Phosphine/Sulfoxide-Carbone, a Ligand with a Flexible Bonding Mode for Early to Late Transition Metals

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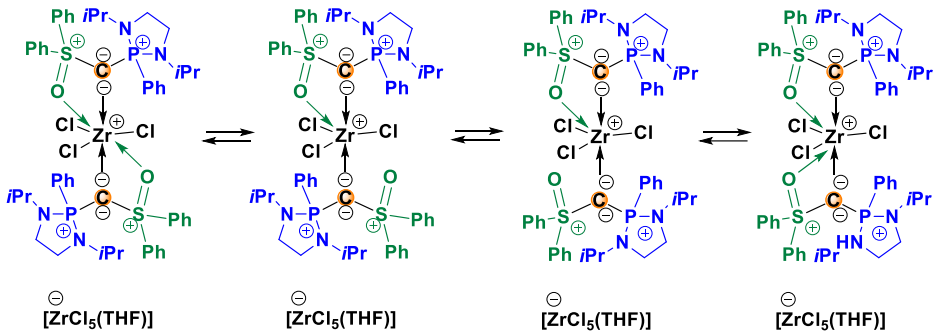
S1. General Methods

All manipulations were performed under an inert atmosphere of argon by using standard Schlenk techniques. Dry and oxygen-free solvents were used. ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded on Brucker Avance 500 or Avance 300 spectrometers. ^{1}H NMR and ^{13}C NMR chemical shifts are reported in parts per million (ppm) relative to Me_4Si as external standard. ^{31}P NMR downfield chemical are expressed in ppm relative to 85 % H_3PO_4. ^{19}F-chemical shifts were reported in ppm relative to C_FCl_3 as an external standard. Mass spectra were recorded on Hewlett Packard 5989A spectrometer. All commercially available reagents were used without further purification otherwise noted. Ylide **1-HOTf** and **7-HOTf** were prepared following previously reported procedures.[1,2]

S2. Additional experimental information



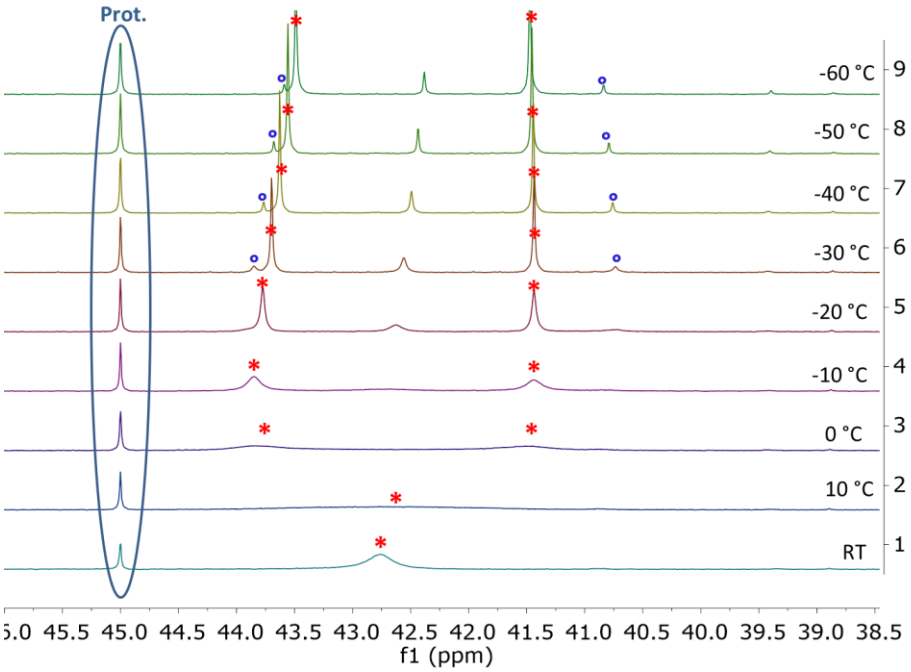


Figure S1: Fluxionnal Behavior of Zr Complex observed by VT experiments.

[1] González, M. L.; Bousquet, L.; Hameury, S.; Alvarez Toledano, C.; Saffon-Merceron, N.; Branchadell, V.; Maerten, E.; Baceiredo, A. Phosphine/Sulfoxide-Supported Carbon(0) Complex. Chem. Eur. J. 2018, 24, 2570–2574.

[2] Dellus, N.; Kato, T.; Bagán, X.; Saffon-Merceron, N.; Branchadell, V.; Baceiredo, A. An isolable mixed P,S-bis(ylide) as an asymmetric carbon atom source. *Angew. Chem. Int. Ed.* **2010**, *49*, 6798–6801

S3. NMR Spectra

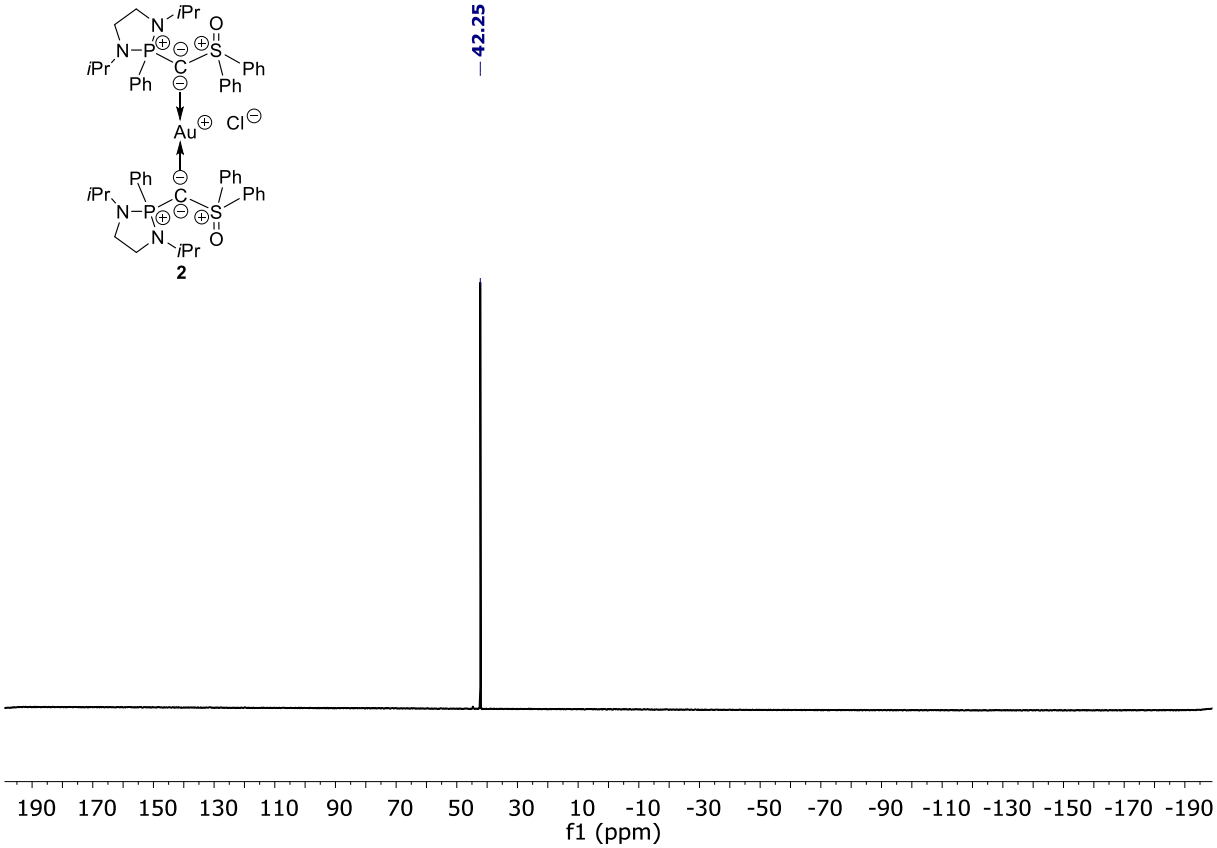


Figure S2: $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 298 K, 202 MHz) of gold(I) complex **2**

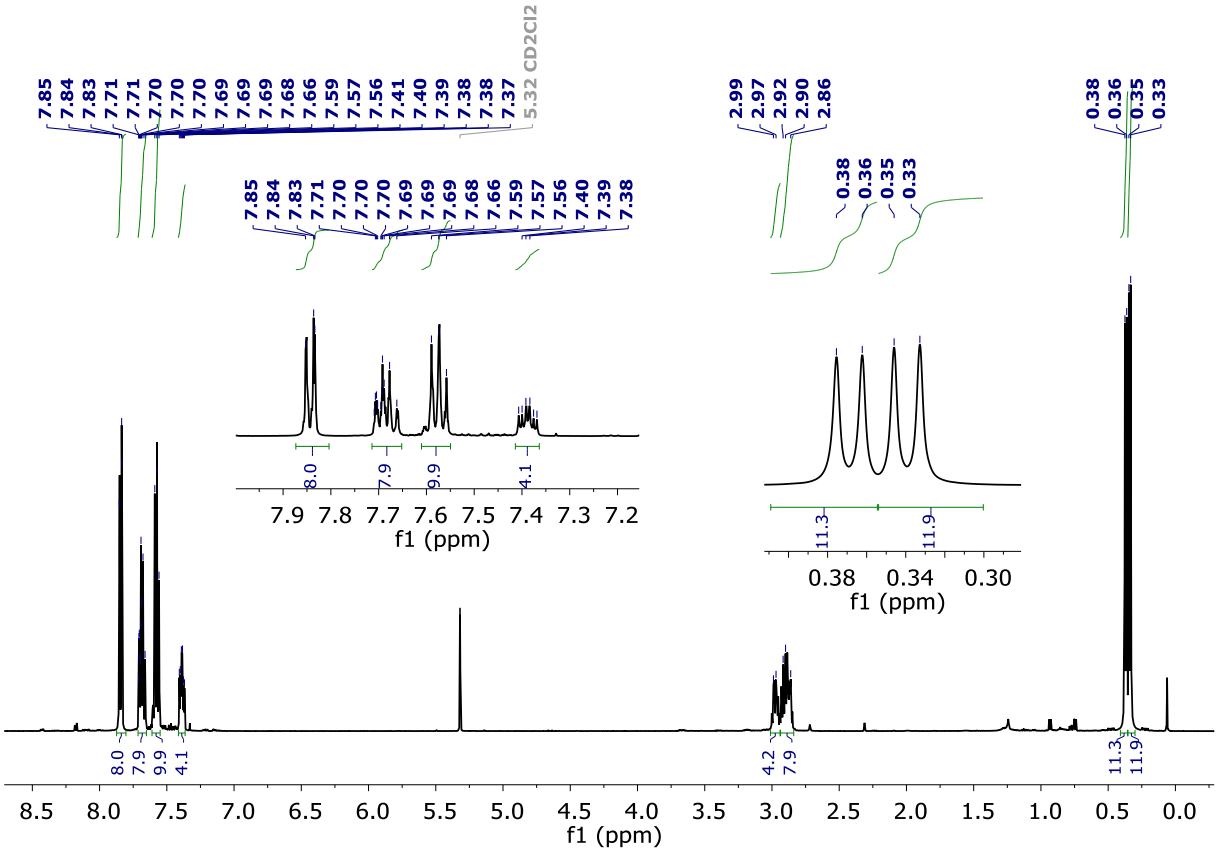


Figure S3: ^{1}H NMR (CD_2Cl_2, 298 K, 500 MHz) of gold(I) complex **2**

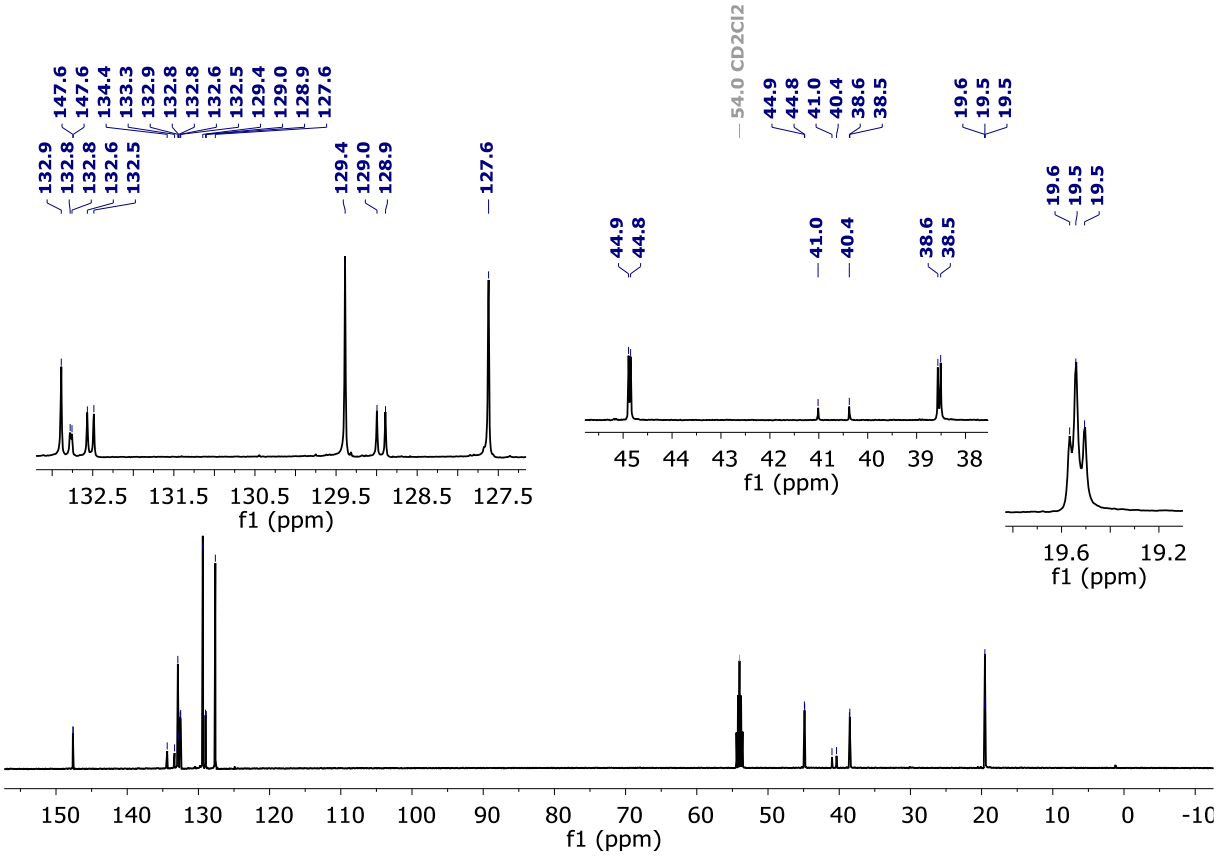


Figure S4: $^{13}C\{^{1}H\}$ NMR (CD_2Cl_2, 298 K, 126 MHz) of gold(I) complex **2**

