Application of Quantitative 1H and 19F NMR to Organometallics

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Ayman Akhdar,[a] Jean-Michel Andanson,[a] Sophie Faure,[a] Mounir Traïkia,[a] and Arnaud Gautier[a*]

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Abstract

Purity assessment of organometallics is particularly important for catalytic applications. While quantitative NMR is a well-known method in pharmaceutic chemistry, the present work illustrates its usefulness for the determination of the ligands and organometallics purities using proton and fluorine NMR. This method is fast, straightforward and provides accuracy results.

Keywords.

Purity, NMR, Analytic Chemistry, Imidazolium, Organometallics.

1. Introduction.

The structure determination of ligands and organometallic complexes is usually performed by NMR, infrared (IR), Ultraviolet-Visible (UV-vis) and X-ray spectroscopies. The purity is determined accurately by HPLC for ligands if analytical pure samples of the constituent(s) of a mixture are available. However, this technique cannot be applied routinely to organometallic complexes. Elemental analysis is useful to establish elemental composition and molecular formula. However, it does not provide a direct measurement of the molar purity. Quantitative NMR (qNMR) presents a valuable alternative because the measured signals represent a direct measurement of the composition. [1] Therefore, in presence of an internal reference of known purity, the composition of a synthetic sample can be determined accurately using a single spectrum/ integration sequence (so-called absolute quantification). Equation 1 affords the purity value after accurate weight and NMR measurements.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} \qquad \text{Eq. 1}$$

(where s and std stand for the sample and the standard respectively; I being the sum of integrals, N the total number of nucleus (^{1}H, ^{19}F for example), M the molecular mass, m the measured masses, and P the purities of the standard). This method gives a direct access to the sample purity regardless the content of water or residual solvents, inorganic or organic chemicals, and sorbents, such as silica gel or celite. qNMR, especially using proton measurement is now a technique of choice routinely used in natural product and pharmaceutical chemistry with a reported accuracy of 1-2%. [1a] Several reviews are available, [1b]

reporting the acquisition parameters and this protocol is recently entered into the guideline for authors of the Journal of Medicinal Chemistry.[2] Chemical suppliers are commercializing 'gold standard' which are used as internal or external standards. Expensive 'gold standards' can also be used to determine the purity of a cheaper or more adequate secondary standard, especially if the commercial standard is not compatible with the chemical stability of the organometallic complexes. The purity is of importance for the synthesis of the desired complexes. For example, the starting material could contain residual water, a metathesis could afford a mixture of counter ions or a transmetallation leading to a contamination. In addition, in the case of kinetic studies the determination of the concentrations in stock solutions (reactants and catalyst) should be taken into consideration to perform accurate measurements. In this communication, the application of ^{1}H and ^{19}F qNMR for ligands and organometallics is reported (ESI for NMR parameters and protocols). In brief, after proper weight measurements, choice of the adequate solvent, recording of ^{1}H and ^{19}F spectrum takes 30 to 60 minutes making this technic attractive for routine determination.

2. Results and discussion.

2.1. Choice of the standard.

To make this method straightforward and broadly applicable to a wide variety of ligands and organometallics, the standard should be cheap and inert in presence of the organometallics. This prohibits standards containing some functional groups that could chelate or react such as carbonyls, reducing agents (formic salts), double bonds, acidic or redox compounds (phenol, quinones). We find that 1,4-dimethoxy

benzene (1,4-DMB) is a good secondary standard for ^{1}H qNMR as it is cheap, easily purified by recrystallization from hexane or sublimation [3] and presents a good solubility in all common NMR solvents. Using maleic acid as primary gold standard (99.995% traceable certified reference materials, TraceCERT®) we find a purity of 99.5 % for our recrystallized sample of 1,4-DMB (Karl Fisher analysis demonstrates the absence of residual water). ^{1}H NMR of 1,4-DMB shows two well defined, sharp singlets at 6.7 and 3.7 ppm. Regarding ^{19}F qNMR, we used the commercially available 'gold standard' 2-chloro-4-fluoro toluene that is found inert in presence of the compounds described below. It is also interesting to note that the chemical shift of this standard is well positioned between the signals displayed by BF_4 and PF_6 anions. PF_6 appears as a doublet due to the coupling with phosphorus whereas BF_4 appears as two close signals, a singlet (^{10}B) and a quadruplet (^{11}B, S=3/2, J=1.1Hz) in a ratio 2:8. Chemical shifts (δ) and relaxation times (T1) are collected in Table 1. The relaxation times show that 30 seconds of inter-pulse delay (>5 T1) could be used for ^{19}F qNMR.

Table 1: Chemical shifts (δ) and relaxation times (T1)

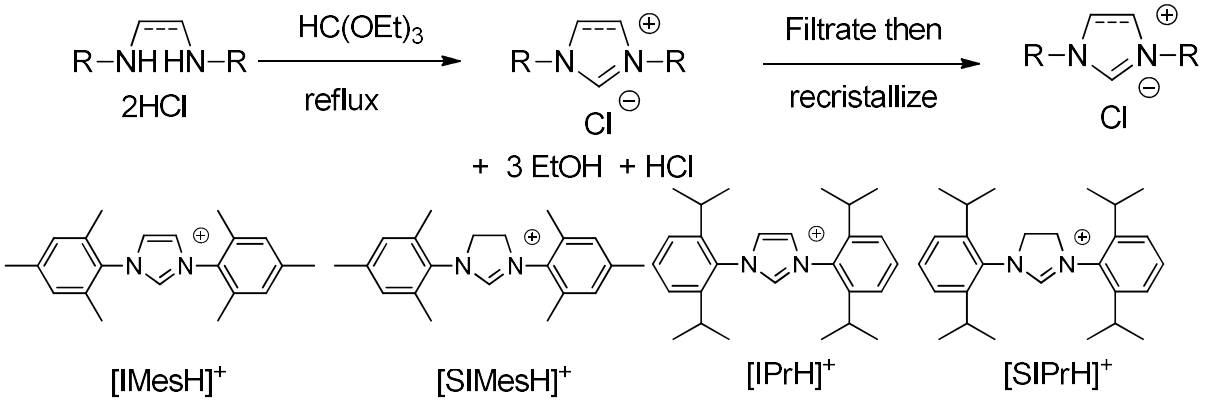
compound	Solvent	δ (ppm)	T1 (s)
2-chloro-4-fluoro Toluene	dmso-d_6	-115.3	3.3[a]
	Acetone-d_6	-117.0	6.5[b]
	MeOH-d_4	-117.7	4.8[a]
	MeCN-d_3	-117.3	4.7[a]
	$CDCl_3$	-115.8	4.4[a]
BF_4^-	dmso-d_6	-148.3	^{10}B: 6.8 ^{11}B: 5.9[c]
	Acetone-d_6	-151.8	^{10}B: 5.5 ^{11}B: 5.2[c]
	MeOH-d_4	-154.8	^{10}B: 5.4 ^{11}B: 4.9[c]
	MeCN-d_3	-151.8	^{10}B: 6.2 ^{11}B: 5.5[c]
	$CDCl_3$	-151.9	^{10}B: 2.9 ^{11}B: 2.8[d]
PF_6^-	dmso-d_6	-70.0	3.8[e]
	Acetone-d_6	-72.6	3.4[e]
	MeOH-d_4	-74.7	3.1[e]

	$MeCN\text{-}d_3$	**-72.8**	**3.5[e]**
	$CDCl_3$	**-72.3**	**2.0[f]**

[a]: taken from Aldrich, TraceCERT® CRMs for quantitative NMR. [b]: this work. [c]: using SIPr.HBF_4. [d]: using $N(Pr)_4$, BF_4. [e]: using SIPr.HPF_6. [f]: using $N(Bu)_4$, PF_6.

2.2.Ligands.

As illustration of this methodology due to their actual increasing interest in catalysis, we decided to focus on imidazolium and imidazolinium that are used for *N*-heterocyclic carbenes (NHCs) and metal-NHCs synthesis.[4] In his seminal communications, Arduengo reports a convenient synthesis of these azoliums that consists of refluxing diamines in triethyl orthoformate (Scheme 1).[4b] After filtration, salt/triethyl orthoformate adduct are obtained and simple purification can be achieved by repeated recrystallizations from acetonitrile/ether.



Scheme 1: Imidazolium and imidazolinium synthesis.

At first, we followed the Arduengo's procedure for SIMes.HCl and find, using 1,4-DMB, a purity of 72% for the crude product after filtration (Table 2, entry 1). Simple recrystallization [4b] and determination of the

water content by Karl-Fisher analysis (0.5 molecule of water, Table 2, entry 2) affords a purity of 91%. For the azolium series, the amount of water varies from 0 to 1 molecule. This first screening of classical imidazolium and imidazolinium highlights the importance of the purity determination that should be corroborated with Karl-Fisher analysis results.

Table 2: purity of azolium chlorides.

Entries[a]	**Azolium**	**Purity[b]**
1	**SIMes.HCl[c]**	**72%**
2	**SIMes.HCl, 0.5H_2O[d]**	**91% (±2%)**
3	**IMes.HCl, 1H_2O**	**98% (± 1%)**
4	**SIPr.HCl, 0H_2O**	**91% (± 2%)**
5	**IPr.HCl, 0.5H_2O**	**96% (± 2%)**

(a): in dmso-d6. (b): uncertainty from 3 experiments. (c): before recrystallization. (d): after recrystallization.

The same procedure was followed for the non-coordinating anions PF_6 and BF_4, commonly used for metal carbene complexes. Importantly, both ^{1}H qNMR and ^{19}F qNMR should give comparable results. We simply synthesize the PF_6 and BF_4 salts by anion metathesis from the corresponding azolium chloride as previously described (Table 3). [5] We obtained high yields and in contrast to the chloride series, Karl-Fisher analysis demonstrates the absence of residual water (less than 0.1%). The results show a good correspondence between both technics with a maximum deviation of 3% (entries 6) and an averaged deviation of 1.8%. In all cases, good purity was observed.

Table3: Purity of azolium with non-coordinating salts.

Entry[(a)]	Azolium	Yield	Purity by ^{1}H qNMR[(b)]	Purity by ^{19}F qNMR[(b)]
1	SIMes.HPF$_6$	90%	98% (± 2%)	97% (± 1%)
2	IMes.HPF$_6$	91%	99% (± 3%)	99% (± 1%)
3	SIPr.HPF$_6$	76%	96% (± 2%)	97% (± 2%)
4	IPr.HPF$_6$	86%	95% (± 2%)	96% (± 1%)
5	SIMes.HBF$_4$	89%	98% (± 2%)	99% (± 1%)
6	IMes. HBF$_4$	90%	94% (± 2%)	97% (± 1%)
7	SIPr. HBF$_4$	78%	95% (± 2%)	97% (± 1%)
8	IPr. HBF$_4$	91%	93% (± 2%)	95% (± 1%)

(a): in dmso-d$_6$. (b) Uncertainty from 2 experiments.

2.3.Metal-NHCs.

The purity of a selection of useful metal-NHCs was determined. We used well-established literature procedures to synthetize silver, copper, gold and palladium complexes. Indeed, Ag(I)-NHCs and Cu(I)-NHCs are important synthons for transmetallation. [6] Silver(I)-NHCs is used in A3 coupling reactions and oxazole synthesis, copper(I)-NHCs are used in CuAAC, reduction, conjugated additions, allylic substitutions, alkene and alkyne transformations, nitrene transfert, allene synthesis, hydrocarboxylation, A3 coupling, oxidative coupling of phenol. [7] Regarding gold(I)-NHCs, they are used in a wide range of catalysis including enyne cycloisomerization, alkyne hydration, C-C coupling reactions; [8] and palladium(II)-NHCs has gained high importance in homogeneous cross-coupling. [9]

IMesAgCl, SIMesAgCl, IPrAgCl and SIPrAgCl were synthesized by the classical silver oxide route [4a], [10] and recrystallized from dichloromethane/pentane affording good purities (Table 4, entries 1-4) in $CDCl_3$. At this stage, we noticed that a decomposition of the complex in presence of the maleic acid standard takes place after few hours; therefore 1,4-DMB as secondary standard was selected. The copper

complexes SIMesCuCl and IPrCuCl (entry 5-6) were obtained by a soft, direct metalation of the azolium using the ammonia protocol. [11] Both complexes are obtained with good purity, 93 and 98% respectively. IPrAuCl was synthetized by transmetallation from IPrAgCl affording 93% purity (entry 7). The cationic complex [IPrAu(MeCN)], BF_4 was obtained from Strem chemical (95%) and analyzed by ^{1}H and ^{19}F qNMR to give an average purity of 95% in perfect agreement with the supplier's specifications (entry 8). Pd(PEPPSI)IPr was synthetized from the well-established procedure from the Organ's group.[12a] We find small amounts of indefinite materials in the crude mixture that were removed by washing with dichloromethane/pentane (1:5). The NMR spectrum shows the presence of 0.8 molecules of CH_2Cl_2 that cannot be removed under reduced pressure (2mbar) for several hours. [12b] The Pd(PEPPSI)IPr's purity in this mixture is found to be 87% and, taking the dichloromethane amount into account, a 96% purity is obtained (entry 9).

Table 4: Purity of metal-NHCs.

Entries	M-NHC	Solvent	Purity
1	IMesAgCl	$CDCl_3$	97% (± 1%)
2	SIMesAgCl	$CDCl_3$	95% (± 2%)
3	IPrAgCl	$CDCl_3$	97% (± 1%)
4	SIPrAgCl	$CDCl_3$	98% (± 1%)
5	SIMesCuCl	$CDCl_3$	93% (± 2%)
6	IPrCuCl	$CDCl_3$	98% (± 1%)
7	IPrAuCl	dmso-d_6	93% (± 1%)
8	[IPrAu,MeCN], BF_4	dmso-d_6	94% (± 1%) [b] 96% (± 1%) [c]
9	Pd(PEPPSI)IPr,[d] 0.8CH_2Cl_2	$CDCl_3$	96% (± 2%)

(a): average of two measurements. (b): by ^{1}H qNMR. (c): by ^{19}F qNMR. (d): Pd(PEPPSI)IPr: Dichloro[1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene)](3-chloropyridyl)palladium(II).

3. Conclusion

In conclusion, we have applied ^{1}H and ^{19}F quantitative NMR to organometallics complexes and their corresponding ligands. According to previous reports, this technique is easy to conduct, affording purity of compounds in approximately 30 minutes to 1h experiment. Care should be taken regarding the choice of the solvent and if possible, Karl Fisher analysis should be considered, especially regarding the ligands.

4. Acknowledgment

Financial support from the Centre National de la Recherche Scientifique (CNRS) and the Université Clermont-Auvergne are acknowledged.

Appendix.

Supplementary material: procedure, NMR sequence and spectra can be found, in the online version, at ...

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S1. Generals.

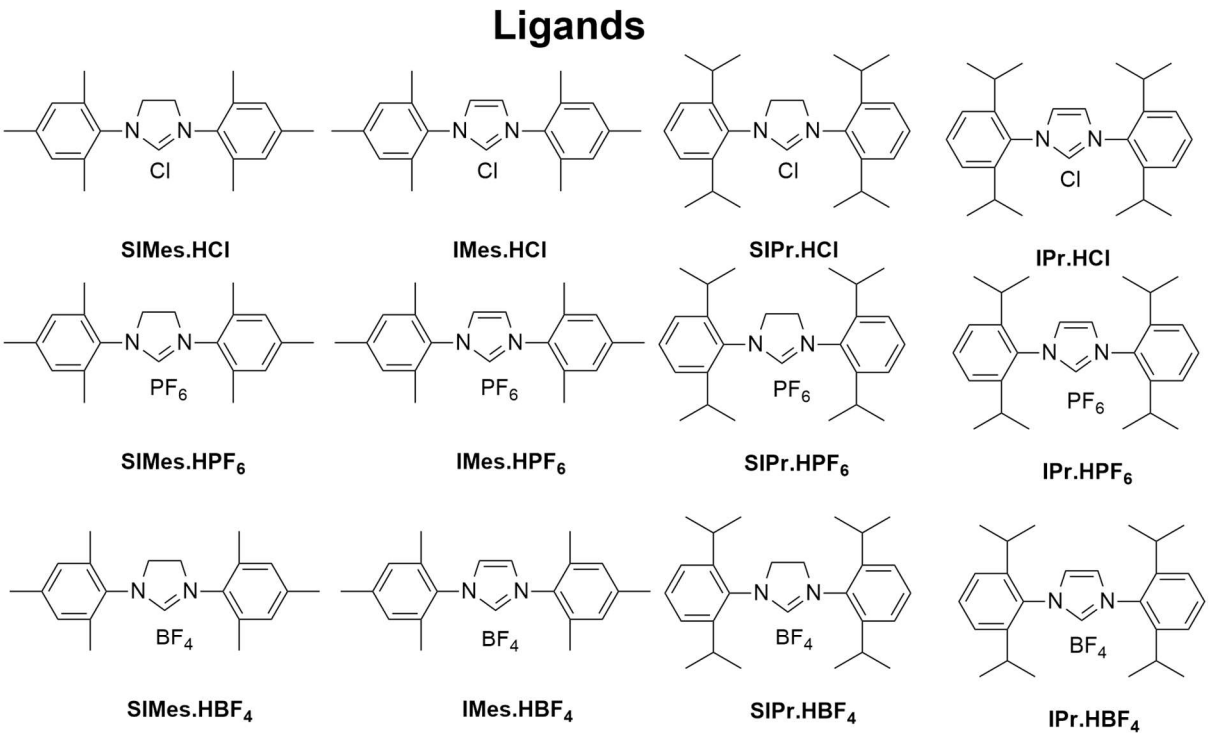
All ligands and complexes were analyzed by ^{1}H and $\{1H\}^{19}F$ NMR spectroscopy on a Bruker advance 400 spectrometer. $CDCl_3$ was neutralized over anhydrous K_2CO_3 prior to use. The water content was measured using a coulometric Karl Fischer titrator (MettlerToledo, DL32).

