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Cruciform π-systems: Effect of aggregation on emission

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SUPPLEMENTARY INFORMATION

X-Ray Structure Determination, $C_{54}H_{60}N_2$

An orange bar was mounted onto the end of a thin glass fiber using inert oil. X-ray intensity data were measured at 200.0(2) K in the nitrogen cold stream of a Bruker SMART APEX CCD-based diffractometer (Mo Kα radiation, λ = 0.71073 Å).[1] Attempts to cool the crystal further resulted in fracturing and destruction of the sample. The raw data frames were integrated with SAINT+,[1] which also applied corrections for Lorentz and polarization effects. The final unit cell parameters are based on the least-squares refinement of 5751 reflections with I > 5(σ)I from the data set. Analysis of the data showed negligible crystal decay during data collection.

Systematic absences in the intensity data were consistent with the space group $P2_1/c$. The structure was solved by a combination of direct methods and difference Fourier syntheses, and refined by full-matrix least-squares against F^2, using the SHELXTL software package.[2] The molecule possesses no crystallographically imposed symmetry. Three of the four butyl groups (C19 – C22, C39 – C42, C43 – C46) are afflicted by disorder. These chains were modeled as occupying two conformations, A and B. The site occupation factors (sofs) for each disorder component were refined, constrained to sum to unity for that component. The final values are near A = 0.60 / B = 0.40 for all three. Additionally, a total of 38 restraints were employed to maintain a chemically reasonable geometry for the disordered groups; however, some displacement parameters of these atoms show highly anisotropic motion, indicating the twofold disorder model is inadequate, and that more conformations are present. These were not modeled. Eventually all non-hydrogen atoms were refined with anisotropic displacement parameters; hydrogen atoms were placed in geometrically calculated positions and refined as standard riding atoms.

(1) SMART Version 5.625 and SAINT+ Version 6.02a. Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1998.

(2) Sheldrick, G. M. SHELXTL Version 5.1; Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1997.

Table 1. Crystal data and structure refinement.

Identification code	j111a1s	
Empirical formula	C54 H60 N2	
Formula weight	737.04	
Temperature	200.0(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 14.7777(7) Å	α= 90°.
	b = 19.5961(9) Å	β= 115.6340(10)°.
	c = 16.7434(8) Å	γ = 90°.
Volume	4371.4(4) $Å^3$	
Z	4	
Density (calculated)	1.120 Mg/m^3	
Absorption coefficient	0.064 mm^{-1}	
F(000)	1592	
Crystal size	0.58 x 0.40 x 0.36 mm^3	
Theta range for data collection	1.70 to 23.28°.	
Index ranges	-14<=h<=16, -21<=k<=21, -18<=l<=18	
Reflections collected	26282	

Independent reflections	6288 [R(int) = 0.0406]
Completeness to theta = 23.28°	99.9 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6288 / 38 / 590
Goodness-of-fit on F^2	0.977
Final R indices [I>2sigma(I)]	R1 = 0.0541, wR2 = 0.1400
R indices (all data)	R1 = 0.0840, wR2 = 0.1519
Extinction coefficient	0.0037(5)
Largest diff. peak and hole	0.235 and -0.160 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for j111a1s. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	950(2)	5456(1)	6161(1)	67(1)
C(2)	1797(2)	5767(1)	6140(1)	69(1)
C(3)	2511(2)	5376(1)	6011(2)	72(1)
C(4)	2429(2)	4672(1)	5904(2)	71(1)
C(5)	1579(2)	4358(1)	5919(1)	69(1)
C(6)	866(2)	4748(1)	6033(2)	72(1)
C(7)	188(2)	5845(1)	6295(2)	72(1)
C(8)	-565(2)	5589(1)	6428(2)	74(1)
C(9)	-1379(2)	5932(1)	6530(2)	70(1)
C(10)	-2103(2)	5556(1)	6656(2)	79(1)
C(11)	-2908(2)	5845(1)	6740(2)	79(1)
C(12)	-3037(2)	6551(1)	6700(2)	84(1)
C(13)	-2311(3)	6931(1)	6578(2)	96(1)
C(14)	-1512(2)	6638(1)	6496(2)	85(1)
C(15)	-4621(2)	6453(2)	6872(2)	103(1)
C(16)	-4285(3)	6245(2)	7850(2)	122(1)
C(17)	-4950(3)	5816(2)	8051(3)	142(1)
C(18)	-4572(3)	5664(2)	9019(2)	151(2)
N(1A)	-3847(2)	6847(1)	6767(2)	113(1)
C(19A)	-3664(7)	7624(3)	7142(6)	103(2)
C(20A)	-4252(5)	8044(4)	6351(5)	130(2)
C(21A)	-4217(13)	8825(6)	6623(12)	179(6)
C(22A)	-3301(13)	9072(9)	6658(14)	228(9)
N(1B)	-3847(2)	6847(1)	6767(2)	113(1)
C(19B)	-4257(5)	7505(4)	6354(5)	65(2)
C(20B)	-3866(9)	7990(4)	7146(6)	87(3)
C(21B)	-4114(12)	8733(4)	6800(11)	85(4)
C(22B)	-3294(7)	9011(7)	6665(10)	77(4)
C(23)	1949(2)	6497(1)	6262(2)	69(1)
C(24)	2073(2)	7092(1)	6348(2)	72(1)
C(25)	2222(2)	7822(1)	6441(2)	67(1)
C(26)	1412(2)	8257(1)	6115(2)	80(1)
C(27)	1549(2)	8949(1)	6211(2)	92(1)
C(28)	2496(3)	9218(1)	6633(2)	87(1)
C(29)	3308(2)	8793(2)	6957(2)	89(1)
C(30)	3176(2)	8096(1)	6859(2)	80(1)
C(31)	3186(2)	4239(1)	5808(2)	83(1)
C(32)	4019(2)	4415(1)	5792(2)	81(1)
C(33)	4792(2)	3952(1)	5752(2)	76(1)
C(34)	5621(2)	4200(1)	5684(2)	78(1)
C(35)	6366(2)	3778(1)	5682(2)	75(1)
C(36)	6329(2)	3068(1)	5768(2)	75(1)
C(37)	5472(3)	2813(1)	5826(2)	93(1)
C(38)	4726(2)	3247(2)	5814(2)	87(1)
C(39A)	7948(2)	2919(1)	5732(2)	85(1)

C(40A)	8637(5)	3124(3)	6726(4)	76(2)
C(41A)	9511(5)	3531(3)	6748(5)	98(2)
C(42A)	10299(15)	3725(10)	7639(9)	119(4)
C(39B)	7948(2)	2919(1)	5732(2)	85(1)
C(40B)	8816(8)	3359(6)	6300(8)	113(4)
C(41B)	9474(11)	3045(5)	7110(6)	113(4)
C(42B)	10280(20)	3518(16)	7738(18)	133(9)
C(43A)	7242(7)	1953(3)	6227(5)	95(2)
C(44A)	7030(8)	1449(3)	5581(5)	144(3)
C(45A)	7354(4)	740(2)	6054(3)	156(2)
C(46A)	8302(3)	479(2)	6135(3)	155(2)
C(43B)	6900(8)	1919(5)	5621(10)	107(4)
C(44B)	7546(11)	1506(6)	6276(12)	244(12)
C(45B)	7354(4)	740(2)	6054(3)	156(2)
C(46B)	8302(3)	479(2)	6135(3)	155(2)
C(47)	1451(2)	3625(1)	5823(2)	72(1)
C(48)	1384(2)	3027(1)	5764(2)	71(1)
C(49)	1338(2)	2296(1)	5673(2)	69(1)
C(50)	1771(2)	1979(1)	5190(2)	86(1)
C(51)	1738(3)	1283(2)	5088(2)	106(1)
C(52)	1281(3)	892(2)	5466(3)	111(1)
C(53)	860(3)	1184(2)	5970(2)	114(1)
C(54)	892(2)	1892(2)	6075(2)	97(1)
N(2)	7084(2)	2651(1)	5786(2)	99(1)

Table 3. Bond lengths [Å] and angles [°].

C(1)-C(6)	1.401(3)	C(26)-C(27)	1.371(3)
C(1)-C(2)	1.407(3)	C(27)-C(28)	1.371(4)
C(1)-C(7)	1.457(3)	C(28)-C(29)	1.365(4)
C(2)-C(3)	1.394(3)	C(29)-C(30)	1.378(3)
C(2)-C(23)	1.448(3)	C(31)-C(32)	1.290(3)
C(3)-C(4)	1.391(3)	C(32)-C(33)	1.482(4)
C(4)-C(5)	1.409(3)	C(33)-C(34)	1.368(3)
C(4)-C(31)	1.465(3)	C(33)-C(38)	1.392(3)
C(5)-C(6)	1.380(3)	C(34)-C(35)	1.378(3)
C(5)-C(47)	1.449(3)	C(35)-C(36)	1.402(3)
C(7)-C(8)	1.323(3)	C(36)-N(2)	1.372(3)
C(8)-C(9)	1.451(3)	C(36)-C(37)	1.403(4)
C(9)-C(10)	1.387(3)	C(37)-C(38)	1.386(4)
C(9)-C(14)	1.395(3)	C(39A)-N(2)	1.419(3)
C(10)-C(11)	1.379(3)	C(39A)-C(40A)	1.582(6)
C(11)-C(12)	1.393(3)	C(40A)-C(41A)	1.504(9)
C(12)-N(1A)	1.379(4)	C(41A)-C(42A)	1.491(12)
C(12)-C(13)	1.392(4)	C(40B)-C(41B)	1.422(14)
C(13)-C(14)	1.372(4)	C(41B)-C(42B)	1.515(17)
C(15)-N(1A)	1.453(4)	C(43A)-C(44A)	1.397(8)
C(15)-C(16)	1.546(4)	C(43A)-N(2)	1.525(7)
C(16)-C(17)	1.439(4)	C(44A)-C(45A)	1.568(8)
C(17)-C(18)	1.498(4)	C(45A)-C(46A)	1.441(5)
N(1A)-C(19A)	1.625(7)	C(43B)-C(44B)	1.366(15)
C(19A)-C(20A)	1.479(10)	C(43B)-N(2)	1.463(11)
C(20A)-C(21A)	1.592(9)	C(47)-C(48)	1.176(3)
C(21A)-C(22A)	1.416(15)	C(48)-C(49)	1.438(4)
C(19B)-C(20B)	1.527(12)	C(49)-C(50)	1.377(3)
C(20B)-C(21B)	1.551(10)	C(49)-C(54)	1.377(3)
C(21B)-C(22B)	1.433(14)	C(50)-C(51)	1.374(4)
C(23)-C(24)	1.180(3)	C(51)-C(52)	1.346(4)
C(24)-C(25)	1.446(3)	C(52)-C(53)	1.370(4)
C(25)-C(26)	1.375(3)	C(53)-C(54)	1.397(4)
C(25)-C(30)	1.382(3)		

