Steps to Demarcate the Effects of Chromophore Aggregation and Planarization in Poly(phenyleneethynylene)s Part 2. The Photophysics of 1,4-Diethynyl-2-fluorobenzene in Solution and in Crystals

Marcia Levitus, Gerardo Zepeda, Hung Dang, Carlos Godinez, Tinh-Alfredo V. Khuong, Kelli Schmieder, and Miguel A. Garcia-Garibay*

Department of Chemistry and Biochemistry,
University of California
Los Angeles, CA 90095-1569

Supplementary Information Contents

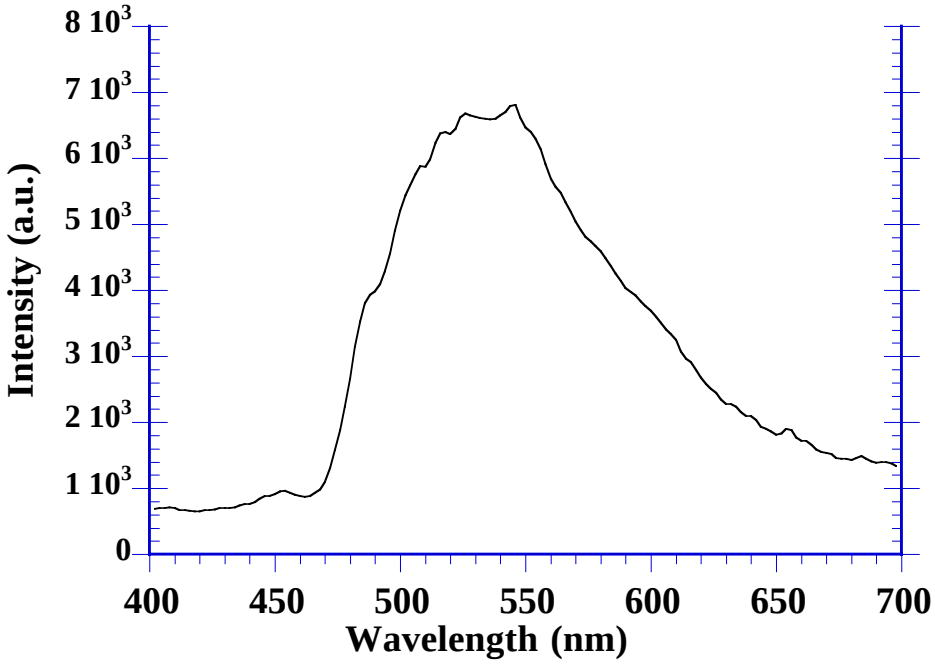

Figure 1: Phosphorescence spectrum of compound **3** in the crystalline solid state.

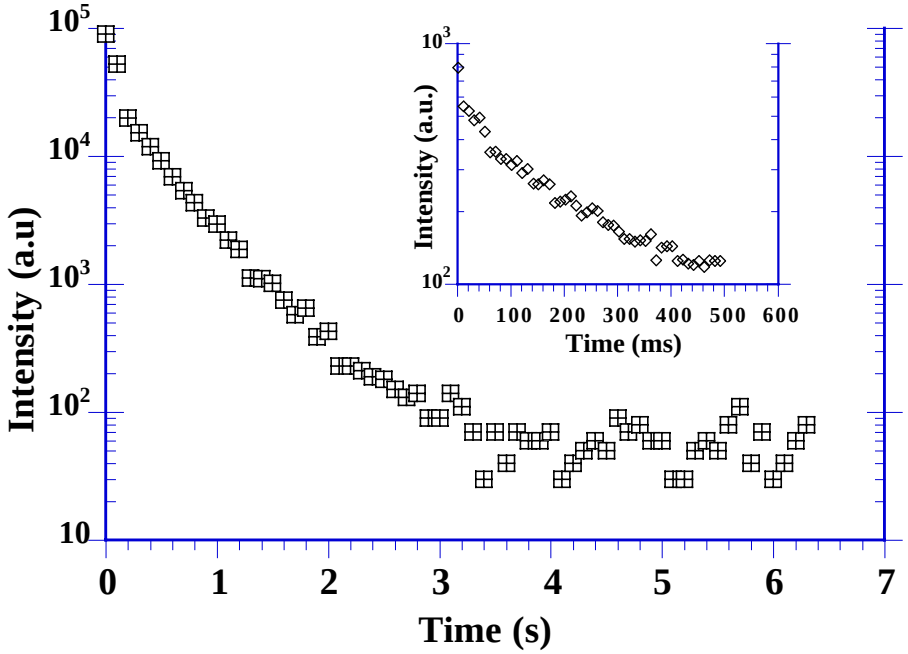

Figure 2: Phosphorescence decay of compound 3 in the crystalline solid state illustrating short and long-lived components. Data acquired during the first 600 ms is shown in the inset.

Figure 3. ORTEP diagram showing four molecules, two on general positions and two sitting on an inversion center. The fluorine atom is disordered through four positions on the benzene ring.

