC 739396)*

O(1)-C(8)	1.3962(7)	C(24)-H(24A)	0.954(13)
O(1)-C(1)	1.4473(8)	C(24)-H(24B)	0.999(13)
O(2)-C(15)	1.3989(7)	C(24)-H(24C)	1.014(14)
O(2)-C(1)	1.4153(8)	C(25)-H(25A)	0.970(12)
O(3)-C(3)	1.3597(7)	C(25)-H(25B)	1.003(12)
O(3)-C(21)	1.4299(8)	C(25)-H(25C)	1.002(11)
N(1)-C(8)	1.2599(8)		
N(1)-C(22)	1.4726(9)	C(8)-O(1)-C(1)	111.20(5)
C(1)-C(2)	1.5160(8)	C(15)-O(2)-C(1)	116.66(4)
C(1)-C(9)	1.5218(8)	C(3)-O(3)-C(21)	117.04(5)
C(2)-C(7)	1.3826(8)	C(8)-N(1)-C(22)	124.50(5)
C(2)-C(3)	1.3961(9)	O(2)-C(1)-O(1)	109.11(4)
C(3)-C(4)	1.4012(8)	O(2)-C(1)-C(2)	114.67(5)
C(4)-C(5)	1.4006(9)	O(1)-C(1)-C(2)	103.49(4)
C(4)-H(4)	0.975(10)	O(2)-C(1)-C(9)	106.51(4)
C(5)-C(6)	1.3843(9)	O(1)-C(1)-C(9)	108.83(5)
C(5)-H(5)	0.975(9)	C(2)-C(1)-C(9)	114.05(5)
C(6)-C(7)	1.3956(8)	C(7)-C(2)-C(3)	120.56(5)
C(6)-H(6)	0.972(10)	C(7)-C(2)-C(1)	109.08(5)
C(7)-C(8)	1.4655(9)	C(3)-C(2)-C(1)	130.25(5)
C(9)-C(10)	1.3914(9)	O(3)-C(3)-C(2)	117.35(5)
C(9)-C(14)	1.3970(9)	O(3)-C(3)-C(4)	125.00(6)
C(10)-C(11)	1.3961(9)	C(2)-C(3)-C(4)	117.65(5)
C(10)-H(10)	0.987(10)	C(3)-C(4)-C(5)	120.70(6)
C(11)-C(12)	1.3930(10)	C(3)-C(4)-H(4)	120.1(5)
C(11)-H(11)	0.977(11)	C(5)-C(4)-H(4)	119.2(5)
C(12)-C(13)	1.3849(11)	C(6)-C(5)-C(4)	121.75(6)
C(12)-H(12)	0.963(10)	C(6)-C(5)-H(5)	121.2(6)
C(13)-C(14)	1.3996(9)	C(4)-C(5)-H(5)	117.0(6)
C(13)-H(13)	0.995(11)	C(5)-C(6)-C(7)	116.73(6)
C(14)-H(14)	0.970(10)	C(5)-C(6)-H(6)	122.9(6)
C(15)-C(16)	1.3861(9)	C(7)-C(6)-H(6)	120.4(6)
C(15)-C(20)	1.3851(10)	C(2)-C(7)-C(6)	122.60(6)
C(16)-C(17)	1.3929(9)	C(2)-C(7)-C(8)	108.60(5)
C(16)-H(16)	0.990(11)	C(6)-C(7)-C(8)	128.78(6)
C(17)-C(18)	1.3846(12)	N(1)-C(8)-O(1)	127.00(6)
C(17)-H(17)	0.947(12)	N(1)-C(8)-C(7)	125.45(5)
C(18)-C(19)	1.3877(12)	O(1)-C(8)-C(7)	107.54(5)
C(18)-H(18)	0.973(10)	C(10)-C(9)-C(14)	119.65(5)
C(19)-C(20)	1.3978(10)	C(10)-C(9)-C(1)	119.45(5)
C(19)-H(19)	0.958(13)	C(14)-C(9)-C(1)	120.90(6)
C(20)-H(20)	0.966(12)	C(9)-C(10)-C(11)	120.24(6)
C(21)-H(21A)	0.970(10)	C(9)-C(10)-H(10)	119.6(6)
C(21)-H(21B)	0.958(9)	C(11)-C(10)-H(10)	120.2(6)
C(21)-H(21C)	1.026(11)	C(12)-C(11)-C(10)	120.19(7)
C(22)-C(24)	1.5246(11)	C(12)-C(11)-H(11)	120.4(5)
C(22)-C(25)	1.5293(10)	C(10)-C(11)-H(11)	119.4(5)
C(22)-C(23)	1.5306(11)	C(13)-C(12)-C(11)	119.57(6)
C(23)-H(23A)	1.033(13)	C(13)-C(12)-H(12)	120.3(7)
C(23)-H(23B)	1.006(13)	C(11)-C(12)-H(12)	120.1(7)
C(23)-H(23C)	1.005(14)	C(12)-C(13)-C(14)	120.65(6)