C(6)-C(1)-C(2)	116.5(2)	C(27)-C(26)-C(25)	120.5(3)
C(6)-C(1)-C(7)	121.2(3)	C(26)-C(27)-C(28)	120.4(3)
C(2)-C(1)-C(7)	122.2(2)	C(29)-C(28)-C(27)	119.8(3)
C(3)-C(2)-C(1)	120.5(2)	C(28)-C(29)-C(30)	120.2(3)
C(3)-C(2)-C(23)	118.9(2)	C(29)-C(30)-C(25)	120.3(3)
C(1)-C(2)-C(23)	120.6(2)	C(32)-C(31)-C(4)	128.9(3)
C(4)-C(3)-C(2)	122.4(3)	C(31)-C(32)-C(33)	126.8(3)
C(3)-C(4)-C(5)	117.4(2)	C(34)-C(33)-C(38)	116.9(3)
C(3)-C(4)-C(31)	124.0(3)	C(34)-C(33)-C(32)	121.5(2)
C(5)-C(4)-C(31)	118.6(2)	C(38)-C(33)-C(32)	121.6(3)
C(6)-C(5)-C(4)	120.1(2)	C(33)-C(34)-C(35)	122.2(2)
C(6)-C(5)-C(47)	119.6(2)	C(34)-C(35)-C(36)	121.9(3)
C(4)-C(5)-C(47)	120.3(2)	N(2)-C(36)-C(35)	121.7(3)
C(5)-C(6)-C(1)	123.2(3)	N(2)-C(36)-C(37)	122.4(3)
C(8)-C(7)-C(1)	126.1(2)	C(35)-C(36)-C(37)	115.9(3)
C(7)-C(8)-C(9)	130.1(2)	C(38)-C(37)-C(36)	121.1(3)
C(10)-C(9)-C(14)	115.3(3)	C(37)-C(38)-C(33)	122.0(3)
C(10)-C(9)-C(8)	120.4(2)	N(2)-C(39A)-C(40A)	102.4(3)
C(14)-C(9)-C(8)	124.3(3)	C(41A)-C(40A)-C(39A)	108.5(6)
C(11)-C(10)-C(9)	123.6(2)	C(42A)-C(41A)-C(40A)	116.8(10)
C(10)-C(11)-C(12)	120.6(3)	C(40B)-C(41B)-C(42B)	114.0(16)
N(1A)-C(12)-C(13)	122.5(3)	C(44A)-C(43A)-N(2)	108.9(6)
N(1A)-C(12)-C(11)	121.3(3)	C(43A)-C(44A)-C(45A)	108.6(6)
C(13)-C(12)-C(11)	116.2(3)	C(46A)-C(45A)-C(44A)	115.7(5)
C(14)-C(13)-C(12)	122.8(3)	C(44B)-C(43B)-N(2)	114.9(12)
C(13)-C(14)-C(9)	121.6(3)	C(48)-C(47)-C(5)	177.6(3)
N(1A)-C(15)-C(16)	111.1(3)	C(47)-C(48)-C(49)	177.1(3)
C(17)-C(16)-C(15)	117.6(3)	C(50)-C(49)-C(54)	118.0(3)
C(16)-C(17)-C(18)	113.4(3)	C(50)-C(49)-C(48)	119.9(2)
C(12)-N(1A)-C(15)	122.9(2)	C(54)-C(49)-C(48)	122.0(3)
C(12)-N(1A)-C(19A)	114.9(4)	C(51)-C(50)-C(49)	121.3(3)
C(15)-N(1A)-C(19A)	117.3(4)	C(52)-C(51)-C(50)	120.3(3)
C(20A)-C(19A)-N(1A)	103.9(7)	C(51)-C(52)-C(53)	120.4(3)
C(19A)-C(20A)-C(21A)	110.2(10)	C(52)-C(53)-C(54)	119.5(3)
C(22A)-C(21A)-C(20A)	104.6(12)	C(49)-C(54)-C(53)	120.5(3)
C(19B)-C(20B)-C(21B)	108.7(8)	C(36)-N(2)-C(39A)	121.6(2)
C(22B)-C(21B)-C(20B)	109.6(10)	C(36)-N(2)-C(43B)	119.8(6)
C(24)-C(23)-C(2)	178.8(2)	C(39A)-N(2)-C(43B)	116.7(6)
C(23)-C(24)-C(25)	179.3(2)	C(36)-N(2)-C(43A)	119.8(3)
C(26)-C(25)-C(30)	118.8(2)	C(39A)-N(2)-C(43A)	114.1(4)
C(26)-C(25)-C(24)	120.3(2)	C(43B)-N(2)-C(43A)	35.8(4)
C(30)-C(25)-C(24)	120.9(2)		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). 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}
C(1)	77(2)	63(2)	59(2)	2(1)	27(1)	1(1)
C(2)	81(2)	66(2)	57(2)	1(1)	28(1)	0(2)
C(3)	75(2)	73(2)	66(2)	-4(1)	29(1)	-8(1)
C(4)	78(2)	70(2)	63(2)	-5(1)	28(1)	5(2)
C(5)	73(2)	65(2)	63(2)	-2(1)	24(1)	-2(2)
C(6)	76(2)	66(2)	71(2)	1(1)	29(1)	0(1)
C(7)	88(2)	60(1)	67(2)	2(1)	32(2)	6(2)
C(8)	87(2)	61(1)	75(2)	4(1)	35(2)	6(2)
C(9)	82(2)	59(2)	66(2)	4(1)	31(1)	10(2)
C(10)	92(2)	59(1)	89(2)	-1(1)	43(2)	3(2)
C(11)	87(2)	69(2)	85(2)	-1(1)	40(2)	5(2)
C(12)	95(2)	77(2)	86(2)	12(1)	46(2)	15(2)

C(13)	127(3)	62(2)	122(2)	19(2)	76(2)	24(2)
C(14)	100(2)	68(2)	101(2)	14(1)	56(2)	8(2)
C(15)	89(2)	107(2)	113(3)	-5(2)	43(2)	9(2)
C(16)	133(3)	133(3)	121(3)	-23(2)	75(3)	-32(2)
C(17)	133(3)	146(3)	149(4)	0(3)	63(3)	-10(3)
C(18)	152(4)	195(4)	108(3)	26(3)	57(3)	14(3)
N(1A)	134(3)	78(2)	163(3)	26(2)	99(2)	35(2)
C(19A)	110(5)	98(6)	99(5)	15(4)	42(4)	33(4)
C(20A)	154(6)	88(5)	123(6)	-2(4)	36(5)	23(4)
C(21A)	184(14)	183(14)	149(9)	16(8)	53(9)	-38(10)
C(22A)	320(20)	143(13)	204(18)	17(11)	102(15)	-43(12)
N(1B)	134(3)	78(2)	163(3)	26(2)	99(2)	35(2)
C(19B)	66(5)	72(6)	54(5)	-3(3)	22(4)	11(4)
C(20B)	94(7)	100(9)	47(5)	11(5)	11(4)	20(6)
C(21B)	110(10)	42(5)	126(10)	-19(5)	71(8)	9(6)
C(22B)	57(6)	71(7)	113(10)	27(7)	46(6)	12(6)
C(23)	81(2)	67(2)	61(2)	-1(1)	32(1)	-3(1)
C(24)	84(2)	76(2)	62(2)	-3(1)	37(1)	-3(2)
C(25)	84(2)	64(2)	57(1)	-3(1)	35(1)	-5(2)
C(26)	79(2)	72(2)	83(2)	7(1)	28(2)	-10(2)
C(27)	95(2)	73(2)	100(2)	12(2)	34(2)	5(2)
C(28)	108(3)	65(2)	84(2)	-4(1)	37(2)	-6(2)
C(29)	89(2)	87(2)	83(2)	-16(2)	29(2)	-20(2)
C(30)	78(2)	77(2)	80(2)	-10(1)	30(2)	-1(2)
C(31)	84(2)	84(2)	81(2)	-10(1)	36(2)	-17(2)
C(32)	97(2)	68(2)	73(2)	-1(1)	32(2)	-5(2)
C(33)	84(2)	67(2)	69(2)	-4(1)	27(2)	-3(2)
C(34)	86(2)	72(2)	81(2)	1(1)	40(2)	-3(2)
C(35)	84(2)	71(2)	75(2)	0(1)	39(2)	-3(2)
C(36)	87(2)	70(2)	78(2)	-6(1)	43(2)	1(2)
C(37)	102(2)	74(2)	114(2)	-13(2)	58(2)	-14(2)
C(38)	76(2)	94(2)	95(2)	-11(2)	40(2)	-19(2)
C(39A)	98(3)	79(2)	89(2)	1(2)	52(2)	5(2)
C(40A)	78(4)	73(3)	87(4)	14(3)	46(4)	0(3)
C(41A)	93(6)	84(4)	121(6)	7(4)	49(5)	-9(4)
C(42A)	103(8)	140(13)	100(6)	3(6)	31(5)	-44(6)
C(39B)	98(3)	79(2)	89(2)	1(2)	52(2)	5(2)
C(40B)	107(10)	106(8)	128(10)	11(8)	53(8)	-1(8)
C(41B)	126(11)	95(7)	122(8)	-1(6)	56(8)	11(7)
C(42B)	92(11)	119(14)	191(19)	7(10)	65(11)	-47(10)
C(43A)	130(6)	79(4)	98(4)	-9(4)	68(5)	8(4)
C(44A)	232(10)	90(4)	103(5)	-4(4)	66(6)	19(5)
C(45A)	188(5)	67(2)	214(5)	14(2)	87(4)	-9(2)
C(46A)	165(4)	171(4)	125(3)	-9(3)	60(3)	26(3)
C(43B)	90(7)	73(8)	120(10)	-4(8)	11(8)	26(6)
C(44B)	218(16)	78(8)	193(15)	25(9)	-140(12)	2(8)
C(45B)	188(5)	67(2)	214(5)	14(2)	87(4)	-9(2)
C(46B)	165(4)	171(4)	125(3)	-9(3)	60(3)	26(3)
C(47)	72(2)	71(2)	71(2)	1(1)	30(1)	4(1)
C(48)	63(2)	72(2)	74(2)	1(1)	26(1)	1(1)
C(49)	65(2)	67(2)	71(2)	7(1)	24(1)	-1(1)
C(50)	92(2)	69(2)	95(2)	6(1)	40(2)	7(2)
C(51)	123(3)	76(2)	111(2)	5(2)	43(2)	17(2)
C(52)	122(3)	73(2)	106(3)	12(2)	19(2)	2(2)
C(53)	111(3)	96(3)	113(3)	34(2)	30(2)	-25(2)
C(54)	102(2)	94(2)	93(2)	11(2)	40(2)	-9(2)
N(2)	117(2)	65(1)	146(2)	6(1)	87(2)	8(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3)

	x	y	z	U(eq)
H(3)	3074	5599	5996	86
H(6)	291	4526	6025	86
H(7)	235	6328	6287	86
H(8)	-573	5105	6462	89
H(10)	-2040	5073	6686	94
H(11)	-3379	5561	6826	94
H(13)	-2371	7414	6549	115
H(14)	-1037	6922	6416	102
H(15A)	-4758	6036	6504	124
H(15B)	-5250	6720	6664	124
H(16A)	-3632	6007	8054	146
H(16B)	-4169	6668	8205	146
H(17A)	-5047	5381	7724	171
H(17B)	-5612	6043	7840	171
H(18A)	-3966	5382	9216	227
H(18B)	-5090	5417	9122	227
H(18C)	-4414	6092	9354	227
H(19A)	-3910	7685	7602	124
H(19B)	-2943	7745	7396	124
H(20A)	-4957	7884	6073	156
H(20B)	-3973	7993	5913	156
H(21A)	-4794	9079	6177	214
H(21B)	-4230	8870	7207	214
H(22A)	-2758	8986	7248	342
H(22B)	-3356	9564	6540	342
H(22C)	-3153	8838	6211	342
H(19C)	-5000	7497	6061	79
H(19D)	-4005	7634	5915	79
H(20C)	-3131	7935	7487	104
H(20D)	-4187	7885	7542	104
H(21C)	-4731	8739	6236	102
H(21D)	-4234	9015	7236	102
H(22D)	-2662	8816	7099	116
H(22E)	-3279	9508	6737	116
H(22F)	-3380	8900	6065	116
H(26)	753	8076	5822	96
H(27)	984	9245	5983	111
H(28)	2588	9698	6701	105
H(29)	3965	8977	7250	107
H(30)	3744	7803	7080	96
H(31)	3042	3764	5750	100
H(32)	4152	4890	5808	97
H(34)	5687	4679	5638	94
H(35)	6920	3973	5619	90
H(37)	5403	2335	5875	111
H(38)	4152	3058	5848	104
H(39A)	7779	3321	5336	102
H(39B)	8276	2570	5518	102
H(40A)	8249	3399	6965	91
H(40B)	8882	2709	7095	91
H(41A)	9835	3265	6441	118
H(41B)	9245	3954	6403	118
H(42A)	10012	4038	7925	179
H(42B)	10855	3951	7574	179
H(42C)	10547	3315	8004	179
H(39C)	7666	3160	5156	102

H(39D)	8277	2507	5636	102
H(40C)	9202	3479	5962	135
H(40D)	8555	3787	6434	135
H(41C)	9801	2649	6976	136
H(41D)	9073	2871	7411	136
H(42D)	10733	3648	7476	199
H(42E)	10661	3283	8302	199
H(42F)	9965	3927	7842	199
H(43A)	7945	1908	6680	114
H(43B)	6794	1900	6523	114
H(44A)	7402	1542	5222	173
H(44B)	6304	1446	5181	173
H(45A)	7387	779	6655	188
H(45B)	6826	402	5723	188
H(46A)	8351	546	5575	232
H(46B)	8349	-9	6274	232
H(46C)	8851	722	6610	232
H(43C)	6208	1819	5535	128
H(43D)	6942	1805	5062	128
H(44C)	7481	1600	6830	293
H(44D)	8243	1617	6382	293
H(45C)	7149	508	6475	188
H(45D)	6823	674	5445	188
H(46D)	8331	517	5563	232
H(46E)	8369	-1	6315	232
H(46F)	8850	743	6581	232
H(50)	2099	2248	4921	103
H(51)	2039	1075	4749	128
H(52)	1250	412	5384	133
H(53)	549	907	6245	136
H(54)	604	2097	6425	117

Table 6. Torsion angles [°]

