Supplementary Information

Copper(I) Iodide Polyphosphine Adducts at Low-Loading for Sonogashira Alkynylation of Demanding Halide Substrates: Ligand Exchange Study between Copper and Palladium

Matthieu Beaupérin, Andre Job , Hélène Cattey, Sylviane Royer, Philippe Meunier and Jean-Cyrille Hierso*

Université de Bourgogne, Institut de Chimie Moléculaire de l'Université de Bourgogne (ICMUB-UMR CNRS 5260), 9 avenue Alain Savary 21078 Dijon, France. Fax: 33 3 8039 3682; Tel: 33 3 8039 6106; E-mail: jean-cyrille.hierso@u-bourgogne.fr

Content

Table 1. Crystal data and structure refinement for copper adduct **4**

Compound	**0906jc19b**
Formula	**$C_{54}H_{53}FeP_3CuI$, 1,5(C_7H_8)**
M	**1179.36**
T; K	**115(2)**
Crystal system	**monoclinic**
Space group	**P 21/c**
a; Å	**13.4559(2)**
b; Å	**18.2967(3)**
c; Å	**23.1577(4)**
β; °	**98.583(1)**
V; Å^3	**5637.54(16)**
Z	**2**
F(000)	**2420**
D_{calc}; g/cm^3	**1.299**
diffractometer	**Enraf–Nonius KappaCCD**
scan type	**mixture of ϕ rotations and ω scans**
λ; Å	**0.71073**
μ; mm^{-1}	**1.306**
Crystal size; mm^3	**0.25 x 0.17 x 0.08**
sin(θ)/λ max; Å^{-1}	**0.65**
Index ranges	**h: –17; 17** **k: –22; 23** **l: –30; 30**
RC = Refl. Collected	**24003**
IRC = independent RC	**12842 [R(int) = 0.0227]**
IRCGT = RC and [I>2σ(I)]	**11046**
Refinement method	**Full–matrix least–squares on F^2**
Data / restraints / parameters	**12482 / 1 / 575**
R for IRCGT	**R1[a] = 0.0408, wR2[b] = 0.0864**
R for IRC	**R1[a] = 0.0515, wR2[b] = 0.0969**
Goodness–of–fit[c]	**1.075**
Largest diff. peak and hole; e.Å^{-3}	**1.404 and –0.682**

[a] $R1=\Sigma(||F_o|-|F_c||)/\Sigma|F_o|$.

[b] $wR2=[\Sigma w(F_o^2-F_c^2)^2/\Sigma[w(F_o^2)^2]^{1/2}$ where $w=1/[\sigma^2(Fo^2)+13.5672\ P+(0.0223P)^2]$ where $P=(Max(Fo^2,0)+2*Fc^2)/3$

[c] Goodness of fit $=[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 0906jc19b.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(68)	8881(11)	-131(9)	9091(6)	119(5)
C(62)	10090(5)	-293(4)	10613(2)	62(2)
C(63)	9477(4)	-380(4)	10078(3)	80(3)
C(64)	9661(5)	24(4)	9597(2)	66(2)
C(65)	10459(5)	514(4)	9650(2)	75(3)
C(66)	11072(4)	601(3)	10185(3)	60(2)
C(67)	10887(5)	197(4)	10666(2)	75(3)
C(61)	916(7)	2846(5)	10100(4)	90(3)
C(55)	1333(4)	3295(3)	9635(2)	66(2)
C(56)	690(4)	3634(4)	9206(3)	103(4)
C(57)	1061(4)	4067(3)	8808(2)	100(3)
C(58)	2073(4)	4161(3)	8839(2)	76(2)
C(59)	2715(3)	3822(3)	9268(2)	62(2)
C(60)	2345(4)	3389(3)	9666(2)	63(2)
C(61B)	1732(5)	4502(4)	8507(3)	47(3)
C(55B)	2509(6)	3664(5)	9348(4)	54(3)
C(56B)	2426(6)	3196(5)	9794(4)	44(3)
C(57B)	1504(6)	3006(4)	9917(3)	42(2)
C(58B)	663(5)	3285(5)	9593(3)	50(3)
C(59B)	745(5)	3754(5)	9147(3)	36(3)
C(60B)	1668(6)	3943(5)	9025(4)	53(3)
P(1)	4699(1)	2233(1)	8020(1)	21(1)
P(2)	7491(1)	2050(1)	8334(1)	24(1)
P(3)	6502(1)	2912(1)	7216(1)	22(1)
Fe	6288(1)	3595(1)	8613(1)	21(1)
Cu	6091(1)	1840(1)	7663(1)	23(1)
I	6003(1)	631(1)	7099(1)	33(1)
C(1)	4978(1)	2997(1)	8519(1)	22(1)
C(2)	5530(1)	2898(1)	9094(1)	23(1)
C(3)	5640(2)	3584(2)	9388(1)	24(1)
C(4)	5175(2)	4116(2)	8988(1)	23(1)
C(5)	4766(2)	3762(2)	8458(1)	24(1)
C(6)	6923(2)	3432(2)	7874(1)	24(1)
C(7)	7464(2)	3040(2)	8370(1)	24(1)
C(8)	7834(2)	3582(2)	8798(1)	26(1)
C(9)	7543(2)	4290(2)	8576(1)	29(1)
C(10)	6960(2)	4188(2)	8012(1)	28(1)
C(11)	6018(2)	3666(2)	10039(1)	28(1)
C(12)	6910(3)	3165(2)	10233(1)	36(1)
C(13)	6318(3)	4451(2)	10209(1)	35(1)
C(14)	5138(3)	3447(2)	10358(1)	35(1)
C(15)	7886(3)	5037(2)	8823(1)	36(1)
C(16)	8488(4)	5392(2)	8376(2)	59(1)
C(17)	8582(3)	4963(2)	9409(2)	45(1)
C(18)	7002(3)	5534(2)	8892(2)	42(1)
C(19)	3989(2)	1631(2)	8442(1)	25(1)
C(20)	4010(2)	884(2)	8343(2)	33(1)
C(21)	3472(3)	409(2)	8650(2)	41(1)
C(22)	2913(3)	678(2)	9056(2)	43(1)
C(23)	2884(3)	1420(2)	9157(2)	45(1)
C(24)	3424(3)	1899(2)	8855(2)	37(1)

