APPENDIX EIGHT

X-Ray Crystallographic Data Relevant to Chapter Four

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Semicarbazone 343 (DCB26)

(CCDC 246585)

Contents:

Figure A8.1 Representation of Semicarbazone **343**

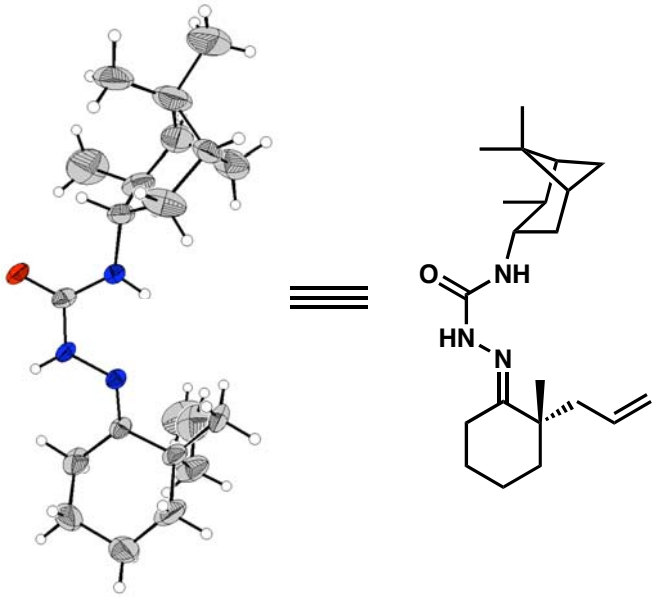

Table 1. Crystal data and structure refinement for DCB26 (CCDC 246585).

Empirical formula	$C_{21}H_{35}N_3O$
Formula weight	345.52
Crystallization Solvent	Ethanol/water
Crystal Habit	Fragment
Crystal size	0.41 x 0.37 x 0.24 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 7110 reflections used in lattice determination	2.31 to 24.12°
Unit cell dimensions	a = 23.1170(16) Å b = 13.6467(9) Å β= 90.396(2)° c = 13.2060(9) Å
Volume	4166.0(5) $Å^3$
Z	8
Crystal system	Monoclinic
Space group	C2
Density (calculated)	1.102 Mg/m^3
F(000)	1520
θ range for data collection	1.73 to 33.55°
Completeness to θ = 33.55°	81.9 %
Index ranges	$-29 \le h \le 34$, $-20 \le k \le 20$, $-18 \le l \le 17$
Data collection scan type	ω scans at 4 ϕ settings
Reflections collected	30377
Independent reflections	12571 [R_{int}= 0.0616]
Absorption coefficient	0.068 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9838 and 0.9726

Table 1 (cont.)

Structure Solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	12571 / 64 / 486
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.972
Final R indices [I>2σ(I), 5761 reflections]	R1 = 0.0873, *w*R2 = 0.1490
R indices (all data)	R1 = 0.1657, *w*R2 = 0.1573
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.002
Average shift/error	0.000
Absolute structure parameter	0.4(16)
Largest diff. peak and hole	0.630 and -0.361 e.Å^{-3}

Special Refinement Details

The data are weak and the structure is disordered, in the allyl of molecule B. These two factors combine to produce a final structure that falls short of the desired quality. Nevertheless, the quality is sufficient to determine the relative stereochemistry around C1 and, given the known stereochemistry of another chiral center, the absolute conformation can be deduced. Care should be taken when including these results in a publication. The allylic fragments were restrained to have similar geometry and the anisotropic displacement factors of the B molecule allyl fragment (only) were restrained to tend towards isotropic behavior.

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 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB26 (CCDC 246585). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
O(1A)	1866(1)	5514(2)	7391(2)	63(1)	1
N(1A)	1997(1)	3514(2)	9031(2)	41(1)	1
N(2A)	2165(1)	4328(2)	8469(2)	42(1)	1
N(3A)	1189(1)	4560(2)	8180(2)	55(1)	1
C(1A)	2171(2)	2117(3)	10056(2)	62(1)	1
C(2A)	2532(2)	1239(3)	9644(4)	94(2)	1
C(3A)	3184(2)	1396(4)	9725(4)	115(2)	1
C(4A)	3351(2)	2343(4)	9182(4)	118(2)	1
C(5A)	3008(2)	3211(4)	9569(3)	83(1)	1
C(6A)	2373(2)	3018(3)	9517(2)	47(1)	1
C(7A)	1528(2)	1903(3)	9804(3)	83(1)	1
C(8A)	2283(2)	2153(3)	11192(3)	104(2)	1
C(9A)	1979(3)	2990(4)	11616(3)	135(2)	1
C(10A)	2111(3)	3520(4)	12331(5)	177(3)	1
C(11A)	1739(2)	4842(3)	7984(2)	50(1)	1
C(12A)	687(2)	5024(3)	7720(3)	62(1)	1
C(13A)	282(2)	4213(4)	7270(3)	96(2)	1
C(14A)	-358(2)	4407(4)	7437(3)	73(1)	1
C(15A)	-446(2)	4624(4)	8568(4)	104(2)	1
C(16A)	-251(2)	5685(4)	8322(5)	117(2)	1
C(17A)	383(2)	5701(3)	8439(4)	92(2)	1
C(18A)	636(3)	6736(4)	8286(6)	179(3)	1
C(19A)	-497(2)	5511(5)	7207(5)	122(2)	1
C(20A)	-236(2)	6069(6)	6377(4)	192(4)	1
C(21A)	-1153(2)	5720(5)	7159(5)	168(3)	1
O(1B)	3303(1)	4867(2)	7779(2)	48(1)	1
N(1B)	3109(1)	6461(2)	5747(2)	60(1)	1
N(2B)	2975(1)	5799(2)	6490(2)	48(1)	1
N(3B)	3956(1)	5670(2)	6811(2)	56(1)	1
C(1B)	2867(2)	7524(3)	4351(4)	90(1)	1
C(2B)	2506(2)	8484(4)	4476(4)	105(2)	1
C(3B)	1857(2)	8263(3)	4489(4)	92(2)	1
C(4B)	1723(2)	7549(4)	5348(3)	100(2)	1
C(5B)	2062(2)	6614(3)	5281(3)	81(1)	1
C(6B)	2705(2)	6838(3)	5208(3)	55(1)	1
C(7B)	2755(3)	7037(5)	3331(4)	141(2)	1
C(8B)	3511(2)	7659(4)	4178(4)	31(2)	0.455(5)
C(9B)	3746(2)	8286(3)	4973(4)	35(2)	0.455(5)
C(10B)	3835(3)	9173(3)	4968(6)	76(3)	0.455(5)
C(8C)	3380(2)	8093(5)	4778(8)	121(4)	0.545(5)
C(9C)	3906(2)	7537(7)	4605(7)	137(4)	0.545(5)
C(10C)	4395(3)	7656(6)	4955(6)	106(3)	0.545(5)
C(11B)	3420(2)	5421(3)	7066(3)	43(1)	1
C(12B)	4459(1)	5290(3)	7340(3)	52(1)	1
C(13B)	4691(1)	4327(3)	6851(3)	61(1)	1
C(14B)	5348(2)	4274(3)	6798(3)	55(1)	1

C(15B)	5567(2)	5213(3)	6268(3)	58(1)	1
C(16B)	5535(1)	5710(3)	7323(2)	47(1)	1
C(17B)	4926(2)	6093(3)	7461(3)	53(1)	1
C(18B)	4835(2)	6662(3)	8438(3)	84(1)	1
C(19B)	5639(2)	4677(3)	7786(3)	56(1)	1
C(20B)	5383(2)	4423(3)	8834(3)	75(1)	1
C(21B)	6293(2)	4428(3)	7801(3)	73(1)	1

Table 3. Bond lengths [Å] and angles [°] for DCB26 (CCDC 246585).