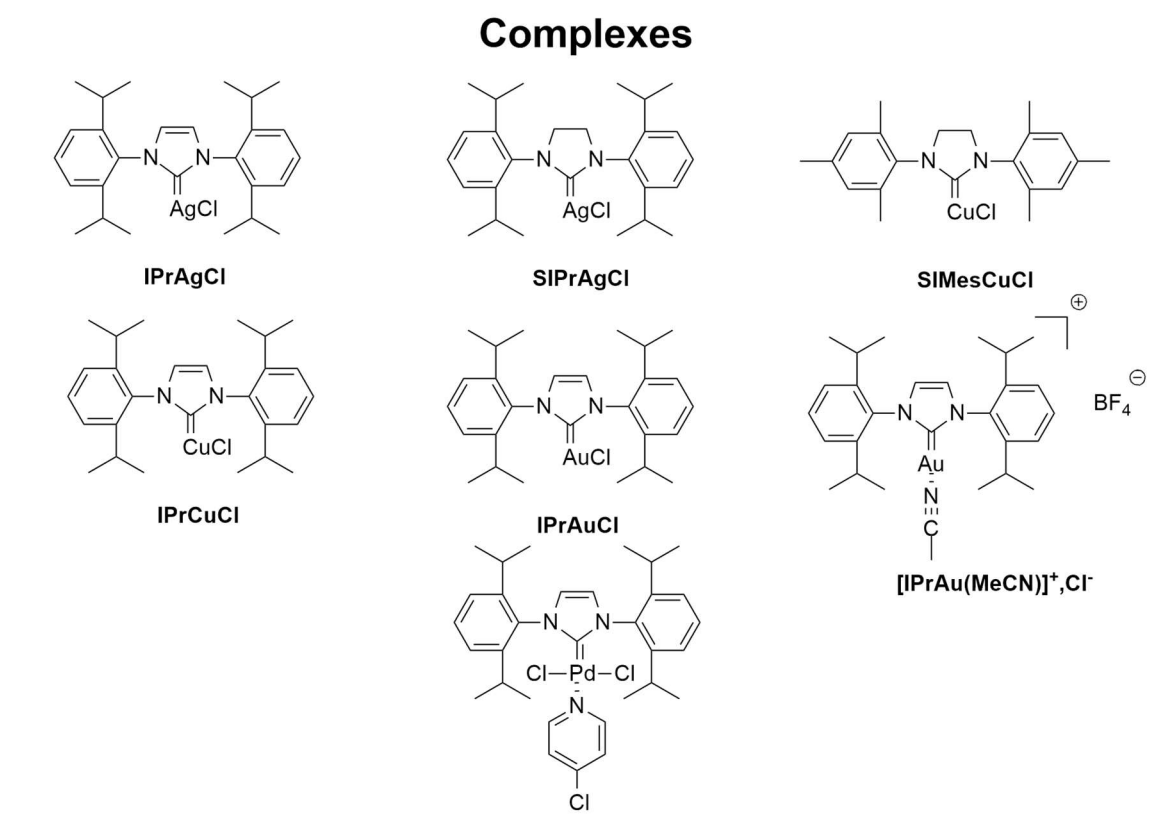
Sample Preparation: The sample and the standard are weighed using a Mettler Toledo balance (0.01 mg accuracy) into a single Eppendorf, then solvent was added and the recipient was gently shaken to ensure a total dissolution. The solution was transferred into a 3 mm standard NMR tube for analysis.

Pulse Program: Sample Temperature: 19 °C (regulated ± 0.1 K), single pulse, without carbon decoupling ('zg' with 90° pulse). Data Points (acquired): 64 K. NS=64. Relaxation delay: D1=60s. Acquisition Time: 4s. Spectral window for proton: SW=30 ppm and O1: 7.5 ppm; for fluorine, PF_6: SW=120 ppm and O_1=90 ppm, BF_4: SW=-130 ppm and O1=100 ppm.

Post-Acquisition Processing was performed with ACDLABS software (1.2, academic version). Zero Filling: to 256K. Line Broadening: LB = 0.1 Hz. Phasing: manually. Baseline Correction, for ^{1}H NMR: 6^{th} order polynomial. For each signal measured a ratio signal/noise>100 is verified.

S2. Structures

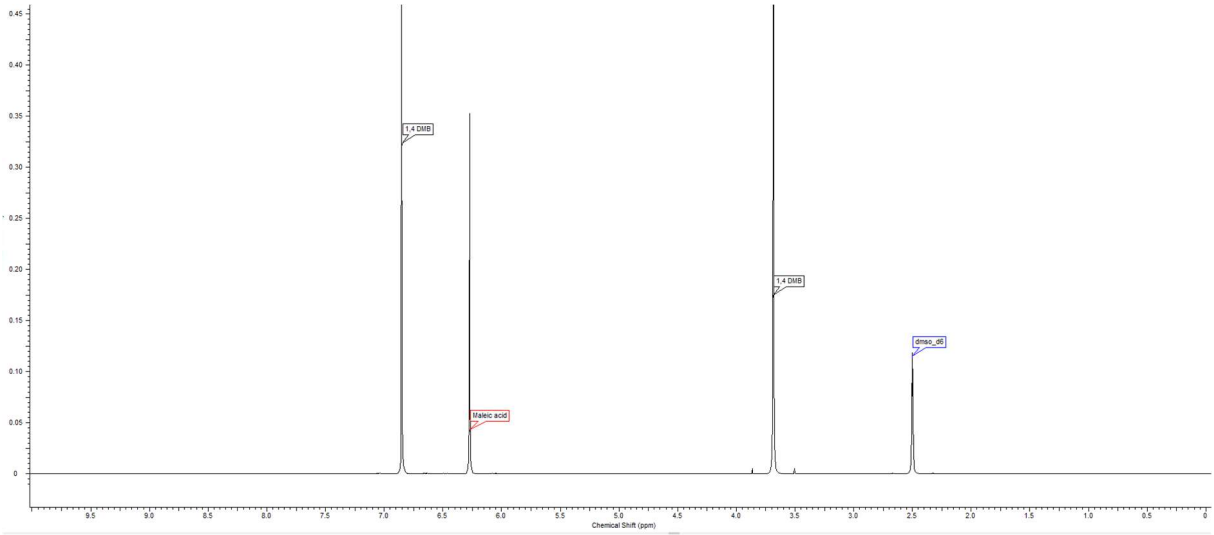




S3. Determination of the purity of 1,4 DMB standard.

Purity was determined as the average of 6 independent measurements in dmso_d_6 from maleic acid gold standard trace certified (Aldrich) as primary standard.

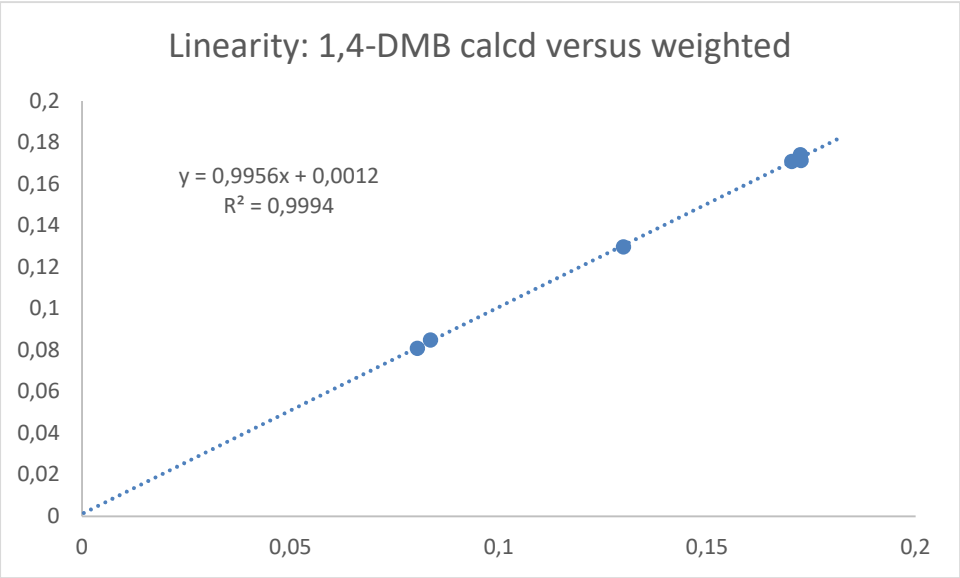
Example of spectrum



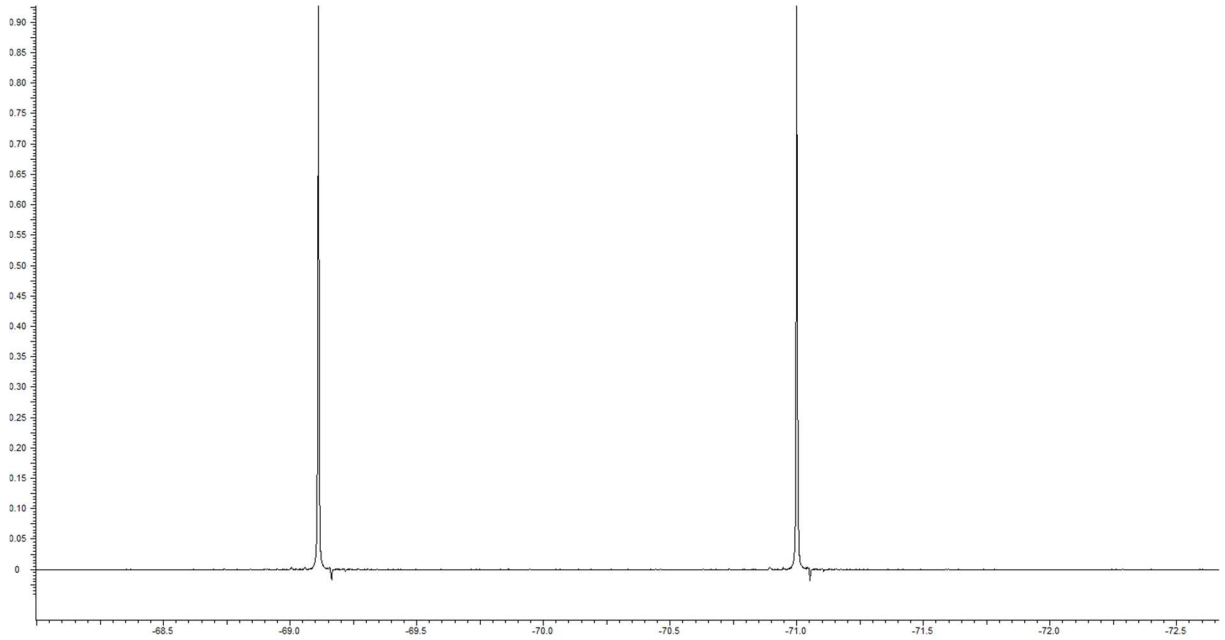
Purity is calculated as the ratio of the number of mol of 1,4-DMB determined by qNMR divided by the number of mol of 1,4-DMB weighted, taking into account of the primary standard purity (99.98 +/- 0.13 %).

Exp	1	2	3	4	5	6
Purity	99.32	98.39	98.91	99.56	100.00	100.04
Average	99.48 %					
Standard deviation	+/- 0.06%					

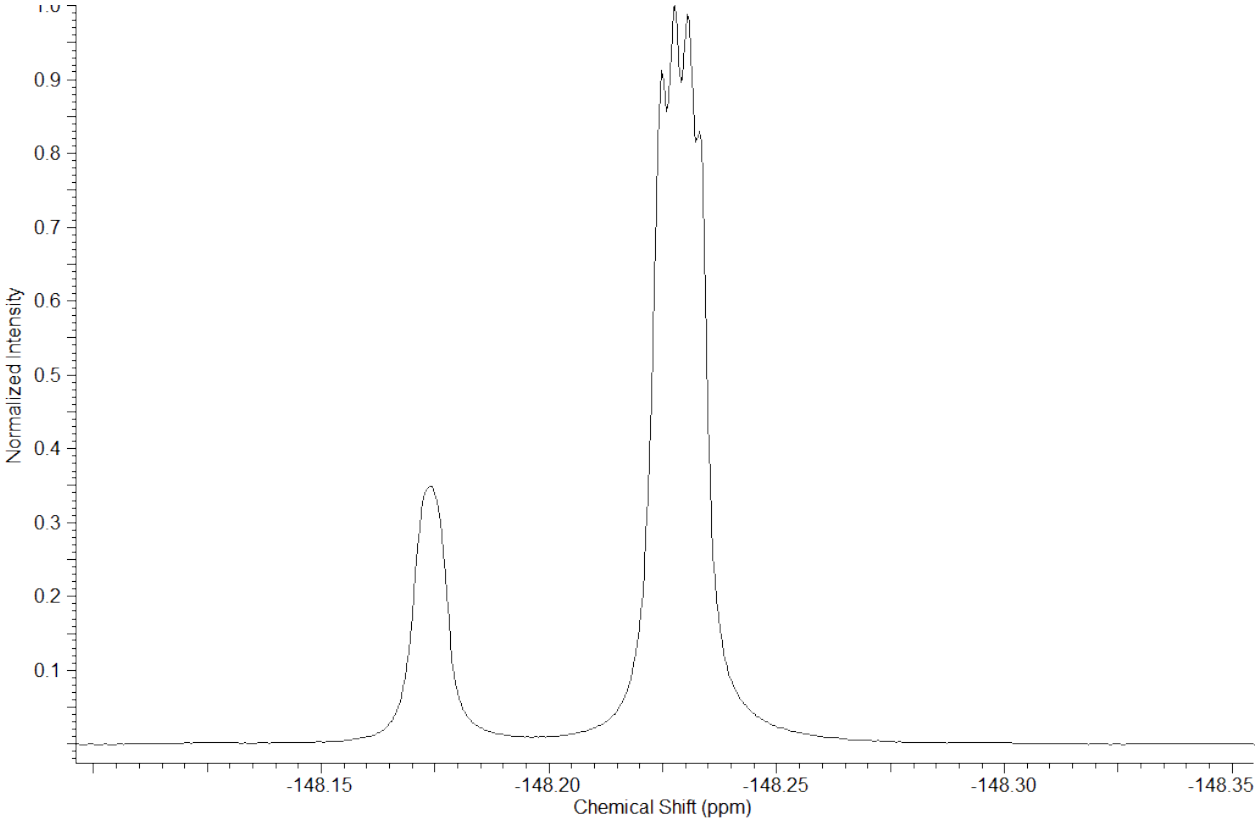
The linearity of the response in the range 10 to 25 mg (0.08 to 0.17 mmol) was verified:


Linearity: 1,4-DMB calcd versus weighted
y = 0,9956x + 0,0012
R² = 0,9994
0,2
0,18
0,16
0,14
0,12
0,1
0,08
0,06
0,04
0,02
0
0
0,05
0,1
0,15
0,2