C(25)	3721(2)	2576(2)	7450(1)	26(1)
C(26)	2932(2)	3018(2)	7569(2)	35(1)
C(27)	2226(3)	3269(2)	7114(2)	45(1)
C(28)	2295(3)	3077(2)	6544(2)	47(1)
C(29)	3063(3)	2626(2)	6424(2)	39(1)
C(30)	3777(2)	2375(2)	6875(1)	31(1)
C(31)	8732(2)	1826(2)	8153(1)	34(1)
C(32)	8890(3)	1124(2)	7961(2)	49(1)
C(33)	9841(4)	921(3)	7837(2)	70(2)
C(34)	10615(3)	1417(4)	7902(2)	73(2)
C(35)	10460(3)	2119(3)	8077(2)	64(1)
C(36)	9517(3)	2327(3)	8200(2)	47(1)
C(37)	7605(2)	1708(2)	9082(1)	26(1)
C(38)	8436(2)	1864(2)	9501(1)	30(1)
C(39)	8494(3)	1596(2)	10064(1)	36(1)
C(40)	7730(3)	1164(2)	10215(1)	37(1)
C(41)	6913(3)	987(2)	9801(1)	35(1)
C(42)	6852(2)	1254(2)	9234(1)	30(1)
C(43)	7685(2)	2887(2)	6908(1)	30(1)
C(44)	8044(2)	2211(2)	6756(1)	38(1)
C(45)	8973(3)	2166(3)	6563(2)	50(1)
C(46)	9541(3)	2778(3)	6531(2)	61(1)
C(47)	9192(3)	3457(3)	6679(2)	58(1)
C(48)	8262(3)	3515(2)	6868(1)	40(1)
C(49)	5691(2)	3499(2)	6713(1)	24(1)
C(50)	5642(2)	3361(2)	6113(1)	29(1)
C(51)	4933(3)	3713(2)	5711(1)	33(1)
C(52)	4270(3)	4209(2)	5899(1)	34(1)
C(53)	4316(2)	4356(2)	6491(1)	31(1)
C(54)	5022(2)	4000(2)	6897(1)	26(1)

Table 3. Bond lengths [A] and angles [deg] for 0906jc19b.

C(68)-C(64)	1.479(13)
C(68)-H(68A)	0.9800
C(68)-H(68B)	0.9800
C(68)-H(68C)	0.9800
C(62)-C(63)	1.3900
C(62)-C(67)	1.3900
C(62)-H(62)	0.9500
C(63)-C(64)	1.3900
C(63)-H(63)	0.9500
C(64)-C(65)	1.3900
C(65)-C(66)	1.3900
C(65)-H(65)	0.9500
C(66)-C(67)	1.3900
C(66)-H(66)	0.9500
C(67)-H(67)	0.9500
C(61)-C(55)	1.527(10)
C(61)-H(61A)	0.9800
C(61)-H(61B)	0.9800
C(61)-H(61C)	0.9800
C(55)-C(56)	1.364(3)
C(55)-C(60)	1.364(3)
C(56)-C(57)	1.364(3)
C(56)-H(56)	0.9500
C(57)-C(58)	1.364(3)
C(57)-H(57)	0.9500
C(58)-C(59)	1.364(3)
C(58)-H(58)	0.9500
C(59)-C(60)	1.364(3)
C(59)-H(59)	0.9500
C(60)-H(60)	0.9500
C(61B)-C(60B)	1.587(10)
C(61B)-H(61D)	0.9800
C(61B)-H(61E)	0.9800
C(61B)-H(61F)	0.9800
C(55B)-C(56B)	1.359(5)
C(55B)-C(60B)	1.360(5)
C(55B)-H(55B)	0.9500
C(56B)-C(57B)	1.359(5)
C(56B)-H(56B)	0.9500
C(57B)-C(58B)	1.359(5)
C(57B)-H(57B)	0.9500
C(58B)-C(59B)	1.359(5)
C(58B)-H(58B)	0.9500
C(59B)-C(60B)	1.359(5)
C(59B)-H(59B)	0.9500
P(1)-C(1)	1.8174
P(1)-C(25)	1.830(3)
P(1)-C(19)	1.834(3)
P(1)-Cu	2.2741
P(2)-C(7)	1.814(3)
P(2)-C(37)	1.826(3)
P(2)-C(31)	1.829(3)
P(2)-Cu	2.2890
P(3)-C(6)	1.813(3)
P(3)-C(49)	1.824(3)
P(3)-C(43)	1.839(3)
P(3)-Cu	2.3214

Fe-C(7)	2.030(3)
Fe-C(6)	2.046(3)
Fe-C(5)	2.049(3)
Fe-C(1)	2.0588
Fe-C(8)	2.060(3)
Fe-C(2)	2.0605
Fe-C(4)	2.071(3)
Fe-C(10)	2.076(3)
Fe-C(3)	2.109(3)
Fe-C(9)	2.126(3)
Cu-I	2.5623
C(1)-C(5)	1.430(3)
C(1)-C(2)	1.4363
C(2)-C(3)	1.427(3)
C(2)-H(2)	0.9500
C(3)-C(4)	1.423(4)
C(3)-C(11)	1.525(4)
C(4)-C(5)	1.424(4)
C(4)-H(4)	0.9500
C(5)-H(5)	0.9500
C(6)-C(10)	1.420(4)
C(6)-C(7)	1.455(4)
C(7)-C(8)	1.437(4)
C(8)-C(9)	1.427(4)
C(8)-H(8)	0.9500
C(9)-C(10)	1.430(4)
C(9)-C(15)	1.526(4)
C(10)-H(10)	0.9500
C(11)-C(12)	1.524(4)
C(11)-C(13)	1.527(4)
C(11)-C(14)	1.539(4)
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-C(18)	1.525(5)
C(15)-C(17)	1.535(5)
C(15)-C(16)	1.548(5)
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-C(20)	1.387(4)
C(19)-C(24)	1.396(4)
C(20)-C(21)	1.392(5)
C(20)-H(20)	0.9500
C(21)-C(22)	1.380(5)
C(21)-H(21)	0.9500
C(22)-C(23)	1.379(5)
C(22)-H(22)	0.9500
C(23)-C(24)	1.390(4)

