SYNLETT

Accounts and
Rapid Communications in
Synthetic Organic Chemistry

SUPPORTING INFORMATION

Supporting Information

for DOI: 10.1055/s-0034-1378348

Thieme

Polyfluorinated cyclopentadienones as Lewis acids.

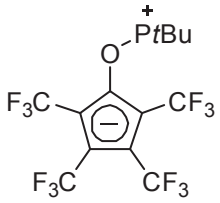
Blanca Inés, Sigrid Holle and Manuel Alcarazo.*

Max-Planck-Institut für Kohlenforschung, Kaiser Wilhelm Platz 1, 45470-Mülheim an der Ruhr, Germany.
Fax: +49 208 306 2428.
E-mail: alcarazo@mpi-muelheim.mpg.de

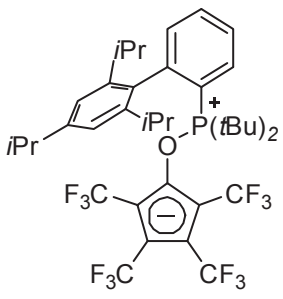
Supporting Information

Table of contents

General procedures: All reactions were carried out in flame-dried glassware under Ar. All the solvents were purified by distillation over the drying agents indicated and were transferred under Ar. CH_2Cl_2 (CaH_2), hexane, toluene (Na/K). Flash chromatography: Merck silica gel 60 (230-400 mesh). IR: Nicolet FT-7199 spectrometer, wavenumbers in cm^{-1}. MS (EI): Finnigan MAT 8200 (70 eV), ESI-MS: Finnigan MAT 95, accurate mass determinations: Bruker APEX III FT-MS (7 T magnet). NMR: Spectra were recorded on a Bruker DPX 300 or AV 400 spectrometer in the solvents indicated; ^1H and ^{13}C chemical shifts (δ) are given in ppm relative to TMS, coupling constants (J) in Hz. The solvent signals were used as references and the chemical shifts converted to the TMS scale. All commercially available compounds (Acros, Fluka, Lancaster, Alfa Aesar, Aldrich) were used as received unless stated otherwise.

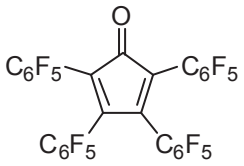


Compound 6. Tris-terbutylphosphine (304 µL, 1.0 M solution in toluene, 0.304 mmol) was added at -78 °C to a solution of ketone **1** (106.8 mg, 0.303 mmol) in toluene (3 mL) and the resulting slurry allowed to warm to r.t. while it was stirred. The solvent was then removed under vacuum and the crude product washed with MeOH to afford **6** as a white solid (73 mg, 43%). **^{1}H NMR** (400 MHz, CD_2Cl_2) δ = 1.69 (d, J = 15.0 Hz, 27H) ppm; **^{13}C NMR** (151 MHz, CD_2Cl_2) δ = 29.5, 42.9 (d, J = 31.3 Hz), 99.9 (d, J = 39.3 Hz), 107.5, 123.9 (q, J = 269.0 Hz), 124.0 (d, J = 267.9 Hz), 135.8 (d, J = 20.4 Hz); **^{31}P NMR** (162 MHz, CD_2Cl_2) δ = 113.9 ppm; **^{19}F NMR** (282 MHz, $CDCl_3$) δ = -46.85, -51.77 ppm; **IR** (neat) ν = 665, 802, 922, 1010, 1084, 1105, 1197, 1269, 1401, 1483, 1738, 2901, 2971, 2988, 3675 cm^{-1}; **HRMS** *calcd.* for $C_{21}H_{27}OF_{12}PNa$: 577.150013; *found*: 577.149952.

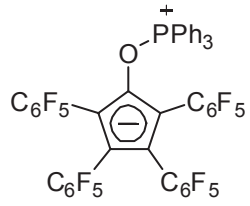


Compound 7. *t*BuXPhos (232 mg, 0.546 mmol) was added in one portion at -78°C to a solution of ketone **1** (192 mg, 0.546 mmol) in CH_2Cl_2 (5mL) and the resulting slurry allowed to warm to r.t. while it was stirred. The solvent was then removed under vacuum and the crude product washed with MeOH to afford **7** as a white solid (321 mg, 76%). **^{1}H NMR** (400 MHz, CD_2Cl_2) δ = 0.96 (br s, 6H), 1.22 (d, J = 6.6 Hz, 6H), 1.28 (d, J = 6.9 Hz, 6H), 1.32 (br s, 18H),

