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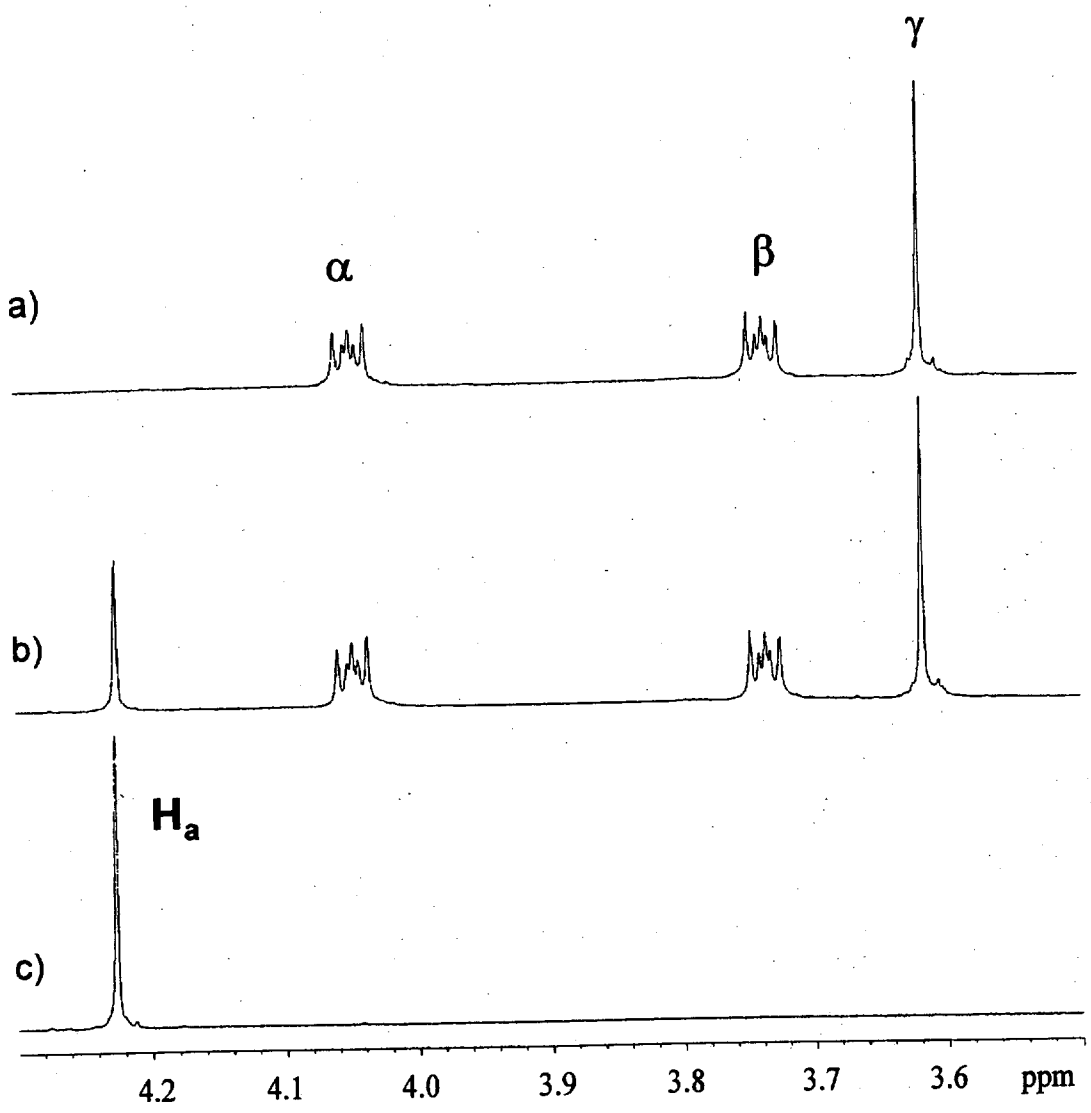

Figure 1S. Stacked ^{1}H NMR plots (400 MHz, CD_3CN, ambient *T*) for a) **1**, b) **1** + **3**·PF_6 (1:1 stoichiometry, 0.01 M), and c) **3**·PF_6.

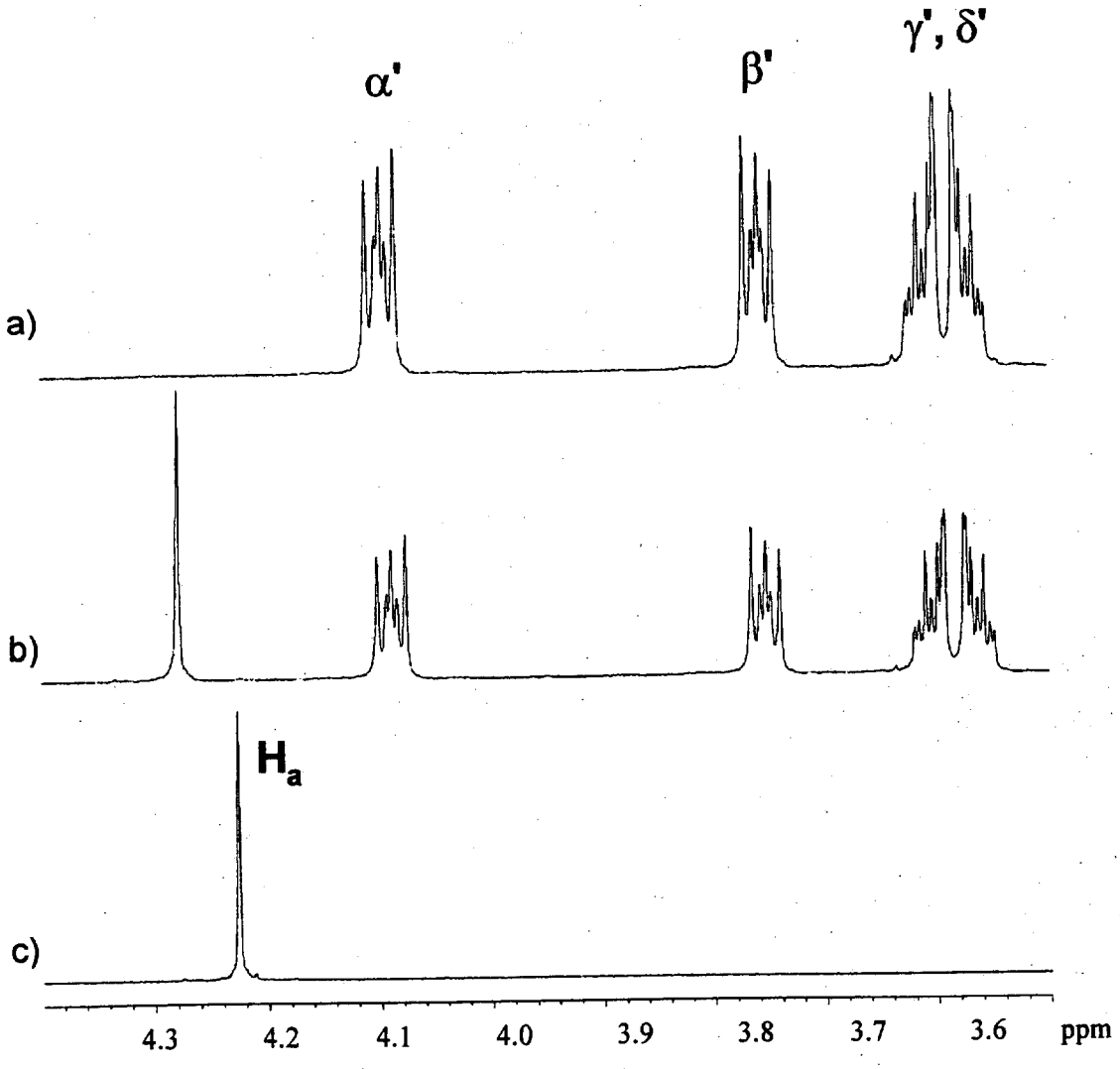

Figure 2S. Stacked ^{1}H NMR plots (400 MHz, CD_3CN, ambient *T*) for a) **2**, b) **2** + **3**·PF_6 (1:1 stoichiometry, 0.01 M), and c) **3**·PF_6.