C(12)-C(13)-H(13)	121.6(7)	H(21A)-C(21)-H(21C)	108.9(8)
C(14)-C(13)-H(13)	117.7(7)	H(21B)-C(21)-H(21C)	111.6(9)
C(9)-C(14)-C(13)	119.68(6)	N(1)-C(22)-C(24)	109.65(6)
C(9)-C(14)-H(14)	120.0(5)	N(1)-C(22)-C(25)	112.64(5)
C(13)-C(14)-H(14)	120.3(5)	C(24)-C(22)-C(25)	110.30(7)
C(16)-C(15)-C(20)	121.38(6)	N(1)-C(22)-C(23)	105.06(6)
C(16)-C(15)-O(2)	118.70(6)	C(24)-C(22)-C(23)	110.07(7)
C(20)-C(15)-O(2)	119.72(6)	C(25)-C(22)-C(23)	108.99(7)
C(15)-C(16)-C(17)	119.13(7)	C(22)-C(23)-H(23A)	108.6(8)
C(15)-C(16)-H(16)	119.3(6)	C(22)-C(23)-H(23B)	110.6(6)
C(17)-C(16)-H(16)	121.6(6)	H(23A)-C(23)-H(23B)	107.1(10)
C(18)-C(17)-C(16)	120.15(7)	C(22)-C(23)-H(23C)	110.4(7)
C(18)-C(17)-H(17)	120.4(8)	H(23A)-C(23)-H(23C)	111.0(10)
C(16)-C(17)-H(17)	119.5(8)	H(23B)-C(23)-H(23C)	109.0(12)
C(17)-C(18)-C(19)	120.31(6)	C(22)-C(24)-H(24A)	110.2(8)
C(17)-C(18)-H(18)	120.8(7)	C(22)-C(24)-H(24B)	109.7(7)
C(19)-C(18)-H(18)	118.7(7)	H(24A)-C(24)-H(24B)	106.2(10)
C(18)-C(19)-C(20)	120.02(7)	C(22)-C(24)-H(24C)	112.6(8)
C(18)-C(19)-H(19)	120.1(6)	H(24A)-C(24)-H(24C)	107.6(12)
C(20)-C(19)-H(19)	119.8(6)	H(24B)-C(24)-H(24C)	110.2(10)
C(15)-C(20)-C(19)	118.98(7)	C(22)-C(25)-H(25A)	110.6(7)
C(15)-C(20)-H(20)	119.2(7)	C(22)-C(25)-H(25B)	110.6(6)
C(19)-C(20)-H(20)	121.8(7)	H(25A)-C(25)-H(25B)	108.2(10)
O(3)-C(21)-H(21A)	111.1(7)	C(22)-C(25)-H(25C)	113.0(7)
O(3)-C(21)-H(21B)	105.3(6)	H(25A)-C(25)-H(25C)	106.5(9)
H(21A)-C(21)-H(21B)	109.5(8)	H(25B)-C(25)-H(25C)	107.8(9)
O(3)-C(21)-H(21C)	110.6(6)		