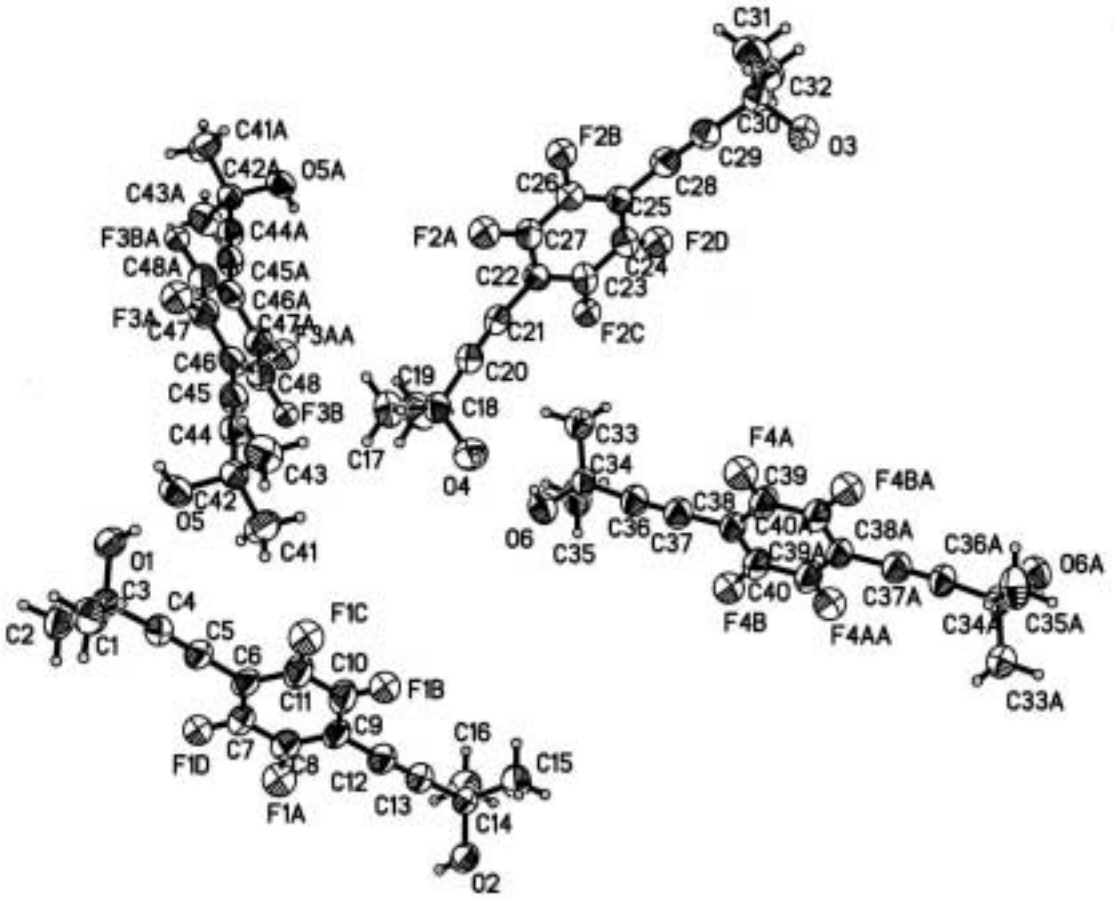

Figure 4.. Stereoview of the packing structure down the shortest direction of the unit cell.