C(6)-C(1)-C(2)-C(3)	-1.0(3)	C(11)-C(12)-C(13)-C(14)	0.3(4)
C(7)-C(1)-C(2)-C(3)	179.9(2)	C(12)-C(13)-C(14)-C(9)	0.2(4)
C(6)-C(1)-C(2)-C(23)	179.8(2)	C(10)-C(9)-C(14)-C(13)	-0.5(4)
C(7)-C(1)-C(2)-C(23)	0.7(3)	C(8)-C(9)-C(14)-C(13)	178.5(2)
C(1)-C(2)-C(3)-C(4)	-0.4(3)	N(1A)-C(15)-C(16)-C(17)	-176.4(3)
C(23)-C(2)-C(3)-C(4)	178.8(2)	C(15)-C(16)-C(17)-C(18)	-178.0(3)
C(2)-C(3)-C(4)-C(5)	0.7(3)	C(13)-C(12)-N(1A)-C(15)	178.4(3)
C(2)-C(3)-C(4)-C(31)	-177.1(2)	C(11)-C(12)-N(1A)-C(15)	-0.7(4)
C(3)-C(4)-C(5)-C(6)	0.3(3)	C(13)-C(12)-N(1A)-C(19A)	-26.9(5)
C(31)-C(4)-C(5)-C(6)	178.2(2)	C(11)-C(12)-N(1A)-C(19A)	153.9(4)
C(3)-C(4)-C(5)-C(47)	-179.2(2)	C(16)-C(15)-N(1A)-C(12)	81.7(4)
C(31)-C(4)-C(5)-C(47)	-1.3(3)	C(16)-C(15)-N(1A)-C(19A)	-72.3(4)
C(4)-C(5)-C(6)-C(1)	-1.7(3)	C(12)-N(1A)-C(19A)-C(20A)	106.8(6)
C(47)-C(5)-C(6)-C(1)	177.8(2)	C(15)-N(1A)-C(19A)-C(20A)	-97.1(6)
C(2)-C(1)-C(6)-C(5)	2.1(3)	N(1A)-C(19A)-C(20A)-C(21A)	175.8(8)
C(7)-C(1)-C(6)-C(5)	-178.8(2)	C(19A)-C(20A)-C(21A)-C(22A)	82.7(16)
C(6)-C(1)-C(7)-C(8)	9.4(4)	C(19B)-C(20B)-C(21B)-C(22B)	-91.3(14)
C(2)-C(1)-C(7)-C(8)	-171.5(2)	C(3)-C(2)-C(23)-C(24)	58(15)
C(1)-C(7)-C(8)-C(9)	-177.0(2)	C(1)-C(2)-C(23)-C(24)	-123(15)
C(7)-C(8)-C(9)-C(10)	179.4(2)	C(2)-C(23)-C(24)-C(25)	16(33)
C(7)-C(8)-C(9)-C(14)	0.4(4)	C(23)-C(24)-C(25)-C(26)	73(22)
C(14)-C(9)-C(10)-C(11)	0.4(4)	C(23)-C(24)-C(25)-C(30)	-107(22)
C(8)-C(9)-C(10)-C(11)	-178.7(2)	C(30)-C(25)-C(26)-C(27)	-0.5(4)
C(9)-C(10)-C(11)-C(12)	0.1(4)	C(24)-C(25)-C(26)-C(27)	179.2(2)
C(10)-C(11)-C(12)-N(1A)	178.8(3)	C(25)-C(26)-C(27)-C(28)	-0.1(4)
C(10)-C(11)-C(12)-C(13)	-0.4(4)	C(26)-C(27)-C(28)-C(29)	0.3(4)
N(1A)-C(12)-C(13)-C(14)	-178.9(3)	C(27)-C(28)-C(29)-C(30)	0.0(4)

C(28)-C(29)-C(30)-C(25)	-0.6(4)	C(47)-C(48)-C(49)-C(50)	-12(6)
C(26)-C(25)-C(30)-C(29)	0.8(4)	C(47)-C(48)-C(49)-C(54)	167(5)
C(24)-C(25)-C(30)-C(29)	-178.9(2)	C(54)-C(49)-C(50)-C(51)	1.7(4)
C(3)-C(4)-C(31)-C(32)	-1.0(4)	C(48)-C(49)-C(50)-C(51)	-179.8(2)
C(5)-C(4)-C(31)-C(32)	-178.8(3)	C(49)-C(50)-C(51)-C(52)	-0.3(5)
C(4)-C(31)-C(32)-C(33)	175.7(2)	C(50)-C(51)-C(52)-C(53)	-1.2(5)
C(31)-C(32)-C(33)-C(34)	176.0(3)	C(51)-C(52)-C(53)-C(54)	1.2(5)
C(31)-C(32)-C(33)-C(38)	-6.4(4)	C(50)-C(49)-C(54)-C(53)	-1.7(4)
C(38)-C(33)-C(34)-C(35)	-0.3(4)	C(48)-C(49)-C(54)-C(53)	179.8(2)
C(32)-C(33)-C(34)-C(35)	177.4(2)	C(52)-C(53)-C(54)-C(49)	0.3(5)
C(33)-C(34)-C(35)-C(36)	-1.5(4)	C(35)-C(36)-N(2)-C(39A)	1.1(4)
C(34)-C(35)-C(36)-N(2)	-178.5(2)	C(37)-C(36)-N(2)-C(39A)	-179.5(3)
C(34)-C(35)-C(36)-C(37)	2.1(4)	C(35)-C(36)-N(2)-C(43B)	-162.5(7)
N(2)-C(36)-C(37)-C(38)	179.5(3)	C(37)-C(36)-N(2)-C(43B)	16.9(8)
C(35)-C(36)-C(37)-C(38)	-1.1(4)	C(35)-C(36)-N(2)-C(43A)	156.1(4)
C(36)-C(37)-C(38)-C(33)	-0.6(4)	C(37)-C(36)-N(2)-C(43A)	-24.6(5)
C(34)-C(33)-C(38)-C(37)	1.4(4)	C(40A)-C(39A)-N(2)-C(36)	83.5(4)
C(32)-C(33)-C(38)-C(37)	-176.3(2)	C(40A)-C(39A)-N(2)-C(43B)	-112.5(7)
N(2)-C(39A)-C(40A)-C(41A)	-170.1(5)	C(40A)-C(39A)-N(2)-C(43A)	-72.8(4)
C(39A)-C(40A)-C(41A)-C(42A)	-175.1(11)	C(44B)-C(43B)-N(2)-C(36)	-124.0(16)
N(2)-C(43A)-C(44A)-C(45A)	171.2(5)	C(44B)-C(43B)-N(2)-C(39A)	72(2)
C(43A)-C(44A)-C(45A)-C(46A)	-100.6(7)	C(44B)-C(43B)-N(2)-C(43A)	-23.2(13)
C(6)-C(5)-C(47)-C(48)	-140(6)	C(44A)-C(43A)-N(2)-C(36)	114.2(6)
C(4)-C(5)-C(47)-C(48)	40(6)	C(44A)-C(43A)-N(2)-C(39A)	-89.1(8)
C(5)-C(47)-C(48)-C(49)	-58(10)	C(44A)-C(43A)-N(2)-C(43B)	13.6(10)

X-Ray Structure Determination, $C_{56}H_{58}F_6N_2$

$C_{56}H_{58}F_6N_2$ crystallizes in the form of brilliant orange plates. Evaluation of several samples showed broad diffraction peaks and weak scattering > 2θ ~ 40º. The crystal judged to be the best of these was mounted onto the end of a thin glass fiber using inert oil. X-ray intensity data were measured at 150.0(2) K on a Bruker SMART APEX CCD-based diffractometer (Mo Kα radiation, λ = 0.71073 Å).[1] The raw data frames were integrated into reflection intensity files with SAINT+,[1] which also applied corrections for Lorentz and polarization effects. The final unit cell parameters are based on the least-squares refinement of 5134 reflections from the data set with I > 5σ(I). Analysis of the data showed negligible crystal decay during data collection. No correction for absorption was applied.

Systematic absences in the intensity data uniquely determined the monoclinic space group $P2_1/n$. The structure was solved by a combination of direct methods and difference Fourier syntheses, and refined by full-matrix least-squares against F^2, using SHELXTL.[2] All atoms reside on positions of general crystallographic symmetry. Severe positional disorder of both butyl groups attached to N2 was observed. A disorder model consisting of two distinct conformations was employed. Occupation factors were adjusted manually and then fixed at values yielding reasonable displacement parameters. A total of 14 geometric restraints were used to maintain a chemically reasonable geometry for these groups. The behavior of the displacement parameters as well as the presence of several diffuse difference peaks in this region indicate more butyl group conformations exist. One atom (C45A) of the minor disorder component was refined isotropically; all other non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were placed in idealized positions and included as riding atoms. The relatively high final residuals reflect the poor crystal quality and side chain disorder.

(1) SMART Version 5.625 and SAINT+ Version 6.02a. Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1998.

(2) Sheldrick, G. M. SHELXTL Version 5.1; Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1997.

Table 1. Crystal data and structure refinement

Identification code	dba1bm	
Empirical formula	C56 H58 F6 N2	
Formula weight	873.04	
Temperature	150.0(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 13.8368(7) Å	α= 90°.
	b = 16.9399(8) Å	β= 97.8490(10)°.
	c = 20.4197(10) Å	γ = 90°.
Volume	4741.4(4) $Å^3$	
Z	4	
Density (calculated)	1.223 Mg/m^3	
Absorption coefficient	0.087 mm^{-1}	
F(000)	1848	
Crystal size	0.38 x 0.28 x 0.10 mm^3	
Theta range for data collection	1.57 to 20.81°.	
Index ranges	-13<=h<=13, -16<=k<=16, -20<=l<=20	
Reflections collected	23049	
Independent reflections	4958 [R(int) = 0.0603]	
Completeness to theta = 20.81°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4958 / 14 / 608	
Goodness-of-fit on F^2	0.971	
Final R indices [I>2sigma(I)]	R1 = 0.0596, wR2 = 0.1407	
R indices (all data)	R1 = 0.1022, wR2 = 0.1581	
Largest diff. peak and hole	0.209 and -0.157 $e.Å^{-3}$	

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

	x	y	z	U(eq)
C(1)	2843(3)	1342(3)	2020(3)	77(1)
C(2)	2453(3)	1217(3)	1352(3)	74(1)
C(3)	3038(3)	1271(2)	863(3)	75(1)
C(4)	4025(3)	1446(2)	987(3)	73(1)
C(5)	4414(3)	1595(2)	1649(3)	73(1)
C(6)	3836(3)	1544(2)	2142(3)	73(1)
C(7)	2237(4)	1286(3)	2551(3)	85(2)
C(8)	2535(3)	1336(3)	3201(3)	88(2)
C(9)	1977(4)	1288(3)	3745(3)	80(2)
C(10)	2455(4)	1351(3)	4383(3)	85(2)
C(11)	1989(3)	1316(3)	4931(3)	85(2)
C(12)	959(4)	1224(3)	4867(3)	74(1)
C(13)	469(4)	1190(3)	4223(3)	80(2)
C(14)	961(4)	1221(3)	3685(3)	80(2)
C(15)	984(3)	1257(3)	6075(2)	84(2)
C(16)	1458(3)	507(3)	6365(2)	80(1)
C(17)	2071(3)	645(3)	7033(2)	92(2)
C(18)	2464(4)	-109(3)	7356(3)	117(2)
C(19)	-523(3)	884(3)	5353(2)	85(2)
C(20)	-1274(3)	1512(3)	5338(3)	97(2)
C(21)	-2300(4)	1137(3)	5305(3)	117(2)
C(22)	-3045(4)	1706(4)	5376(3)	139(2)
C(23)	1420(3)	1046(3)	1159(2)	77(1)