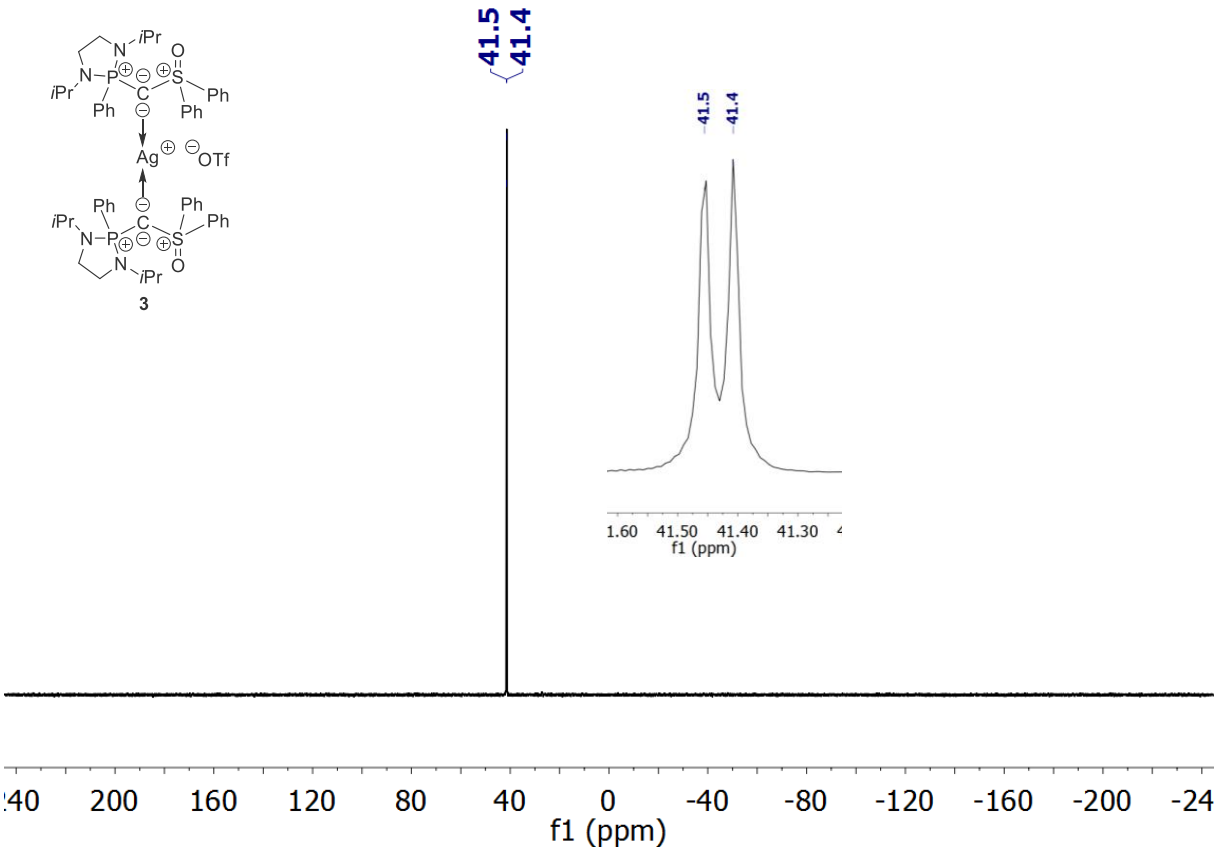


Figure S5: $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 298 K, 121 MHz) of Silver complex **3**

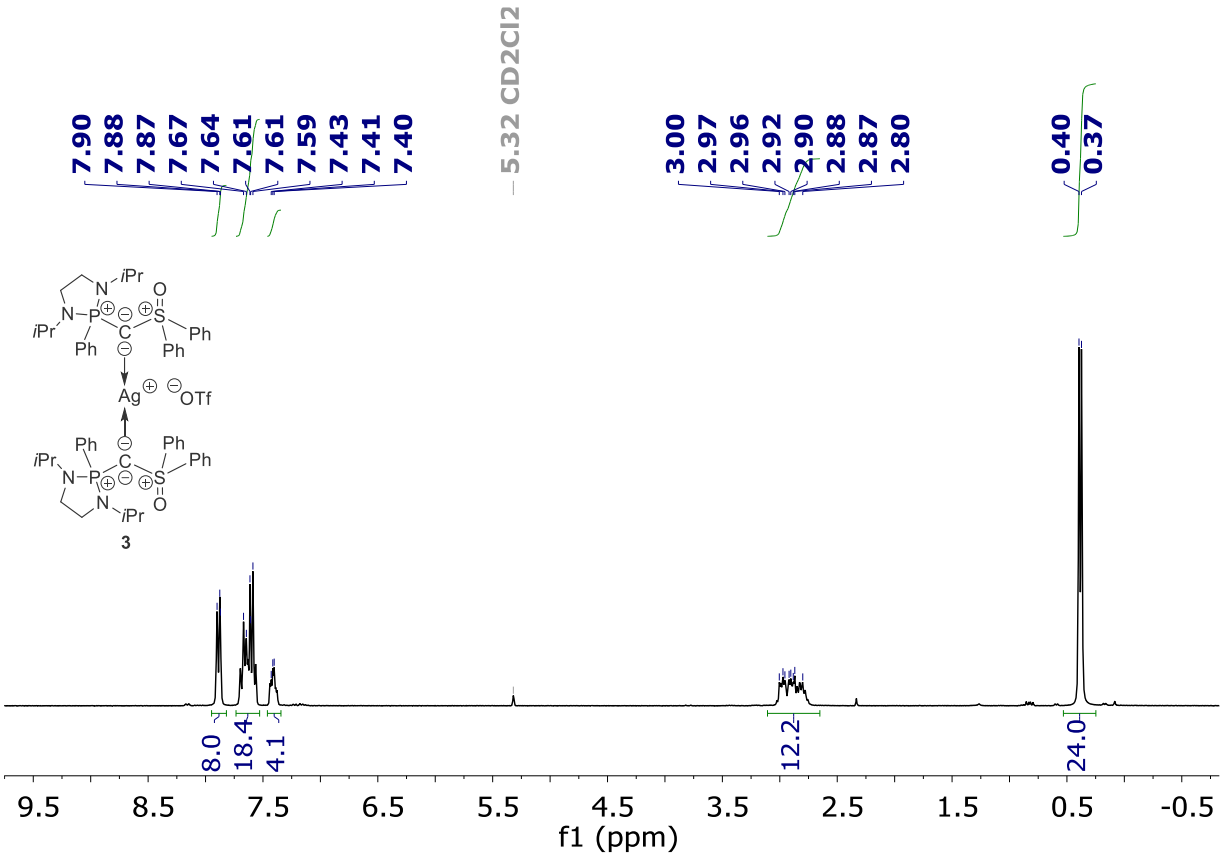


Figure S6: ^{1}H NMR (CD_2Cl_2, 298 K, 300 MHz) of Silver complex **3**

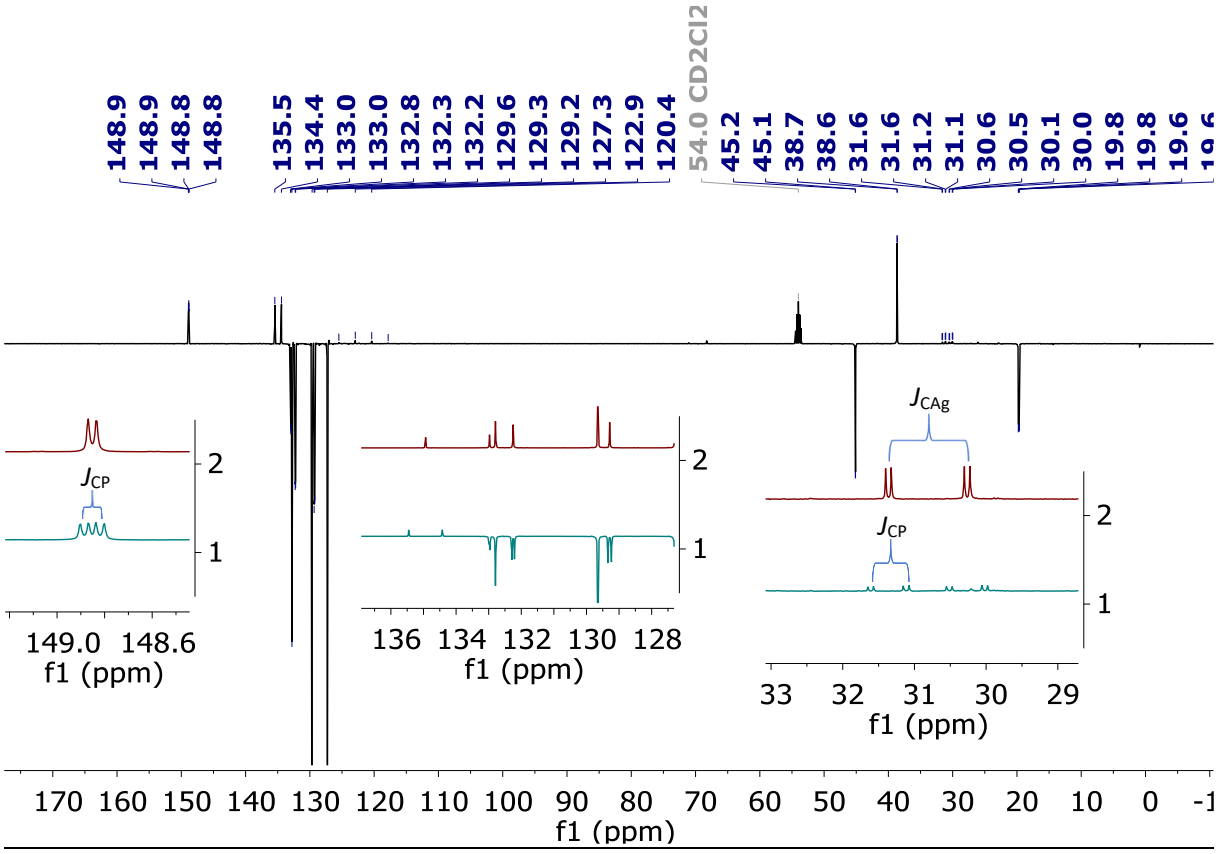


Figure S7: J-mod $^{13}C\{^{1}H\}$ NMR (CD_2Cl_2, 298 K, 75 MHz) of Silver complex **3.** Zoom on classical ^{13}C NMR (green) and J-mod $^{13}C\{^{1}H, ^{31}P\}$ (red).

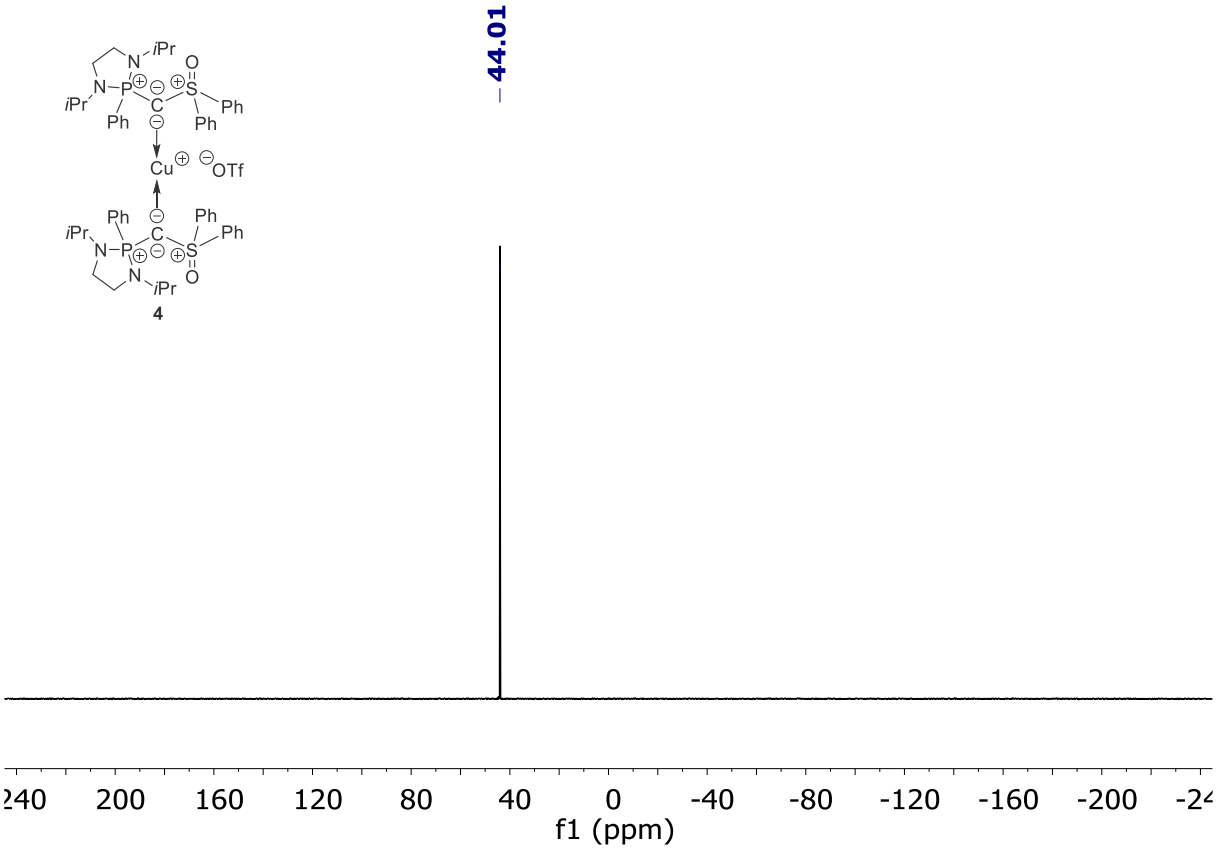


Figure S8: $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 298 K, 121 MHz) of Copper complex **4**