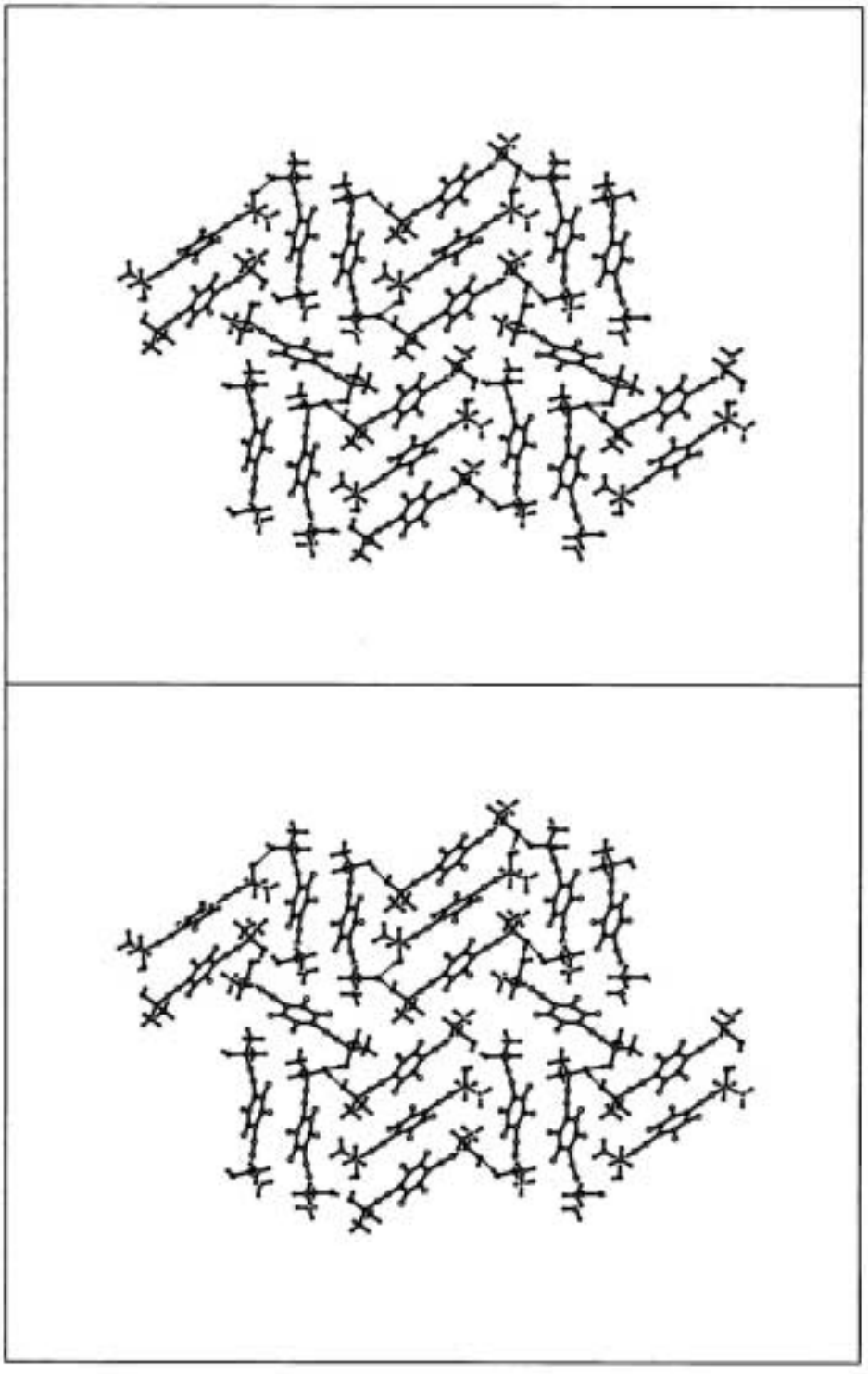

Table 1. Crystal data and structure refinement for Compound **4**.

Identification code	Compound **4**	
Empirical formula	C16 H17 F O2	
Formula weight	260.30	
Temperature	298(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1bar	
Unit cell dimensions	a = 6.0599(10) Å	a= 101.422(3)°.
	b = 17.450(3) Å	b= 95.053(3)°.
	c = 21.186(3) Å	g = 92.618(3)°.
Volume	2182.7(6) $Å^3$	
Z	6	
Density (calculated)	1.188 Mg/m^3	
Absorption coefficient	0.086 mm^{-1}	
F(000)	828	
Crystal size	0.6 x 0.2 x 0.1 mm^3	
Theta range for data collection	1.19 to 28.31°.	
Index ranges	-7<=h<=8, -23<=k<=21, -28<=l<=20	
Reflections collected	14183	
Independent reflections	9779 [R(int) = 0.0289]	
Completeness to theta = 28.31°	90.2 %	
Absorption correction	Empirical	
Max. and min. transmission	1.000 and .9234	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9779 / 0 / 553	
Goodness-of-fit on F^2	0.818	
Final R indices [I>2sigma(I)]	R1 = 0.0578, wR2 = 0.1322	
R indices (all data)	R1 = 0.1715, wR2 = 0.1621	
Largest diff. peak and hole	0.378 and -0.147 $e.Å^{-3}$	

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

	x	y	z	U(eq)
F(1A)	175(8)	7911(3)	4987(2)	75(1)
F(1B)	5235(19)	6154(7)	4728(6)	61(3)
F(1C)	7032(12)	6783(4)	3945(3)	74(2)
F(1D)	1872(12)	8552(4)	4140(4)	58(2)
F(2A)	11385(7)	1779(3)	701(2)	59(1)
F(2B)	13405(7)	452(2)	384(2)	55(1)
F(2C)	6171(13)	614(4)	1573(4)	51(2)
F(2D)	8310(40)	-625(15)	1284(13)	63(7)
F(3A)	8726(8)	5676(2)	-197(2)	63(1)
F(3B)	3921(17)	4996(6)	1142(6)	41(3)
F(4A)	7725(10)	306(3)	4200(3)	70(2)
F(4B)	1411(15)	737(5)	5077(4)	67(2)
O(1)	7377(3)	8304(1)	2077(1)	58(1)
O(2)	-253(3)	6267(1)	6742(1)	58(1)
O(3)	13919(3)	-2522(1)	1258(1)	62(1)
O(4)	6079(3)	3262(1)	2625(1)	64(1)
O(5)	10791(3)	7332(1)	2095(1)	58(1)
O(6)	3070(3)	2557(1)	3354(1)	63(1)
C(1)	9597(5)	9183(2)	2943(2)	74(1)
C(2)	5667(6)	9431(2)	2565(2)	81(1)
C(3)	7267(5)	8824(2)	2694(2)	52(1)
C(4)	6355(5)	8395(2)	3155(2)	54(1)
C(5)	5546(5)	8090(2)	3542(2)	56(1)
C(6)	4559(5)	7714(2)	4000(1)	54(1)
C(7)	2705(6)	7989(2)	4281(2)	61(1)
C(8)	1756(6)	7619(2)	4712(2)	63(1)
C(9)	2599(5)	6954(2)	4879(1)	55(1)
C(10)	4460(6)	6682(2)	4602(2)	69(1)
C(11)	5427(6)	7054(2)	4174(2)	68(1)
C(12)	1570(5)	6559(2)	5317(2)	60(1)
C(13)	717(5)	6211(2)	5673(2)	58(1)