O(1A)-C(11A)	1.242(4)	C(15B)-C(16B)	1.552(4)
N(1A)-C(6A)	1.272(4)	C(16B)-C(17B)	1.515(4)
N(1A)-N(2A)	1.392(3)	C(16B)-C(19B)	1.555(4)
N(2A)-C(11A)	1.365(4)	C(17B)-C(18B)	1.522(5)
N(3A)-C(11A)	1.355(4)	C(19B)-C(20B)	1.548(5)
N(3A)-C(12A)	1.452(4)	C(19B)-C(21B)	1.548(5)
C(1A)-C(6A)	1.497(5)		
C(1A)-C(8A)	1.521(4)	C(6A)-N(1A)-N(2A)	120.1(3)
C(1A)-C(7A)	1.549(5)	C(11A)-N(2A)-N(1A)	117.3(3)
C(1A)-C(2A)	1.560(5)	C(11A)-N(3A)-C(12A)	123.0(3)
C(2A)-C(3A)	1.525(6)	C(6A)-C(1A)-C(8A)	113.0(3)
C(3A)-C(4A)	1.528(6)	C(6A)-C(1A)-C(7A)	110.8(3)
C(4A)-C(5A)	1.516(7)	C(8A)-C(1A)-C(7A)	111.9(3)
C(5A)-C(6A)	1.492(5)	C(6A)-C(1A)-C(2A)	107.2(3)
C(8A)-C(9A)	1.455(5)	C(8A)-C(1A)-C(2A)	106.3(3)
C(9A)-C(10A)	1.226(5)	C(7A)-C(1A)-C(2A)	107.2(3)
C(12A)-C(17A)	1.503(6)	C(3A)-C(2A)-C(1A)	113.5(4)
C(12A)-C(13A)	1.564(5)	C(2A)-C(3A)-C(4A)	109.8(3)
C(13A)-C(14A)	1.521(6)	C(5A)-C(4A)-C(3A)	111.6(4)
C(14A)-C(15A)	1.536(6)	C(6A)-C(5A)-C(4A)	111.4(4)
C(14A)-C(19A)	1.569(7)	N(1A)-C(6A)-C(5A)	126.7(3)
C(15A)-C(16A)	1.551(7)	N(1A)-C(6A)-C(1A)	117.6(3)
C(16A)-C(17A)	1.473(7)	C(5A)-C(6A)-C(1A)	115.6(3)
C(16A)-C(19A)	1.592(8)	C(9A)-C(8A)-C(1A)	109.1(3)
C(17A)-C(18A)	1.542(7)	C(10A)-C(9A)-C(8A)	129.9(5)
C(19A)-C(20A)	1.467(7)	O(1A)-C(11A)-N(3A)	123.8(3)
C(19A)-C(21A)	1.544(7)	O(1A)-C(11A)-N(2A)	120.2(3)
O(1B)-C(11B)	1.238(3)	N(3A)-C(11A)-N(2A)	116.0(3)
N(1B)-C(6B)	1.279(4)	N(3A)-C(12A)-C(17A)	112.3(3)
N(1B)-N(2B)	1.370(3)	N(3A)-C(12A)-C(13A)	109.0(3)
N(2B)-C(11B)	1.376(4)	C(17A)-C(12A)-C(13A)	113.1(4)
N(3B)-C(11B)	1.331(4)	C(14A)-C(13A)-C(12A)	113.7(4)
N(3B)-C(12B)	1.448(4)	C(13A)-C(14A)-C(15A)	108.0(3)
C(1B)-C(7B)	1.523(6)	C(13A)-C(14A)-C(19A)	109.7(4)
C(1B)-C(8B)	1.521(4)	C(15A)-C(14A)-C(19A)	88.6(4)
C(1B)-C(8C)	1.522(4)	C(14A)-C(15A)-C(16A)	86.3(4)
C(1B)-C(6B)	1.517(5)	C(17A)-C(16A)-C(15A)	106.3(4)
C(1B)-C(2B)	1.562(6)	C(17A)-C(16A)-C(19A)	116.6(5)
C(2B)-C(3B)	1.530(6)	C(15A)-C(16A)-C(19A)	87.3(5)
C(3B)-C(4B)	1.529(6)	C(16A)-C(17A)-C(12A)	113.2(4)
C(4B)-C(5B)	1.500(6)	C(16A)-C(17A)-C(18A)	112.2(4)
C(5B)-C(6B)	1.521(5)	C(12A)-C(17A)-C(18A)	107.5(5)
C(8B)-C(9B)	1.456(5)	C(20A)-C(19A)-C(21A)	106.4(5)
C(9B)-C(10B)	1.228(5)	C(20A)-C(19A)-C(14A)	124.0(5)
C(8C)-C(9C)	1.454(5)	C(21A)-C(19A)-C(14A)	112.5(5)
C(9C)-C(10C)	1.228(5)	C(20A)-C(19A)-C(16A)	117.9(5)
C(12B)-C(17B)	1.545(4)	C(21A)-C(19A)-C(16A)	110.7(5)
C(12B)-C(13B)	1.562(5)	C(14A)-C(19A)-C(16A)	83.8(4)
C(13B)-C(14B)	1.524(5)	C(6B)-N(1B)-N(2B)	119.7(3)
C(14B)-C(15B)	1.547(5)	N(1B)-N(2B)-C(11B)	118.1(3)
C(14B)-C(19B)	1.564(5)	C(11B)-N(3B)-C(12B)	122.2(3)

C(7B)-C(1B)-C(8B)	94.6(4)
C(7B)-C(1B)-C(8C)	132.6(5)
C(8B)-C(1B)-C(8C)	39.8(4)
C(7B)-C(1B)-C(6B)	110.4(4)
C(8B)-C(1B)-C(6B)	115.7(4)
C(8C)-C(1B)-C(6B)	103.6(5)
C(7B)-C(1B)-C(2B)	111.8(4)
C(8B)-C(1B)-C(2B)	116.1(4)
C(8C)-C(1B)-C(2B)	87.1(4)
C(6B)-C(1B)-C(2B)	107.7(4)
C(3B)-C(2B)-C(1B)	111.2(4)
C(4B)-C(3B)-C(2B)	109.6(4)
C(5B)-C(4B)-C(3B)	113.0(4)
C(4B)-C(5B)-C(6B)	110.1(4)
N(1B)-C(6B)-C(5B)	126.5(3)
N(1B)-C(6B)-C(1B)	118.7(3)
C(5B)-C(6B)-C(1B)	114.7(3)
C(9B)-C(8B)-C(1B)	108.8(3)
C(10B)-C(9B)-C(8B)	129.6(5)
C(9C)-C(8C)-C(1B)	109.1(3)
C(10C)-C(9C)-C(8C)	129.9(5)
O(1B)-C(11B)-N(3B)	123.8(3)
O(1B)-C(11B)-N(2B)	118.9(3)
N(3B)-C(11B)-N(2B)	117.3(3)
N(3B)-C(12B)-C(17B)	110.7(2)
N(3B)-C(12B)-C(13B)	112.2(3)
C(17B)-C(12B)-C(13B)	113.5(3)
C(14B)-C(13B)-C(12B)	113.8(3)
C(13B)-C(14B)-C(15B)	108.1(3)
C(13B)-C(14B)-C(19B)	111.6(3)
C(15B)-C(14B)-C(19B)	86.9(2)
C(14B)-C(15B)-C(16B)	86.5(2)
C(17B)-C(16B)-C(15B)	108.0(3)
C(17B)-C(16B)-C(19B)	114.0(3)
C(15B)-C(16B)-C(19B)	87.0(2)
C(18B)-C(17B)-C(16B)	114.3(3)
C(18B)-C(17B)-C(12B)	110.5(3)
C(16B)-C(17B)-C(12B)	113.1(3)
C(20B)-C(19B)-C(21B)	108.6(3)
C(20B)-C(19B)-C(16B)	119.7(3)
C(21B)-C(19B)-C(16B)	110.6(3)
C(20B)-C(19B)-C(14B)	120.1(3)
C(21B)-C(19B)-C(14B)	110.3(3)
C(16B)-C(19B)-C(14B)	85.8(2)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB26 (CCDC 246585). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1A)	638(16)	458(13)	802(18)	329(13)	-72(14)	-23(11)
N(1A)	548(18)	313(13)	371(16)	24(12)	-86(14)	9(12)
N(2A)	486(16)	322(13)	450(16)	90(13)	-135(13)	-71(12)
N(3A)	467(19)	535(17)	650(20)	230(15)	-54(15)	9(14)
C(1A)	790(30)	570(20)	510(30)	211(18)	110(20)	190(20)
C(2A)	1350(50)	610(30)	860(30)	320(20)	230(30)	320(30)
C(3A)	850(40)	1450(50)	1140(40)	790(40)	170(30)	490(30)
C(4A)	570(30)	1630(50)	1350(50)	820(40)	160(30)	390(30)
C(5A)	680(30)	1020(30)	790(30)	480(30)	-340(20)	-120(30)
C(6A)	580(20)	512(19)	318(19)	86(16)	-87(17)	-35(17)
C(7A)	880(30)	550(20)	1050(40)	340(20)	220(30)	-120(20)
C(8A)	1590(50)	910(30)	630(30)	230(30)	170(30)	360(30)
C(9A)	1730(60)	1700(60)	630(40)	280(40)	-120(40)	50(50)
C(10A)	2220(80)	1770(60)	1300(60)	-300(50)	-820(60)	170(60)
C(11A)	570(20)	451(19)	460(20)	26(18)	-130(18)	3(18)
C(12A)	580(20)	680(20)	610(30)	110(20)	-40(20)	210(20)
C(13A)	840(30)	1190(40)	860(30)	-370(30)	-240(30)	350(30)
C(14A)	420(20)	1180(30)	570(30)	180(30)	-38(19)	150(20)
C(15A)	790(30)	1360(50)	980(40)	-90(30)	200(30)	370(30)
C(16A)	920(40)	860(40)	1740(60)	270(40)	420(40)	450(30)
C(17A)	1090(40)	630(30)	1040(40)	80(30)	-40(30)	360(30)
C(18A)	1730(60)	580(30)	3050(90)	-50(40)	-240(60)	100(40)
C(19A)	710(40)	1620(60)	1320(50)	170(50)	-90(40)	520(30)
C(20A)	790(40)	3690(110)	1280(50)	1540(70)	210(40)	790(50)
C(21A)	930(40)	2030(70)	2070(70)	510(60)	-40(40)	670(40)
O(1B)	552(14)	414(12)	461(14)	115(11)	-81(11)	-33(10)
N(1B)	630(20)	529(17)	650(20)	303(16)	115(17)	143(15)
N(2B)	542(18)	382(14)	504(17)	139(13)	-6(14)	20(13)
N(3B)	499(19)	519(16)	670(20)	330(15)	89(16)	96(14)
C(1B)	830(30)	880(30)	980(30)	410(30)	-10(20)	200(20)
C(2B)	660(30)	950(30)	1540(50)	620(30)	80(30)	330(30)
C(3B)	850(40)	820(30)	1080(40)	320(30)	70(30)	320(30)
C(4B)	940(40)	1270(40)	800(30)	310(30)	40(30)	280(30)
C(5B)	760(30)	1010(30)	670(30)	220(20)	-180(20)	80(30)
C(6B)	630(30)	580(20)	460(20)	131(18)	60(20)	120(20)
C(7B)	1700(40)	1730(40)	790(30)	430(30)	160(30)	410(40)
C(8B)	160(30)	420(30)	360(30)	130(30)	80(30)	160(20)
C(9B)	350(30)	250(30)	430(40)	120(30)	30(30)	20(30)
C(10B)	690(40)	780(40)	820(50)	-100(40)	-180(40)	80(40)
C(8C)	1470(60)	980(50)	1190(60)	330(40)	340(40)	170(40)
C(9C)	1460(60)	1350(60)	1310(60)	280(40)	-30(40)	-410(50)
C(10C)	1520(50)	730(40)	960(50)	170(40)	460(40)	110(40)
C(11B)	590(20)	312(16)	400(20)	67(15)	-18(18)	53(16)
C(12B)	540(20)	353(17)	670(30)	127(17)	20(19)	-4(16)
C(13B)	500(20)	490(20)	830(30)	0(20)	-170(20)	-76(18)
C(14B)	530(20)	487(19)	630(20)	-87(18)	-98(19)	39(17)