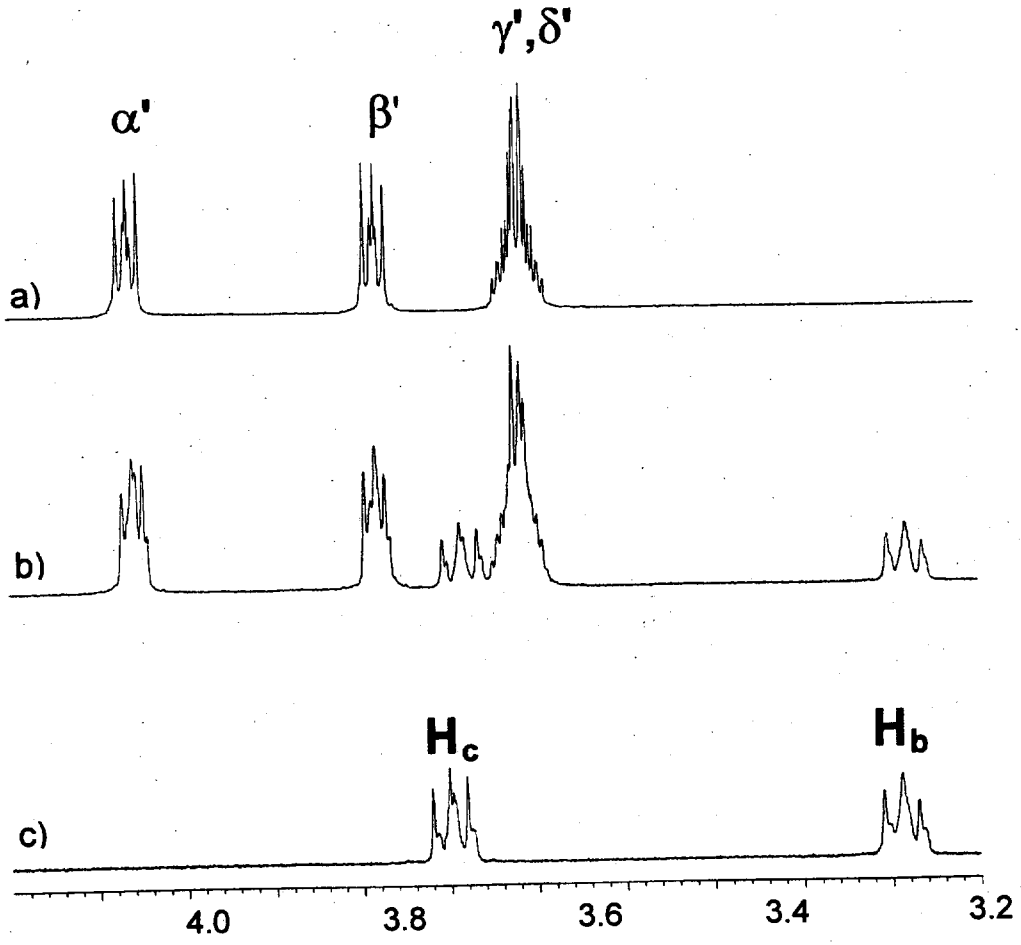

Figure 3S. Stacked ^{1}H NMR plots (400 MHz, acetone-d_6, ambient *T*) for a) **2**, b) **2** + **5**·PF_6 (1:1 stoichiometry, 0.01 M), and c) **5**·PF_6.

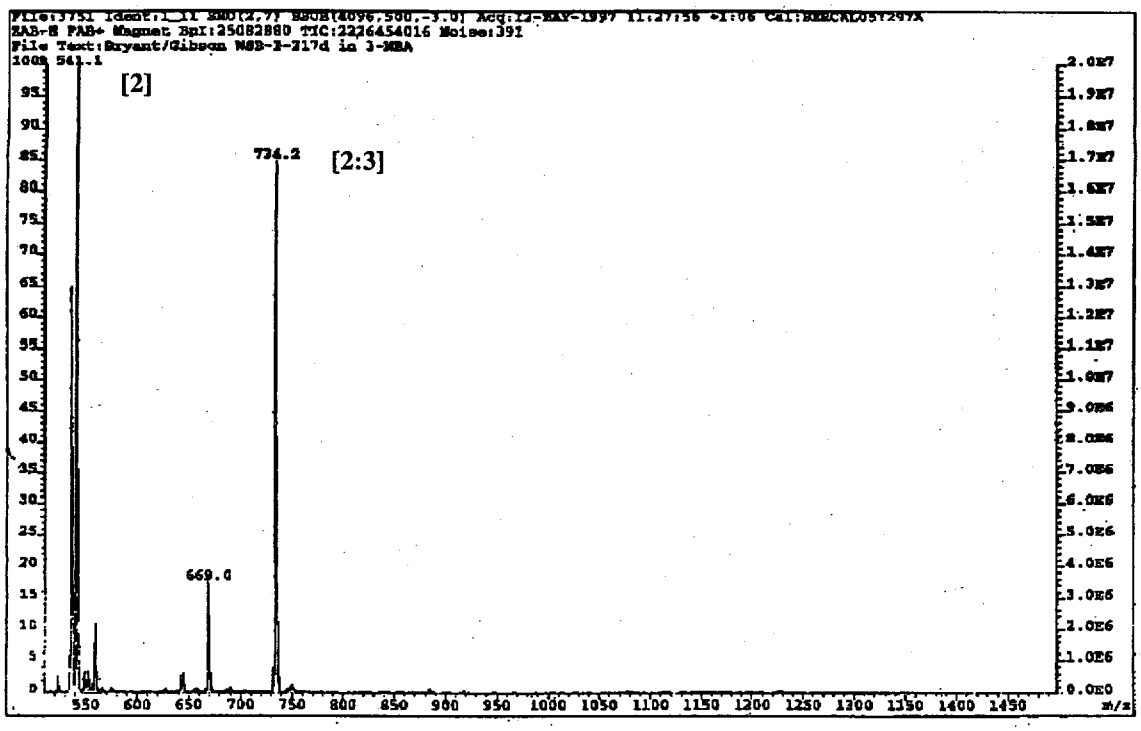

Figure 4S. LRFAB-MS for **2** + **3**·PF_6.