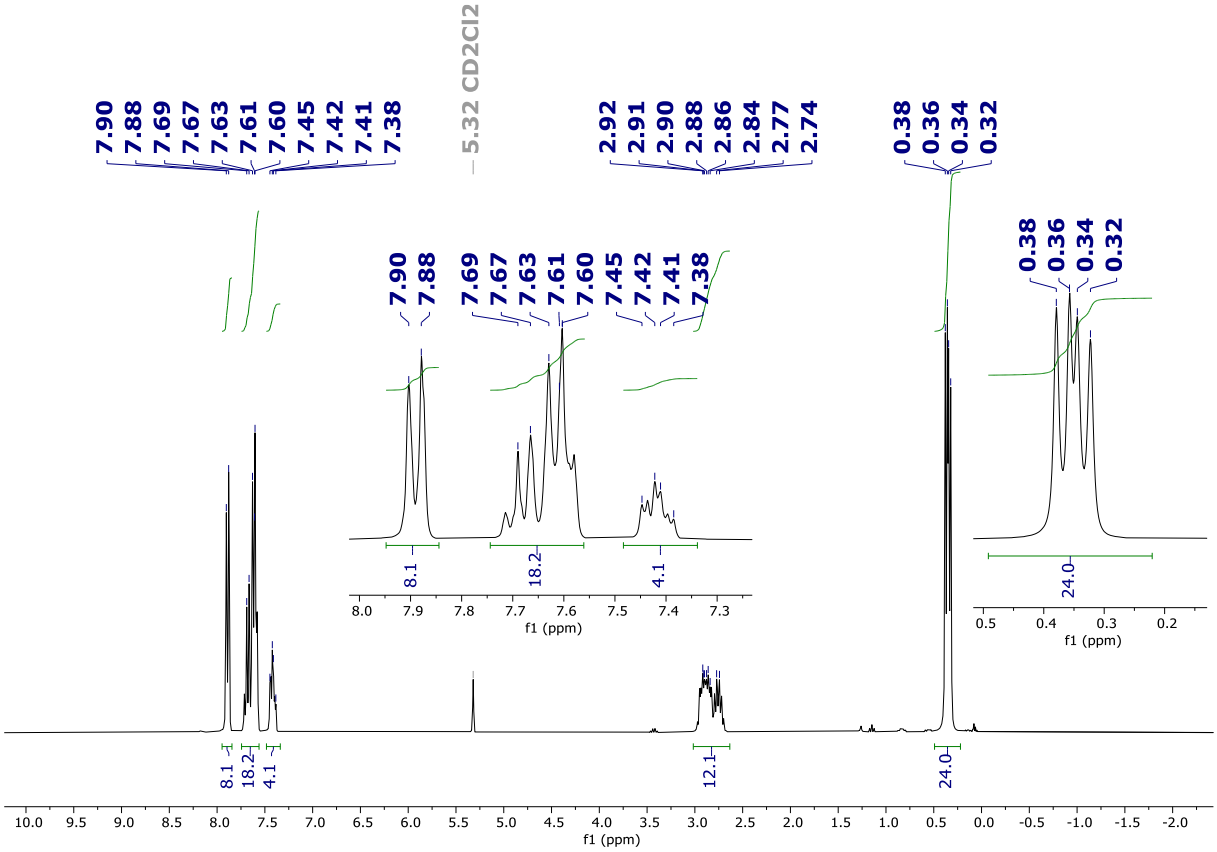


Figure S9: ^{1}H NMR (CD_2Cl_2, 298 K, 300 MHz) of Copper complex **4**

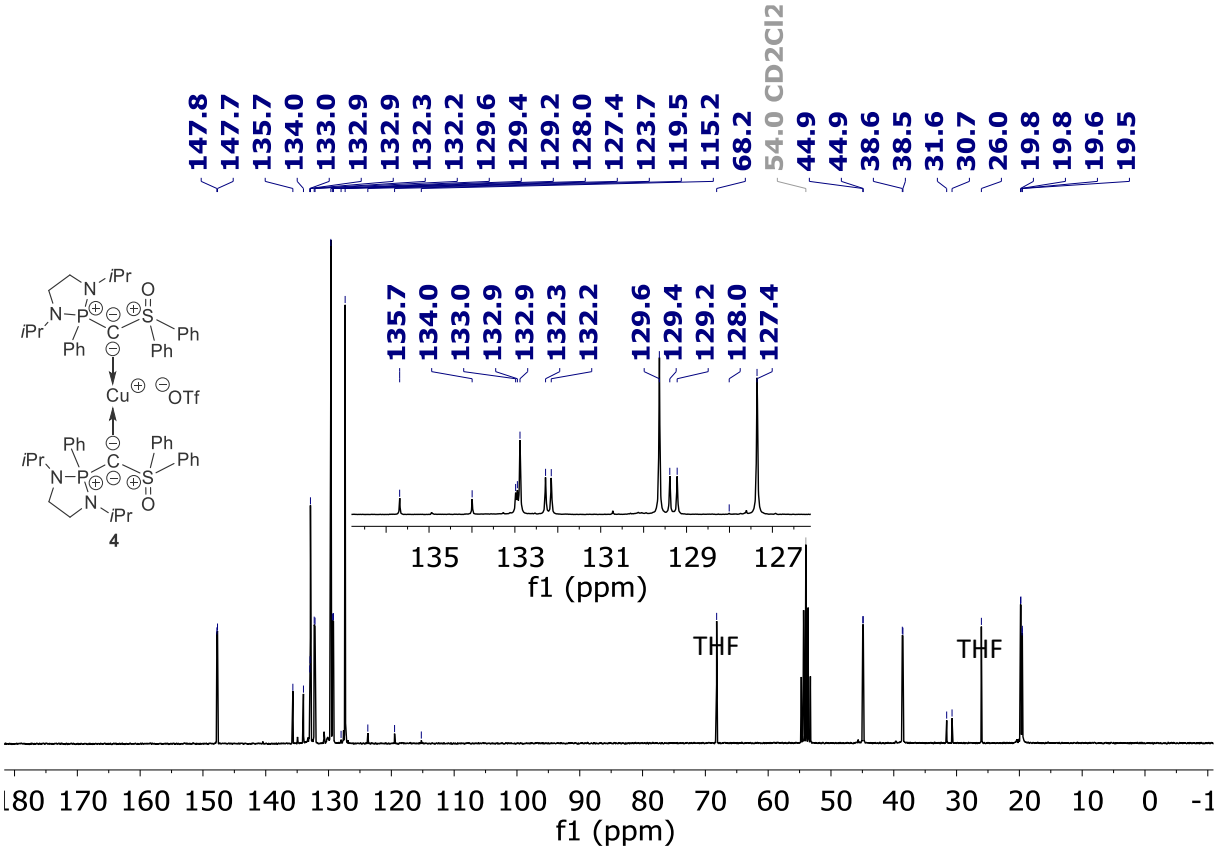


Figure S10: $^{13}C\{^{1}H\}$ NMR (CD_2Cl_2, 298 K, 75 MHz) of Copper complex **4**

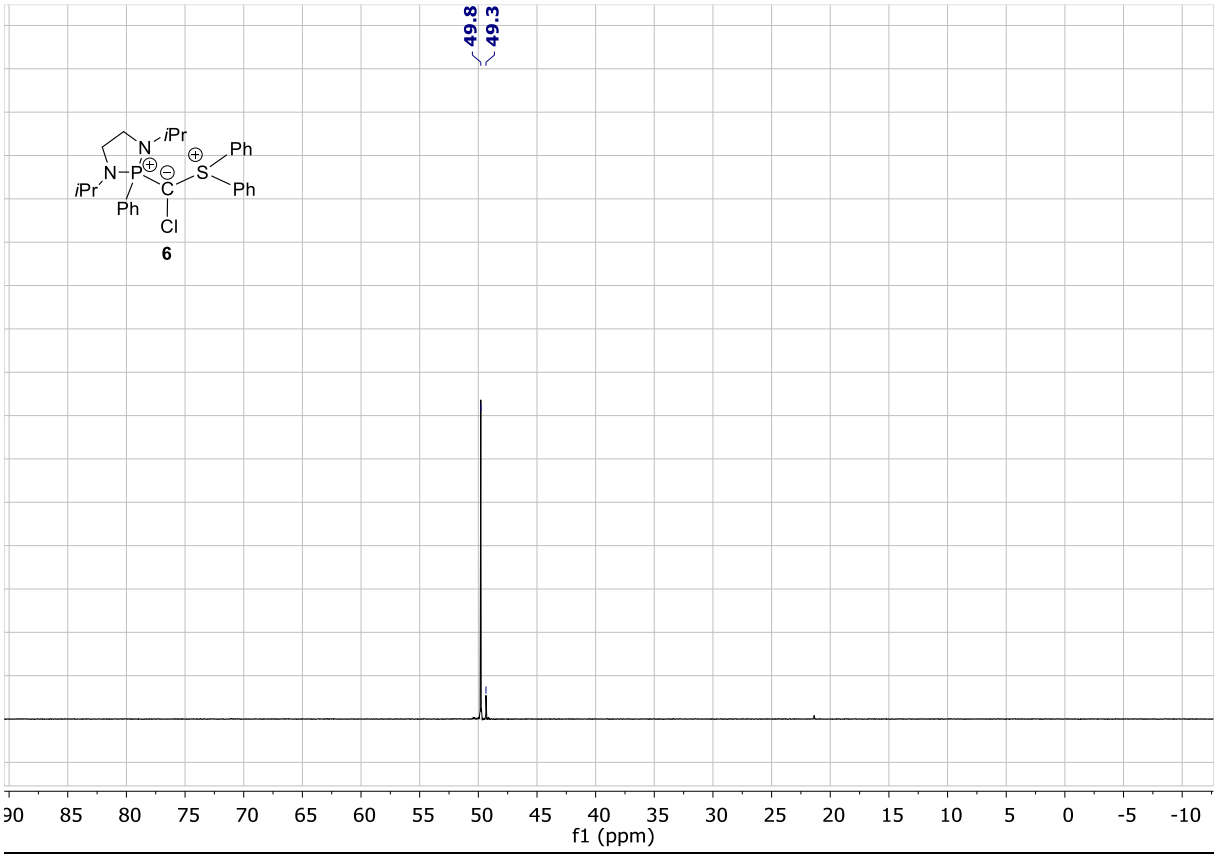


Figure S11: $^{31}P\{^{1}H\}$ NMR ($CDCl_3$, 298 K, 121 MHz) of chlorinated ylide **6**