C(15B)	540(20)	830(30)	370(20)	-18(18)	-73(17)	40(20)
C(16B)	560(20)	444(18)	400(20)	38(16)	30(17)	-76(17)
C(17B)	560(20)	323(17)	700(30)	186(17)	117(19)	26(16)
C(18B)	1080(40)	430(20)	1020(30)	-60(20)	420(30)	-150(20)
C(19B)	650(20)	418(19)	620(20)	136(17)	-92(19)	-23(17)
C(20B)	1090(30)	600(20)	570(30)	230(20)	-180(20)	-190(20)
C(21B)	560(20)	670(20)	960(30)	60(20)	-310(20)	20(20)

Table 5. Hydrogen bonds for DCB26 (CCDC 246585) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(2A)-H(2A)...O(1B)	0.88	2.05	2.886(3)	158.8
N(2B)-H(2B)...O(1A)	0.88	2.04	2.860(4)	155.6

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Semicarbazone 344 (DCB27)

(CCDC 248956)

Contents:

Figure A8.2 Representation of Semicarbazone **344**

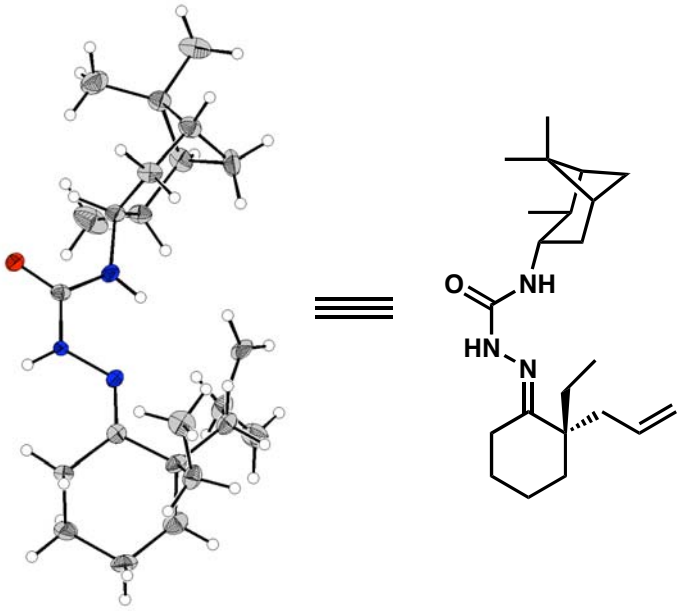

Table 1. Crystal data and structure refinement for DCB27 (CCDC 248956).

Empirical formula	$C_{22}H_{37}N_3O$
Formula weight	359.55
Crystallization Solvent	Acetone
Crystal Habit	Fragment
Crystal size	0.39 x 0.37 x 0.24 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 13615 reflections used in lattice determination	2.25 to 21.58°
Unit cell dimensions	a = 13.4105(11) Å b = 13.4433(11) Å c = 24.353(2) Å
Volume	4390.4(6) $Å^3$
Z	8
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Density (calculated)	1.088 Mg/m^3
F(000)	1584
θ range for data collection	1.67 to 28.34°
Completeness to θ = 28.34°	94.5 %
Index ranges	$-17 \le h \le 17$, $-17 \le k \le 17$, $-32 \le l \le 30$
Data collection scan type	ω scans at 5 ϕ settings
Reflections collected	63444
Independent reflections	10086 [R_{int}= 0.0909]
Absorption coefficient	0.067 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9841 and 0.9744

Table 1 (cont.)

Structure Solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	10086 / 447 / 570
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	2.208
Final R indices [$I>2\sigma(I)$, 6214 reflections]	R1 = 0.0842, *w*R2 = 0.1195
R indices (all data)	R1 = 0.1330, *w*R2 = 0.1224
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 parameter	0.6(17)
Largest diff. peak and hole	0.271 and -0.287 e.Å^{-3}

Special Refinement Details

The diffraction intensities fall off sharply past $2\theta=40^\circ$, presumably because the structure is disordered. The asymmetric unit contains two molecules (hydrogen bonded to each other and of the same configuration) disordered in different ways. Molecule A is disordered about the terminal carbon (C11) of the allyl ligand (see figures 1 and 2). Both orientations were modeled, including riding hydrogen atoms, with the only restraint being a total occupancy of 1.0 for C11A and C11C. Molecule B is disordered in the camphene moiety, C13B-C22B. The disorder manifests as a rotation of the camphene around the N3B-C13B bond (see figures 3 and 4). Both orientations were restrained to have geometry similar to the corresponding part of the A molecule, using the SAME command. Additional restraints were imposed in this portion of molecule B as follows; 1) SIMU – to restrained bonded atoms to have similar displacement parameters and 2) ISOR – to restrain the anisotropic displacement parameters, U_{ij}, to approximate isotropic behavior without placing restraint on the refined value of the isotropic U.

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 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB27 (CCDC 248956). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
O(1A)	2809(2)	2335(2)	1779(1)	32(1)	1
N(1A)	817(2)	3903(2)	2093(1)	27(1)	1
N(2A)	1655(2)	3309(2)	2175(1)	24(1)	1
N(3A)	1824(2)	3282(2)	1236(1)	34(1)	1
C(1A)	-518(2)	4936(2)	2388(1)	33(1)	1
C(2A)	-437(3)	5977(2)	2642(2)	47(1)	1
C(3A)	-112(3)	5977(2)	3235(2)	49(1)	1
C(4A)	882(3)	5438(3)	3291(2)	57(1)	1
C(5A)	795(3)	4365(2)	3076(1)	42(1)	1
C(6A)	420(2)	4360(2)	2493(1)	28(1)	1
C(7A)	-1419(2)	4400(2)	2646(1)	39(1)	1
C(8A)	-1470(3)	3264(2)	2527(1)	49(1)	1
C(9A)	-735(2)	5058(2)	1763(1)	39(1)	1
C(10A)	12(3)	5597(3)	1434(2)	48(1)	1
C(11A)	185(5)	5261(5)	885(3)	54(3)	0.511(7)
C(11C)	-125(6)	6553(7)	1281(3)	66(3)	0.489(7)
C(12A)	2114(2)	2934(2)	1725(1)	27(1)	1
C(13A)	2259(2)	2928(2)	725(1)	34(1)	1
C(14A)	1576(2)	2156(2)	446(1)	38(1)	1
C(15A)	1565(2)	2226(3)	-177(1)	42(1)	1
C(16A)	1262(3)	3289(3)	-322(2)	54(1)	1
C(17A)	2374(3)	3537(2)	-258(1)	39(1)	1
C(18A)	2554(2)	3800(2)	349(1)	36(1)	1
C(19A)	3603(3)	4144(3)	469(2)	77(1)	1
C(20A)	2622(2)	2441(2)	-411(1)	38(1)	1
C(21A)	3522(3)	1891(3)	-180(2)	59(1)	1
C(22A)	2637(3)	2320(3)	-1038(1)	61(1)	1
O(1B)	2342(2)	2440(2)	3187(1)	34(1)	1
N(1B)	4099(2)	678(2)	2784(1)	37(1)	1
N(2B)	3295(2)	1310(2)	2738(1)	37(1)	1
N(3B)	3367(2)	1432(2)	3679(1)	60(1)	1
C(1B)	5294(3)	-479(3)	2425(2)	48(1)	1
C(2B)	6017(3)	-264(3)	1958(2)	88(2)	1
C(3B)	5500(4)	-304(3)	1385(2)	81(2)	1
C(4B)	4622(3)	410(3)	1357(2)	63(1)	1
C(5B)	3895(3)	189(3)	1803(2)	54(1)	1
C(6B)	4383(3)	205(3)	2358(2)	46(1)	1
C(7B)	4962(3)	-1591(3)	2413(2)	72(1)	1
C(8B)	4290(3)	-1903(3)	2885(2)	72(1)	1
C(9B)	5798(3)	-326(3)	2981(2)	72(1)	1
C(10B)	6309(3)	682(4)	3049(2)	81(2)	1
C(11B)	7251(3)	828(3)	3119(2)	103(2)	1
C(12B)	2981(2)	1775(3)	3210(1)	35(1)	1
C(13B)	3160(7)	2001(7)	4235(3)	34(3)	0.515(3)
C(14B)	2103(6)	1773(7)	4394(3)	71(3)	0.515(3)
C(15B)	1912(8)	1565(8)	5018(3)	97(4)	0.515(3)

