Síntesi estereoselectiva de fosfines amb quiralitat al fòsfor. Aplicacions en catàlisis

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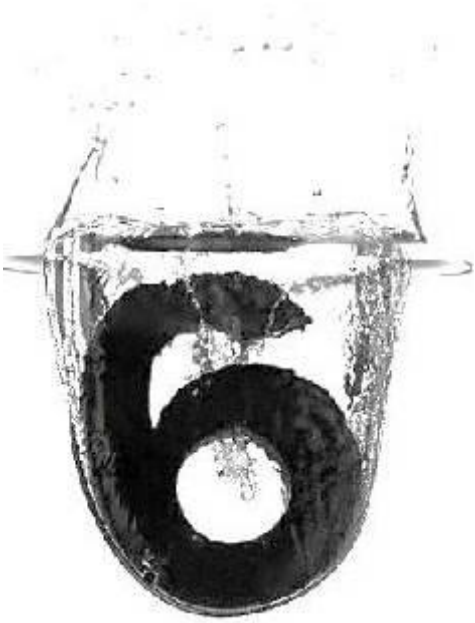
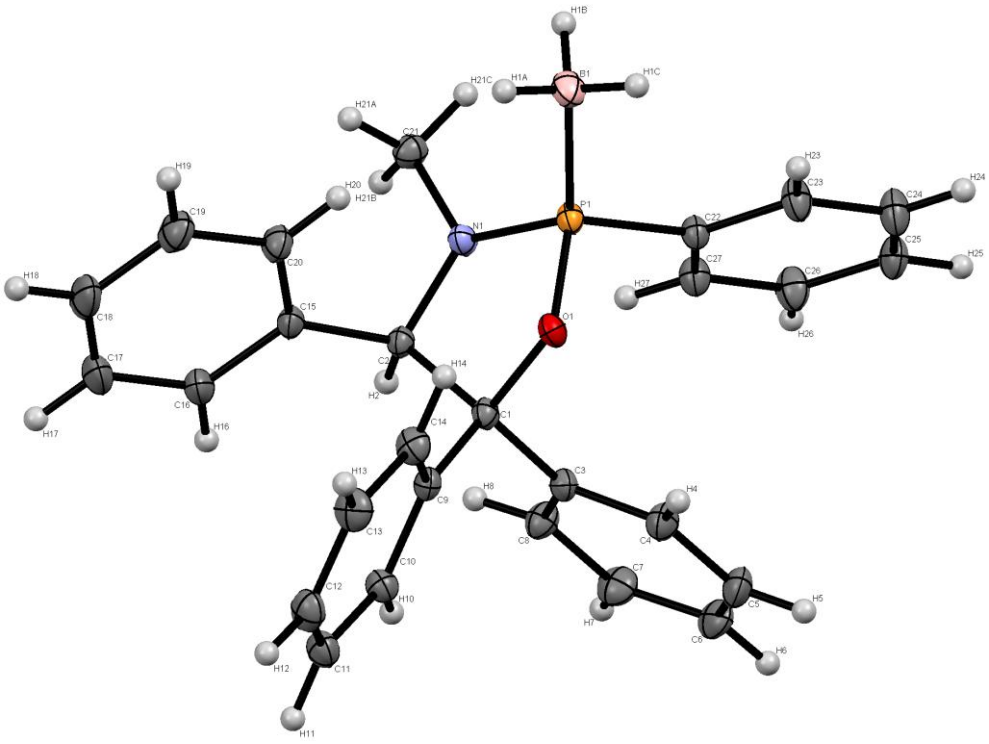
Dades de difracció de raigs-X



... Imagination is more important than knowledge ...