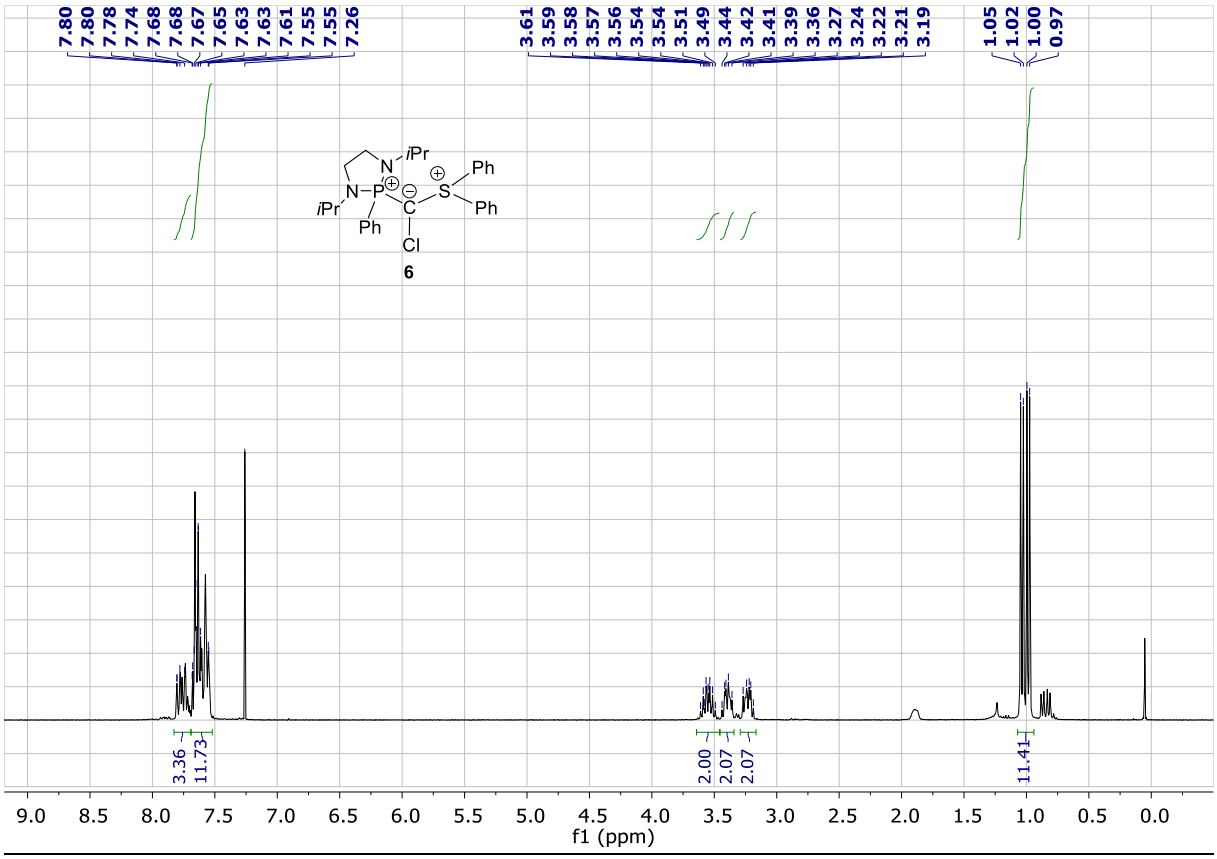


Figure S12: ^{1}H NMR ($CDCl_3$, 298 K, 300 MHz) of chlorinated ylide **6**

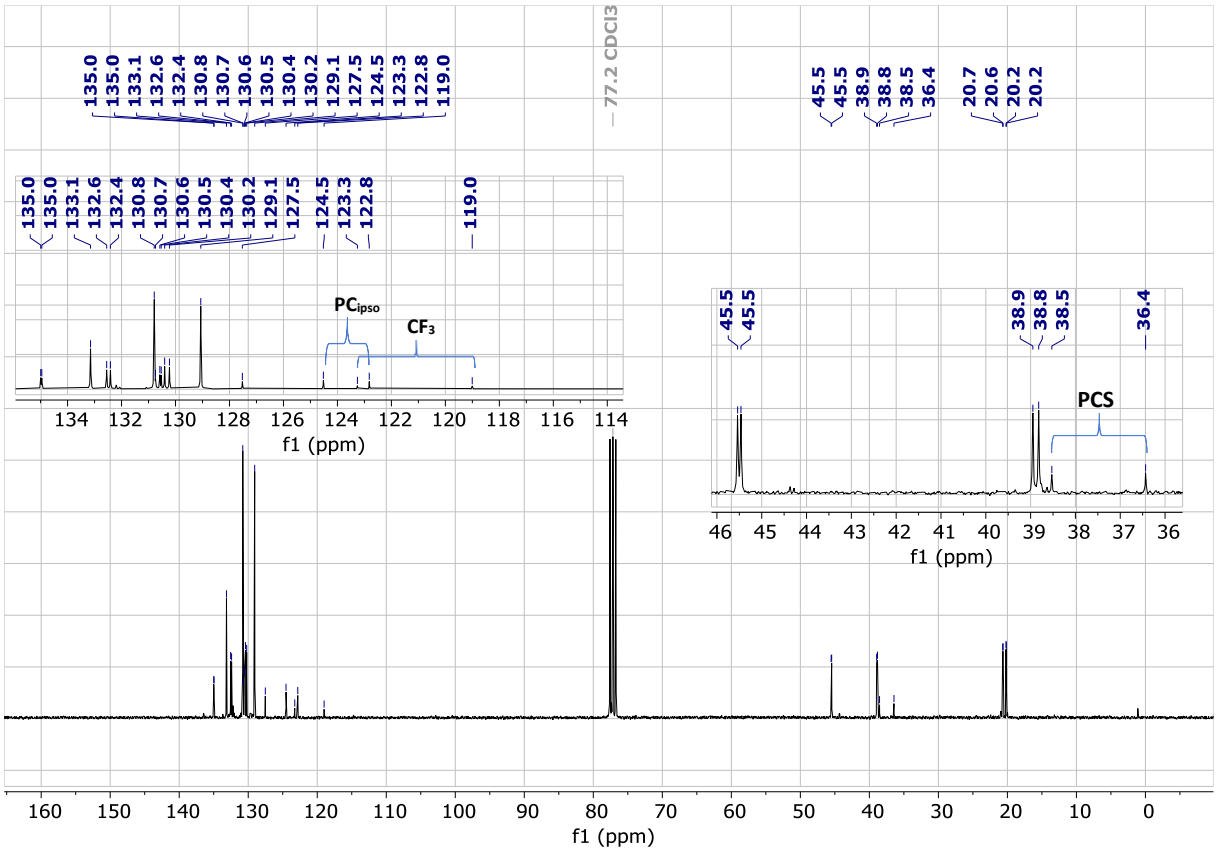


Figure S13: $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 298 K, 75 MHz) of chlorinated ylide **6**

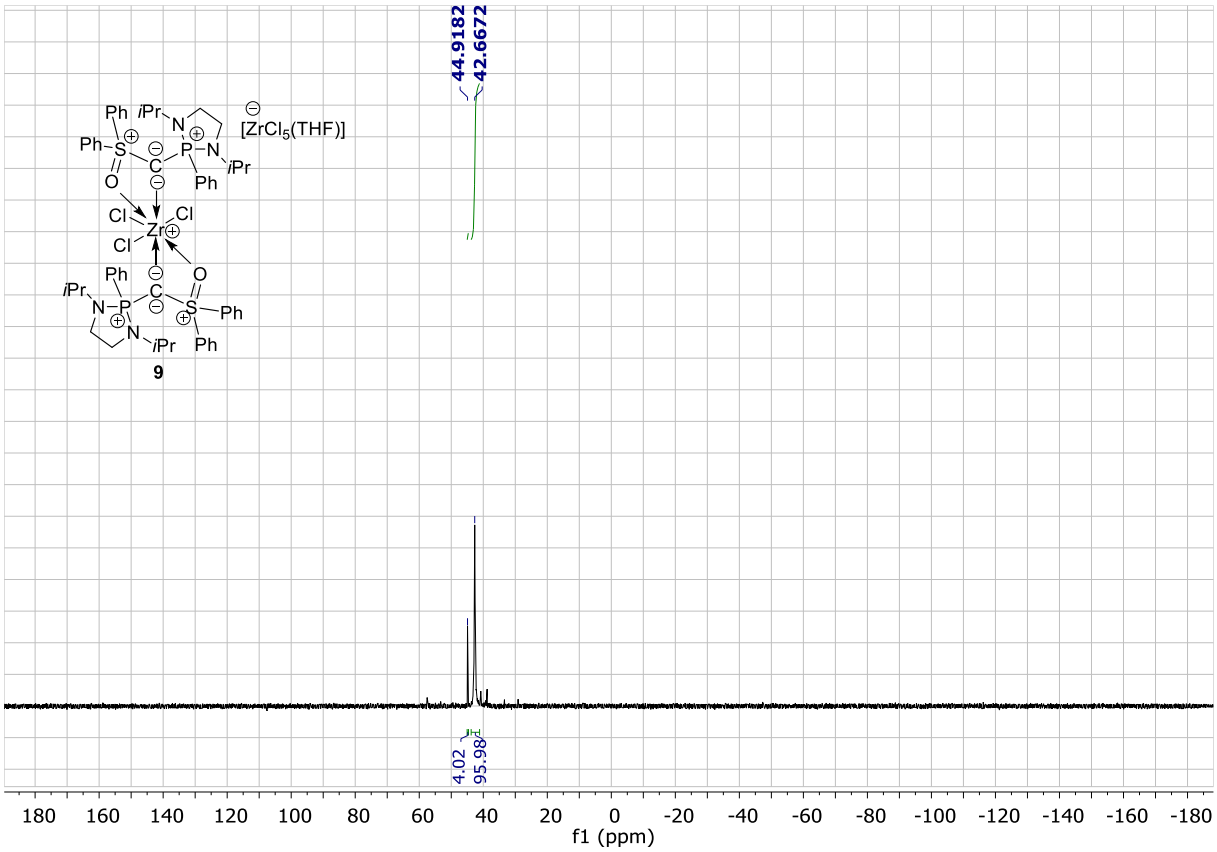


Figure S14: $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 298 K, 121 MHz) of Zirconium complex **9**

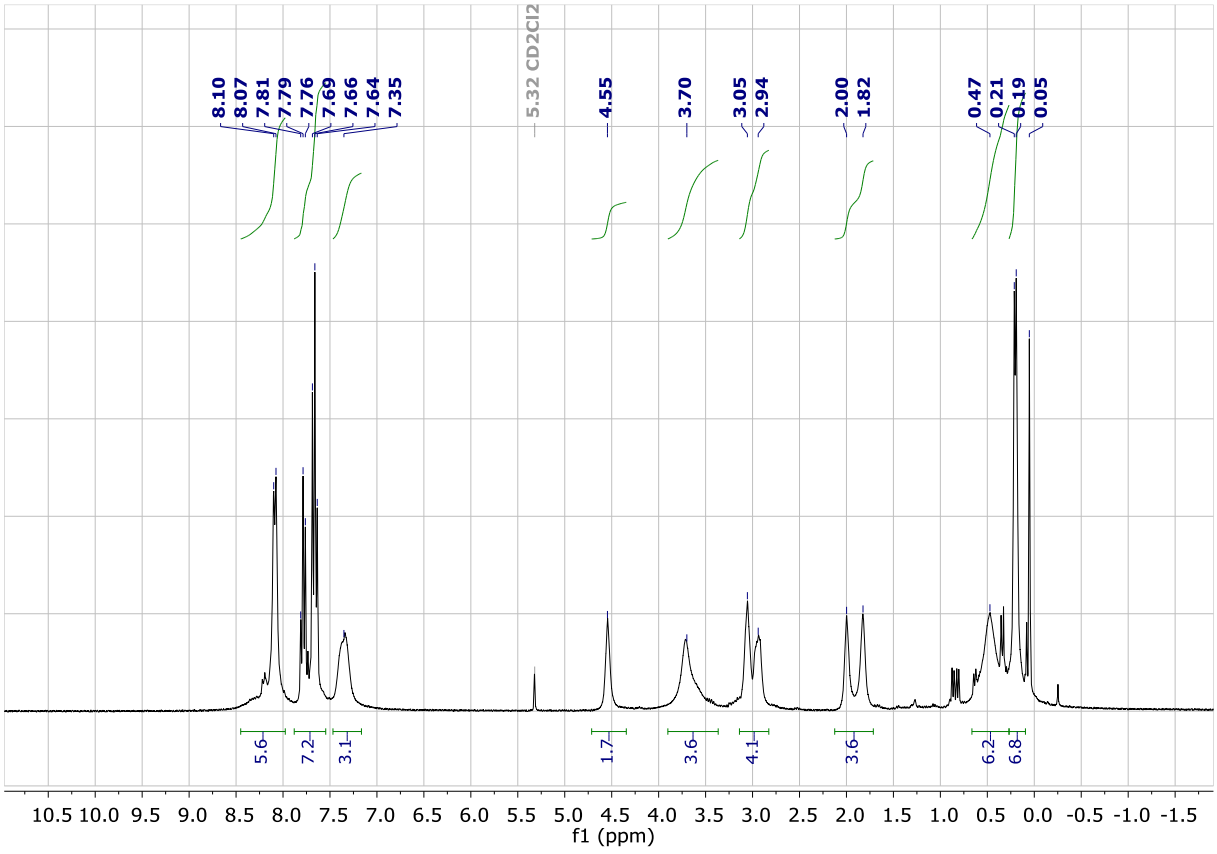


Figure S15: ^{1}H NMR (CD_2Cl_2, 298 K, 300 MHz) of Zirconium complex **9**

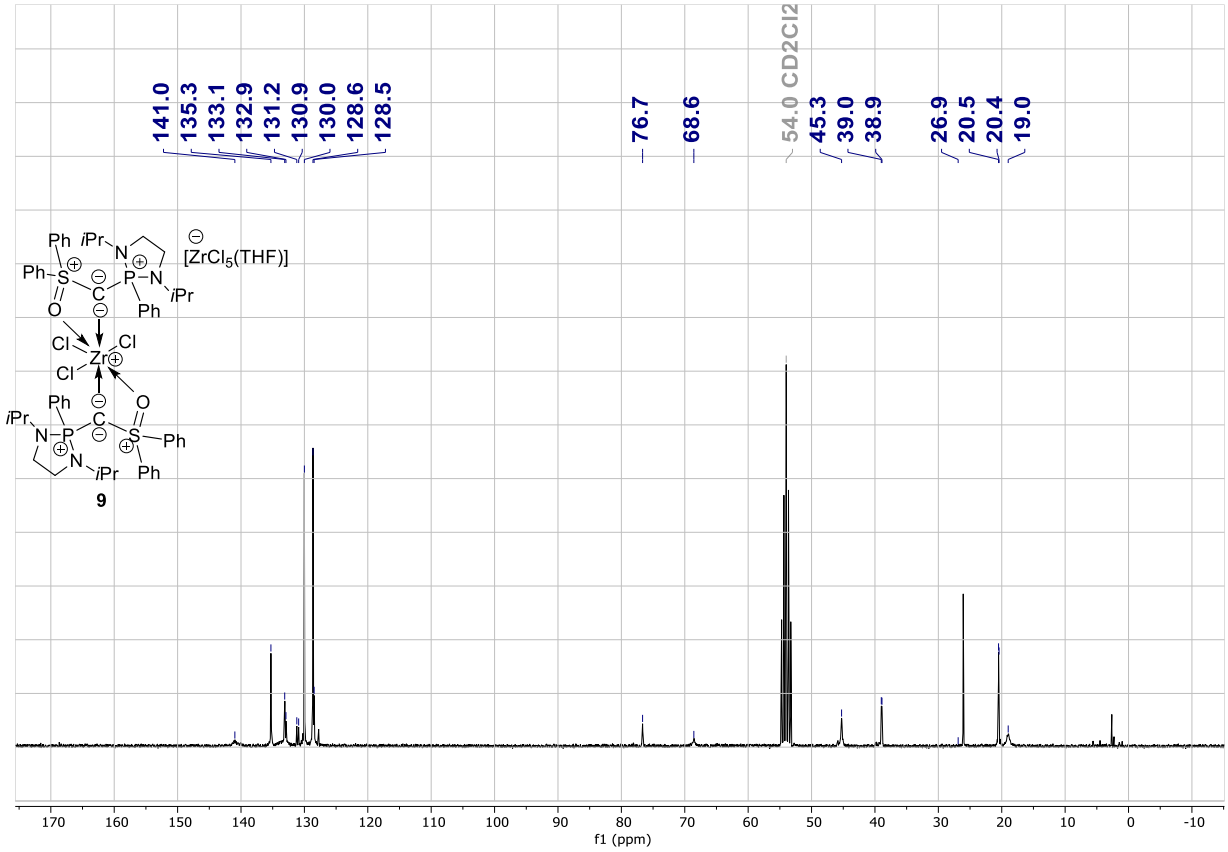


Figure S16: $^{13}C\{^{1}H\}$ NMR (CD_2Cl_2, 298 K, 75 MHz) of Zirconium complex **9**

S4. X-ray analysis

The details of data collection and crystal structures refinement are summarized in Table S1.