C(16B)	2704(8)	702(6)	5131(3)	82(3)	0.515(3)
C(17B)	3526(10)	1499(10)	5209(4)	72(4)	0.515(3)
C(18B)	3936(6)	1742(9)	4670(4)	80(3)	0.515(3)
C(19B)	4820(8)	2450(9)	4663(4)	117(4)	0.515(3)
C(20B)	2622(8)	2162(6)	5387(3)	54(3)	0.515(3)
C(21B)	2403(7)	1991(7)	5992(3)	92(4)	0.515(3)
C(22B)	2701(5)	3269(5)	5261(3)	60(2)	0.515(3)
C(13C)	2948(10)	1526(7)	4183(4)	45(3)	0.485(3)
C(14C)	3136(8)	604(5)	4553(3)	88(3)	0.485(3)
C(15C)	3848(9)	798(7)	5059(3)	77(3)	0.485(3)
C(16C)	4712(8)	1489(8)	4870(5)	108(4)	0.485(3)
C(17C)	3935(8)	2363(7)	4946(4)	80(3)	0.485(3)
C(18C)	3386(6)	2503(5)	4419(3)	35(2)	0.485(3)
C(19C)	2648(6)	3361(5)	4450(3)	65(3)	0.485(3)
C(20C)	3451(10)	1701(11)	5380(4)	68(4)	0.485(3)
C(21C)	3921(10)	1826(10)	5948(3)	160(6)	0.485(3)
C(22C)	2319(9)	1719(9)	5426(6)	99(6)	0.485(3)

Table 3. Bond lengths [Å] and angles [°] for DCB27 (CCDC 248956).

O(1A)-C(12A)	1.238(3)	C(15B)-C(16B)	1.597(11)
N(1A)-C(6A)	1.268(4)	C(16B)-C(17B)	1.549(12)
N(1A)-N(2A)	1.394(3)	C(17B)-C(18B)	1.460(12)
N(2A)-C(12A)	1.354(4)	C(17B)-C(20B)	1.565(12)
N(3A)-C(12A)	1.337(4)	C(18B)-C(19B)	1.521(11)
N(3A)-C(13A)	1.455(4)	C(20B)-C(21B)	1.521(10)
C(1A)-C(6A)	1.499(4)	C(20B)-C(22B)	1.523(10)
C(1A)-C(2A)	1.534(4)	C(13C)-C(18C)	1.549(9)
C(1A)-C(7A)	1.541(4)	C(13C)-C(14C)	1.554(11)
C(1A)-C(9A)	1.557(4)	C(14C)-C(15C)	1.580(11)
C(2A)-C(3A)	1.510(4)	C(15C)-C(20C)	1.539(13)
C(3A)-C(4A)	1.523(5)	C(15C)-C(16C)	1.554(12)
C(4A)-C(5A)	1.540(4)	C(16C)-C(17C)	1.582(11)
C(5A)-C(6A)	1.507(4)	C(17C)-C(18C)	1.490(10)
C(7A)-C(8A)	1.556(4)	C(17C)-C(20C)	1.527(13)
C(9A)-C(10A)	1.474(4)	C(18C)-C(19C)	1.521(9)
C(10A)-C(11C)	1.350(9)	C(20C)-C(22C)	1.523(13)
C(10A)-C(11A)	1.431(7)	C(20C)-C(21C)	1.527(12)
C(13A)-C(18A)	1.540(4)		
C(13A)-C(14A)	1.542(4)	C(6A)-N(1A)-N(2A)	120.4(3)
C(14A)-C(15A)	1.521(4)	C(12A)-N(2A)-N(1A)	117.7(3)
C(15A)-C(16A)	1.526(4)	C(12A)-N(3A)-C(13A)	122.0(3)
C(15A)-C(20A)	1.555(4)	C(6A)-C(1A)-C(2A)	110.1(3)
C(16A)-C(17A)	1.537(4)	C(6A)-C(1A)-C(7A)	110.3(2)
C(17A)-C(18A)	1.538(4)	C(2A)-C(1A)-C(7A)	108.5(3)
C(17A)-C(20A)	1.556(4)	C(6A)-C(1A)-C(9A)	112.2(3)
C(18A)-C(19A)	1.509(4)	C(2A)-C(1A)-C(9A)	108.1(3)
C(20A)-C(21A)	1.523(4)	C(7A)-C(1A)-C(9A)	107.5(3)
C(20A)-C(22A)	1.535(4)	C(3A)-C(2A)-C(1A)	113.9(3)
O(1B)-C(12B)	1.240(3)	C(2A)-C(3A)-C(4A)	109.8(3)
N(1B)-C(6B)	1.275(4)	C(3A)-C(4A)-C(5A)	110.4(3)
N(1B)-N(2B)	1.377(3)	C(6A)-C(5A)-C(4A)	110.5(3)
N(2B)-C(12B)	1.373(4)	N(1A)-C(6A)-C(1A)	118.1(3)
N(3B)-C(12B)	1.337(4)	N(1A)-C(6A)-C(5A)	125.9(3)
N(3B)-C(13C)	1.355(12)	C(1A)-C(6A)-C(5A)	116.0(3)
N(3B)-C(13B)	1.579(9)	C(1A)-C(7A)-C(8A)	114.7(3)
C(1B)-C(2B)	1.521(5)	C(10A)-C(9A)-C(1A)	117.1(3)
C(1B)-C(9B)	1.528(5)	C(11C)-C(10A)-C(11A)	93.6(5)
C(1B)-C(6B)	1.538(4)	C(11C)-C(10A)-C(9A)	121.7(5)
C(1B)-C(7B)	1.561(5)	C(11A)-C(10A)-C(9A)	117.5(4)
C(2B)-C(3B)	1.559(6)	O(1A)-C(12A)-N(3A)	122.8(3)
C(3B)-C(4B)	1.521(5)	O(1A)-C(12A)-N(2A)	119.9(3)
C(4B)-C(5B)	1.488(4)	N(3A)-C(12A)-N(2A)	117.2(3)
C(5B)-C(6B)	1.503(5)	N(3A)-C(13A)-C(18A)	111.3(3)
C(7B)-C(8B)	1.520(5)	N(3A)-C(13A)-C(14A)	111.0(3)
C(9B)-C(10B)	1.526(5)	C(18A)-C(13A)-C(14A)	113.8(2)
C(10B)-C(11B)	1.290(5)	C(15A)-C(14A)-C(13A)	113.8(3)
C(13B)-C(14B)	1.501(10)	C(14A)-C(15A)-C(16A)	107.0(3)
C(13B)-C(18B)	1.526(10)	C(14A)-C(15A)-C(20A)	111.6(3)
C(14B)-C(15B)	1.566(9)	C(16A)-C(15A)-C(20A)	89.1(3)
C(15B)-C(20B)	1.535(11)	C(15A)-C(16A)-C(17A)	85.5(2)