C(24)	600(3)	915(3)	952(2)	80(2)
C(25)	-399(3)	791(3)	676(2)	72(1)
C(26)	-955(3)	220(3)	931(2)	83(2)
C(27)	-1930(3)	105(3)	682(2)	79(2)
C(28)	-2352(3)	548(3)	172(3)	88(2)
C(29)	-1816(3)	1120(3)	-96(2)	89(2)
C(30)	-854(3)	1242(3)	146(3)	74(1)
C(31)	-304(4)	1878(4)	-133(3)	95(2)
C(32)	4636(3)	1477(3)	454(3)	76(2)
C(33)	4458(3)	1070(3)	-106(3)	73(1)
C(34)	4968(3)	1077(3)	-676(3)	70(1)
C(35)	4771(3)	503(3)	-1164(3)	76(1)
C(36)	5209(4)	485(3)	-1713(3)	87(2)
C(37)	5919(3)	1053(3)	-1810(4)	82(2)
C(38)	6121(3)	1632(3)	-1330(3)	83(2)
C(39)	5666(3)	1639(3)	-788(3)	75(2)
C(40A)	6145(4)	489(4)	-2860(4)	121(2)
C(41A)	5259(6)	697(3)	-3326(3)	121(2)
C(42A)	5010(5)	80(6)	-3837(4)	179(3)
C(43A)	4211(6)	292(6)	-4307(4)	135(3)
C(40B)	6145(4)	489(4)	-2860(4)	121(2)
C(41B)	5259(6)	697(3)	-3326(3)	121(2)
C(42B)	5010(5)	80(6)	-3837(4)	179(3)
C(43B)	4394(18)	-565(13)	-4037(14)	154(12)
C(44A)	7094(5)	1644(3)	-2486(4)	139(2)
C(45A)	7895(17)	1324(19)	-2831(12)	292(16)
C(46A)	8531(11)	872(12)	-2343(17)	188(15)
C(47A)	9515(15)	923(19)	-2529(17)	166(5)
C(44B)	7094(5)	1644(3)	-2486(4)	139(2)
C(45B)	8102(5)	1596(6)	-2087(6)	125(4)
C(46B)	8712(16)	934(12)	-2023(8)	267(16)
C(47B)	9136(10)	691(12)	-2609(10)	166(5)
C(48)	5433(3)	1779(2)	1834(2)	73(1)
C(49)	6263(3)	1931(2)	2004(2)	72(1)
C(50)	7287(3)	2127(2)	2177(2)	65(1)
C(51)	7904(3)	2045(2)	1705(2)	72(1)
C(52)	8888(3)	2226(3)	1841(3)	79(1)
C(53)	9253(3)	2479(3)	2458(3)	80(2)
C(54)	8659(3)	2571(3)	2942(2)	76(1)
C(55)	7675(3)	2394(3)	2808(2)	68(1)
C(56)	7055(4)	2514(4)	3330(3)	91(2)
F(1)	-45(2)	2463(2)	293(2)	131(1)
F(2)	-801(2)	2216(2)	-675(2)	147(1)
F(3)	529(2)	1638(2)	-341(1)	108(1)
F(4)	7511(2)	2821(2)	3880(2)	131(1)
F(5)	6641(2)	1842(2)	3503(2)	133(1)
F(6)	6283(2)	2980(2)	3158(2)	128(1)
N(1)	477(3)	1177(2)	5406(2)	83(1)
N(2)	6383(3)	1063(3)	-2363(3)	112(2)

Table 3. Bond lengths [Å] and angles [°]

C(1)-C(6)	1.405(6)	C(46A)-C(47A)	1.465(14)	C(30)-C(29)-C(28)	120.8(5)
C(1)-C(2)	1.413(6)	C(45B)-C(46B)	1.398(13)	C(29)-C(30)-C(25)	120.5(5)
C(1)-C(7)	1.462(6)	C(46B)-C(47B)	1.461(13)	C(29)-C(30)-C(31)	119.8(5)
C(2)-C(3)	1.372(6)	C(48)-C(49)	1.182(5)	C(25)-C(30)-C(31)	119.7(4)
C(2)-C(23)	1.458(6)	C(49)-C(50)	1.451(6)	F(1)-C(31)-F(3)	105.4(4)
C(3)-C(4)	1.387(6)	C(50)-C(51)	1.378(6)	F(1)-C(31)-F(2)	106.4(5)
C(4)-C(5)	1.409(6)	C(50)-C(55)	1.402(5)	F(3)-C(31)-F(2)	103.4(5)
C(4)-C(32)	1.467(6)	C(51)-C(52)	1.386(6)	F(1)-C(31)-C(30)	113.4(5)
C(5)-C(6)	1.372(6)	C(52)-C(53)	1.362(6)	F(3)-C(31)-C(30)	114.1(5)
C(5)-C(48)	1.442(6)	C(53)-C(54)	1.376(6)	F(2)-C(31)-C(30)	113.2(5)
C(7)-C(8)	1.338(6)	C(54)-C(55)	1.385(5)	C(33)-C(32)-C(4)	124.2(5)
C(8)-C(9)	1.438(7)	C(55)-C(56)	1.471(7)	C(32)-C(33)-C(34)	129.5(5)
C(9)-C(10)	1.384(6)	C(56)-F(4)	1.318(5)	C(35)-C(34)-C(39)	115.5(5)
C(9)-C(14)	1.398(6)	C(56)-F(6)	1.336(6)	C(35)-C(34)-C(33)	120.1(5)
C(10)-C(11)	1.367(6)	C(56)-F(5)	1.342(6)	C(39)-C(34)-C(33)	124.3(6)
C(11)-C(12)	1.422(6)	C(6)-C(1)-C(2)	116.4(5)	C(36)-C(35)-C(34)	123.0(5)
C(12)-N(1)	1.364(6)	C(6)-C(1)-C(7)	122.0(5)	C(35)-C(36)-C(37)	120.3(6)
C(12)-C(13)	1.395(6)	C(2)-C(1)-C(7)	121.6(5)	N(2)-C(37)-C(38)	120.0(5)
C(13)-C(14)	1.371(6)	C(3)-C(2)-C(1)	120.4(4)	N(2)-C(37)-C(36)	122.6(6)
C(15)-N(1)	1.455(5)	C(3)-C(2)-C(23)	118.0(5)	C(38)-C(37)-C(36)	117.4(6)
C(15)-C(16)	1.513(6)	C(1)-C(2)-C(23)	121.5(5)	C(39)-C(38)-C(37)	121.0(5)
C(16)-C(17)	1.523(5)	C(2)-C(3)-C(4)	123.2(5)	C(38)-C(39)-C(34)	122.7(5)
C(17)-C(18)	1.505(6)	C(3)-C(4)-C(5)	116.8(5)	N(2)-C(40A)-C(41A)	112.8(5)
C(19)-N(1)	1.461(5)	C(3)-C(4)-C(32)	121.7(5)	C(42A)-C(41A)-C(40A)	112.0(7)
C(19)-C(20)	1.485(6)	C(5)-C(4)-C(32)	121.5(4)	C(43A)-C(42A)-C(41A)	112.7(8)
C(20)-C(21)	1.549(6)	C(6)-C(5)-C(4)	120.6(4)	N(2)-C(44A)-C(45A)	113.8(14)
C(21)-C(22)	1.432(7)	C(6)-C(5)-C(48)	117.9(5)	C(46A)-C(45A)-C(44A)	106.7(16)
C(23)-C(24)	1.177(5)	C(4)-C(5)-C(48)	121.5(5)	C(45A)-C(46A)-C(47A)	107(2)
C(24)-C(25)	1.435(6)	C(5)-C(6)-C(1)	122.6(5)	C(45B)-C(46B)-C(47B)	117.1(16)
C(25)-C(26)	1.382(6)	C(8)-C(7)-C(1)	127.0(5)	C(49)-C(48)-C(5)	178.1(6)
C(25)-C(30)	1.402(6)	C(7)-C(8)-C(9)	129.5(5)	C(48)-C(49)-C(50)	177.0(5)
C(26)-C(27)	1.389(5)	C(10)-C(9)-C(14)	115.7(6)	C(51)-C(50)-C(55)	118.7(4)
C(27)-C(28)	1.349(6)	C(10)-C(9)-C(8)	119.0(5)	C(51)-C(50)-C(49)	118.9(4)
C(28)-C(29)	1.379(6)	C(14)-C(9)-C(8)	125.2(5)	C(55)-C(50)-C(49)	122.4(5)
C(29)-C(30)	1.371(5)	C(11)-C(10)-C(9)	123.3(5)	C(50)-C(51)-C(52)	121.5(4)
C(30)-C(31)	1.477(7)	C(10)-C(11)-C(12)	120.6(5)	C(53)-C(52)-C(51)	119.0(5)
C(31)-F(1)	1.334(6)	N(1)-C(12)-C(13)	122.0(5)	C(52)-C(53)-C(54)	121.1(4)
C(31)-F(3)	1.345(5)	N(1)-C(12)-C(11)	121.7(5)	C(53)-C(54)-C(55)	120.1(5)
C(31)-F(2)	1.348(5)	C(13)-C(12)-C(11)	116.3(5)	C(54)-C(55)-C(50)	119.6(5)
C(32)-C(33)	1.328(6)	C(14)-C(13)-C(12)	121.5(5)	C(54)-C(55)-C(56)	118.9(5)
C(33)-C(34)	1.441(6)	C(13)-C(14)-C(9)	122.5(5)	C(50)-C(55)-C(56)	121.5(4)
C(34)-C(35)	1.393(6)	N(1)-C(15)-C(16)	114.6(4)	F(4)-C(56)-F(6)	105.3(5)
C(34)-C(39)	1.398(6)	C(15)-C(16)-C(17)	112.3(4)	F(4)-C(56)-F(5)	106.4(5)
C(35)-C(36)	1.346(6)	C(18)-C(17)-C(16)	112.7(4)	F(6)-C(56)-F(5)	102.5(5)
C(36)-C(37)	1.408(6)	N(1)-C(19)-C(20)	114.3(4)	F(4)-C(56)-C(55)	114.4(5)
C(37)-N(2)	1.374(6)	C(19)-C(20)-C(21)	110.0(4)	F(6)-C(56)-C(55)	114.5(5)
C(37)-C(38)	1.388(7)	C(22)-C(21)-C(20)	112.8(5)	F(5)-C(56)-C(55)	112.6(5)
C(38)-C(39)	1.345(6)	C(24)-C(23)-C(2)	174.7(5)	C(12)-N(1)-C(15)	121.8(4)
C(40A)-N(2)	1.412(7)	C(23)-C(24)-C(25)	177.0(5)	C(12)-N(1)-C(19)	121.5(4)
C(40A)-C(41A)	1.488(7)	C(26)-C(25)-C(30)	117.3(4)	C(15)-N(1)-C(19)	115.6(4)
C(41A)-C(42A)	1.484(9)	C(26)-C(25)-C(24)	120.7(4)	C(37)-N(2)-C(40A)	119.5(5)
C(42A)-C(43A)	1.407(8)	C(30)-C(25)-C(24)	122.0(5)	C(37)-N(2)-C(44A)	124.1(6)
C(44A)-N(2)	1.436(5)	C(25)-C(26)-C(27)	121.5(4)	C(40A)-N(2)-C(44A)	116.4(6)
C(44A)-C(45A)	1.494(14)	C(28)-C(27)-C(26)	120.1(5)		
C(45A)-C(46A)	1.453(15)	C(27)-C(28)-C(29)	119.8(4)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). 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}
C(1)	54(3)	61(3)	106(4)	14(3)	-18(3)	-2(2)
C(2)	45(3)	64(3)	104(4)	11(3)	-23(3)	-1(2)
C(3)	50(3)	63(3)	102(4)	8(3)	-22(3)	-3(2)
C(4)	50(3)	57(3)	101(4)	17(3)	-28(3)	-8(2)
C(5)	46(3)	53(3)	110(4)	15(3)	-22(3)	-10(2)
C(6)	49(3)	64(3)	97(4)	15(3)	-20(3)	-9(2)
C(7)	55(3)	73(3)	118(5)	15(3)	-23(4)	-3(3)
C(8)	55(3)	83(4)	116(5)	22(4)	-26(4)	-11(3)
C(9)	61(4)	67(3)	104(5)	22(3)	-20(3)	-10(3)
C(10)	61(3)	81(4)	106(4)	29(3)	-13(4)	-13(3)
C(11)	63(4)	68(3)	113(5)	20(3)	-28(3)	-11(3)
C(12)	62(4)	56(3)	99(4)	6(3)	-11(3)	-2(2)
C(13)	62(3)	58(3)	113(5)	11(3)	-13(4)	-6(3)
C(14)	60(4)	64(3)	106(5)	13(3)	-26(3)	-9(3)
C(15)	70(3)	67(3)	110(4)	-10(3)	-2(3)	-5(3)
C(16)	68(3)	68(3)	100(4)	-11(3)	-3(3)	-6(3)
C(17)	76(3)	91(4)	104(4)	-8(3)	-5(3)	-9(3)
C(18)	119(5)	109(5)	113(5)	16(4)	-21(4)	3(4)
C(19)	66(3)	69(3)	115(4)	-4(3)	-3(3)	-6(3)
C(20)	73(3)	81(4)	132(5)	-9(3)	-7(3)	4(3)
C(21)	63(3)	95(4)	185(6)	-14(4)	-8(4)	4(3)
C(22)	98(5)	117(5)	201(7)	-7(5)	19(4)	-1(4)
C(23)	49(3)	66(3)	108(4)	2(3)	-14(3)	1(2)
C(24)	41(3)	78(3)	111(4)	-12(3)	-21(3)	0(2)
C(25)	46(3)	61(3)	103(4)	-5(3)	-16(3)	4(3)
C(26)	55(3)	80(4)	105(4)	6(3)	-19(3)	-1(3)
C(27)	51(3)	68(3)	109(4)	10(3)	-23(3)	-12(2)
C(28)	45(3)	73(3)	133(5)	6(3)	-29(3)	-11(3)
C(29)	47(3)	91(4)	120(4)	14(3)	-24(3)	3(3)
C(30)	43(3)	57(3)	116(4)	2(3)	-4(3)	-10(2)
C(31)	52(3)	104(5)	124(5)	8(4)	-8(3)	19(4)
C(32)	47(3)	63(3)	109(4)	16(3)	-23(3)	-6(2)
C(33)	44(3)	61(3)	103(4)	18(3)	-26(3)	-9(2)
C(34)	44(3)	54(3)	103(4)	29(3)	-20(3)	-7(3)
C(35)	54(3)	53(3)	118(5)	20(4)	-2(3)	-11(3)
C(36)	75(4)	59(3)	125(5)	15(3)	14(3)	-6(3)
C(37)	52(3)	59(4)	136(5)	38(4)	13(3)	5(3)
C(38)	41(3)	54(4)	145(5)	25(4)	-16(3)	-5(3)
C(39)	41(3)	64(3)	111(4)	23(3)	-27(3)	-6(3)
C(40A)	84(4)	104(5)	182(7)	35(5)	43(5)	13(4)
C(41A)	162(7)	88(4)	123(5)	-29(4)	63(5)	-37(5)
C(42A)	112(6)	205(9)	219(9)	6(8)	25(6)	-5(6)
C(43A)	84(6)	182(10)	130(7)	-12(7)	-14(5)	-11(6)
C(40B)	84(4)	104(5)	182(7)	35(5)	43(5)	13(4)
C(41B)	162(7)	88(4)	123(5)	-29(4)	63(5)	-37(5)
C(42B)	112(6)	205(9)	219(9)	6(8)	25(6)	-5(6)
C(43B)	100(19)	140(30)	210(30)	20(20)	-13(19)	-80(20)
C(44A)	132(6)	88(4)	215(7)	16(5)	84(5)	-30(4)
C(46A)	49(11)	101(18)	420(50)	40(20)	63(17)	-36(13)
C(47A)	93(14)	223(16)	186(12)	-41(11)	33(12)	-101(11)
C(44B)	132(6)	88(4)	215(7)	16(5)	84(5)	-30(4)
C(45B)	36(5)	109(8)	232(13)	73(9)	23(6)	20(5)
C(46B)	490(40)	190(20)	104(12)	14(12)	-26(17)	60(20)
C(47B)	93(14)	223(16)	186(12)	-41(11)	33(12)	-101(11)
C(48)	55(3)	59(3)	97(4)	16(3)	-21(3)	-9(2)
C(49)	49(3)	65(3)	94(4)	19(3)	-20(3)	-11(2)

