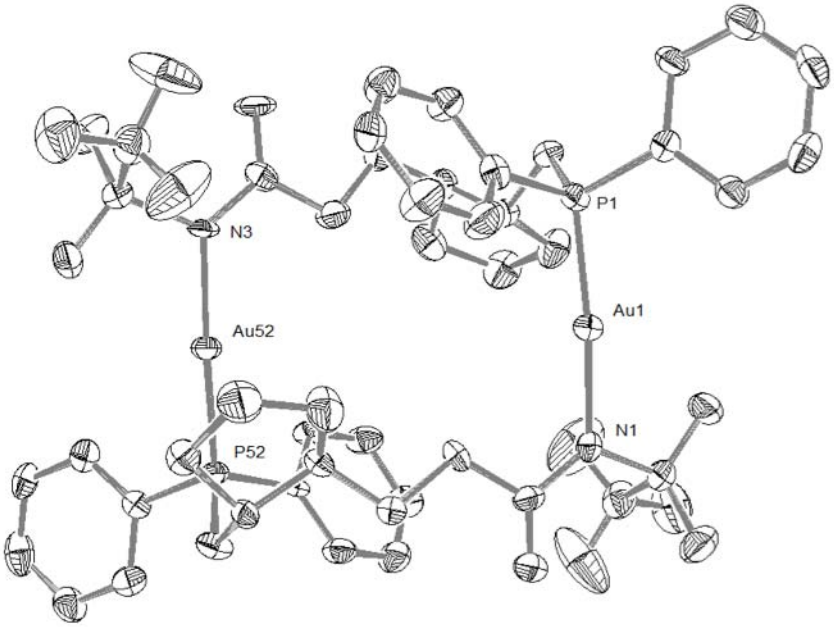
Electronic Supplementary Material (ESI) for Dalton Transactions

Insights in the Mechanism for Gold Catalysis: Behaviour of Gold(I) Amide Complexes in Solution

Mariusz Bobin, Iain J. Day, Mark Roe, Eddy M. E. Viseux*

Experimental: Table of content

1. X-Ray crystallographic studies



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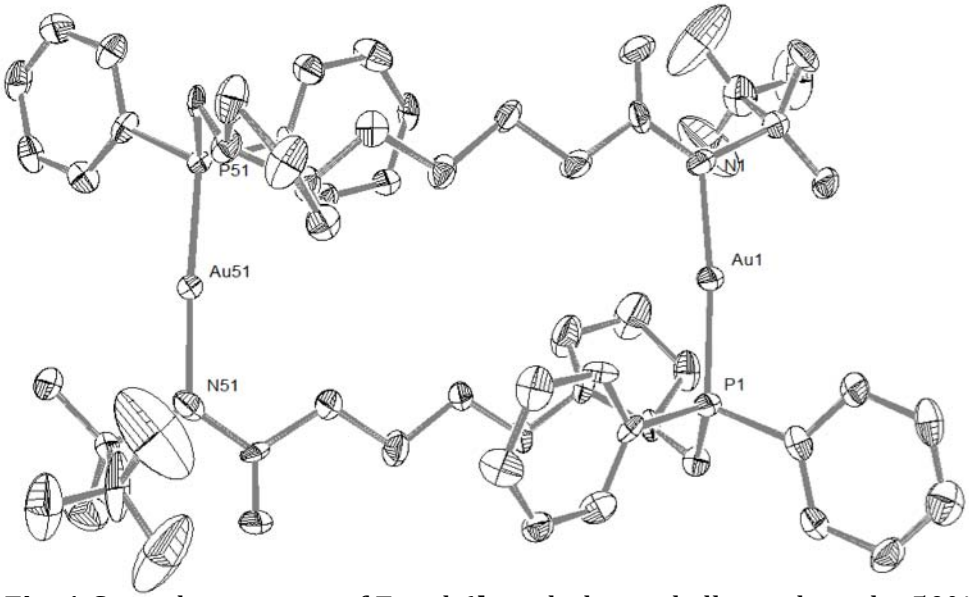


6b

Fig. 1 Crystal structures of **7** and **6b** with thermal ellipsoids at the 50% probability level. Hydrogen atoms are omitted for clarity.

Diffraction data were collected at 173(2) K on an Enraf–Nonius CAD4 diffractometer using monochromated Mo-Kα radiation (λ = 0.71073 Å). Single crystals were coated in oil and then directly mounted on the diffractometer under a stream of cold nitrogen gas at -100°C. All structures were solved with SHELXS[1] and were refined on F^2 with H atoms in riding mode, using SHELXL97[1]. All solvent molecules were able to be refined anisotropically. Molecular drawings were made with ORTEP-3v2[2]. Crystal data and refinement parameters are shown in Table Y. Crystallographic data for the structures reported in this paper have been deposited with the Crystallographic Data Centre.

Table 1 Crystal and structure refinement data for **7** and **6b**.

Compound	**7**	**6b**
CCDC	915634	916508
Formula	$C_{72}H_{72}Au_2F_6N_4O_6P_2S_2$	$C_{46}H_{54}Au_2F_6N_4O_6P_2S_2$
M	1723.33	696.46
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1$
a (Å)	10.5355(3)	10.4641(2)
b (Å)	11.8539(4)	17.3566(5)
c (Å)	15.7927(5)	14.2244(4)
α (°)	94.235(2)	90
β (°)	100.106(1)	90.861(1)
γ (°)	112.490(2)	90
V (A^3)	1772.4(1)	2583.2(1)
Z	1	4
Absorption coefficient (mm^{-1})	4.31	5.89
Unique reflections, R_{int}	14989, 0.040	11184, 0.073
Final R indices $I > 2\sigma(I)$ R_1, wR_2	0.031, 0.068	0.051, 0.105
R indices (all data) R_1, wR_2	0.037, 0.071	0.067, 0.110

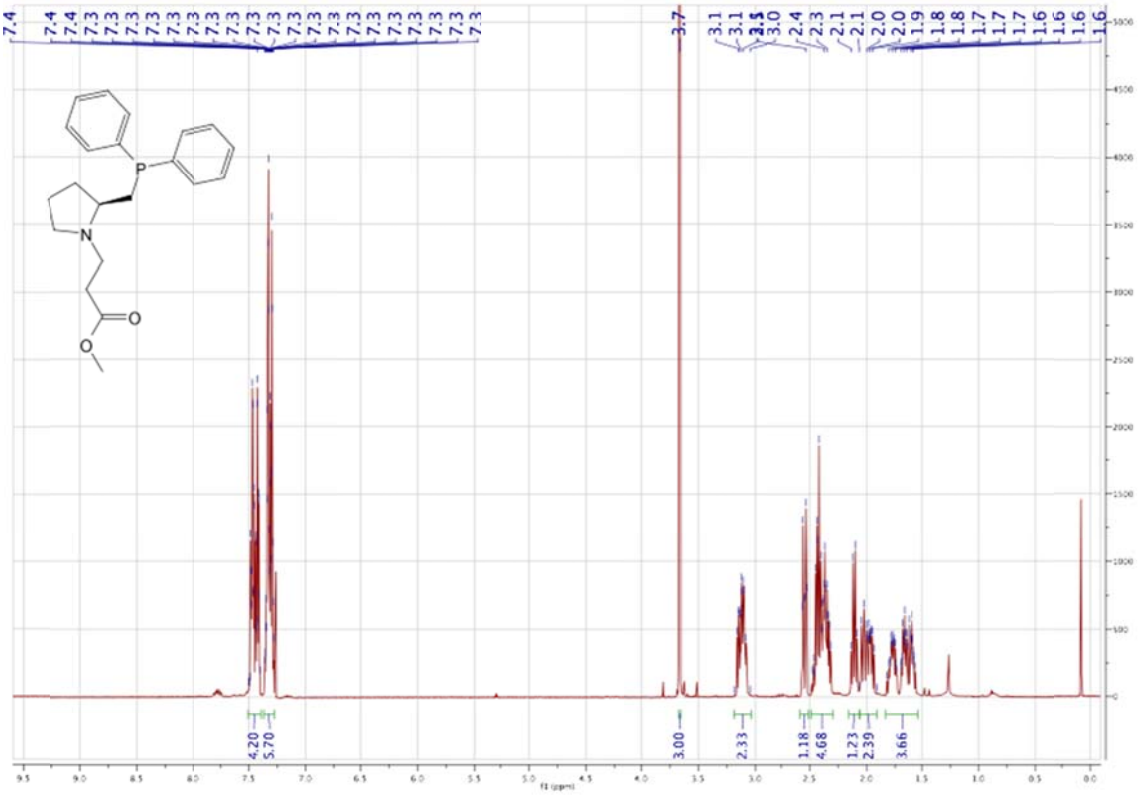
1 G. M. Sheldrick, *SHELXL-97, Program for refinement of crystal structures*, University of Göttingen, Germany, 1997
2 Ortep-3 for Windows: L.J. Farrugia, J. Appl. Cryst., (1997), **30**, 565.

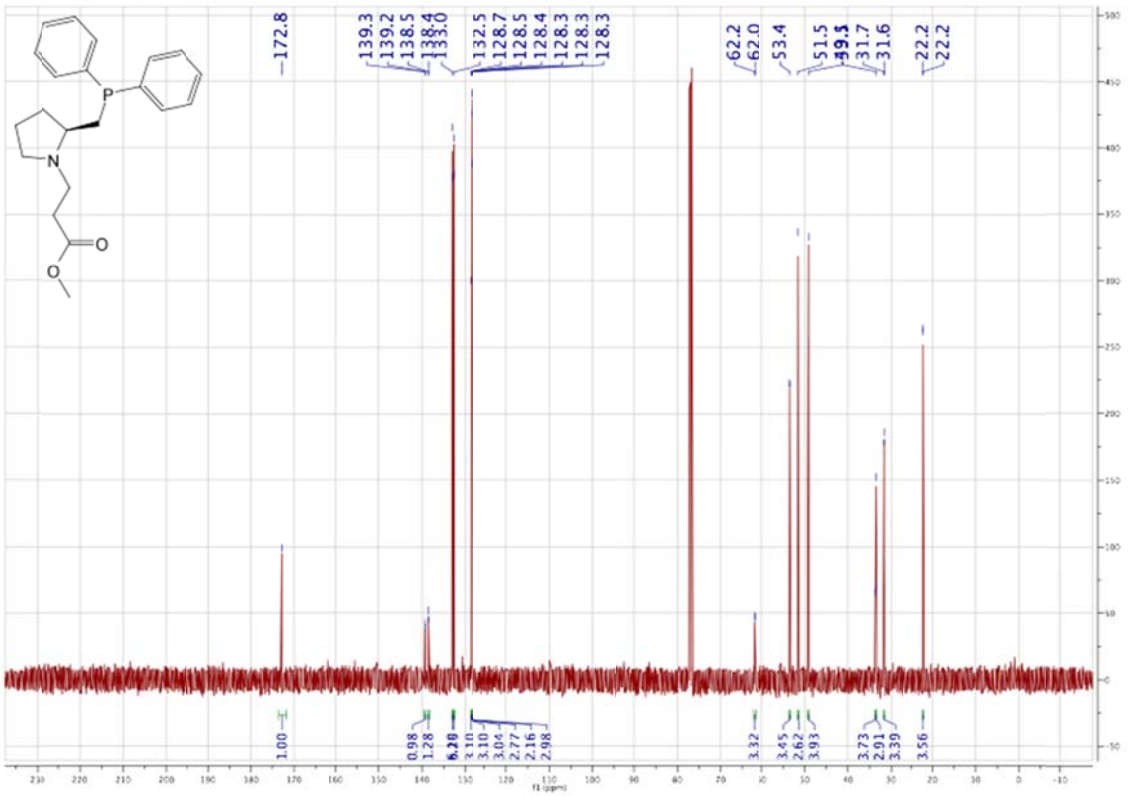
Discussion

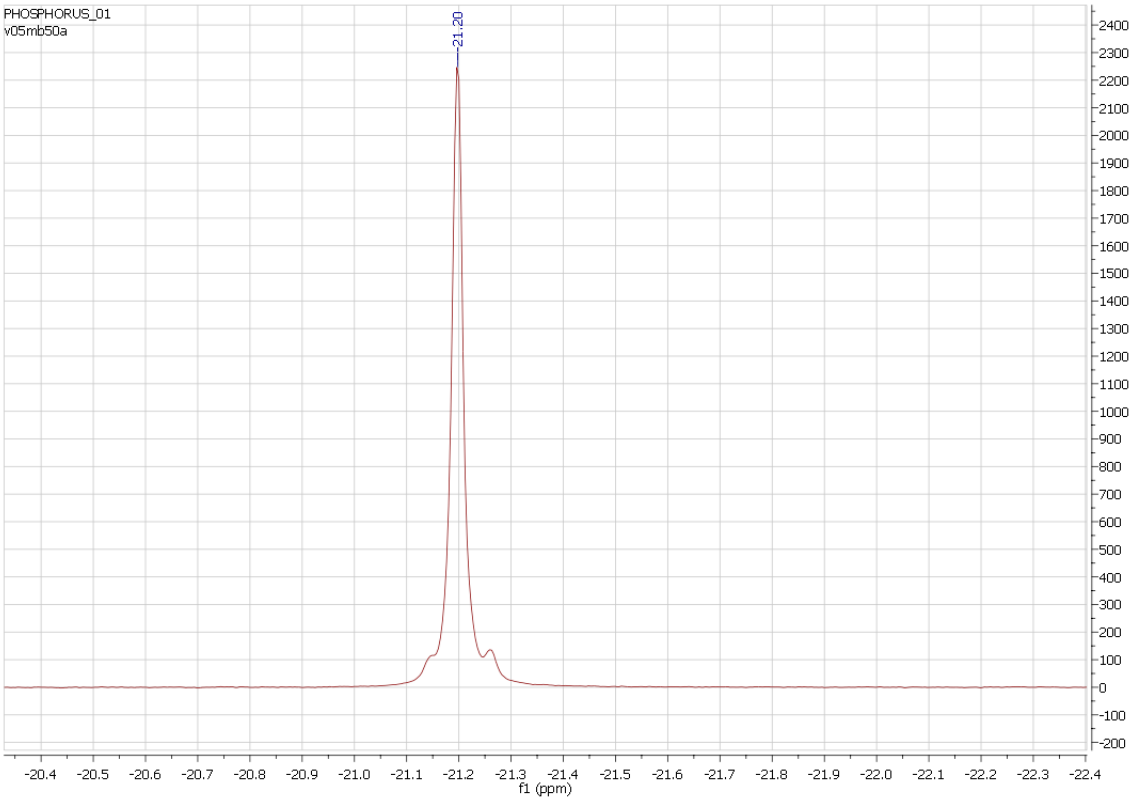
	7	**6b**
Au-N (Å)	2.091(7)	2.093(9)
	2.094(8)	2.076(9)
Au-P (Å)	2.236(3)	2.238(3)
	2.242(2)	2.232(2)
P-Au-N (°)	173.9(2)	174.1(4)
	177.4(2)	176.1(4)
Au-Au (Å)	6.613	9.077
Longest dimension (Å)	18.803	20.255

2. NMR spectra

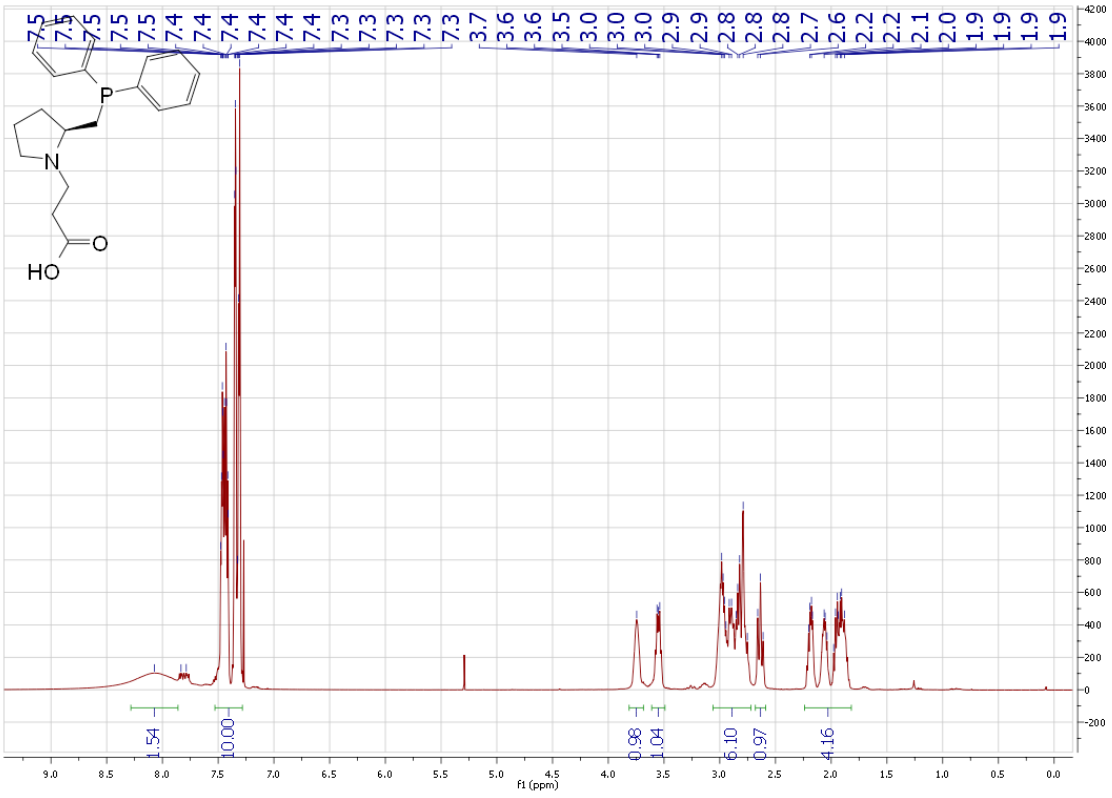
Methyl 3-{(2*S*)-2-[(diphenylphosphino)methyl]pyrrolidin-1-yl} propanoate