Supplementary crystallographic data for CCDC-2366566 (**2**), CCDC- 2366567 (**3**), CCDC- 2366568 (**4**), CCDC- 2366569 (**6**), CCDC- 2366570 (**9**) and CCDC- 2366571 (**11**) can be obtained free of charge from The Cambridge Crystallographic Data Centre via https://www.ccdc.cam.ac.uk/structures/.

Table S1. Crystallographic data for the compounds **2**, **3**, **4**, **6**, **9** and **11**

Compound	**2**	**3**	**4**	**6**	**9**	**11**
Chemical formula	$C_{54}H_{66}N_4O_2P_2S_2Au$,Cl, solvent	$C_{54}H_{66}AgN_4O_2P_2S_2$,CF_3O_3S, C_4H_8O	$C_{54}H_{66}CuN_4O_2P_2S_2$,CF_3O_3S	$C_{27}H_{33}ClN_2PS$, CF_3O_3S	$C_{54}H_{66}Cl_3N_4O_2P_2S_2Zr$, $C_4H_8Cl_5OZr$, $3(C_4H_8O)$	$C_{54}H_{66}Cl_3HfN_4O_2P_2S_2$, $C_4H_8Cl_5HfO$, 4 (C_4H_8O), solvent
M_r	1284.50	1258.21	1141.77	633.10	1683.62	1930.26
Crystal system	Monoclinic	Monoclinic	Triclinic	Monoclinic	Monoclinic	Monoclinic
Space group	*P* 2_1/n	*P* 2_1/c	*P* $\bar{1}$	*P* 2_1/c	Cm	Cc
a [Å]	13.4000(6)	10.3704(6)	10.9404(3)	15.2207(17)	18.201(3)	18.214(3)
b [Å]	18.8462(7)	36.154(2)	15.3548(5)	11.0109(12)	43.730(6)	43.716(8)
c [Å]	24.0889(10)	15.7364(9)	17.1108(5)	18.964(2)	10.6729(15)	21.330(4)
α [°]	90	90	93.6895(12)	90	90	90
β [°]	104.8736(16)	92.685(3)	95.1242(11)	107.449(4)	93.578(4)	93.557(4)
γ [°]	90	90	99.2602(11)	90	90	90
V [$Å^3$]	5879.6(4)	5893.6(6)	2816.53(15)	3032.0(6)	8478(2)	16952(5)
Z	4	4	2	4	4	8
ρ [g cm^{-3}]	1.451	1.418	1.346	1.387	1.319	1.513
μ($Mo_{Kα}$) [mm^{-1}]	2.821	0.565	0.615	0.368	0.632	2.838
Reflections collected	258304	85575	97773	66525	72613	217422
Independent reflections	14644 R(int)=0.0922	11189 R(int)=0.0678	17199 R(int)=0.0499	5327 R(int)=0.1603	13343 R(int)=0.1295	36889 R(int)=0.0635
Data/ restraints/ parameters	14644/311/757	11189/258/783	17199/425/739	4334/0/365	13343/723/1087	36889/1541/2176
Crystal size [mm^3]	0.20x0.12x0.06	0.20x0.10×0.08	0.36×0.20×0.15	0.12×0.06×0.03	0.20×0.10×0.02	0.20×0.15×0.06
GOOF on F^2	1.046	1.042	1.026	1.033	1.038	1.037
R (I > 2σ(I))	0.0296	0.0410	0.0484	0.0447	0.0736	0.0393
wR2 (all data)	0.0506	0.0859	0.1304	0.0948	0.2153	0.0990
Largest difference peak and hole, [e $Å^{-3}$]	0.437 and -0.525	0.353 and -0.507	0.696 and -0.794	0.266 and -0.320	1.182 and -0.808	1.053 and -1.286
CCDC number	2366566	2366567	2366568	2366569	2366570	2366571

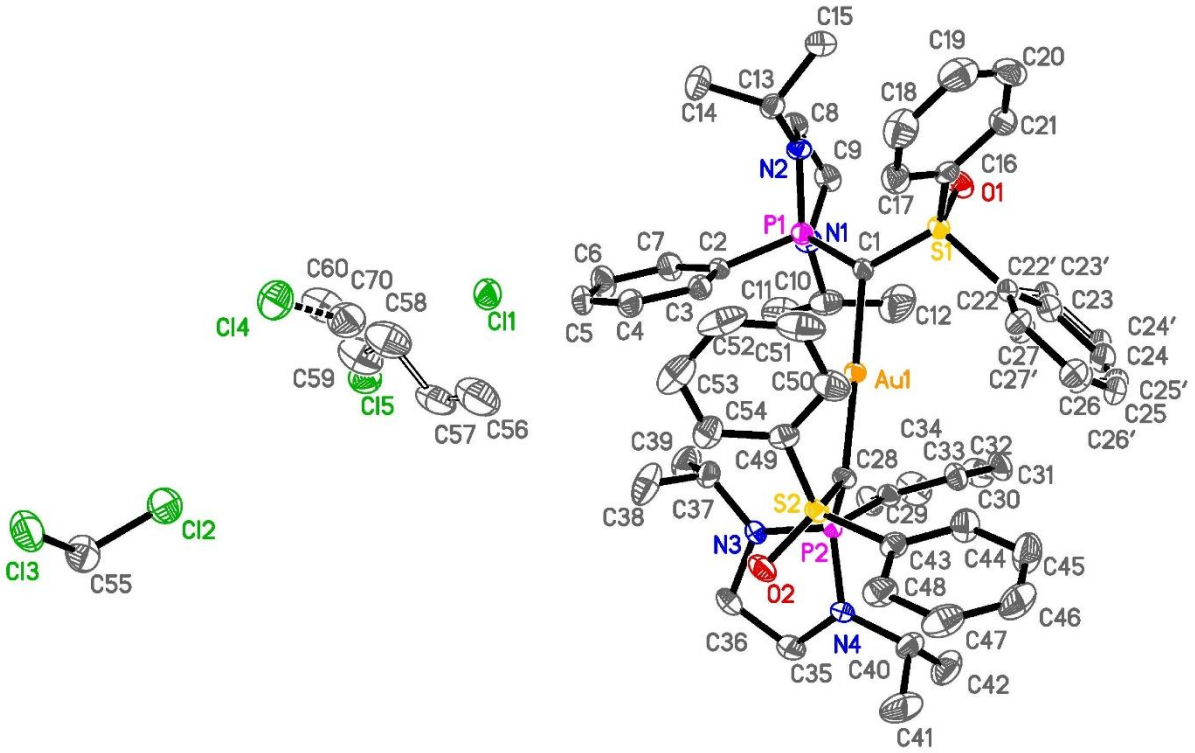


Figure S17: Molecular structure of **2**. Thermal ellipsoids represent 30 % probability. H were omitted for clarity. Selected bond lengths [Å] and angles [°]: Au1-C1 2.043(2), Au1-C28 2.051(2), P1-C1 1.706(3), P2-C28 1.698(3), S1-C1 1.649(3), S2-C28 1.644(2), C1-Au1-C28 177.41(11), S1-C1-P1 116.26(15), S2-C28-P2 117.44(15), P1-C1-Au1 120.40(14), P2-C28-Au1 115.87(13), S1-C1-Au1 120.86(14), S2-C28-Au1 122.37(14).

IUCr's Checkcif provided a B level error

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PROBLEM: Missing # of FCF Reflection(s) Below Theta(Min). 17 Note

RESPONSE: Theta min = 2.90 deg. The theta value of missing reflections is below 2.85 deg. These reflections are therefore excluded.

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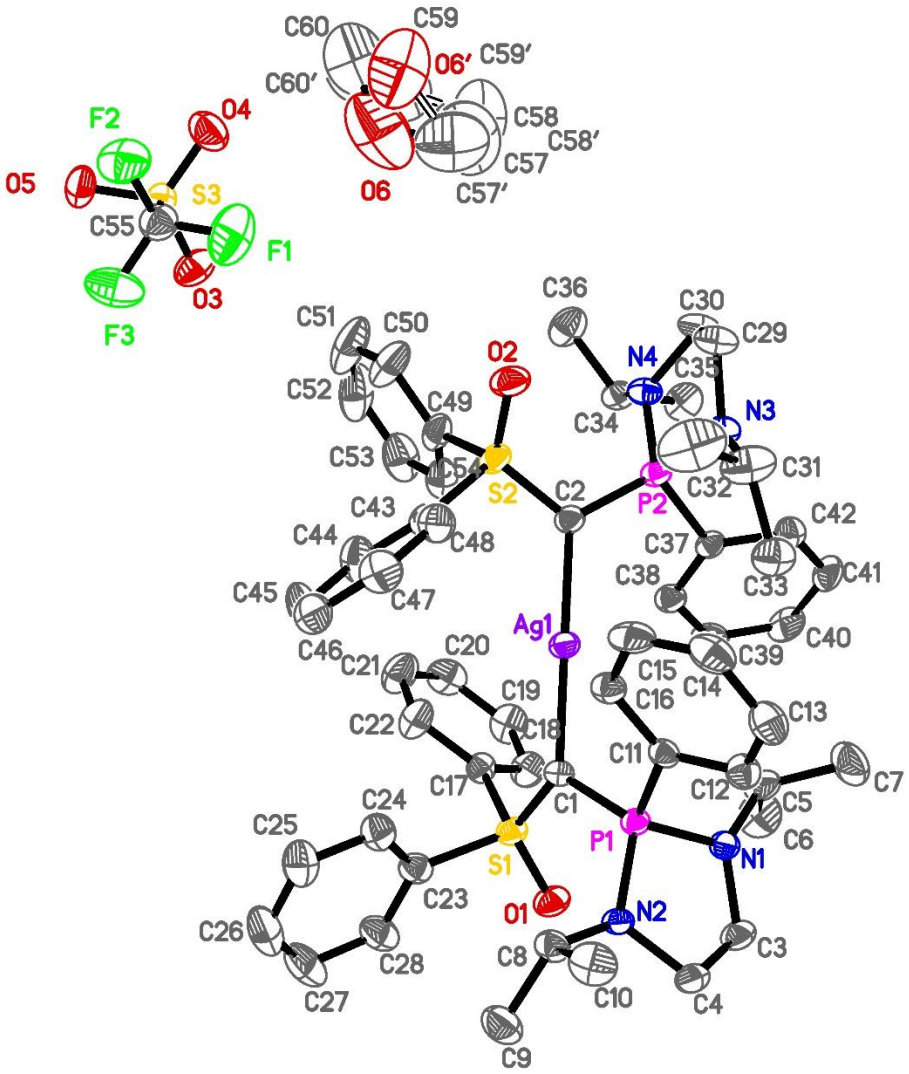

Figure S18: Molecular structure of **3**. Thermal ellipsoids represent 30 % probability. H atoms were omitted for clarity. Selected bond lengths [Å] and angles [°]: Ag1-C2 2.110(3), Ag1-C1 2.114(3), P1-C1 1.695(3), P2-C2 1.694(3), S1-C1 1.636(3), S2-C2 1.634(3), C1-Ag1-C2 179.31(11), S1-C1-P1 115.58(16), S2-C2-P2 116.42(18), S1-C1-Ag1 118.59(15), S2-C2-Ag1 119.01(16), P1-C1-Ag1 120.38(16), P2-C2-Ag1 122.75(15).

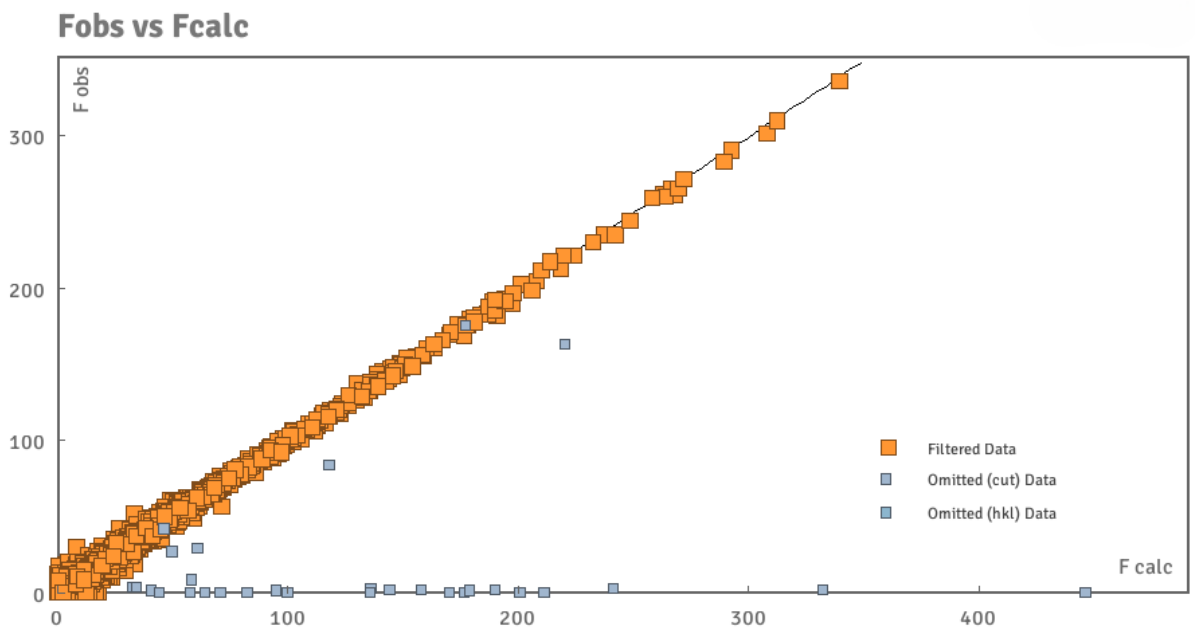

Figure S19: Plot of Fobs versus Fcalc for compound **3**.