Albert Einstein (1879-1955)

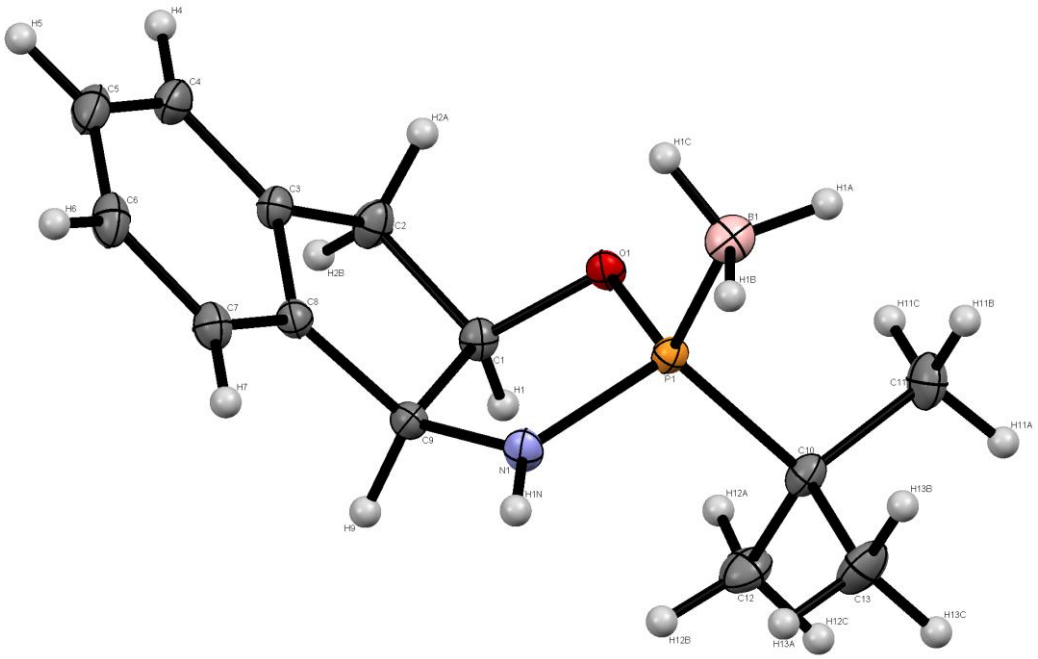
Dades del cristall i refinament de l'estructura de 80



6 Dades de difracció de raigs-X

Empirical formula	C27 H27 B N O P	
Formula weight	423.28	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 6.7240(4) Å	α= 90.00 °.
	b = 14.2225(9) Å	β = 94.213(3) °.
	c = 12.2470(9) Å	γ = 90.00 °.
Volume	1168.04(13) $Å^3$	
Z	2	
Density (calculated)	1.203 Mg/m^3	
Absorption coefficient	0.136 mm^{-1}	
F(000)	448	
Crystal size	0.40 x 0.40 x 0.40 mm^3	
Theta range for data collection	1.67 to 1.67°.	
Index ranges	-10 <=h<=10 ,0 <=k<=21 ,0 <=l<=18	
Reflections collected	8687	
Independent reflections	8159 [R(int) = 0.0000]	
Completeness to theta =33.27°	0.904 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9475 and 0.9474	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8687 / 1 / 283	
Goodness-of-fit on F^2	1.087	
Final R indices [I>2sigma(I)]	R1 = 0.0528 , wR2 = 0.1376	
R indices (all data)	R1 = 0.0561 , wR2 = 0.1419	
Largest diff. peak and hole	0.487 and -0.539 $e.Å^{-3}$	