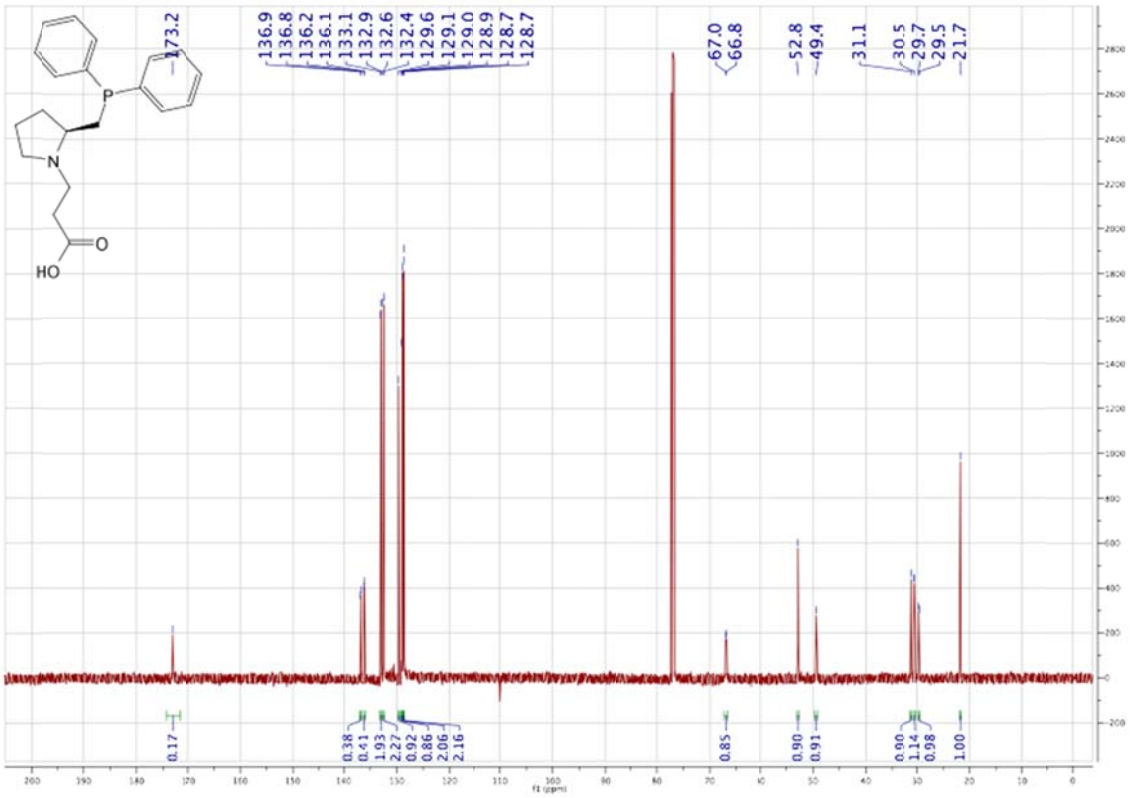


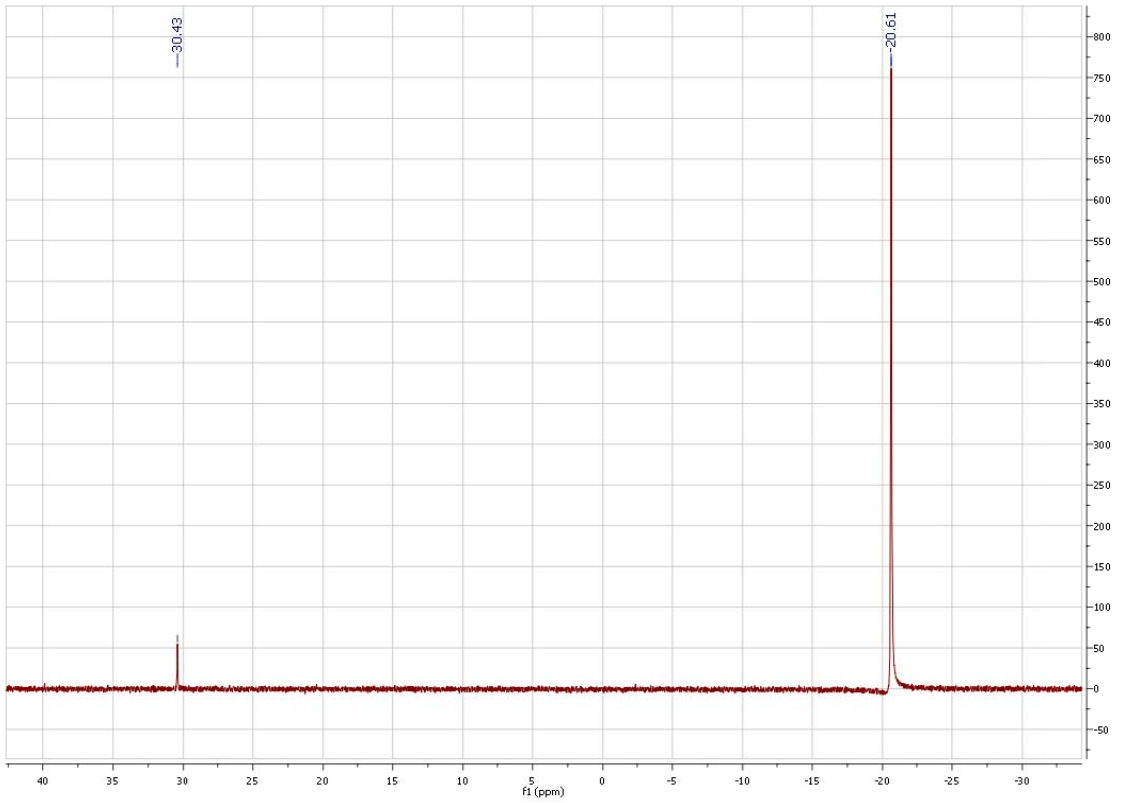




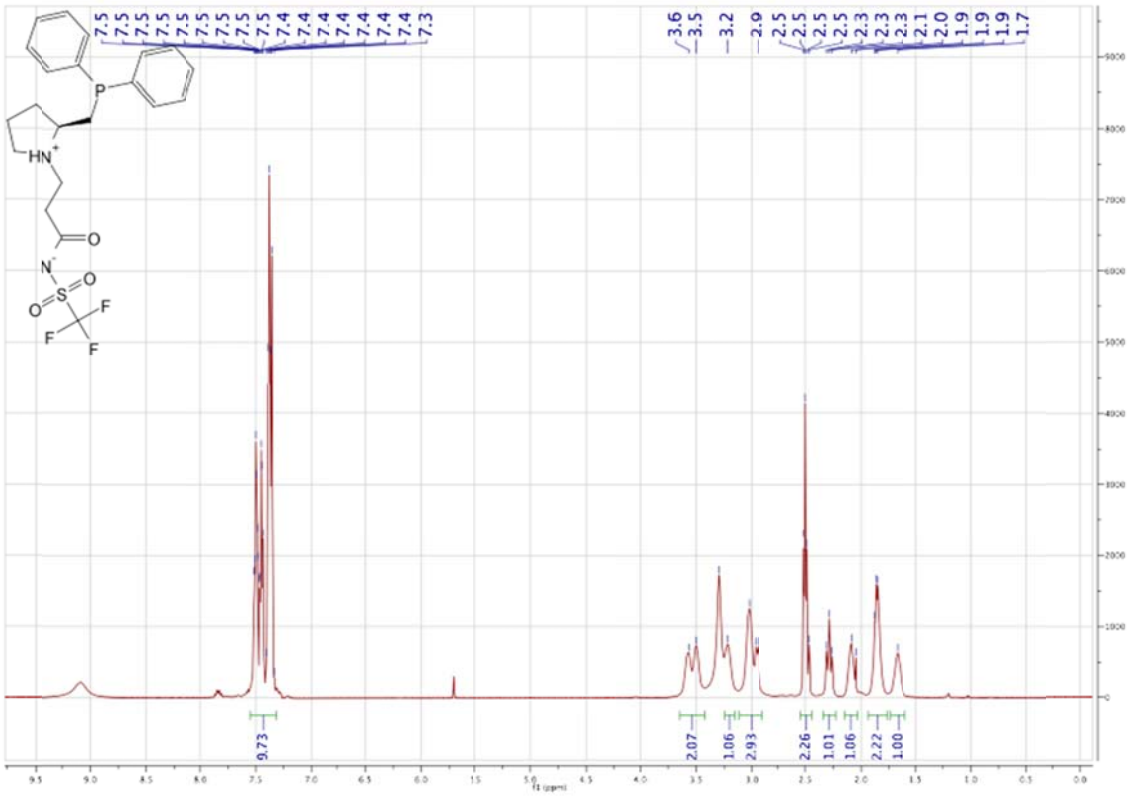
3-{(2*S*)-2-[(Diphenylphosphino)methyl]pyrrolidin-1-yl}propanoic acid

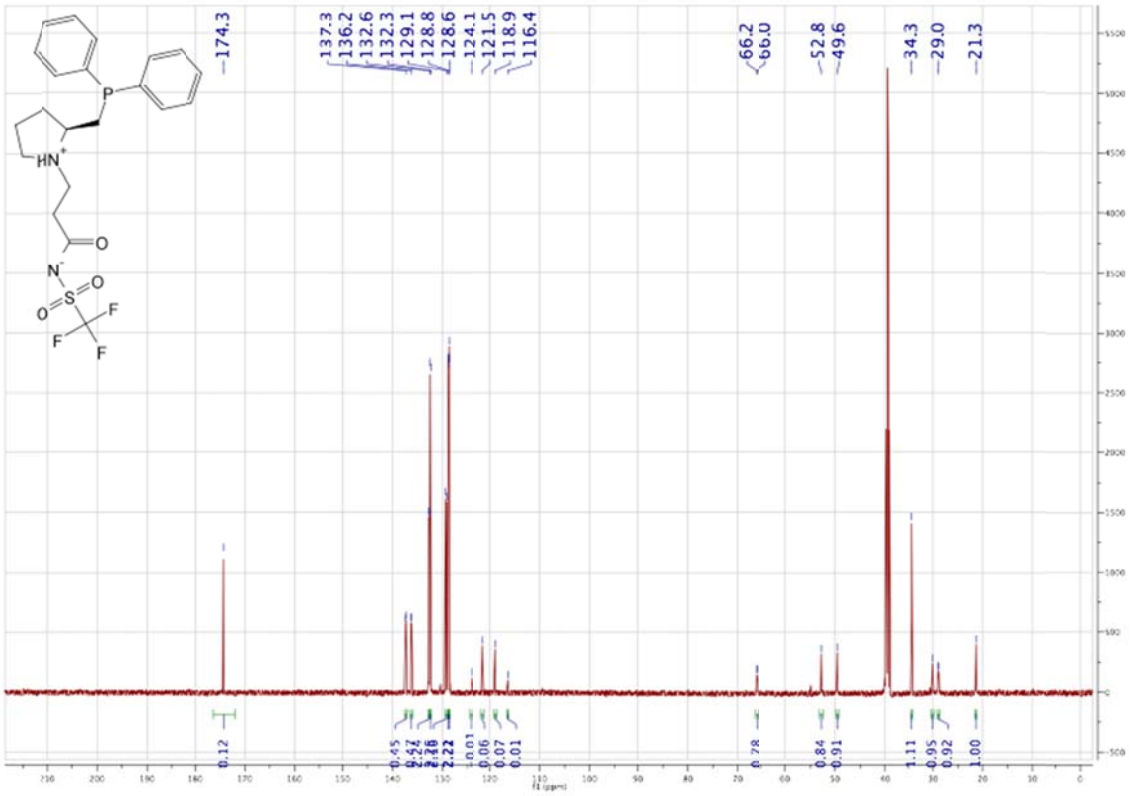


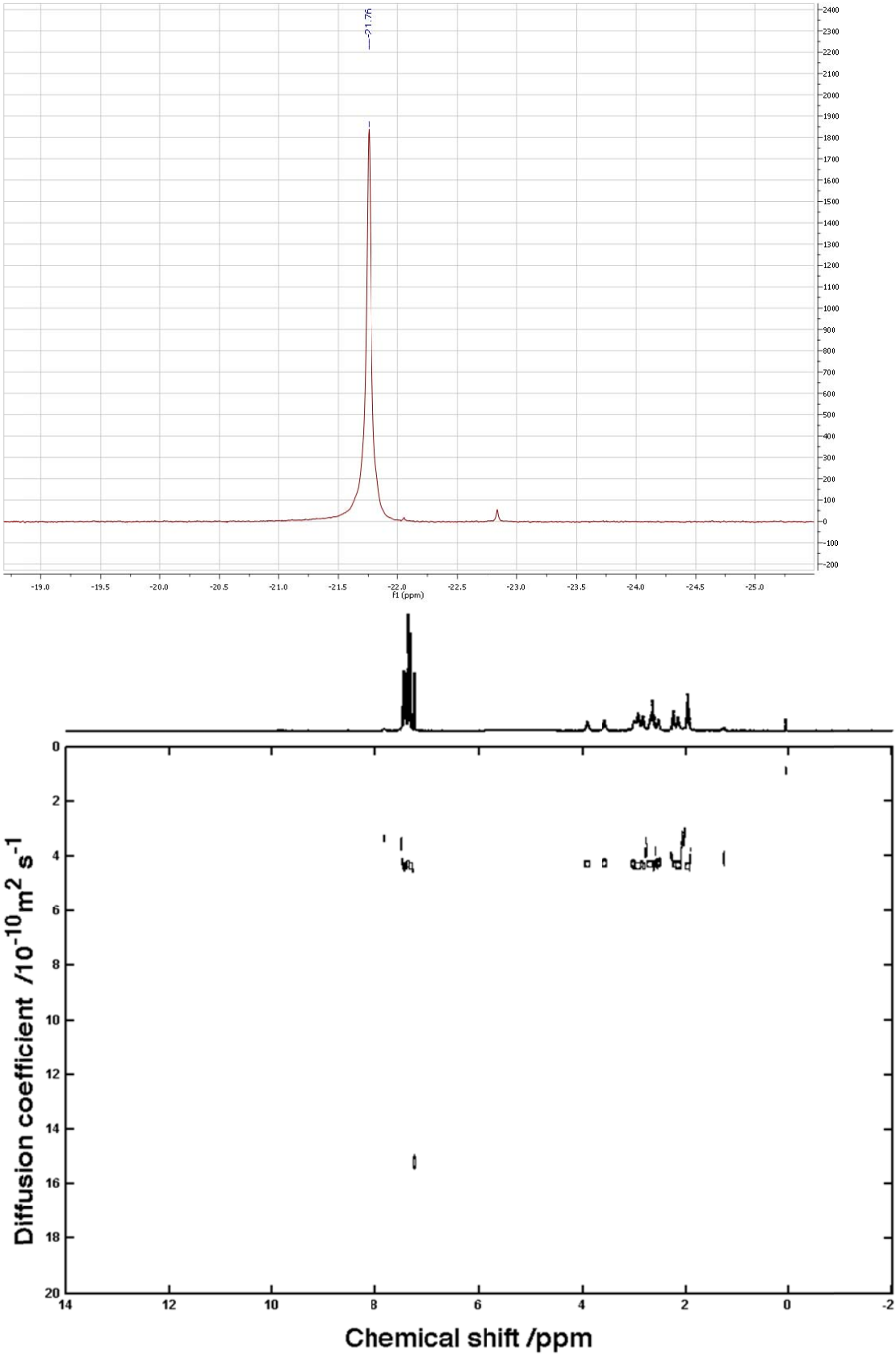




(3-((2S)-2-((Diphenylphosphino)methyl)pyrrolidin-1-ium-1-yl)propanoyl)((trifluoromethyl)sulfonyl)amide (15)







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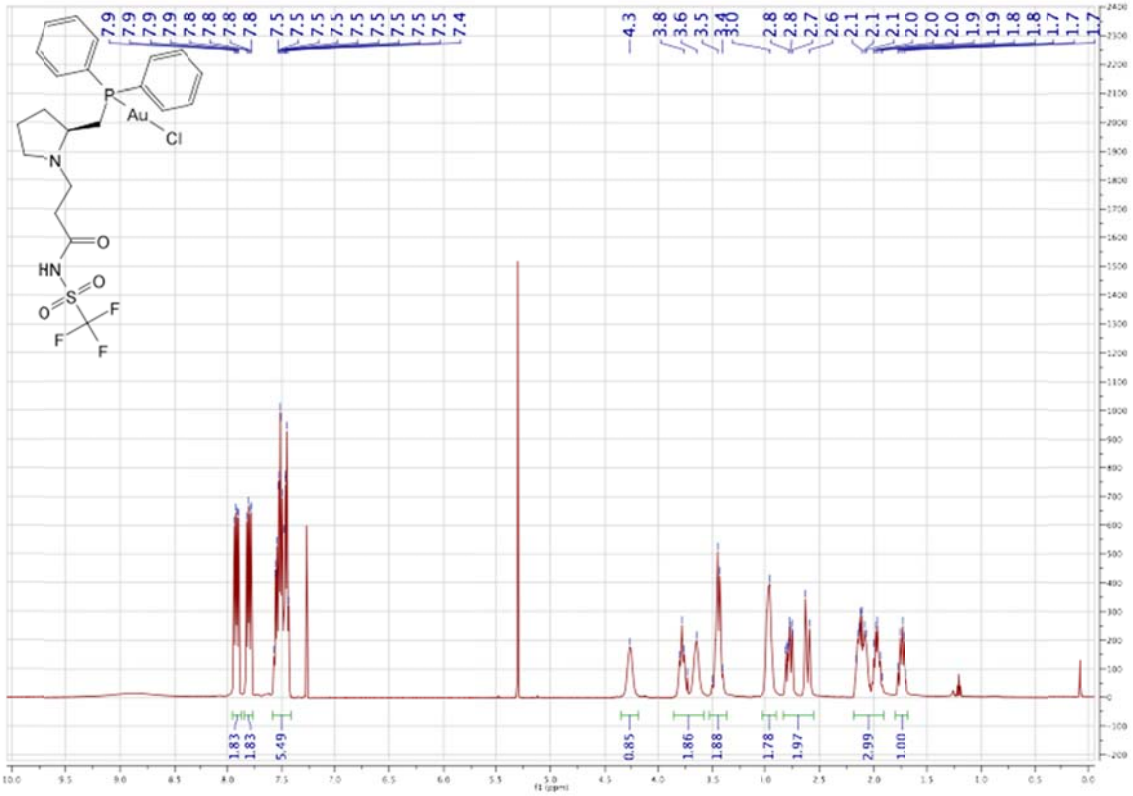
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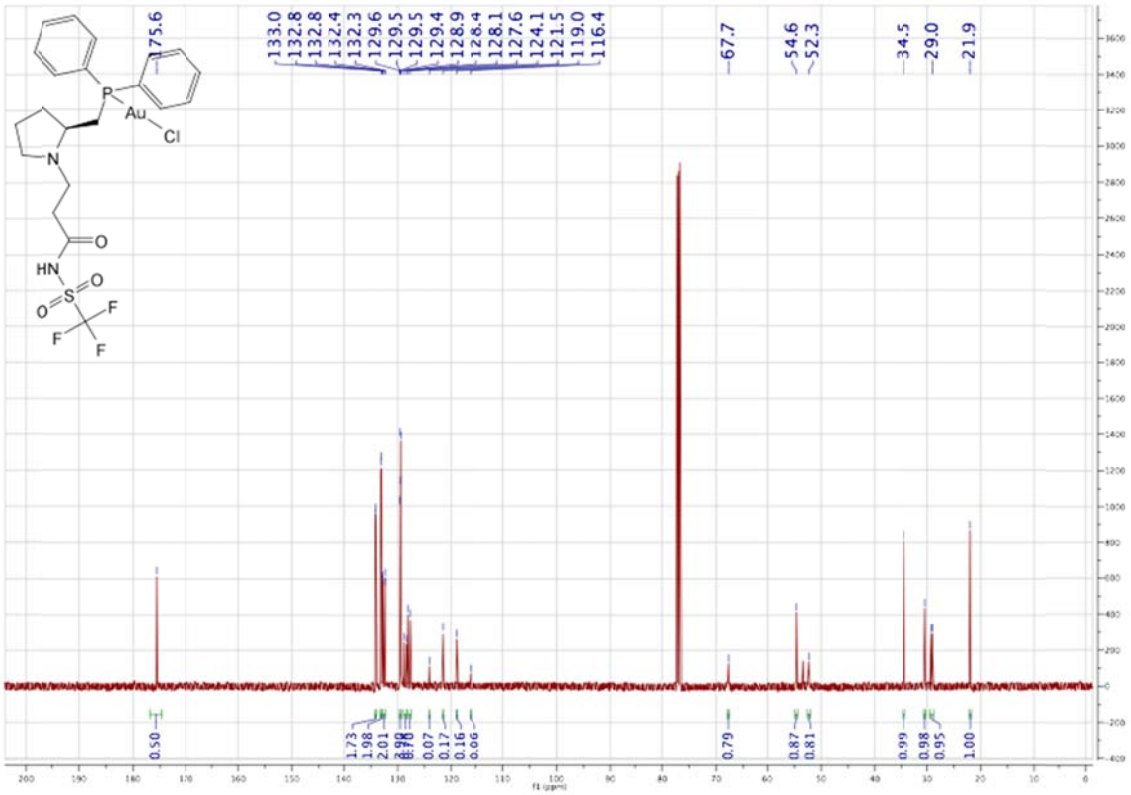
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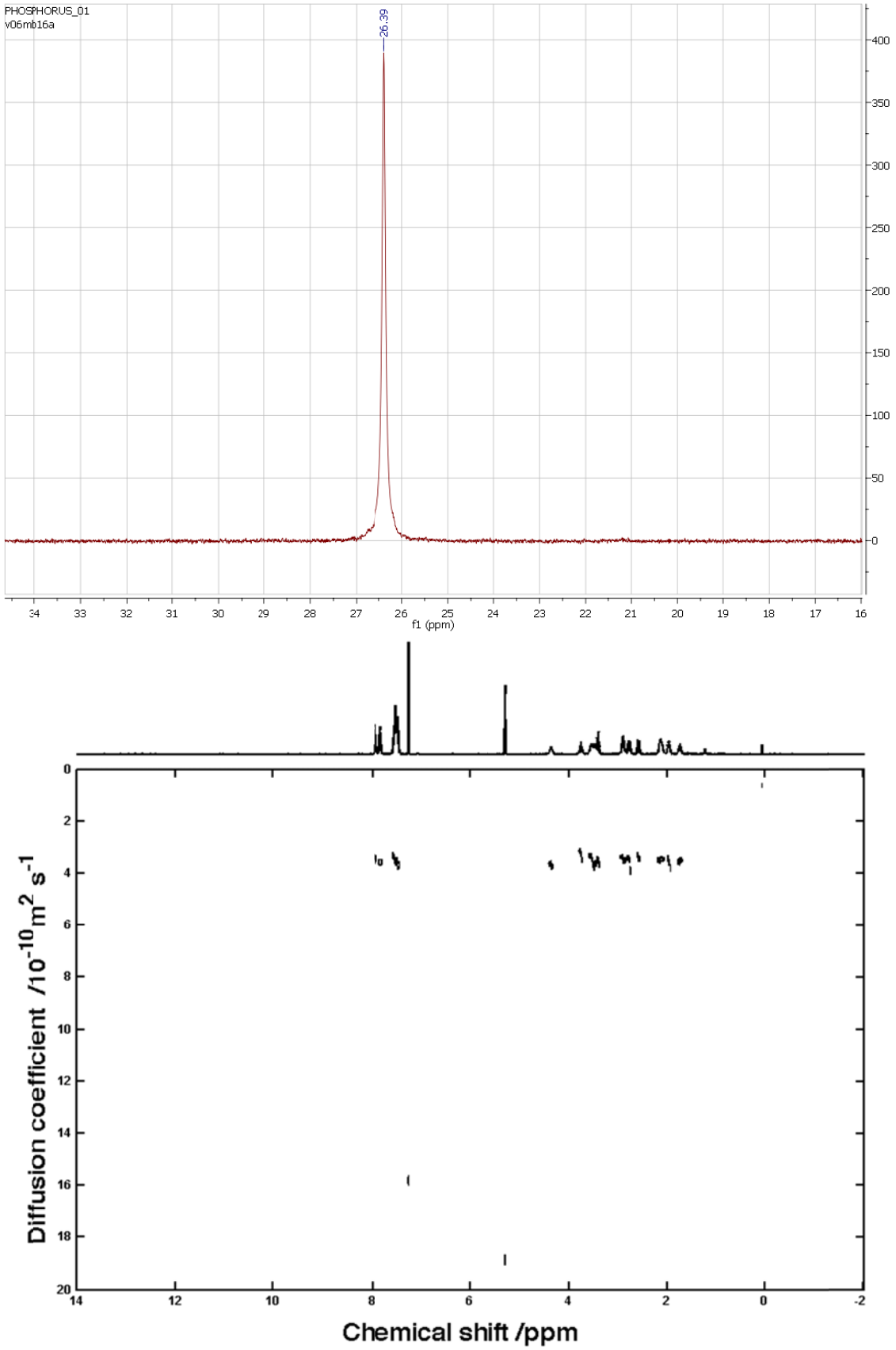