C(16A)-C(17A)-C(18A)	107.5(3)
C(16A)-C(17A)-C(20A)	88.7(3)
C(18A)-C(17A)-C(20A)	114.5(3)
C(19A)-C(18A)-C(17A)	113.8(3)
C(19A)-C(18A)-C(13A)	110.9(3)
C(17A)-C(18A)-C(13A)	110.8(3)
C(21A)-C(20A)-C(22A)	107.8(3)
C(21A)-C(20A)-C(15A)	119.8(3)
C(22A)-C(20A)-C(15A)	110.9(3)
C(21A)-C(20A)-C(17A)	122.7(3)
C(22A)-C(20A)-C(17A)	110.0(3)
C(15A)-C(20A)-C(17A)	83.9(2)
C(6B)-N(1B)-N(2B)	118.4(3)
C(12B)-N(2B)-N(1B)	116.9(3)
C(12B)-N(3B)-C(13C)	125.5(6)
C(12B)-N(3B)-C(13B)	119.8(4)
C(2B)-C(1B)-C(9B)	110.8(4)
C(2B)-C(1B)-C(6B)	108.2(3)
C(9B)-C(1B)-C(6B)	111.4(3)
C(2B)-C(1B)-C(7B)	110.5(3)
C(9B)-C(1B)-C(7B)	105.8(3)
C(6B)-C(1B)-C(7B)	110.1(3)
C(1B)-C(2B)-C(3B)	112.3(4)
C(4B)-C(3B)-C(2B)	111.2(4)
C(5B)-C(4B)-C(3B)	110.4(3)
C(4B)-C(5B)-C(6B)	111.6(3)
N(1B)-C(6B)-C(5B)	127.6(3)
N(1B)-C(6B)-C(1B)	116.7(3)
C(5B)-C(6B)-C(1B)	115.7(3)
C(8B)-C(7B)-C(1B)	114.8(3)
C(10B)-C(9B)-C(1B)	114.4(3)
C(11B)-C(10B)-C(9B)	126.1(4)
O(1B)-C(12B)-N(3B)	123.7(3)
O(1B)-C(12B)-N(2B)	120.2(3)
N(3B)-C(12B)-N(2B)	116.1(3)
C(14B)-C(13B)-C(18B)	114.7(7)
C(14B)-C(13B)-N(3B)	106.8(7)
C(18B)-C(13B)-N(3B)	111.4(7)
C(13B)-C(14B)-C(15B)	116.2(7)
C(20B)-C(15B)-C(14B)	111.9(7)
C(20B)-C(15B)-C(16B)	82.3(6)
C(14B)-C(15B)-C(16B)	100.8(8)
C(17B)-C(16B)-C(15B)	89.5(7)
C(18B)-C(17B)-C(16B)	108.2(8)
C(18B)-C(17B)-C(20B)	114.4(10)
C(16B)-C(17B)-C(20B)	82.9(7)
C(17B)-C(18B)-C(19B)	116.4(9)
C(17B)-C(18B)-C(13B)	114.7(8)
C(19B)-C(18B)-C(13B)	112.4(9)
C(21B)-C(20B)-C(22B)	110.8(7)
C(21B)-C(20B)-C(15B)	111.6(8)
C(22B)-C(20B)-C(15B)	115.9(7)
C(21B)-C(20B)-C(17B)	109.4(8)
C(22B)-C(20B)-C(17B)	116.5(9)
C(15B)-C(20B)-C(17B)	91.2(7)
N(3B)-C(13C)-C(18C)	105.0(9)
N(3B)-C(13C)-C(14C)	112.6(8)
C(18C)-C(13C)-C(14C)	113.5(8)
C(13C)-C(14C)-C(15C)	114.7(6)
C(20C)-C(15C)-C(16C)	86.4(8)
C(20C)-C(15C)-C(14C)	108.6(8)
C(16C)-C(15C)-C(14C)	108.6(7)
C(15C)-C(16C)-C(17C)	85.3(8)
C(18C)-C(17C)-C(20C)	117.4(8)
C(18C)-C(17C)-C(16C)	108.5(8)
C(20C)-C(17C)-C(16C)	85.8(8)
C(17C)-C(18C)-C(19C)	112.0(7)
C(17C)-C(18C)-C(13C)	113.6(7)
C(19C)-C(18C)-C(13C)	114.5(8)
C(22C)-C(20C)-C(17C)	117.7(10)
C(22C)-C(20C)-C(21C)	110.1(11)
C(17C)-C(20C)-C(21C)	112.8(10)
C(22C)-C(20C)-C(15C)	113.2(11)
C(17C)-C(20C)-C(15C)	87.8(8)
C(21C)-C(20C)-C(15C)	113.8(10)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB27 (CCDC 248956). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1A)	329(13)	309(13)	330(13)	-1(11)	10(11)	59(11)
N(1A)	235(16)	241(15)	326(17)	-5(13)	4(13)	24(13)
N(2A)	267(15)	245(15)	201(15)	-20(12)	-28(13)	47(13)
N(3A)	423(17)	357(17)	248(17)	44(14)	32(14)	173(14)
C(1A)	320(20)	218(19)	450(20)	-51(17)	42(18)	11(16)
C(2A)	460(20)	330(20)	610(30)	-30(20)	40(20)	-18(18)
C(3A)	510(20)	370(20)	580(30)	-210(20)	60(20)	35(18)
C(4A)	480(20)	620(30)	600(30)	-350(20)	-130(20)	80(20)
C(5A)	440(20)	450(20)	360(20)	-177(18)	-78(18)	138(19)
C(6A)	248(19)	238(19)	370(20)	6(17)	16(17)	-84(16)
C(7A)	260(20)	460(20)	450(20)	105(19)	38(17)	26(17)
C(8A)	560(30)	340(20)	560(20)	58(19)	40(20)	-201(19)
C(9A)	261(19)	410(20)	510(20)	10(20)	50(20)	45(17)
C(10A)	460(30)	430(30)	550(30)	150(20)	50(20)	150(20)
C(11A)	800(60)	420(50)	390(50)	90(40)	0(40)	410(40)
C(11C)	640(60)	940(80)	410(50)	-110(50)	50(50)	70(60)
C(12A)	268(19)	252(18)	300(20)	-59(17)	26(17)	-49(15)
C(13A)	390(20)	410(20)	229(18)	-16(17)	54(17)	86(18)
C(14A)	450(20)	320(20)	380(20)	-4(17)	100(19)	-82(18)
C(15A)	380(20)	480(20)	400(20)	-55(19)	10(19)	-66(19)
C(16A)	500(30)	750(30)	380(20)	60(20)	-150(20)	150(20)
C(17A)	560(30)	340(20)	280(20)	129(17)	30(20)	-97(18)
C(18A)	450(20)	318(19)	300(20)	2(17)	77(18)	-30(17)
C(19A)	670(30)	990(40)	660(30)	-280(30)	60(30)	-430(30)
C(20A)	370(20)	430(20)	330(20)	4(18)	87(18)	-30(18)
C(21A)	460(30)	650(30)	670(30)	-100(20)	130(20)	70(20)
C(22A)	790(30)	560(30)	490(20)	-140(20)	160(20)	-110(20)
O(1B)	331(13)	372(14)	312(13)	-13(11)	50(11)	46(11)
N(1B)	301(17)	392(18)	430(20)	56(15)	58(15)	132(15)
N(2B)	281(16)	544(19)	283(17)	-60(14)	-40(14)	184(16)
N(3B)	600(20)	950(30)	246(19)	125(18)	18(18)	350(20)
C(1B)	330(20)	410(20)	700(30)	180(20)	130(20)	206(19)
C(2B)	530(30)	830(40)	1270(50)	220(30)	380(30)	350(30)
C(3B)	960(40)	610(30)	850(40)	60(30)	500(30)	240(30)
C(4B)	620(30)	720(30)	560(30)	210(20)	250(20)	130(20)
C(5B)	520(30)	480(30)	620(30)	80(20)	120(20)	74(19)
C(6B)	390(20)	430(20)	550(30)	80(20)	80(20)	185(19)
C(7B)	620(30)	480(30)	1070(40)	150(30)	280(30)	170(20)
C(8B)	640(30)	760(30)	760(30)	370(30)	90(30)	-130(20)
C(9B)	470(30)	520(30)	1170(40)	280(30)	-120(30)	210(20)
C(10B)	400(30)	750(40)	1290(40)	260(30)	-230(30)	10(20)
C(11B)	550(30)	680(30)	1850(60)	500(30)	-40(40)	-50(30)
C(12B)	290(20)	390(20)	360(20)	43(19)	18(18)	29(17)
C(13B)	530(70)	260(60)	220(50)	-90(50)	-100(40)	130(60)
C(14B)	920(70)	720(60)	490(60)	-200(50)	130(50)	-240(60)
C(15B)	1490(100)	1050(80)	370(50)	170(60)	-200(60)	-640(80)

C(16B)	1720(100)	370(50)	380(50)	60(40)	-20(60)	-340(60)
C(17B)	1300(100)	830(90)	30(60)	40(60)	-110(60)	-60(80)
C(18B)	770(70)	1070(80)	570(60)	-190(60)	-230(60)	200(70)
C(19B)	890(80)	1720(120)	890(80)	240(80)	60(70)	280(90)
C(20B)	690(70)	560(70)	360(60)	-190(50)	-170(50)	-90(60)
C(21B)	1150(80)	1480(90)	120(40)	-130(50)	220(50)	-600(70)
C(22B)	520(50)	520(50)	770(60)	-320(50)	20(50)	80(40)
C(13C)	590(70)	350(60)	410(60)	-90(50)	-170(50)	-100(60)
C(14C)	1880(100)	280(50)	480(50)	0(40)	310(60)	-350(60)
C(15C)	1460(100)	550(70)	300(60)	220(50)	190(60)	370(70)
C(16C)	1400(110)	890(80)	950(90)	-210(70)	-550(80)	440(80)
C(17C)	1100(90)	470(60)	810(80)	-190(60)	-560(70)	-90(60)
C(18C)	460(50)	180(50)	410(60)	90(40)	-50(40)	-140(40)
C(19C)	960(70)	430(50)	550(60)	190(40)	170(50)	280(50)
C(20C)	1100(100)	800(90)	150(70)	120(60)	-160(60)	-80(80)
C(21C)	2130(140)	2230(140)	450(70)	0(80)	-370(80)	880(120)
C(22C)	1330(130)	620(90)	1040(110)	310(80)	270(100)	-550(80)