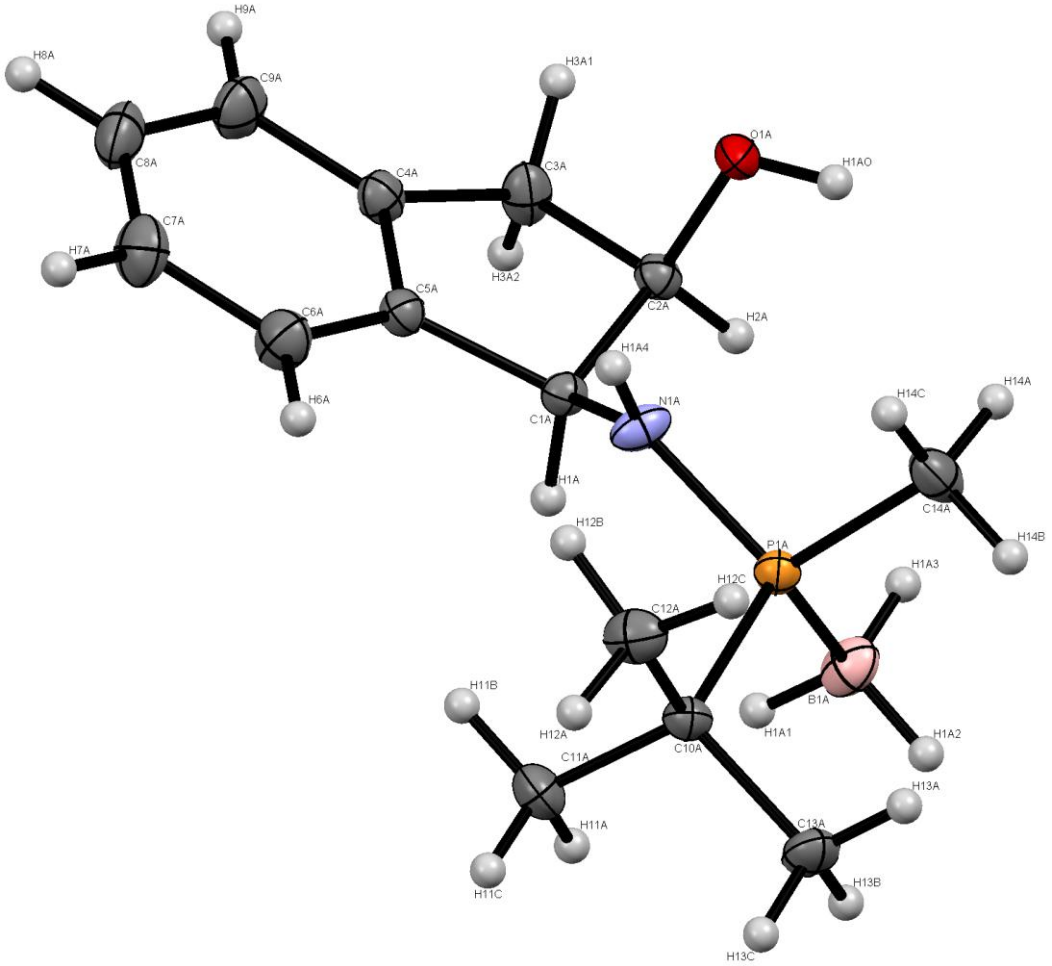
Dades del cristall i refinament de l'estructura de 83



6 Dades de difracció de raigs-X

Empirical formula	C13 H21 B N O P	
Formula weight	249.09	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 8.6793(5) Å	α= 90.00 °.
	b = 10.6231(6) Å	β = 90.00 °.
	c = 15.0930(9) Å	γ = 90.00 °.
Volume	1391.59(14) $Å^3$	
Z	4	
Density (calculated)	1.189 Mg/m^3	
Absorption coefficient	0.181 mm^{-1}	
F(000)	536	
Crystal size	0.10 x 0.05 x 0.03 mm^3	
Theta range for data collection	2.34 to 36.50 °.	
Index ranges	-14 <=h<=13 ,-17 <=k<=17 ,-24 <=l<=23	
Reflections collected	6628	
Independent reflections	6143 [R(int) = 0.0447]	
Completeness to theta =36.50°	0.984 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9946 and 0.9821	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6628 / 0 / 158	
Goodness-of-fit on F^2	1.073	
Final R indices [I>2sigma(I)]	R1 = 0.0332 , wR2 = 0.0892	
R indices (all data)	R1 = 0.0376 , wR2 = 0.0918	
Absolute Structure Flack parameter	x =-0.03(5)	
Largest diff. peak and hole	0.523 and -0.437 $e.Å^{-3}$	