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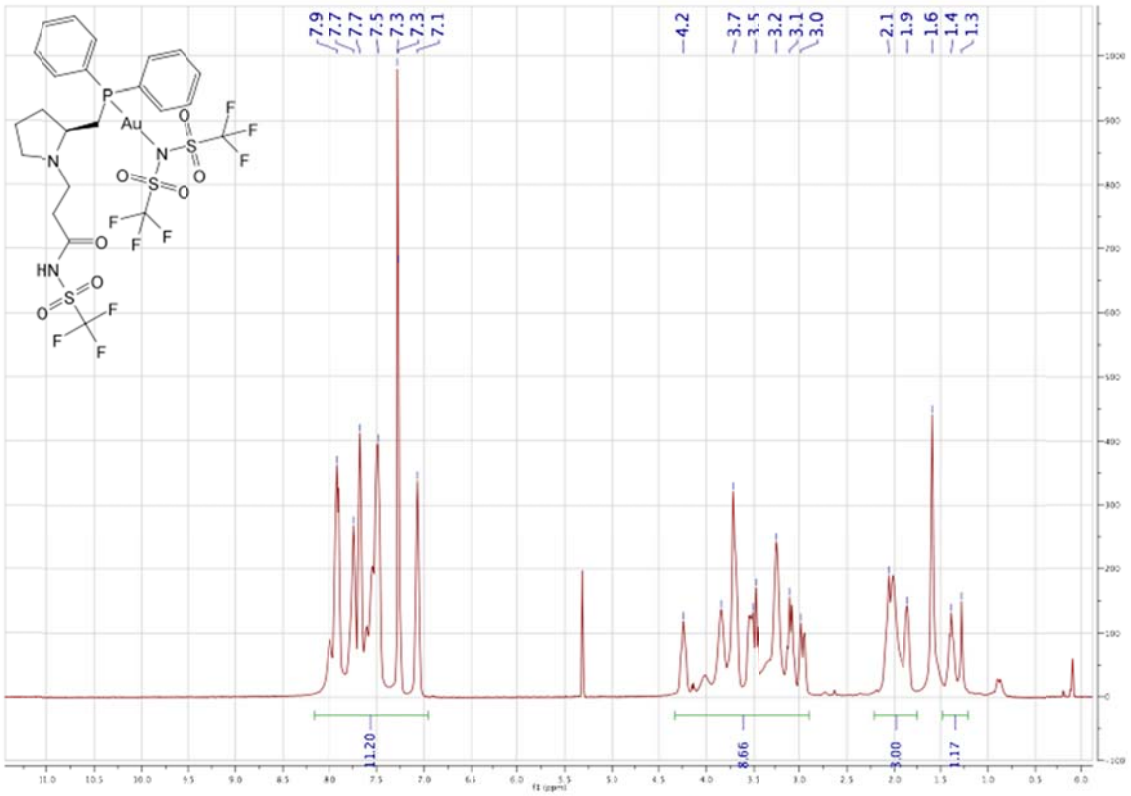
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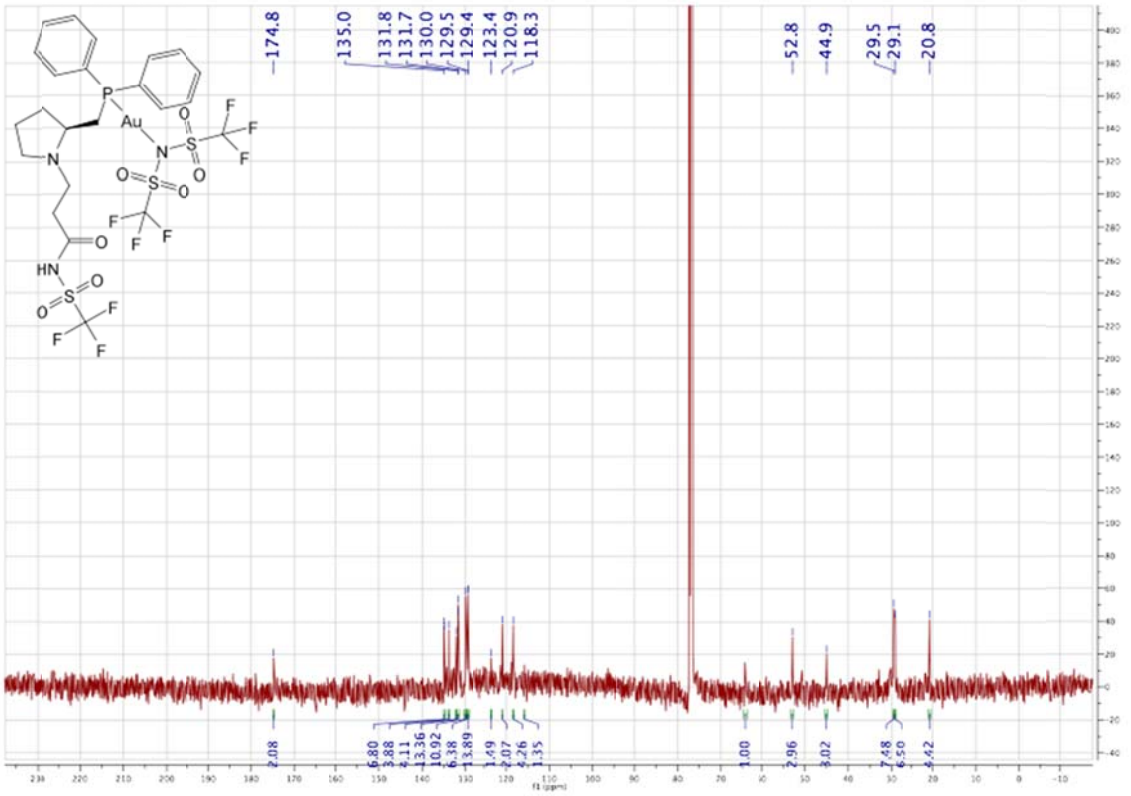
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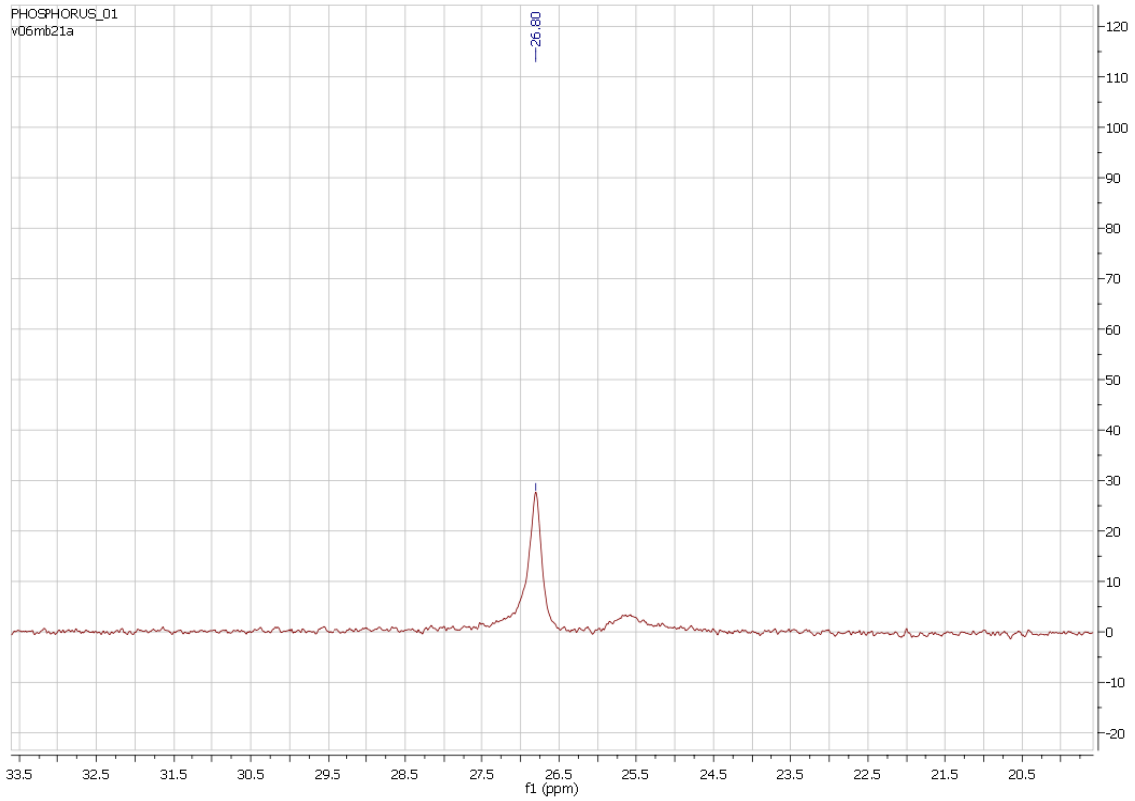

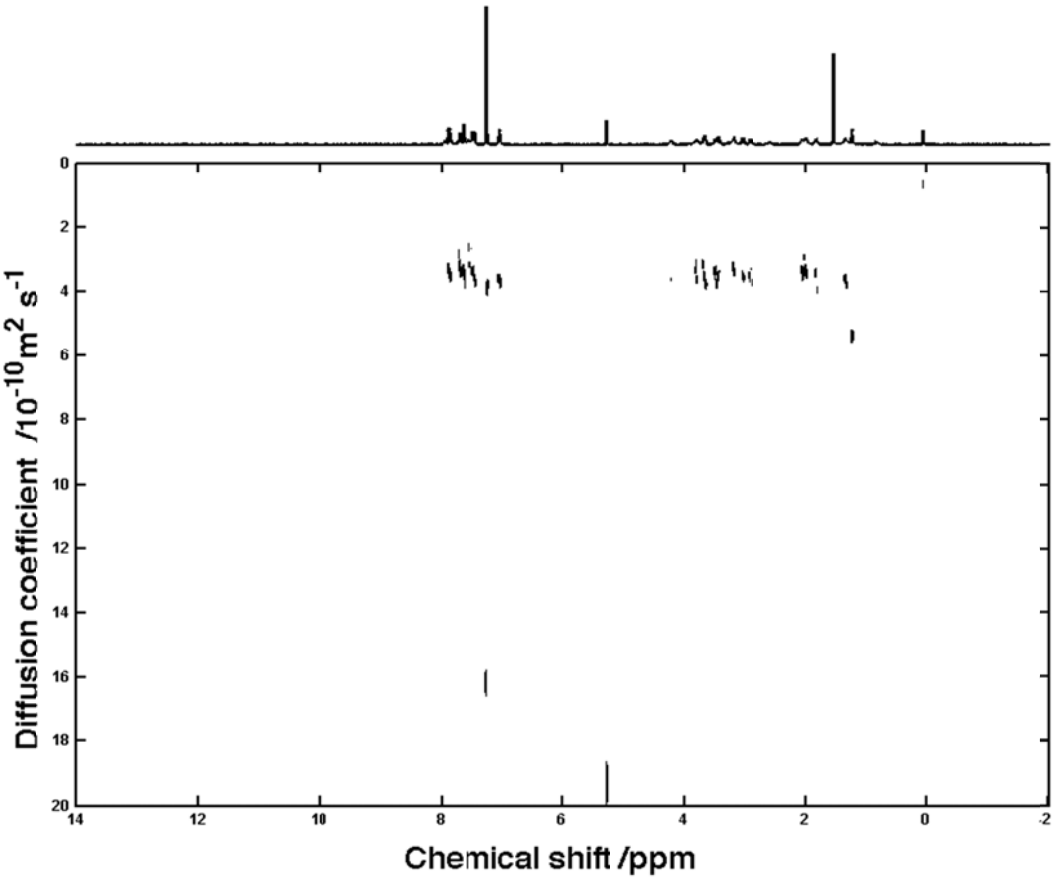

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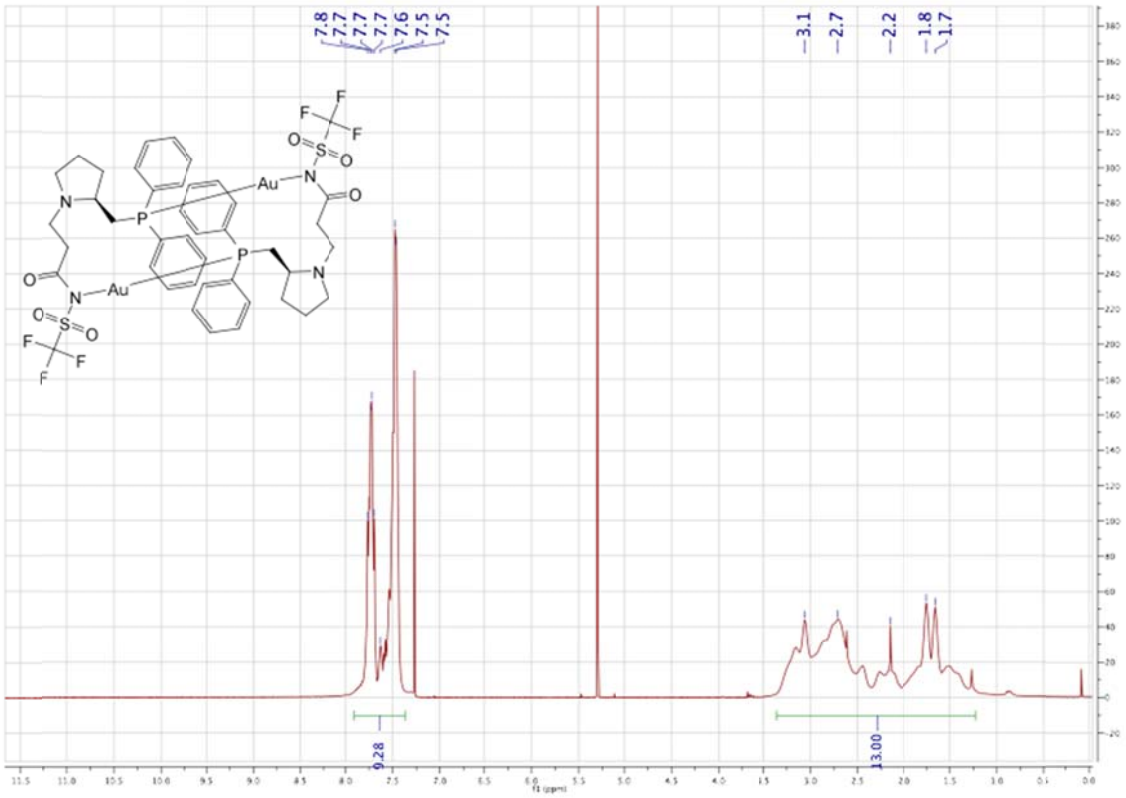
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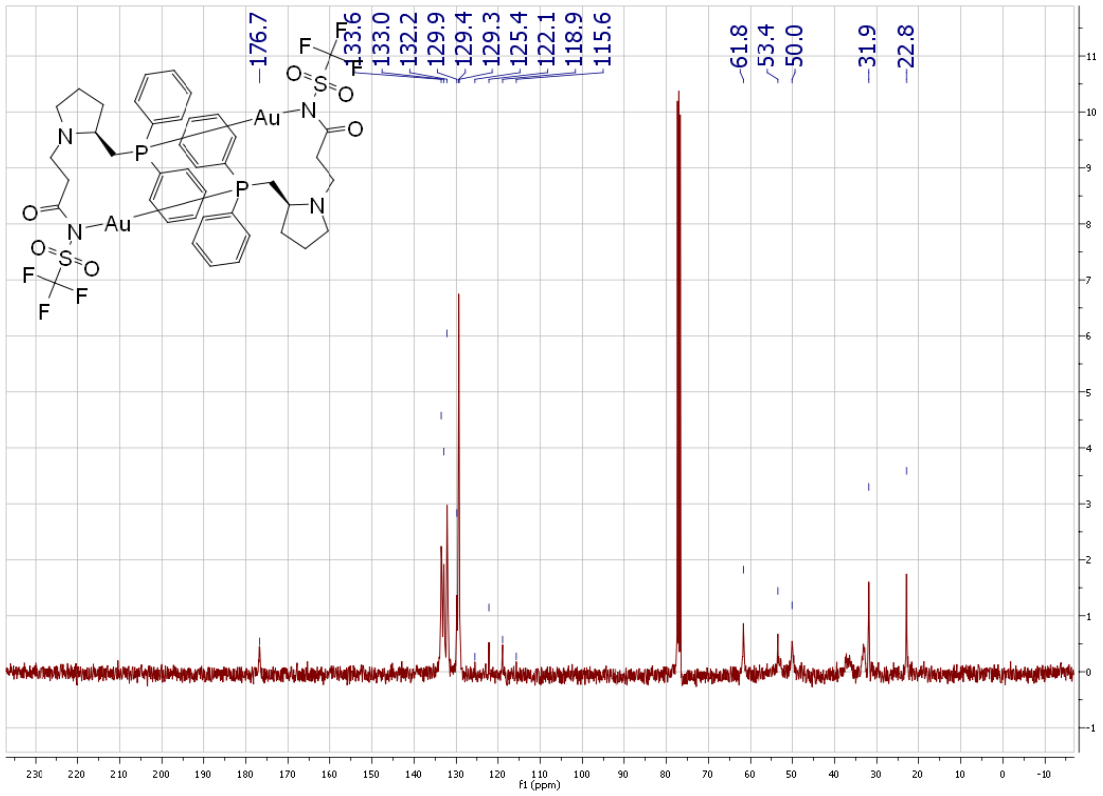
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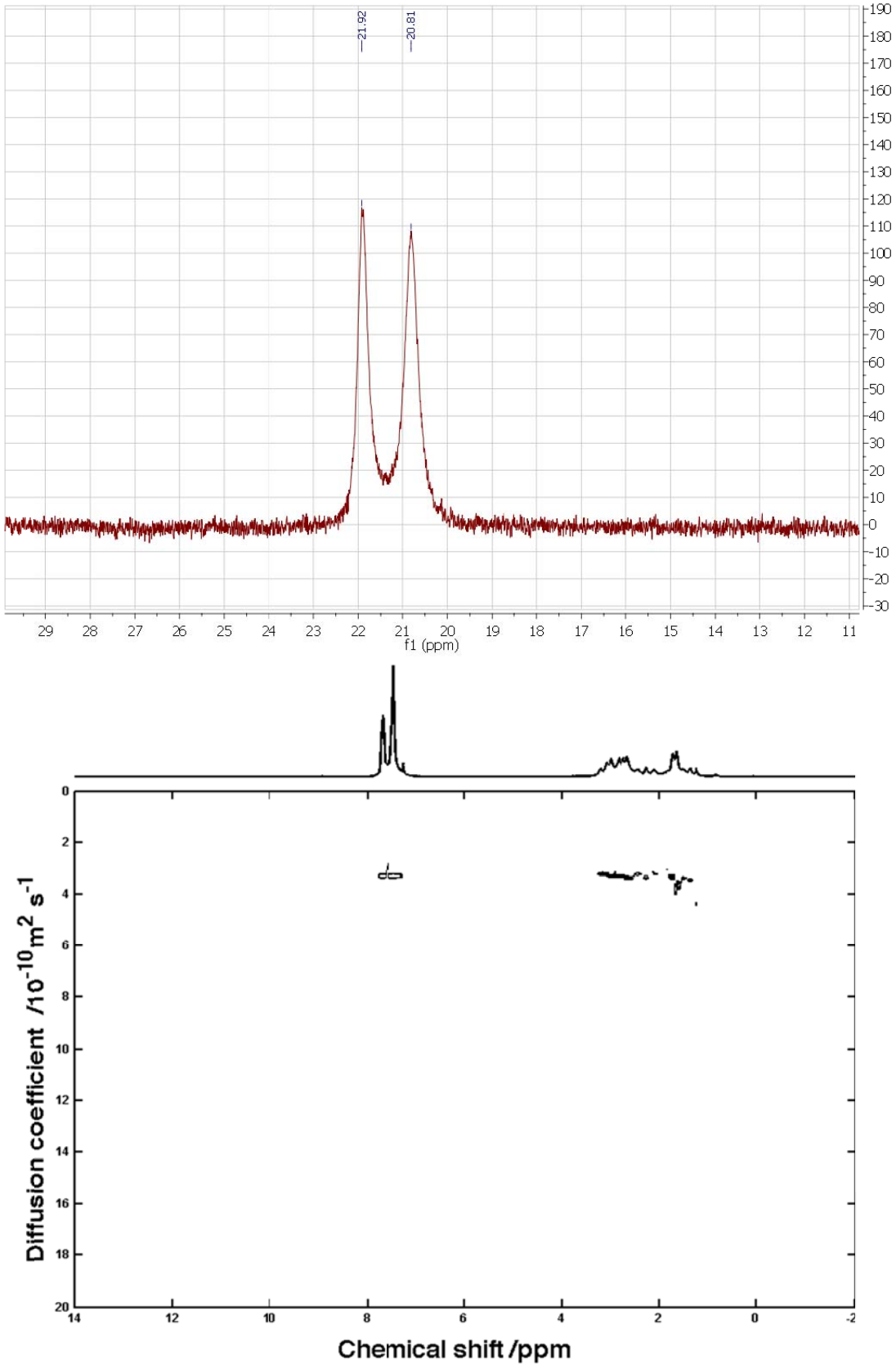
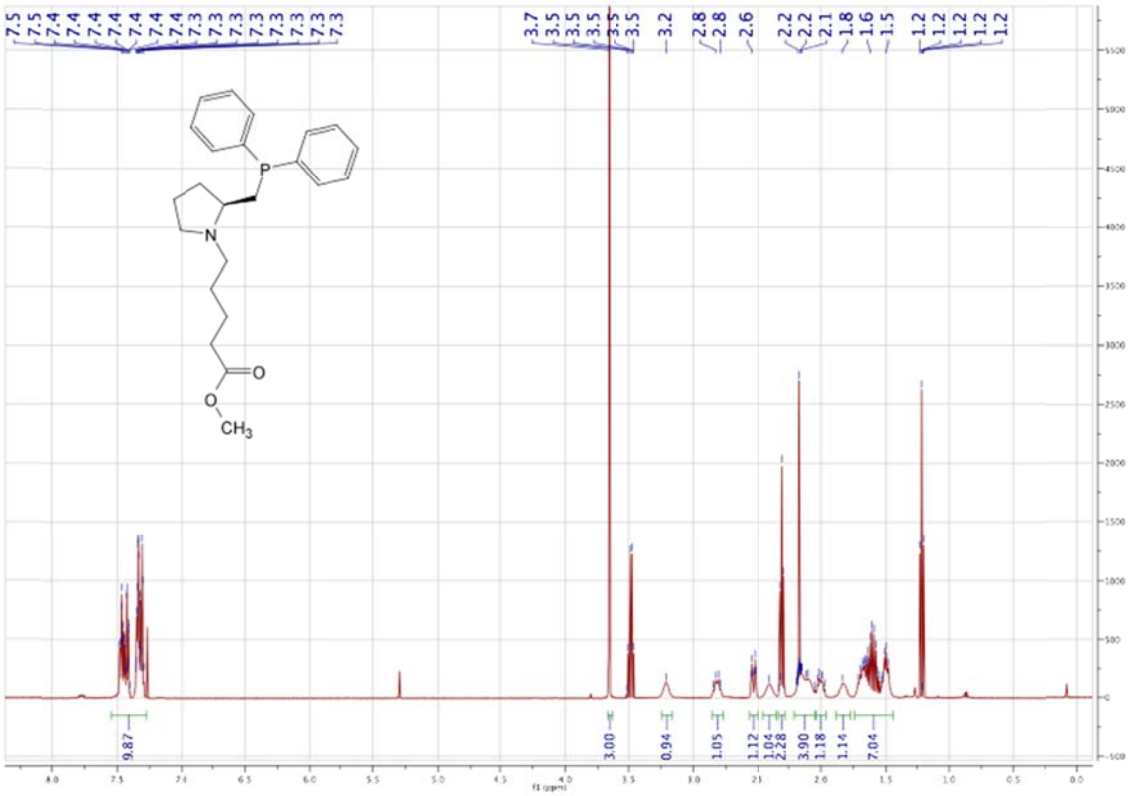