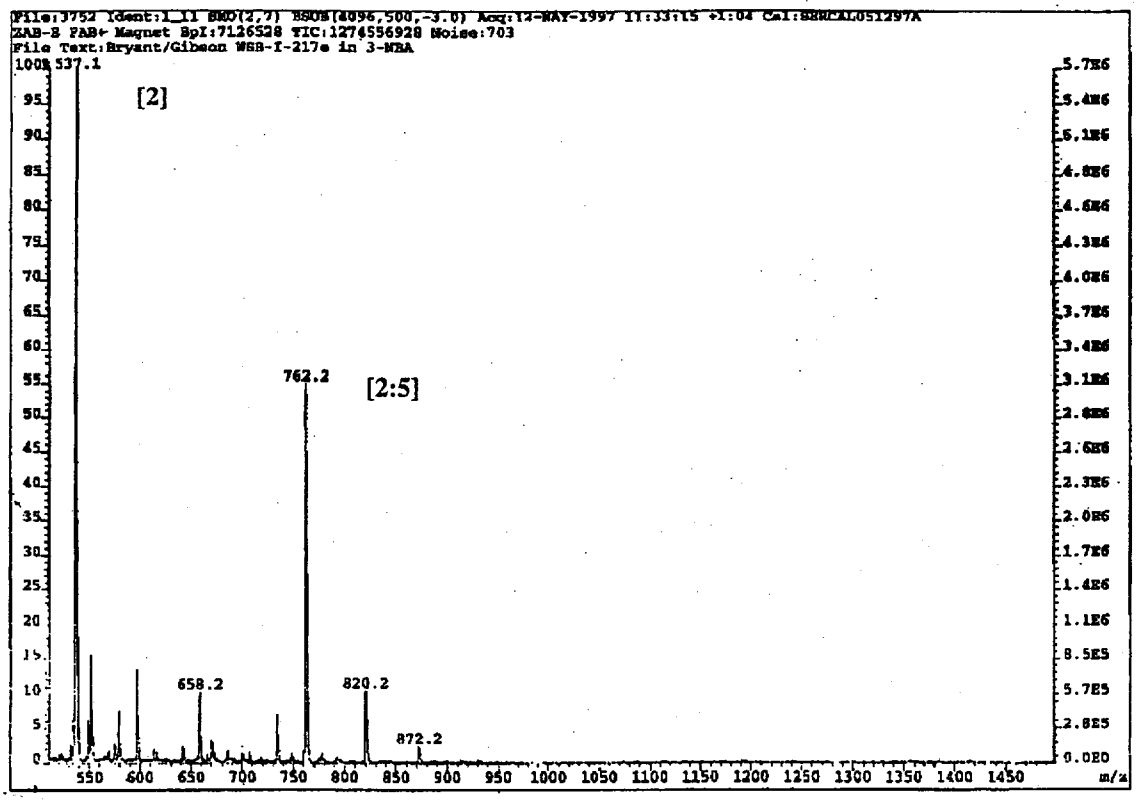

Figure 5S. LRFAB-MS for **2** + **5**·PF_6.

Table 1S. Crystal data and structure refinement for **1**.

Identification code	giba1
Empirical formula	C24 H32 O8
Formula weight	448.50
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	a = 11.382(3) Å alpha = 90° b = 16.111(2) Å beta = 102.479(11)° c = 12.8971(11) Å gamma = 90°
Volume, Z	2309.2(7) $Å^3$, 4
Density (calculated)	1.290 g/cm^3
Absorption coefficient	0.096 mm^{-1}
F(000)	960
Crystal size	0.2 x 0.3 x 0.8 mm
θ range for data collection	2.05 to 22.50°
Limiting indices	-1<=h<=12, -1<=k<=17, -13<=l<=13
Reflections collected	3757
Independent reflections	2913 [R(int) = 0.0216]
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2912 / 0 / 290
Goodness-of-fit on F^2	1.044
Final R indices [$I>2\sigma(I)$]	R1 = 0.0426, wR2 = 0.0925
R indices (all data)	R1 = 0.0728, wR2 = 0.1092
Extinction coefficient	0.0097(10)
Largest diff. peak and hole	0.185 and -0.143 e. $Å^{-3}$

Table 2S. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **1**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	2027(2)	1593(1)	4409(2)	61(1)
C(1)	1575(2)	786(2)	4094(2)	61(1)
C(2)	243(2)	850(2)	3736(2)	68(1)
O(2)	-276(2)	1080(1)	4585(1)	57(1)
C(3)	-1527(2)	1255(2)	4275(2)	55(1)
C(4)	-1798(2)	2123(2)	3880(2)	60(1)
O(3)	-1475(2)	2722(1)	4701(2)	63(1)
C(5)	-322(2)	3070(2)	4780(2)	60(1)
C(6)	-151(2)	3703(2)	5651(2)	57(1)
O(4)	1033(2)	4017(1)	5757(2)	68(1)
C(7)	1406(2)	4643(2)	6470(2)	49(1)
C(8)	741(2)	4942(2)	7174(2)	53(1)
C(9)	1219(3)	5575(2)	7849(2)	61(1)
C(10)	2326(2)	5918(2)	7849(2)	53(1)
C(11)	2972(2)	5616(2)	7142(2)	45(1)
C(12)	2517(2)	4975(2)	6462(2)	52(1)
O(5)	4076(2)	5906(1)	7051(1)	56(1)
C(13)	4628(2)	6540(2)	7768(2)	53(1)
C(14)	5825(2)	6732(2)	7523(2)	57(1)
O(6)	6635(2)	6075(1)	7888(1)	53(1)
C(15)	7724(2)	6132(2)	7507(2)	59(1)
C(16)	7641(2)	5742(2)	6453(2)	56(1)
O(7)	7507(2)	4865(1)	6481(2)	56(1)
C(17)	6305(2)	4589(2)	6171(2)	56(1)
C(18)	6337(2)	3678(2)	6001(3)	65(1)
O(8)	5120(2)	3399(1)	5836(2)	68(1)
C(19)	4844(2)	2636(2)	5382(2)	47(1)
C(20)	5673(2)	2090(2)	5133(2)	56(1)
C(21)	5267(3)	1360(2)	4635(3)	65(1)
C(22)	4065(2)	1159(2)	4377(2)	61(1)
C(23)	3249(2)	1707(2)	4640(2)	46(1)
C(24)	3632(2)	2441(2)	5154(2)	46(1)

Table 3S. Bond lengths [Å] and angles [°] for **1**.

O(1)-C(23)	1.371(3)
O(1)-C(1)	1.424(3)
C(1)-C(2)	1.490(4)
C(2)-O(2)	1.402(3)
O(2)-C(3)	1.421(3)
C(3)-C(4)	1.498(4)
C(4)-O(3)	1.421(3)
O(3)-C(5)	1.411(3)
C(5)-C(6)	1.498(4)
C(6)-O(4)	1.418(3)
O(4)-C(7)	1.370(3)
C(7)-C(12)	1.376(4)
C(7)-C(8)	1.388(4)
C(8)-C(9)	1.375(4)
C(9)-C(10)	1.375(4)
C(10)-C(11)	1.378(3)
C(11)-O(5)	1.369(3)
C(11)-C(12)	1.380(4)
O(5)-C(13)	1.429(3)
C(13)-C(14)	1.496(4)
C(14)-O(6)	1.416(3)
O(6)-C(15)	1.431(3)
C(15)-C(16)	1.482(4)
C(16)-O(7)	1.422(3)
O(7)-C(17)	1.412(3)
C(17)-C(18)	1.485(4)
C(18)-O(8)	1.428(3)
O(8)-C(19)	1.368(3)
C(19)-C(20)	1.378(3)
C(19)-C(24)	1.384(3)
C(20)-C(21)	1.371(4)
C(21)-C(22)	1.375(4)
C(22)-C(23)	1.375(4)
C(23)-C(24)	1.379(3)