C(50)	52(3)	56(3)	79(4)	9(3)	-19(3)	-9(2)
C(51)	61(3)	68(3)	79(3)	-14(3)	-13(3)	-6(3)
C(52)	59(3)	74(3)	100(4)	-18(3)	-4(3)	-5(3)
C(53)	51(3)	72(3)	111(4)	-12(3)	-14(3)	-7(3)
C(54)	58(3)	75(3)	86(4)	-5(3)	-18(3)	-14(3)
C(55)	54(3)	71(3)	74(4)	2(3)	-9(3)	-10(2)
C(56)	85(4)	94(5)	88(4)	18(4)	-3(4)	-13(4)
F(1)	97(2)	85(2)	215(4)	-24(2)	37(2)	-26(2)
F(2)	85(2)	134(3)	211(4)	79(3)	-19(2)	-5(2)
F(3)	73(2)	121(2)	127(2)	8(2)	9(2)	5(2)
F(4)	116(2)	187(3)	85(2)	-33(2)	1(2)	-30(2)
F(5)	142(3)	142(3)	117(3)	27(2)	21(2)	-39(2)
F(6)	101(2)	172(3)	115(2)	23(2)	29(2)	29(2)
N(1)	62(3)	76(3)	108(4)	-9(3)	-9(3)	-6(2)
N(2)	107(4)	58(3)	179(6)	7(4)	46(4)	-7(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3)

	x	y	z	U(eq)
H(3)	2753	1184	419	90
H(6)	4119	1650	2584	88
H(7)	1559	1205	2421	102
H(8)	3216	1416	3322	106
H(10)	3142	1423	4444	102
H(11)	2356	1353	5359	102
H(13)	-222	1144	4156	96
H(14)	599	1196	3255	96
H(15A)	513	1444	6365	101
H(15B)	1494	1668	6076	101
H(16A)	1877	286	6054	96
H(16B)	944	114	6417	96
H(17A)	2624	996	6972	110
H(17B)	1668	917	7329	110
H(18A)	1920	-455	7426	175
H(18B)	2851	12	7783	175
H(18C)	2877	-375	7071	175
H(19A)	-578	531	5732	102
H(19B)	-661	564	4945	102
H(20A)	-1250	1855	4947	116
H(20B)	-1139	1843	5739	116
H(21A)	-2462	863	4876	140
H(21B)	-2289	738	5660	140
H(22A)	-2912	1954	5812	208
H(22B)	-3681	1442	5332	208
H(22C)	-3051	2110	5032	208
H(26)	-664	-102	1286	99
H(27)	-2300	-286	870	95
H(28)	-3017	465	-1	105
H(29)	-2117	1434	-452	107
H(32)	5195	1808	513	91
H(33)	3914	724	-135	87
H(35)	4305	105	-1106	91
H(36)	5039	87	-2036	104
H(38)	6588	2029	-1385	99
H(39)	5826	2043	-469	90
H(40A)	6036	-24	-2649	145
H(40B)	6702	424	-3112	145
H(41A)	4702	770	-3075	145
H(41B)	5371	1204	-3545	145

H(42A)	5584	-17	-4067	214
H(42B)	4856	-417	-3619	214
H(43A)	3621	321	-4092	202
H(43B)	4122	-106	-4659	202
H(43C)	4335	807	-4497	202
H(40C)	6036	-24	-2649	145
H(40D)	6702	424	-3112	145
H(41C)	4702	770	-3075	145
H(41D)	5371	1204	-3545	145
H(42C)	4908	409	-4242	214
H(42D)	5658	-164	-3846	214
H(43D)	4623	-1033	-3779	232
H(43E)	4407	-670	-4508	232
H(43F)	3726	-440	-3963	232
H(44A)	7383	1876	-2059	167
H(44B)	6759	2073	-2758	167
H(45A)	7625	982	-3204	351
H(45B)	8262	1761	-3005	351
H(46A)	8315	315	-2343	226
H(46B)	8520	1094	-1896	226
H(47A)	9523	676	-2962	249
H(47B)	9975	648	-2198	249
H(47C)	9706	1479	-2550	249
H(44C)	6821	2171	-2410	167
H(44D)	7173	1614	-2960	167
H(45C)	8487	2023	-2256	150
H(45D)	8016	1748	-1630	150
H(46C)	8335	484	-1881	320
H(46D)	9253	1040	-1664	320
H(47D)	8616	513	-2951	249
H(47E)	9597	258	-2492	249
H(47F)	9478	1139	-2776	249
H(51)	7649	1860	1277	86
H(52)	9301	2176	1508	94
H(53)	9929	2594	2557	96
H(54)	8925	2756	3368	91

Table 6. Torsion angles [°].

C(6)-C(1)-C(2)-C(3)	1.8(6)	C(9)-C(10)-C(11)-C(12)	-1.2(8)
C(7)-C(1)-C(2)-C(3)	-179.8(4)	C(10)-C(11)-C(12)-N(1)	179.2(5)
C(6)-C(1)-C(2)-C(23)	-176.6(4)	C(10)-C(11)-C(12)-C(13)	-1.2(7)
C(7)-C(1)-C(2)-C(23)	1.7(7)	N(1)-C(12)-C(13)-C(14)	-178.7(5)
C(1)-C(2)-C(3)-C(4)	0.2(7)	C(11)-C(12)-C(13)-C(14)	1.8(7)
C(23)-C(2)-C(3)-C(4)	178.6(4)	C(12)-C(13)-C(14)-C(9)	0.0(8)
C(2)-C(3)-C(4)-C(5)	-2.0(6)	C(10)-C(9)-C(14)-C(13)	-2.3(7)
C(2)-C(3)-C(4)-C(32)	178.1(4)	C(8)-C(9)-C(14)-C(13)	-179.0(5)
C(3)-C(4)-C(5)-C(6)	1.8(6)	N(1)-C(15)-C(16)-C(17)	173.6(4)
C(32)-C(4)-C(5)-C(6)	-178.3(4)	C(15)-C(16)-C(17)-C(18)	174.3(4)
C(3)-C(4)-C(5)-C(48)	179.6(4)	N(1)-C(19)-C(20)-C(21)	-178.1(4)
C(32)-C(4)-C(5)-C(48)	-0.5(6)	C(19)-C(20)-C(21)-C(22)	172.4(5)
C(4)-C(5)-C(6)-C(1)	0.2(7)	C(3)-C(2)-C(23)-C(24)	-8(5)
C(48)-C(5)-C(6)-C(1)	-177.7(4)	C(1)-C(2)-C(23)-C(24)	171(5)
C(2)-C(1)-C(6)-C(5)	-2.0(6)	C(2)-C(23)-C(24)-C(25)	-46(14)
C(7)-C(1)-C(6)-C(5)	179.6(4)	C(23)-C(24)-C(25)-C(26)	-174(100)
C(6)-C(1)-C(7)-C(8)	-6.9(8)	C(23)-C(24)-C(25)-C(30)	5(11)
C(2)-C(1)-C(7)-C(8)	174.9(5)	C(30)-C(25)-C(26)-C(27)	-1.2(7)
C(1)-C(7)-C(8)-C(9)	179.8(5)	C(24)-C(25)-C(26)-C(27)	178.3(4)
C(7)-C(8)-C(9)-C(10)	179.4(5)	C(25)-C(26)-C(27)-C(28)	1.0(7)
C(7)-C(8)-C(9)-C(14)	-4.1(9)	C(26)-C(27)-C(28)-C(29)	-0.7(8)
C(14)-C(9)-C(10)-C(11)	2.9(7)	C(27)-C(28)-C(29)-C(30)	0.5(8)
C(8)-C(9)-C(10)-C(11)	179.8(5)	C(28)-C(29)-C(30)-C(25)	-0.7(7)