(*S*)-Methyl 5-(2-((diphenylphosphino)methyl)pyrrolidin-1-yl)pentanoate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O
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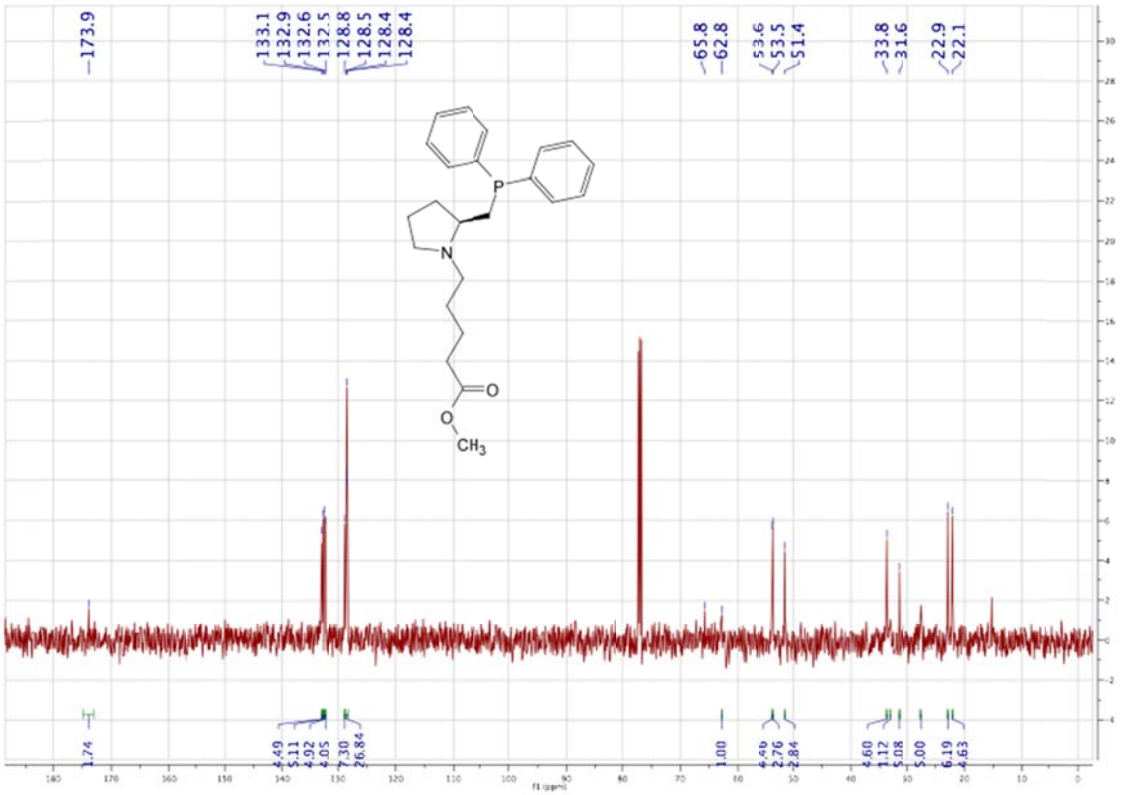
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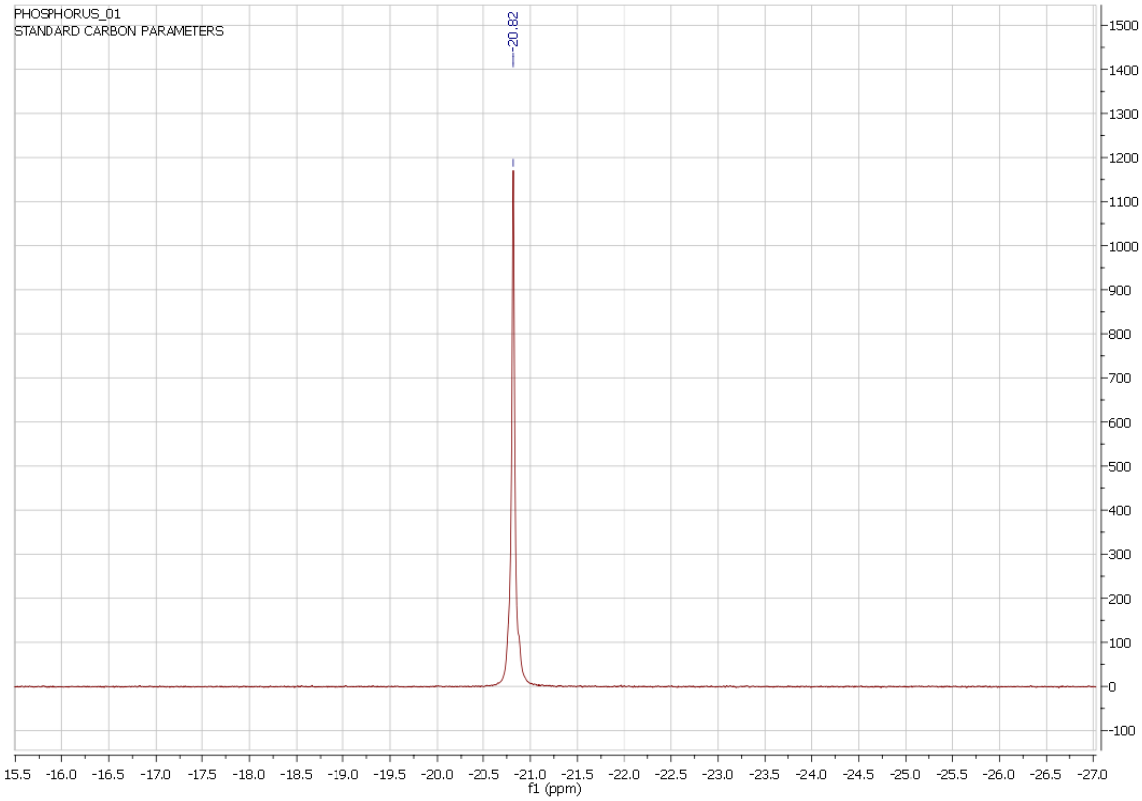
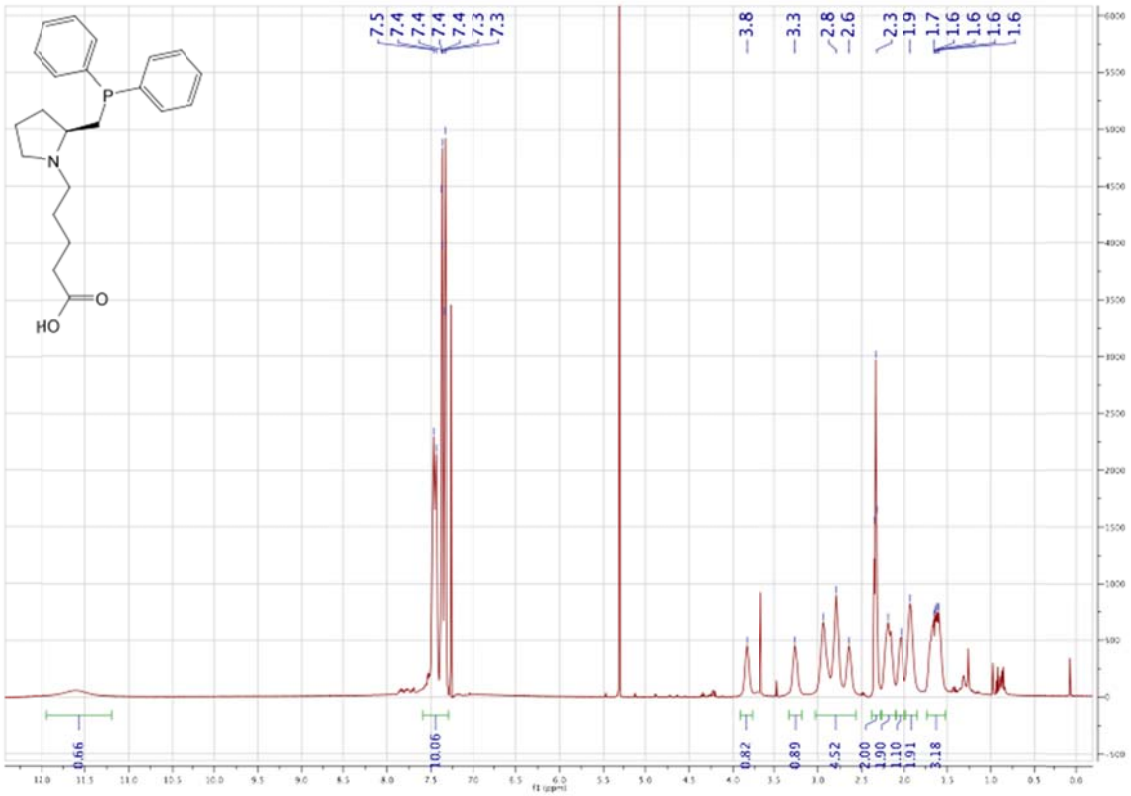
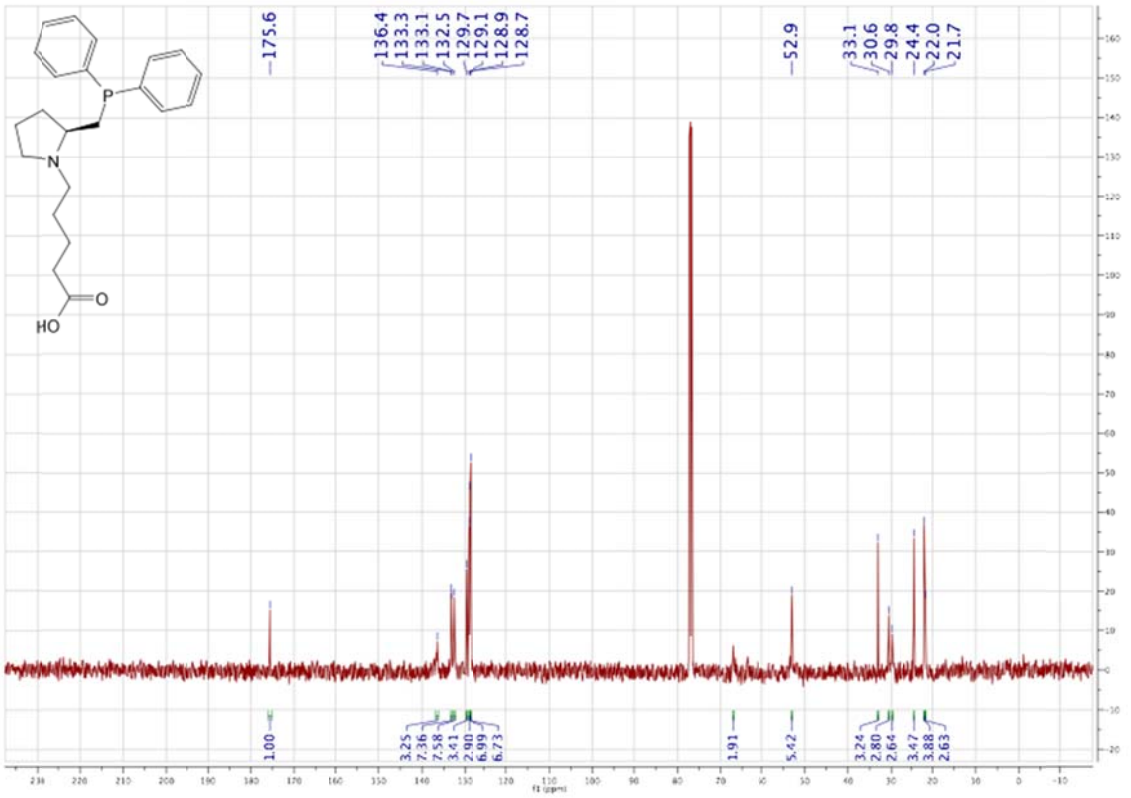


(*S*)-5-(2-((Diphenylphosphino)methyl)pyrrolidin-1-yl)pentanoic acid





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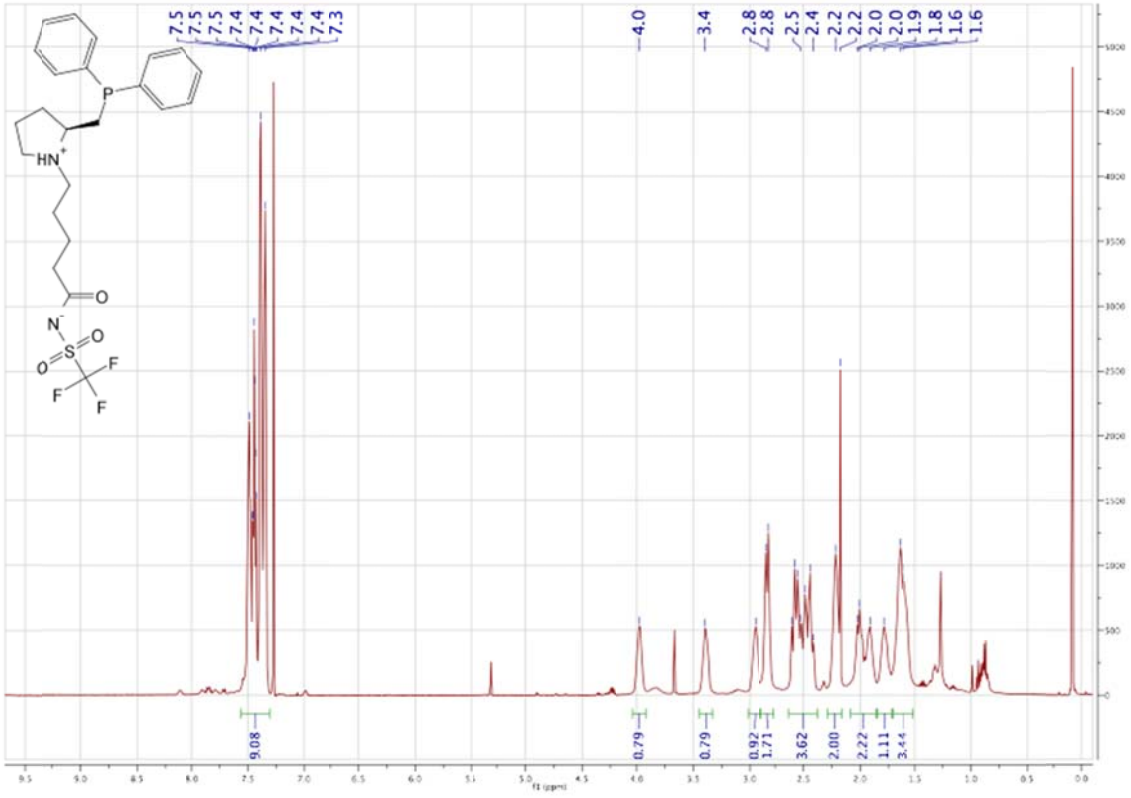