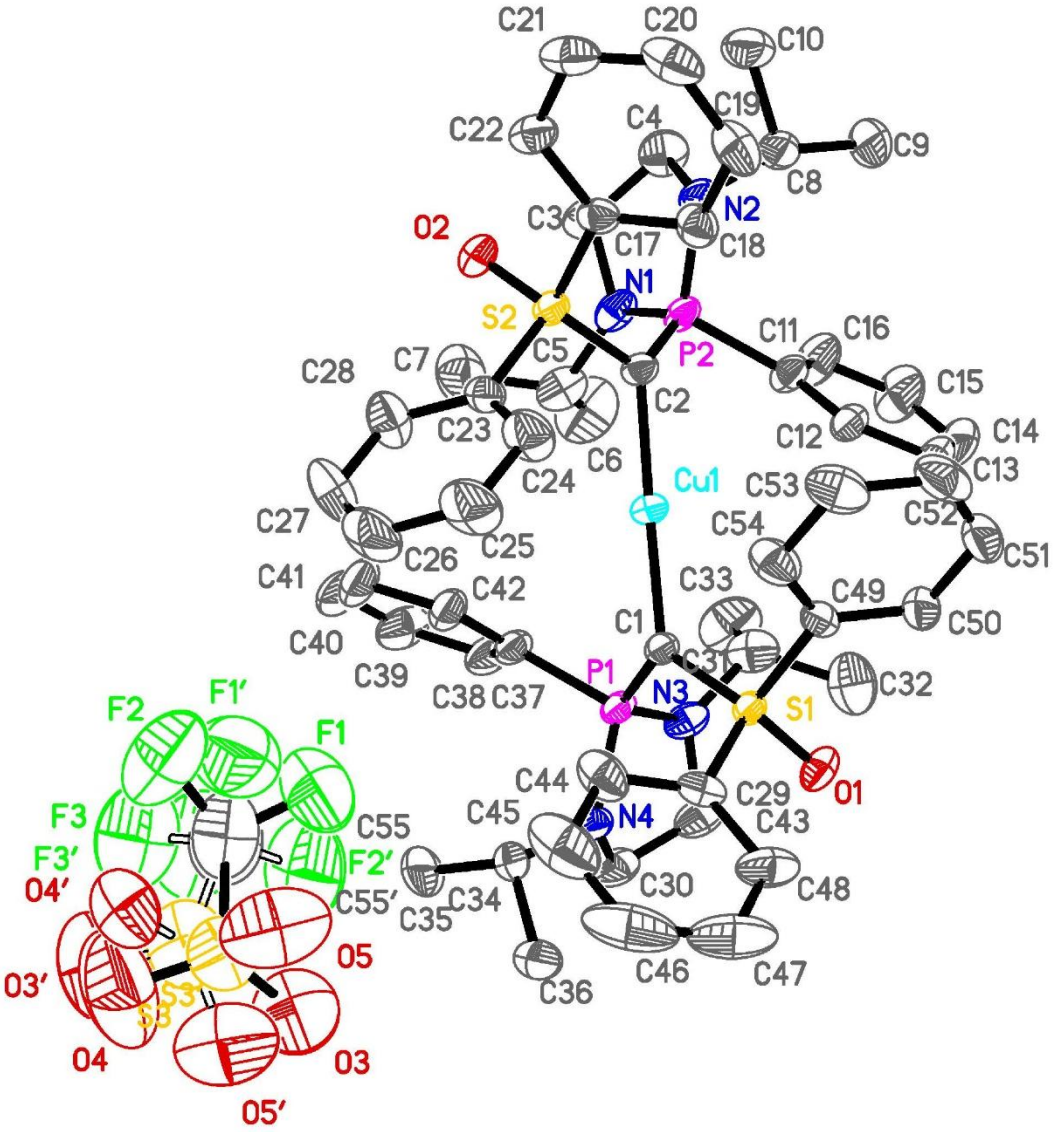

Figure S20: Molecular structure of **4**. Thermal ellipsoids represent 30 % probability. H atoms were omitted for clarity. Selected bond lengths [Å] and angles [°]: Cu1-C2 1.919(2), Cu1-C1 1.918(2), P1-C1 1.702(2), P2-C2 1.706(2), S1-C1 1.638(2), S2-C2 1.641(2), C1-Cu1-C2 178.34(9), S1-C1-P1 114.70(11), S2-C2-P2 114.49(11), S1-C1-Cu1 121.66(11), S2-C2-Cu1 121.44(11), P1-C1-Cu1 119.31(10), P2-C2-Cu1 118.16(11).

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PROBLEM: Missing # of FCF Reflection(s) Below Theta(Min). 16 Note

RESPONSE: Theta min = 2.86 deg. The theta value of missing reflections is below 2.84 deg. These reflections are therefore excluded.

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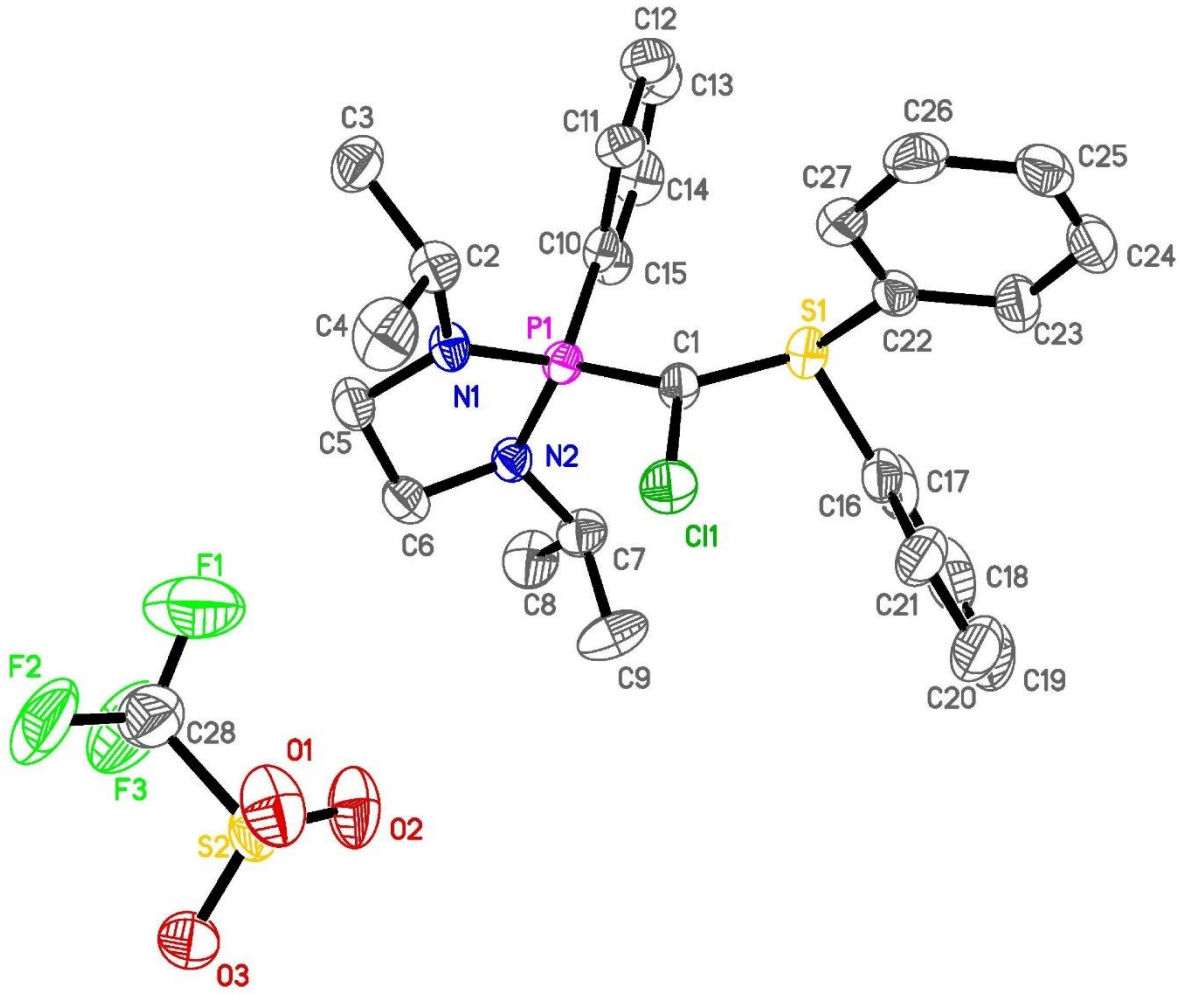

Figure S21: Molecular structure of **6**. Thermal ellipsoids represent 30 % probability. H atoms were omitted for clarity. Selected bond lengths [Å] and angles [°]: P1-C1 1.710(3), S1-C1 1.684(3), Cl1-C1 1.748(3), S1-C1-P1 120.3(2), P1-C1-Cl1 118.0(2), S1-C1-Cl1 119.0(2).

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PROBLEM: The value of sine(theta_max)/wavelength is less than 0.575

RESPONSE: The crystals were very weakly diffracting and the overall

quality of the data is poor due to crystal quality.

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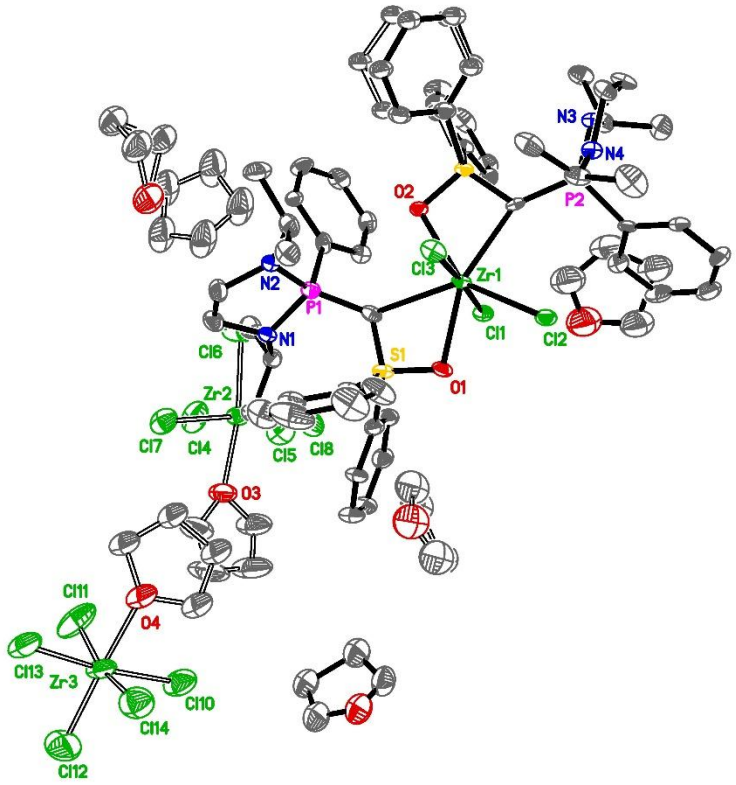

Figure S22: Molecular structure of **9**. Thermal ellipsoids represent 30 % probability. H atoms were omitted for clarity. Selected bond lengths [Å] and angles [°]: P1-C1 1.689(15), P2-C28 1.726(13), S1-C1 1.619(14), S2-C28 1.602(12), Zr1-C1 2.438(13), Zr1-O1 2.295(9), Zr1-C28 2.383(12), Zr1-O2 2.244(8), Zr1-Cl1 2.491(3), Zr1-Cl2 2.446(4), Zr1-Cl3 2.453(4), S1-C1-P1 126.4(8), S2-C28-P2 124.2(7) S1-C1-Zr1 91.6(6), S2-C28-Zr1 92.3(5), P1-C1-Zr1 140.2(7), P2-C28-Zr1 141.4(6).

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PROBLEM: Low Bond Precision on C-C Bonds ..... 0.02363 Ang.

RESPONSE: This alert is generated because of the large amount of disorder in the structure and the relatively weak diffraction of the crystal.