Table 5. Hydrogen bonds for DCB27 (CCDC 248956) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(2A)-H(2AA)...O(1B)	0.88	2.03	2.880(3)	161.8
N(2B)-H(2BA)...O(1A)	0.88	2.01	2.789(3)	146.4

CALIFORNIA INSTITUTE OF TECHNOLOGY

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Crystal Structure Analysis of:

Pd(II)(allyl)PHOX•PF_6 salt 356 (DCB24)

(CCDC 245187)

Contents:

Figure A8.3 Representation of Pd(II)(allyl)PHOX•PF_6 salt **356**

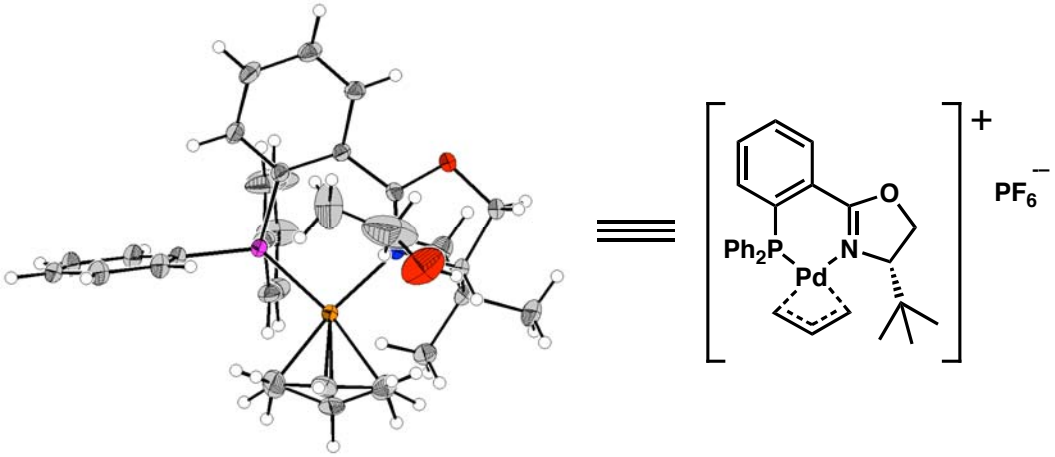

Figure A8.4 Representation of the unit cell of Pd(II)(allyl)PHOX•PF_6 salt **356**

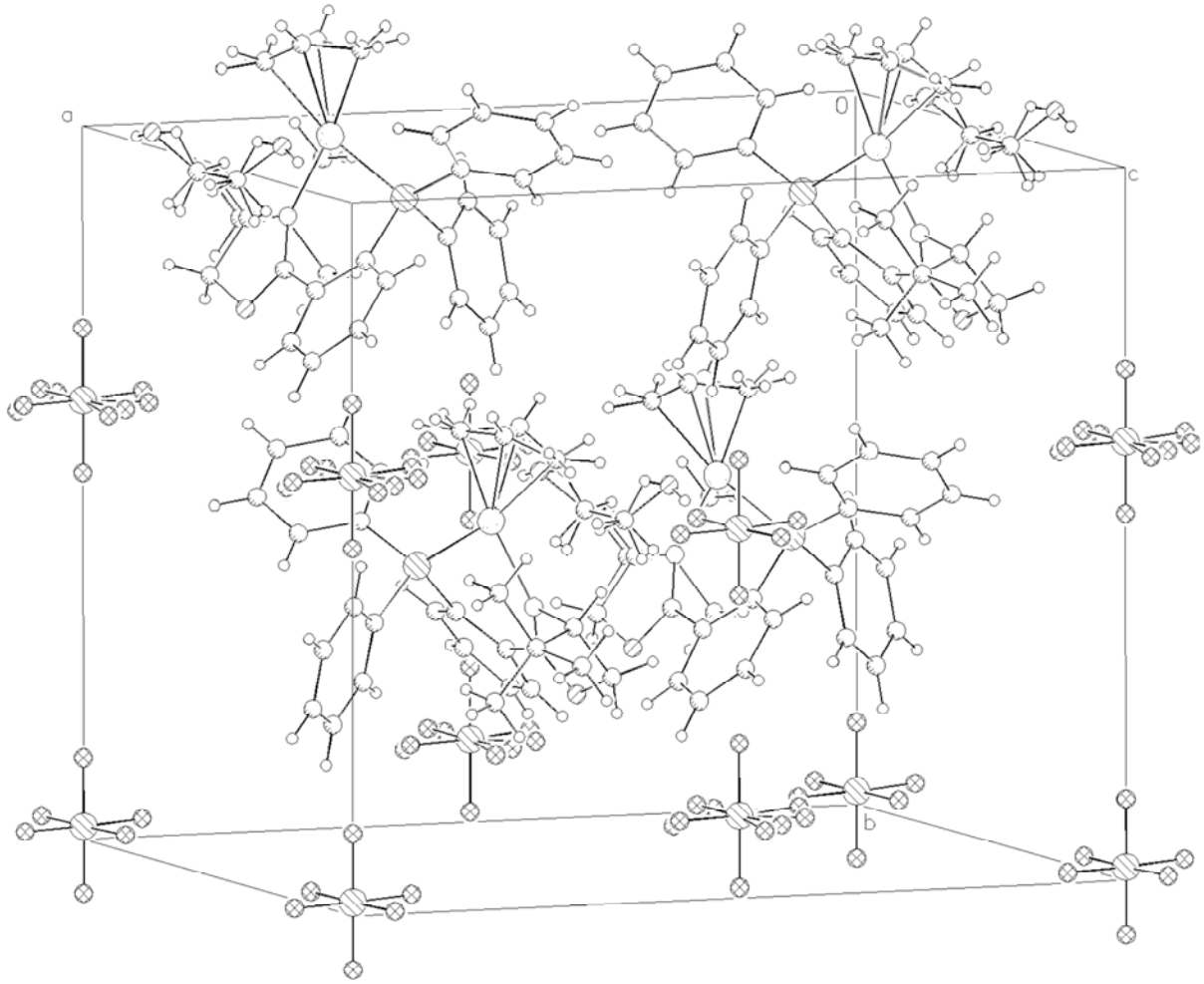

Table 1. Crystal data and structure refinement for DCB24 (CCDC 245187).

Empirical formula	$[C_{28}H_{31}NOPPd]^+ PF_6^- \bullet \frac{1}{2}C_2H_5OH$
Formula weight	702.91
Crystallization Solvent	Ethanol
Crystal Habit	Fragment
Crystal size	0.35 x 0.34 x 0.23 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 15322 reflections used in lattice determination	2.31 to 41.00°
Unit cell dimensions	a = 17.5183(6) Å b = 15.7792(5) Å β= 107.0990(10)° c = 11.3736(4) Å
Volume	3004.98(18) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	C2
Density (calculated)	1.554 Mg/m^3
F(000)	1428
θ range for data collection	1.77 to 42.31°
Completeness to θ = 42.31°	85.0 %
Index ranges	$-32 \le h \le 32$, $-28 \le k \le 29$, $-20 \le l \le 15$
Data collection scan type	ω scans at 3 ϕ settings of 2θ=-28° and 2 at 2θ=-59°
Reflections collected	28501
Independent reflections	15572 [R_{int}= 0.0351]
Absorption coefficient	0.787 mm^{-1}
Absorption correction	SADABS
Max. and min. transmission	0.8397 and 0.7702

Table 1 (cont.)

Structure Solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	15572 / 1 / 408
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.343
Final R indices [I>2σ(I), 13582 reflections]	R1 = 0.0373, *w*R2 = 0.0725
R indices (all data)	R1 = 0.0459, *w*R2 = 0.0748
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.004
Average shift/error	0.000
Absolute structure parameter	-0.019(13)
Largest diff. peak and hole	1.422 and -0.710 e.Å^{-3}

Special Refinement Details

The propyl ligand, C26-C27-C28, is disordered in two alternate orientations, differing by "up-down" positions for C27. Additional disorder is observed in one PF_6 counterion and an included solvent molecule, modeled as ethanol hydrogen bonded to a fluorine of one counterion.