S4. ^{19}F NMR spectra of SIPr.HPF_6

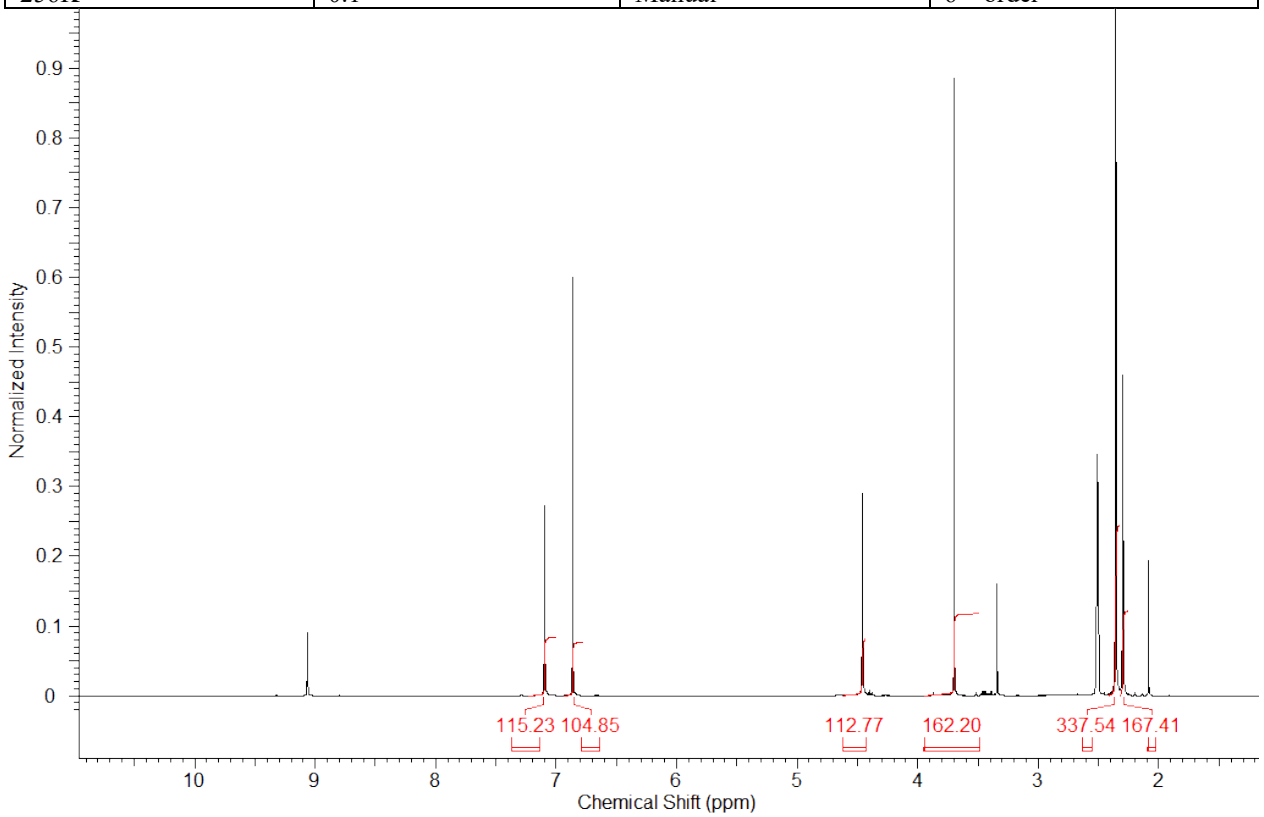


S5. ^{19}F NMR spectra of SIPr.HBF$_4$



S6. SIMes.HCl 0.5H_2O Verifié!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
36.61	12.52	

Signals standard (ppm)	6.86	3.7		
Integration	104.8	162.2		
Signals sample (ppm)	7.1	4.45	2.35	2.3
Integration	115.2	112.7	337.5	167.4

Somme of the integral: sample, 732.8; standard, 267.

Number of protons: sample, 26; standard, 10.

Molecular weight: sample, 351.9; standard, 138.2.

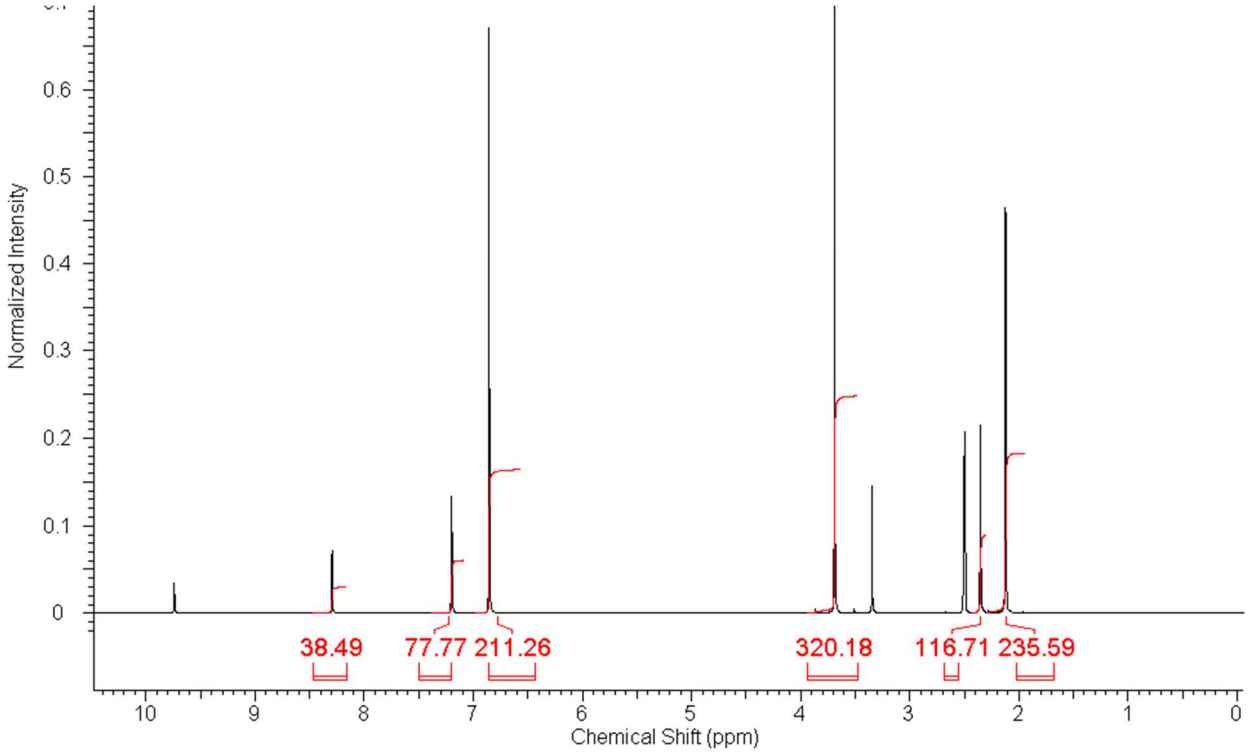
Mass of: sample, 36.61; standard: 12.52.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{732.8}{267} . \frac{10}{26} . \frac{351.9}{138.2} . \frac{12.52}{36.61} . 0.995 \ = 0.914$$

Purity: 91%

S7. IMes.HCl, 1H_2O ^1H qNMR Verifié!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
13.48	13.96	

Signals standard (ppm)	6.86	3.7		
Integration	211.3	320.2		
Signals sample (ppm)	8.3	7.2	2.4	2.1
Integration	38.5	77.7	116.7	235.6

Somme of the integral: sample, 468.5; standard, 531.5.

Number of protons: sample, 24; standard, 10.

Molecular weight: sample, 358.9; standard, 138.2.

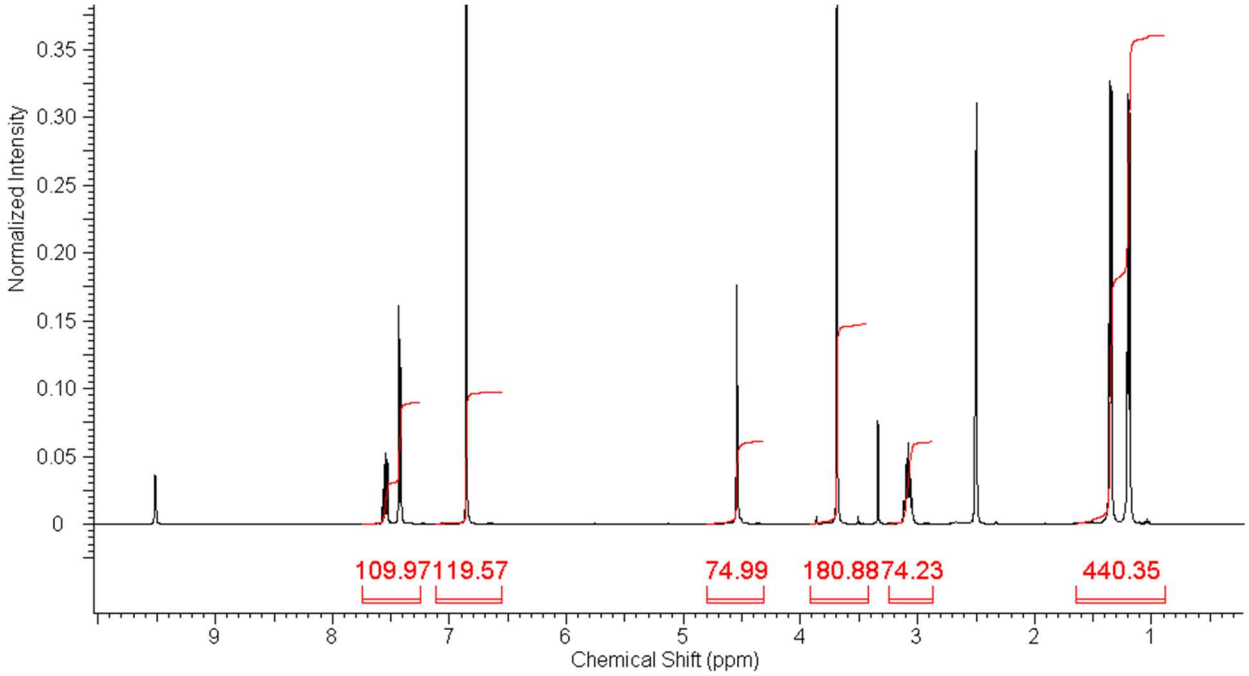
Mass of: sample, 13.48; standard: 13.96.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{468.5}{531.5} . \frac{10}{24} . \frac{358.9}{138.2} . \frac{13.96}{13.48} . 0.995 = 0.983$$

Purity: 98%

S8. SIPr.HCl, 0H_2O ^1H qNMR Verifié!!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
31.98	15.53	

Signals standard (ppm)	6.86	3.7		
Integration	119.6	180.9		
Signals sample (ppm)	7.5	4.5	3.1	2.3
Integration	110	75	74.2	440.4

Somme of the integral: sample, 699.6; standard, 300.5.

Number of protons: sample, 40; standard, 10.

Molecular weight: sample, 445.1; standard, 138.2.

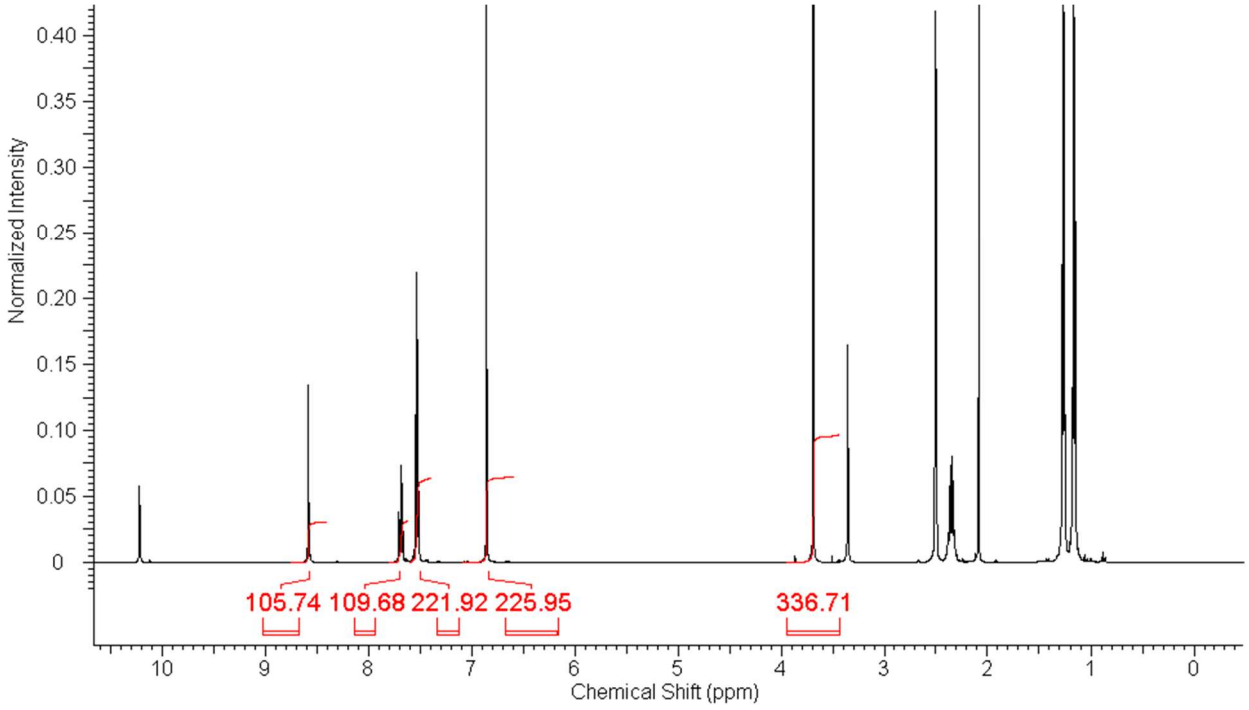
Mass of: sample, 31.98; standard: 15.53.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{699.6}{300.5} . \frac{10}{40} . \frac{445.1}{138.2} . \frac{15.53}{31.98} . 0.995 = 0.906$$

Purity: 91%

S9. IPr.HCl, 0.5H_2O ^1H qNMR Verifié!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
31.75	11.27	

Signals standard (ppm)	6.86	3.7	
Integration	226	336.7	
Signals sample (ppm)	8.6	7.7	3.7
Integration	105.7	109.7	221.9

Somme of the integral: sample, 437.3; standard, 562.7.

Number of protons: sample, 8; standard, 10.

Molecular weight: sample, 434.1; standard, 138.2.

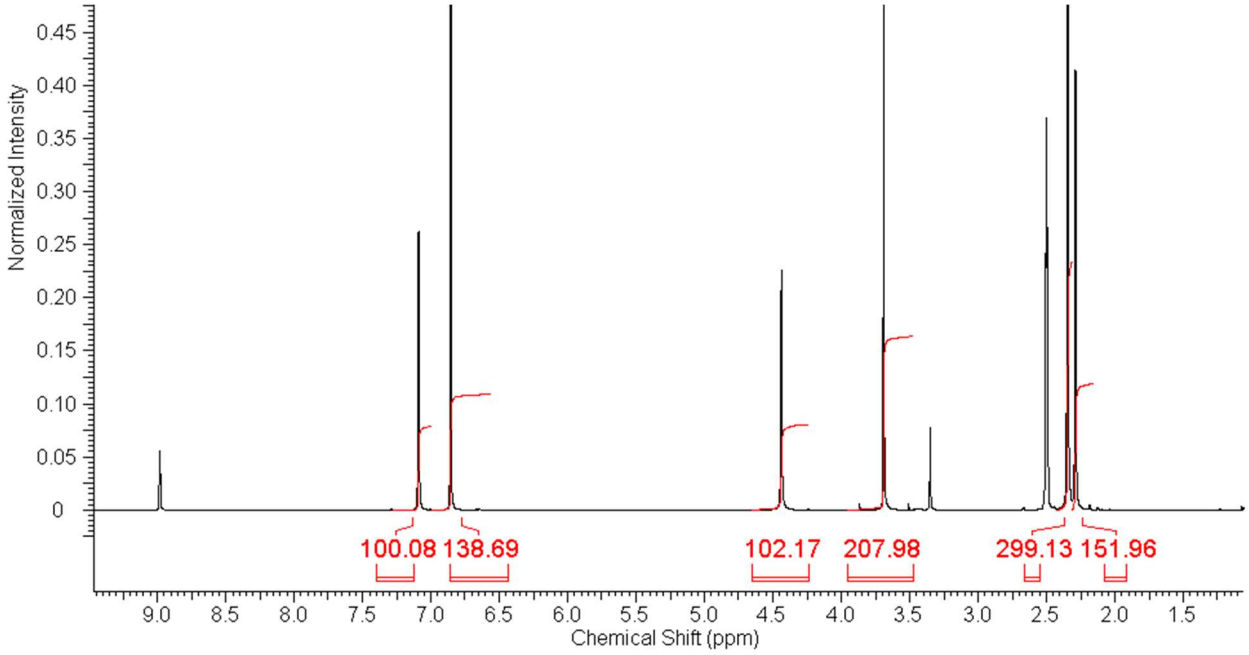
Mass of: sample, 31.75; standard: 11.27.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{552.1}{562.7} . \frac{10}{8} . \frac{434.1}{138.2} . \frac{11.27}{31.75} . 0.995 = 0.96$$

Purity: 96%

S10. SIMes.HPF$_6$ ^{1}H qNMR Verifié !!!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
31.00	12.98	

Signals standard (ppm)	6.86	3.7		
Integration	138.7	208		
Signals sample (ppm)	7.1	4.43	2.34	2.29
Integration	100.1	102.7	299.1	152

Somme of the integral: sample, 653.9; standard, 346.7.