2.52 (br s, 2H), 2.95 (sep, *J* = 6.9 Hz, 1H), 7.12 (s, 2H), 7.52 (dd, *J* = 6.6, 1.5 Hz, 1H), 7.61 (dt, *J* = 7.9, 1.7 Hz, 1H), 7.73 (dt, *J* = 7.5, 1.6 Hz, 1H), 8.09 (dd, *J* = 11.3, 1.1 Hz, 1H) ppm; **^{13}C NMR** (101 MHz, CD_2Cl_2) δ = 21.8, 24.1, 26.8, 28.2, 31.2, 34.7, 41.9, 99.0 (9, *J* = 35.8 Hz), 106.2 (br), 121.7, 123.7 (q, *J* = 266.5 Hz), 124.0 (q, *J* = 268.6 Hz), 125.0 (d, *J* = 65.3 Hz), 127.0 (d, *J* = 9.8 Hz), 132.2 (d, *J* = 19.2 Hz), 132.9 (d, *J* = 3.0 Hz), 136.4, 136.5, 136.8 (d, *J* = 4.4 Hz), 143.4 (d, *J* = 10.5 Hz), 148.3, 151.6 ppm; **^{31}P NMR** (162 MHz, CD_2Cl_2) δ = 101.0 ppm; **^{19}F NMR** (282 MHz, $CDCl_3$) δ = -51.6, -49.2 ppm; **IR** (neat) ν = 666, 772, 938, 1038, 1118, 1200, 1284, 1409, 1463, 1503, 2968; **HRMS** *calcd.* for $C_{38}H_{45}OF_{12}PNa$: 799.290869; *found*: 799.290480.

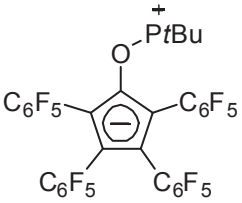


Compound 9. To a young-key equiped flask charged with $Co_2(CO)_8$ (496.5 mg, 1.451 mmol) and 1,2-bis(pentafluorophenyl)acetylene (500 mg, 1.396 mmol) was added degassed *o*-xylene (3.9 mL). The flask was then degassed and the deep red suspension was stirred at room temperature overnight and then heated to 160 °C until the formation of the product was observed by TLC. The reaction crude was then filtered through a plug of neutral Al_2O_3, the solvent was removed under vacuum and the crude product purified by flash cromatography (Al_2O_3: *n*-pentane) to afford ketone **1** as red solid (475.4 mg, 92%). **^{13}C NMR** (151 MHz, CD_2Cl_2) δ = 104.1 (td, *J* = 18.2, 3.3 Hz), 106.4 (t, *J* = 17.3 Hz), 122.5, 138.2 (dm, *J* = 255.7 Hz), 138.3 (dm, *J* = 253.2 Hz), 142.8 (dm, *J* = 257.9 Hz), 143.2 (dm, *J* = 259.3 Hz), 143.6 (dm, *J* = 249.3 Hz), 144.9 (dm, *J* = 254.9 Hz), 146.3, 189.3 ppm; **^{19}F NMR** (282 MHz, $CDCl_3$) δ = -(159-69-159.54) (m, 4F), -(158.10-157.95) (m, 4F), -149.07 (t, *J* = 21.0 Hz, 2F), -146.89 (t, *J* = 21.0 Hz, 2F), -137.27 (d, *J* = 19.0 Hz, 2F), -136.99 (d, *J* = 15.2 Hz, 2F) ppm; **IR** (neat) ν = 931, 994, 1099, 1341, 1442, 1497, 1523, 1653, 1733 cm^{-1}; **HRMS** *calcd.* for $C_{29}O_1F_{20}$: 743.962986; *found*: 743.962315.

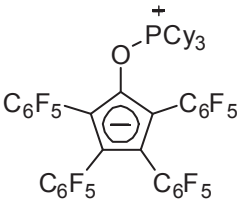


Compound 11. Triphenylphosphine (7 mg, 0.027 mmol) was added in one portion at room temperature to a solution of ketone **9** (20 mg, 0.027 mmol) in toluene (1 mL) and the resulting slurry was stirred at the same temperature overnight. The solvent was then removed under vacuum and the crude product washed with *n*-pentane (3 x 1 mL) to afford **3** as yellow solid (26.5 mg, 98%). **^{1}H NMR** (400 MHz, CD_2Cl_2) parcial δ

= 7.34-7.39 (m, 6H), 7.51-7.55 (m, 6H), 7.79 (t, *J* = 7.1 Hz, 3H) ppm; **^{13}C NMR** (101 MHz, CD_2Cl_2) δ = 83.2, 96.6, 103.7, 112.4, 114.0, 120.5, 129.9 (d, *J* = 12.9 Hz), 131.2 (dm, *J* = 242.2 Hz), 134.2 (d, *J* = 11.4 Hz), 135.5 (dm, *J* = 243.2 Hz), 136.2 (d, *J* = 2.9 Hz), 137.5 (dm, *J* = 241.3 Hz), 144.5 (dm, *J* = 243.2 Hz), 145.4 (dm, *J* = 245.6 Hz) ppm; **^{31}P NMR** (162 MHz, CD_2Cl_2) δ = 62.3 ppm; **^{19}F NMR** (282 MHz, $CDCl_3$) δ = -165.18 (t, *J* = 18.9 Hz, 4F), -(164.61-164.47) (m, 4F), -160.25 (t, *J* = 21.0 Hz, 2F), -159.37 (t, *J* = 20.9 Hz, 2F), -140.50 (d, *J* = 13.6 Hz, 4F), -139.54 (d, *J* = 24.6 Hz, 4F) ppm; **IR** (neat) ν = 657, 686, 733, 748, 781, 790, 875, 922, 949, 984, 1050, 1088, 1104, 1122, 1166, 1355, 1399, 1441, 1469, 1494, 1520, 1535, 1592 cm^{-1}; **HRMS** *calcd.* for $C_{47}H_{16}O_1F_{20}P_1$: 1007.061403; *found*: 1007.061418.