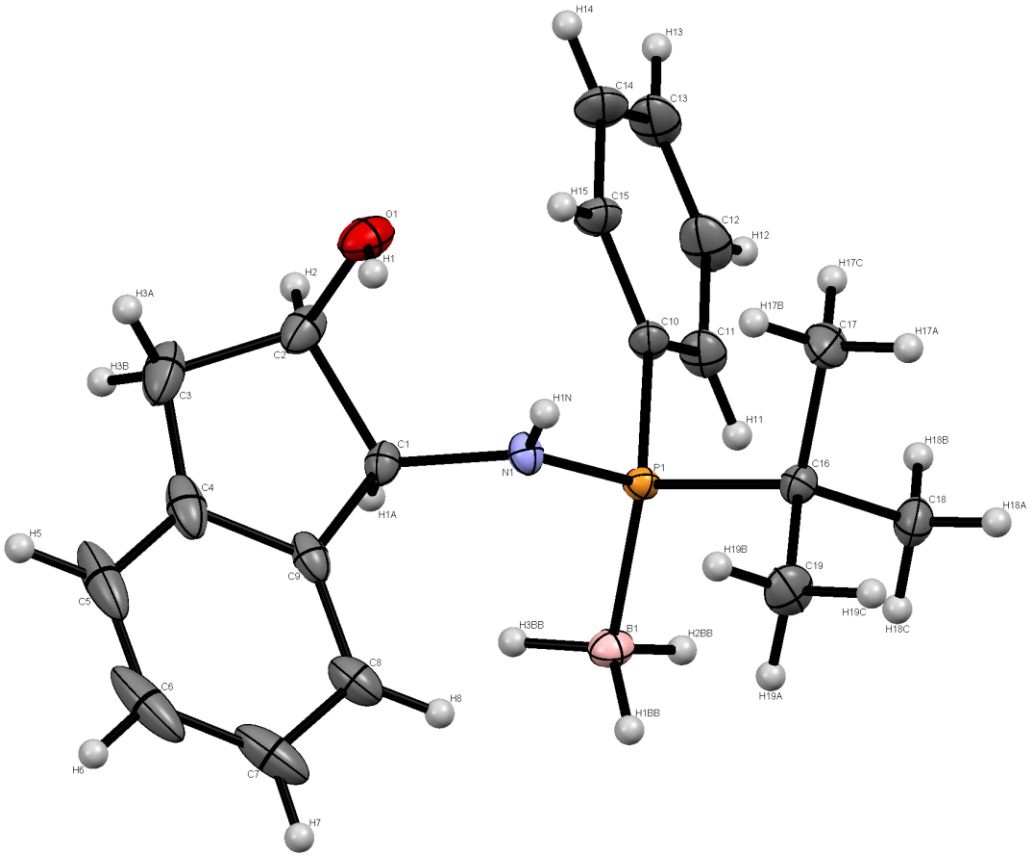
Dades del cristall i refinament de l'estructura de 94



6 Dades de difracció de raigs-X

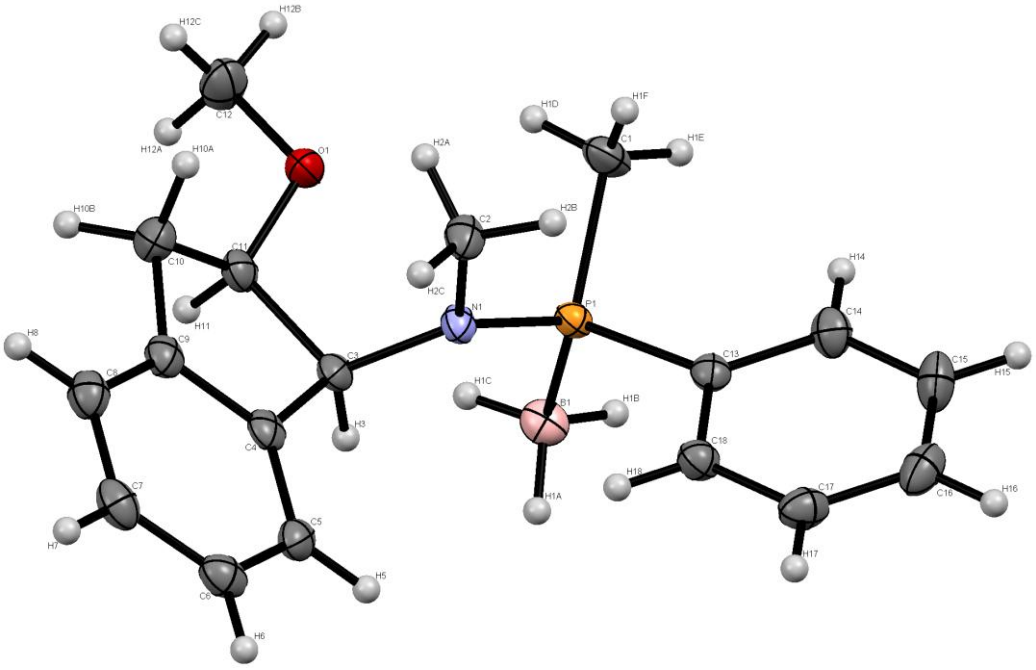
Empirical formula	C14 H25 B N O P	
Formula weight	265.13	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Trigonal	
Space group	P3(2)21	
Unit cell dimensions	a = 12.7622(4) Å	α= 90.00 °.
	b = 12.7622(4) Å	β = 90.00 °.
	c = 33.2409(10) Å	γ = 120.00 °.
Volume	4688.7(3) $Å^3$	
Z	12	
Density (calculated)	1.127 Mg/m^3	
Absorption coefficient	0.165 mm^{-1}	
F(000)	1728	
Crystal size	0.40 x 0.40 x 0.30 mm^3	
Theta range for data collection	1.84 to 35.07 °.	
Index ranges	-18 <=h<=20 ,-10 <=k<=17 ,-53 <=l<=49	
Reflections collected	13701	
Independent reflections	11835 [R(int) = 0.0377]	
Completeness to theta =35.07°	0.998 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9521 and 0.9369	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	13701 / 0 / 337	
Goodness-of-fit on F^2	1.041	
Final R indices [I>2sigma(I)]	R1 = 0.0455 , wR2 = 0.1077	
R indices (all data)	R1 = 0.0564 , wR2 = 0.1140	
Absolute Structure Flack parameter	x =-0.02(5)	
Largest diff. peak and hole	0.447 and -0.258 $e.Å^{-3}$	