C(23)-H(23)	0.9500
C(24)-H(24)	0.9500
C(25)-C(26)	1.394(4)
C(25)-C(30)	1.395(4)
C(26)-C(27)	1.387(5)
C(26)-H(26)	0.9500
C(27)-C(28)	1.385(6)
C(27)-H(27)	0.9500
C(28)-C(29)	1.383(6)
C(28)-H(28)	0.9500
C(29)-C(30)	1.388(4)
C(29)-H(29)	0.9500
C(30)-H(30)	0.9500
C(31)-C(32)	1.387(5)
C(31)-C(36)	1.390(5)
C(32)-C(33)	1.402(5)
C(32)-H(32)	0.9500
C(33)-C(34)	1.372(8)
C(33)-H(33)	0.9500
C(34)-C(35)	1.371(8)
C(34)-H(34)	0.9500
C(35)-C(36)	1.394(5)
C(35)-H(35)	0.9500
C(36)-H(36)	0.9500
C(37)-C(42)	1.395(4)
C(37)-C(38)	1.397(4)
C(38)-C(39)	1.384(4)
C(38)-H(38)	0.9500
C(39)-C(40)	1.382(5)
C(39)-H(39)	0.9500
C(40)-C(41)	1.385(5)
C(40)-H(40)	0.9500
C(41)-C(42)	1.392(4)
C(41)-H(41)	0.9500
C(42)-H(42)	0.9500
C(43)-C(44)	1.393(5)
C(43)-C(48)	1.398(5)
C(44)-C(45)	1.391(5)
C(44)-H(44)	0.9500
C(45)-C(46)	1.364(7)
C(45)-H(45)	0.9500
C(46)-C(47)	1.388(7)
C(46)-H(46)	0.9500
C(47)-C(48)	1.390(5)
C(47)-H(47)	0.9500
C(48)-H(48)	0.9500
C(49)-C(54)	1.396(4)
C(49)-C(50)	1.402(4)
C(50)-C(51)	1.388(4)
C(50)-H(50)	0.9500
C(51)-C(52)	1.388(5)
C(51)-H(51)	0.9500
C(52)-C(53)	1.387(4)
C(52)-H(52)	0.9500
C(53)-C(54)	1.394(4)
C(53)-H(53)	0.9500
C(54)-H(54)	0.9500

C(63)-C(62)-C(67)	120.0
C(63)-C(62)-H(62)	120.0
C(67)-C(62)-H(62)	120.0

C(64)-C(63)-C(62)	120.0
C(64)-C(63)-H(63)	120.0
C(62)-C(63)-H(63)	120.0
C(65)-C(64)-C(63)	120.0
C(65)-C(64)-C(68)	129.9(9)
C(63)-C(64)-C(68)	110.0(9)
C(64)-C(65)-C(66)	120.0
C(64)-C(65)-H(65)	120.0
C(66)-C(65)-H(65)	120.0
C(67)-C(66)-C(65)	120.0
C(67)-C(66)-H(66)	120.0
C(65)-C(66)-H(66)	120.0
C(66)-C(67)-C(62)	120.0
C(66)-C(67)-H(67)	120.0
C(62)-C(67)-H(67)	120.0
C(56)-C(55)-C(60)	120.0
C(56)-C(55)-C(61)	119.9(5)
C(60)-C(55)-C(61)	120.0(5)
C(55)-C(56)-C(57)	120.0
C(55)-C(56)-H(56)	120.0
C(57)-C(56)-H(56)	120.0
C(58)-C(57)-C(56)	120.0
C(58)-C(57)-H(57)	120.0
C(56)-C(57)-H(57)	120.0
C(59)-C(58)-C(57)	120.0
C(59)-C(58)-H(58)	120.0
C(57)-C(58)-H(58)	120.0
C(58)-C(59)-C(60)	120.0
C(58)-C(59)-H(59)	120.0
C(60)-C(59)-H(59)	120.0
C(59)-C(60)-C(55)	120.0
C(59)-C(60)-H(60)	120.0
C(55)-C(60)-H(60)	120.0
C(60B)-C(61B)-H(61D)	109.5
C(60B)-C(61B)-H(61E)	109.5
H(61D)-C(61B)-H(61E)	109.5
C(60B)-C(61B)-H(61F)	109.5
H(61D)-C(61B)-H(61F)	109.5
H(61E)-C(61B)-H(61F)	109.5
C(56B)-C(55B)-C(60B)	120.0
C(56B)-C(55B)-H(55B)	120.0
C(60B)-C(55B)-H(55B)	120.0
C(57B)-C(56B)-C(55B)	120.0
C(57B)-C(56B)-H(56B)	120.0
C(55B)-C(56B)-H(56B)	120.0
C(56B)-C(57B)-C(58B)	120.0
C(56B)-C(57B)-H(57B)	120.0
C(58B)-C(57B)-H(57B)	120.0
C(59B)-C(58B)-C(57B)	120.0
C(59B)-C(58B)-H(58B)	120.0
C(57B)-C(58B)-H(58B)	120.0
C(58B)-C(59B)-C(60B)	120.0
C(58B)-C(59B)-H(59B)	120.0
C(60B)-C(59B)-H(59B)	120.0
C(59B)-C(60B)-C(55B)	120.0
C(59B)-C(60B)-C(61B)	118.4(6)
C(55B)-C(60B)-C(61B)	121.6(6)
C(1)-P(1)-C(25)	104.62(10)
C(1)-P(1)-C(19)	101.37(9)
C(25)-P(1)-C(19)	102.40(13)
C(1)-P(1)-Cu	111.8