C(28)-C(29)-C(30)-C(31)	-177.7(5)	C(48)-C(49)-C(50)-C(55)	154(9)
C(26)-C(25)-C(30)-C(29)	1.0(7)	C(55)-C(50)-C(51)-C(52)	-0.7(6)
C(24)-C(25)-C(30)-C(29)	-178.5(4)	C(49)-C(50)-C(51)-C(52)	179.7(4)
C(26)-C(25)-C(30)-C(31)	178.0(5)	C(50)-C(51)-C(52)-C(53)	1.1(7)
C(24)-C(25)-C(30)-C(31)	-1.5(7)	C(51)-C(52)-C(53)-C(54)	-1.3(7)
C(29)-C(30)-C(31)-F(1)	112.9(5)	C(52)-C(53)-C(54)-C(55)	1.0(7)
C(25)-C(30)-C(31)-F(1)	-64.1(6)	C(53)-C(54)-C(55)-C(50)	-0.5(7)
C(29)-C(30)-C(31)-F(3)	-126.4(5)	C(53)-C(54)-C(55)-C(56)	-178.7(5)
C(25)-C(30)-C(31)-F(3)	56.6(7)	C(51)-C(50)-C(55)-C(54)	0.4(6)
C(29)-C(30)-C(31)-F(2)	-8.4(8)	C(49)-C(50)-C(55)-C(54)	179.9(4)
C(25)-C(30)-C(31)-F(2)	174.6(5)	C(51)-C(50)-C(55)-C(56)	178.5(4)
C(3)-C(4)-C(32)-C(33)	-27.3(7)	C(49)-C(50)-C(55)-C(56)	-2.0(7)
C(5)-C(4)-C(32)-C(33)	152.8(4)	C(54)-C(55)-C(56)-F(4)	2.9(7)
C(4)-C(32)-C(33)-C(34)	176.6(4)	C(50)-C(55)-C(56)-F(4)	-175.2(4)
C(32)-C(33)-C(34)-C(35)	168.4(4)	C(54)-C(55)-C(56)-F(6)	124.6(5)
C(32)-C(33)-C(34)-C(39)	-12.3(7)	C(50)-C(55)-C(56)-F(6)	-53.5(7)
C(39)-C(34)-C(35)-C(36)	-0.6(6)	C(54)-C(55)-C(56)-F(5)	-118.7(5)
C(33)-C(34)-C(35)-C(36)	178.8(4)	C(50)-C(55)-C(56)-F(5)	63.2(6)
C(34)-C(35)-C(36)-C(37)	1.4(7)	C(13)-C(12)-N(1)-C(15)	-176.5(4)
C(35)-C(36)-C(37)-N(2)	-179.6(5)	C(11)-C(12)-N(1)-C(15)	3.0(7)
C(35)-C(36)-C(37)-C(38)	-1.6(7)	C(13)-C(12)-N(1)-C(19)	16.5(7)
N(2)-C(37)-C(38)-C(39)	179.2(4)	C(11)-C(12)-N(1)-C(19)	-164.0(4)
C(36)-C(37)-C(38)-C(39)	1.1(6)	C(16)-C(15)-N(1)-C(12)	-83.4(6)
C(37)-C(38)-C(39)-C(34)	-0.3(7)	C(16)-C(15)-N(1)-C(19)	84.3(5)
C(35)-C(34)-C(39)-C(38)	0.0(6)	C(20)-C(19)-N(1)-C(12)	-100.7(5)
C(33)-C(34)-C(39)-C(38)	-179.3(4)	C(20)-C(19)-N(1)-C(15)	91.5(5)
N(2)-C(40A)-C(41A)-C(42A)	-179.0(6)	C(38)-C(37)-N(2)-C(40A)	-177.6(5)
C(40A)-C(41A)-C(42A)-C(43A)	-176.2(7)	C(36)-C(37)-N(2)-C(40A)	0.4(7)
N(2)-C(44A)-C(45A)-C(46A)	-75(2)	C(38)-C(37)-N(2)-C(44A)	0.5(8)
C(44A)-C(45A)-C(46A)-C(47A)	-150(2)	C(36)-C(37)-N(2)-C(44A)	178.5(5)
C(6)-C(5)-C(48)-C(49)	-3(14)	C(41A)-C(40A)-N(2)-C(37)	83.0(6)
C(4)-C(5)-C(48)-C(49)	179(100)	C(41A)-C(40A)-N(2)-C(44A)	-95.3(6)
C(5)-C(48)-C(49)-C(50)	-162(10)	C(45A)-C(44A)-N(2)-C(37)	144.6(12)
C(48)-C(49)-C(50)-C(51)	-27(10)	C(45A)-C(44A)-N(2)-C(40A)	-37.3(13)

Symmetry transformations used to generate equivalent atoms:

X-Ray Structure Determination, $C_{58}H_{56}F_{12}N_2$

An orange plate was mounted onto the end of a thin glass fiber using inert oil. X-ray intensity data covering the full sphere of reciprocal space were measured at 150(1) K on a Bruker SMART APEX CCD-based diffractometer (Mo Kα radiation, λ = 0.71073 Å).[1] The raw data frames were integrated with SAINT+,[1] which also applied corrections for Lorentz and polarization effects. The final unit cell parameters are based on the least-squares refinement of 9712 reflections from the data set with $I > 5(\sigma)I$. Analysis of the data showed negligible crystal decay during data collection.

Systematic absences in the intensity data were consistent with the space groups C2/c and Cc; intensity statistics indicated centricity. The structure was solved in C2/c by a combination of direct methods and difference Fourier syntheses, and refined by full-matrix least-squares against F^2, using SHELXTL.[2] The molecule resides on a center of symmetry. All non-hydrogen atoms were refined with anisotropic displacement parameters; hydrogen atoms were placed in geometrically idealized positions and refined with isotropic displacement parameters as riding atoms.

(1) SMART Version 5.625 and SAINT+ Version 6.02a. Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1998.

(2) Sheldrick, G. M. SHELXTL Version 5.1; Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1997.

Table 1. Crystal data and structure refinement

Identification code	jw06s	
Empirical formula	C58 H56 F12 N2	
Formula weight	1009.05	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 42.3302(15) Å	α= 90°.
	b = 5.1937(2) Å	β= 128.2140(10)°.
	c = 28.8208(11) Å	γ = 90°.
Volume	4978.4(3) $Å^3$	
Z	4	
Density (calculated)	1.346 Mg/m^3	
Absorption coefficient	0.109 mm^{-1}	
F(000)	2104	
Crystal size	0.42 x 0.30 x 0.10 mm^3	
Theta range for data collection	1.42 to 25.04°.	
Index ranges	-50<=h<=49, -6<=k<=6, -34<=l<=34	
Reflections collected	16856	
Independent reflections	4406 [R(int) = 0.0428]	
Completeness to theta = 25.04°	99.8 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4406 / 0 / 356	
Goodness-of-fit on F^2	1.056	
Final R indices [I>2sigma(I)]	R1 = 0.0541, wR2 = 0.1423	
R indices (all data)	R1 = 0.0675, wR2 = 0.1497	
Extinction coefficient	0.0006(2)	
Largest diff. peak and hole	0.667 and -0.385 e.$Å^{-3}$	

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

	x	y	z	U(eq)
C(1)	2824(1)	6204(4)	5522(1)	28(1)
C(2)	2893(1)	8240(4)	5282(1)	29(1)
C(3)	2582(1)	9520(4)	4772(1)	27(1)
C(4)	2672(1)	11575(4)	4539(1)	30(1)
C(5)	2725(1)	13297(4)	4318(1)	31(1)
C(6)	2755(1)	15322(4)	4009(1)	30(1)
C(7)	3125(1)	16143(4)	4176(1)	31(1)
C(8)	3141(1)	18048(4)	3851(1)	32(1)
C(9)	2791(1)	19130(4)	3361(1)	33(1)
C(10)	2424(1)	18337(4)	3201(1)	31(1)
C(11)	2405(1)	16457(4)	3523(1)	31(1)
C(12)	3534(1)	18902(5)	4015(1)	39(1)
C(13)	2047(1)	19485(4)	2664(1)	36(1)
C(14)	3147(1)	4764(4)	6041(1)	32(1)
C(15)	3542(1)	5073(4)	6361(1)	33(1)
C(16)	3847(1)	3448(4)	6857(1)	32(1)
C(17)	3754(1)	1651(4)	7117(1)	40(1)
C(18)	4035(1)	25(4)	7559(1)	41(1)
C(19)	4436(1)	31(4)	7772(1)	34(1)
C(20)	4536(1)	1896(4)	7530(1)	34(1)
C(21)	4249(1)	3547(4)	7090(1)	35(1)
C(22)	4605(1)	-3446(4)	8473(1)	39(1)
C(23)	4616(1)	-2218(5)	8962(1)	43(1)

C(24)	4428(1)	-3878(5)	9162(1)	48(1)
C(25)	4511(1)	-2914(6)	9727(1)	60(1)
C(26)	5120(1)	-1811(4)	8382(1)	39(1)
C(27)	5412(1)	132(4)	8853(1)	38(1)
C(28)	5838(1)	-187(5)	9063(1)	42(1)
C(29)	6113(1)	1985(5)	9463(1)	49(1)
F(1)	3848(1)	17995(3)	4536(1)	59(1)
F(2)	3570(1)	21460(3)	4050(1)	54(1)
F(3)	3587(1)	18189(4)	3625(1)	68(1)
F(4)	2030(1)	21978(3)	2693(1)	85(1)
F(5)	1718(1)	18608(5)	2556(1)	117(1)
F(6)	2009(1)	19052(4)	2183(1)	83(1)
N(1)	4712(1)	-1714(3)	8191(1)	37(1)

Table 3. Bond lengths [Å] and angles [°]

C(1)-C(2)	1.388(3)	C(22)-N(1)	1.460(3)	F(1)-C(12)-F(2)	106.24(19)
C(1)-C(3)#1	1.421(3)	C(22)-C(23)	1.521(3)	F(3)-C(12)-C(8)	112.89(19)
C(1)-C(14)	1.462(3)	C(23)-C(24)	1.511(4)	F(1)-C(12)-C(8)	113.05(19)
C(2)-C(3)	1.394(3)	C(24)-C(25)	1.519(4)	F(2)-C(12)-C(8)	111.90(19)
C(3)-C(1)#1	1.421(3)	C(26)-N(1)	1.452(3)	F(4)-C(13)-F(5)	105.6(2)
C(3)-C(4)	1.433(3)	C(26)-C(27)	1.522(3)	F(4)-C(13)-F(6)	105.2(2)
C(4)-C(5)	1.197(3)	C(27)-C(28)	1.515(3)	F(5)-C(13)-F(6)	105.8(2)
C(5)-C(6)	1.431(3)	C(28)-C(29)	1.516(3)	F(4)-C(13)-C(10)	113.3(2)
C(6)-C(11)	1.391(3)	C(2)-C(1)-C(3)#1	117.03(19)	F(5)-C(13)-C(10)	113.88(19)
C(6)-C(7)	1.392(3)	C(2)-C(1)-C(14)	123.22(19)	F(6)-C(13)-C(10)	112.25(19)
C(7)-C(8)	1.392(3)	C(3)#1-C(1)-C(14)	119.72(19)	C(15)-C(14)-C(1)	128.9(2)
C(8)-C(9)	1.385(3)	C(1)-C(2)-C(3)	122.5(2)	C(14)-C(15)-C(16)	125.5(2)
C(8)-C(12)	1.490(3)	C(2)-C(3)-C(1)#1	120.44(19)	C(21)-C(16)-C(17)	115.6(2)
C(9)-C(10)	1.381(3)	C(2)-C(3)-C(4)	119.94(19)	C(21)-C(16)-C(15)	121.8(2)
C(10)-C(11)	1.383(3)	C(1)#1-C(3)-C(4)	119.62(19)	C(17)-C(16)-C(15)	122.5(2)
C(10)-C(13)	1.497(3)	C(5)-C(4)-C(3)	176.3(2)	C(18)-C(17)-C(16)	122.7(2)
C(12)-F(3)	1.326(3)	C(4)-C(5)-C(6)	175.2(2)	C(17)-C(18)-C(19)	121.6(2)
C(12)-F(1)	1.332(3)	C(11)-C(6)-C(7)	119.13(19)	N(1)-C(19)-C(20)	122.6(2)
C(12)-F(2)	1.334(3)	C(11)-C(6)-C(5)	119.3(2)	N(1)-C(19)-C(18)	121.4(2)
C(13)-F(4)	1.302(3)	C(7)-C(6)-C(5)	121.5(2)	C(20)-C(19)-C(18)	116.0(2)
C(13)-F(5)	1.306(3)	C(8)-C(7)-C(6)	119.8(2)	C(21)-C(20)-C(19)	121.5(2)
C(13)-F(6)	1.311(3)	C(9)-C(8)-C(7)	120.6(2)	C(20)-C(21)-C(16)	122.5(2)
C(14)-C(15)	1.326(3)	C(9)-C(8)-C(12)	118.9(2)	N(1)-C(22)-C(23)	114.49(19)
C(15)-C(16)	1.462(3)	C(7)-C(8)-C(12)	120.5(2)	C(24)-C(23)-C(22)	113.2(2)
C(16)-C(21)	1.390(3)	C(10)-C(9)-C(8)	119.5(2)	C(23)-C(24)-C(25)	113.1(2)
C(16)-C(17)	1.398(3)	C(9)-C(10)-C(11)	120.3(2)	N(1)-C(26)-C(27)	115.76(19)
C(17)-C(18)	1.369(3)	C(9)-C(10)-C(13)	119.14(19)	C(28)-C(27)-C(26)	113.13(19)
C(18)-C(19)	1.405(3)	C(11)-C(10)-C(13)	120.5(2)	C(27)-C(28)-C(29)	112.7(2)
C(19)-N(1)	1.378(3)	C(10)-C(11)-C(6)	120.6(2)	C(19)-N(1)-C(26)	121.26(19)
C(19)-C(20)	1.401(3)	F(3)-C(12)-F(1)	106.5(2)	C(19)-N(1)-C(22)	120.43(19)
C(20)-C(21)	1.383(3)	F(3)-C(12)-F(2)	105.74(19)	C(26)-N(1)-C(22)	118.25(18)