Dades del cristall i refinament de l'estructura de 102



Empirical formula	C19 H27 B N O P	
Formula weight	327.20	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 9.8349(6) Å	α= 90.00 °.
	b = 13.0080(8) Å	β = 90.00 °.
	c = 14.6147(11) Å	γ = 90.00 °.
Volume	1869.7(2) $Å^3$	
Z	4	
Density (calculated)	1.162 Mg/m^3	
Absorption coefficient	0.151 mm^{-1}	
F(000)	704	
Crystal size	0.80 x 0.80 x 0.80 mm^3	
Theta range for data collection	2.10 to 37.06°.	
Index ranges	-16 <=h<=7 ,-19 <=k<=22 ,-20 <=l<=13	
Reflections collected	7628	
Independent reflections	6982 [R(int) = 0.0232]	
Completeness to theta =37.06°	0.864 %	
Absorption correction	Empirical	
Max. and min. transmission	0.8890 and 0.8890	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7628 / 0 / 228	
Goodness-of-fit on F^2	1.107	
Final R indices [I>2sigma(I)]	R1 = 0.0372 , wR2 = 0.0945	
R indices (all data)	R1 = 0.0424 , wR2 = 0.0979	
Absolute Structure Flack parameter	x =0.03(5)	
Largest diff. peak and hole	0.624 and -0.201 $e.Å^{-3}$	