Number of protons: sample, 26; standard, 10.

Molecular weight: sample, 452.4; standard, 138.2.

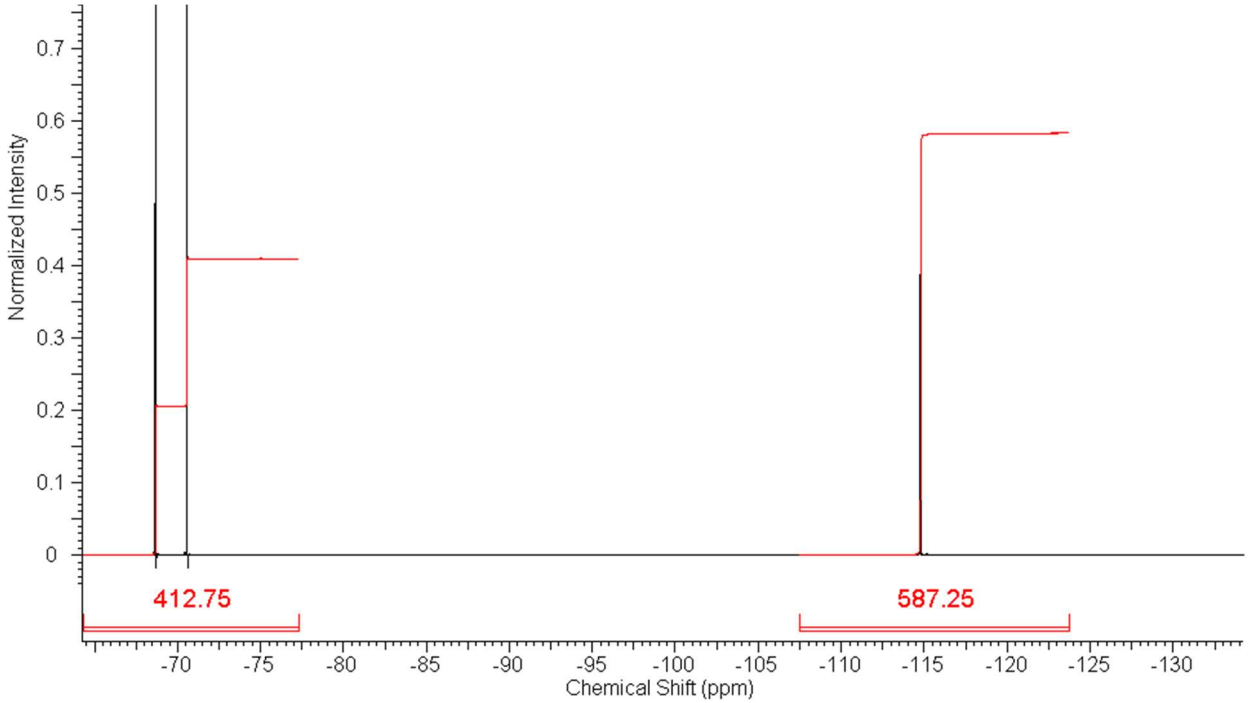
Mass of: sample, 30.00; standard: 12.98.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{653.9}{346.7} . \frac{10}{26} . \frac{452.4}{138.2} . \frac{12.98}{31} . 0.995 = 0.98$$

Purity: 98%

S11. SIMes.HPF$_6$ ^{19}F qNMR Vérifié

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
10.64	28.14	

Signals standard (ppm)	-115
Integration	587.3
Signals sample (ppm)	-70
Integration	412.8

Somme of the integral: sample, 412.8; standard, 587.3.

Number of fluorine: sample, 6; standard, 1.

Molecular weight: sample, 453.4; standard, 144.6.

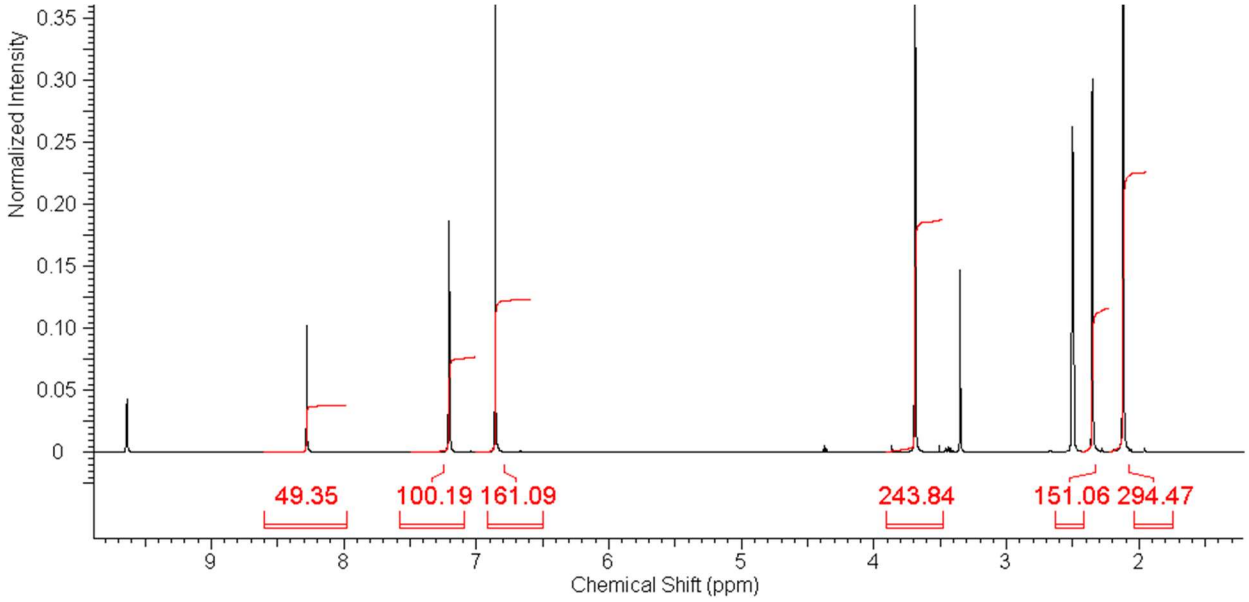
Mass of: sample, 10.64; standard: 28.4.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{412.8}{587.3} . \frac{1}{6} . \frac{453.4}{144.6} . \frac{28.4}{10.64} . 0.996 = 0.96$$

Purity: 96%

S12. IMes.HPF$_6$ ^{1}H qNMR verifié !!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
31.51	15.25	

Signals standard (ppm)	6.86	3.7		
Integration	161.1	243.8		
Signals sample (ppm)	8.28	7.2	2.34	2.12
Integration	49.35	100.2	151.1	294.5

Somme of the integral: sample, 595.2; standard, 404.9.

Number of protons: sample, 24; standard, 10.

Molecular weight: sample, 450.4; standard, 138.2.

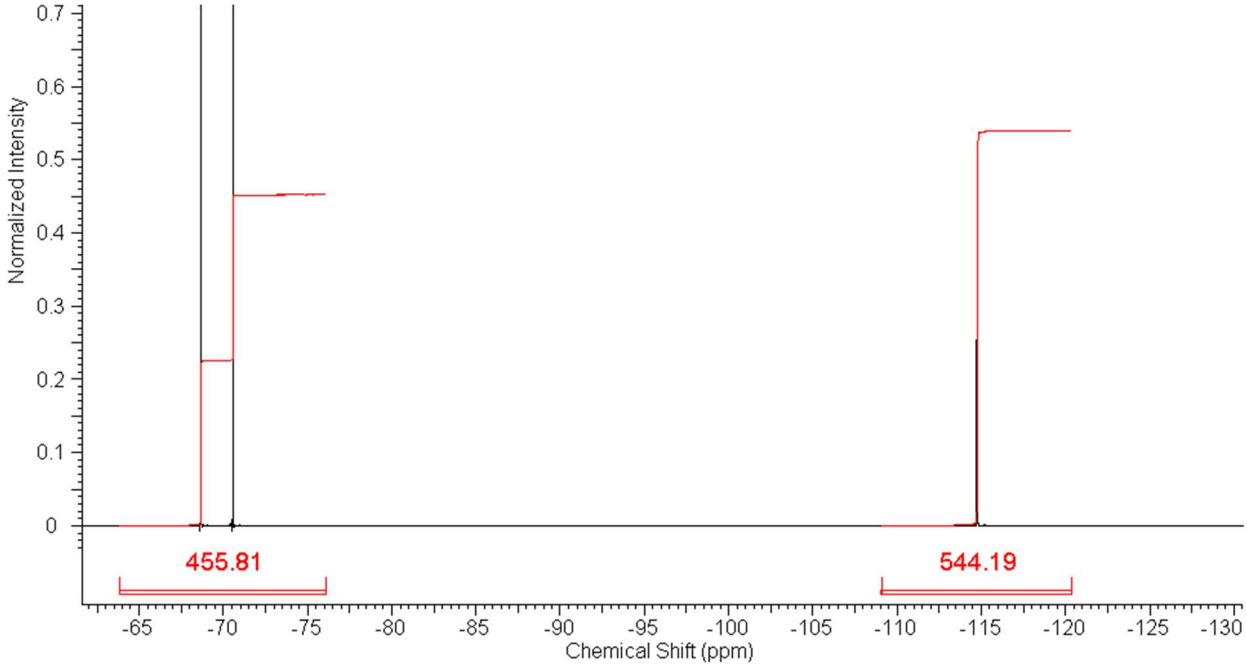
Mass of: sample, 31.51; standard: 15.25.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{595.2}{404.9} . \frac{10}{24} . \frac{450.4}{138.2} . \frac{15.25}{31.51} . 0.995 \ = 0.96$$

Purity: 96%

S13. IMes.HPF$_6$ ^{19}F qNMR Verifié!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
11.27	25.82	

Signals standard (ppm)	-115
Integration	544.2
Signals sample (ppm)	-70
Integration	455.8

Somme of the integral: sample, 455.8; standard, 544.2.

Number of fluorine: sample, 6; standard, 1.

Molecular weight: sample, 450.4; standard, 144.6.

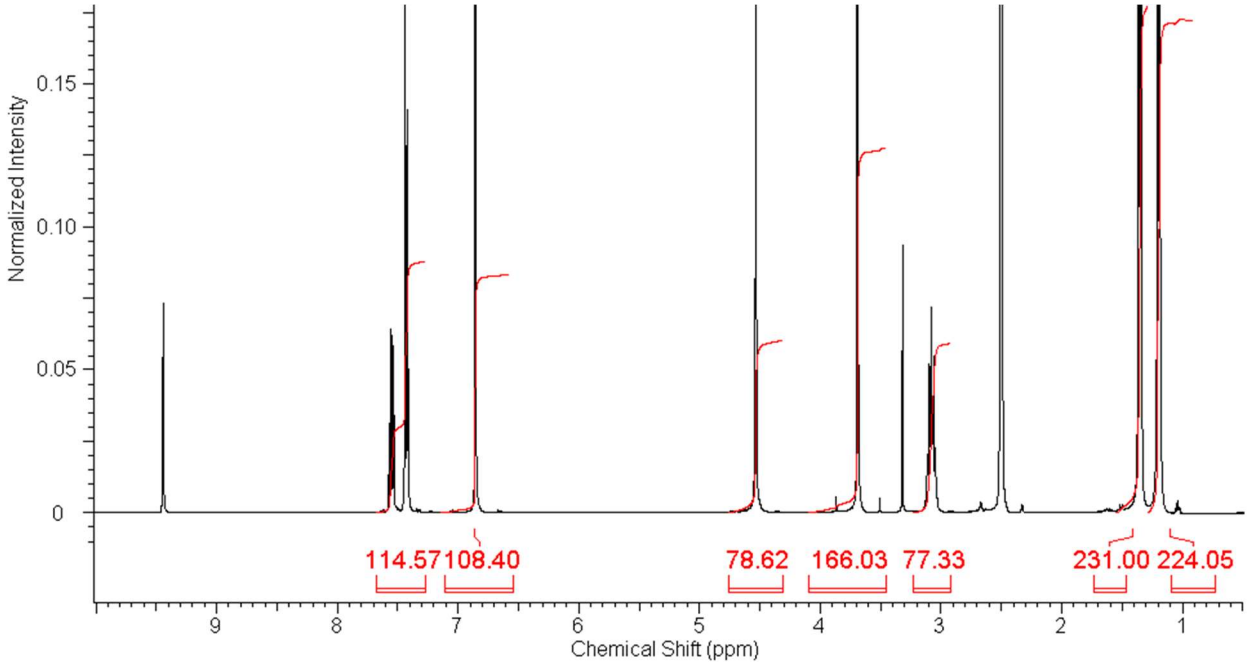
Mass of: sample, 11.27; standard: 25.82.

$$P_s = \frac{I_s}{I_{std}} \cdot \frac{N_{std}}{N_s} \cdot \frac{M_s}{M_{std}} \cdot \frac{m_{std}}{m_s} \cdot P_{std} = \frac{455.8}{544.2} \cdot \frac{1}{6} \cdot \frac{450.4}{144.6} \cdot \frac{25.82}{11.27} \cdot 0.996 = 0.99$$

Purity: 99%

S14. SIPr.HPF$_6$ ^{1}H qNMR Vérifié !!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
32.09	11.51	

Signals standard (ppm)	6.86	3.7			
Integration	108.4	166			
Signals sample (ppm)	7.49	4.54	3.1	11.35	1.19
Integration	114.6	78.6	77.3	231	224.1

Somme of the integral: sample, 725.6; standard, 274.4.

Number of protons: sample, 38; standard, 10.

Molecular weight: sample, 536.6; standard, 138.2.

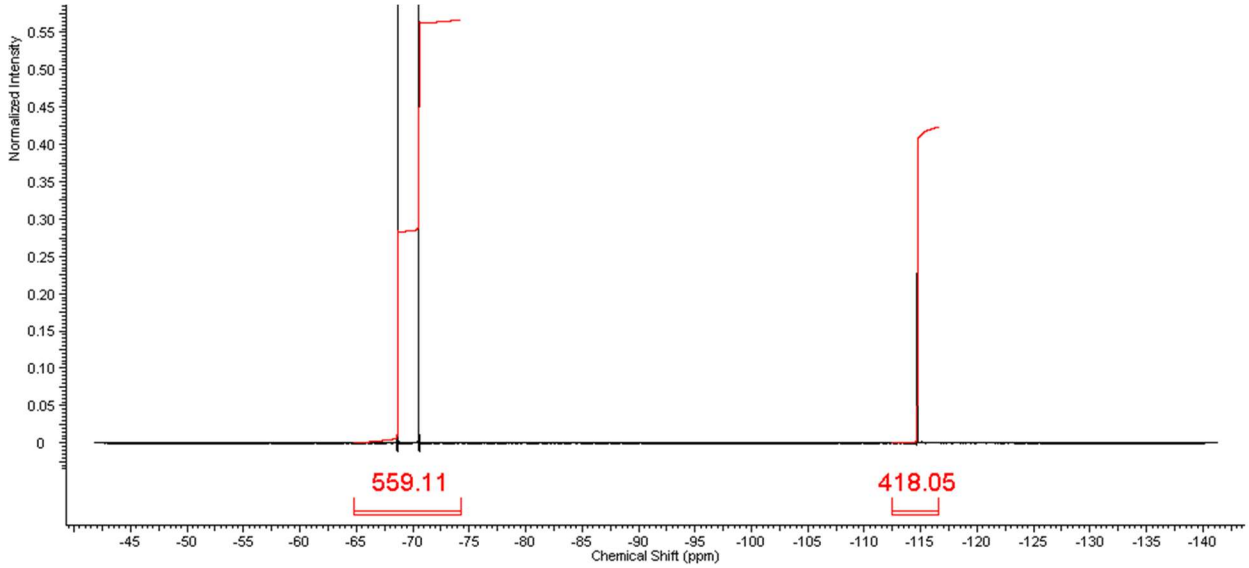
Mass of: sample, 32.09; standard: 11.51.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{725.6}{274.4} . \frac{10}{38} . \frac{536.6}{138.2} . \frac{11.51}{32.09} . 0.995 = 0.96$$

Purity: 96%

S15. SIPr.HPF$_6$ ^{19}F qNMR Vérifié

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
11.56	13.6	

Signals standard (ppm)	-115
Integration	418.1
Signals sample (ppm)	-70
Integration	559.1

Somme of the integral: sample, 559.1; standard, 418.1.