C(25)-P(1)-Cu	112.90(10)
C(19)-P(1)-Cu	121.85(9)
C(7)-P(2)-C(37)	107.37(12)
C(7)-P(2)-C(31)	104.99(15)
C(37)-P(2)-C(31)	101.04(13)
C(7)-P(2)-Cu	100.27(9)
C(37)-P(2)-Cu	122.07(10)
C(31)-P(2)-Cu	119.58(10)
C(6)-P(3)-C(49)	108.09(13)
C(6)-P(3)-C(43)	99.16(13)
C(49)-P(3)-C(43)	103.67(13)
C(6)-P(3)-Cu	97.72(9)
C(49)-P(3)-Cu	128.49(9)
C(43)-P(3)-Cu	115.44(11)
C(7)-Fe-C(6)	41.81(11)
C(7)-Fe-C(5)	145.70(11)
C(6)-Fe-C(5)	113.92(11)
C(7)-Fe-C(1)	113.30(8)
C(6)-Fe-C(1)	106.94(8)
C(5)-Fe-C(1)	40.76(8)
C(7)-Fe-C(8)	41.14(11)
C(6)-Fe-C(8)	68.72(11)
C(5)-Fe-C(8)	171.90(12)
C(1)-Fe-C(8)	146.97(9)
C(7)-Fe-C(2)	108.03(8)
C(6)-Fe-C(2)	131.58(8)
C(5)-Fe-C(2)	67.96(8)
C(1)-Fe-C(2)	40.8
C(8)-Fe-C(2)	116.57(8)
C(7)-Fe-C(4)	171.45(11)
C(6)-Fe-C(4)	146.64(11)
C(5)-Fe-C(4)	40.43(11)
C(1)-Fe-C(4)	68.15(8)
C(8)-Fe-C(4)	133.57(12)
C(2)-Fe-C(4)	67.18(8)
C(7)-Fe-C(10)	68.89(12)
C(6)-Fe-C(10)	40.30(12)
C(5)-Fe-C(10)	109.15(12)
C(1)-Fe-C(10)	131.18(8)
C(8)-Fe-C(10)	67.50(12)
C(2)-Fe-C(10)	170.48(8)
C(4)-Fe-C(10)	117.00(12)
C(7)-Fe-C(3)	132.09(11)
C(6)-Fe-C(3)	171.07(12)
C(5)-Fe-C(3)	67.76(11)
C(1)-Fe-C(3)	68.22(8)
C(8)-Fe-C(3)	110.91(11)
C(2)-Fe-C(3)	39.99(8)
C(4)-Fe-C(3)	39.80(11)
C(10)-Fe-C(3)	148.44(12)
C(7)-Fe-C(9)	68.36(12)
C(6)-Fe-C(9)	67.66(11)
C(5)-Fe-C(9)	133.05(12)
C(1)-Fe-C(9)	170.51(8)
C(8)-Fe-C(9)	39.81(12)
C(2)-Fe-C(9)	148.53(8)
C(4)-Fe-C(9)	111.70(12)
C(10)-Fe-C(9)	39.78(11)
C(3)-Fe-C(9)	118.18(11)
P(1)-Cu-P(2)	109.8
P(1)-Cu-P(3)	99.2

P(2)-Cu-P(3)	86.2
P(1)-Cu-I	118.7
P(2)-Cu-I	117.5
P(3)-Cu-I	119.9
C(5)-C(1)-C(2)	106.48(11)
C(5)-C(1)-P(1)	132.09(11)
C(2)-C(1)-P(1)	121.4
C(5)-C(1)-Fe	69.24(12)
C(2)-C(1)-Fe	69.7
P(1)-C(1)-Fe	124.6
C(3)-C(2)-C(1)	109.49(11)
C(3)-C(2)-Fe	71.85(11)
C(1)-C(2)-Fe	69.5
C(3)-C(2)-H(2)	125.3
C(1)-C(2)-H(2)	125.3
Fe-C(2)-H(2)	124.9
C(4)-C(3)-C(2)	106.7(2)
C(4)-C(3)-C(11)	128.8(3)
C(2)-C(3)-C(11)	123.7(2)
C(4)-C(3)-Fe	68.66(15)
C(2)-C(3)-Fe	68.16(10)
C(11)-C(3)-Fe	136.1(2)
C(3)-C(4)-C(5)	109.0(2)
C(3)-C(4)-Fe	71.54(16)
C(5)-C(4)-Fe	68.93(16)
C(3)-C(4)-H(4)	125.5
C(5)-C(4)-H(4)	125.5
Fe-C(4)-H(4)	125.6
C(4)-C(5)-C(1)	108.3(2)
C(4)-C(5)-Fe	70.63(17)
C(1)-C(5)-Fe	70.00(12)
C(4)-C(5)-H(5)	125.8
C(1)-C(5)-H(5)	125.8
Fe-C(5)-H(5)	125.1
C(10)-C(6)-C(7)	107.8(2)
C(10)-C(6)-P(3)	134.2(2)
C(7)-C(6)-P(3)	117.5(2)
C(10)-C(6)-Fe	70.98(16)
C(7)-C(6)-Fe	68.49(15)
P(3)-C(6)-Fe	131.50(15)
C(8)-C(7)-C(6)	106.5(3)
C(8)-C(7)-P(2)	135.6(2)
C(6)-C(7)-P(2)	117.9(2)
C(8)-C(7)-Fe	70.57(17)
C(6)-C(7)-Fe	69.70(16)
P(2)-C(7)-Fe	122.28(15)
C(9)-C(8)-C(7)	109.3(3)
C(9)-C(8)-Fe	72.58(17)
C(7)-C(8)-Fe	68.29(16)
C(9)-C(8)-H(8)	125.3
C(7)-C(8)-H(8)	125.3
Fe-C(8)-H(8)	125.3
C(8)-C(9)-C(10)	107.1(3)
C(8)-C(9)-C(15)	128.8(3)
C(10)-C(9)-C(15)	123.5(3)
C(8)-C(9)-Fe	67.61(17)
C(10)-C(9)-Fe	68.23(16)
C(15)-C(9)-Fe	135.9(2)
C(6)-C(10)-C(9)	109.2(3)
C(6)-C(10)-Fe	68.72(16)
C(9)-C(10)-Fe	71.99(16)