C(23)-O(1)-C(1)	118.1(2)
O(1)-C(1)-C(2)	107.8(2)
O(2)-C(2)-C(1)	110.4(2)
C(2)-O(2)-C(3)	113.5(2)
O(2)-C(3)-C(4)	113.6(2)
O(3)-C(4)-C(3)	112.3(2)
C(5)-O(3)-C(4)	114.2(2)
O(3)-C(5)-C(6)	107.0(2)
O(4)-C(6)-C(5)	106.4(2)
C(7)-O(4)-C(6)	118.5(2)
O(4)-C(7)-C(12)	115.6(2)
O(4)-C(7)-C(8)	124.1(2)
C(12)-C(7)-C(8)	120.2(3)
C(9)-C(8)-C(7)	118.3(3)
C(8)-C(9)-C(10)	122.4(3)
C(9)-C(10)-C(11)	118.6(3)
O(5)-C(11)-C(10)	124.5(2)
O(5)-C(11)-C(12)	115.2(2)
C(10)-C(11)-C(12)	120.3(2)
C(7)-C(12)-C(11)	120.3(2)
C(11)-O(5)-C(13)	118.2(2)
O(5)-C(13)-C(14)	107.7(2)
O(6)-C(14)-C(13)	109.4(2)

C(14)-O(6)-C(15)	112.9(2)
O(6)-C(15)-C(16)	113.6(2)
O(7)-C(16)-C(15)	112.6(2)
C(17)-O(7)-C(16)	114.1(2)
O(7)-C(17)-C(18)	107.5(2)
O(8)-C(18)-C(17)	106.2(2)
C(19)-O(8)-C(18)	118.1(2)
O(8)-C(19)-C(20)	124.6(2)
O(8)-C(19)-C(24)	114.9(2)
C(20)-C(19)-C(24)	120.4(2)
C(21)-C(20)-C(19)	118.6(3)
C(20)-C(21)-C(22)	122.2(3)
C(23)-C(22)-C(21)	118.6(3)
O(1)-C(23)-C(22)	124.6(2)
O(1)-C(23)-C(24)	114.8(2)
C(22)-C(23)-C(24)	120.6(2)
C(23)-C(24)-C(19)	119.6(2)

Table 4S. Anisotropic displacement parameters ($Å^2$ x 10^3) for **1**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
O(1)	44(1)	45(1)	93(2)	-10(1)	14(1)	-6(1)
C(1)	52(2)	55(2)	76(2)	-19(2)	11(2)	-7(2)
C(2)	50(2)	85(2)	69(2)	-32(2)	14(2)	-13(2)
O(2)	49(1)	63(1)	60(1)	-10(1)	11(1)	-5(1)
C(3)	42(2)	56(2)	68(2)	-9(2)	9(1)	-9(1)
C(4)	48(2)	69(2)	59(2)	1(2)	4(1)	-5(2)
O(3)	53(1)	54(1)	85(1)	-11(1)	22(1)	-12(1)
C(5)	54(2)	59(2)	71(2)	-5(2)	19(2)	-12(2)
C(6)	51(2)	50(2)	74(2)	2(2)	20(2)	-6(2)
O(4)	55(1)	75(2)	78(1)	-24(1)	25(1)	-18(1)
C(7)	51(2)	52(2)	44(2)	1(1)	12(1)	-1(2)
C(8)	44(2)	58(2)	59(2)	2(2)	17(1)	-1(2)
C(9)	54(2)	71(2)	64(2)	-9(2)	24(2)	3(2)
C(10)	50(2)	58(2)	51(2)	-8(1)	13(1)	4(2)
C(11)	41(2)	53(2)	41(2)	6(1)	7(1)	1(1)
C(12)	48(2)	65(2)	45(2)	-7(2)	18(1)	-2(2)
O(5)	48(1)	71(1)	50(1)	-13(1)	14(1)	-12(1)
C(13)	52(2)	50(2)	55(2)	-5(2)	8(1)	3(1)
C(14)	54(2)	49(2)	67(2)	-3(2)	12(2)	-3(2)
O(6)	50(1)	57(1)	55(1)	0(1)	15(1)	7(1)
C(15)	47(2)	57(2)	74(2)	-8(2)	12(2)	-8(2)
C(16)	53(2)	50(2)	71(2)	-1(2)	24(2)	-9(2)
O(7)	42(1)	49(1)	75(1)	-8(1)	9(1)	-4(1)
C(17)	42(2)	64(2)	61(2)	-2(2)	10(1)	-5(2)
C(18)	46(2)	62(2)	88(2)	-20(2)	15(2)	-13(2)
O(8)	49(1)	62(1)	99(2)	-29(1)	25(1)	-15(1)
C(19)	49(2)	43(2)	50(2)	-3(1)	13(1)	-4(1)
C(20)	41(2)	60(2)	65(2)	-9(2)	11(1)	-2(2)
C(21)	47(2)	60(2)	91(2)	-15(2)	20(2)	7(2)
C(22)	52(2)	49(2)	83(2)	-19(2)	17(2)	-2(2)
C(23)	40(2)	47(2)	51(2)	3(1)	11(1)	-3(1)
C(24)	45(2)	40(2)	57(2)	2(1)	18(1)	2(1)

Table 6s. Crystal data and structure refinement for **2**:$(\mathbf{3})_2$.

Identification	gib04
Empirical formula	$C_{56}H_{72}F_{12}N2010_{P2}$
Formula weight	1223.10
Temperature	222(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 11.0034(2) Å alpha = 102.9690(10)°
	b = 11.88170(10) Å beta = 113.7160(10)°
	c = 13.1435(2) Å gamma = 96.0480(10)°
Volume, Z	1495.81(4) Å^3, 1
Density (calculated)	1.358 g/cm^3
Absorption coefficient	0.167 mm^{-1}
F(000)	640
Crystal size	0.50 x 0.45 x 0.35 mm
Crystal color	colorless
θ range for data collection	1.77 to 28.12°
Limiting indices	$-14 \leq h \leq 14$, $-15 \leq k \leq 15$, $-17 \leq l \leq 17$
Reflections collected	8892
Independent reflections	6409 (R_{int} = 0.0447)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6403 / 0 / 370
Goodness-of-fit on F^2	1.238
Final R indices [I>2σ(I)]	R1 = 0.0557, wR2 = 0.2235
R indices (all data)	R1 = 0.0683, wR2 = 0.2566
Largest diff. peak and hole	0.532 and -0.463 eÅ^{-3}