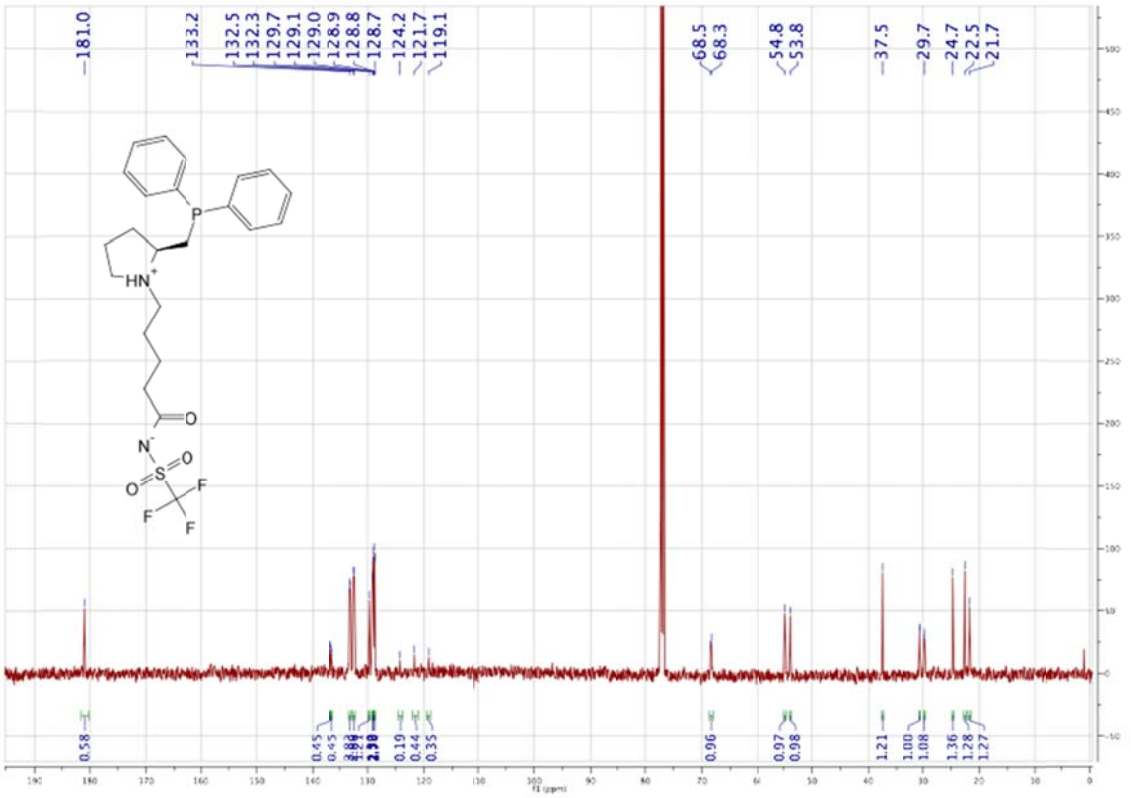
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21.7
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N-
S
O
F
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0.45
0.45
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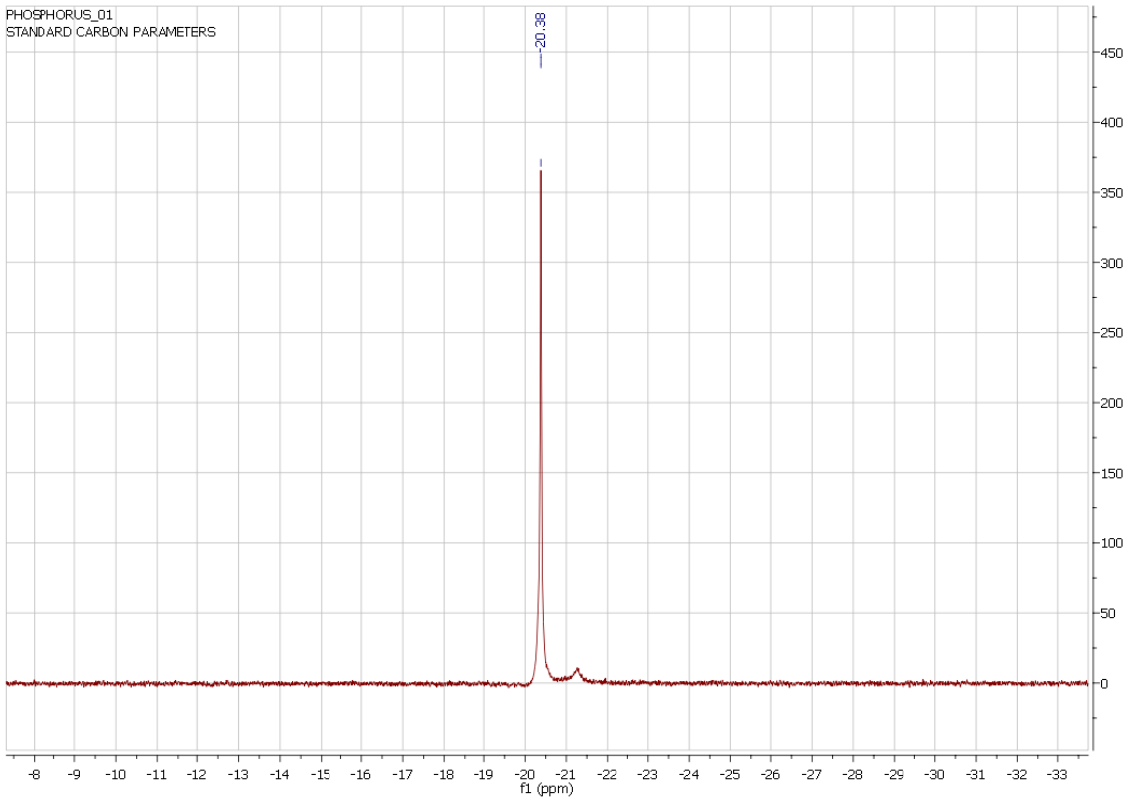
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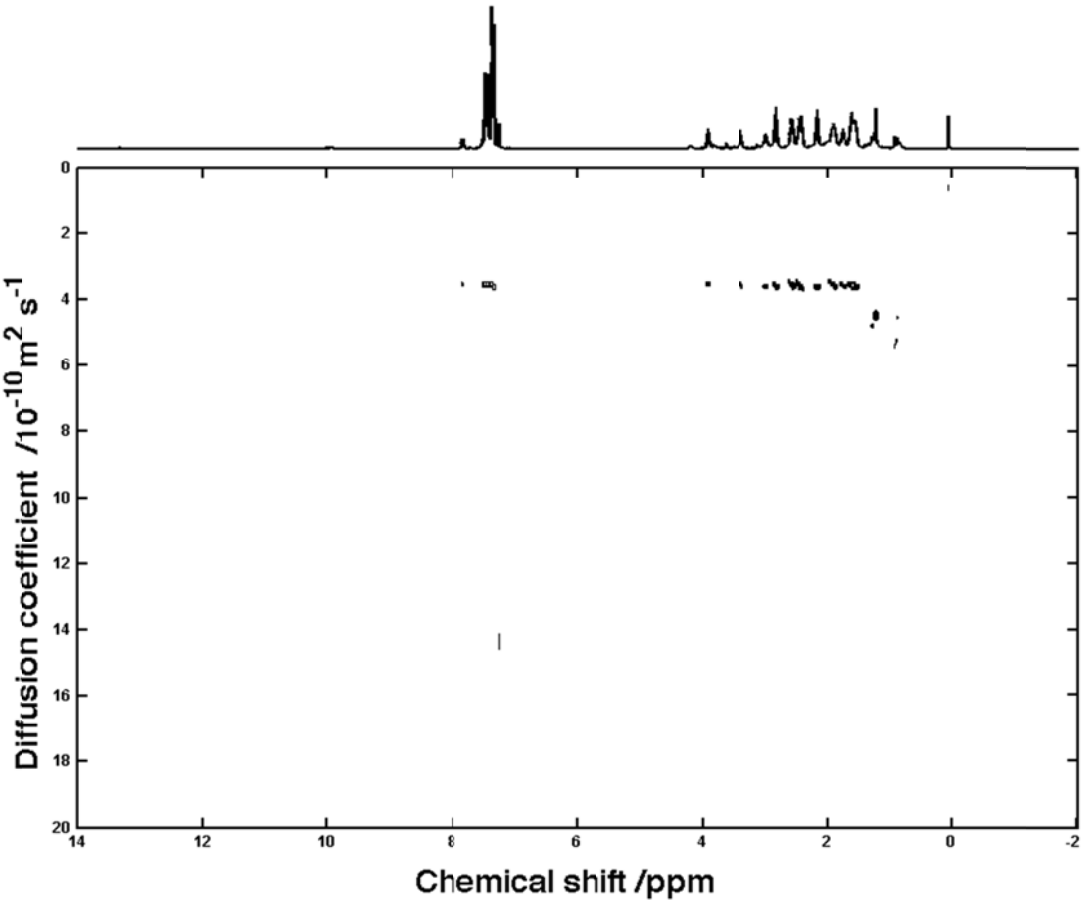
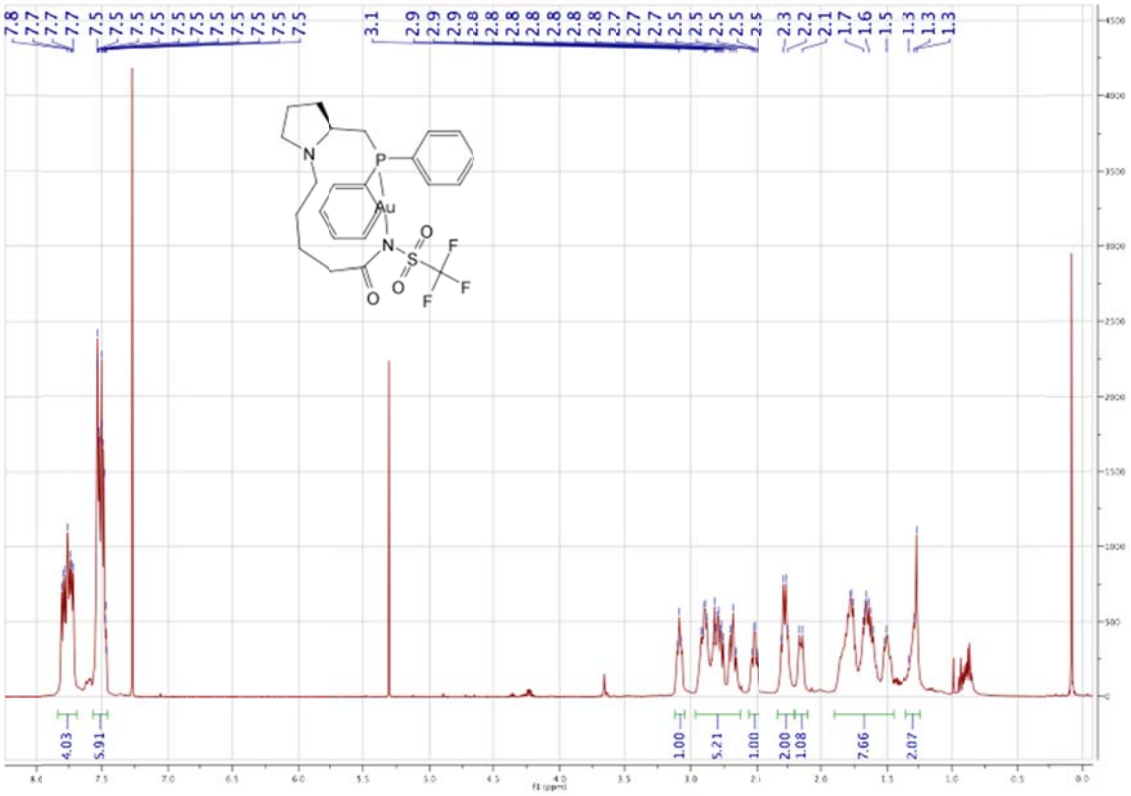
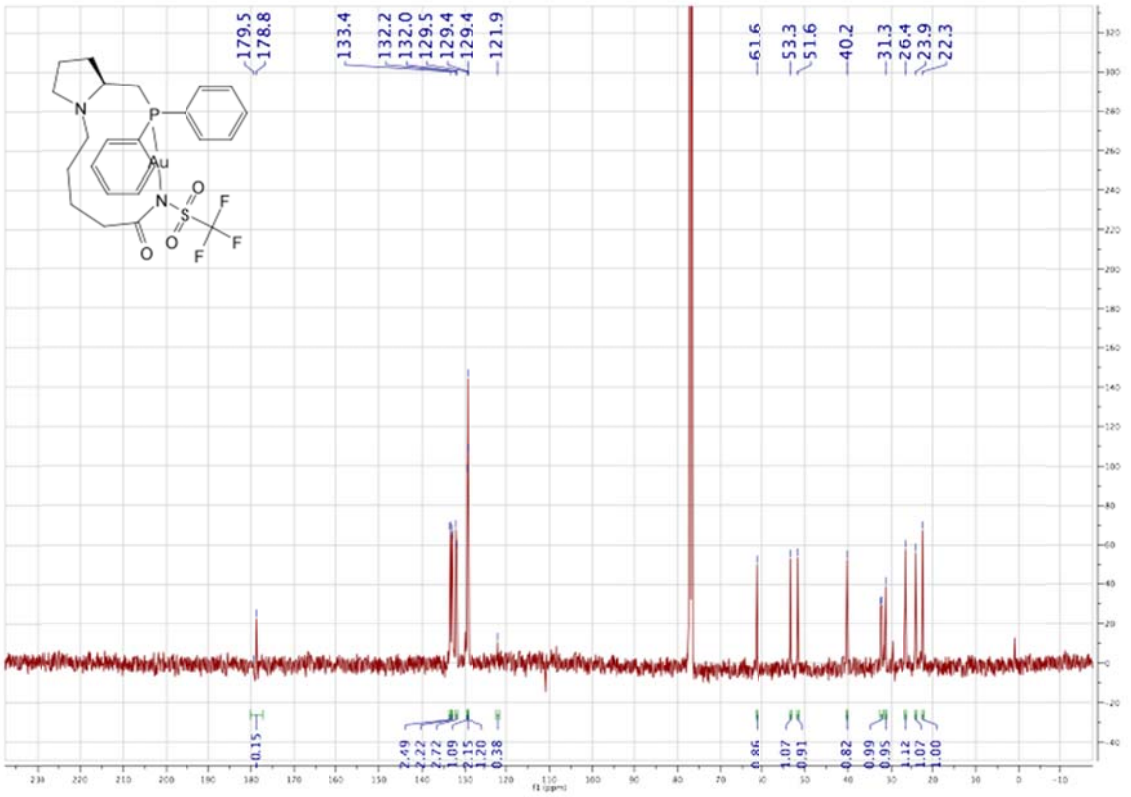
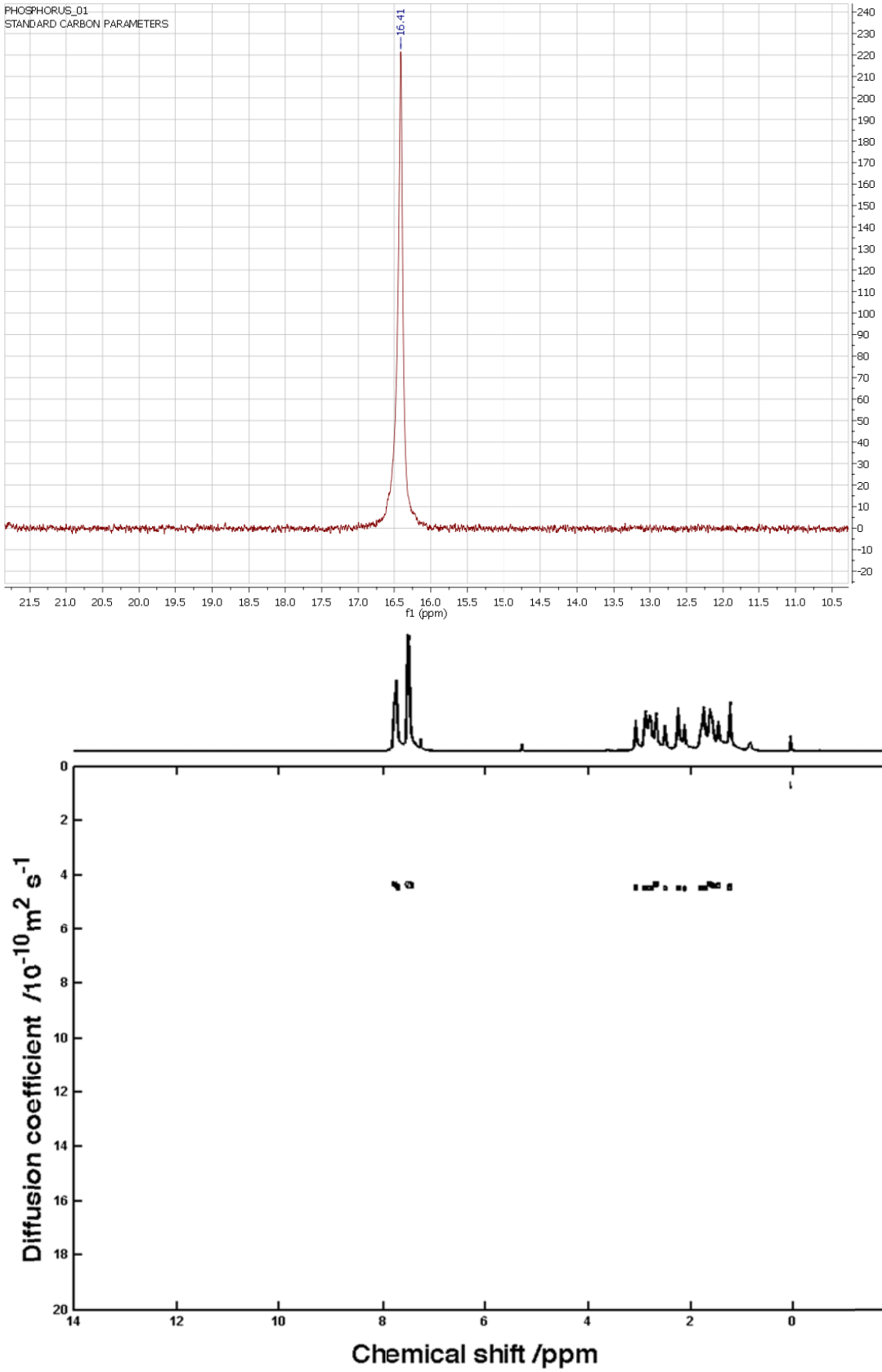


Bidentate gold(I) complex (6a/6b)







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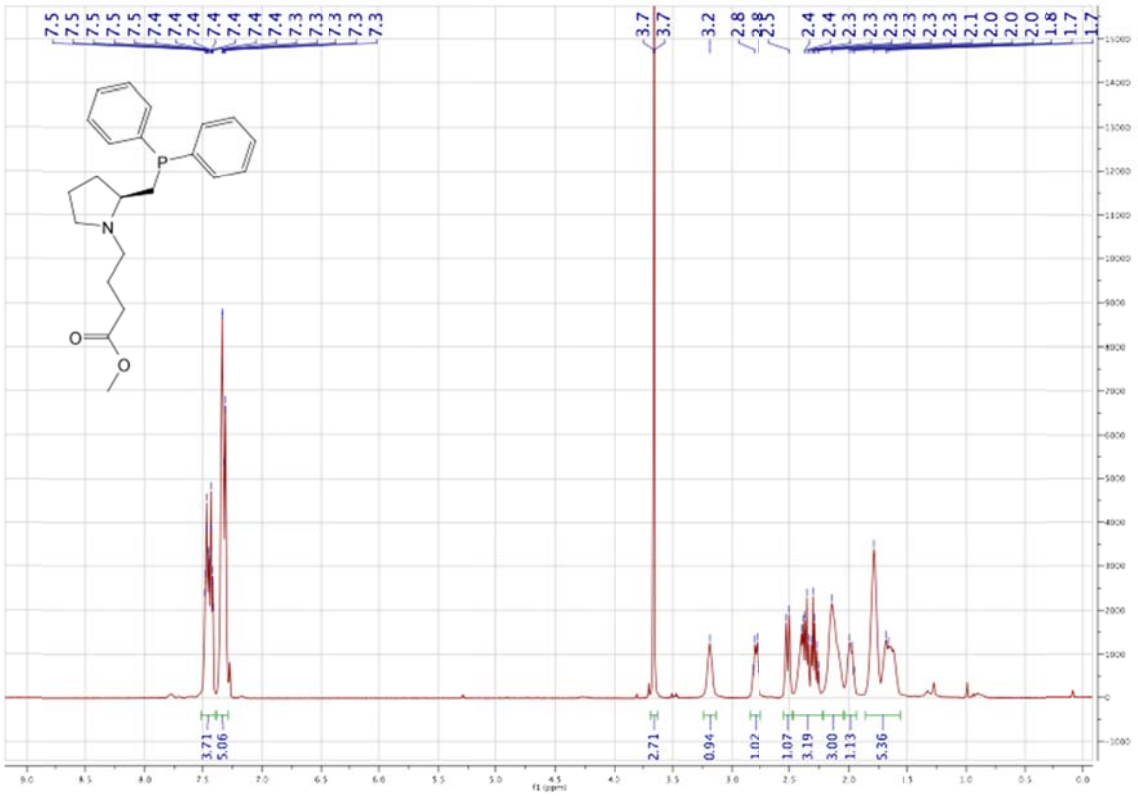
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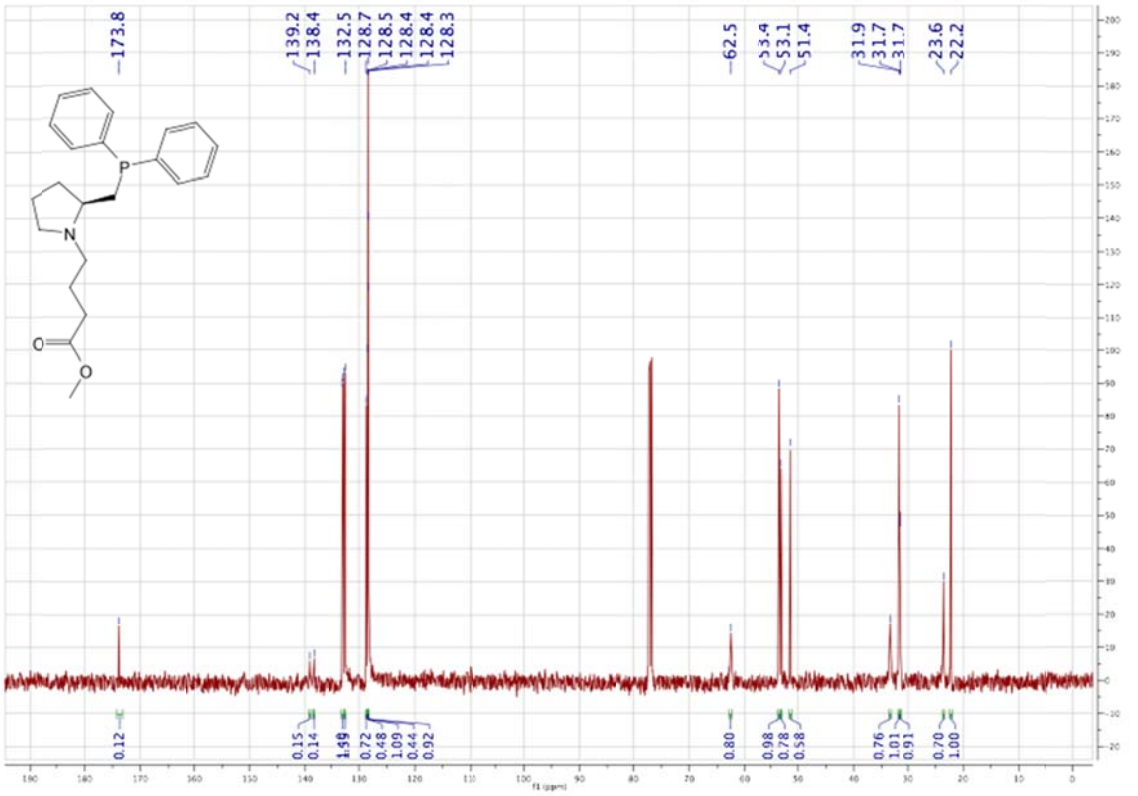
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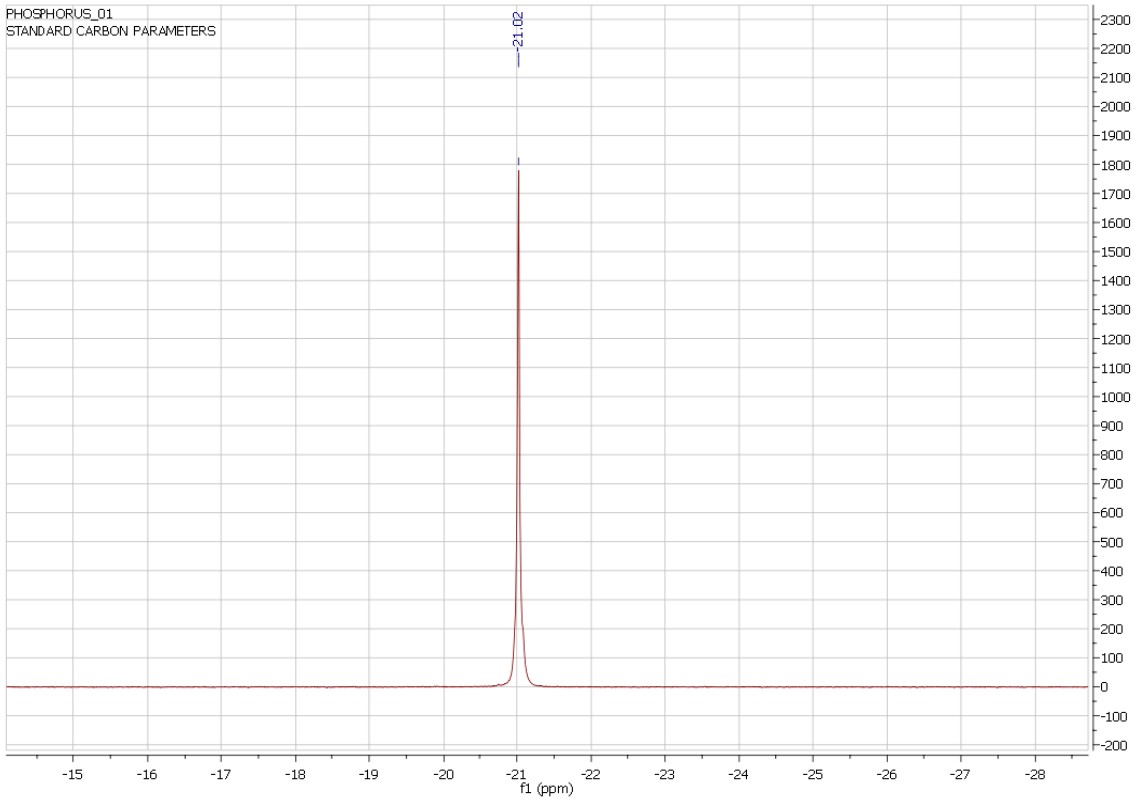
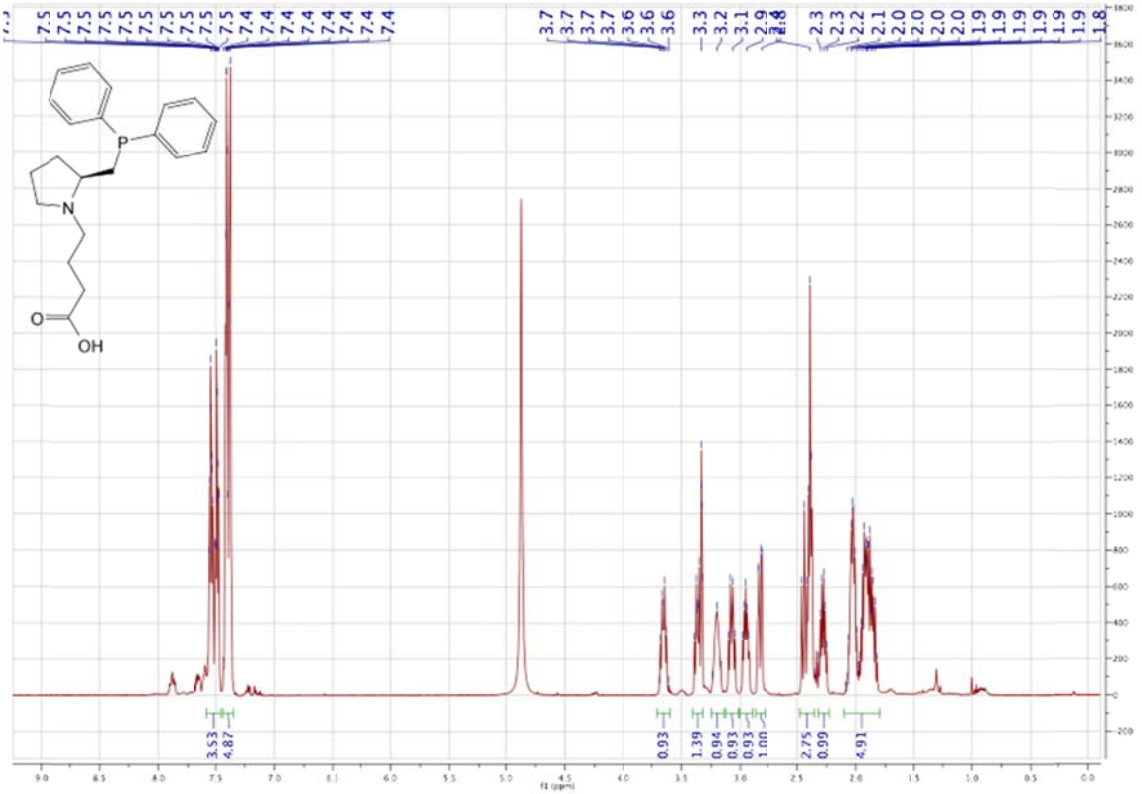
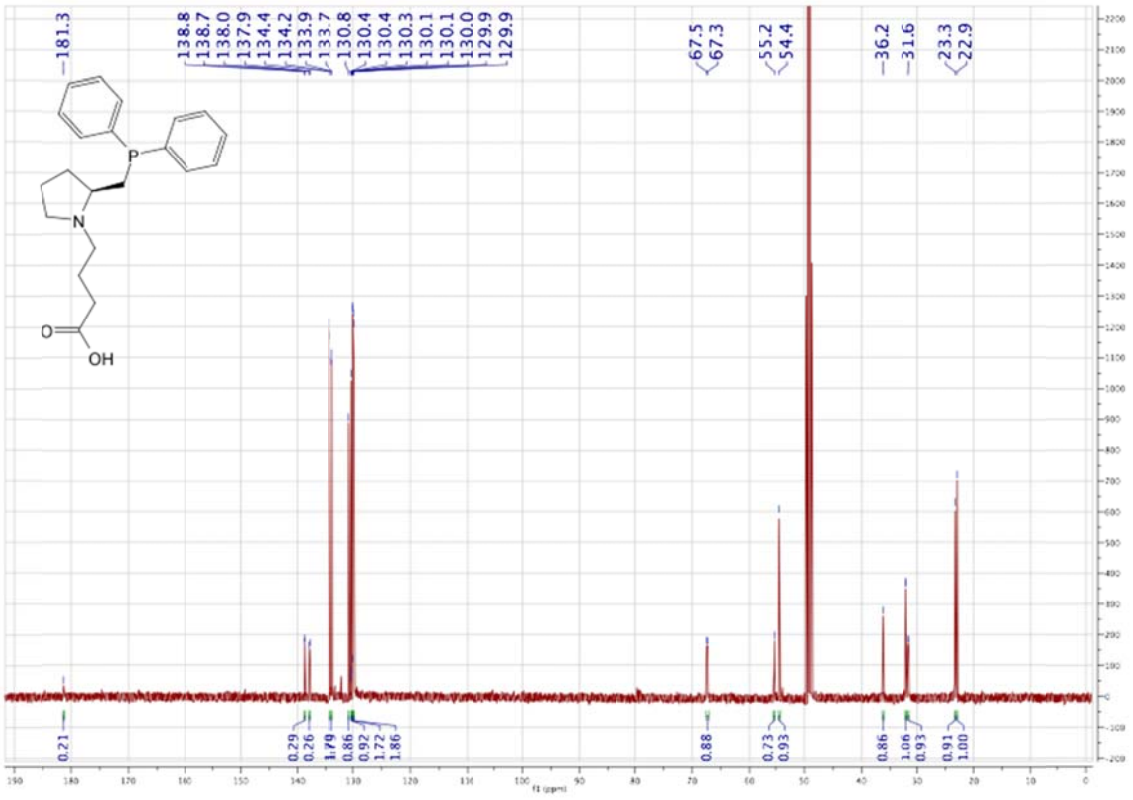
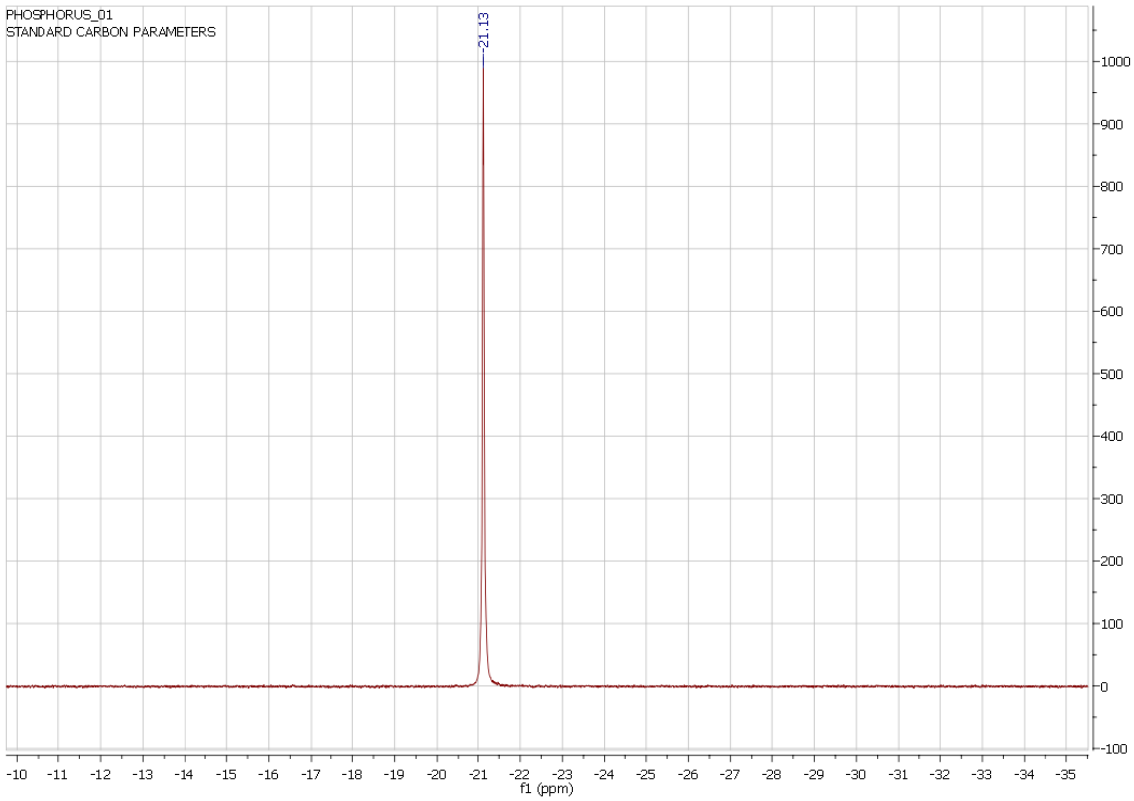