C(6)-C(10)-H(10)	125.4
C(9)-C(10)-H(10)	125.4
Fe-C(10)-H(10)	125.5
C(12)-C(11)-C(3)	111.3(2)
C(12)-C(11)-C(13)	108.9(3)
C(3)-C(11)-C(13)	112.5(2)
C(12)-C(11)-C(14)	109.2(3)
C(3)-C(11)-C(14)	106.4(3)
C(13)-C(11)-C(14)	108.4(3)
C(11)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-H(13A)	109.5
C(11)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(11)-C(14)-H(14A)	109.5
C(11)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(11)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(18)-C(15)-C(9)	112.1(3)
C(18)-C(15)-C(17)	110.0(3)
C(9)-C(15)-C(17)	111.3(3)
C(18)-C(15)-C(16)	108.3(3)
C(9)-C(15)-C(16)	106.5(3)
C(17)-C(15)-C(16)	108.5(3)
C(15)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(15)-C(17)-H(17A)	109.5
C(15)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(15)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(15)-C(18)-H(18A)	109.5
C(15)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(15)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(20)-C(19)-C(24)	119.0(3)
C(20)-C(19)-P(1)	118.6(2)
C(24)-C(19)-P(1)	122.4(2)
C(19)-C(20)-C(21)	120.4(3)
C(19)-C(20)-H(20)	119.8
C(21)-C(20)-H(20)	119.8
C(22)-C(21)-C(20)	120.1(3)
C(22)-C(21)-H(21)	119.9
C(20)-C(21)-H(21)	119.9
C(23)-C(22)-C(21)	120.0(3)

C(23)-C(22)-H(22)	120.0
C(21)-C(22)-H(22)	120.0
C(22)-C(23)-C(24)	120.3(3)
C(22)-C(23)-H(23)	119.8
C(24)-C(23)-H(23)	119.8
C(23)-C(24)-C(19)	120.1(3)
C(23)-C(24)-H(24)	119.9
C(19)-C(24)-H(24)	119.9
C(26)-C(25)-C(30)	119.5(3)
C(26)-C(25)-P(1)	123.0(2)
C(30)-C(25)-P(1)	117.6(2)
C(27)-C(26)-C(25)	119.9(3)
C(27)-C(26)-H(26)	120.0
C(25)-C(26)-H(26)	120.0
C(28)-C(27)-C(26)	120.3(4)
C(28)-C(27)-H(27)	119.9
C(26)-C(27)-H(27)	119.9
C(29)-C(28)-C(27)	120.1(3)
C(29)-C(28)-H(28)	119.9
C(27)-C(28)-H(28)	119.9
C(28)-C(29)-C(30)	120.1(3)
C(28)-C(29)-H(29)	120.0
C(30)-C(29)-H(29)	120.0
C(29)-C(30)-C(25)	120.1(3)
C(29)-C(30)-H(30)	119.9
C(25)-C(30)-H(30)	119.9
C(32)-C(31)-C(36)	119.0(3)
C(32)-C(31)-P(2)	118.0(3)
C(36)-C(31)-P(2)	123.0(3)
C(31)-C(32)-C(33)	119.9(4)
C(31)-C(32)-H(32)	120.1
C(33)-C(32)-H(32)	120.1
C(34)-C(33)-C(32)	120.2(5)
C(34)-C(33)-H(33)	119.9
C(32)-C(33)-H(33)	119.9
C(35)-C(34)-C(33)	120.4(4)
C(35)-C(34)-H(34)	119.8
C(33)-C(34)-H(34)	119.8
C(34)-C(35)-C(36)	119.9(5)
C(34)-C(35)-H(35)	120.0
C(36)-C(35)-H(35)	120.0
C(31)-C(36)-C(35)	120.5(4)
C(31)-C(36)-H(36)	119.8
C(35)-C(36)-H(36)	119.8
C(42)-C(37)-C(38)	118.9(3)
C(42)-C(37)-P(2)	118.8(2)
C(38)-C(37)-P(2)	122.3(2)
C(39)-C(38)-C(37)	120.6(3)
C(39)-C(38)-H(38)	119.7
C(37)-C(38)-H(38)	119.7
C(40)-C(39)-C(38)	120.1(3)
C(40)-C(39)-H(39)	120.0
C(38)-C(39)-H(39)	120.0
C(39)-C(40)-C(41)	120.2(3)
C(39)-C(40)-H(40)	119.9
C(41)-C(40)-H(40)	119.9
C(40)-C(41)-C(42)	120.0(3)
C(40)-C(41)-H(41)	120.0
C(42)-C(41)-H(41)	120.0
C(41)-C(42)-C(37)	120.3(3)
C(41)-C(42)-H(42)	119.8

C(37)-C(42)-H(42)	119.8
C(44)-C(43)-C(48)	119.7(3)
C(44)-C(43)-P(3)	118.3(3)
C(48)-C(43)-P(3)	121.8(3)
C(45)-C(44)-C(43)	119.8(4)
C(45)-C(44)-H(44)	120.1
C(43)-C(44)-H(44)	120.1
C(46)-C(45)-C(44)	120.5(4)
C(46)-C(45)-H(45)	119.8
C(44)-C(45)-H(45)	119.8
C(45)-C(46)-C(47)	120.5(4)
C(45)-C(46)-H(46)	119.8
C(47)-C(46)-H(46)	119.8
C(46)-C(47)-C(48)	120.1(4)
C(46)-C(47)-H(47)	120.0
C(48)-C(47)-H(47)	120.0
C(47)-C(48)-C(43)	119.5(4)
C(47)-C(48)-H(48)	120.2
C(43)-C(48)-H(48)	120.2
C(54)-C(49)-C(50)	118.9(3)
C(54)-C(49)-P(3)	122.9(2)
C(50)-C(49)-P(3)	117.5(2)
C(51)-C(50)-C(49)	120.4(3)
C(51)-C(50)-H(50)	119.8
C(49)-C(50)-H(50)	119.8
C(52)-C(51)-C(50)	120.2(3)
C(52)-C(51)-H(51)	119.9
C(50)-C(51)-H(51)	119.9
C(53)-C(52)-C(51)	120.0(3)
C(53)-C(52)-H(52)	120.0
C(51)-C(52)-H(52)	120.0
C(52)-C(53)-C(54)	120.1(3)
C(52)-C(53)-H(53)	120.0
C(54)-C(53)-H(53)	120.0
C(53)-C(54)-C(49)	120.4(3)
C(53)-C(54)-H(54)	119.8
C(49)-C(54)-H(54)	119.8