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). 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}
C(1)	32(1)	28(1)	26(1)	1(1)	19(1)	3(1)
C(2)	28(1)	30(1)	29(1)	0(1)	18(1)	1(1)
C(3)	31(1)	24(1)	27(1)	1(1)	18(1)	1(1)
C(4)	30(1)	30(1)	27(1)	1(1)	16(1)	4(1)
C(5)	32(1)	31(1)	30(1)	0(1)	18(1)	2(1)
C(6)	38(1)	26(1)	29(1)	-1(1)	23(1)	0(1)
C(7)	34(1)	29(1)	29(1)	-1(1)	19(1)	2(1)
C(8)	36(1)	29(1)	33(1)	-3(1)	23(1)	-2(1)
C(9)	44(1)	28(1)	34(1)	0(1)	27(1)	-3(1)
C(10)	37(1)	29(1)	28(1)	-1(1)	21(1)	1(1)
C(11)	32(1)	31(1)	32(1)	-3(1)	20(1)	-2(1)
C(12)	39(1)	40(1)	38(1)	-3(1)	24(1)	-5(1)
C(13)	39(1)	37(1)	34(1)	2(1)	23(1)	0(1)
C(14)	33(1)	30(1)	31(1)	5(1)	19(1)	0(1)
C(15)	35(1)	33(1)	32(1)	6(1)	22(1)	3(1)
C(16)	30(1)	31(1)	30(1)	2(1)	16(1)	2(1)
C(17)	25(1)	45(1)	41(1)	10(1)	16(1)	1(1)
C(18)	32(1)	38(1)	41(2)	11(1)	17(1)	-2(1)
C(19)	32(1)	29(1)	30(1)	-3(1)	14(1)	1(1)
C(20)	26(1)	42(1)	31(1)	1(1)	16(1)	2(1)
C(21)	33(1)	41(1)	31(1)	3(1)	21(1)	-1(1)
C(22)	40(1)	29(1)	34(1)	5(1)	15(1)	4(1)
C(23)	38(1)	38(1)	40(1)	-2(1)	18(1)	2(1)
C(24)	41(1)	49(2)	49(2)	-1(1)	24(1)	1(1)
C(25)	47(2)	77(2)	55(2)	0(2)	31(2)	6(2)
C(26)	34(1)	35(1)	34(1)	2(1)	14(1)	9(1)
C(27)	36(1)	37(1)	34(1)	-1(1)	19(1)	2(1)
C(28)	39(1)	39(1)	39(1)	1(1)	19(1)	4(1)
C(29)	39(2)	51(2)	49(2)	-4(1)	23(1)	-5(1)
F(1)	36(1)	63(1)	61(1)	17(1)	22(1)	-2(1)
F(2)	47(1)	40(1)	71(1)	0(1)	35(1)	-10(1)
F(3)	60(1)	100(1)	72(1)	-33(1)	53(1)	-28(1)
F(4)	83(1)	46(1)	55(1)	-6(1)	7(1)	26(1)
F(5)	39(1)	165(2)	105(2)	90(2)	23(1)	10(1)
F(6)	97(1)	98(1)	33(1)	8(1)	29(1)	54(1)
N(1)	32(1)	32(1)	35(1)	5(1)	14(1)	5(1)

Table 5. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$)

	x	y	z	U(eq)
H(2)	3162	8782	5474	33(6)
H(7)	3367	15405	4511	29(6)
H(9)	2803	20410	3137	34(6)
H(11)	2151	15936	3411	32(6)
H(14)	3063	3421	6166	50(7)
H(15)	3634	6469	6260	56(8)
H(17)	3485	1555	6982	41(7)
H(18)	3956	-1136	7726	62(8)
H(20)	4806	2030	7672	34(6)
H(21)	4329	4796	6941	40(7)
H(22A)	4791	-4932	8639	31(6)
H(22B)	4330	-4116	8167	46(7)
H(23A)	4900	-1870	9304	43(7)

H(23B)	4473	-547	8821	52(7)
H(24A)	4532	-5656	9226	42(7)
H(24B)	4133	-3934	8845	57(8)
H(25A)	4801	-2956	10049	56(8)
H(25B)	4373	-4021	9826	83(11)
H(25C)	4413	-1144	9669	85(11)
H(26A)	5109	-1543	8033	48(7)
H(26B)	5228	-3561	8535	38(6)
H(27A)	5317	1892	8693	49(7)
H(27B)	5412	-51	9195	62(8)
H(28A)	5833	-266	8715	53(8)
H(28B)	5947	-1839	9278	59(8)
H(29A)	6019	3606	9242	65(9)
H(29B)	6387	1628	9607	78(10)
H(29C)	6110	2123	9800	74(10)

Table 6. Torsion angles [°]

C(3)#1-C(1)-C(2)-C(3)	-1.0(3)	C(11)-C(10)-C(13)-F(5)	2.1(3)
C(14)-C(1)-C(2)-C(3)	177.07(19)	C(9)-C(10)-C(13)-F(6)	60.3(3)
C(1)-C(2)-C(3)-C(1)#1	1.1(3)	C(11)-C(10)-C(13)-F(6)	-118.1(2)
C(1)-C(2)-C(3)-C(4)	-178.74(19)	C(2)-C(1)-C(14)-C(15)	-0.6(4)
C(2)-C(3)-C(4)-C(5)	174(100)	C(3)#1-C(1)-C(14)-C(15)	177.5(2)
C(1)#1-C(3)-C(4)-C(5)	-6(4)	C(1)-C(14)-C(15)-C(16)	-176.3(2)
C(3)-C(4)-C(5)-C(6)	-11(6)	C(14)-C(15)-C(16)-C(21)	166.2(2)
C(4)-C(5)-C(6)-C(11)	22(3)	C(14)-C(15)-C(16)-C(17)	-12.2(4)
C(4)-C(5)-C(6)-C(7)	-157(3)	C(21)-C(16)-C(17)-C(18)	-2.2(4)
C(11)-C(6)-C(7)-C(8)	-1.0(3)	C(15)-C(16)-C(17)-C(18)	176.2(2)
C(5)-C(6)-C(7)-C(8)	177.5(2)	C(16)-C(17)-C(18)-C(19)	-1.3(4)
C(6)-C(7)-C(8)-C(9)	-0.2(3)	C(17)-C(18)-C(19)-N(1)	-176.0(2)
C(6)-C(7)-C(8)-C(12)	-178.48(19)	C(17)-C(18)-C(19)-C(20)	3.7(4)
C(7)-C(8)-C(9)-C(10)	1.0(3)	N(1)-C(19)-C(20)-C(21)	176.9(2)
C(12)-C(8)-C(9)-C(10)	179.3(2)	C(18)-C(19)-C(20)-C(21)	-2.8(3)
C(8)-C(9)-C(10)-C(11)	-0.6(3)	C(19)-C(20)-C(21)-C(16)	-0.6(4)
C(8)-C(9)-C(10)-C(13)	-178.92(19)	C(17)-C(16)-C(21)-C(20)	3.1(3)
C(9)-C(10)-C(11)-C(6)	-0.6(3)	C(15)-C(16)-C(21)-C(20)	-175.3(2)
C(13)-C(10)-C(11)-C(6)	177.67(19)	N(1)-C(22)-C(23)-C(24)	168.7(2)
C(7)-C(6)-C(11)-C(10)	1.4(3)	C(22)-C(23)-C(24)-C(25)	168.6(2)
C(5)-C(6)-C(11)-C(10)	-177.11(19)	N(1)-C(26)-C(27)-C(28)	176.1(2)
C(9)-C(8)-C(12)-F(3)	-67.9(3)	C(26)-C(27)-C(28)-C(29)	171.5(2)
C(7)-C(8)-C(12)-F(3)	110.4(2)	C(20)-C(19)-N(1)-C(26)	-3.7(3)
C(9)-C(8)-C(12)-F(1)	171.2(2)	C(18)-C(19)-N(1)-C(26)	176.0(2)
C(7)-C(8)-C(12)-F(1)	-10.5(3)	C(20)-C(19)-N(1)-C(22)	173.7(2)
C(9)-C(8)-C(12)-F(2)	51.3(3)	C(18)-C(19)-N(1)-C(22)	-6.6(3)
C(7)-C(8)-C(12)-F(2)	-130.4(2)	C(27)-C(26)-N(1)-C(19)	82.6(3)
C(9)-C(10)-C(13)-F(4)	-58.8(3)	C(27)-C(26)-N(1)-C(22)	-94.9(2)
C(11)-C(10)-C(13)-F(4)	122.9(2)	C(23)-C(22)-N(1)-C(19)	-79.1(3)
C(9)-C(10)-C(13)-F(5)	-179.5(2)	C(23)-C(22)-N(1)-C(26)	98.4(2)

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

X-Ray Structure Determination, $C_{42}H_{22}F_{12}$

A yellow-green blade was mounted onto the end of a thin glass fiber using inert oil. X-ray intensity data covering the full sphere of reciprocal space were measured at 150(1) K on a Bruker SMART APEX CCD-based diffractometer (Mo Kα radiation, λ = 0.71073 Å).[1] The raw data frames were integrated with SAINT+,[1] which also applied corrections for Lorentz and polarization effects. The final unit cell parameters are based on the least-squares refinement of 6553 reflections from the data set with I > 5(σ)I. Analysis of the data showed negligible crystal decay during data collection.

Systematic absences in the intensity data were consistent with the space groups P2/c and Pc; intensity statistics indicated centricity. The structure was solved in P2/c by a combination of direct methods and difference Fourier syntheses, and

refined by full-matrix least-squares against F^2, using SHELXTL.[2] The molecule resides on a center of symmetry. One $-CF_3$ group (C21, F4 – F6) is rotationally disordered over two orientations in the proportion 0.68(1) / 0.32 and was refined with the aid of 18 restraints (SHELX SAME). All non-hydrogen atoms were refined with anisotropic displacement parameters; hydrogen atoms were placed in geometrically idealized positions and refined with isotropic displacement parameters as riding atoms.

(1) SMART Version 5.625 and SAINT+ Version 6.02a. Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1998.

(2) Sheldrick, G. M. SHELXTL Version 5.1; Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1997.

Table 1. Crystal data and structure refinement.

Identification code	jw05s	
Empirical formula	C42 H22 F12	
Formula weight	754.60	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2/c	
Unit cell dimensions	a = 17.8688(14) Å	α= 90°.
	b = 5.0625(4) Å	β= 97.617(2)°.
	c = 18.6933(15) Å	γ = 90°.
Volume	1676.1(2) $Å^3$	
Z	2	
Density (calculated)	1.495 Mg/m^3	
Absorption coefficient	0.133 mm^{-1}	
F(000)	764	
Crystal size	0.72 x 0.30 x 0.04 mm^3	
Theta range for data collection	2.20 to 24.11°.	
Index ranges	-20<=h<=20, -5<=k<=5, -21<=l<=21	
Reflections collected	9956	
Independent reflections	2664 [R(int) = 0.0360]	
Completeness to theta = 24.11°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2664 / 18 / 284	
Goodness-of-fit on F^2	1.010	
Final R indices [I>2sigma(I)]	R1 = 0.0546, wR2 = 0.1344	
R indices (all data)	R1 = 0.0662, wR2 = 0.1421	
Extinction coefficient	0.0027(10)	
Largest diff. peak and hole	0.196 and -0.204 e.$Å^{-3}$	

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

	x	y	z	U(eq)
C(1)	4235(1)	307(5)	5128(1)	35(1)
C(2)	4816(1)	1805(5)	5501(1)	38(1)
C(3)	5566(1)	1541(5)	5380(1)	34(1)
C(4)	6127(1)	3185(5)	5792(1)	35(1)
C(5)	6557(1)	4623(5)	6135(1)	36(1)
C(6)	7069(1)	6492(5)	6510(1)	35(1)
C(7)	7785(1)	6841(5)	6305(2)	41(1)
C(8)	8274(2)	8727(6)	6631(2)	47(1)
C(9)	8067(2)	10284(6)	7168(2)	47(1)
C(10)	7365(2)	9952(6)	7391(2)	45(1)
C(11)	6868(1)	8072(5)	7075(1)	36(1)
C(13)	3447(1)	625(5)	5244(1)	37(1)
C(14)	3168(1)	2525(5)	5618(1)	39(1)

C(15)	2380(1)	2868(5)	5737(1)	37(1)
C(16)	1795(1)	1220(6)	5434(2)	43(1)
C(17)	1068(2)	1598(6)	5571(2)	47(1)
C(18)	894(2)	3636(6)	6018(2)	43(1)
C(19)	1464(2)	5293(6)	6317(2)	46(1)
C(20)	2193(2)	4911(6)	6175(2)	44(1)
C(12)	6119(2)	7697(6)	7337(1)	46(1)
F(1)	5540(1)	8112(4)	6834(1)	66(1)
F(2)	6035(1)	5252(4)	7582(1)	61(1)
F(3)	6033(1)	9295(4)	7888(1)	79(1)
C(21A)	107(2)	4005(6)	6173(2)	59(1)
F(4A)	-413(3)	3338(16)	5652(2)	99(3)
F(5A)	-38(4)	6350(10)	6410(7)	155(4)
F(6A)	-33(3)	2405(12)	6702(3)	80(2)
C(21B)	107(2)	4005(6)	6173(2)	59(1)
F(4B)	-18(9)	3860(40)	6837(5)	147(9)
F(5B)	-397(7)	2530(30)	5777(9)	135(8)
F(6B)	-121(7)	6430(18)	5957(7)	87(4)

Table 3. Bond lengths [Å] and angles [°].