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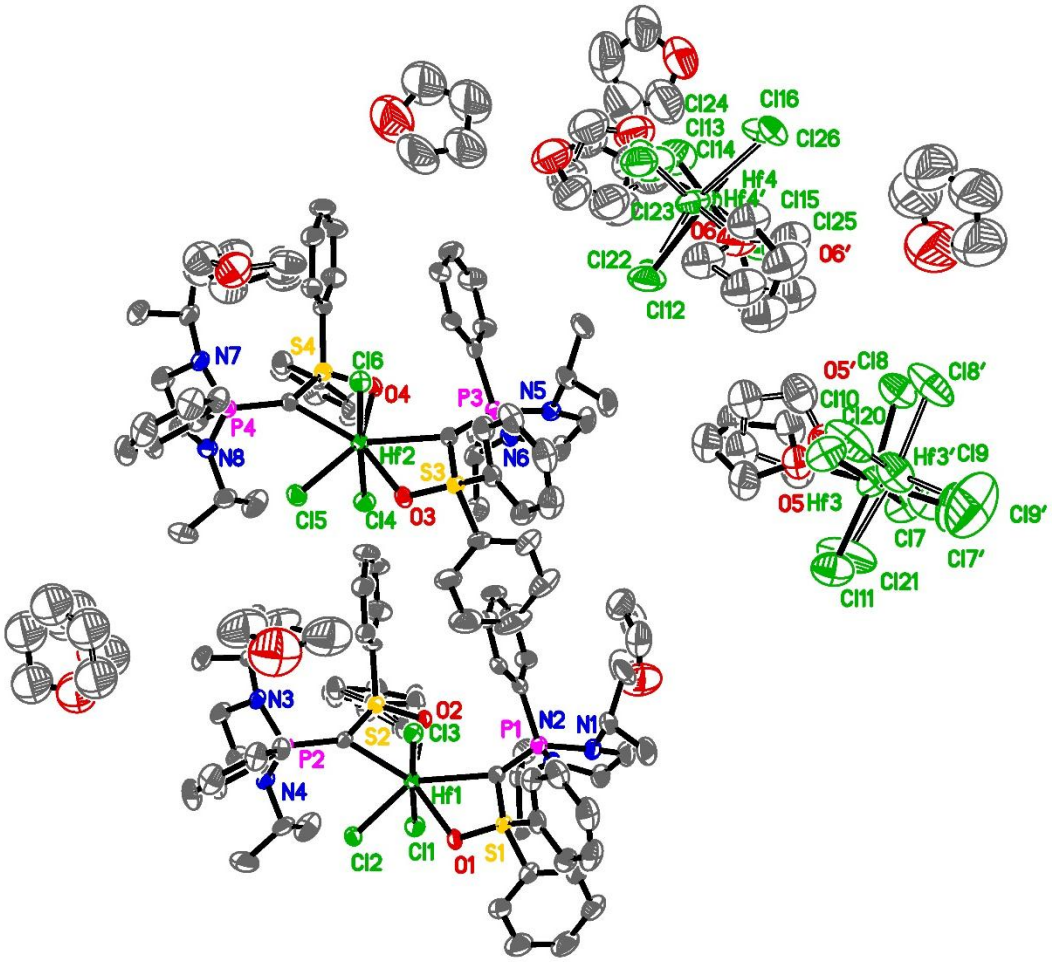

Figure S23: Molecular structure of **11**. Thermal ellipsoids represent 30 % probability. H atoms were omitted for clarity. Selected bond lengths [Å] and angles [°]: P1-C1 1.708(7), P2-C28 1.717(7), S1-C1 1.610(7), S2-C28 1.606(7), Hf1-C1 2.415(7), Hf1-O1 2.282(5), Hf1-C28 2.392(7), Hf1-O2 2.214(5), Hf1-Cl1 2.440(2), Hf1-Cl2 2.437(2), Hf1-Cl3 2.471(2), S1-C1-P1 125.1(4), S2-C28-P2 125.7(4) S1-C1-Hf1 92.2(3), S2-C28-Hf1 91.4(3), P1-C1-Hf1 140.5(4), P2-C28-Hf1 141.0(4).

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CME

Measurement Conditions

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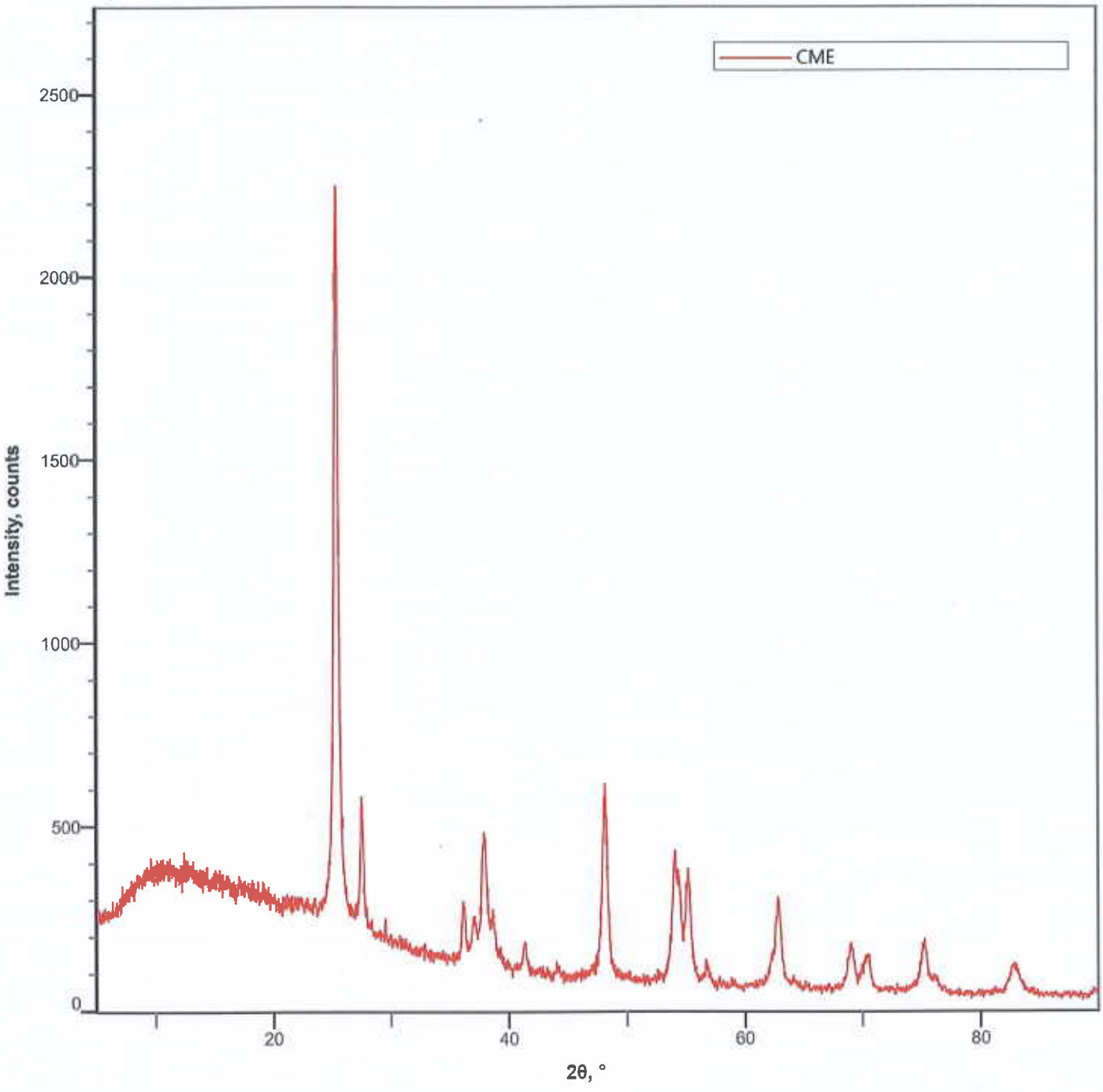
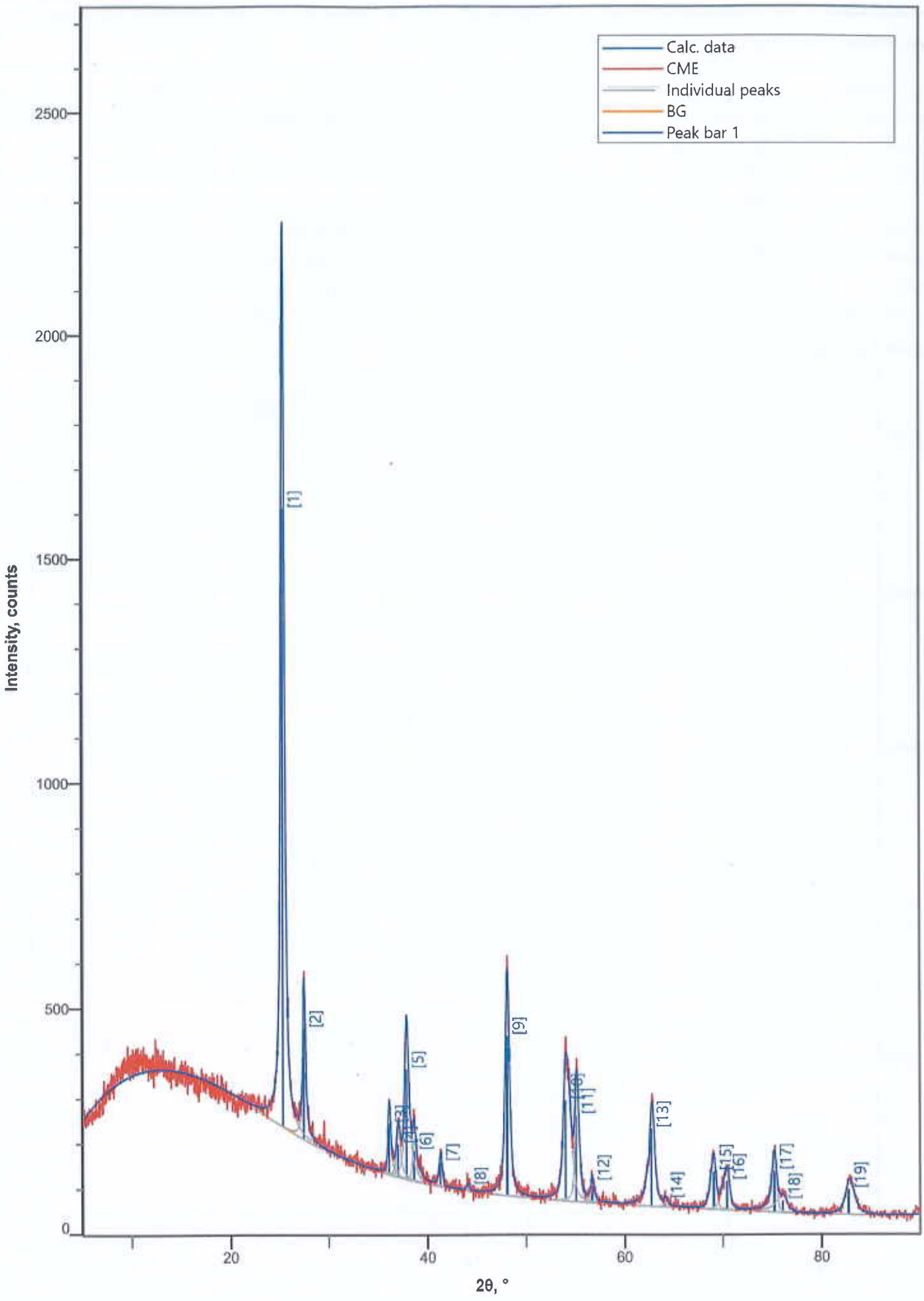
Goniometer	MiniFlex 300/600	Step width	0.02 °
Attachment	ASC-6	Scan axis	θ/2θ
Filter	None	Scan range	5 ~ 90 °
Selection slit	None	Incident slit box	1.25deg
Diffracted beam mono	None	Length-limiting slit	10 mm
Detector	D/teX Ultra2	Receiving slit box #1	Open
Optics attribute	None	Receiving slit box #2	Open

Measured profile view



Peak Profile View



Qualitative Analysis Results

Phase name	Chemical formula	FOM	Phase reg. detail	Space Group	DB Card Number
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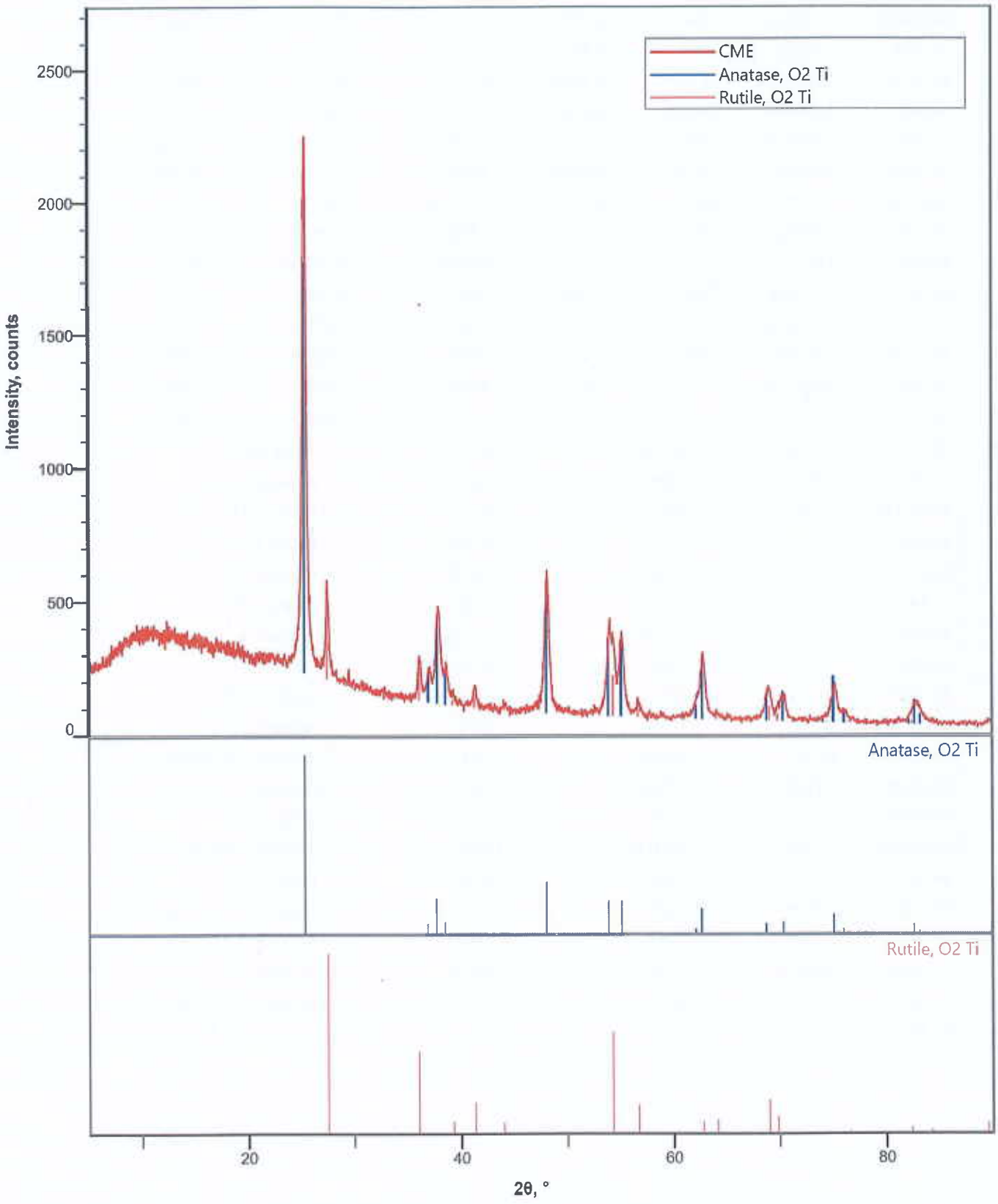
Phase Data View