C(14)	-341(5)	5774(2)	6110(1)	50(1)
C(15)	971(6)	5071(2)	6192(2)	77(1)
C(16)	-2735(5)	5520(2)	5859(2)	75(1)
C(17)	7435(5)	3987(2)	1854(2)	70(1)
C(18)	6067(5)	3272(2)	1946(2)	54(1)
C(19)	3650(5)	3303(2)	1699(2)	80(1)
C(20)	6993(5)	2549(2)	1622(2)	55(1)
C(21)	7787(5)	1962(2)	1385(1)	54(1)
C(22)	8748(5)	1225(2)	1169(1)	50(1)
C(23)	7860(6)	549(2)	1326(2)	61(1)
C(24)	8887(6)	-147(2)	1192(2)	58(1)
C(25)	10836(5)	-186(2)	885(1)	51(1)
C(26)	11682(5)	484(2)	708(1)	54(1)
C(27)	10655(5)	1174(2)	845(2)	56(1)
C(28)	11906(5)	-916(2)	772(2)	59(1)
C(29)	12695(5)	-1533(2)	715(2)	56(1)
C(30)	13618(5)	-2312(2)	635(2)	52(1)
C(31)	15812(5)	-2307(2)	343(2)	72(1)
C(32)	11960(5)	-2928(2)	226(2)	70(1)
C(33)	3134(6)	1351(2)	2569(2)	71(1)
C(34)	2333(5)	1740(2)	3209(2)	54(1)
C(35)	-177(5)	1701(2)	3181(2)	74(1)
C(36)	3234(5)	1353(2)	3724(2)	61(1)
C(37)	3811(5)	978(2)	4114(2)	63(1)
C(38)	4426(6)	485(2)	4567(2)	57(1)
C(39)	6404(6)	125(2)	4555(2)	66(1)
C(40)	3014(6)	352(2)	5019(2)	65(1)
C(41)	9124(6)	6535(2)	2715(2)	72(1)
C(42)	10438(5)	6527(2)	2139(1)	49(1)
C(43)	12655(5)	6180(2)	2233(2)	74(1)
C(44)	9097(5)	6101(2)	1549(2)	52(1)
C(45)	7937(5)	5765(2)	1087(2)	56(1)
C(46)	6453(5)	5372(2)	533(2)	53(1)
C(47)	6978(5)	5352(2)	-94(2)	58(1)
C(48)	4433(6)	5008(2)	622(2)	57(1)

Table 3. Bond lengths [Å] and angles [°] for Compound **4**

F(1A)-C(8)	1.242(5)
F(1B)-C(10)	1.120(12)
F(1C)-C(11)	1.200(7)
F(1D)-C(7)	1.201(7)
F(2A)-C(27)	1.229(5)
F(2B)-C(26)	1.296(5)
F(2C)-C(23)	1.190(7)
F(2D)-C(24)	0.95(3)
F(3A)-C(47)	1.234(5)
F(3B)-C(48)	1.176(11)
F(4A)-C(39)	1.217(6)
F(4B)-C(40)	1.208(9)
O(1)-C(3)	1.445(3)
O(1)-H(1)	0.8200
O(2)-C(14)	1.437(3)
O(2)-H(2)	0.8200
O(3)-C(30)	1.437(3)
O(3)-H(3)	0.8200
O(4)-C(18)	1.441(3)
O(4)-H(4)	0.8200
O(5)-C(42)	1.433(3)
O(5)-H(5)	0.8200
O(6)-C(34)	1.438(3)
O(6)-H(6)	0.8200
C(1)-C(3)	1.524(4)
C(1)-H(1A)	0.9600
C(1)-H(1B)	0.9600
C(1)-H(1C)	0.9600
C(2)-C(3)	1.516(4)
C(2)-H(2A)	0.9600
C(2)-H(2B)	0.9600
C(2)-H(2C)	0.9600
C(3)-C(4)	1.472(4)
C(4)-C(5)	1.189(4)

C(5)-C(6)	1.432(4)
C(6)-C(7)	1.379(4)
C(6)-C(11)	1.388(4)
C(7)-C(8)	1.370(4)
C(8)-C(9)	1.384(4)
C(9)-C(10)	1.377(4)
C(9)-C(12)	1.430(4)
C(10)-C(11)	1.371(4)
C(12)-C(13)	1.194(4)
C(13)-C(14)	1.479(4)
C(14)-C(16)	1.515(4)
C(14)-C(15)	1.521(4)
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(16)-H(16A)	0.9600
C(16)-H(16B)	0.9600
C(16)-H(16C)	0.9600
C(17)-C(18)	1.520(4)
C(17)-H(17A)	0.9600
C(17)-H(17B)	0.9600
C(17)-H(17C)	0.9600
C(18)-C(20)	1.473(4)
C(18)-C(19)	1.519(4)
C(19)-H(19A)	0.9600
C(19)-H(19B)	0.9600
C(19)-H(19C)	0.9600
C(20)-C(21)	1.189(4)
C(21)-C(22)	1.444(4)
C(22)-C(23)	1.386(4)
C(22)-C(27)	1.392(4)
C(23)-C(24)	1.382(4)
C(24)-C(25)	1.396(4)
C(25)-C(26)	1.387(4)
C(25)-C(28)	1.442(4)
C(26)-C(27)	1.372(4)

C(28)-C(29)	1.187(4)
C(29)-C(30)	1.477(4)
C(30)-C(31)	1.515(4)
C(30)-C(32)	1.515(4)
C(31)-H(31A)	0.9600
C(31)-H(31B)	0.9600
C(31)-H(31C)	0.9600
C(32)-H(32A)	0.9600
C(32)-H(32B)	0.9600
C(32)-H(32C)	0.9600
C(33)-C(34)	1.522(4)
C(33)-H(33A)	0.9600
C(33)-H(33B)	0.9600
C(33)-H(33C)	0.9600
C(34)-C(36)	1.471(4)
C(34)-C(35)	1.515(4)
C(35)-H(35A)	0.9600
C(35)-H(35B)	0.9600
C(35)-H(35C)	0.9600
C(36)-C(37)	1.191(4)
C(37)-C(38)	1.446(4)
C(38)-C(39)	1.379(4)
C(38)-C(40)	1.386(4)
C(39)-C(40)#1	1.377(4)
C(40)-C(39)#1	1.377(4)
C(41)-C(42)	1.513(4)
C(41)-H(41A)	0.9600
C(41)-H(41B)	0.9600
C(41)-H(41C)	0.9600
C(42)-C(44)	1.474(4)
C(42)-C(43)	1.512(4)
C(43)-H(43A)	0.9600
C(43)-H(43B)	0.9600
C(43)-H(43C)	0.9600
C(44)-C(45)	1.187(4)
C(45)-C(46)	1.448(4)