Number of fluorine: sample, 6; standard, 1.

Molecular weight: sample, 534.6; standard, 144.6.

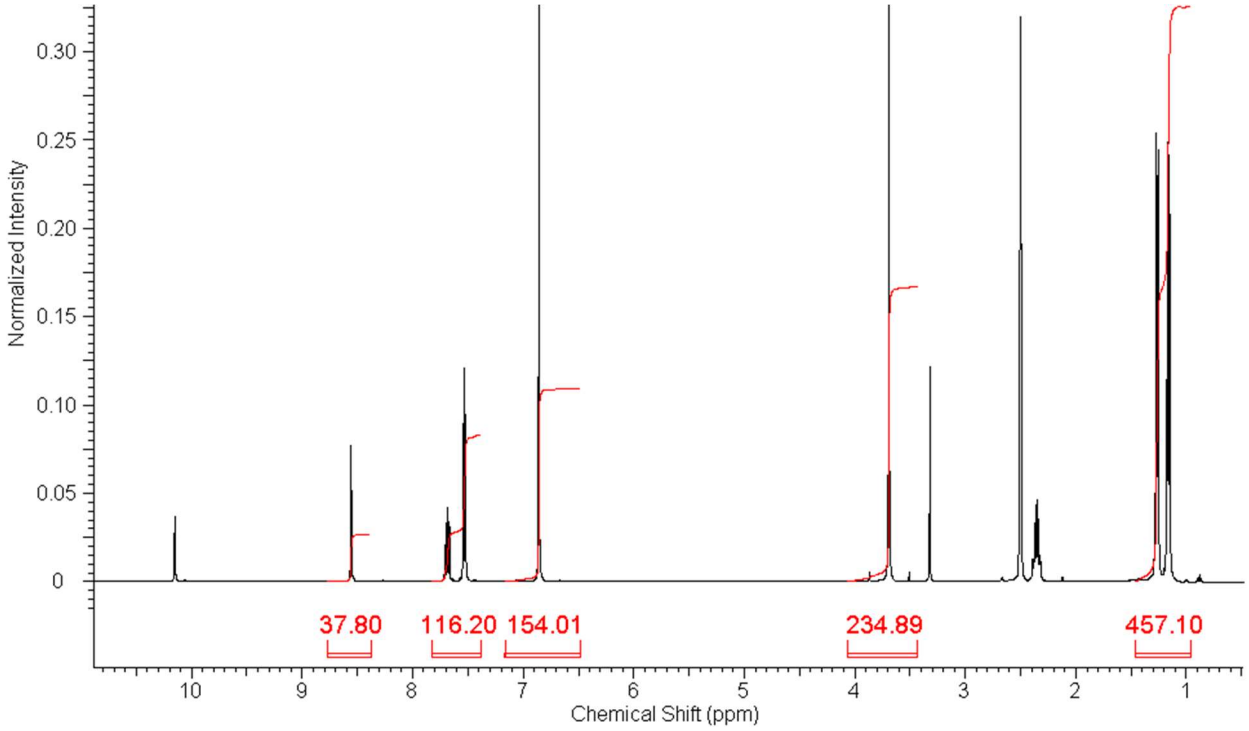
Mass of: sample, 11.56; standard: 13.6.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{559.1}{418.1} . \frac{1}{6} . \frac{534.6}{144.6} . \frac{13.6}{11.56} . 0.996 = 0.97$$

Purity: 97%

S16. IPr.HPF$_6$ ^{1}H qNMR Verifié!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
41.33	20.79	

Signals standard (ppm)	6.86	3.7	
Integration	154.0	234.9	
Signals sample (ppm)	8.56	7.61	1.21
Integration	37.8	116.2	457.1

Somme of the integral: sample, 611.1; standard, 388.9.

Number of protons: sample, 32; standard, 10.

Molecular weight: sample, 536.6; standard, 138.2.

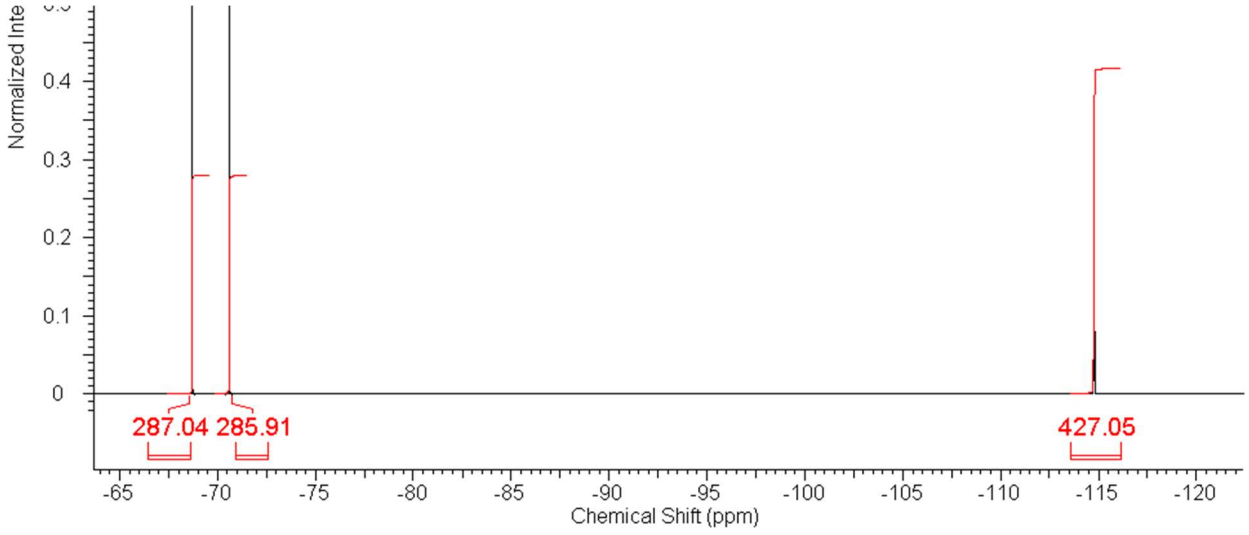
Mass of: sample, 41.33; standard: 20.79.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{611.1}{388.9} . \frac{10}{32} . \frac{536.6}{138.2} . \frac{20.79}{41.33} . 0.995 \ = 0.95$$

Purity: 95%

S17. IPr.HPF$_6$ ^{19}F qNMR Verifié

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
12.80	14.98	

Signals standard (ppm)	-115	
Integration	427	
Signals sample (ppm)	-70	-70
Integration	287	285.9

Somme of the integral: sample, 572.9; standard, 427.

Number of fluorine: sample, 6; standard, 1.

Molecular weight: sample, 534.6; standard, 144.6.

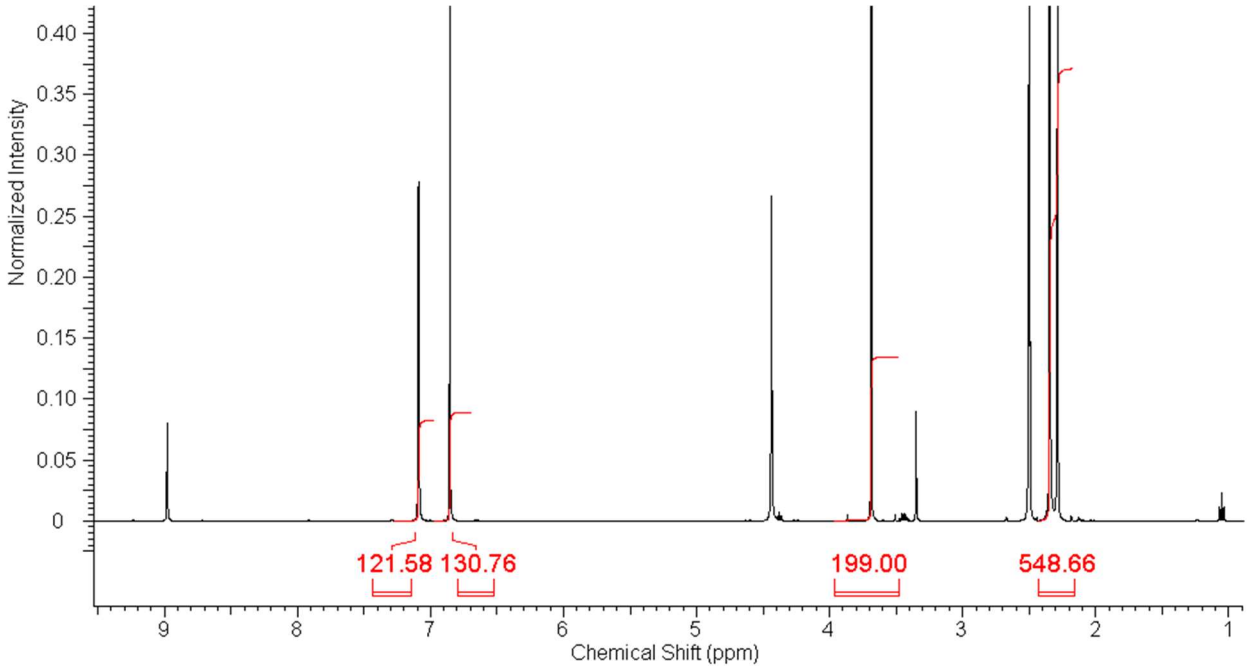
Mass of: sample, 12.8; standard: 14.98.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{572.9}{427} . \frac{1}{6} . \frac{534.6}{144.6} . \frac{14.98}{12.8} . 0.996 = 0.96$$

Purity : 96%

S18. SIMes.HBF$_4$ ^{1}H qNMR Verifié !!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
31.18	11.75	

Signals standard (ppm)	6.86	3.7
Integration	130.8	199
Signals sample (ppm)	7.09	2.32
Integration	121.6	548.7

Somme of the integral: sample, 670.3; standard, 329.8.

Number of protons: sample, 22; standard, 10.

Molecular weight: sample, 394.3; standard, 138.2.

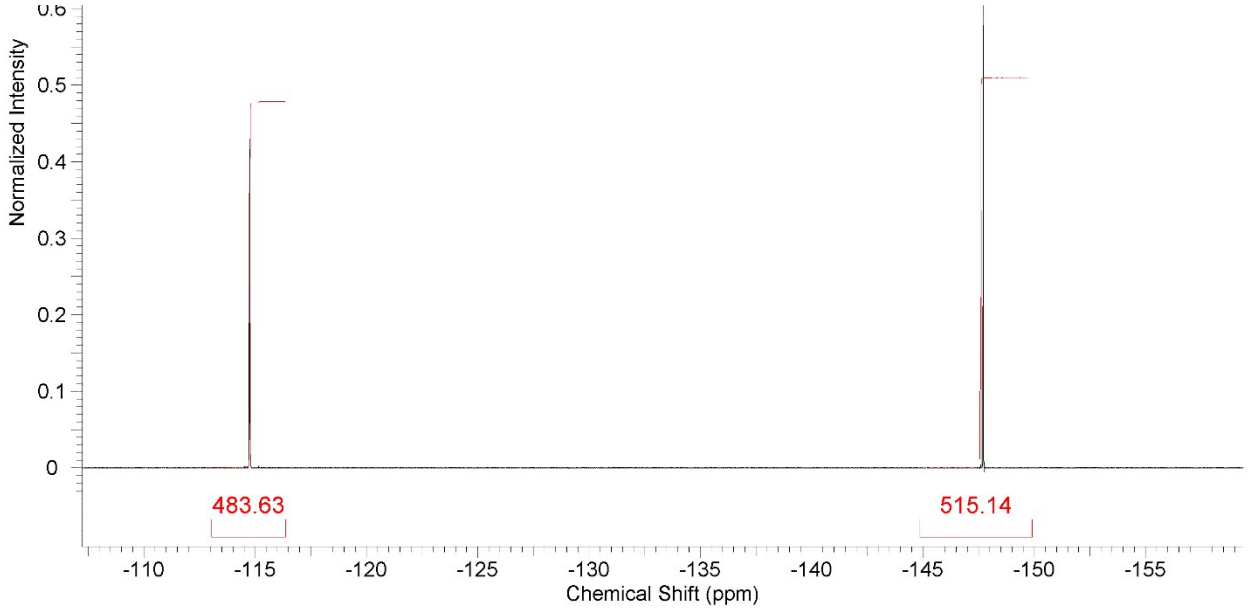
Mass of: sample, 31.18; standard: 11.75.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{670.3}{329.8} . \frac{10}{22} . \frac{394.3}{138.2} . \frac{11.75}{31.18} . 0.995 = 0.98$$

Purity: 98%

S19. SIMes.HBF$_4$ ^{19}F qNMR Verifié

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
10.62	14.58	

Signals standard (ppm)	-115
Integration	484
Signals sample (ppm)	-148
Integration	515

Somme of the integral: sample, 515; standard, 484.

Number of fluorine: sample, 4; standard, 1.

Molecular weight: sample, 394.2; standard, 144.6.

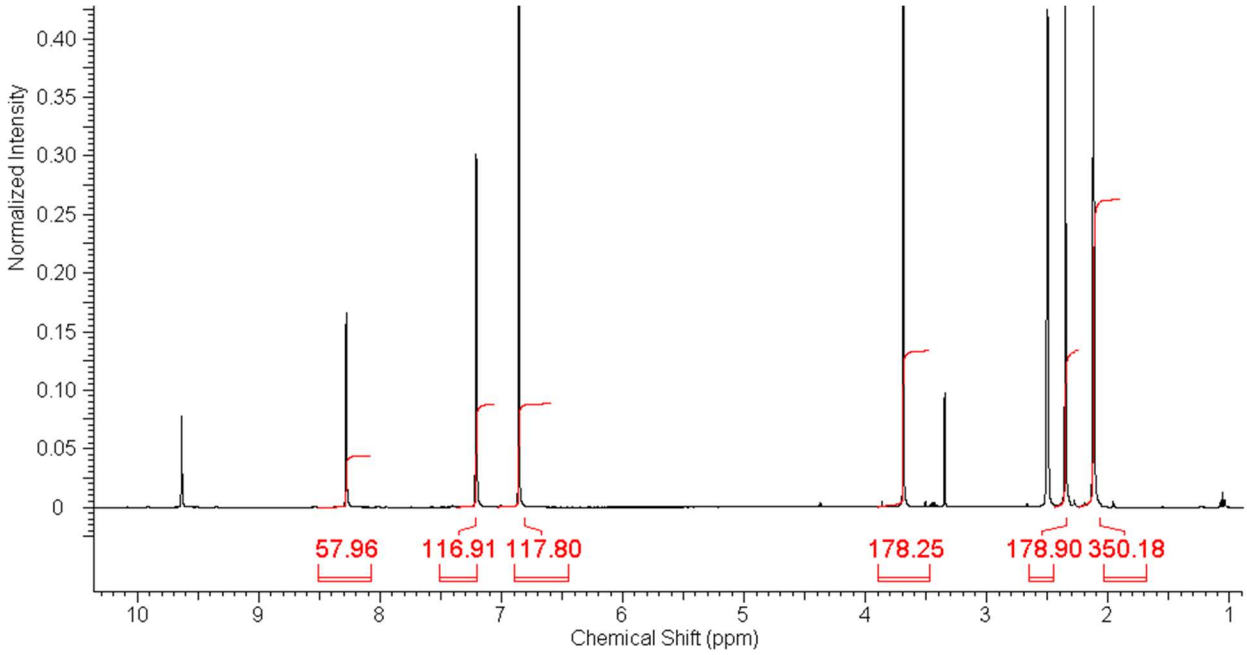
Mass of: sample, 10.62; standard: 14.58.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{515}{484} . \frac{1}{4} . \frac{394.2}{144.6} . \frac{14.58}{10.62} . 0.996 \ = 0.99$$

Purity: 99%

S20. IMes.HBF$_4$ ^{1}H qNMR Verifié !!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
35.55	12.02	

Signals standard (ppm)	6.86	3.7		
Integration	117.8	178.3		
Signals sample (ppm)	8.3	7.2	2.4	2.1
Integration	58	116.9	178.9	350.2

Somme of the integral: sample, 704; standard, 296.1.

Number of protons: sample, 24; standard, 10.

Molecular weight: sample, 392.2; standard, 138.2.