Table 7S. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for **2**:(**3**)$_2$. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
P	5596(1)	3201(1)	1677(1)	42(1)
F(1)	4915(3)	1913(2)	799(2)	128(1)
F(2)	4750(2)	3781(2)	704(2)	96(1)
F(3)	4464(2)	3123(2)	2129(2)	99(1)
F(4)	6492(3)	2670(2)	2674(2)	91(1)
F(5)	6300(2)	4490(1)	2567(1)	72(1)
F(6)	6757(2)	3263(2)	1233(2)	69(1)
O(1)	-3162(1)	5101(1)	-3944(1)	37(1)
O(2)	-3937(2)	2542(1)	-4182(1)	39(1)
O(3)	-3177(2)	701(1)	-3249(1)	42(1)
O(4)	-2502(1)	891(1)	-849(1)	34(1)
O(5)	64(1)	1409(1)	1325(1)	34(1)
N	-3265(1)	2966(1)	-1753(1)	28(1)
C(1)	-2055(2)	5935(2)	-3731(2)	30(1)
C(2)	-1380(2)	5878(2)	-4439(2)	37(1)
C(3)	-203(2)	6741(2)	-4066(2)	43(1)
C(4)	294(2)	7650(2)	-3034(2)	39(1)
C(5)	-424(2)	7719(2)	-2357(2)	29(1)
C(6)	-1594(2)	6855(2)	-2700(2)	30(1)
C(7)	-3749(2)	4149(2)	-5013(2)	37(1)
C(8)	-4690(2)	3220(2)	-4895(2)	40(1)
C(9)	-3700(4)	1520(2)	-4839(2)	63(1)
C(10)	-2662(4)	1020(2)	-4018(2)	63(1)
C(11)	-2378(2)	-4(2)	-2618(2)	41(1)
C(12)	-2858(2)	-213(2)	-1738(2)	38(1)
C(13)	-2419(2)	725(2)	229(2)	36(1)
C(14)	-1077(2)	442(2)	931(2)	35(1)
C(15)	22(2)	3093(2)	-510(2)	39(1)
C(16)	1202(2)	2876(2)	-573(2)	53(1)
C(17)	1582(3)	3267(3)	-1342(3)	67(1)
C(18)	798(3)	3886(3)	-2030(2)	76(1)
C(19)	-411(3)	4096(2)	-1964(2)	55(1)
C(20)	-792(2)	3706(2)	-1196(2)	34(1)
C(21)	-2049(2)	3990(2)	-1083(2)	50(1)
C(22)	-4515(2)	3284(2)	-1652(2)	38(1)
C(23)	-5745(2)	2284(2)	-2353(2)	32(1)
C(24)	-6843(2)	2388(2)	-3319(2)	40(1)
C(25)	-7998(2)	1478(2)	-3936(2)	50(1)
C(26)	-8053(2)	434(2)	-3631(2)	51(1)
C(27)	-6953(2)	306(2)	-2688(2)	47(1)
C(28)	-5818(2)	1227(2)	-2053(2)	39(1)

Table 8S. Bond lengths [Å] and angles [°] for **2**:$(3)_2$.

P-F(1)	1.583(2)
P-F(3)	1.584(2)
P-F(5)	1.592(2)
P-F(4)	1.597(2)
P-F(2)	1.597(2)
P-F(6)	1.606(2)
O(1)-C(1)	1.375(2)
O(1)-C(7)	1.440(2)
O(2)-C(9)	1.436(3)
O(2)-C(8)	1.439(2)
O(3)-C(11)	1.430(2)
O(3)-C(10)	1.440(3)
O(4)-C(12)	1.441(2)
O(4)-C(13)	1.442(2)
O(5)-C(5)#1	1.386(2)
O(5)-C(14)	1.439(2)
N-C(21)	1.508(2)
N-C(22)	1.512(2)
C(1)-C(6)	1.398(2)
C(1)-C(2)	1.401(3)
C(2)-C(3)	1.397(3)
C(3)-C(4)	1.388(3)
C(4)-C(5)	1.404(3)
C(5)-O(5)#1	1.386(2)
C(5)-C(6)	1.400(2)
C(7)-C(8)	1.509(3)
C(9)-C(10)	1.504(4)
C(11)-C(12)	1.507(3)
C(13)-C(14)	1.514(3)
C(15)-C(16)	1.381(3)
C(15)-C(20)	1.389(3)
C(16)-C(17)	1.386(4)
C(17)-C(18)	1.381(5)
C(18)-C(19)	1.412(4)
C(19)-C(20)	1.386(3)
C(20)-C(21)	1.513(3)
C(22)-C(23)	1.503(2)
C(23)-C(24)	1.398(3)
C(23)-C(28)	1.403(3)
C(24)-C(25)	1.389(3)
C(25)-C(26)	1.390(4)
C(26)-C(27)	1.393(3)
C(27)-C(28)	1.385(3)

F(1)-P-F(3)	90.89(13)
F(1)-P-F(5)	179.2(2)
F(3)-P-F(5)	89.60(12)
F(1)-P-F(4)	90.9(2)
F(3)-P-F(4)	88.48(13)
F(5)-P-F(4)	88.51(11)
F(1)-P-F(2)	91.2(2)
F(3)-P-F(2)	92.71(13)
F(5)-P-F(2)	89.36(11)
F(4)-P-F(2)	177.55(13)
F(1)-P-F(6)	88.82(12)
F(3)-P-F(6)	178.93(12)
F(5)-P-F(6)	90.68(10)

F(4)-P-F(6)	90.49(11)
F(2)-P-F(6)	88.33(10)
C(1)-O(1)-C(7)	118.49(14)
C(9)-O(2)-C(8)	112.9(2)
C(11)-O(3)-C(10)	110.9(2)
C(12)-O(4)-C(13)	112.52(13)
C(5)#1-O(5)-C(14)	118.99(14)
C(21)-N-C(22)	111.53(13)
O(1)-C(1)-C(6)	114.5(2)
O(1)-C(1)-C(2)	124.6(2)
C(6)-C(1)-C(2)	120.9(2)
C(3)-C(2)-C(1)	118.2(2)
C(4)-C(3)-C(2)	122.0(2)
C(3)-C(4)-C(5)	119.0(2)
O(5)#1-C(5)-C(6)	114.7(2)
O(5)#1-C(5)-C(4)	125.1(2)
C(6)-C(5)-C(4)	120.2(2)
C(1)-C(6)-C(5)	119.6(2)
O(1)-C(7)-C(8)	107.3(2)
O(2)-C(8)-C(7)	111.2(2)
O(2)-C(9)-C(10)	109.3(2)
O(3)-C(10)-C(9)	108.5(2)
O(3)-C(11)-C(12)	109.2(2)
O(4)-C(12)-C(11)	108.6(2)
O(4)-C(13)-C(14)	112.0(2)
O(5)-C(14)-C(13)	112.60(14)
C(16)-C(15)-C(20)	121.4(2)
C(15)-C(16)-C(17)	119.6(2)
C(18)-C(17)-C(16)	120.4(2)
C(17)-C(18)-C(19)	119.6(2)
C(20)-C(19)-C(18)	120.1(2)
C(19)-C(20)-C(15)	118.9(2)
C(19)-C(20)-C(21)	120.0(2)
C(15)-C(20)-C(21)	121.1(2)
N-C(21)-C(20)	112.6(2)
C(23)-C(22)-N	111.92(14)
C(24)-C(23)-C(28)	118.5(2)
C(24)-C(23)-C(22)	120.7(2)
C(28)-C(23)-C(22)	120.8(2)
C(25)-C(24)-C(23)	120.4(2)
C(26)-C(25)-C(24)	120.4(2)
C(25)-C(26)-C(27)	119.9(2)
C(28)-C(27)-C(26)	119.7(2)
C(27)-C(28)-C(23)	121.1(2)

Symmetry transformations used to generate equivalent atoms:

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

Table 9S. Anisotropic displacement parameters [$Å^2 \times 10^3$] for **2**:$(\mathbf{3})_2$. The anisotropic displacement factor exponent takes the form: $-2\pi 2[h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
P	43(1)	41(1)	39(1)	3(1)	18(1)	6(1)
F(1)	174(2)	80(1)	102(2)	-44(1)	91(2)	-58(1)
F(2)	83(1)	157(2)	59(1)	44(1)	27(1)	66(1)
F(3)	65(1)	151(2)	75(1)	1(1)	48(1)	-2(1)
F(4)	128(2)	90(1)	96(1)	59(1)	63(1)	61(1)
F(5)	97(1)	45(1)	61(1)	3(1)	31(1)	2(1)
F(6)	68(1)	74(1)	88(1)	29(1)	52(1)	27(1)
O(1)	36(1)	33(1)	35(1)	2(1)	15(1)	-3(1)
O(2)	52(1)	29(1)	31(1)	9(1)	13(1)	9(1)
O(3)	56(1)	36(1)	34(1)	12(1)	16(1)	21(1)
O(4)	36(1)	25(1)	32(1)	6(1)	7(1)	6(1)
O(5)	29(1)	32(1)	32(1)	5(1)	9(1)	1(1)
N	24(1)	23(1)	30(1)	2(1)	8(1)	3(1)
C(1)	28(1)	28(1)	30(1)	10(1)	9(1)	6(1)
C(2)	40(1)	38(1)	31(1)	7(1)	15(1)	5(1)
C(3)	44(1)	48(1)	41(1)	11(1)	25(1)	4(1)
C(4)	34(1)	40(1)	40(1)	11(1)	16(1)	0(1)
C(5)	26(1)	30(1)	28(1)	10(1)	7(1)	6(1)
C(6)	28(1)	29(1)	32(1)	10(1)	12(1)	7(1)
C(7)	42(1)	29(1)	29(1)	7(1)	9(1)	2(1)
C(8)	38(1)	33(1)	37(1)	13(1)	5(1)	1(1)
C(9)	116(2)	37(1)	38(1)	11(1)	34(1)	29(1)
C(10)	111(2)	51(1)	60(1)	26(1)	57(2)	45(1)
C(11)	51(1)	33(1)	38(1)	10(1)	16(1)	21(1)
C(12)	43(1)	26(1)	35(1)	7(1)	8(1)	6(1)
C(13)	31(1)	35(1)	35(1)	9(1)	10(1)	4(1)
C(14)	34(1)	28(1)	35(1)	9(1)	9(1)	2(1)
C(15)	34(1)	34(1)	41(1)	12(1)	9(1)	-2(1)
C(16)	38(1)	41(1)	63(1)	7(1)	8(1)	13(1)
C(17)	40(1)	86(2)	62(2)	-2(1)	23(1)	13(1)
C(18)	64(2)	109(2)	51(1)	17(2)	33(1)	-10(2)
C(19)	45(1)	66(2)	47(1)	30(1)	8(1)	4(1)
C(20)	26(1)	27(1)	37(1)	2(1)	8(1)	-1(1)
C(21)	28(1)	32(1)	66(1)	-13(1)	15(1)	-4(1)
C(22)	32(1)	32(1)	45(1)	-1(1)	19(1)	6(1)
C(23)	26(1)	34(1)	37(1)	5(1)	17(1)	7(1)
C(24)	36(1)	42(1)	42(1)	14(1)	16(1)	10(1)
C(25)	34(1)	61(1)	42(1)	13(1)	7(1)	6(1)
C(26)	34(1)	55(1)	53(1)	7(1)	16(1)	-6(1)
C(27)	41(1)	41(1)	63(1)	18(1)	27(1)	3(1)
C(28)	28(1)	45(1)	45(1)	17(1)	15(1)	9(1)

Table 10S. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **2**:(**3**)$_2$.

	x	y	z	U(eq)
H(0A)	-3077(1)	2336(1)	-1477(1)	34
H(0B)	-3434(1)	2751(1)	-2515(1)	34
H(2A)	-1710(2)	5276(2)	-5147(2)	45
H(3A)	266(2)	6705(2)	-4529(2)	52
H(4A)	1099(2)	8211(2)	-2793(2)	47
H(6A)	-2067(2)	6895(2)	-2240(2)	36
H(7A)	-3033(2)	3814(2)	-5150(2)	44
H(7B)	-4258(2)	4439(2)	-5668(2)	44
H(8A)	-5235(2)	3605(2)	-4548(2)	48
H(8B)	-5314(2)	2689(2)	-5668(2)	48
H(9A)	-3370(4)	1743(2)	-5372(2)	75
H(9B)	-4552(4)	922(2)	-5301(2)	75
H(10A)	-2494(4)	321(2)	-4457(2)	76
H(10B)	-1803(4)	1610(2)	-3568(2)	76
H(11A)	-1416(2)	404(2)	-2222(2)	49
H(11B)	-2468(2)	-763(2)	-3155(2)	49
H(12A)	-3846(2)	-514(2)	-2117(2)	46
H(12B)	-2427(2)	-802(2)	-1393(2)	46
H(13A)	-3166(2)	78(2)	60(2)	43
H(13B)	-2526(2)	1447(2)	689(2)	43
H(14A)	-1109(2)	245(2)	1607(2)	42
H(14B)	-950(2)	-255(2)	454(2)	42
H(15A)	-235(2)	2820(2)	8(2)	47
H(16A)	1745(2)	2465(2)	-98(2)	64
H(17A)	2378(3)	3111(3)	-1395(3)	80
H(18A)	1068(3)	4165(3)	-2539(2)	91
H(19A)	-959(3)	4502(2)	-2443(2)	66
H(21A)	-1857(2)	4207(2)	-260(2)	60
H(21B)	-2266(2)	4674(2)	-1365(2)	60
H(22A)	-4343(2)	3488(2)	-834(2)	45
H(22B)	-4695(2)	3981(2)	-1920(2)	45
H(24A)	-6800(2)	3078(2)	-3551(2)	48
H(25A)	-8747(2)	1569(2)	-4565(2)	60
H(26A)	-8831(2)	-184(2)	-4059(2)	61
H(27A)	-6982(2)	-402(2)	-2483(2)	57
H(28A)	-5084(2)	1142(2)	-1409(2)	47

Table 12S. Crystal data and structure refinement for **2**:(**5**)$_2$.

Identification code	gib09
Empirical formula	$C_{60}H_{80}F_{12}N_2O_{10}P_2$
Formula weight	1279.20
Temperature	243(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 12.250(2) Å alpha = 90°
	b = 11.294(4) Å beta = 98.522(5)°
	c = 23.473(3) Å gamma = 90°
Volume, Z	3211.8(12) Å^3, 2
Density (calculated)	1.323 g/cm^3
Absorption coefficient	0.158 mm^{-1}
F(000)	1344
Crystal size	0.40 x 0.30 x 0.30 mm
Crystal color	colorless
θ range for data collection	2.01 to 22.51°
Limiting indices	$-1 < h < 13$, $-1 < k < 12$, $-25 < l < 25$
Reflections collected	5476
Independent reflections	4182 (Rint = 0.1029)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4179 / 6 / 388
Goodness-of-fit on F^2	0.989
Final R indices [I>2σ(I)]	R1 = 0.0667, wR2 = 0.1589
R indices (all data)	R1 = 0.1095, wR2 = 0.1722
Extinction coefficient	0.0111(11)
Largest diff. peak and hole	0.722 and -0.300 eÅ^{-3}