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 0906jc19b. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
P(1)	24(1)	22(1)	19(1)	0(1)	5(1)	-1(1)
P(2)	25(1)	31(1)	17(1)	-2(1)	3(1)	5(1)
P(3)	25(1)	28(1)	15(1)	0(1)	5(1)	-2(1)
Fe	26(1)	22(1)	15(1)	0(1)	5(1)	-2(1)
Cu	26(1)	24(1)	17(1)	-2(1)	4(1)	1(1)
I	48(1)	26(1)	25(1)	-6(1)	9(1)	-2(1)
C(1)	24(1)	23(1)	20(1)	1(1)	7(1)	-1(1)
C(2)	27(1)	24(1)	18(1)	3(1)	8(1)	4(1)
C(3)	29(2)	25(1)	18(1)	0(1)	8(1)	2(1)
C(4)	32(2)	21(1)	19(1)	2(1)	9(1)	3(1)
C(5)	26(1)	25(1)	21(1)	3(1)	7(1)	1(1)
C(6)	24(1)	32(2)	16(1)	0(1)	5(1)	-4(1)
C(7)	22(1)	32(2)	18(1)	-2(1)	6(1)	-3(1)
C(8)	25(2)	36(2)	19(1)	-2(1)	5(1)	-4(1)
C(9)	31(2)	33(2)	23(1)	-3(1)	7(1)	-10(1)
C(10)	35(2)	30(2)	19(1)	2(1)	6(1)	-7(1)
C(11)	37(2)	31(2)	17(1)	0(1)	6(1)	4(1)
C(12)	42(2)	43(2)	20(1)	2(1)	2(1)	10(2)
C(13)	49(2)	36(2)	20(1)	-5(1)	4(1)	0(2)
C(14)	48(2)	40(2)	21(1)	2(1)	14(1)	3(2)
C(15)	45(2)	32(2)	31(2)	-6(1)	6(1)	-13(2)
C(16)	76(3)	54(2)	50(2)	-8(2)	23(2)	-38(2)
C(17)	43(2)	48(2)	43(2)	-13(2)	-1(2)	-13(2)
C(18)	61(2)	28(2)	36(2)	-4(1)	0(2)	-10(2)
C(19)	24(1)	28(2)	25(1)	3(1)	7(1)	-1(1)
C(20)	36(2)	26(2)	41(2)	0(1)	16(1)	2(1)
C(21)	47(2)	24(2)	58(2)	5(2)	22(2)	1(2)
C(22)	49(2)	30(2)	55(2)	14(2)	25(2)	1(2)
C(23)	54(2)	37(2)	51(2)	5(2)	33(2)	1(2)
C(24)	50(2)	24(2)	42(2)	0(1)	26(2)	-2(1)
C(25)	25(2)	27(1)	27(1)	5(1)	2(1)	-6(1)
C(26)	26(2)	40(2)	39(2)	2(1)	4(1)	-2(1)
C(27)	28(2)	52(2)	52(2)	8(2)	-2(2)	4(2)
C(28)	33(2)	58(2)	45(2)	17(2)	-10(2)	-5(2)
C(29)	39(2)	47(2)	29(2)	8(1)	-5(1)	-11(2)
C(30)	30(2)	34(2)	27(2)	4(1)	1(1)	-5(1)
C(31)	28(2)	57(2)	16(1)	-2(1)	4(1)	12(2)
C(32)	48(2)	66(3)	33(2)	-12(2)	8(2)	22(2)
C(33)	63(3)	100(4)	48(2)	-14(2)	15(2)	43(3)
C(34)	40(2)	140(5)	43(2)	-6(3)	17(2)	36(3)
C(35)	29(2)	117(4)	48(2)	-5(3)	11(2)	3(2)
C(36)	31(2)	79(3)	32(2)	-4(2)	8(1)	4(2)
C(37)	30(2)	27(1)	21(1)	-2(1)	5(1)	9(1)
C(38)	31(2)	35(2)	22(1)	-3(1)	2(1)	6(1)
C(39)	42(2)	40(2)	22(1)	-2(1)	-3(1)	12(2)
C(40)	57(2)	30(2)	25(2)	4(1)	8(1)	13(2)
C(41)	50(2)	26(2)	33(2)	1(1)	14(2)	4(1)
C(42)	37(2)	28(2)	26(2)	-1(1)	5(1)	6(1)
C(43)	25(2)	54(2)	12(1)	0(1)	5(1)	-3(1)
C(44)	30(2)	65(2)	20(1)	-7(2)	4(1)	1(2)
C(45)	34(2)	93(3)	24(2)	-12(2)	7(1)	11(2)

C(46)	29(2)	128(4)	27(2)	-6(2)	8(2)	-4(2)
C(47)	37(2)	105(4)	31(2)	3(2)	6(2)	-30(2)
C(48)	34(2)	64(2)	23(2)	2(2)	4(1)	-15(2)
C(49)	27(1)	26(1)	18(1)	2(1)	4(1)	-5(1)
C(50)	36(2)	32(2)	18(1)	-1(1)	4(1)	-2(1)
C(51)	43(2)	38(2)	16(1)	2(1)	1(1)	-2(1)
C(52)	38(2)	34(2)	27(2)	7(1)	-1(1)	-1(1)
C(53)	34(2)	26(2)	32(2)	1(1)	5(1)	-1(1)
C(54)	34(2)	28(2)	19(1)	1(1)	9(1)	-4(1)

Table 1. Crystal data and structure refinement for copper adduct **6**

Compound	**0906jc20**
Formula	**$C_{44}H_{49}CuFeIP_3$, 1.5(C_7H_8)**
M	**1055.23**
T; K	**115(2)**
Crystal system	**Triclinic**
Space group	**P -1**
a; Å	**12.9274(3)**
b; Å	**14.1887(3)**
c; Å	**14.7538(3)**
α; °	**93.026(1)**
β; °	**91.082(1)**
χ; °	**112.970(1)**
V; $Å^3$	**2485.84(9)**
Z	**2**
F(000)	**1082**
D_{calc}; g/cm^3	**1.410**
diffractometer	**Enraf-Nonius KappaCCD**
scan type	**mixture of ϕ rotations and ω scans**
λ; Å	**0.71073**
μ; mm^{-1}	**1.472**
Crystal size; mm^3	**0.3 x 0.2 x 0.125**
sin(θ)/λ max; $Å^{-1}$	**0.65**
Index ranges	**h: -16; 16** **k: -18; 18** **l: -19; 18**
RC = Refl. Collected	**21650**
IRC = independent RC	**11348 [R(int) = 0.0137]**
IRCGT = RC and [I>2σ(I)]	**10474**
Refinement method	**Full-matrix least-squares on F^2**
Data / restraints / parameters	**11348 / 0 / 569**
R for IRCGT	**$R1^a$ = 0.0239, $wR2^b$ = 0.0537**
R for IRC	**$R1^a$ = 0.0273, $wR2^b$ = 0.0559**
Goodness-of-fit[c]	**1.078**
Largest diff. peak and hole; $e.Å^{-3}$	**0.718 and -0.348**

[a] $R1=\Sigma(||F_o|-|F_c||)/\Sigma|F_o|$.