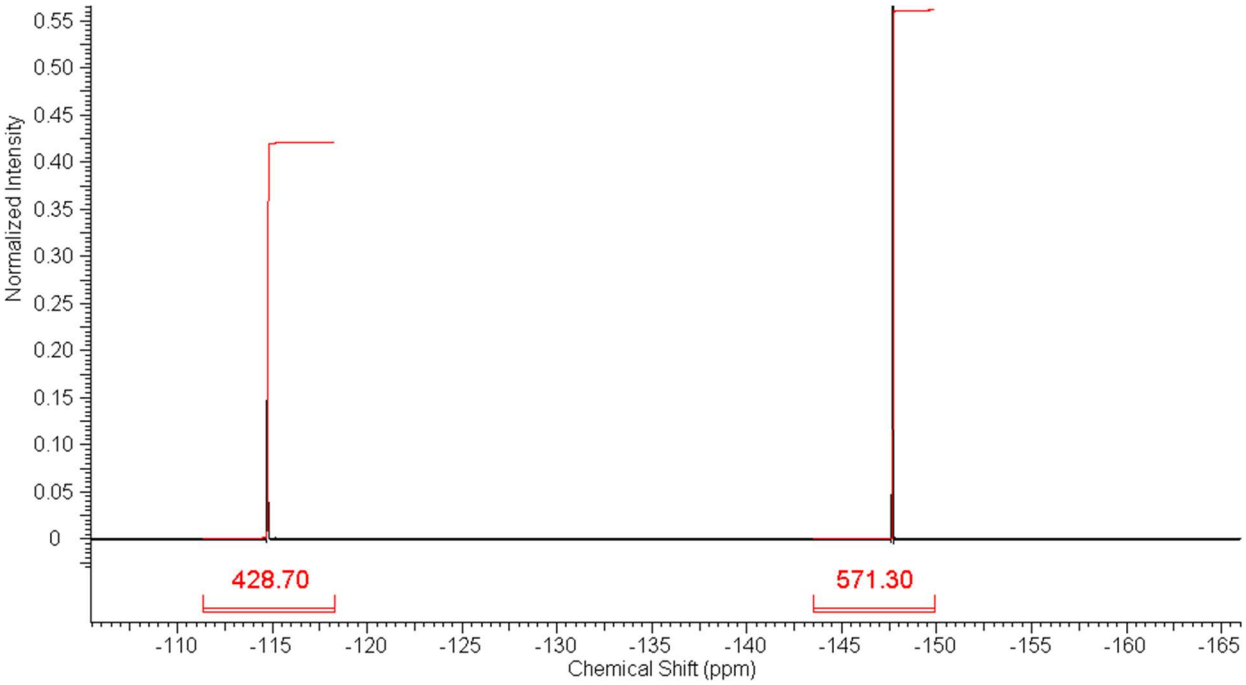
Mass of: sample, 35.55; standard: 12.02.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{704}{296.1} . \frac{10}{24} . \frac{392.2}{138.2} . \frac{12.02}{35.55} . 0.995 = 0.94$$

Purity: 94%

S21. IMes.HBF$_4$ ^{19}F qNMR Verifié

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d$_6$
11.18	12.1	

Signals standard (ppm)	-115
Integration	429
Signals sample (ppm)	-148
Integration	571

Somme of the integral: sample, 571; standard, 429.

Number of fluorine: sample, 4; standard, 1.

Molecular weight: sample, 392.2; standard, 144.6.

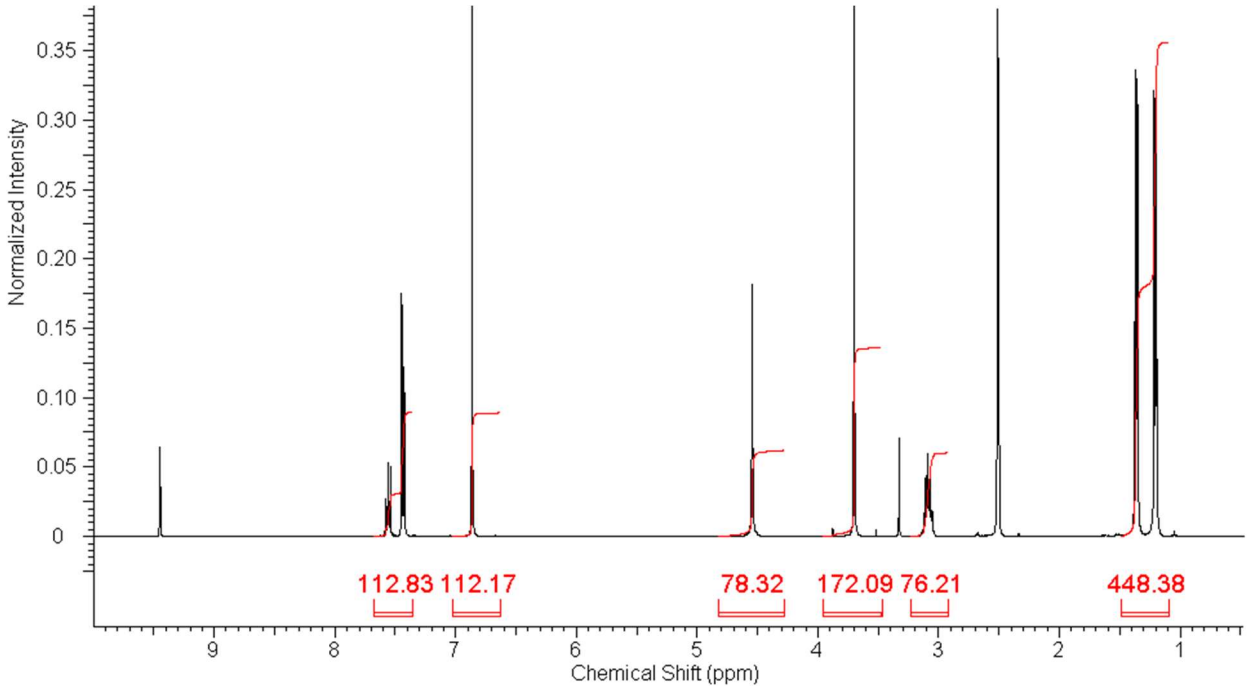
Mass of: sample, 11.18; standard: 12.1.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{571}{429} . \frac{1}{4} . \frac{392.2}{144.6} . \frac{12.1}{11.18} . 0.996 = 0.97$$

Purity: 97%

S22. SIPr. HBF_4 ^1H qNMR Verifié!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
30.8	12.92	

Signals standard (ppm)	6.86	3.7		
Integration	112.2	172.2		
Signals sample (ppm)	7.5	4.6	3.1	1.29
Integration	112.8	78.3	76.2	448.4

Somme of the integral: sample, 715.7; standard, 284.4.

Number of protons: sample, 38; standard, 10.

Molecular weight: sample, 478.4; standard, 138.2.

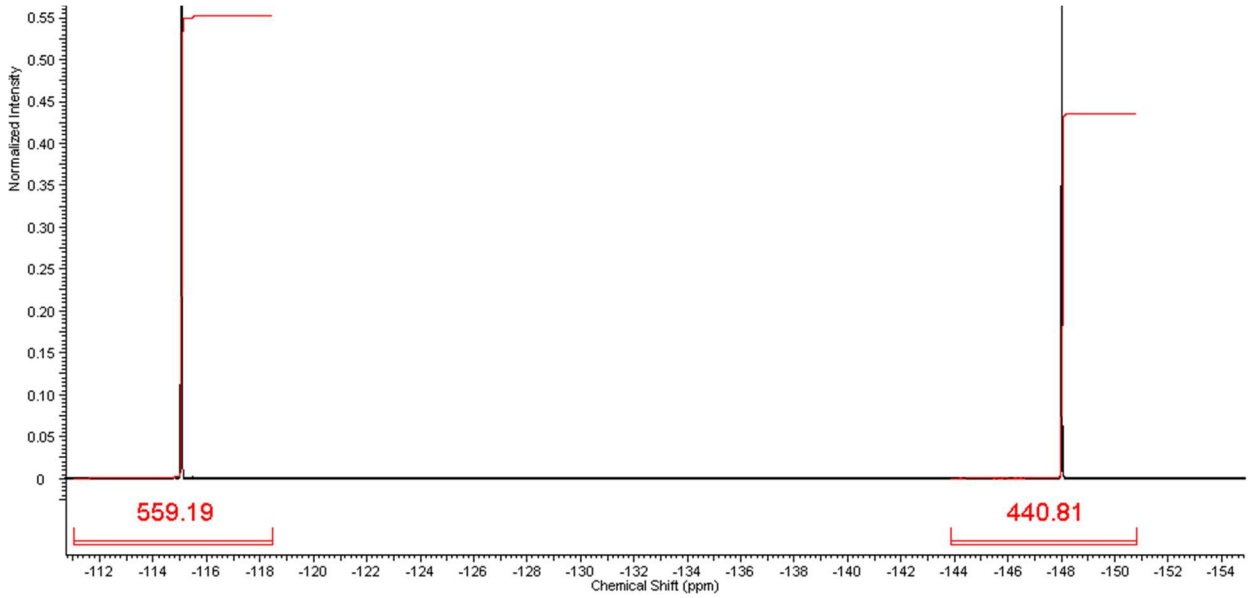
Mass of: sample, 30.8; standard: 12.92.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{715.7}{284.4} . \frac{10}{38} . \frac{478.4}{138.2} . \frac{12.92}{30.8} . 0.995 \ = 0.95$$

Purity: 95%

S23. SIPr. HBF_4, ^{19}F qNMR Verifié

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6^{Th} order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
10.1	15.2	

Signals standard (ppm)	-115
Integration	559.2
Signals sample (ppm)	-148
Integration	440.8

Somme of the integral: sample, 440.8; standard, 559.2.

Number of fluorine: sample, 4; standard, 1.

Molecular weight: sample, 478.4; standard, 144.6.

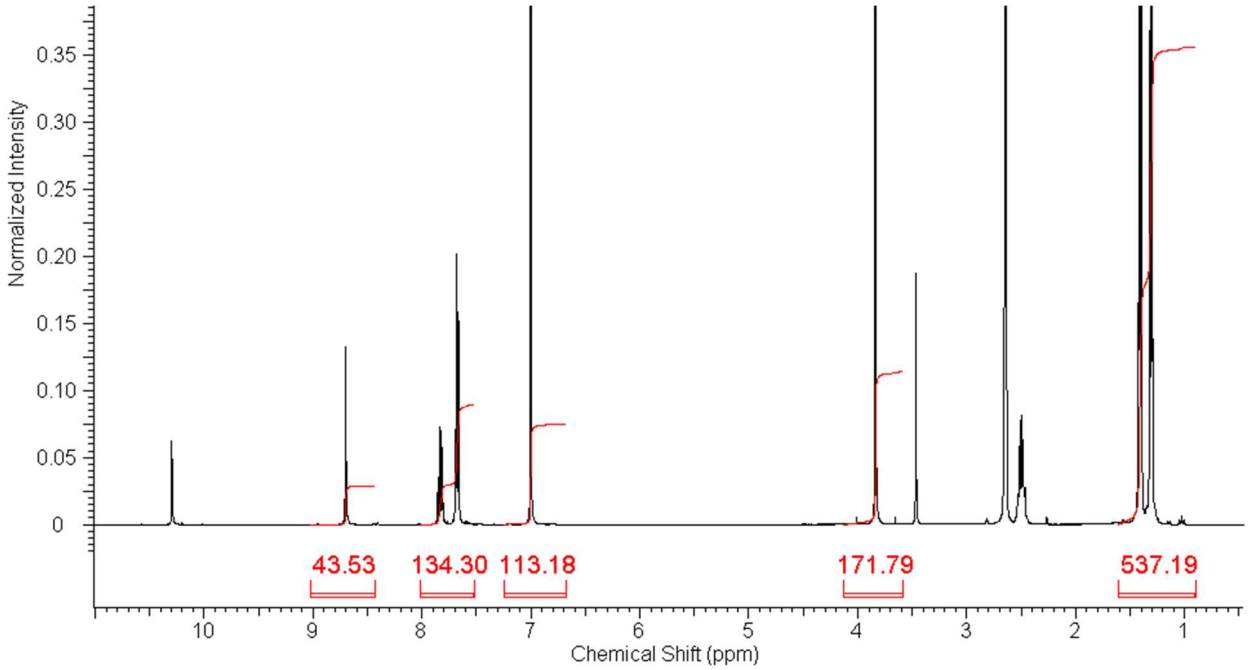
Mass of: sample, 10.1; standard: 15.2.

$$P_s = \frac{I_s}{I_{std}} \cdot \frac{N_{std}}{N_s} \cdot \frac{M_s}{M_{std}} \cdot \frac{m_{std}}{m_s} \cdot P_{std} = \frac{440.8}{559.2} \cdot \frac{1}{4} \cdot \frac{478.4}{144.6} \cdot \frac{15.2}{10.1} \cdot 0.996 = 0.97$$

Purity: 97%

S24. IPr. HBF_4, ^1H qNMR Verifié!!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6^{Th} order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
31.58	10.96	

Signals standard (ppm)	6.86	3.7	
Integration	113.2	171.8	
Signals sample (ppm)	8.7	7.8	1.4
Integration	45.53	134.3	537.2

Somme of the integral: sample, 717.3; standard, 285.

Number of protons: sample, 32; standard, 10.

Molecular weight: sample, 476.4; standard, 138.2.

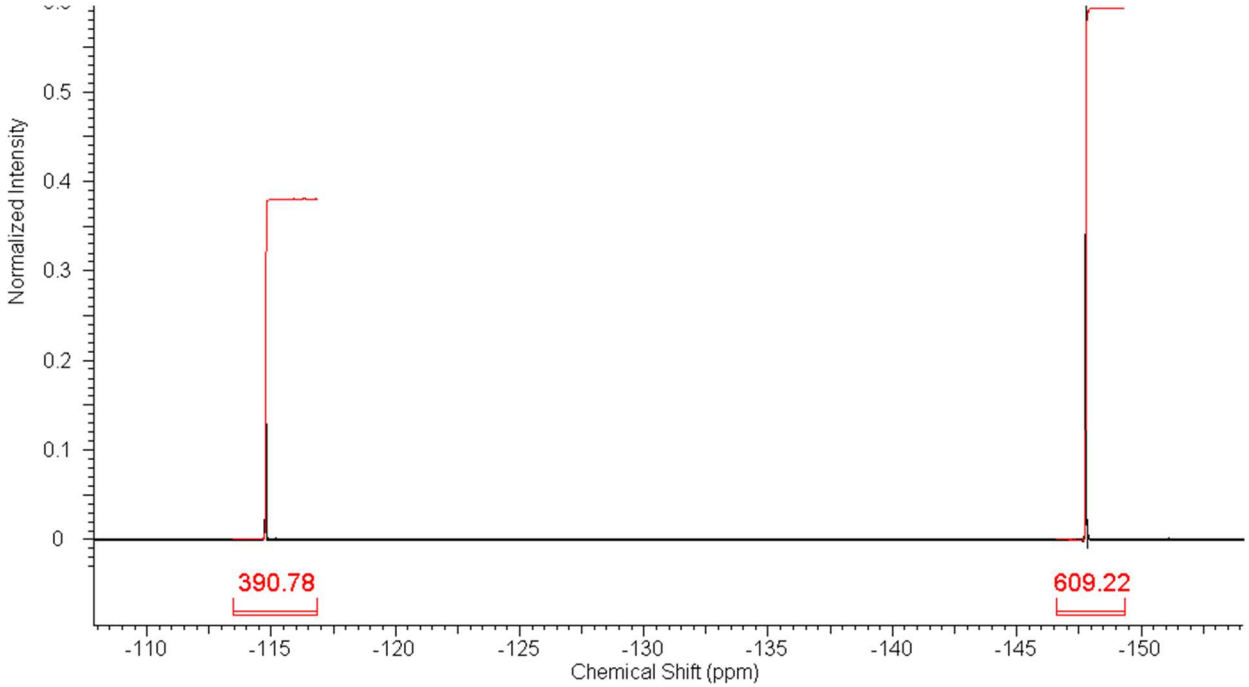
Mass of: sample, 31.58; standard: 10.96.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{717.3}{285} . \frac{10}{32} . \frac{476.4}{138.2} . \frac{10.96}{31.58} . 0.995 \ = 0.93$$

Purity: 93%

S25. IPr. HBF_4, ^{19}F qNMR Verifié!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6^{Th} order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
14.9	11.2	

Signals standard (ppm)	-115
Integration	390.8
Signals sample (ppm)	-148
Integration	609.2

Somme of the integral: sample, 609.2; standard, 390.8.

Number of fluorine: sample, 4; standard, 1.

Molecular weight: sample, 476.4; standard, 144.6.