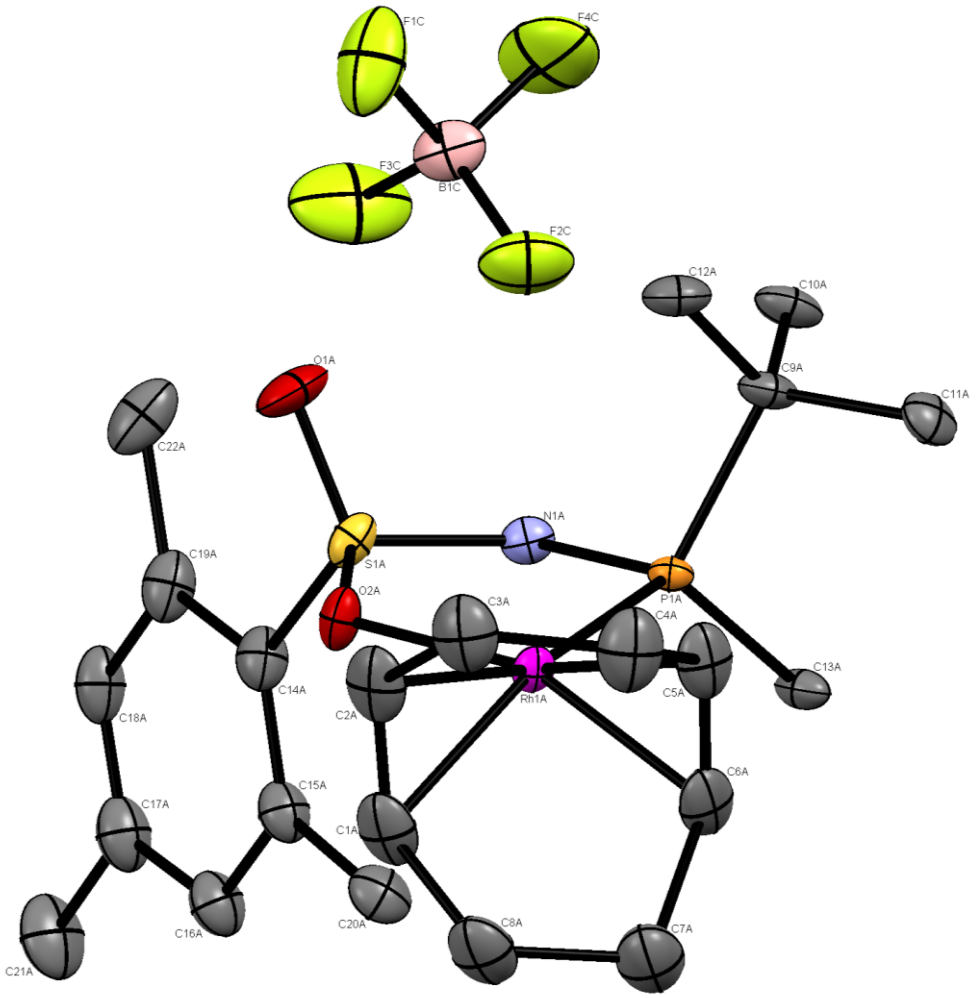
Dades del cristall i refinament de l'estructura de 93a



6 Dades de difracció de raigs-X

Empirical formula	C18 H25 B N O P	
Formula weight	313.17	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 7.0769(6) Å	α= 90.00°.
	b = 15.3364(18) Å	β = 98.595(4)°.
	c = 8.3414(7) Å	γ = 90.00°.
Volume	895.16(15) $Å^3$	
Z	2	
Density (calculated)	1.162 Mg/m^3	
Absorption coefficient	0.154 mm^{-1}	
F(000)	336	
Crystal size	0.40 x 0.20 x 0.05 mm^3	
Theta range for data collection	2.47 to 27.52°.	
Index ranges	-9 <=h<=7 ,-19 <=k<=19 ,-10 <=l<=10	
Reflections collected	6967	
Independent reflections	3773 [R(int) = 0.0272]	
Completeness to theta =27.52°	0.982 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9923 and 0.9408	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3773 / 1 / 203	
Goodness-of-fit on F^2	1.095	
Final R indices [I>2sigma(I)]	R1 = 0.0373 , wR2 = 0.0887	
R indices (all data)	R1 = 0.0401 , wR2 = 0.0968	
Absolute Structure Flack parameter	x =0.07(8)	
Largest diff. peak and hole	0.239 and -0.341 $e.Å^{-3}$	