C(46)-C(47)	1.388(4)
C(46)-C(48)	1.397(4)
C(47)-C(48)#2	1.371(4)
C(48)-C(47)#2	1.371(4)

C(3)-O(1)-H(1)	109.5
C(14)-O(2)-H(2)	109.5
C(30)-O(3)-H(3)	109.5
C(18)-O(4)-H(4)	109.5
C(42)-O(5)-H(5)	109.5
C(34)-O(6)-H(6)	109.5
C(3)-C(1)-H(1A)	109.5
C(3)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(3)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
C(3)-C(2)-H(2A)	109.5
C(3)-C(2)-H(2B)	109.5
H(2A)-C(2)-H(2B)	109.5
C(3)-C(2)-H(2C)	109.5
H(2A)-C(2)-H(2C)	109.5
H(2B)-C(2)-H(2C)	109.5
O(1)-C(3)-C(4)	110.5(2)
O(1)-C(3)-C(2)	105.8(2)
C(4)-C(3)-C(2)	108.4(3)
O(1)-C(3)-C(1)	108.3(2)
C(4)-C(3)-C(1)	111.1(3)
C(2)-C(3)-C(1)	112.6(3)
C(5)-C(4)-C(3)	175.7(3)
C(4)-C(5)-C(6)	179.1(3)
C(7)-C(6)-C(11)	117.6(3)
C(7)-C(6)-C(5)	121.3(3)
C(11)-C(6)-C(5)	121.1(3)
F(1D)-C(7)-C(8)	118.9(5)
F(1D)-C(7)-C(6)	120.2(5)

C(8)-C(7)-C(6)	120.8(3)
F(1A)-C(8)-C(7)	119.1(4)
F(1A)-C(8)-C(9)	119.1(4)
C(7)-C(8)-C(9)	121.7(3)
C(10)-C(9)-C(8)	117.5(3)
C(10)-C(9)-C(12)	121.0(3)
C(8)-C(9)-C(12)	121.5(3)
F(1B)-C(10)-C(11)	118.4(7)
F(1B)-C(10)-C(9)	120.6(7)
C(11)-C(10)-C(9)	121.0(3)
F(1C)-C(11)-C(10)	117.8(5)
F(1C)-C(11)-C(6)	120.9(5)
C(10)-C(11)-C(6)	121.3(3)
C(13)-C(12)-C(9)	178.4(3)
C(12)-C(13)-C(14)	179.4(3)
O(2)-C(14)-C(13)	108.9(2)
O(2)-C(14)-C(16)	109.6(2)
C(13)-C(14)-C(16)	111.0(3)
O(2)-C(14)-C(15)	106.2(2)
C(13)-C(14)-C(15)	110.2(3)
C(16)-C(14)-C(15)	110.9(3)
C(14)-C(15)-H(15A)	109.5
C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(14)-C(16)-H(16A)	109.5
C(14)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(14)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(18)-C(17)-H(17A)	109.5
C(18)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5

C(18)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
O(4)-C(18)-C(20)	108.5(2)
O(4)-C(18)-C(19)	105.9(3)
C(20)-C(18)-C(19)	111.0(3)
O(4)-C(18)-C(17)	110.2(3)
C(20)-C(18)-C(17)	110.4(3)
C(19)-C(18)-C(17)	110.7(2)
C(18)-C(19)-H(19A)	109.5
C(18)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
C(18)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(21)-C(20)-C(18)	177.2(3)
C(20)-C(21)-C(22)	173.4(3)
C(23)-C(22)-C(27)	118.0(3)
C(23)-C(22)-C(21)	119.6(3)
C(27)-C(22)-C(21)	122.2(3)
F(2C)-C(23)-C(24)	123.4(5)
F(2C)-C(23)-C(22)	115.5(4)
C(24)-C(23)-C(22)	121.2(3)
F(2D)-C(24)-C(23)	123.5(16)
F(2D)-C(24)-C(25)	116.1(16)
C(23)-C(24)-C(25)	120.3(3)
C(26)-C(25)-C(24)	118.5(3)
C(26)-C(25)-C(28)	122.6(3)
C(24)-C(25)-C(28)	118.9(3)
F(2B)-C(26)-C(27)	119.4(3)
F(2B)-C(26)-C(25)	119.8(3)
C(27)-C(26)-C(25)	120.8(3)
F(2A)-C(27)-C(26)	122.1(4)
F(2A)-C(27)-C(22)	116.6(3)
C(26)-C(27)-C(22)	121.3(3)
C(29)-C(28)-C(25)	174.9(3)