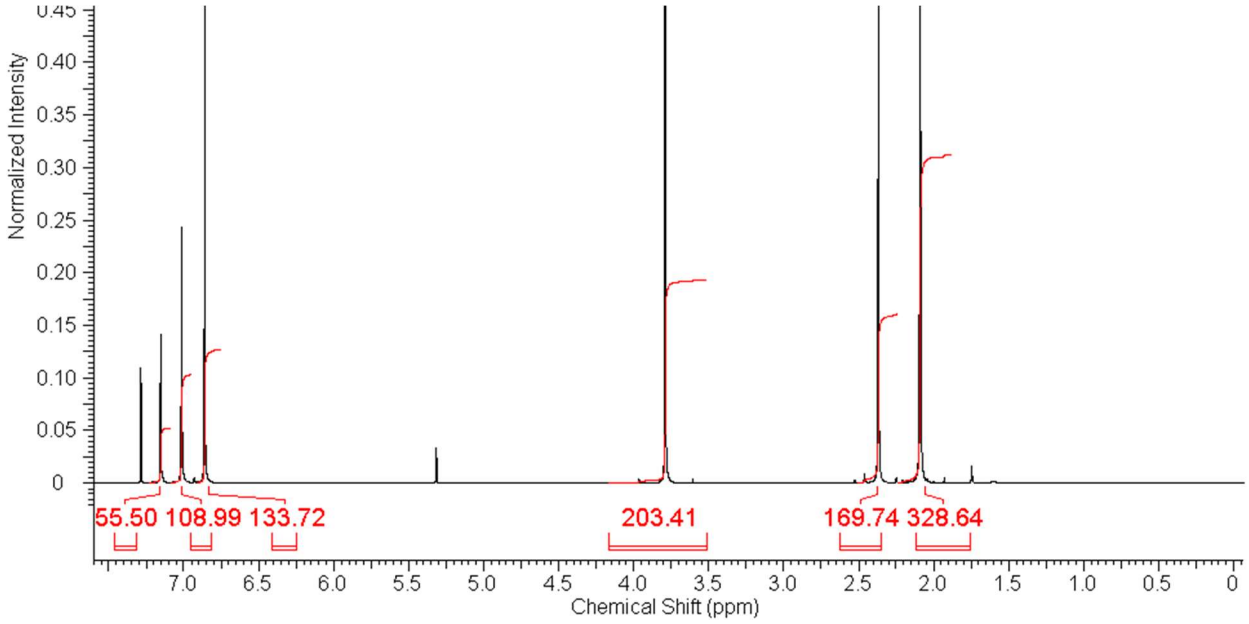
Mass of: sample, 14.9; standard: 11.12.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{609.2}{390.8} . \frac{1}{4} . \frac{476.4}{144.6} . \frac{11.12}{14.9} . 0.996 = 0.95$$

Purity: 95%

S26. IMesAgCl, ^{1}H qNMR Vérifié!!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: $CDCl_3$
41.72	14.9	

Signals standard (ppm)	6.86	3.7		
Integration	133.72	203.4		
Signals sample (ppm)	7.14	7.0	2.36	2.07
Integration	55.5	109	169.7	328.6

Somme of the integral: sample, 662.8; standard, 327.1.

Number of protons: sample, 24; standard, 10.

Molecular weight: sample, 449.7; standard, 138.2.

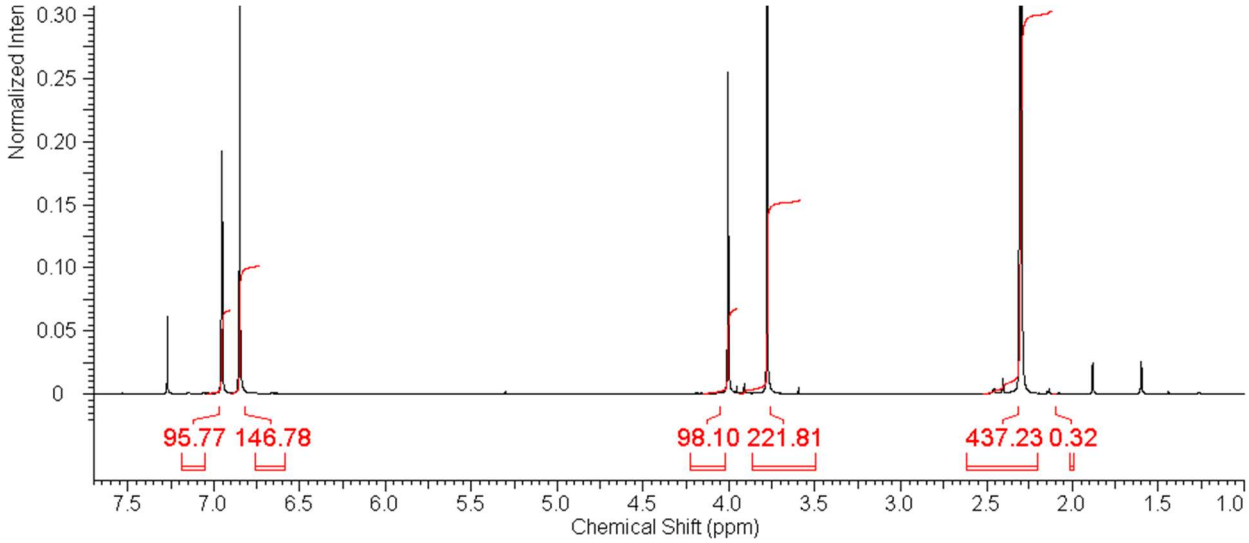
Mass of: sample, 41.72; standard: 14.9.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{662.8}{337.4} . \frac{10}{24} . \frac{449.7}{138.2} . \frac{14.9}{41.72} . 0.995 \ = 0.97$$

Purity: 97%

S27. SIMesAgCl, ^{1}H qNMR Vérifié !!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6^{Th} order



Masse sample (mg)	Masse Standard (mg)	Solvent: $CDCl_3$
28.79	12.74	

Signals standard (ppm)	6.86	3.7	
Integration	146.8	221.8	
Signals sample (ppm)	6.95	4	2.3
Integration	95.8	98.1	437.2

Somme of the integral: sample, 631.1; standard, 368.6.

Number of protons: sample, 26; standard, 10.

Molecular weight: sample, 449.8; standard, 138.2.

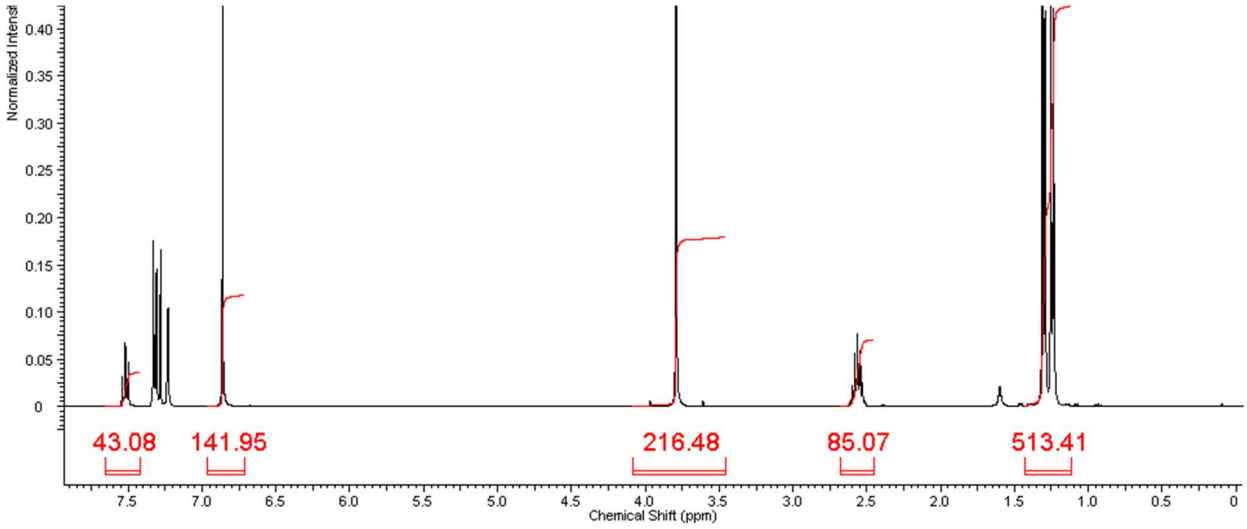
Mass of: sample, 28.79; standard: 12.74.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{631.1}{368.7} . \frac{10}{26} . \frac{449.8}{138.2} . \frac{12.74}{28.79} . 0.995 = 0.95$$

Purity: 95%

S28. IPrAgCl, ^{1}H qNMR Vérifié !

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: $CDCl_3$
23.89	10.20	

Signals standard (ppm)	6.86	3.7	
Integration	142.0	216.5	
Signals sample (ppm)	7.7	7.5	7.4
Integration	43.1	85.1	513.4

Somme of the integral: sample, 641.6; standard, 358.5.

Number of protons: sample, 30; standard, 10.

Molecular weight: sample, 531.9 standard, 138.2.

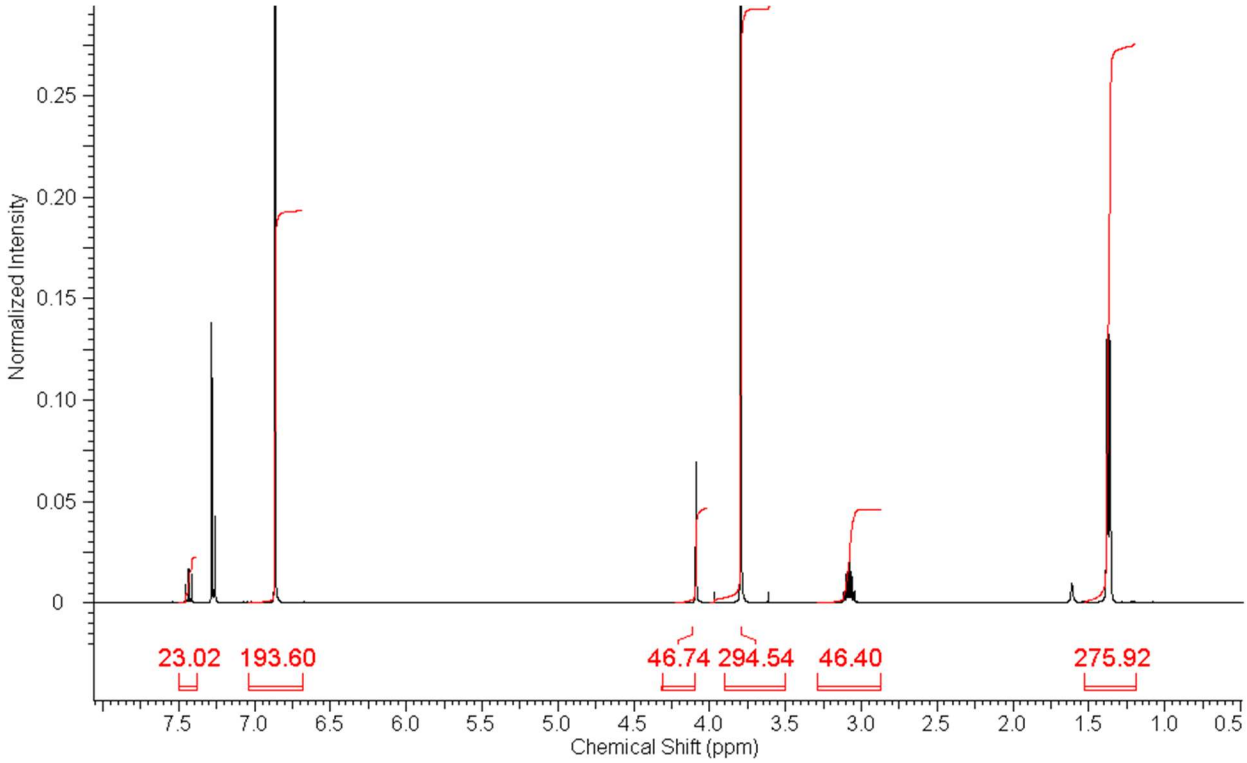
Mass of: sample, 23.89; standard: 10.2.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{641.6}{358.5} . \frac{10}{30} . \frac{531.9}{138.2} . \frac{10.2}{23.89} . 0.995 \ = 0.97$$

Purity: 97%

S29. SIPrAgCl ^{1}H qNMR Vérifié !!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: $CDCl_3$
11.86	12.83	

Signals standard (ppm)	6.86	3.7		
Integration	193.6	294.5		
Signals sample (ppm)	7.43	4.1	3.1	1.4
Integration	23.0	46.7	46.4	275.9

Somme of the integral: sample, 392; standard, 488.1.

Number of protons: sample, 34; standard, 10.

Molecular weight: sample, 533.9; standard, 138.2.

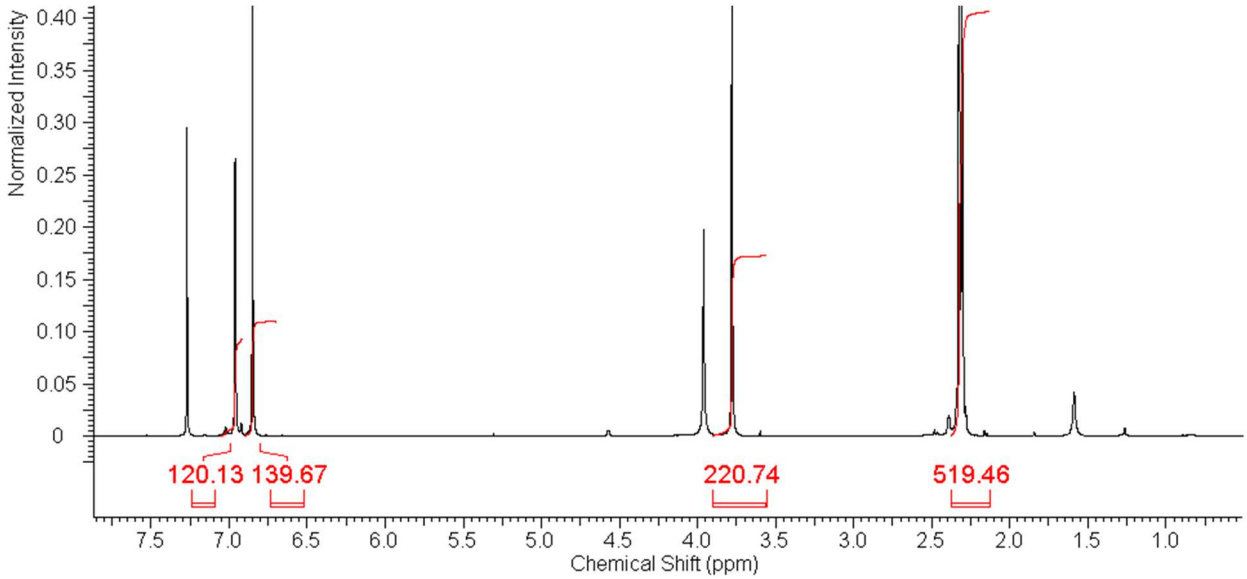
Mass of: sample, 11.86; standard: 12.83.

$$P_s = \frac{I_s}{I_{std}} \cdot \frac{N_{std}}{N_s} \cdot \frac{M_s}{M_{std}} \cdot \frac{m_{std}}{m_s} \cdot P_{std} = \frac{392}{488.1} \cdot \frac{10}{34} \cdot \frac{533.9}{138.2} \cdot \frac{12.83}{11.86} \cdot 0.995 = 0.98$$

Purity: 98%

S30. SIMesCuCl, ^{1}H qNMR Vérifié!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6^{Th} order



Masse sample (mg)	Masse Standard (mg)	Solvent: CDCl3
33.25	13.18	

Signals standard (ppm)	6.86	3.7
Integration	139.7	220.7
Signals sample (ppm)	7.0	2.3
Integration	120.1	519.5

Somme of the integral: sample, 639.6; standard, 360.4.

Number of protons: sample, 22; standard, 10.

Molecular weight: sample, 405.4; standard, 138.2.