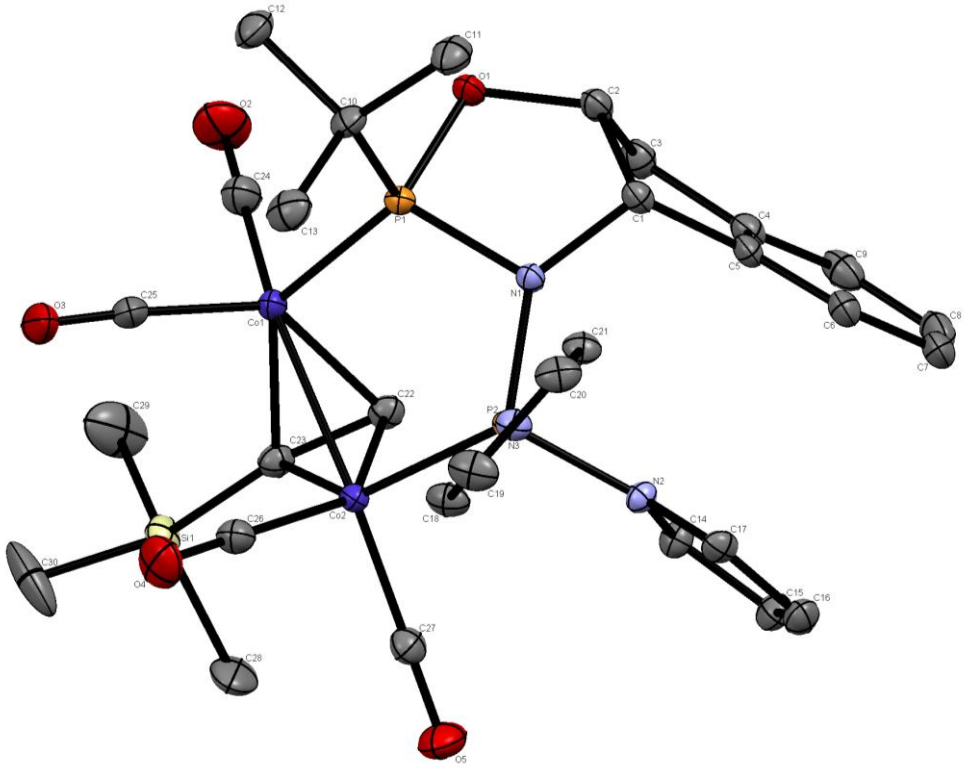
Dades del cristall i refinament de l'estructura de 206



6 Dades de difracció de raigs-X

Empirical formula	C22 H35 B F4 N O2 P Rh S	
Formula weight	598.26	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	P4(1)	
Unit cell dimensions	a = 9.4687(4) Å	α= 90.00°.
	b = 9.4687(4) Å	β = 90.00°.
	c = 56.674(2) Å	γ = 90.00°.
Volume	5081.2(4) $Å^3$	
Z	8	
Density (calculated)	1.564 Mg/m^3	
Absorption coefficient	0.867 mm^{-1}	
F(000)	2456	
Crystal size	0.25 x 0.15 x 0.10 mm^3	
Theta range for data collection	2.15 to 29.57°.	
Index ranges	-8 <=h<=12 ,-11 <=k<=12 ,-48 <=l<=69	
Reflections collected	16879	
Independent reflections	9843 [R(int) = 0.0355]	
Completeness to theta =29.57°	0.857 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9183 and 0.8125	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9843 / 740 / 700	
Goodness-of-fit on F^2	1.140	
Final R indices [I>2sigma(I)]	R1 = 0.0704 , wR2 = 0.2042	
R indices (all data)	R1 = 0.0875 , wR2 = 0.2560	
Absolute Structure Flack parameter	x =0.07(6)	
Largest diff. peak and hole	1.616 and -1.735 $e.Å^{-3}$	