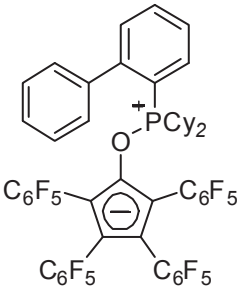


Compound 12. Tris-terbutylphosphine (81 µL, 1.0 M solution in toluene, 0.081 mmol) was added at room temperature to a solution of ketone **9** (60 mg, 0.081 mmol) in toluene (3 mL) and the resulting slurry was stirred at the same temperature overnight. The solvent was then removed under vacuum and the crude product washed with *n*-pentane (3 x 2 mL) to afford **2** as pale yellow solid (65.9 mg, 86%). **^{1}H NMR** (400 MHz, CD_2Cl_2) δ = 1.40 (d, *J* = 14.7 Hz, 27H) ppm; **^{13}C NMR** (151 MHz, CD_2Cl_2) δ = 29.2, 42.1 (d, *J* = 33.9 Hz), 77.9, 96.1, 104.2, 113.6 (m), 114.0 (m), 137.2 (dm, *J* = 250.4 Hz), 137.9 (dm, *J* = 251.8 Hz), 139.6 (dm, *J* = 249.0 Hz), 139.9 (dm, *J* = 249.0 Hz), 144.7 (dm, *J* = 246.2 Hz), 145.5 (dm, *J* = 243.4 Hz) ppm; **^{31}P NMR** (162 MHz, CD_2Cl_2) δ = 106.7 ppm; **^{19}F NMR** (282 MHz, $CDCl_3$) δ = -(165.21-165.02) (m, 4F), -(164.00-163.80) (m, 4F), -(158.86-158.52) (m, 4F), -(140.25-140.14) (m, 4F), -(139.00-138.85) (m, 4F) ppm; **IR** (neat) ν = 742, 856, 926, 991, 1053, 1093, 1104, 1347, 1402, 1494, 1504, 1523, 1978 cm^{-1}; **HRMS** *calcd.* for $C_{41}H_{27}O_1F_{20}P_1Na$: 969.137244; *found*: 969.137923.



Compound 13. Tricyclohexylphosphine (15 mg, 0.053 mmol) was added in one portion to a solution of ketone **9** (40 mg, 0.053 mmol) in toluene (1.5 mL) at room temperature and the resulting mixture was stirred at the same temperature overnight. The solvent was then removed under vacuum and the crude product washed with *n*-pentane (3 x 1 mL) to afford **5** as brown-yellow solid (48.7 mg, 89%). **^{1}H NMR** (400 MHz, CD_2Cl_2) δ = 1.00-1.46 (m, 16H), 1.73-2.07 (m, 17H) ppm; **^{13}C NMR** (101 MHz, CD_2Cl_2) parcial δ