Table 13S. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for **2**:(**5**)$_2$. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
P(1)	5071(1)	-595(1)	2182(1)	56(1)
F(1)	4930(3)	-864(4)	2814(1)	114(2)
F(2)	5209(3)	-346(4)	1534(1)	112(1)
F(3)	3845(2)	-166(3)	2045(2)	104(1)
F(4)	4650(3)	-1894(3)	2009(2)	98(1)
F(5)	6292(2)	-1038(3)	2305(2)	100(1)
F(6)	5504(3)	689(3)	2337(2)	90(1)
O(1)	1908(2)	-1288(3)	-1108(1)	49(1)
O(2)	1125(3)	-266(3)	-2238(1)	63(1)
O(3)	-872(3)	947(3)	-2645(1)	61(1)
O(4)	-1278(2)	2798(3)	-1932(1)	51(1)
O(5)	-1874(2)	2590(3)	-780(1)	53(1)
N(1)	-197(3)	-1043(3)	1396(1)	40(1)
C(1)	1947(4)	-1896(4)	-150(2)	45(1)
C(2)	2486(4)	-2092(4)	395(2)	43(1)
C(3)	3595(4)	-1796(5)	538(2)	57(1)
C(4)	4124(4)	-1305(5)	126(2)	61(2)
C(5)	3605(4)	-1102(5)	-430(2)	52(1)
C(6)	2510(4)	-1412(4)	-569(2)	40(1)
C(7)	2536(4)	-1144(5)	-1570(2)	54(1)
C(8)	1797(4)	-1279(5)	-2119(2)	61(2)
C(9)	441(6)	-551(6)	-2766(2)	66(3)
C(10)	-148(6)	512(7)	-3018(3)	59(3)
C(9')	786(12)	88(13)	-2819(2)	70(5)
C(10')	13(9)	1084(13)	-2970(6)	72(5)
C(11)	-1397(5)	2000(5)	-2870(2)	71(2)
C(12)	-2015(4)	2507(5)	-2437(2)	66(2)
C(13)	-1630(4)	3729(5)	-1595(2)	59(2)
C(14)	-2449(4)	3323(5)	-1225(2)	59(2)
C(15)	978(5)	2620(6)	946(2)	64(2)
C(16)	1341(6)	3512(5)	616(3)	76(2)
C(17)	2248(6)	3341(7)	364(2)	83(2)
C(18)	2821(6)	2304(7)	446(2)	82(2)
C(19)	2474(5)	1422(5)	769(2)	68(2)
C(20)	1537(4)	1567(5)	1033(2)	48(1)
C(21)	1146(4)	588(5)	1387(2)	62(2)
C(22)	327(3)	-218(4)	1024(2)	40(1)
C(23)	-1048(4)	-1839(4)	1096(2)	42(1)
C(24)	-1623(4)	-2523(4)	1517(2)	45(1)
C(25)	-2379(4)	-3450(4)	1234(2)	39(1)
C(26)	-3448(4)	-3205(5)	990(2)	47(1)
C(27)	-4135(5)	-4062(7)	721(2)	70(2)
C(28)	-3759(6)	-5202(7)	695(2)	80(2)
C(29)	-2726(7)	-5462(5)	927(3)	82(2)
C(30)	-2026(5)	-4601(5)	1200(2)	62(2)

Table 14S. Bond lengths [Å] and angles [°] for **2**:$(5)_2$.

P(1)-F(1)	1.549(3)
P(1)-F(5)	1.563(3)
P(1)-F(3)	1.565(3)
P(1)-F(6)	1.568(3)
P(1)-F(2)	1.580(3)
P(1)-F(4)	1.588(4)
O(1)-C(6)	1.373(5)
O(1)-C(7)	1.429(5)
O(2)-C(8)	1.414(6)
O(2)-C(9')	1.4239(10)
O(2)-C(9)	1.4255(10)
O(3)-C(11)	1.416(6)
O(3)-C(10)	1.4233(10)
O(3)-C(10')	1.4241(11)
O(4)-C(12)	1.417(5)
O(4)-C(13)	1.421(5)
O(5)-C(2)#1	1.377(5)
O(5)-C(14)	1.433(5)
N(1)-C(23)	1.476(5)
N(1)-C(22)	1.486(5)
C(1)-C(2)	1.369(6)
C(1)-C(6)	1.393(6)
C(2)-O(5)#1	1.377(5)
C(2)-C(3)	1.391(6)
C(3)-C(4)	1.359(6)
C(4)-C(5)	1.383(6)
C(5)-C(6)	1.379(6)
C(7)-C(8)	1.469(6)
C(9)-C(10)	1.4787(11)
C(9')-C(10')	1.4795(11)
C(11)-C(12)	1.471(7)
C(13)-C(14)	1.494(6)
C(15)-C(20)	1.372(7)
C(15)-C(16)	1.383(8)
C(16)-C(17)	1.348(8)
C(17)-C(18)	1.364(9)
C(18)-C(19)	1.359(8)
C(19)-C(20)	1.392(7)
C(20)-C(21)	1.504(6)
C(21)-C(22)	1.519(6)
C(23)-C(24)	1.509(6)
C(24)-C(25)	1.488(6)
C(25)-C(30)	1.377(7)
C(25)-C(26)	1.377(6)
C(26)-C(27)	1.374(7)
C(27)-C(28)	1.371(9)
C(28)-C(29)	1.334(8)
C(29)-C(30)	1.388(8)

F(1)-P(1)-F(5)	90.0(2)
F(1)-P(1)-F(3)	91.0(2)
F(5)-P(1)-F(3)	178.7(2)
F(1)-P(1)-F(6)	92.3(2)
F(5)-P(1)-F(6)	88.3(2)
F(3)-P(1)-F(6)	92.5(2)
F(1)-P(1)-F(2)	178.9(2)

F(5)-P(1)-F(2)	90.0(2)
F(3)-P(1)-F(2)	89.0(2)
F(6)-P(1)-F(2)	88.8(2)
F(1)-P(1)-F(4)	89.2(2)
F(5)-P(1)-F(4)	91.0(2)
F(3)-P(1)-F(4)	88.2(2)
F(6)-P(1)-F(4)	178.4(2)
F(2)-P(1)-F(4)	89.8(2)
C(6)-O(1)-C(7)	115.8(3)
C(8)-O(2)-C(9')	119.7(6)
C(8)-O(2)-C(9)	103.8(4)
C(11)-O(3)-C(10)	110.5(4)
C(11)-O(3)-C(10')	92.8(6)
C(12)-O(4)-C(13)	115.4(4)
C(2)#1-O(5)-C(14)	117.2(3)
C(23)-N(1)-C(22)	115.9(3)
C(2)-C(1)-C(6)	120.2(4)
C(1)-C(2)-O(5)#1	116.5(4)
C(1)-C(2)-C(3)	120.3(4)
O(5)#1-C(2)-C(3)	123.2(4)
C(4)-C(3)-C(2)	118.5(5)
C(3)-C(4)-C(5)	122.6(5)
C(6)-C(5)-C(4)	118.4(5)
O(1)-C(6)-C(5)	124.3(4)
O(1)-C(6)-C(1)	115.8(4)
C(5)-C(6)-C(1)	119.9(4)
O(1)-C(7)-C(8)	108.9(4)
O(2)-C(8)-C(7)	110.7(4)
O(2)-C(9)-C(10)	110.7(6)
O(3)-C(10)-C(9)	110.2(6)
O(2)-C(9')-C(10')	121.9(10)
O(3)-C(10')-C(9')	107.5(9)
O(3)-C(11)-C(12)	108.8(4)
O(4)-C(12)-C(11)	109.8(4)
O(4)-C(13)-C(14)	112.4(4)
O(5)-C(14)-C(13)	107.5(4)
C(20)-C(15)-C(16)	121.3(5)
C(17)-C(16)-C(15)	119.7(6)
C(16)-C(17)-C(18)	120.1(6)
C(19)-C(18)-C(17)	120.7(6)
C(18)-C(19)-C(20)	120.6(6)
C(15)-C(20)-C(19)	117.6(5)
C(15)-C(20)-C(21)	121.8(5)
C(19)-C(20)-C(21)	120.6(5)
C(20)-C(21)-C(22)	111.6(4)
N(1)-C(22)-C(21)	110.7(3)
N(1)-C(23)-C(24)	111.3(3)
C(25)-C(24)-C(23)	112.8(3)
C(30)-C(25)-C(26)	116.9(5)
C(30)-C(25)-C(24)	120.7(5)
C(26)-C(25)-C(24)	122.4(4)
C(27)-C(26)-C(25)	122.0(5)
C(28)-C(27)-C(26)	119.7(6)
C(29)-C(28)-C(27)	119.5(6)
C(28)-C(29)-C(30)	121.2(6)
C(25)-C(30)-C(29)	120.7(5)