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 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB24 (CCDC 245187). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Pd(1)	2838(1)	4922(1)	2994(1)	18(1)	1
P(1)	2327(1)	5673(1)	4282(1)	19(1)	1
O(1)	3784(1)	7349(1)	2545(1)	22(1)	1
N(1)	3187(1)	6087(1)	2419(2)	17(1)	1
C(1)	3099(1)	6431(1)	5056(2)	18(1)	1
C(2)	3275(1)	6569(1)	6320(2)	23(1)	1
C(3)	3845(1)	7170(1)	6920(2)	24(1)	1
C(4)	4208(1)	7669(1)	6242(2)	25(1)	1
C(5)	4040(1)	7544(1)	4982(2)	21(1)	1
C(6)	3512(1)	6903(1)	4381(2)	17(1)	1
C(7)	3458(1)	6748(1)	3074(2)	17(1)	1
C(8)	3836(1)	6994(1)	1381(2)	25(1)	1
C(9)	3264(1)	6242(1)	1155(2)	20(1)	1
C(10)	2441(1)	6383(1)	190(2)	23(1)	1
C(11)	2594(2)	6514(2)	-1064(2)	33(1)	1
C(12)	1931(1)	5586(2)	100(2)	29(1)	1
C(13)	2015(1)	7156(1)	510(2)	28(1)	1
C(14)	2024(1)	5153(1)	5500(2)	26(1)	1
C(15)	2543(2)	4578(2)	6254(2)	33(1)	1
C(16)	2337(2)	4162(2)	7193(2)	41(1)	1
C(17)	1591(2)	4315(2)	7372(3)	45(1)	1
C(18)	1070(2)	4884(2)	6615(2)	43(1)	1
C(19)	1277(2)	5303(2)	5676(2)	35(1)	1
C(20)	1492(1)	6337(1)	3474(2)	22(1)	1
C(21)	888(1)	5982(1)	2497(2)	24(1)	1
C(22)	256(1)	6481(2)	1823(2)	33(1)	1
C(23)	218(2)	7332(2)	2102(3)	50(1)	1
C(24)	812(2)	7686(2)	3061(4)	63(1)	1
C(25)	1439(1)	7192(2)	3747(3)	44(1)	1
C(26)	3314(2)	3965(2)	1935(3)	41(1)	1
C(27A)	3201(3)	3620(2)	2979(5)	30(1)	0.563(11)
C(27B)	2776(4)	3655(3)	2334(7)	32(2)	0.437(11)
C(28)	2576(3)	3683(2)	3388(3)	56(1)	1
P(2)	0	3840(1)	0	18(1)	1
F(1)	0	2828(1)	0	36(1)	1
F(2)	0	4853(2)	0	32(1)	1
F(3)	955(3)	3812(3)	530(5)	49(1)	0.77(3)
F(4)	-75(4)	3827(4)	1359(6)	51(1)	0.77(3)
F(3B)	900(13)	3927(11)	140(50)	100(9)	0.23(3)
F(4B)	170(30)	3897(16)	1430(20)	94(10)	0.23(3)
P(3)	5000	4813(1)	0	36(1)	1
F(5)	5000	3850(3)	0	210(4)	1
F(6)	5000	5758(3)	0	223(5)	1
F(7)	4919(1)	4802(3)	1329(2)	120(2)	1
F(8)	4076(1)	4798(4)	-481(2)	120(1)	1

C(30)	4962(4)	5145(5)	5617(6)	55(4)	0.50
C(31)	5035(8)	5160(5)	4359(7)	106(8)	0.50
O(30)	5453(3)	4644(3)	3892(5)	68(2)	0.50

Table 3. Selected bond lengths [Å] and angles [°] for DCB24 (CCDC 245187).

Pd(1)-C(28)	2.087(2)	C(28)-Pd(1)-N(1)	171.47(9)
Pd(1)-N(1)	2.1020(15)	C(28)-Pd(1)-C(27B)	37.19(19)
Pd(1)-C(27B)	2.127(4)	N(1)-Pd(1)-C(27B)	134.59(17)
Pd(1)-C(27A)	2.153(4)	C(28)-Pd(1)-C(27A)	36.07(16)
Pd(1)-C(26)	2.239(2)	N(1)-Pd(1)-C(27A)	135.98(13)
Pd(1)-P(1)	2.2639(5)	C(27B)-Pd(1)-C(27A)	23.7(2)
		C(28)-Pd(1)-C(26)	67.92(10)
		N(1)-Pd(1)-C(26)	103.55(9)
		C(27B)-Pd(1)-C(26)	33.40(17)
		C(27A)-Pd(1)-C(26)	36.35(15)
		C(28)-Pd(1)-P(1)	101.37(8)
		N(1)-Pd(1)-P(1)	87.15(5)
		C(27B)-Pd(1)-P(1)	136.98(16)
		C(27A)-Pd(1)-P(1)	132.75(14)
		C(26)-Pd(1)-P(1)	168.99(7)

Table 4. Bond lengths [Å] and angles [°] for DCB24 (CCDC 245187).

Pd(1)-C(28)	2.087(2)	P(3)-F(6)	1.492(5)
Pd(1)-N(1)	2.1020(15)	P(3)-F(5)	1.520(5)
Pd(1)-C(27B)	2.127(4)	P(3)-F(8)	1.549(2)
Pd(1)-C(27A)	2.153(4)	P(3)-F(8)#2	1.549(2)
Pd(1)-C(26)	2.239(2)	P(3)-F(7)#2	1.5592(19)
Pd(1)-P(1)	2.2639(5)	P(3)-F(7)	1.5592(19)
P(1)-C(20)	1.814(2)	C(30)-C(31)	1.4740
P(1)-C(14)	1.8179(19)	C(31)-O(30)	1.3080
P(1)-C(1)	1.8262(18)		
O(1)-C(7)	1.337(2)	C(28)-Pd(1)-N(1)	171.47(9)
O(1)-C(8)	1.465(2)	C(28)-Pd(1)-C(27B)	37.19(19)
N(1)-C(7)	1.287(2)	N(1)-Pd(1)-C(27B)	134.59(17)
N(1)-C(9)	1.502(2)	C(28)-Pd(1)-C(27A)	36.07(16)
C(1)-C(2)	1.397(3)	N(1)-Pd(1)-C(27A)	135.98(13)
C(1)-C(6)	1.412(2)	C(27B)-Pd(1)-C(27A)	23.7(2)
C(2)-C(3)	1.399(3)	C(28)-Pd(1)-C(26)	67.92(10)
C(3)-C(4)	1.382(3)	N(1)-Pd(1)-C(26)	103.55(9)
C(4)-C(5)	1.390(3)	C(27B)-Pd(1)-C(26)	33.40(17)
C(5)-C(6)	1.406(2)	C(27A)-Pd(1)-C(26)	36.35(15)
C(6)-C(7)	1.482(3)	C(28)-Pd(1)-P(1)	101.37(8)
C(8)-C(9)	1.526(3)	N(1)-Pd(1)-P(1)	87.15(5)
C(9)-C(10)	1.549(3)	C(27B)-Pd(1)-P(1)	136.98(16)
C(10)-C(13)	1.529(3)	C(27A)-Pd(1)-P(1)	132.75(14)
C(10)-C(12)	1.529(3)	C(26)-Pd(1)-P(1)	168.99(7)
C(10)-C(11)	1.541(3)	C(20)-P(1)-C(14)	105.92(10)
C(14)-C(15)	1.388(3)	C(20)-P(1)-C(1)	103.80(8)
C(14)-C(19)	1.401(3)	C(14)-P(1)-C(1)	105.63(9)
C(15)-C(16)	1.388(3)	C(20)-P(1)-Pd(1)	112.83(6)
C(16)-C(17)	1.401(4)	C(14)-P(1)-Pd(1)	121.10(7)
C(17)-C(18)	1.386(5)	C(1)-P(1)-Pd(1)	106.03(6)
C(18)-C(19)	1.391(3)	C(7)-O(1)-C(8)	106.45(14)
C(20)-C(25)	1.394(3)	C(7)-N(1)-C(9)	107.51(15)
C(20)-C(21)	1.407(3)	C(7)-N(1)-Pd(1)	128.28(14)
C(21)-C(22)	1.391(3)	C(9)-N(1)-Pd(1)	123.75(12)
C(22)-C(23)	1.386(4)	C(2)-C(1)-C(6)	118.94(16)
C(23)-C(24)	1.385(4)	C(2)-C(1)-P(1)	120.43(14)
C(24)-C(25)	1.385(4)	C(6)-C(1)-P(1)	120.61(14)
C(26)-C(27B)	1.259(6)	C(1)-C(2)-C(3)	121.19(18)
C(26)-C(27A)	1.372(6)	C(4)-C(3)-C(2)	119.57(19)
C(27A)-C(28)	1.314(6)	C(3)-C(4)-C(5)	120.13(18)
C(27B)-C(28)	1.345(7)	C(4)-C(5)-C(6)	120.84(18)
P(2)-F(3B)	1.543(18)	C(5)-C(6)-C(1)	119.03(17)
P(2)-F(3B)#1	1.543(18)	C(5)-C(6)-C(7)	116.43(15)
P(2)-F(4B)	1.57(2)	C(1)-C(6)-C(7)	124.49(15)
P(2)-F(4B)#1	1.57(2)	N(1)-C(7)-O(1)	116.61(16)
P(2)-F(4)	1.590(5)	N(1)-C(7)-C(6)	128.16(16)
P(2)-F(4)#1	1.590(5)	O(1)-C(7)-C(6)	115.06(14)
P(2)-F(2)	1.598(2)	O(1)-C(8)-C(9)	103.71(14)
P(2)-F(1)	1.5982(18)	N(1)-C(9)-C(8)	101.78(15)
P(2)-F(3)	1.603(4)	N(1)-C(9)-C(10)	112.07(15)
P(2)-F(3)#1	1.603(4)	C(8)-C(9)-C(10)	115.76(17)