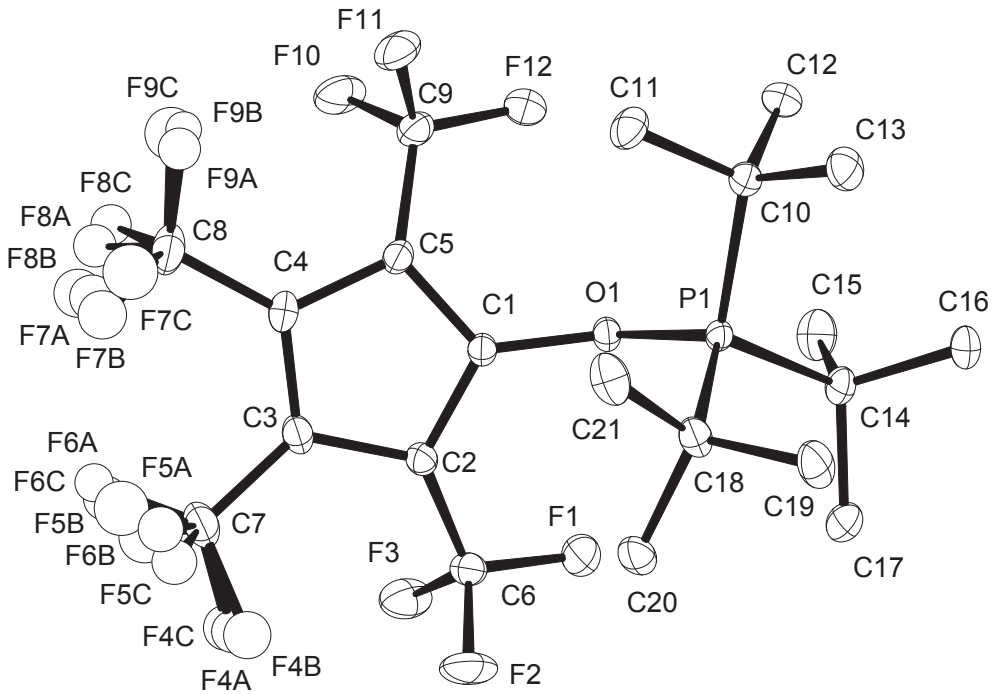
= 25.6 (d, *J* = 1.4 Hz), 26.2 (d, *J* = 3.3 Hz), 26.8 (d, *J* = 12.9 Hz), 36.2 (d, *J* = 47.7 Hz), 96.0, 103.6, 112.8, 113.8, 137.4 (dm, *J* = 248.4 Hz), 138.1 (dm, *J* = 252.2 Hz), 139.5 (dm, *J* = 252.2 Hz), 139.9 (dm, *J* = 243.2 Hz), 144.6 (dm, *J* = 246.5 Hz), 145.5 (dm, *J* = 243.7 Hz) ppm; **^{31}P NMR** (162 MHz, CD_2Cl_2) δ = 89.9 ppm; **^{19}F NMR** (282 MHz, $CDCl_3$) δ = -(165.15-165.02) (m, 4F), -(163.61-163.42) (m, 4F), -159.16 (m, 4F), -(140.43-140.24) (m, 4F), -139.71 (dt, *J* = 25.3, 8.8 Hz, 4F) ppm; **IR** (neat) ν = 731, 872, 922, 998, 1056, 1092, 1103, 1356, 1450, 1472, 1492, 1504, 1520, 2491 cm^{-1}; **HRMS** *calcd.* for $C_{47}H_{34}O_1F_{20}P_1$: 1025.202248; *found*: 1025.202439.



Compound 14. (2-Biphenyl)dicyclohexylphosphine (24 mg, 0.067 mmol) was added in one portion at room temperature to a solution of ketone **9** (50 mg, 0.067 mmol) in toluene (2 mL) and the resulting mixture was stirred at the same temperature overnight. The solvent was then removed under vacuum and the crude product washed with *n*-pentane (3 x 2 mL) to afford **4** as pale yellow solid (68.3 mg, 93%). **^{1}H NMR** (400 MHz, CD_2Cl_2) δ = 0.83-1.00 (m, 4H), 1.07-1.27 (m, 6H), 1.38-1.72 (m, 10H), 2.01-2.12 (m, 2H), 7.26-7.29 (m, 1H), 7.32-7.38 (m, 3H), 7.44-7.48 (m, 1H), 7.55-7.57 (m, 3H), 7.70-7.74 (m, 1H) ppm; **^{13}C NMR** (101 MHz, CD_2Cl_2) parcial δ = 25.7 (d, *J* = 1.4 Hz), 26.3 (d, *J* = 3.8 Hz), 26.9 (d, *J* = 13.3 Hz), 37.2 (d, *J* = 51.9 Hz), 83.2, 95.5, 103.5, 113.0, 114.0, 127.7 (d, *J* = 11.4 Hz), 129.2, 129.7, 130.3, 131.9 (d, *J* = 10.0 Hz), 134.6 (d, *J* = 14.3 Hz), 134.7, 137.5 (dm, *J* = 242.7 Hz), 137.9 (dm, *J* = 247.0 Hz), 139.5 (d, *J* = 2.4 Hz), 144.7 (dm, *J* = 242.7 Hz), 145.5 (dm, *J* = 240.3 Hz), 148.5 (d, *J* = 8.6 Hz), ppm; **^{31}P NMR** (162 MHz, CD_2Cl_2) δ = 80.5 ppm; **^{19}F NMR** (282 MHz, $CDCl_3$) δ = -(165.21-165.09) (m, 4F), -(163.99-163.84) (m, 4F), -159.68 (t, *J* = 21.1 Hz, 2F), -159.36 (t, *J* = 21.0 Hz, 2F), -(140.52-140.34) (m, 4F), -139.77 (dt, *J* = 25.0, 8.7 Hz, 4F) ppm; **IR** (neat) ν = 696, 704, 731, 753, 768, 790, 852, 864, 894, 924, 969, 991, 1060, 1094, 1106, 1287, 1358, 1403, 1475, 1490, 1501, 1522, 1535, 2862, 2933; **HRMS** *calcd.* for $C_{53}H_{31}O_1F_{20}P_1Na$: 1117.168543; *found*: 1117.169092.