C(28)-C(29)-C(30)	178.4(3)
O(3)-C(30)-C(29)	108.6(2)
O(3)-C(30)-C(31)	110.0(2)
C(29)-C(30)-C(31)	111.1(3)
O(3)-C(30)-C(32)	105.7(2)
C(29)-C(30)-C(32)	110.1(2)
C(31)-C(30)-C(32)	111.2(3)
C(30)-C(31)-H(31A)	109.5
C(30)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
C(30)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
C(30)-C(32)-H(32A)	109.5
C(30)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
C(30)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(34)-C(33)-H(33A)	109.5
C(34)-C(33)-H(33B)	109.5
H(33A)-C(33)-H(33B)	109.5
C(34)-C(33)-H(33C)	109.5
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
O(6)-C(34)-C(36)	110.2(2)
O(6)-C(34)-C(35)	106.8(2)
C(36)-C(34)-C(35)	109.4(3)
O(6)-C(34)-C(33)	109.3(3)
C(36)-C(34)-C(33)	109.3(2)
C(35)-C(34)-C(33)	111.9(3)
C(34)-C(35)-H(35A)	109.5
C(34)-C(35)-H(35B)	109.5
H(35A)-C(35)-H(35B)	109.5
C(34)-C(35)-H(35C)	109.5
H(35A)-C(35)-H(35C)	109.5

H(35B)-C(35)-H(35C)	109.5
C(37)-C(36)-C(34)	173.4(3)
C(36)-C(37)-C(38)	176.7(3)
C(39)-C(38)-C(40)	118.1(3)
C(39)-C(38)-C(37)	121.4(3)
C(40)-C(38)-C(37)	120.6(3)
F(4A)-C(39)-C(40)#1	120.6(4)
F(4A)-C(39)-C(38)	117.5(4)
C(40)#1-C(39)-C(38)	121.5(3)
F(4B)-C(40)-C(39)#1	121.4(5)
F(4B)-C(40)-C(38)	117.7(5)
C(39)#1-C(40)-C(38)	120.4(3)
C(42)-C(41)-H(41A)	109.5
C(42)-C(41)-H(41B)	109.5
H(41A)-C(41)-H(41B)	109.5
C(42)-C(41)-H(41C)	109.5
H(41A)-C(41)-H(41C)	109.5
H(41B)-C(41)-H(41C)	109.5
O(5)-C(42)-C(44)	109.1(2)
O(5)-C(42)-C(41)	105.6(2)
C(44)-C(42)-C(41)	109.1(2)
O(5)-C(42)-C(43)	109.5(2)
C(44)-C(42)-C(43)	111.7(2)
C(41)-C(42)-C(43)	111.6(3)
C(42)-C(43)-H(43A)	109.5
C(42)-C(43)-H(43B)	109.5
H(43A)-C(43)-H(43B)	109.5
C(42)-C(43)-H(43C)	109.5
H(43A)-C(43)-H(43C)	109.5
H(43B)-C(43)-H(43C)	109.5
C(45)-C(44)-C(42)	177.2(3)
C(44)-C(45)-C(46)	177.8(3)
C(47)-C(46)-C(48)	118.5(3)
C(47)-C(46)-C(45)	121.4(3)
C(48)-C(46)-C(45)	120.2(3)
F(3A)-C(47)-C(48)#2	117.5(4)

F(3A)-C(47)-C(46)	120.8(4)
C(48)#2-C(47)-C(46)	121.7(3)
F(3B)-C(48)-C(47)#2	118.8(6)
F(3B)-C(48)-C(46)	121.3(6)
C(47)#2-C(48)-C(46)	119.8(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1 #2 -x+1,-y+1,-z

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

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	60(1)	68(1)	50(1)	17(1)	10(1)	15(1)
O(2)	63(1)	63(1)	52(1)	17(1)	10(1)	13(1)
O(3)	54(1)	69(2)	67(2)	28(1)	4(1)	3(1)
O(4)	57(1)	72(1)	63(2)	12(1)	10(1)	0(1)
O(5)	56(1)	49(1)	67(2)	3(1)	14(1)	-1(1)
O(6)	65(2)	52(1)	74(2)	17(1)	11(1)	5(1)
C(1)	71(2)	69(2)	81(3)	13(2)	19(2)	-9(2)
C(2)	93(3)	85(2)	86(3)	48(2)	41(2)	43(2)
C(3)	60(2)	49(2)	51(2)	16(2)	18(2)	6(2)
C(4)	61(2)	50(2)	55(2)	15(2)	11(2)	2(2)
C(5)	68(2)	53(2)	49(2)	13(2)	13(2)	-4(2)
C(6)	64(2)	54(2)	45(2)	12(2)	11(2)	-9(2)
C(7)	76(2)	59(2)	54(2)	21(2)	17(2)	2(2)
C(8)	71(2)	64(2)	58(2)	17(2)	20(2)	6(2)
C(9)	68(2)	57(2)	42(2)	15(2)	8(2)	-10(2)
C(10)	81(3)	67(2)	70(2)	32(2)	25(2)	10(2)
C(11)	74(2)	73(2)	67(2)	30(2)	21(2)	8(2)
C(12)	72(2)	56(2)	54(2)	17(2)	15(2)	-6(2)
C(13)	66(2)	55(2)	54(2)	15(2)	9(2)	1(2)
C(14)	58(2)	49(2)	46(2)	14(2)	11(2)	1(2)
C(15)	98(3)	66(2)	77(3)	27(2)	24(2)	19(2)
C(16)	68(2)	82(2)	75(2)	22(2)	1(2)	-11(2)
C(17)	76(2)	57(2)	80(3)	22(2)	6(2)	7(2)
C(18)	53(2)	53(2)	58(2)	17(2)	5(2)	8(2)
C(19)	65(2)	73(2)	98(3)	10(2)	-7(2)	17(2)
C(20)	63(2)	52(2)	53(2)	16(2)	7(2)	11(2)
C(21)	64(2)	54(2)	47(2)	17(2)	3(2)	10(2)
C(22)	61(2)	47(2)	41(2)	8(2)	-1(2)	13(2)
C(23)	74(2)	48(2)	63(2)	17(2)	9(2)	7(2)
C(24)	69(2)	46(2)	61(2)	12(2)	10(2)	7(2)
C(25)	58(2)	51(2)	43(2)	10(2)	-1(2)	12(2)