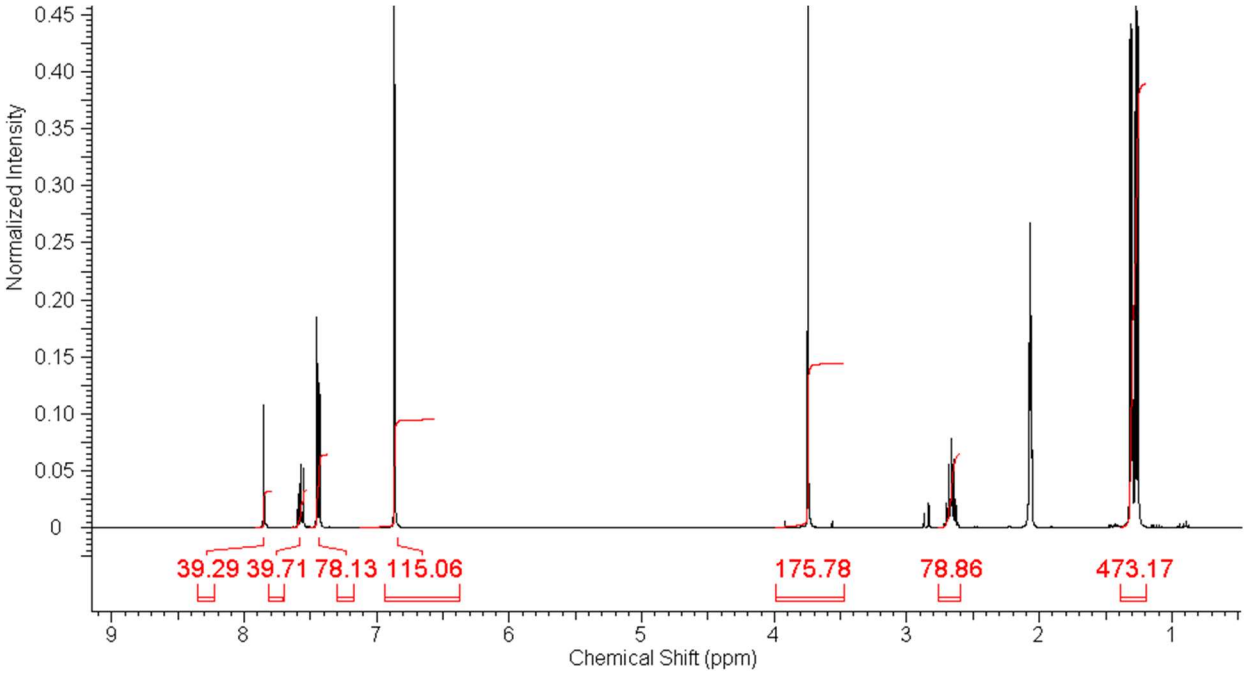
Mass of: sample, 33.25; standard: 13.18.

$$P_s = \frac{I_s}{I_{std}} \cdot \frac{N_{std}}{N_s} \cdot \frac{M_s}{M_{std}} \cdot \frac{m_{std}}{m_s} \cdot P_{std} = \frac{639.6}{360.4} \cdot \frac{10}{22} \cdot \frac{405.4}{138.2} \cdot \frac{13.18}{33.25} \cdot 0.995 = 0.93$$

Purity: 93%

S31. IPrCuCl, ^{1}H qNMR VERIFIER !

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: Acetone-d_6
31.38	13.00	

Signals standard (ppm)	6.86	3.7			
Integration	115.1	175.8			
Signals sample (ppm)	7.9	7.6	7.4	2.7	1.3
Integration	39.3	39.7	78.1	78.9	473.2

Somme of the integral: sample, 709.2; standard, 290.9.

Number of protons: sample, 36; standard, 10.

Molecular weight: sample, 489.6; standard, 138.2.

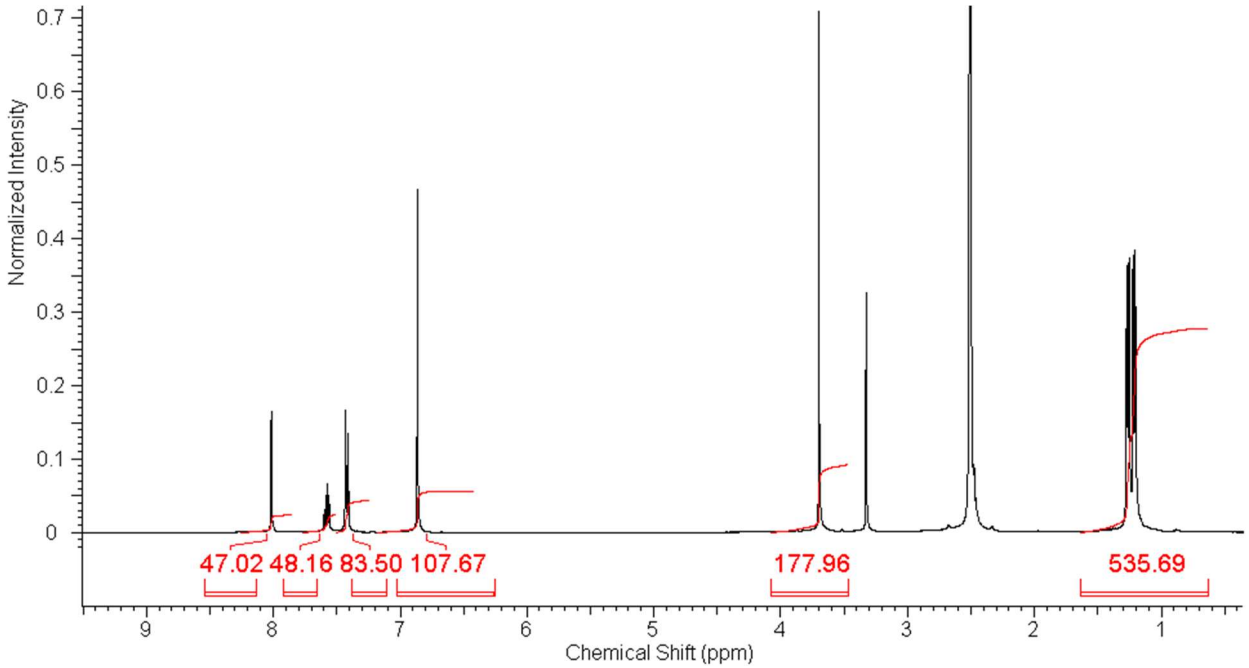
Mass of: sample, 31.38; standard: 13.00.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{709.2}{290.9} . \frac{10}{36} . \frac{489.6}{138.2} . \frac{13.00}{31.38} . 0.995 \ = 0.98$$

Purity: 98%

S32. IPrAuCl, ^{1}H qNMR Vérifié !!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
50.83	13.52	

Signals standard (ppm)	6.86	3.7		
Integration	107.7	178.0		
Signals sample (ppm)	8.0	7.6	7.4	1.2
Integration	47	48.2	83.5	535.7

Somme of the integral: sample, 714.4; standard, 285.7.

Number of protons: sample, 32; standard, 10.

Molecular weight: sample, 621; standard, 138.2.

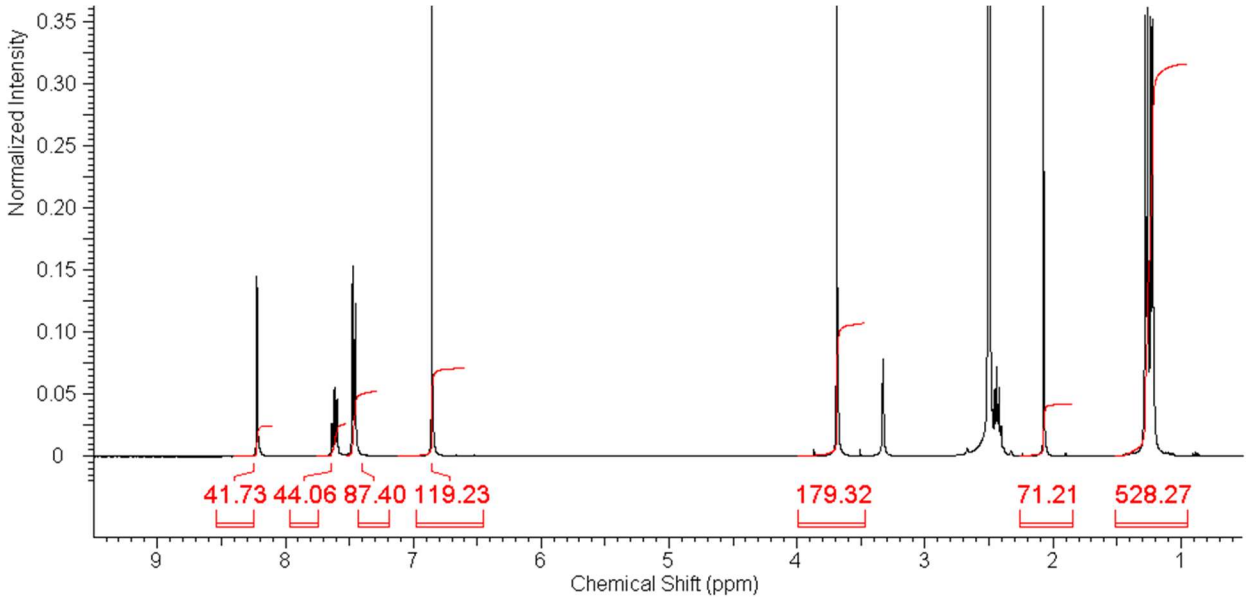
Mass of: sample, 50.83; standard: 13.52.

$$P_s = \frac{I_s}{I_{std}} \cdot \frac{N_{std}}{N_s} \cdot \frac{M_s}{M_{std}} \cdot \frac{m_{std}}{m_s} \cdot P_{std} = \frac{714.4}{285.7} \cdot \frac{10}{32} \cdot \frac{621.0}{138.2} \cdot \frac{13.52}{50.83} \cdot 0.995 = 0.92$$

Purity: 92%

S33. [IPrAu,MeCN], BF_4 ,^{1}H qNMR, Vérifié!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6Th order



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
49.3	12.21	

Signals standard (ppm)	6.86	3.7			
Integration	119.2	179.3			
Signals sample (ppm)	8.2	7.6	7.5	2.0	1.3
Integration	41.7	44.1	87.4	71.2	528.3

Somme of the integral: sample, 772.71; standard, 289.5.

Number of protons: sample, 35; standard, 10.

Molecular weight: sample, 713.4; standard, 138.2.

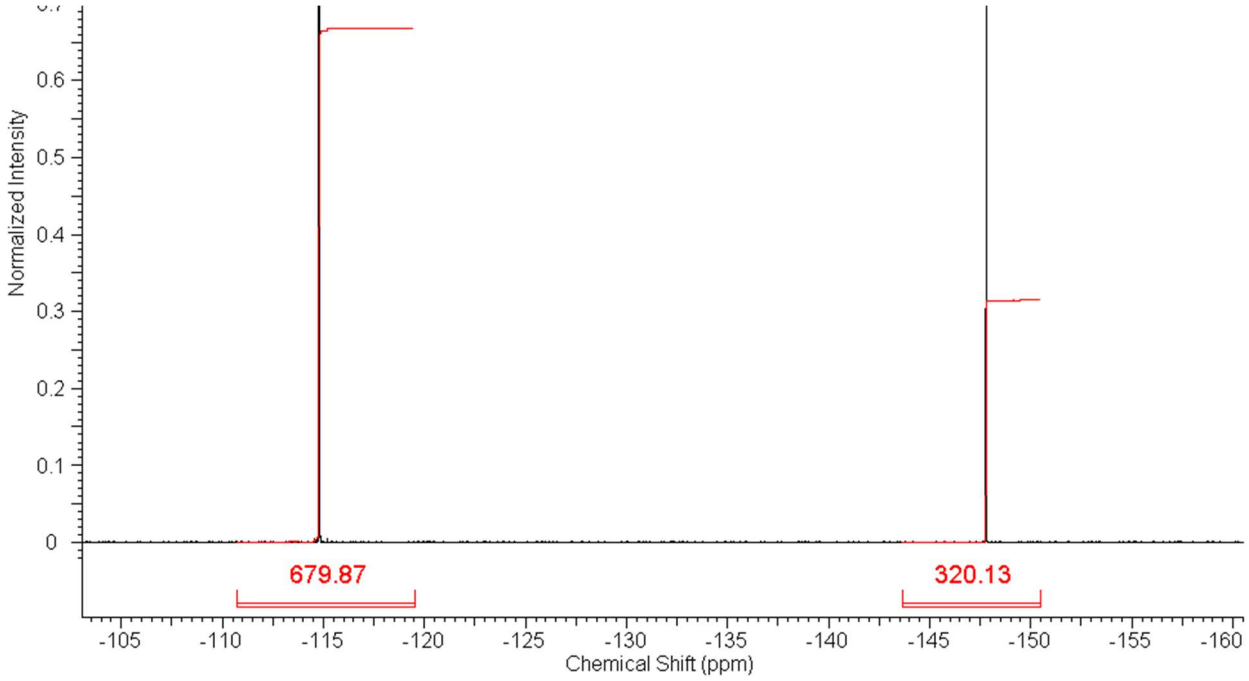
Mass of: sample, 49.3; standard: 12.21.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{772.7}{289.5} . \frac{10}{35} . \frac{713.4}{138.2} . \frac{12.21}{49.3} . 0.995 \ = 0.93$$

Purity: 93%

S34. [IPrAu,MeCN], BF_4, ^{19}F qNMR Verifié

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	FID reconstruction



Masse sample (mg)	Masse Standard (mg)	Solvent: dmso-d_6
16.74	26.74	

Signals standard (ppm)	-115
Integration	679.9
Signals sample (ppm)	-148
Integration	320.1

Somme of the integral: sample, 320.1; standard, 679.9.

Number of fluorine: sample, 4; standard, 1.

Molecular weight: sample, 713.4; standard, 144.6.

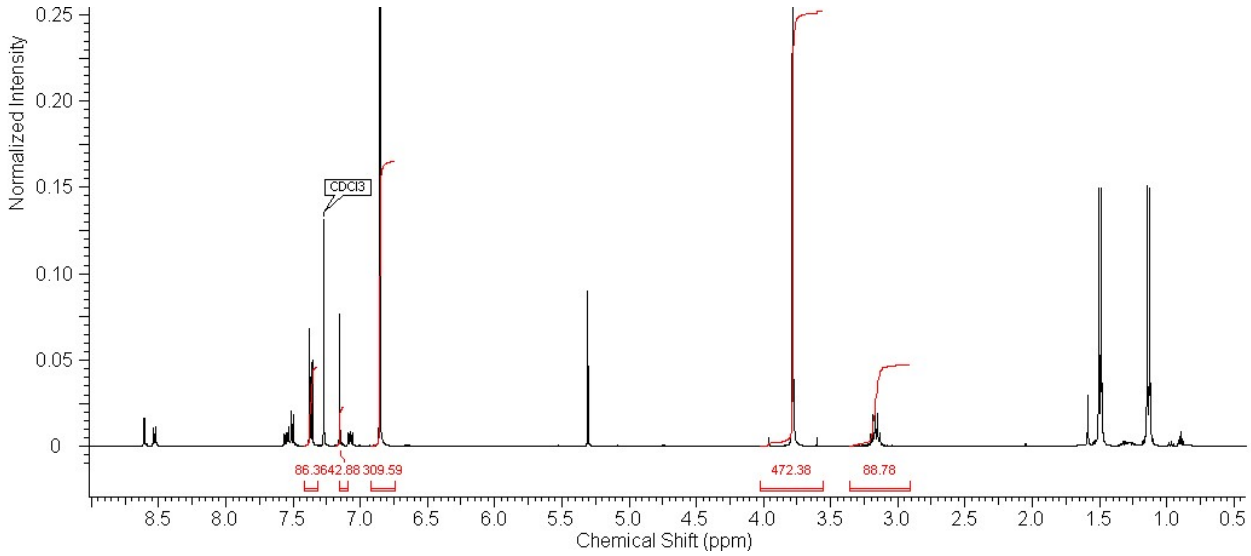
Mass of: sample, 16.74; standard: 26.74.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{st} = \frac{320.1}{679.9} . \frac{1}{4} . \frac{713.4}{144.6} . \frac{26.74}{16.74} . 0.996 = 0.95$$

Purity: 95%

S35. PEPSSI. 0.8 CH_2Cl_2, ^1H qNMR Verifié!!

Zero filling	LB	Phase	Baseline
256K	0.1	Manual	6^{Th} order



Masse sample (mg)	Masse Standard (mg)	Solvent: $CDCl_3$
19.73	13.43	

Signals standard (ppm)	6.7	3.7	
Integration	309.6	472.4	
Signals sample (ppm)	7.37	7.15	3.16
Integration	86.4	42.9	88.8

Somme of the integral: sample, 307.1; standard, 782.

Number of protons: sample, 14; standard, 10.

Molecular weight: sample, 745; standard, 138.2.

Mass of: sample (0.8 dcm), 19.73; standard: 12.43.

$$P_s = \frac{I_s}{I_{std}} . \frac{N_{std}}{N_s} . \frac{M_s}{M_{std}} . \frac{m_{std}}{m_s} . P_{std} = \frac{307}{782} . \frac{10}{14} . \frac{745}{138.2} . \frac{12.43}{19.73} . 0.995 \ = 0.96$$

Purity: 96%