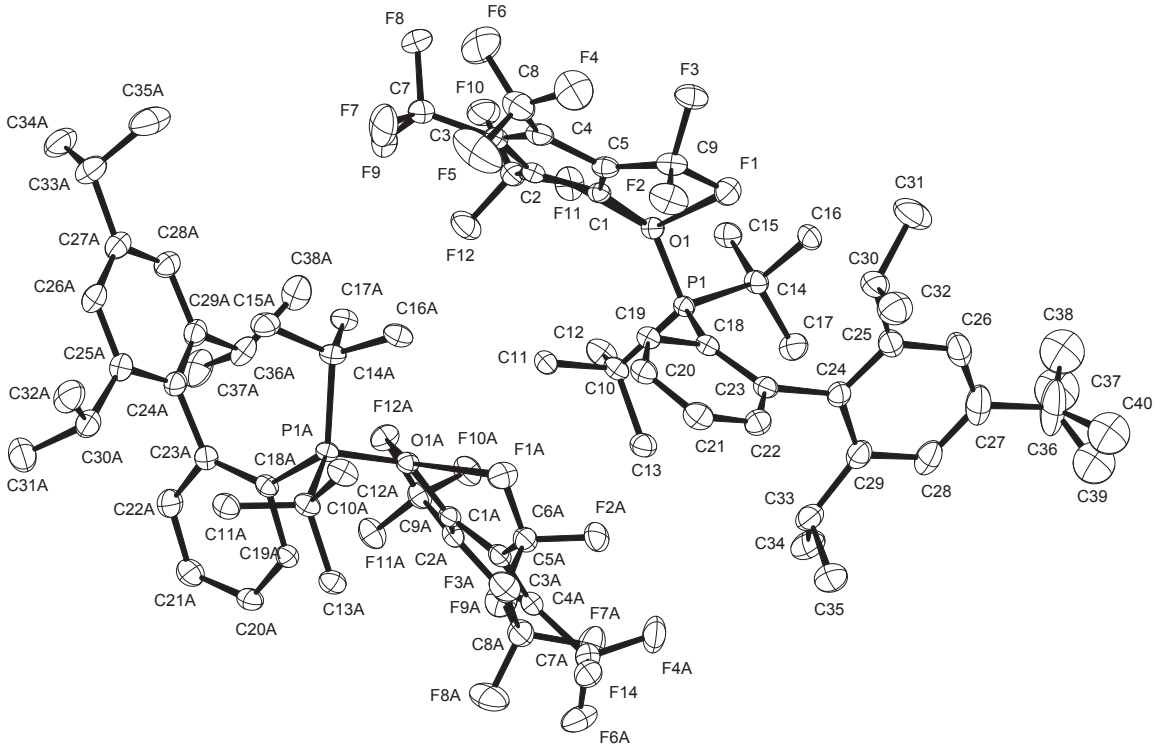
X-ray structures

Compound 6:



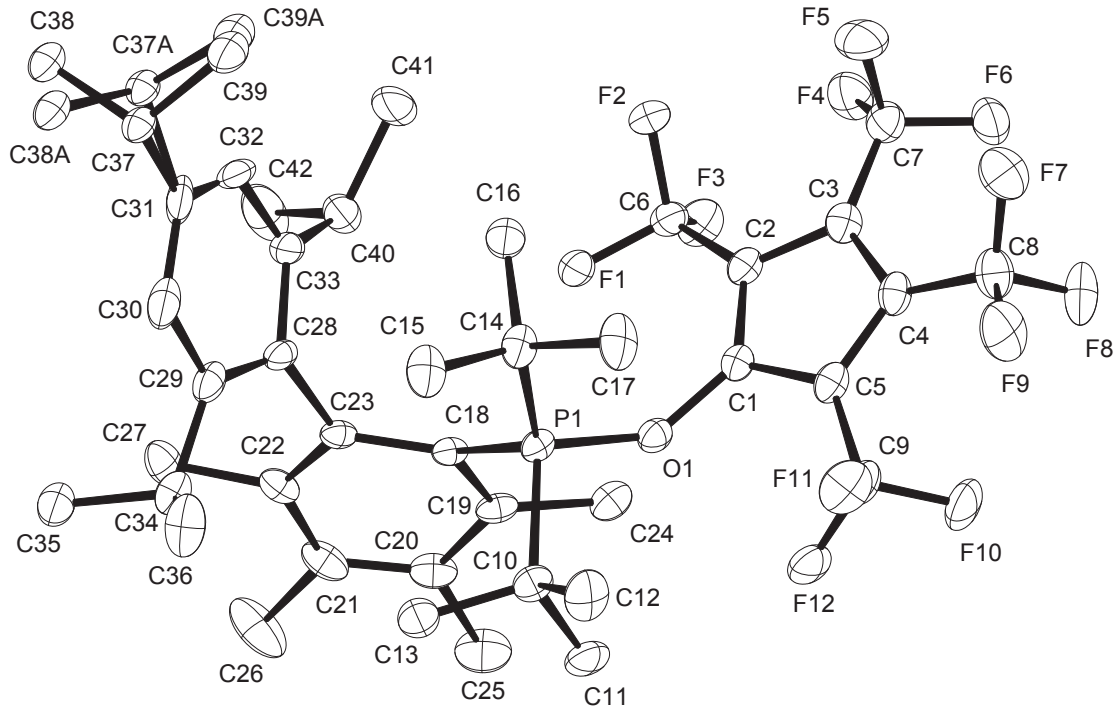
Empirical formula	$C_{21}H_{27}F_{12}OP$	
Color	colorless	
Formula weight	554.40 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	MONOCLINIC	
Space group	**$P2_1/n$, (no. 14)**	
Unit cell dimensions	a = 12.6772(15) Å	α= 90°.
	b = 10.2913(12) Å	β= 106.058(2)°.
	c = 18.812(2) Å	γ = 90°.
Volume	2358.6(5) Å^3	
Z	4	
Density (calculated)	1.561 Mg · m^{-3}	
Absorption coefficient	0.224 mm^{-1}	
F(000)	1136 e	
Crystal size	0.50 x 0.44 x 0.26 mm^3	
θ range for data collection	1.74 to 33.80°.	
Index ranges	-19 ≤ h ≤ 19, -15≤ k ≤ 15, -29 ≤ l ≤ 29	
Reflections collected	75377	
Independent reflections	9402 [R_{int} = 0.0509]	
Reflections with I>2σ(I)	8290	
Completeness to θ = 27.50°	100.0 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.95 and 0.91	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9402 / 0 / 343	
Goodness-of-fit on F^2	1.042	
Final R indices [I>2σ(I)]	R_1 = 0.0539	wR^2 = 0.1415
R indices (all data)	R_1 = 0.0608	wR^2 = 0.1482
Largest diff. peak and hole	1.052 and -1.099 e · Å^{-3}	