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y,-z

Table 15S. Anisotropic displacement parameters [$Å^2 \times 10^3$] for **2**:$(\mathbf{5})_2$. The anisotropic displacement factor exponent takes the form: $-2\pi 2[h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
P(1)	50(1)	65(1)	53(1)	-8(1)	7(1)	-5(1)
F(1)	103(3)	184(4)	59(2)	14(2)	27(2)	-31(3)
F(2)	118(3)	161(4)	60(2)	-6(2)	24(2)	-66(3)
F(3)	46(2)	116(3)	144(3)	27(3)	-3(2)	8(2)
F(4)	101(3)	72(2)	119(3)	-22(2)	13(2)	-21(2)
F(5)	55(2)	100(3)	145(3)	24(2)	17(2)	14(2)
F(6)	81(2)	72(2)	116(3)	-35(2)	7(2)	-9(2)
O(1)	40(2)	67(2)	40(2)	-1(2)	7(2)	3(2)
O(2)	61(2)	100(3)	28(2)	3(2)	6(2)	23(2)
O(3)	63(2)	80(3)	39(2)	5(2)	4(2)	21(2)
O(4)	49(2)	58(2)	45(2)	5(2)	9(2)	7(2)
O(5)	48(2)	70(2)	42(2)	9(2)	11(2)	9(2)
N(1)	45(2)	44(2)	32(2)	2(2)	4(2)	-5(2)
C(1)	38(3)	55(3)	43(3)	-6(3)	9(2)	2(3)
C(2)	47(3)	45(3)	36(3)	-6(2)	8(2)	9(3)
C(3)	53(3)	70(4)	43(3)	4(3)	-3(3)	-8(3)
C(4)	44(3)	79(4)	55(3)	8(3)	-9(3)	-18(3)
C(5)	44(3)	60(4)	53(3)	3(3)	7(3)	-4(3)
C(6)	43(3)	39(3)	38(3)	-5(2)	3(2)	7(2)
C(7)	50(3)	70(4)	42(3)	4(3)	12(3)	10(3)
C(8)	62(4)	76(4)	47(3)	-6(3)	15(3)	8(3)
C(11)	77(4)	90(5)	42(3)	6(3)	-6(3)	15(4)
C(12)	54(3)	73(4)	64(4)	7(3)	-14(3)	11(3)
C(13)	74(4)	51(3)	58(3)	9(3)	25(3)	4(3)
C(14)	64(3)	56(4)	60(3)	11(3)	16(3)	16(3)
C(15)	62(4)	73(4)	56(3)	-5(3)	6(3)	-8(4)
C(16)	109(5)	50(4)	66(4)	0(3)	-2(4)	-7(4)
C(17)	116(6)	83(5)	53(4)	-1(4)	26(4)	-36(5)
C(18)	95(5)	100(6)	61(4)	-3(4)	44(4)	-21(5)
C(19)	80(4)	67(4)	61(3)	-8(3)	25(3)	-11(3)
C(20)	53(3)	53(4)	37(3)	-3(3)	1(2)	-18(3)
C(21)	62(3)	76(4)	45(3)	11(3)	3(3)	-28(3)
C(22)	39(3)	47(3)	35(2)	5(2)	8(2)	-1(2)
C(23)	47(3)	47(3)	32(2)	-4(2)	0(2)	-9(3)
C(24)	53(3)	47(3)	33(2)	4(2)	5(2)	-7(3)
C(25)	48(3)	42(3)	29(2)	4(2)	9(2)	-2(3)
C(26)	54(3)	48(3)	40(3)	1(3)	10(2)	-3(3)
C(27)	67(4)	91(5)	50(3)	3(4)	5(3)	-30(4)
C(28)	107(6)	85(6)	48(4)	-11(4)	11(4)	-56(5)
C(29)	148(7)	41(4)	62(4)	-5(3)	27(4)	-14(5)
C(30)	73(4)	51(4)	61(4)	7(3)	12(3)	-2(3)

Table 16S. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **2**:$(\mathbf{5})_2$.

	x	y	z	U(eq)
H(1A)	342(3)	-1495(3)	1598(1)	48
H(1C)	-508(3)	-606(3)	1656(1)	48
H(1B)	1195(4)	-2088(4)	-242(2)	54
H(3A)	3971(4)	-1932(5)	911(2)	68
H(4A)	4871(4)	-1096(5)	223(2)	73
H(5A)	3991(4)	-760(5)	-706(2)	63
H(7A)	3123(4)	-1739(5)	-1541(2)	64
H(7B)	2878(4)	-358(5)	-1549(2)	64
H(8A)	2237(4)	-1400(5)	-2430(2)	73
H(8B)	1330(4)	-1978(5)	-2101(2)	73
H(9C)	-97(6)	-1156(6)	-2695(2)	79
H(9D)	896(6)	-877(6)	-3038(2)	79
H(10C)	388(6)	1127(7)	-3078(3)	71
H(10D)	-572(6)	312(7)	-3393(3)	71
H(9'A)	457(12)	-609(13)	-3027(2)	84
H(9'B)	1459(12)	273(13)	-2982(2)	84
H(1'A)	390(9)	1840(13)	-2877(6)	87
H(1'B)	-267(9)	1073(13)	-3384(6)	87
H(11A)	-1902(5)	1824(5)	-3224(2)	85
H(11B)	-844(5)	2567(5)	-2962(2)	85
H(12A)	-2407(4)	3220(5)	-2592(2)	79
H(12B)	-2562(4)	1934(5)	-2343(2)	79
H(13A)	-1961(4)	4359(5)	-1851(2)	71
H(13B)	-986(4)	4062(5)	-1350(2)	71
H(14A)	-2777(4)	4005(5)	-1056(2)	71
H(14B)	-3041(4)	2872(5)	-1455(2)	71
H(15A)	335(5)	2737(6)	1113(2)	77
H(16A)	957(6)	4235(5)	569(3)	92
H(17A)	2486(6)	3937(7)	132(2)	100
H(18A)	3462(6)	2198(7)	276(2)	99
H(19A)	2870(5)	707(5)	816(2)	82
H(21A)	797(4)	932(5)	1699(2)	74
H(21B)	1782(4)	120(5)	1563(2)	74
H(22A)	-243(3)	262(4)	795(2)	48
H(22B)	709(3)	-674(4)	758(2)	48
H(23A)	-704(4)	-2395(4)	856(2)	51
H(23B)	-1593(4)	-1372(4)	842(2)	51
H(24A)	-1066(4)	-2898(4)	1803(2)	54
H(24B)	-2046(4)	-1970(4)	1721(2)	54
H(26A)	-3714(4)	-2427(5)	1007(2)	56
H(27A)	-4860(5)	-3869(7)	556(2)	84
H(28A)	-4226(6)	-5795(7)	514(2)	96
H(29A)	-2468(7)	-6242(5)	906(3)	99
H(30A)	-1303(5)	-4807(5)	1363(2)	74