C(1)-C(2)	1.395(4)	C(12)-F(3)	1.334(3)	C(13)-C(14)-C(15)	127.1(2)
C(1)-C(3)#1	1.412(4)	C(12)-F(2)	1.335(3)	C(20)-C(15)-C(16)	117.4(2)
C(1)-C(13)	1.463(4)	C(21A)-F(4A)	1.298(5)	C(20)-C(15)-C(14)	119.3(2)
C(2)-C(3)	1.395(4)	C(21A)-F(5A)	1.305(5)	C(16)-C(15)-C(14)	123.4(2)
C(3)-C(1)#1	1.412(4)	C(21A)-F(6A)	1.328(5)	C(17)-C(16)-C(15)	121.1(3)
C(3)-C(4)	1.444(4)	C(2)-C(1)-C(3)#1	117.4(2)	C(16)-C(17)-C(18)	120.7(3)
C(4)-C(5)	1.183(4)	C(2)-C(1)-C(13)	122.3(2)	C(19)-C(18)-C(17)	118.9(3)
C(5)-C(6)	1.434(4)	C(3)#1-C(1)-C(13)	120.4(2)	C(19)-C(18)-C(21A)	120.8(3)
C(6)-C(7)	1.395(4)	C(1)-C(2)-C(3)	122.3(2)	C(17)-C(18)-C(21A)	120.3(3)
C(6)-C(11)	1.408(4)	C(2)-C(3)-C(1)#1	120.3(2)	C(20)-C(19)-C(18)	120.2(3)
C(7)-C(8)	1.381(4)	C(2)-C(3)-C(4)	118.2(2)	C(19)-C(20)-C(15)	121.8(3)
C(8)-C(9)	1.366(4)	C(1)#1-C(3)-C(4)	121.4(2)	F(1)-C(12)-F(3)	107.2(2)
C(9)-C(10)	1.383(4)	C(5)-C(4)-C(3)	176.5(3)	F(1)-C(12)-F(2)	106.2(2)
C(10)-C(11)	1.380(4)	C(4)-C(5)-C(6)	176.0(3)	F(3)-C(12)-F(2)	105.5(2)
C(11)-C(12)	1.497(4)	C(7)-C(6)-C(11)	118.0(2)	F(1)-C(12)-C(11)	113.4(2)
C(13)-C(14)	1.325(4)	C(7)-C(6)-C(5)	119.9(2)	F(3)-C(12)-C(11)	112.0(2)
C(14)-C(15)	1.465(4)	C(11)-C(6)-C(5)	122.1(2)	F(2)-C(12)-C(11)	112.0(2)
C(15)-C(20)	1.386(4)	C(8)-C(7)-C(6)	121.1(3)	F(4A)-C(21A)-F(5A)	109.7(5)
C(15)-C(16)	1.397(4)	C(9)-C(8)-C(7)	120.4(3)	F(4A)-C(21A)-F(6A)	102.3(4)
C(16)-C(17)	1.370(4)	C(8)-C(9)-C(10)	119.8(3)	F(5A)-C(21A)-F(6A)	103.7(5)
C(17)-C(18)	1.388(4)	C(11)-C(10)-C(9)	120.8(3)	F(4A)-C(21A)-C(18)	115.0(4)
C(18)-C(19)	1.379(4)	C(10)-C(11)-C(6)	119.9(2)	F(5A)-C(21A)-C(18)	114.6(4)
C(18)-C(21A)	1.485(4)	C(10)-C(11)-C(12)	120.0(2)	F(6A)-C(21A)-C(18)	110.2(3)
C(19)-C(20)	1.378(4)	C(6)-C(11)-C(12)	120.1(2)		
C(12)-F(1)	1.318(3)	C(14)-C(13)-C(1)	126.5(2)		

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). 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}
C(1)	34(1)	35(2)	35(1)	4(1)	0(1)	-2(1)
C(2)	40(1)	36(2)	36(1)	-1(1)	2(1)	-1(1)
C(3)	35(1)	32(1)	34(1)	5(1)	-1(1)	-4(1)
C(4)	32(1)	35(2)	37(1)	4(1)	3(1)	0(1)
C(5)	36(1)	36(2)	37(1)	4(1)	6(1)	2(1)
C(6)	35(1)	31(1)	36(1)	4(1)	-1(1)	-1(1)
C(7)	39(1)	39(2)	46(2)	-1(1)	5(1)	0(1)
C(8)	37(2)	45(2)	58(2)	6(2)	3(1)	-6(1)
C(9)	46(2)	38(2)	54(2)	3(1)	-8(1)	-11(1)
C(10)	57(2)	36(2)	41(2)	-3(1)	0(1)	0(1)
C(11)	42(1)	27(1)	38(1)	4(1)	0(1)	-1(1)
C(13)	38(1)	35(2)	35(1)	4(1)	-2(1)	-7(1)
C(14)	37(1)	38(2)	40(1)	1(1)	-1(1)	-5(1)
C(15)	41(1)	35(2)	35(1)	6(1)	2(1)	1(1)
C(16)	41(2)	42(2)	46(2)	-4(1)	7(1)	-2(1)
C(17)	41(2)	44(2)	57(2)	1(2)	6(1)	-5(1)
C(18)	42(2)	40(2)	49(2)	11(1)	9(1)	3(1)
C(19)	51(2)	41(2)	47(2)	-3(1)	9(1)	7(1)
C(20)	44(2)	41(2)	47(2)	-1(1)	1(1)	-1(1)
C(12)	48(2)	46(2)	43(2)	1(1)	4(1)	-2(1)
F(1)	39(1)	89(2)	69(1)	17(1)	8(1)	12(1)
F(2)	54(1)	60(1)	71(1)	18(1)	18(1)	-5(1)
F(3)	76(1)	85(2)	83(1)	-36(1)	38(1)	-10(1)
C(21A)	53(2)	59(3)	67(2)	1(2)	18(2)	7(2)
F(4A)	40(3)	193(8)	61(2)	12(3)	0(2)	30(3)
F(5A)	82(4)	69(4)	338(14)	-63(5)	115(7)	-9(3)
F(6A)	56(2)	119(4)	71(3)	10(3)	32(2)	-6(2)
C(21B)	53(2)	59(3)	67(2)	1(2)	18(2)	7(2)
F(4B)	91(8)	300(30)	54(5)	-4(11)	26(5)	80(15)
F(5B)	61(7)	81(7)	270(20)	-102(10)	61(10)	-45(6)
F(6B)	55(5)	76(7)	140(9)	36(7)	48(6)	40(5)

Table 5. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$).

	x	y	z	U(eq)
H(2)	4696	3049	5849	36(7)
H(7)	7938	5763	5934	44(8)
H(8)	8758	8944	6480	59(9)
H(9)	8403	11589	7388	66(10)
H(10)	7223	11032	7766	37(7)
H(13)	3099	-659	5029	42(7)
H(14)	3517	3803	5833	47(8)
H(16)	1903	-188	5128	47(8)
H(17)	680	455	5359	51(8)
H(19)	1353	6701	6623	48(8)
H(20)	2578	6079	6382	45(8)

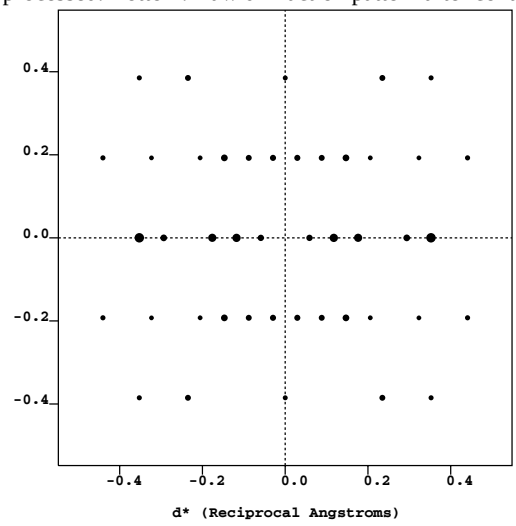
Table 6. Torsion angles [°]

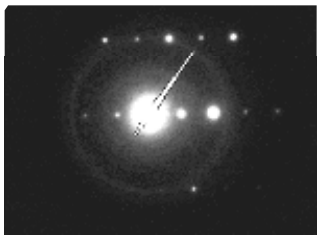
C(3)#1-C(1)-C(2)-C(3)	0.6(4)	C(13)-C(14)-C(15)-C(16)	-2.6(4)
C(13)-C(1)-C(2)-C(3)	-178.7(2)	C(20)-C(15)-C(16)-C(17)	-0.6(4)
C(1)-C(2)-C(3)-C(1)#1	-0.6(4)	C(14)-C(15)-C(16)-C(17)	178.9(3)
C(1)-C(2)-C(3)-C(4)	179.9(2)	C(15)-C(16)-C(17)-C(18)	-0.1(4)
C(2)-C(3)-C(4)-C(5)	-22(5)	C(16)-C(17)-C(18)-C(19)	0.6(4)
C(1)#1-C(3)-C(4)-C(5)	159(4)	C(16)-C(17)-C(18)-C(21A)	-179.0(3)
C(3)-C(4)-C(5)-C(6)	-71(7)	C(17)-C(18)-C(19)-C(20)	-0.3(4)
C(4)-C(5)-C(6)-C(7)	-75(4)	C(21A)-C(18)-C(19)-C(20)	179.4(3)
C(4)-C(5)-C(6)-C(11)	103(4)	C(18)-C(19)-C(20)-C(15)	-0.5(4)
C(11)-C(6)-C(7)-C(8)	-1.8(4)	C(16)-C(15)-C(20)-C(19)	1.0(4)
C(5)-C(6)-C(7)-C(8)	176.5(2)	C(14)-C(15)-C(20)-C(19)	-178.6(3)
C(6)-C(7)-C(8)-C(9)	0.5(4)	C(10)-C(11)-C(12)-F(1)	119.9(3)
C(7)-C(8)-C(9)-C(10)	0.5(4)	C(6)-C(11)-C(12)-F(1)	-60.7(3)
C(8)-C(9)-C(10)-C(11)	-0.3(4)	C(10)-C(11)-C(12)-F(3)	-1.5(4)
C(9)-C(10)-C(11)-C(6)	-1.0(4)	C(6)-C(11)-C(12)-F(3)	177.9(2)
C(9)-C(10)-C(11)-C(12)	178.4(3)	C(10)-C(11)-C(12)-F(2)	-119.9(3)
C(7)-C(6)-C(11)-C(10)	2.0(4)	C(6)-C(11)-C(12)-F(2)	59.6(3)
C(5)-C(6)-C(11)-C(10)	-176.2(2)	C(19)-C(18)-C(21A)-F(4A)	148.3(5)
C(7)-C(6)-C(11)-C(12)	-177.5(2)	C(17)-C(18)-C(21A)-F(4A)	-32.1(5)
C(5)-C(6)-C(11)-C(12)	4.4(4)	C(19)-C(18)-C(21A)-F(5A)	19.8(8)
C(2)-C(1)-C(13)-C(14)	9.9(4)	C(17)-C(18)-C(21A)-F(5A)	-160.6(7)
C(3)#1-C(1)-C(13)-C(14)	-169.3(3)	C(19)-C(18)-C(21A)-F(6A)	-96.7(4)
C(1)-C(13)-C(14)-C(15)	179.9(2)	C(17)-C(18)-C(21A)-F(6A)	82.9(4)
C(13)-C(14)-C(15)-C(20)	176.9(3)		

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

Simulated and observed electron diffraction of a microcrystal of 3 ($C_{42}H_{22}F_{12}$).

Top left: simulated electron diffraction obtained by cerius[2]. Top right: corresponding experimental electron diffraction pattern, processed. Bottom: Raw diffraction pattern after contrast enhancement by Microsoft Word program.





The crystals of **3** were very small and of comparatively low quality. They were slightly bent and not perfectly oriented with respect to the diffraction plane. As a consequence only a section of the expected diffraction pattern is visible. The calculated values are in perfect match when comparing experiment and simulation. The crystals are oriented such that one looks onto a plane that is defined by the crystallographic axes *a* and *b*.