Compound 7:



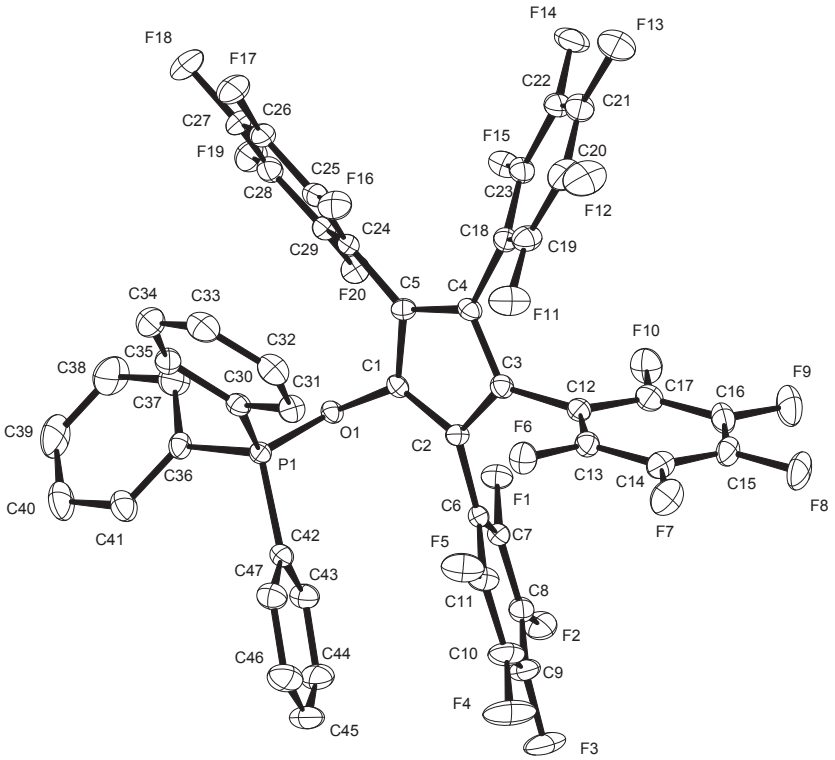
Empirical formula	$C_{76}H_{89}F_{24}O_2P_2$	
Color	yellow	
Formula weight	1552.41 g · mol^{-1}	
Temperature	100 K	
Wavelength	1.54178 Å	
Crystal system	MONOCLINIC	
Space group	**$P2_1/c$, (no. 14)**	
Unit cell dimensions	a = 19.2158(10) Å	α= 90°.
	b = 12.5485(6) Å	β= 102.368(2)°.
	c = 32.3281(16) Å	γ = 90°.
Volume	7614.3(7) Å^3	
Z	4	
Density (calculated)	1.354 Mg · m^{-3}	
Absorption coefficient	1.429 mm^{-1}	
F(000)	3228 e	
Crystal size	0.25 x 0.25 x 0.24 mm^3	
θ range for data collection	2.35 to 67.32°.	
Index ranges	-22 ≤ h ≤ 20, -14≤ k ≤ 15, -38 ≤ l ≤ 38	
Reflections collected	170666	
Independent reflections	13525 [R_{int} = 0.0550]	
Reflections with I>2σ(I)	12380	
Completeness to θ = 67.32°	99.0 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.79 and 0.69	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	13525 / 0 / 960	
Goodness-of-fit on F^2	1.017	
Final R indices [I>2σ(I)]	R_1 = 0.0418	wR^2 = 0.1084
R indices (all data)	R_1 = 0.0451	wR^2 = 0.1115
Largest diff. peak and hole	1.102 and -0.557 e · Å^{-3}	

Compound 8:



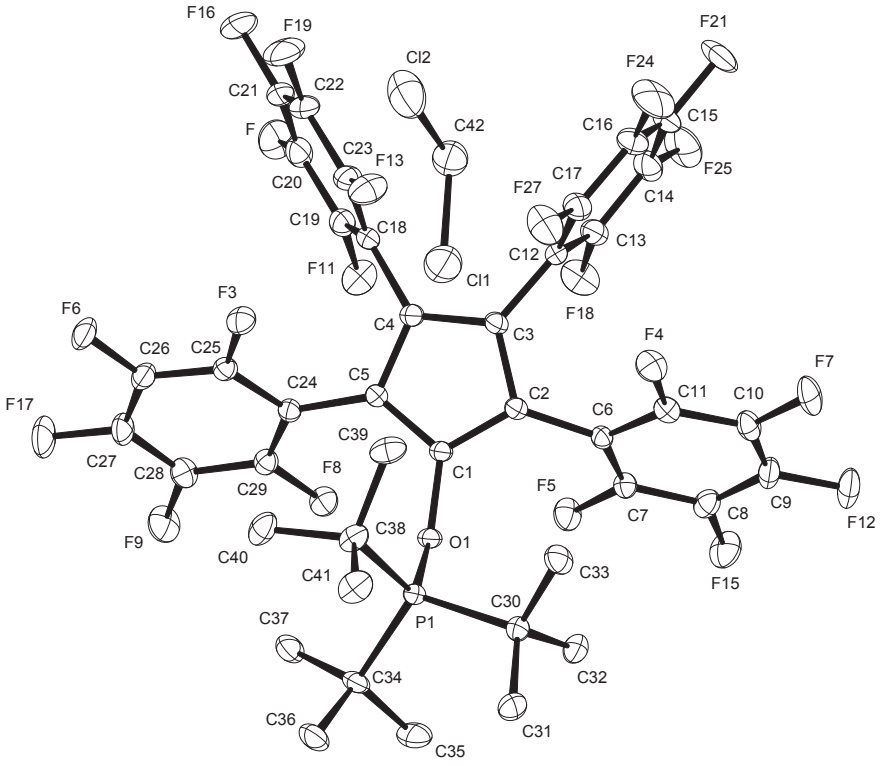
Empirical formula	$C_{42}H_{54}F_{12}OP$	
Color	colourless	
Formula weight	833.82 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	MONOCLINIC	
Space group	**$P2_1/c$, (no. 14)**	
Unit cell dimensions	a = 11.1478(13) Å	α= 90°.
	b = 20.897(3) Å	β= 101.691(16)°.
	c = 17.374(4) Å	γ = 90°.
Volume	3963.4(12) Å^3	
Z	4	
Density (calculated)	1.397 Mg · m^{-3}	
Absorption coefficient	0.159 mm^{-1}	
F(000)	1748 e	
Crystal size	0.35 x 0.24 x 0.16 mm^3	
θ range for data collection	2.60 to 27.50°.	
Index ranges	-14 ≤ h ≤ 14, -27≤ k ≤ 27, -22 ≤ l ≤ 22	
Reflections collected	76323	
Independent reflections	9107 [R_{int} = 0.0843]	
Reflections with I>2σ(I)	6916	
Completeness to θ = 27.50°	99.9 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.98 and 0.95	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9107 / 0 / 533	
Goodness-of-fit on F^2	1.059	
Final R indices [I>2σ(I)]	R_1 = 0.0574	wR^2 = 0.1300
R indices (all data)	R_1 = 0.0817	wR^2 = 0.1491
Largest diff. peak and hole	0.726 and -0.813 e · Å^{-3}	

Compound 11:



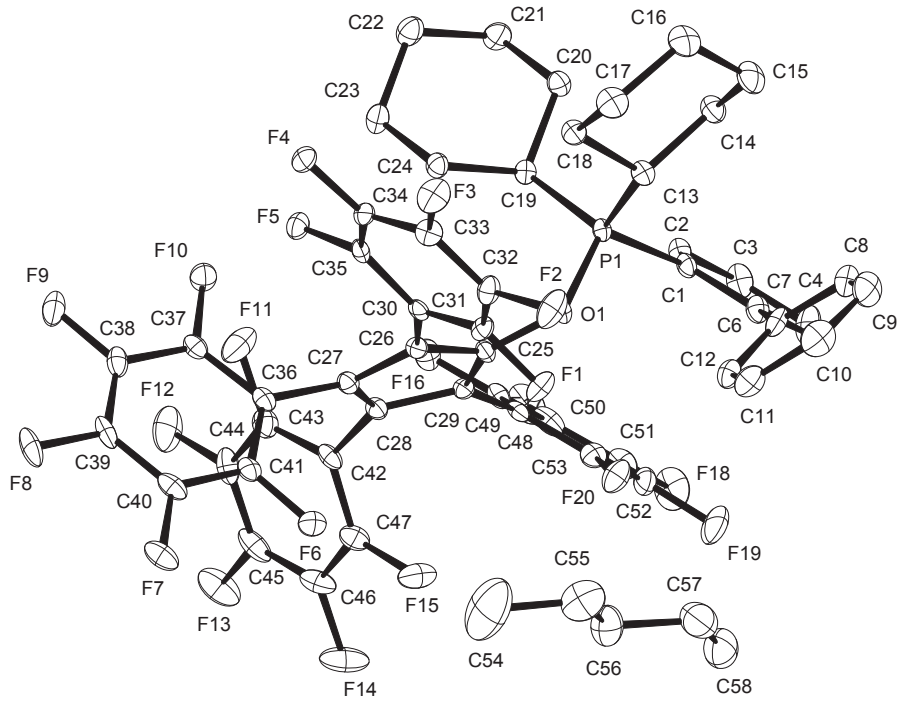
Empirical formula	$C_{47}H_{15}F_{20}OP$	
Color	yellow	
Formula weight	1006.56 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	ORTHORHOMBIC	
Space group	**Pbca, (no. 61)**	
Unit cell dimensions	a = 20.950(2) Å	α= 90°.
	b = 17.1156(16) Å	β= 90°.
	c = 22.707(3) Å	γ = 90°.
Volume	8142.2(16) Å^3	
Z	8	
Density (calculated)	1.642 Mg · m^{-3}	
Absorption coefficient	0.199 mm^{-1}	
F(000)	4000 e	
Crystal size	0.35 x 0.2 x 0.06 mm^3	
θ range for data collection	2.65 to 29.98°.	
Index ranges	-28 ≤ h ≤ 29, -24≤ k ≤ 23, -31 ≤ l ≤ 31	
Reflections collected	139035	
Independent reflections	11830 [R_{int} = 0.0742]	
Reflections with I>2σ(I)	8468	
Completeness to θ = 27.50°	99.9 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.99 and 0.95	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11830 / 0 / 622	
Goodness-of-fit on F^2	1.084	
Final R indices [I>2σ(I)]	R_1 = 0.0487	wR^2 = 0.0943
R indices (all data)	R_1 = 0.0814	wR^2 = 0.1086
Largest diff. peak and hole	0.379 and -0.367 e · Å^{-3}	

Compound 12:



Empirical formula	$C_{42}H_{29}Cl_2F_{20}OP$	
Color	yellow	
Formula weight	1031.52 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	MONOCLINIC	
Space group	**$P2_1/c$, (no. 14)**	
Unit cell dimensions	a = 12.4613(7) Å	α= 90°.
	b = 21.0904(19) Å	β= 91.457(4)°.
	c = 16.0828(9) Å	γ = 90°.
Volume	4225.4(5) Å^3	
Z	4	
Density (calculated)	1.622 Mg · m^{-3}	
Absorption coefficient	0.315 mm^{-1}	
F(000)	2072 e	
Crystal size	0.26 x 0.18 x 0.05 mm^3	
θ range for data collection	2.71 to 30.07°.	
Index ranges	-17 ≤ h ≤ 17, -29≤ k ≤ 29, -22 ≤ l ≤ 22	
Reflections collected	102917	
Independent reflections	12396 [R_{int} = 0.0635]	
Reflections with I>2σ(I)	8928	
Completeness to θ = 27.50°	99.9 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.99 and 0.95	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12396 / 0 / 599	
Goodness-of-fit on F^2	1.072	
Final R indices [I>2σ(I)]	R_1 = 0.0552	wR^2 = 0.1254
R indices (all data)	R_1 = 0.0867	wR^2 = 0.1419
Largest diff. peak and hole	1.056 and -1.265 e · Å^{-3}	

Compound 14:



Empirical formula	$C_{58}H_{43}F_{20}OP$	
Color	yellow	
Formula weight	1166.89 g · mol^{-1}	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	MONOCLINIC	
Space group	**$P2_1/n$, (no. 14)**	
Unit cell dimensions	a = 14.423(2) Å	α= 90°.
	b = 19.032(3) Å	β= 97.003(11)°.
	c = 18.747(2) Å	γ = 90°.
Volume	5107.8(12) Å^3	
Z	4	
Density (calculated)	1.517 Mg · m^{-3}	
Absorption coefficient	0.170 mm^{-1}	
F(000)	2376 e	
Crystal size	0.21 x 0.16 x 0.10 mm^3	
θ range for data collection	2.68 to 27.50°.	
Index ranges	-18 ≤ h ≤ 18, -24≤ k ≤ 23, -24 ≤ l ≤ 24	
Reflections collected	45838	
Independent reflections	11709 [R_{int} = 0.0535]	
Reflections with I>2σ(I)	8163	
Completeness to θ = 27.50°	99.8 %	
Absorption correction	Gaussian	
Max. and min. transmission	0.99 and 0.96	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11709 / 0 / 723	
Goodness-of-fit on F^2	1.087	
Final R indices [I>2σ(I)]	R_1 = 0.0507	wR^2 = 0.0945
R indices (all data)	R_1 = 0.0872	wR^2 = 0.1102
Largest diff. peak and hole	0.820 and -0.351 e · Å^{-3}	