C(26)	64(2)	53(2)	47(2)	11(2)	8(2)	9(2)
C(27)	67(2)	50(2)	53(2)	15(2)	4(2)	5(2)
C(28)	67(2)	60(2)	50(2)	11(2)	5(2)	14(2)
C(29)	56(2)	56(2)	58(2)	12(2)	10(2)	10(2)
C(30)	57(2)	48(2)	53(2)	15(2)	12(2)	7(2)
C(31)	65(2)	70(2)	87(3)	16(2)	32(2)	8(2)
C(32)	72(2)	57(2)	76(2)	7(2)	-2(2)	3(2)
C(33)	88(3)	67(2)	59(2)	18(2)	0(2)	14(2)
C(34)	62(2)	48(2)	52(2)	16(2)	-2(2)	6(2)
C(35)	58(2)	71(2)	98(3)	35(2)	-1(2)	2(2)
C(36)	68(2)	62(2)	54(2)	19(2)	-5(2)	10(2)
C(37)	74(2)	59(2)	54(2)	15(2)	-3(2)	3(2)
C(38)	75(2)	52(2)	44(2)	12(2)	-10(2)	8(2)
C(39)	71(2)	76(2)	56(2)	24(2)	3(2)	17(2)
C(40)	72(2)	70(2)	56(2)	23(2)	2(2)	20(2)
C(41)	80(2)	80(2)	55(2)	12(2)	10(2)	-4(2)
C(42)	52(2)	44(2)	48(2)	7(2)	3(2)	0(1)
C(43)	68(2)	74(2)	76(2)	10(2)	-10(2)	18(2)
C(44)	56(2)	47(2)	51(2)	9(2)	-4(2)	-1(2)
C(45)	64(2)	46(2)	57(2)	11(2)	-5(2)	-1(2)
C(46)	65(2)	40(2)	50(2)	6(2)	-10(2)	7(2)
C(47)	64(2)	53(2)	57(2)	10(2)	3(2)	0(2)
C(48)	63(2)	53(2)	52(2)	9(2)	-2(2)	1(2)

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

	x	y	z	U(eq)
H(1)	8276	7976	2118	87
H(2)	-992	6646	6723	87
H(3)	14979	-2258	1474	92
H(4)	7354	3346	2799	96
H(5)	11745	7372	1849	87
H(6)	4227	2616	3191	94
H(1A)	10546	8777	3016	111
H(1B)	9542	9553	3342	111
H(1C)	10169	9446	2629	111
H(2A)	6276	9735	2283	121
H(2B)	5434	9769	2966	121
H(2C)	4276	9172	2363	121
H(15A)	2423	5249	6402	116
H(15B)	1104	4750	5774	116
H(15C)	213	4770	6450	116
H(16A)	-3390	5244	6153	113
H(16B)	-2789	5182	5440	113
H(16C)	-3542	5973	5826	113
H(17A)	6801	4454	2063	105
H(17B)	7435	3983	1401	105
H(17C)	8931	3976	2042	105
H(19A)	2815	2848	1762	121
H(19B)	3543	3314	1246	121
H(19C)	3064	3766	1931	121
H(31A)	16872	-1946	630	108
H(31B)	15607	-2148	-66	108
H(31C)	16348	-2823	280	108
H(32A)	12584	-3430	171	104
H(32B)	11618	-2798	-190	104
H(32C)	10627	-2946	438	104
H(33A)	2630	1625	2238	106

H(33B)	2549	816	2454	106
H(33C)	4726	1367	2612	106
H(35A)	-618	1976	3584	110
H(35B)	-735	1163	3103	110
H(35C)	-769	1939	2836	110
H(41A)	10005	6800	3102	108
H(41B)	8735	6007	2749	108
H(41C)	7796	6804	2658	108
H(43A)	13476	6208	1870	111
H(43B)	12419	5642	2266	111
H(43C)	13477	6468	2622	111

Table 6. Torsion angles [°] for Compound **4**.

O(1)-C(3)-C(4)-C(5)	-142(4)
C(2)-C(3)-C(4)-C(5)	-26(5)
C(1)-C(3)-C(4)-C(5)	98(4)
C(3)-C(4)-C(5)-C(6)	126(23)
C(4)-C(5)-C(6)-C(7)	-108(25)
C(4)-C(5)-C(6)-C(11)	72(25)
C(11)-C(6)-C(7)-F(1D)	-179.0(5)
C(5)-C(6)-C(7)-F(1D)	0.5(6)
C(11)-C(6)-C(7)-C(8)	-0.4(5)
C(5)-C(6)-C(7)-C(8)	179.1(3)
F(1D)-C(7)-C(8)-F(1A)	-6.0(7)
C(6)-C(7)-C(8)-F(1A)	175.4(4)
F(1D)-C(7)-C(8)-C(9)	178.1(5)
C(6)-C(7)-C(8)-C(9)	-0.5(5)
F(1A)-C(8)-C(9)-C(10)	-175.0(4)
C(7)-C(8)-C(9)-C(10)	0.9(5)
F(1A)-C(8)-C(9)-C(12)	5.6(5)
C(7)-C(8)-C(9)-C(12)	-178.5(3)
C(8)-C(9)-C(10)-F(1B)	178.7(7)
C(12)-C(9)-C(10)-F(1B)	-1.9(9)
C(8)-C(9)-C(10)-C(11)	-0.4(5)
C(12)-C(9)-C(10)-C(11)	179.0(3)
F(1B)-C(10)-C(11)-F(1C)	1.6(9)
C(9)-C(10)-C(11)-F(1C)	-179.3(5)
F(1B)-C(10)-C(11)-C(6)	-179.6(7)
C(9)-C(10)-C(11)-C(6)	-0.5(5)
C(7)-C(6)-C(11)-F(1C)	179.6(5)
C(5)-C(6)-C(11)-F(1C)	0.1(6)
C(7)-C(6)-C(11)-C(10)	1.0(5)
C(5)-C(6)-C(11)-C(10)	-178.6(3)
C(10)-C(9)-C(12)-C(13)	-44(13)
C(8)-C(9)-C(12)-C(13)	136(13)
C(9)-C(12)-C(13)-C(14)	-9(48)
C(12)-C(13)-C(14)-O(2)	170(100)