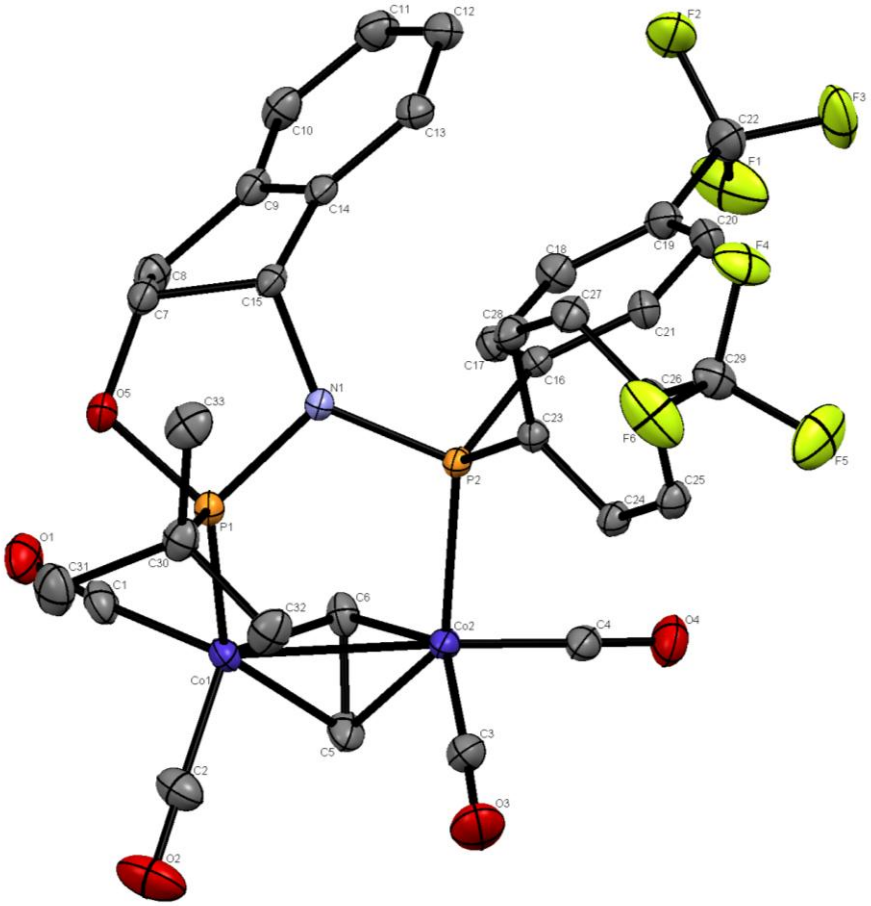
Dades del cristall i refinament de l'estructura de 264



6 Dades de difracció de raigs-X

Empirical formula	C30 H37 Co2 N3 O5 P2 Si	
Formula weight	727.52	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 8.8649(8) Å	α= 90.00°.
	b = 20.8055(19) Å	β = 101.487(3)°.
	c = 9.1083(8) Å	γ = 90.00°.
Volume	1646.3(3) $Å^3$	
Z	2	
Density (calculated)	1.468 Mg/m^3	
Absorption coefficient	1.183 mm^{-1}	
F(000)	752	
Crystal size	0.15 x 0.05 x 0.05 mm^3	
Theta range for data collection	1.96 to 30.02°.	
Index ranges	-11 <=h<=12 ,-29 <=k<=28 ,-12 <=l<=12	
Reflections collected	31075	
Independent reflections	8453 [R(int) = 0.0249]	
Completeness to theta =30.02°	0.918 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9432 and 0.8425	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8453 / 85 / 400	
Goodness-of-fit on F^2	1.013	
Final R indices [I>2sigma(I)]	R1 = 0.0225 , wR2 = 0.0584	
R indices (all data)	R1 = 0.0234 , wR2 = 0.0591	
Absolute Structure Flack parameter	x =-0.007(6)	
Largest diff. peak and hole	0.477 and -0.745 $e.Å^{-3}$	

Dades del cristall i refinament de l'estructura de 275



6 Dades de difracció de raigs-X

Empirical formula	C36 H34 Co2 F6 N O5 P2	
Formula weight	854.44	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2	
Unit cell dimensions	a = 15.8829(6) Å	α= 90°.
	b = 19.5684(9) Å	β= 90°.
	c = 11.5831(5) Å	γ = 90°.
Volume	3600.1(3) $Å^3$	
Z	4	
Density (calculated)	1.576 Mg/m^3	
Absorption coefficient	1.085 mm^{-1}	
F(000)	1740	
Crystal size	0.20 x 0.20 x 0.15 mm^3	
Theta range for data collection	1.65 to 37.49°.	
Index ranges	-26<=h<=26, -31<=k<=32, -19<=l<=19	
Reflections collected	51852	
Independent reflections	17881 [R(int) = 0.0348]	
Completeness to theta = 32.50°	99.9 %	
Max. and min. transmission	0.8542 and 0.8122	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	17881 / 169 / 553	
Goodness-of-fit on F^2	1.046	
Final R indices [I>2sigma(I)]	R1 = 0.0350, wR2 = 0.0860	
R indices (all data)	R1 = 0.0475, wR2 = 0.0937	
Absolute structure parameter	-0.002(6)	
Largest diff. peak and hole	1.228 and -1.009 $e.Å^{-3}$	