C(13)-C(10)-C(12)	110.76(17)
C(13)-C(10)-C(11)	110.13(18)
C(12)-C(10)-C(11)	107.80(17)
C(13)-C(10)-C(9)	111.34(16)
C(12)-C(10)-C(9)	109.42(17)
C(11)-C(10)-C(9)	107.27(17)
C(15)-C(14)-C(19)	119.5(2)
C(15)-C(14)-P(1)	118.74(16)
C(19)-C(14)-P(1)	121.78(19)
C(14)-C(15)-C(16)	120.8(2)
C(15)-C(16)-C(17)	119.7(3)
C(18)-C(17)-C(16)	119.5(2)
C(17)-C(18)-C(19)	120.8(2)
C(18)-C(19)-C(14)	119.7(3)
C(25)-C(20)-C(21)	118.5(2)
C(25)-C(20)-P(1)	122.91(16)
C(21)-C(20)-P(1)	118.53(15)
C(22)-C(21)-C(20)	120.12(19)
C(23)-C(22)-C(21)	120.4(2)
C(24)-C(23)-C(22)	119.8(2)
C(25)-C(24)-C(23)	120.2(3)
C(24)-C(25)-C(20)	120.9(2)
C(28)-C(27A)-C(26)	128.5(5)
C(26)-C(27B)-C(28)	136.7(6)
F(3B)-P(2)-F(3B)#1	169.8(13)
F(3B)-P(2)-F(4B)	90.7(14)
F(3B)#1-P(2)-F(4B)	88.7(14)
F(3B)-P(2)-F(4B)#1	88.7(14)
F(3B)#1-P(2)-F(4B)#1	90.7(14)
F(4B)-P(2)-F(4B)#1	173.5(18)
F(3B)-P(2)-F(4)	105.8(18)
F(3B)#1-P(2)-F(4)	74.3(18)
F(4B)#1-P(2)-F(4)	165(2)
F(3B)-P(2)-F(4)#1	74.3(18)
F(3B)#1-P(2)-F(4)#1	105.8(18)
F(4)-P(2)-F(4)#1	178.4(5)
F(3B)-P(2)-F(2)	84.9(7)
F(3B)#1-P(2)-F(2)	84.9(7)
F(4B)-P(2)-F(2)	86.8(9)
F(4B)#1-P(2)-F(2)	86.8(9)
F(4)-P(2)-F(2)	90.8(3)
F(4)#1-P(2)-F(2)	90.8(3)
F(3B)-P(2)-F(1)	95.1(7)
F(3B)#1-P(2)-F(1)	95.1(7)
F(4B)-P(2)-F(1)	93.2(9)
F(4B)#1-P(2)-F(1)	93.2(9)
F(4)-P(2)-F(1)	89.2(3)
F(4)#1-P(2)-F(1)	89.2(3)
F(2)-P(2)-F(1)	180.0
F(3B)#1-P(2)-F(3)	164.4(18)
F(4B)-P(2)-F(3)	75.9(19)
F(4B)#1-P(2)-F(3)	104.3(19)
F(4)-P(2)-F(3)	90.6(3)
F(4)#1-P(2)-F(3)	89.4(3)
F(2)-P(2)-F(3)	91.6(2)
F(1)-P(2)-F(3)	88.4(2)
F(3B)-P(2)-F(3)#1	164.4(18)
F(4B)-P(2)-F(3)#1	104.3(19)
F(4B)#1-P(2)-F(3)#1	75.9(19)
F(4)-P(2)-F(3)#1	89.4(4)
F(4)#1-P(2)-F(3)#1	90.6(3)
F(2)-P(2)-F(3)#1	91.6(2)
F(1)-P(2)-F(3)#1	88.4(2)
F(3)-P(2)-F(3)#1	176.8(4)
F(6)-P(3)-F(5)	180.000(1)
F(6)-P(3)-F(8)	90.9(2)
F(5)-P(3)-F(8)	89.1(2)
F(6)-P(3)-F(8)#2	90.9(2)
F(5)-P(3)-F(8)#2	89.1(2)
F(8)-P(3)-F(8)#2	178.3(4)
F(6)-P(3)-F(7)#2	90.64(19)
F(5)-P(3)-F(7)#2	89.36(19)
F(8)-P(3)-F(7)#2	92.36(12)
F(8)#2-P(3)-F(7)#2	87.63(12)
F(6)-P(3)-F(7)	90.64(19)
F(5)-P(3)-F(7)	89.36(19)
F(8)-P(3)-F(7)	87.63(12)
F(8)#2-P(3)-F(7)	92.36(12)
F(7)#2-P(3)-F(7)	178.7(4)
O(30)-C(31)-C(30)	127.1

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

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB24 (CCDC 245187). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	249(1)	152(1)	169(1)	-17(1)	102(1)	-31(1)
P(1)	209(2)	217(2)	157(2)	-38(2)	86(2)	-57(2)
O(1)	270(6)	206(6)	201(7)	-11(5)	116(5)	-71(5)
N(1)	204(7)	184(6)	156(8)	-27(5)	87(6)	-26(5)
C(1)	165(7)	215(7)	168(9)	-20(6)	54(6)	-17(5)
C(2)	219(8)	303(9)	177(9)	-41(7)	90(6)	-23(6)
C(3)	205(8)	354(10)	166(9)	-79(7)	51(7)	-6(7)
C(4)	184(8)	309(9)	235(10)	-102(7)	43(6)	-28(6)
C(5)	183(7)	219(7)	217(9)	-44(6)	61(6)	-25(5)
C(6)	152(6)	186(6)	157(8)	-25(5)	45(5)	-7(5)
C(7)	156(6)	171(6)	187(8)	0(5)	69(5)	-16(5)
C(8)	339(10)	257(9)	205(10)	-22(7)	156(8)	-94(7)
C(9)	261(8)	209(7)	169(9)	-16(6)	122(6)	-43(6)
C(10)	297(9)	266(8)	146(9)	-36(6)	90(7)	-33(7)
C(11)	459(13)	381(12)	182(11)	8(8)	141(9)	-24(9)
C(12)	310(10)	332(10)	208(10)	-58(8)	62(7)	-86(8)
C(13)	301(10)	319(10)	203(10)	15(8)	67(7)	20(7)
C(14)	351(10)	292(9)	196(10)	-70(6)	156(8)	-132(7)
C(15)	479(13)	311(10)	261(12)	-10(8)	201(10)	-86(9)
C(16)	698(18)	313(11)	271(13)	-1(9)	233(12)	-136(11)
C(17)	830(20)	342(12)	308(13)	-148(10)	375(14)	-305(13)
C(18)	540(13)	487(13)	403(12)	-178(16)	351(11)	-278(15)
C(19)	403(12)	419(12)	300(12)	-104(9)	225(10)	-149(9)
C(20)	172(7)	261(8)	228(10)	-94(7)	67(6)	-52(6)
C(21)	232(8)	267(8)	226(10)	-87(7)	70(7)	-72(6)
C(22)	221(9)	419(12)	325(13)	-131(9)	26(8)	-40(8)
C(23)	261(11)	445(14)	680(20)	-170(14)	-48(11)	86(10)
C(24)	311(13)	421(15)	980(30)	-389(17)	-102(15)	122(11)
C(25)	221(10)	410(13)	591(18)	-309(12)	-44(10)	35(8)
C(26)	668(17)	230(10)	422(16)	-52(9)	325(14)	39(10)
C(27A)	400(30)	150(14)	370(30)	4(14)	120(20)	-16(13)
C(27B)	440(40)	154(18)	410(40)	-103(18)	190(30)	-37(18)
C(28)	1130(30)	165(9)	595(19)	10(10)	600(20)	-74(12)
P(2)	208(3)	164(2)	159(3)	0	52(2)	0
F(1)	453(11)	158(7)	586(14)	0	334(10)	0
F(2)	458(9)	159(8)	316(9)	0	61(7)	0
F(3)	201(11)	356(18)	770(30)	35(16)	-63(14)	-7(11)
F(4)	890(30)	440(20)	310(20)	47(14)	357(19)	125(16)
F(3B)	390(70)	340(50)	2400(300)	500(110)	640(110)	170(50)
F(4B)	2200(300)	320(60)	120(60)	130(40)	150(120)	130(130)
P(3)	413(4)	349(6)	402(5)	0	235(3)	0
F(5)	2790(100)	370(20)	3230(110)	0	1010(80)	0
F(6)	4250(130)	290(20)	3080(100)	0	2540(100)	0
F(7)	772(14)	2450(40)	439(11)	130(20)	293(10)	-630(20)
F(8)	562(12)	2360(50)	671(14)	-350(30)	185(11)	-300(20)

C(30)	250(30)	870(110)	530(50)	330(50)	120(30)	250(40)
C(31)	890(100)	540(90)	1800(200)	-350(90)	380(130)	-80(80)
O(30)	770(30)	560(30)	640(30)	-10(20)	130(30)	380(20)

Table 6. Hydrogen bonds for DCB24 (CCDC 245187) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(30)-H(30)...F(7)	0.85	1.99	2.799(6)	157.8

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