[b] $wR2=[\Sigma w(F_o^2-F_c^2)^2/\Sigma[w(F_o^2)^2]^{1/2}$ where $w=1/[\sigma^2(Fo^2)+(0.0138P)^2+2.1392P]$ where $P=(Max(Fo^2,0)+2*Fc^2)/3$

[c] Goodness of fit $=[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **0906jc20.**
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(106)	5085(6)	4077(6)	−1223(6)	54(4)
C(100)	4911(4)	5895(3)	1114(3)	79(6)
C(105)	4043(3)	4967(3)	889(3)	47(1)
C(104)	4098(3)	4379(3)	135(3)	44(5)
C(103)	5020(3)	4718(3)	−393(3)	47(1)
C(102)	5888(3)	5646(4)	−168(3)	55(6)
C(101)	5833(4)	6234(3)	585(3)	67(2)
P(1)	7685(1)	3449(1)	1881(1)	20(1)
P(2)	7843(1)	1033(1)	2101(1)	17(1)
P(3)	10231(1)	2880(1)	2187(1)	18(1)
Fe	8483(1)	2790(1)	3927(1)	18(1)
Cu	8610(1)	2483(1)	1312(1)	18(1)
I	8659(1)	2189(1)	−421(1)	24(1)
C(1)	7803(2)	3577(1)	3120(1)	21(1)
C(2)	6995(2)	2988(1)	3739(1)	25(1)
C(3)	7437(2)	3322(2)	4641(1)	29(1)
C(4)	8519(2)	4117(2)	4598(1)	30(1)
C(5)	8755(2)	4270(1)	3663(1)	24(1)
C(6)	8516(1)	1536(1)	3207(1)	19(1)
C(7)	9612(1)	2387(1)	3242(1)	19(1)
C(8)	9974(1)	2635(1)	4178(1)	21(1)
C(9)	9144(2)	1957(1)	4724(1)	22(1)
C(10)	8255(2)	1282(1)	4124(1)	22(1)
C(11)	6139(2)	2838(2)	1671(1)	26(1)
C(12)	5854(2)	2573(2)	656(2)	44(1)
C(13)	5415(2)	3373(2)	2086(2)	35(1)
C(14)	8277(2)	4817(1)	1607(1)	29(1)
C(15)	8397(2)	4924(2)	590(2)	38(1)
C(16)	7722(2)	5494(2)	2036(2)	38(1)
C(17)	6331(1)	383(1)	2204(1)	21(1)
C(18)	5743(2)	−103(2)	1395(2)	38(1)
C(19)	4585(2)	−606(2)	1362(2)	46(1)
C(20)	3989(2)	−636(2)	2137(2)	36(1)
C(21)	4557(2)	−143(2)	2935(1)	31(1)
C(22)	5725(2)	368(1)	2972(1)	25(1)
C(23)	8235(1)	−57(1)	1873(1)	19(1)
C(24)	7810(2)	−928(1)	2368(1)	28(1)
C(25)	8162(2)	−1723(2)	2203(2)	34(1)
C(26)	8961(2)	−1643(1)	1563(1)	28(1)
C(27)	9390(2)	−780(1)	1079(1)	26(1)
C(28)	9014(2)	7(1)	1221(1)	22(1)
C(29)	11137(1)	2181(1)	1967(1)	21(1)
C(30)	11585(2)	2231(1)	1109(1)	26(1)
C(31)	12237(2)	1682(2)	882(1)	33(1)
C(32)	12438(2)	1069(2)	1503(2)	35(1)
C(33)	11996(2)	1011(2)	2354(2)	36(1)
C(34)	11351(2)	1568(2)	2591(1)	28(1)
C(35)	11260(1)	4186(1)	2458(1)	19(1)
C(36)	11200(2)	4961(1)	1948(1)	26(1)
C(37)	11973(2)	5966(2)	2112(2)	33(1)

C(38)	12813(2)	6198(2)	2780(1)	32(1)
C(39)	12898(2)	5427(1)	3280(1)	28(1)
C(40)	12132(1)	4427(1)	3115(1)	23(1)
C(41)	9259(2)	1866(2)	5738(1)	27(1)
C(42)	9876(2)	1150(2)	5860(2)	49(1)
C(43)	9904(2)	2910(2)	6236(2)	50(1)
C(44)	8108(2)	1390(2)	6149(1)	39(1)
C(45)	6156(2)	6700(2)	4620(2)	47(1)
C(46)	5818(2)	7112(2)	3890(2)	48(1)
C(47)	5551(2)	7961(2)	4014(2)	53(1)
C(48)	5617(2)	8429(2)	4865(2)	53(1)
C(49)	5952(2)	8029(2)	5596(2)	55(1)
C(50)	6212(2)	7176(2)	5476(2)	53(1)
C(51)	6439(3)	5767(3)	4490(2)	69(1)