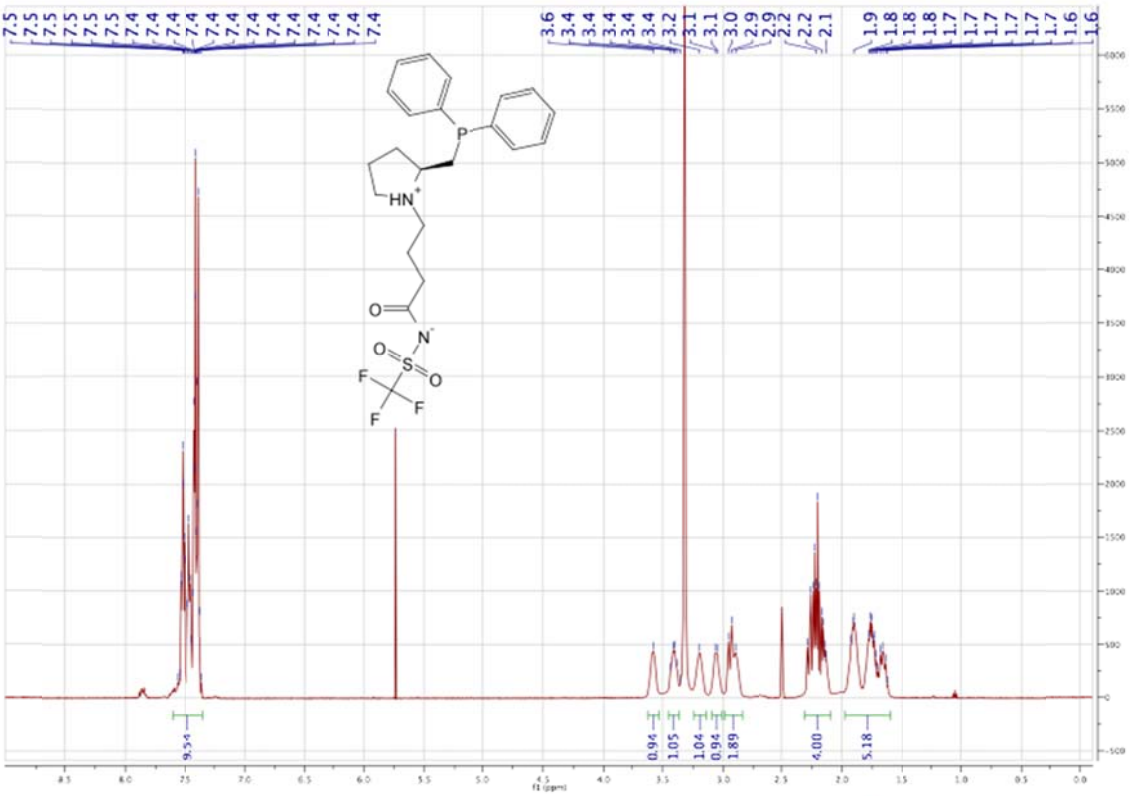
(S)-4-(2-((Diphenylphosphino)methyl)pyrrolidin-1-yl)butanoic acid

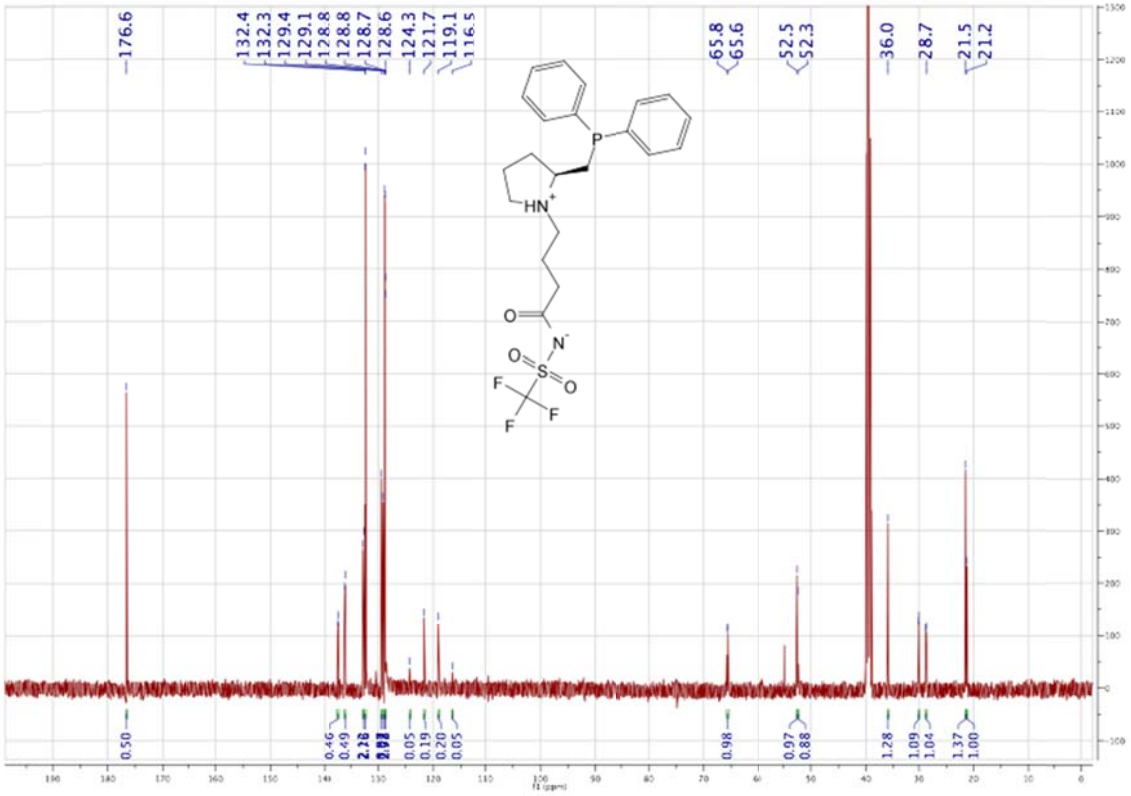


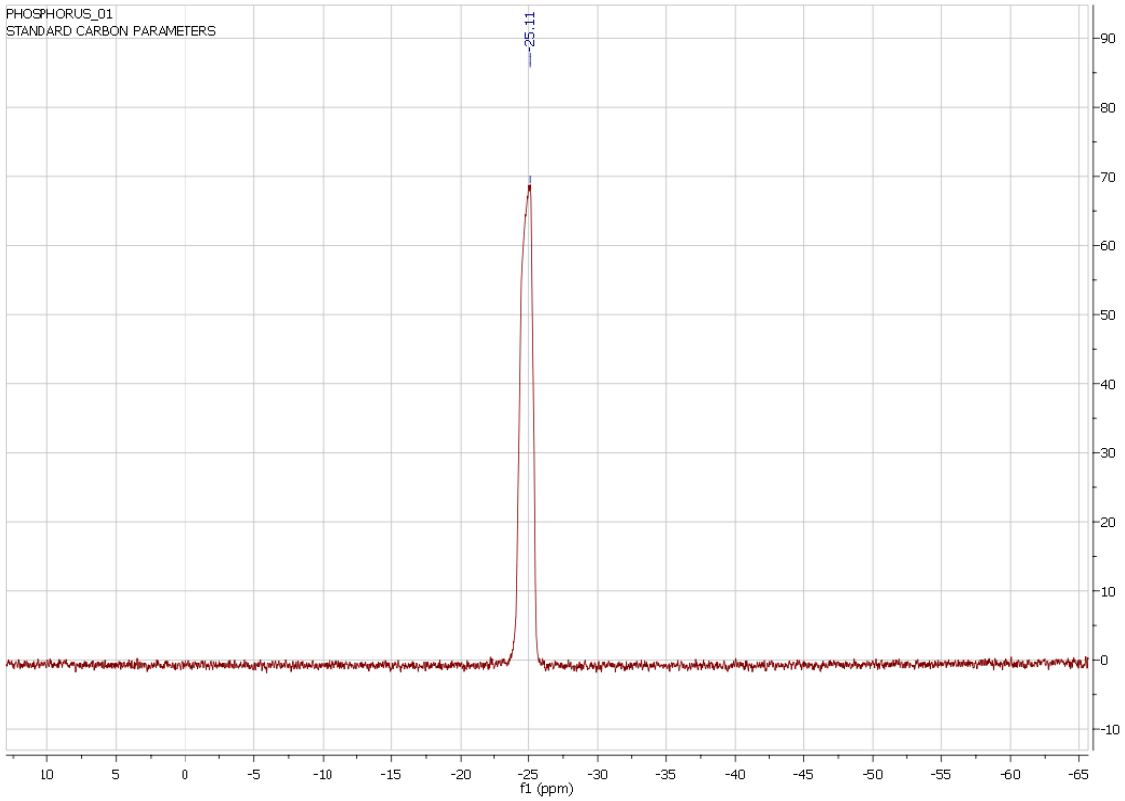




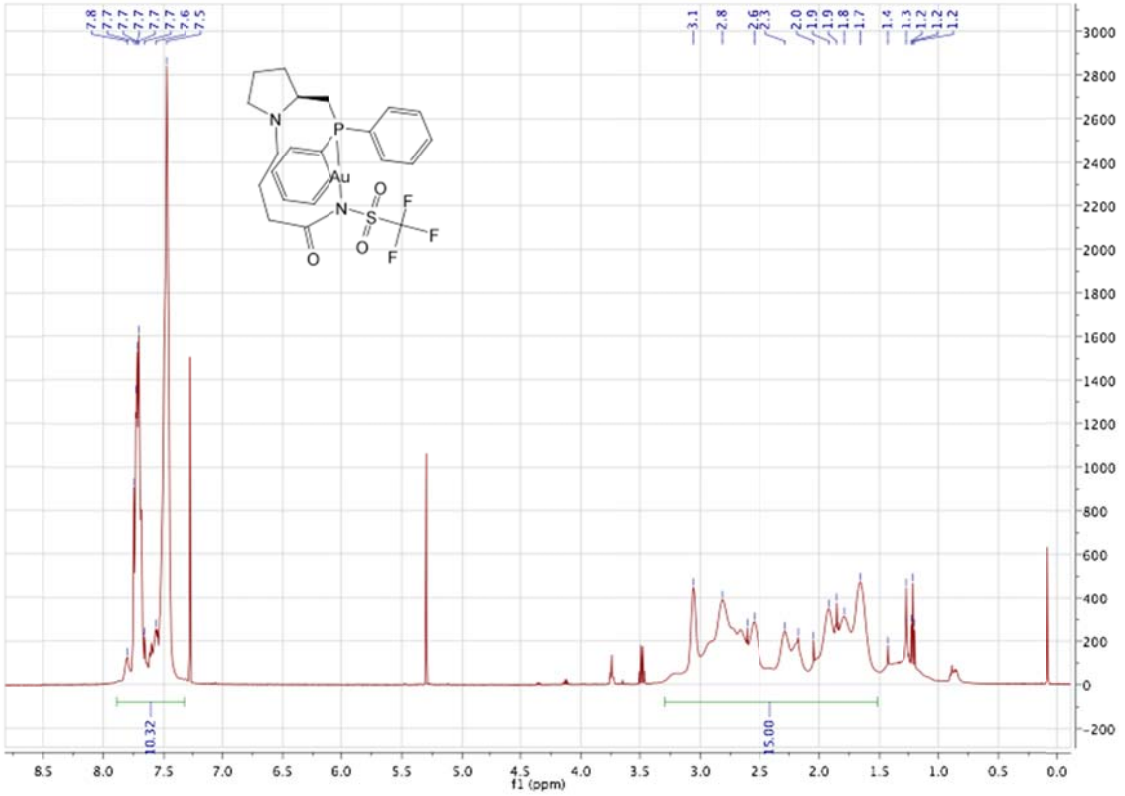
(4-((2S)-2-((Diphenylphosphino)methyl)pyrrolidin-1-ium-1-yl)butanoyl)((trifluoromethyl)sulfonyl)amide (22)