Peak list

No.	2θ, °	d, Å	Height, cou...	FWHM, °	Int. I., counts°	Int. W., °	Asymmetry
1	25.341(4)	3.5118(6)	1374(29)	0.400(4)	790(4)	0.575(15)	1.08(5)
2	27.453(16)	3.2463(18)	245(10)	0.282(16)	111(2)	0.45(3)	0.8(2)
3	36.090(10)	2.4867(7)	113(7)	0.29(2)	50(2)	0.44(4)	0.85(11)
4	36.990(18)	2.4283(11)	72(5)	0.45(4)	49(4)	0.68(9)	0.85(11)
5	37.845(14)	2.3753(9)	243(11)	0.480(15)	176(6)	0.72(6)	0.85(11)
6	38.610(16)	2.3300(9)	66(4)	0.37(5)	37(5)	0.56(11)	0.85(11)
7	41.26(4)	2.186(2)	53(4)	0.30(5)	21.3(18)	0.40(6)	0.7(5)
8	44.14(3)	2.0501(15)	11.1(10)	0.34(10)	4.0(16)	0.36(17)	2(4)
9	48.082(12)	1.8908(4)	353(15)	0.466(10)	236(2)	0.67(3)	1.17(13)
10	53.999(16)	1.6967(5)	221(11)	0.812(18)	233(5)	1.05(7)	0.56(6)
11	55.056(8)	1.6666(2)	191(10)	0.441(15)	109(3)	0.57(5)	0.56(6)
12	56.670(15)	1.6229(4)	46(4)	0.20(4)	19.0(17)	0.41(7)	1.2(4)
13	62.707(13)	1.4804(3)	171(11)	0.50(2)	147(3)	0.86(7)	0.76(9)
14	64.07(2)	1.4522(5)	16.7(17)	0.19(10)	6.4(15)	0.38(13)	0.6(10)
15	69.03(2)	1.3594(4)	83(6)	0.59(3)	61(2)	0.74(8)	3.0(7)
16	70.39(3)	1.3365(5)	64(5)	0.77(4)	62(2)	0.97(12)	3.0(7)
17	75.15(3)	1.2632(4)	91(7)	0.47(3)	62(9)	0.68(14)	1.8(6)
18	76.00(8)	1.2511(11)	26(3)	1.3(3)	49(9)	1.9(6)	1.8(6)
19	82.70(5)	1.1659(6)	55(5)	0.99(5)	79(2)	1.43(17)	0.8(2)

No.	2θ, °	Decay(ηL/mL)	Decay(ηH/mH)	Size, Å	Phase Name
1	25.341(4)	0.79(3)	0.82(3)	213(2)	Anatase: 1 0 1
2	27.453(16)	1.14(19)	0.98(14)	303(17)	Rutile: 1 1 0
3	36.090(10)	0.72(13)	1.05(11)	296(20)	Rutile: 1 0 1
4	36.990(18)	0.72(13)	1.05(11)	193(18)	Anatase: 1 0 3
5	37.845(14)	0.72(13)	1.05(11)	183(6)	Anatase: 0 0 4
6	38.610(16)	0.72(13)	1.05(11)	235(32)	Anatase: 1 1 2
7	41.26(4)	1.1(6)	0.1(4)	295(45)	Rutile: 1 1 1
8	44.14(3)	0.0(14)	0(2)	264(79)	Rutile: 2 1 0
9	48.082(12)	0.79(6)	0.81(7)	195(4)	Anatase: 2 0 0
10	53.999(16)	0.78(7)	0.41(6)	115(3)	Anatase: 1 0 5,Rutile: 2 1 1
11	55.056(8)	0.78(7)	0.41(6)	212(7)	Anatase: 2 1 1
12	56.670(15)	1.5(3)	1.5(4)	483(92)	Rutile: 2 2 0
13	62.707(13)	1.55(8)	0.71(11)	193(8)	Anatase: 2 0 4,Rutile: 0 0 2
14	64.07(2)	1.5(6)	1.3(6)	506(265)	Rutile: 3 1 0
15	69.03(2)	0.30(9)	0.9(2)	171(8)	Anatase: 1 1 6,Rutile: 3 0 1
16	70.39(3)	0.30(9)	0.9(2)	131(7)	Anatase: 2 2 0,Rutile: 1 1 2
17	75.15(3)	0.95(19)	0.6(3)	223(15)	Anatase: 2 1 5
18	76.00(8)	0.95(19)	0.6(3)	81(19)	Anatase: 3 0 1,Rutile: 2 0 2
19	82.70(5)	0.93(19)	0.69(15)	111(5)	Anatase: 2 2 4,Rutile: 3 2 1

No.	2θ, °	Chemical Formula	Card No	Norm. I.	Profile Type	
1	25.341(4)	O2 Ti	9015929	100.00	Split Pseudo-Voigt	
2	27.453(16)	O2 Ti	9004141	14.09	Split Pseudo-Voigt	
3	36.090(10)	O2 Ti	9004141	6.36	Split Pseudo-Voigt	
4	36.990(18)	O2 Ti	9015929	6.22	Split Pseudo-Voigt	
5	37.845(14)	O2 Ti	9015929	22.25	Split Pseudo-Voigt	
6	38.610(16)	O2 Ti	9015929	4.72	Split Pseudo-Voigt	
7	41.26(4)	O2 Ti	9004141	2.70	Split Pseudo-Voigt	
8	44.14(3)	O2 Ti	9004141	0.51	Split Pseudo-Voigt	
9	48.082(12)	O2 Ti	9015929	29.87	Split Pseudo-Voigt	
10	53.999(16)	O2 Ti,O2 Ti	9015929,90041...	29.49	Split Pseudo-Voigt	
11	55.056(8)	O2 Ti	9015929	13.80	Split Pseudo-Voigt	
12	56.670(15)	O2 Ti	9004141	2.40	Split Pseudo-Voigt	
13	62.707(13)	O2 Ti,O2 Ti	9015929,90041...	18.61	Split Pseudo-Voigt	
14	64.07(2)	O2 Ti	9004141	0.81	Split Pseudo-Voigt	
15	69.03(2)	O2 Ti,O2 Ti	9015929,90041...	7.75	Split Pseudo-Voigt	
16	70.39(3)	O2 Ti,O2 Ti	9015929,90041...	7.83	Split Pseudo-Voigt	
17	75.15(3)	O2 Ti	9015929	7.83	Split Pseudo-Voigt	
18	76.00(8)	O2 Ti,O2 Ti	9015929,90041...	6.19	Split Pseudo-Voigt	
19	82.70(5)	O2 Ti,O2 Ti	9015929,90041...	9.98	Split Pseudo-Voigt	

No.	2θ, °	Distribution R.S.D.	Strain, %	Degree of Orientation	
1	25.341(4)	-	-	-	
2	27.453(16)	-	-	-	
3	36.090(10)	-	-	-	
4	36.990(18)	-	-	-	
5	37.845(14)	-	-	-	
6	38.610(16)	-	-	-	
7	41.26(4)	-	-	-	
8	44.14(3)	-	-	-	
9	48.082(12)	-	-	-	
10	53.999(16)	-	-	-	
11	55.056(8)	-	-	-	
12	56.670(15)	-	-	-	
13	62.707(13)	-	-	-	
14	64.07(2)	-	-	-	
15	69.03(2)	-	-	-	
16	70.39(3)	-	-	-	
17	75.15(3)	-	-	-	
18	76.00(8)	-	-	-	
19	82.70(5)	-	-	-	

No.	2θ, °	Ring Factor	β Cluster	Custom Label
1	25.341(4)	-	-	
2	27.453(16)	-	-	
3	36.090(10)	-	-	
4	36.990(18)	-	-	
5	37.845(14)	-	-	
6	38.610(16)	-	-	
7	41.26(4)	-	-	
8	44.14(3)	-	-	
9	48.082(12)	-	-	
10	53.999(16)	-	-	
11	55.056(8)	-	-	
12	56.670(15)	-	-	
13	62.707(13)	-	-	
14	64.07(2)	-	-	
15	69.03(2)	-	-	
16	70.39(3)	-	-	
17	75.15(3)	-	-	
18	76.00(8)	-	-	
19	82.70(5)	-	-	

Lattice parameters

Phase name	a, Å	b, Å	c, Å	α, °	β, °
Anatase	3.78205	3.78205	9.49162	90.000	90.000
Rutile	4.59236	4.59236	2.95760	90.000	90.000

d-I List

Anatase

No.	2θ, °	d, Å	h k l	Norm. I.	Height, cou...	FWHM, °	Int. I(cal)	Int. I(obs)
1	25.32940	3.51340	1 0 1	100.00	1374(41)	0.4015	1191(36)	1303(5)
2	37.01443	2.42671	1 0 3	6.62	86(3)	0.4253	79(2)	83(5)
3	37.88531	2.37291	0 0 4	20.42	264(8)	0.4285	243(7)	277(8)
4	38.61276	2.32986	1 1 2	7.75	100(3)	0.4314	92(3)	68(8)
5	48.07616	1.89102	2 0 0	28.95	336(10)	0.4805	345(10)	381(3)
6	52.01519	1.75670	2 0 2	0.00	0.0	0.5074	0.00	0.00
7	54.00423	1.69660	1 0 5	18.60	199(6)	0.5224	221(7)	272(4)
8	55.10982	1.66515	2 1 1	18.47	195(6)	0.5311	220(7)	176(4)
9	62.18449	1.49162	2 1 3	3.27	31.0(9)	0.5938	38.9(12)	43.5(5)
10	62.78159	1.47886	2 0 4	14.55	137(4)	0.5996	173(5)	194(2)
11	68.90812	1.36156	1 1 6	6.55	55.7(17)	0.6643	78(2)	61.8(14)
12	70.34934	1.33716	2 2 0	7.21	59.9(18)	0.6808	86(3)	80.7(19)
13	74.24122	1.27639	1 0 7	0.62	4.87(15)	0.7281	7.4(2)	5.94(11)
14	75.17499	1.26284	2 1 5	11.19	86(3)	0.7400	133(4)	106(2)
15	76.10475	1.24971	3 0 1	3.04	23.0(7)	0.7521	36.2(11)	56.2(19)
16	78.81744	1.21336	2 0 6	0.00	0.0	0.7888	0.00	0.00
17	80.96915	1.18645	0 0 8	0.54	3.74(11)	0.8193	6.39(19)	0.00
18	82.25478	1.17113	3 0 3	0.77	5.27(16)	0.8382	9.2(3)	12.9(3)
19	82.78887	1.16493	2 2 4	5.79	39.1(12)	0.8462	69(2)	96.5(19)
20	83.24259	1.15973	3 1 2	2.34	15.7(5)	0.8531	27.9(8)	0.00

Rutile

No.	2θ, °	d, Å	h k l	Norm. I.	Height, cou...	FWHM, °	Int. I(cal)	Int. I(obs)
1	27.44414	3.24729	1 1 0	100.00	246(10)	0.2906	164(6)	177(3)
2	36.09264	2.48655	1 0 1	46.14	96(4)	0.3498	76(3)	71(3)
3	39.20212	2.29618	2 0 0	7.21	14.0(5)	0.3745	11.8(5)	0.00
4	41.25382	2.18660	1 1 1	18.08	33.8(13)	0.3917	29.6(12)	24(2)
5	44.05675	2.05376	2 1 0	6.50	11.5(4)	0.4161	10.7(4)	7(2)
6	54.33911	1.68693	2 1 1	55.97	81(3)	0.5158	92(4)	107.8(14)
7	56.64381	1.62364	2 2 0	16.53	22.9(9)	0.5403	27.1(11)	31.0(15)
8	62.78433	1.47880	0 0 2	7.77	9.7(4)	0.6094	12.7(5)	15.15(18)
9	64.06808	1.45223	3 1 0	8.16	9.9(4)	0.6246	13.4(5)	10.7(11)
10	65.53247	1.42328	2 2 1	0.58	0.68(3)	0.6423	0.94(4)	0.00
11	69.02824	1.35948	3 0 1	19.63	21.9(9)	0.6860	32.2(13)	24.9(6)
12	69.83031	1.34582	1 1 2	9.80	10.8(4)	0.6964	16.1(6)	14.8(4)
13	72.44398	1.30356	3 1 1	1.03	1.09(4)	0.7309	1.70(7)	0.00
14	74.42535	1.27369	3 2 0	0.21	0.217(8)	0.7580	0.350(14)	0.275(5)
15	76.56970	1.24327	2 0 2	2.28	2.24(9)	0.7883	3.74(15)	4.92(17)
16	79.86358	1.20007	2 1 2	1.11	1.03(4)	0.8368	1.83(7)	0.00
17	82.36690	1.16982	3 2 1	4.49	4.00(16)	0.8754	7.4(3)	10.3(2)
18	84.27900	1.14809	4 0 0	2.87	2.48(10)	0.9060	4.71(18)	0.00
19	87.51174	1.11381	4 1 0	0.96	0.79(3)	0.9600	1.58(6)	0.00

Rutile

No.	2θ, °	d, Å	h k l	Norm. I.	Height, cou...	FWHM, °	Int. I(cal)	Int. I(obs)
20	89.58842	1.09330	2 2 2	6.92	5.5(2)	0.9964	11.4(4)	0.00