C(12)-C(13)-C(14)-C(16)	-70(38)
C(12)-C(13)-C(14)-C(15)	54(38)
O(4)-C(18)-C(20)-C(21)	-24(7)
C(19)-C(18)-C(20)-C(21)	-140(7)
C(17)-C(18)-C(20)-C(21)	97(7)
C(18)-C(20)-C(21)-C(22)	30(9)
C(20)-C(21)-C(22)-C(23)	45(3)
C(20)-C(21)-C(22)-C(27)	-130(3)
C(27)-C(22)-C(23)-F(2C)	-176.4(4)
C(21)-C(22)-C(23)-F(2C)	8.3(6)
C(27)-C(22)-C(23)-C(24)	2.9(4)
C(21)-C(22)-C(23)-C(24)	-172.4(3)
F(2C)-C(23)-C(24)-F(2D)	1(2)
C(22)-C(23)-C(24)-F(2D)	-178.7(19)
F(2C)-C(23)-C(24)-C(25)	178.2(5)
C(22)-C(23)-C(24)-C(25)	-1.0(5)
F(2D)-C(24)-C(25)-C(26)	176.6(17)
C(23)-C(24)-C(25)-C(26)	-1.2(4)
F(2D)-C(24)-C(25)-C(28)	-4.3(18)
C(23)-C(24)-C(25)-C(28)	177.9(3)
C(24)-C(25)-C(26)-F(2B)	-175.5(3)
C(28)-C(25)-C(26)-F(2B)	5.5(5)
C(24)-C(25)-C(26)-C(27)	1.5(4)
C(28)-C(25)-C(26)-C(27)	-177.6(3)
F(2B)-C(26)-C(27)-F(2A)	-4.0(5)
C(25)-C(26)-C(27)-F(2A)	179.0(4)
F(2B)-C(26)-C(27)-C(22)	177.4(3)
C(25)-C(26)-C(27)-C(22)	0.4(4)
C(23)-C(22)-C(27)-F(2A)	178.7(3)
C(21)-C(22)-C(27)-F(2A)	-6.1(5)
C(23)-C(22)-C(27)-C(26)	-2.6(4)
C(21)-C(22)-C(27)-C(26)	172.6(3)
C(26)-C(25)-C(28)-C(29)	160(4)
C(24)-C(25)-C(28)-C(29)	-19(4)
C(25)-C(28)-C(29)-C(30)	75(14)
C(28)-C(29)-C(30)-O(3)	-112(13)

C(28)-C(29)-C(30)-C(31)	127(13)
C(28)-C(29)-C(30)-C(32)	3(13)
O(6)-C(34)-C(36)-C(37)	166(3)
C(35)-C(34)-C(36)-C(37)	49(3)
C(33)-C(34)-C(36)-C(37)	-74(3)
C(34)-C(36)-C(37)-C(38)	7(9)
C(36)-C(37)-C(38)-C(39)	102(7)
C(36)-C(37)-C(38)-C(40)	-77(7)
C(40)-C(38)-C(39)-F(4A)	-173.0(4)
C(37)-C(38)-C(39)-F(4A)	8.1(5)
C(40)-C(38)-C(39)-C(40)#1	-0.4(5)
C(37)-C(38)-C(39)-C(40)#1	-179.3(3)
C(39)-C(38)-C(40)-F(4B)	171.7(5)
C(37)-C(38)-C(40)-F(4B)	-9.4(6)
C(39)-C(38)-C(40)-C(39)#1	0.3(5)
C(37)-C(38)-C(40)-C(39)#1	179.3(3)
O(5)-C(42)-C(44)-C(45)	-90(6)
C(41)-C(42)-C(44)-C(45)	24(6)
C(43)-C(42)-C(44)-C(45)	148(6)
C(42)-C(44)-C(45)-C(46)	21(14)
C(44)-C(45)-C(46)-C(47)	126(9)
C(44)-C(45)-C(46)-C(48)	-53(9)
C(48)-C(46)-C(47)-F(3A)	178.8(3)
C(45)-C(46)-C(47)-F(3A)	-0.6(5)
C(48)-C(46)-C(47)-C(48)#2	0.5(5)
C(45)-C(46)-C(47)-C(48)#2	-179.0(3)
C(47)-C(46)-C(48)-F(3B)	178.7(6)
C(45)-C(46)-C(48)-F(3B)	-1.9(7)
C(47)-C(46)-C(48)-C(47)#2	-0.5(5)
C(45)-C(46)-C(48)-C(47)#2	179.0(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1 #2 -x+1,-y+1,-z