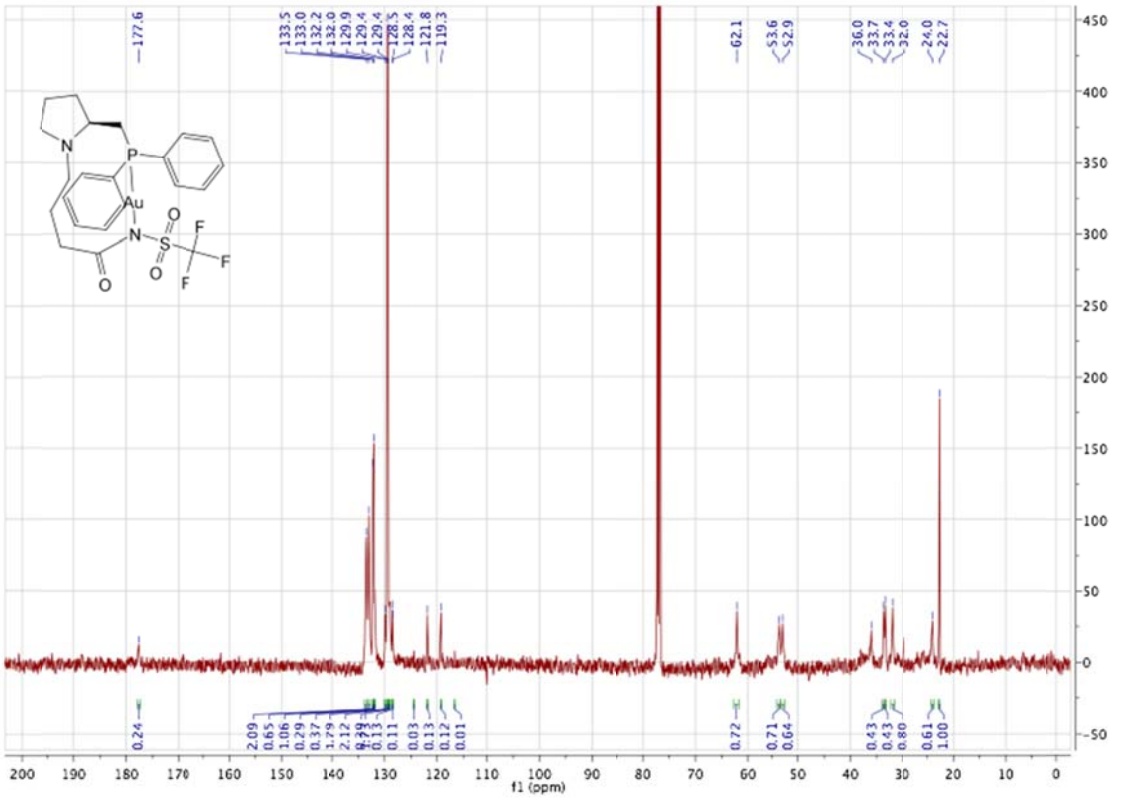

7.5
7.4
3.6
3.4
3.2
3.1
3.0
2.9
2.2
2.1
1.9
1.8
1.7
1.6
HN
P
O
N
S
F
9.34
0.94
1.05
1.04
0.94
1.89
4.00
5.18

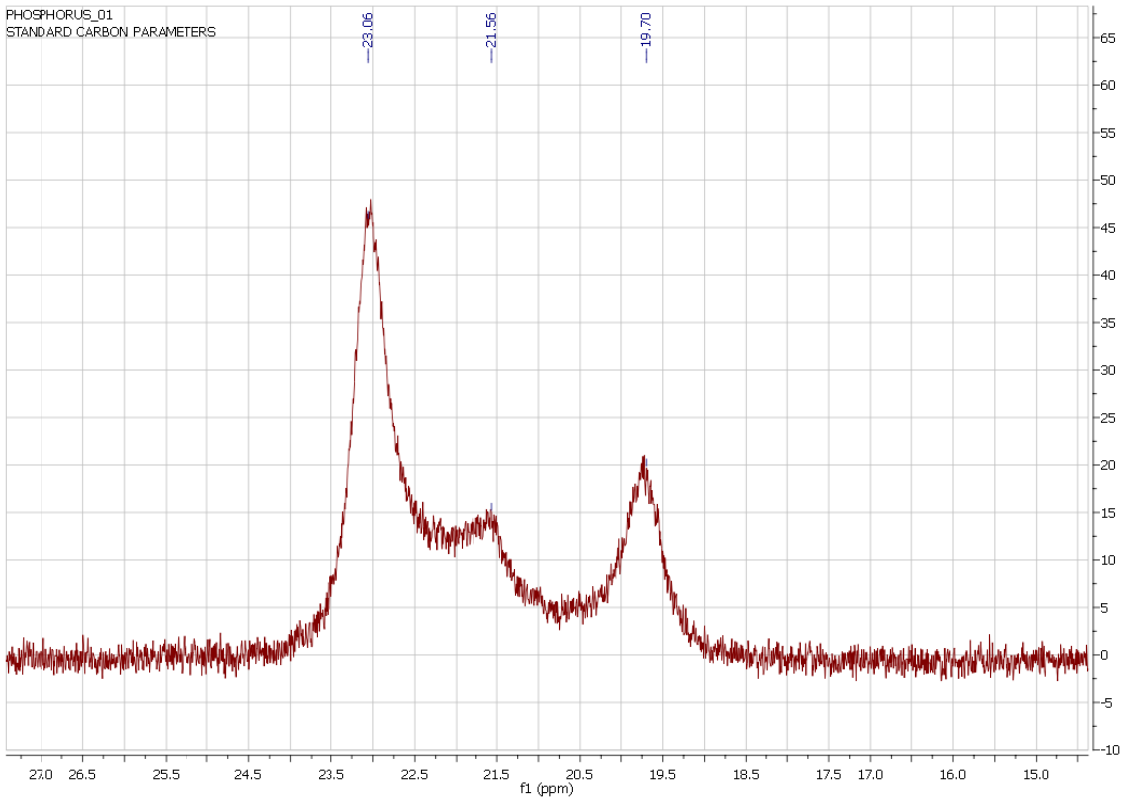

176.6
132.4
132.3
129.4
129.1
128.8
128.8
128.7
128.6
124.3
121.7
119.1
116.5
65.8
65.6
52.5
52.3
36.0
28.7
21.5
21.2
HN
P
O
N
S
F
0.50
0.46
0.49
0.05
0.19
0.20
0.05
0.98
0.97
0.88
1.28
1.09
1.04
1.37
1.00

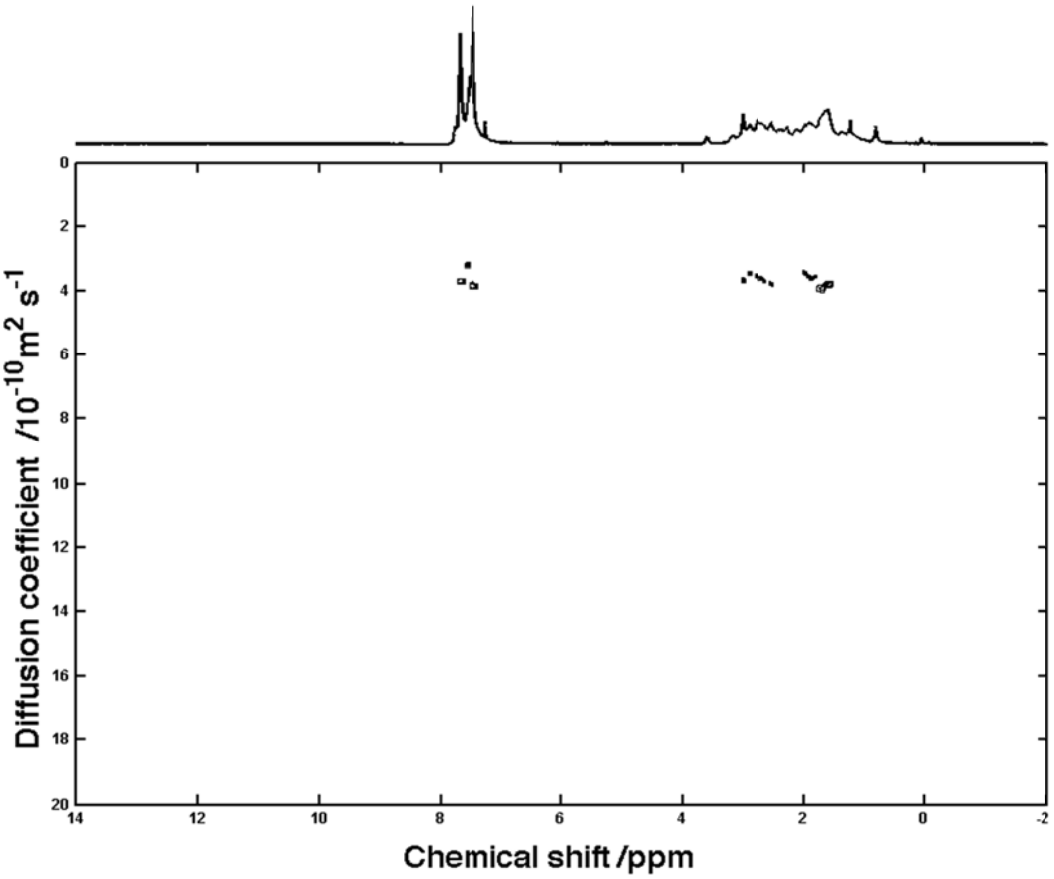


Bidentate Gold(I) complex (5a/5b)

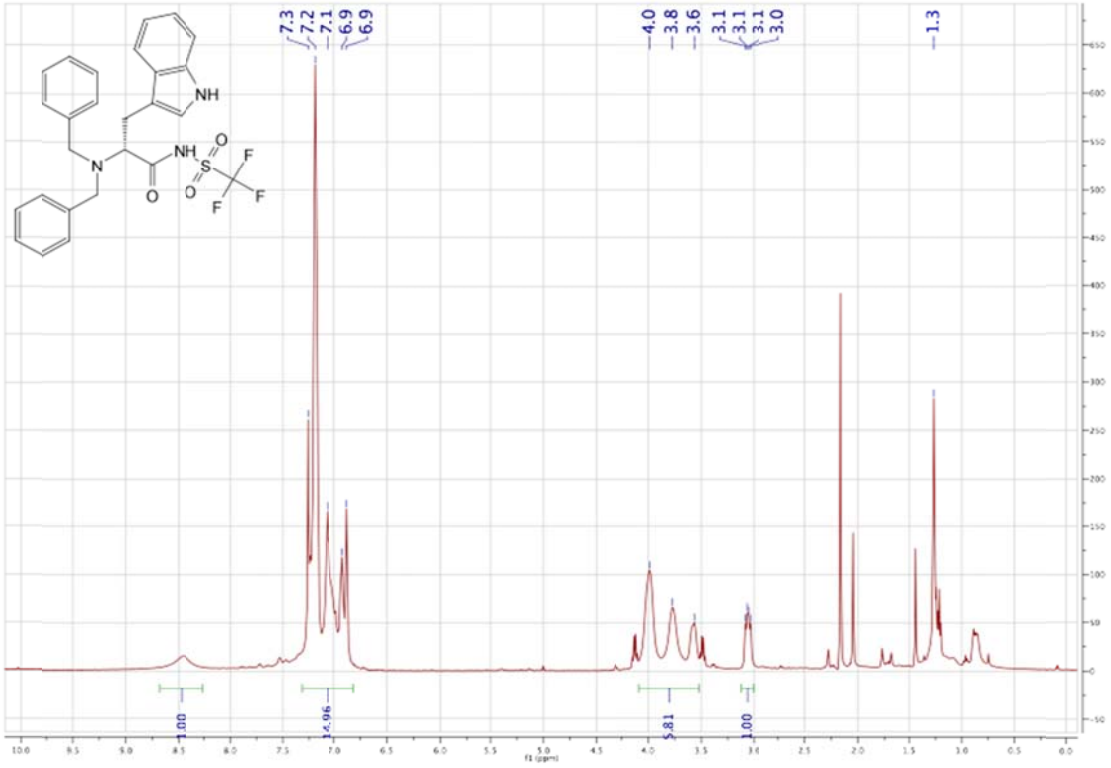


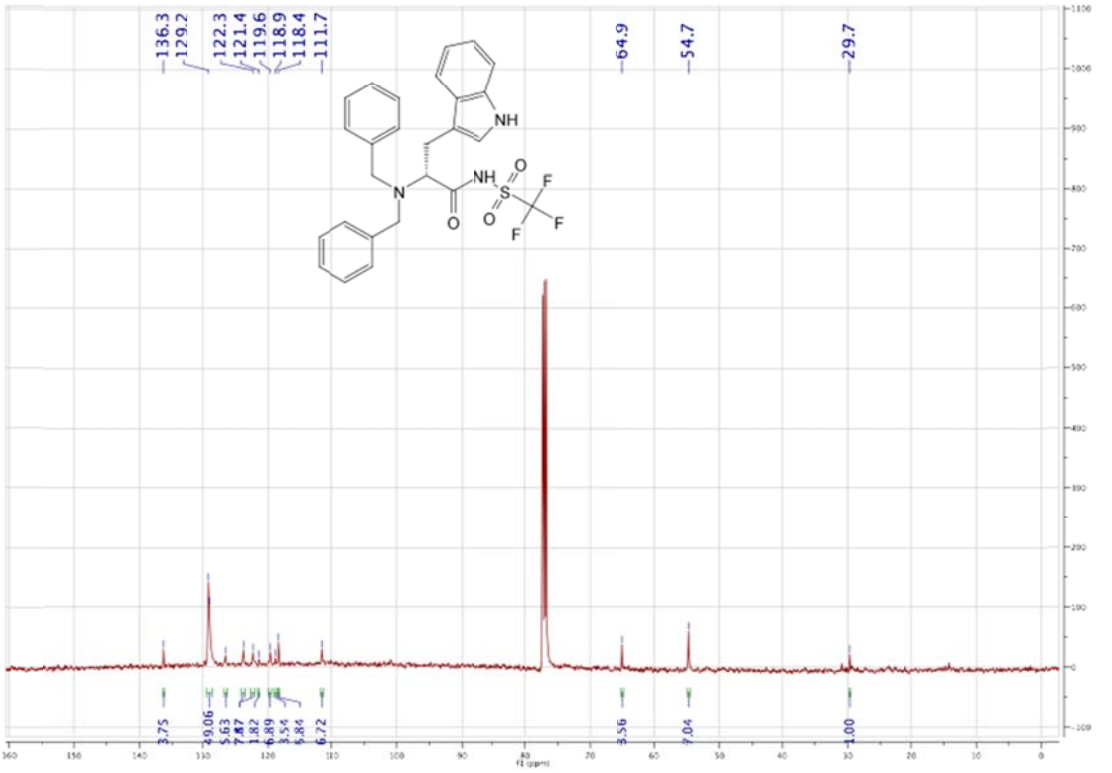

177.6
133.5
133.0
132.2
132.0
129.9
129.4
129.4
128.5
128.4
121.8
119.3
62.1
53.6
52.9
36.0
33.7
33.4
32.0
24.0
22.7
f1 (ppm)


PHOSPHORUS_01
STANDARD CARBON PARAMETERS
23.06
21.56
19.70
f1 (ppm)

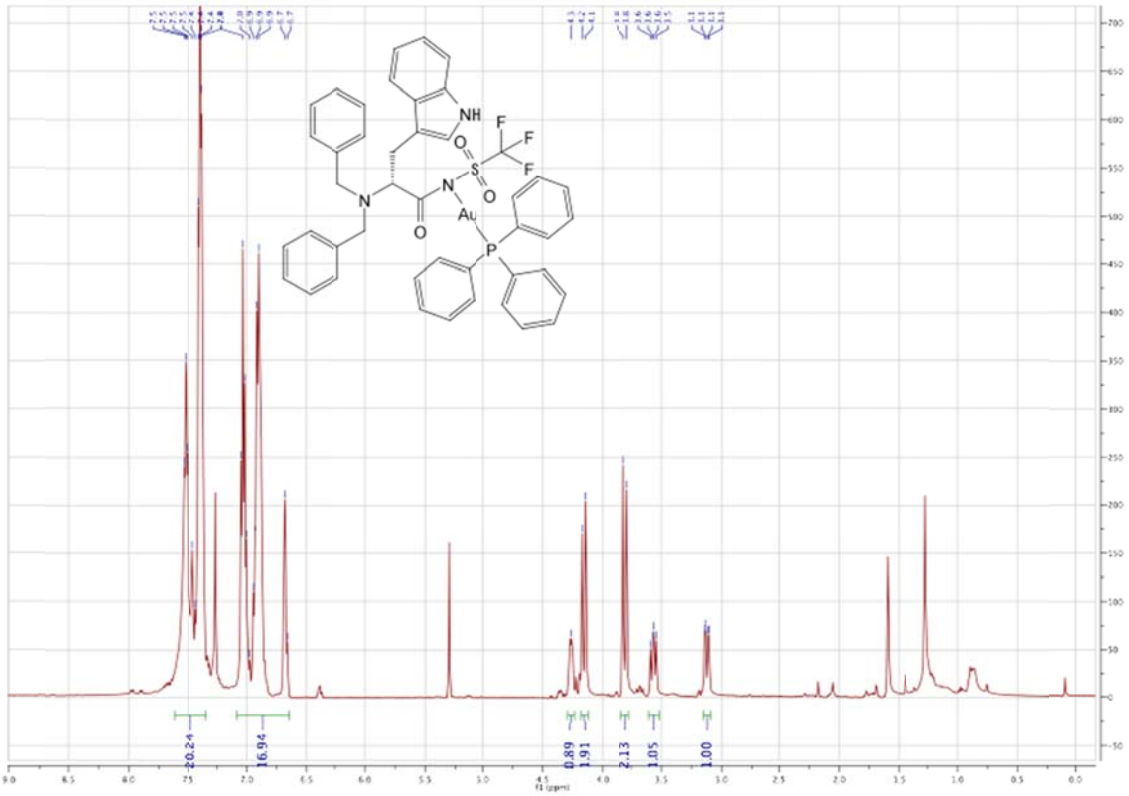


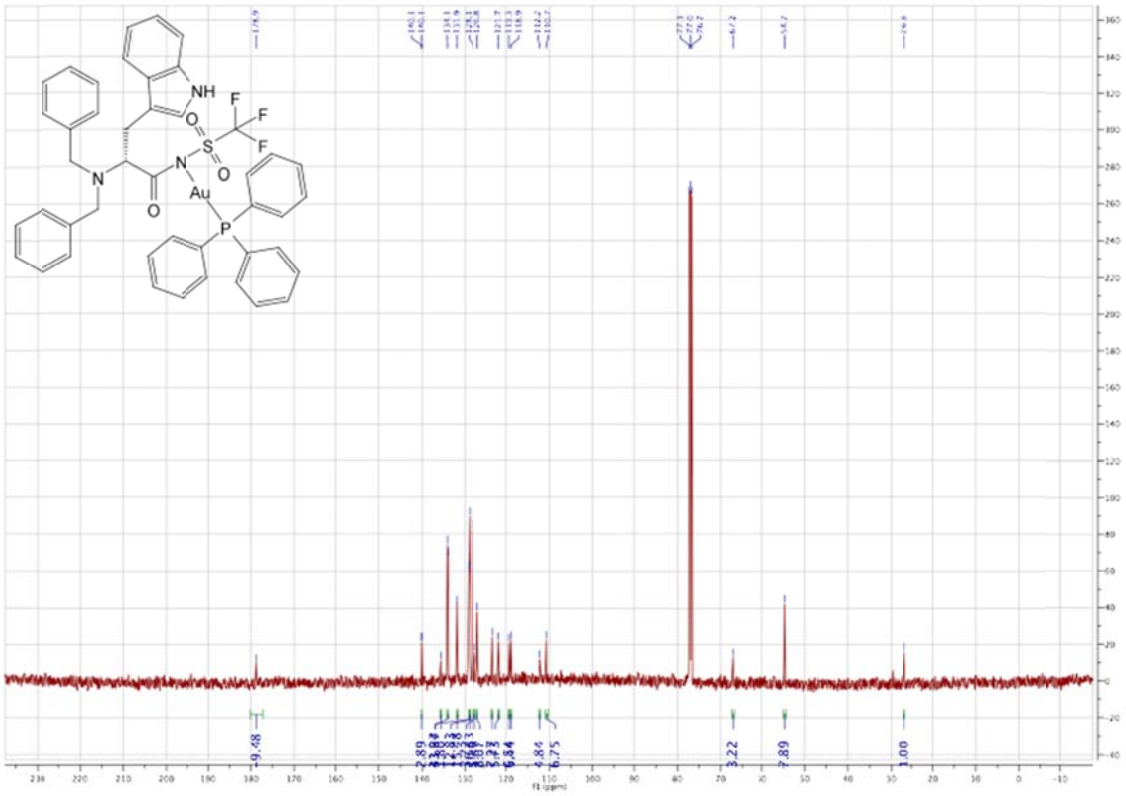
_N_,_N_-Dibenzyl-_N_[(trifluoromethyl)sulfonyl]-(_D_)-tryptophanamide