Table A7.4. Anisotropic displacement parameters ($Å^2$ x 10^4) for ***548p*** *(CCDC 739396). 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)	130(2)	128(2)	105(2)	-46(1)	-1(2)	-40(1)
O(2)	100(2)	105(2)	174(2)	-44(2)	-28(2)	-3(1)
O(3)	151(2)	196(2)	119(2)	-70(2)	-24(2)	-45(2)
N(1)	172(2)	145(2)	104(2)	-52(2)	-10(2)	-32(2)
C(1)	102(2)	120(2)	104(2)	-37(2)	-15(2)	-13(2)
C(2)	103(2)	109(2)	105(2)	-35(2)	-12(2)	-7(2)
C(3)	111(2)	121(2)	107(2)	-40(2)	-24(2)	-2(2)
C(4)	134(2)	148(2)	104(3)	-33(2)	-7(2)	-13(2)
C(5)	145(3)	164(2)	126(3)	-38(2)	8(2)	-50(2)
C(6)	146(3)	146(2)	132(3)	-53(2)	-1(2)	-49(2)
C(7)	115(2)	115(2)	105(2)	-42(2)	-3(2)	-12(2)
C(8)	117(2)	106(2)	113(2)	-39(2)	-7(2)	-16(2)
C(9)	113(2)	106(2)	115(3)	-32(2)	1(2)	-11(2)
C(10)	155(3)	133(2)	161(3)	-36(2)	-36(2)	-1(2)
C(11)	187(3)	143(3)	177(3)	-22(2)	-32(2)	25(2)
C(12)	212(3)	113(2)	178(3)	-39(2)	21(2)	-4(2)
C(13)	200(3)	129(2)	186(3)	-67(2)	-2(2)	-34(2)
C(14)	153(3)	135(2)	153(3)	-50(2)	-22(2)	-17(2)
C(15)	106(2)	109(2)	159(3)	-30(2)	-27(2)	-12(2)
C(16)	221(3)	163(3)	166(3)	-53(2)	-46(2)	37(2)
C(17)	273(4)	185(3)	275(4)	-113(3)	-93(3)	76(2)
C(18)	206(3)	129(3)	371(5)	-42(3)	-111(3)	10(2)
C(19)	155(3)	186(3)	280(4)	64(3)	-56(3)	-24(2)
C(20)	130(3)	184(3)	174(3)	6(2)	-20(2)	-10(2)
C(21)	174(3)	201(3)	120(3)	-72(2)	-38(2)	-9(2)
C(22)	229(3)	178(3)	103(3)	-51(2)	-11(2)	-49(2)
C(23)	500(6)	392(5)	148(4)	-90(3)	-54(4)	-231(4)
C(24)	295(4)	383(4)	175(4)	-97(3)	29(3)	71(3)
C(25)	369(4)	209(3)	143(3)	-3(2)	-26(3)	-58(3)

Table A7.5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for ***548p*** *(CCDC 739396)*

	x	y	z	U_{iso}
H(4)	195(11)	7865(9)	5989(8)	16(2)
H(5)	-949(11)	6529(9)	5152(8)	19(2)
H(6)	-126(12)	6554(10)	3263(9)	24(2)
H(10)	1514(12)	11163(10)	1610(9)	24(2)
H(11)	817(12)	13634(10)	1389(9)	22(2)
H(12)	2027(12)	14956(10)	2256(9)	24(2)
H(13)	3978(13)	13807(11)	3336(9)	28(3)
H(14)	4579(11)	11346(9)	3594(8)	15(2)
H(16)	6670(12)	7893(10)	1388(9)	24(2)
H(17)	7961(15)	5535(12)	1525(11)	40(3)
H(18)	8013(13)	4068(11)	3201(9)	31(3)
H(19)	6660(14)	4920(11)	4740(10)	34(3)
H(20)	5290(14)	7260(11)	4623(10)	35(3)
H(21A)	899(12)	9846(10)	6330(9)	22(2)
H(21B)	2656(12)	10148(10)	6345(8)	20(2)
H(21C)	2231(12)	8528(10)	6677(9)	24(2)
H(23A)	1742(16)	6048(13)	-88(12)	51(4)
H(23B)	2069(15)	7207(12)	-1187(11)	38(3)
H(23C)	467(18)	7546(13)	-279(12)	58(4)
H(24A)	5072(16)	7598(13)	224(12)	51(4)
H(24B)	4858(14)	7095(11)	-786(10)	37(3)
H(24C)	4511(17)	6092(14)	400(12)	55(4)
H(25A)	1484(14)	9829(11)	-271(10)	37(3)
H(25B)	3018(13)	9472(10)	-1169(10)	28(3)
H(25C)	3247(13)	9878(11)	-80(10)	31(3)