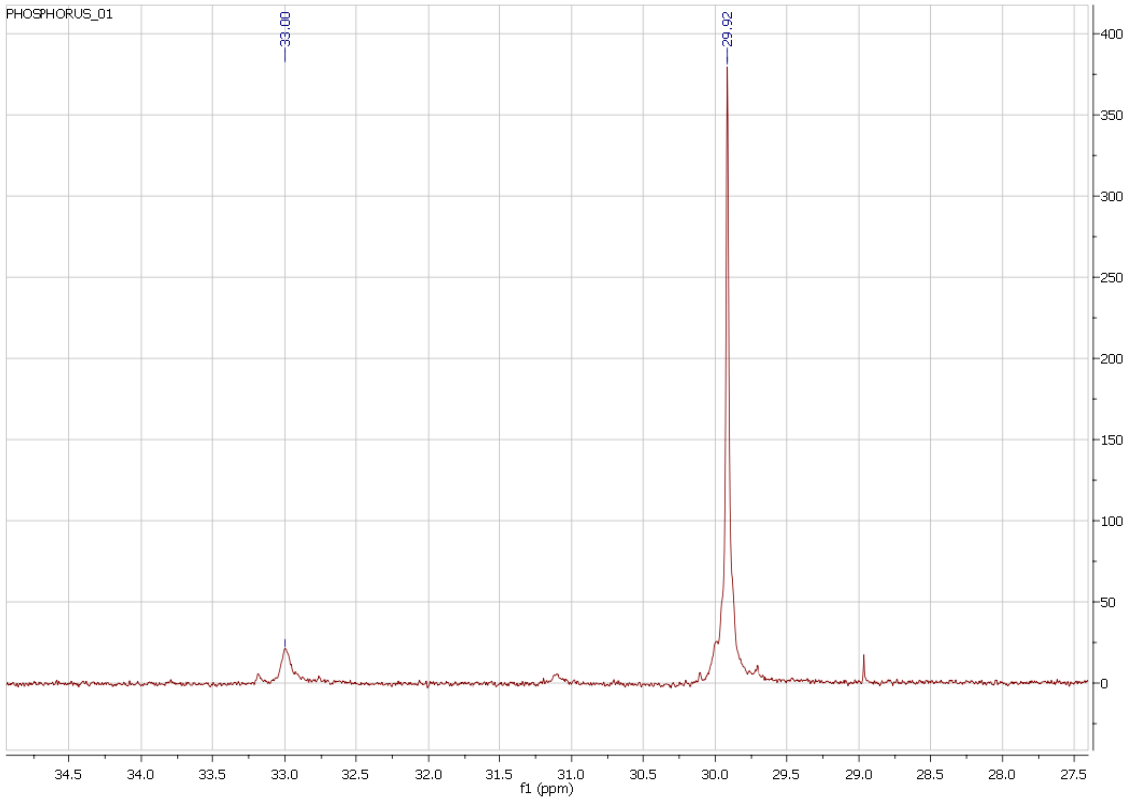




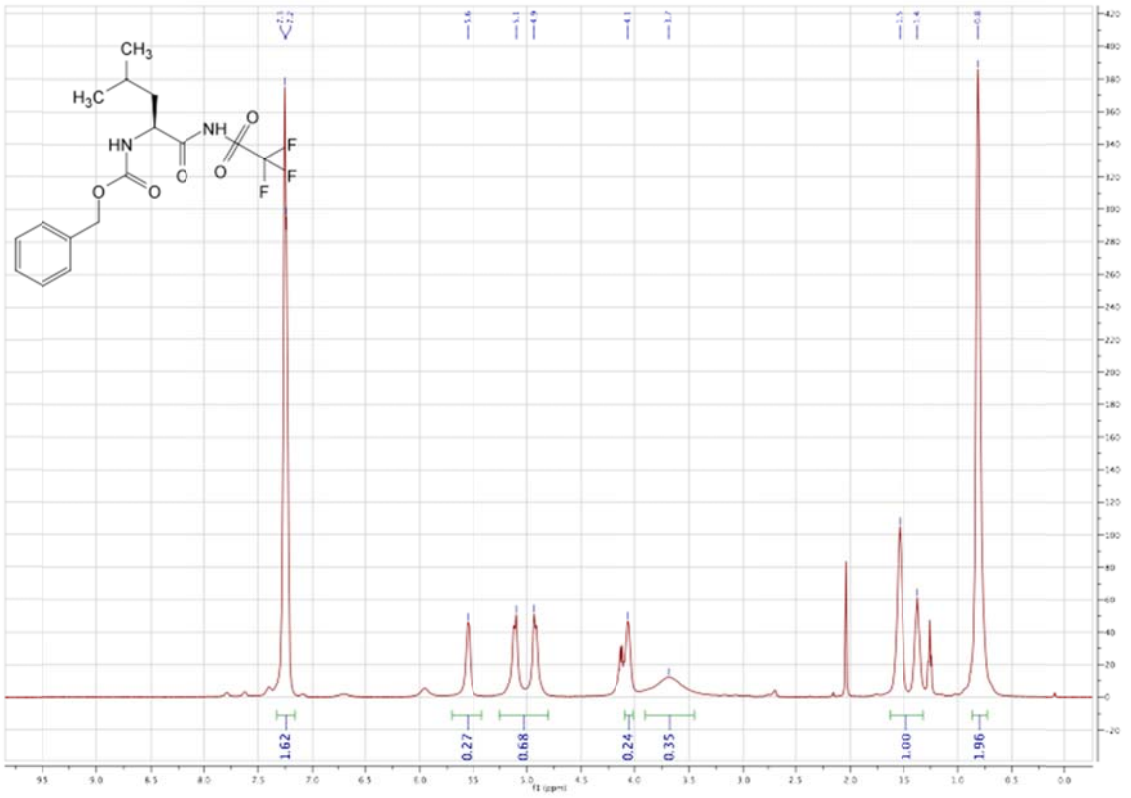
Triphenyphosphine gold _N_,_N_-dibenzyl-_N_[(trifluoromethyl)sulfonyl]-(_D_)-tryptophanamide (4)

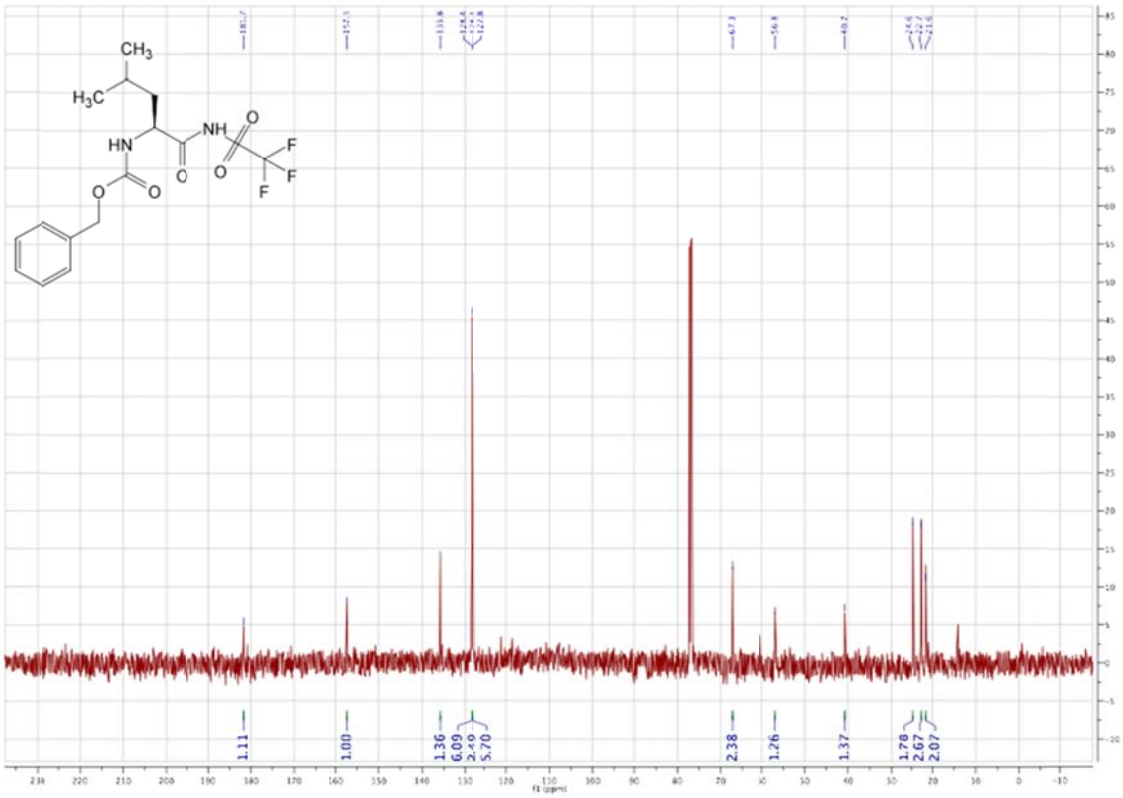

NH
F
F
F
O
S
O
N
Au
P
O
N
20.24
16.94
0.89
1.91
2.13
1.05
1.00


NH
F
F
F
O
S
O
N
Au
P
O
N
9.48
2.89
4.86
6.75
3.22
2.89
1.00

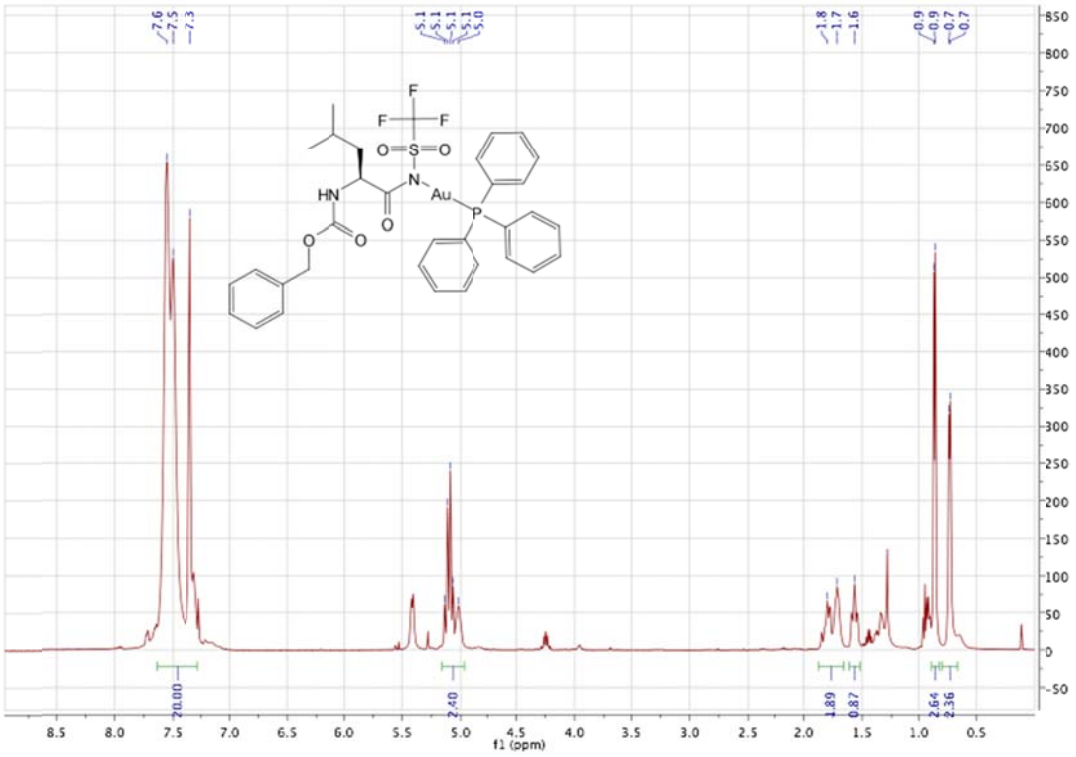


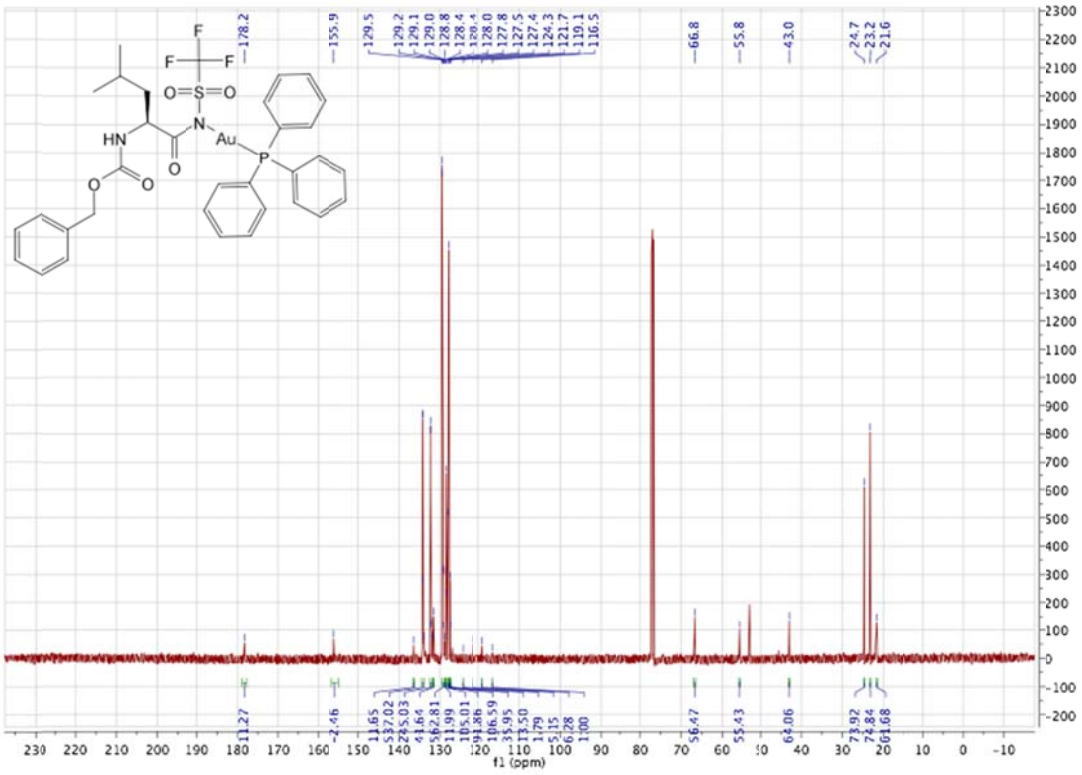
N^2-[(Benzyloxy)carbonyl}-N^1-[(trifluoromethyl)sulfonyl]-(*L*)-leucinamide

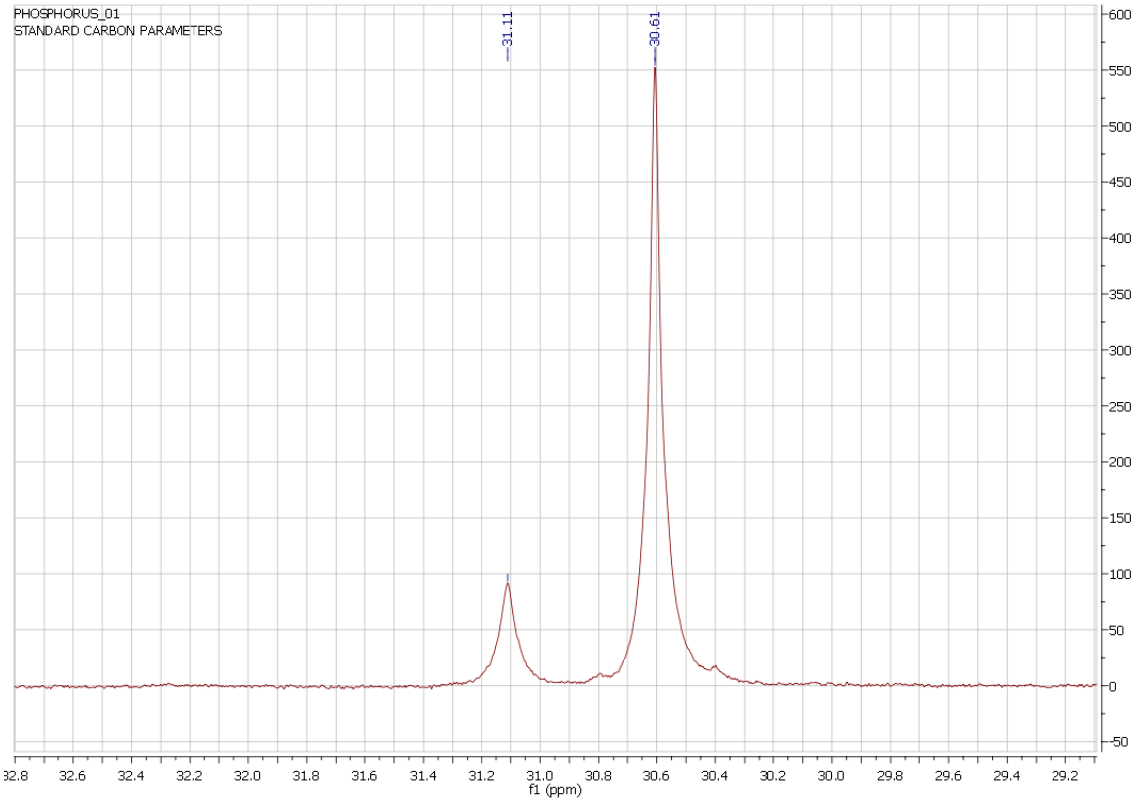




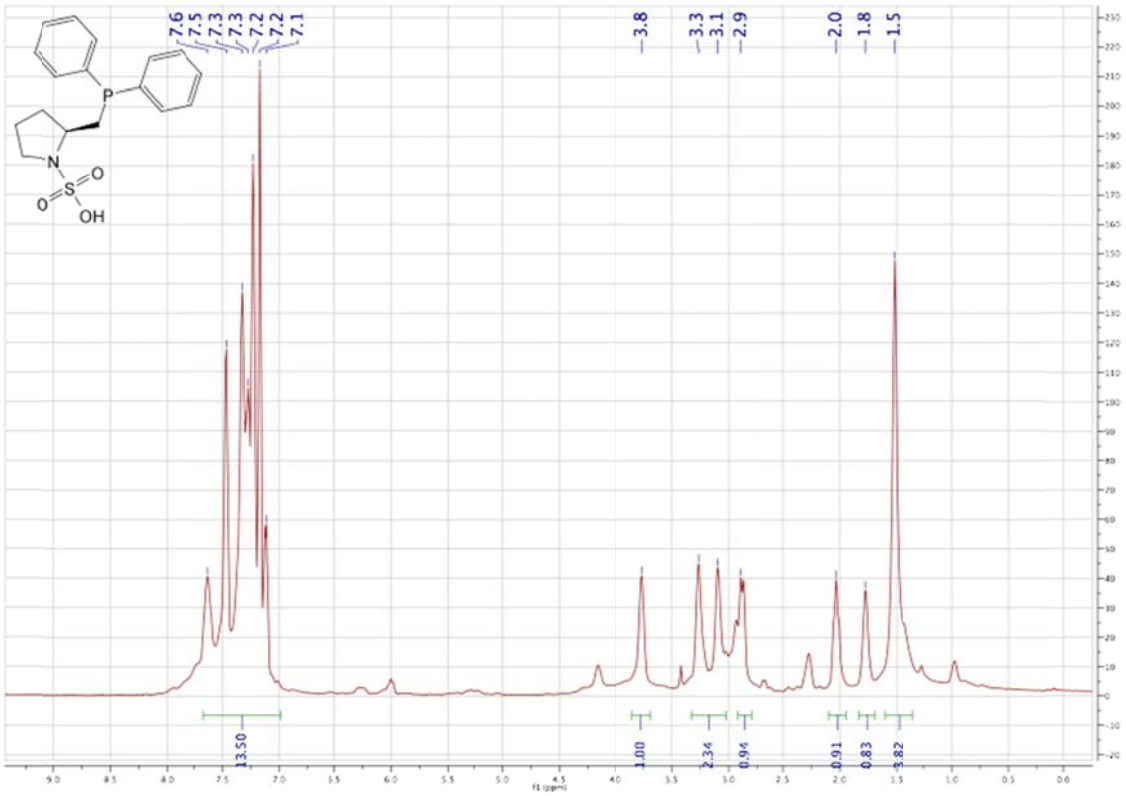
Triphenylphosphine gold N^2-[(benzyloxy)carbonyl}-N^1-[(trifluoromethyl)sulfonyl]-(*L*)-leucinamide (3)

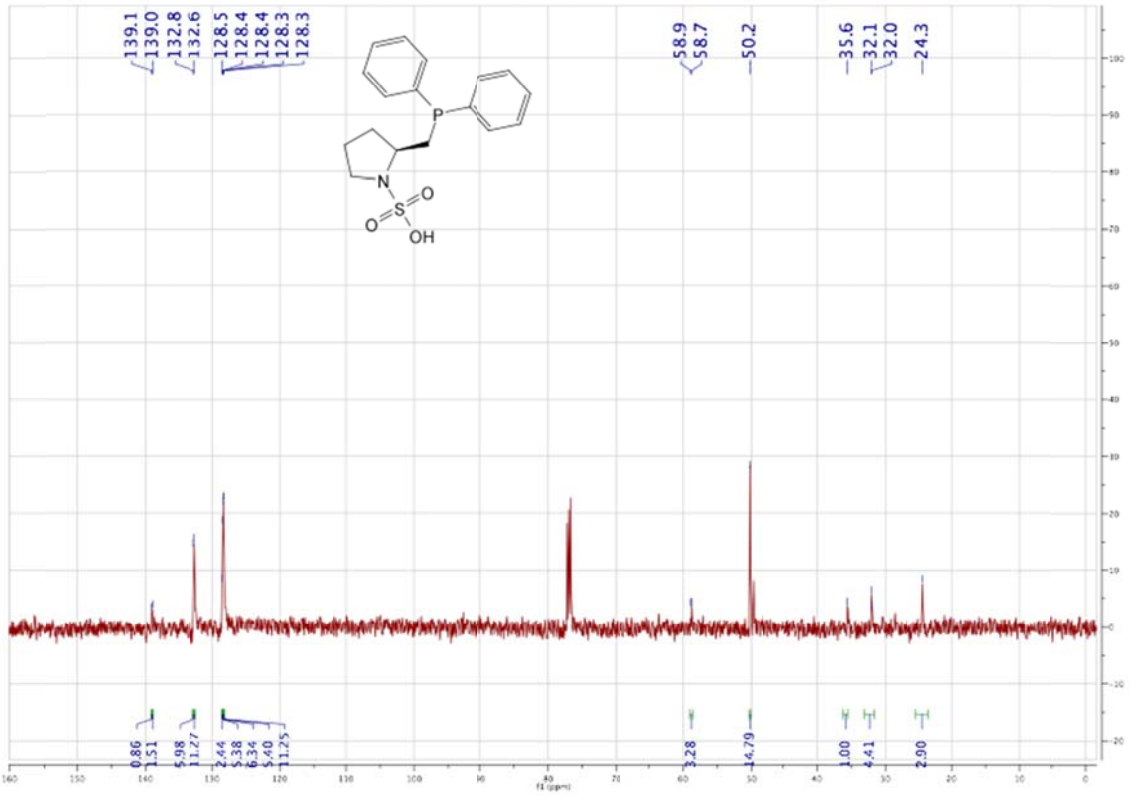






(*S*)-2-((Diphenylphosphino)methyl)pyrrolidine-1-sulfonic acid


7.6
7.5
7.3
7.3
7.2
7.2
7.1
3.8
3.3
3.1
2.9
2.0
1.8
1.5
13.50
1.00
2.34
0.94
0.91
0.83
3.82
P
N
S
O
O
OH


139.1
139.0
132.8
132.6
128.5
128.4
128.4
128.3
128.3
58.9
58.7
50.2
35.6
32.1
32.0
24.3
0.86
1.51
5.98
11.27
2.44
5.38
6.34
5.40
11.25
3.28
14.79
1.00
4.41
2.90
P
N
S
O
O
OH