Table 3. Bond lengths [A] and angles [deg] for **0906jc20.**

C(106)-C(103)	1.511(9)
C(106)-H(10A)	0.9600
C(106)-H(10B)	0.9600
C(106)-H(10C)	0.9600
C(100)-C(105)	1.374(2)
C(100)-C(101)	1.374(2)
C(100)-H(100)	0.9300
C(105)-C(104)	1.374(2)
C(105)-H(105)	0.9300
C(104)-C(103)	1.374(2)
C(104)-H(104)	0.9300
C(103)-C(102)	1.374(2)
C(102)-C(101)	1.374(2)
C(102)-H(102)	0.9300
C(101)-H(101)	0.9300
P(1)-C(1)	1.8249(18)
P(1)-C(11)	1.8545(19)
P(1)-C(14)	1.8583(19)
P(1)-Cu	2.2810(5)
P(2)-C(6)	1.8084(17)
P(2)-C(17)	1.8208(17)
P(2)-C(23)	1.8249(17)
P(2)-Cu	2.2964(5)
P(3)-C(7)	1.8102(17)
P(3)-C(35)	1.8283(17)
P(3)-C(29)	1.8306(17)
P(3)-Cu	2.2968(5)
Fe-C(7)	2.0303(17)
Fe-C(6)	2.0385(16)
Fe-C(5)	2.0501(17)
Fe-C(8)	2.0524(17)
Fe-C(4)	2.0643(18)
Fe-C(2)	2.0654(18)
Fe-C(3)	2.0681(18)
Fe-C(1)	2.0747(18)
Fe-C(10)	2.0799(17)
Fe-C(9)	2.0996(17)
Cu-I	2.5743(2)
C(1)-C(2)	1.433(2)
C(1)-C(5)	1.435(2)
C(2)-C(3)	1.420(3)
C(2)-H(2)	0.9300
C(3)-C(4)	1.419(3)
C(3)-H(3)	0.9300
C(4)-C(5)	1.428(3)
C(4)-H(4)	0.9300
C(5)-H(5)	0.9300
C(6)-C(10)	1.430(2)
C(6)-C(7)	1.457(2)
C(7)-C(8)	1.430(2)
C(8)-C(9)	1.427(2)
C(8)-H(8)	0.9300
C(9)-C(10)	1.427(2)
C(9)-C(41)	1.518(2)
C(10)-H(10)	0.9300
C(11)-C(12)	1.527(3)

C(11)-C(13)	1.535(3)
C(11)-H(11)	0.9800
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(14)-C(15)	1.521(3)
C(14)-C(16)	1.526(3)
C(14)-H(14)	0.9800
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(16)-H(16A)	0.9600
C(16)-H(16B)	0.9600
C(16)-H(16C)	0.9600
C(17)-C(22)	1.385(2)
C(17)-C(18)	1.395(3)
C(18)-C(19)	1.383(3)
C(18)-H(18)	0.9300
C(19)-C(20)	1.384(3)
C(19)-H(19)	0.9300
C(20)-C(21)	1.377(3)
C(20)-H(20)	0.9300
C(21)-C(22)	1.395(2)
C(21)-H(21)	0.9300
C(22)-H(22)	0.9300
C(23)-C(28)	1.387(2)
C(23)-C(24)	1.393(2)
C(24)-C(25)	1.384(3)
C(24)-H(24)	0.9300
C(25)-C(26)	1.388(3)
C(25)-H(25)	0.9300
C(26)-C(27)	1.377(3)
C(26)-H(26)	0.9300
C(27)-C(28)	1.388(2)
C(27)-H(27)	0.9300
C(28)-H(28)	0.9300
C(29)-C(34)	1.394(2)
C(29)-C(30)	1.396(2)
C(30)-C(31)	1.388(3)
C(30)-H(30)	0.9300
C(31)-C(32)	1.384(3)
C(31)-H(31)	0.9300
C(32)-C(33)	1.382(3)
C(32)-H(32)	0.9300
C(33)-C(34)	1.392(3)
C(33)-H(33)	0.9300
C(34)-H(34)	0.9300
C(35)-C(36)	1.390(2)
C(35)-C(40)	1.397(2)
C(36)-C(37)	1.391(3)
C(36)-H(36)	0.9300
C(37)-C(38)	1.380(3)
C(37)-H(37)	0.9300
C(38)-C(39)	1.388(3)
C(38)-H(38)	0.9300

C(39)-C(40)	1.383(2)
C(39)-H(39)	0.9300
C(40)-H(40)	0.9300
C(41)-C(43)	1.525(3)
C(41)-C(44)	1.527(3)
C(41)-C(42)	1.532(3)
C(42)-H(42A)	0.9600
C(42)-H(42B)	0.9600
C(42)-H(42C)	0.9600
C(43)-H(43A)	0.9600
C(43)-H(43B)	0.9600
C(43)-H(43C)	0.9600
C(44)-H(44A)	0.9600
C(44)-H(44B)	0.9600
C(44)-H(44C)	0.9600
C(45)-C(50)	1.388(4)
C(45)-C(46)	1.392(4)
C(45)-C(51)	1.509(4)
C(46)-C(47)	1.382(4)
C(46)-H(46)	0.9300
C(47)-C(48)	1.375(4)
C(47)-H(47)	0.9300
C(48)-C(49)	1.383(4)
C(48)-H(48)	0.9300
C(49)-C(50)	1.380(4)
C(49)-H(49)	0.9300
C(50)-H(50)	0.9300
C(51)-H(51A)	0.9600
C(51)-H(51B)	0.9600
C(51)-H(51C)	0.9600

C(105)-C(100)-C(101)	120.0
C(105)-C(100)-H(100)	120.0
C(101)-C(100)-H(100)	120.0
C(100)-C(105)-C(104)	120.0
C(100)-C(105)-H(105)	120.0
C(104)-C(105)-H(105)	120.0
C(105)-C(104)-C(103)	120.0
C(105)-C(104)-H(104)	120.0
C(103)-C(104)-H(104)	120.0
C(102)-C(103)-C(104)	120.0
C(102)-C(103)-C(106)	119.7(4)
C(104)-C(103)-C(106)	120.3(4)
C(101)-C(102)-C(103)	120.0
C(101)-C(102)-H(102)	120.0
C(103)-C(102)-H(102)	120.0
C(102)-C(101)-C(100)	120.0
C(102)-C(101)-H(101)	120.0
C(100)-C(101)-H(101)	120.0
C(1)-P(1)-C(11)	101.55(8)
C(1)-P(1)-C(14)	101.00(8)
C(11)-P(1)-C(14)	112.36(9)
C(1)-P(1)-Cu	111.22(6)
C(11)-P(1)-Cu	113.55(6)
C(14)-P(1)-Cu	115.49(6)
C(6)-P(2)-C(17)	109.91(8)
C(6)-P(2)-C(23)	102.06(8)
C(17)-P(2)-C(23)	100.93(7)

C(6)-P(2)-Cu	99.57(5)
C(17)-P(2)-Cu	121.86(6)
C(23)-P(2)-Cu	120.71(6)
C(7)-P(3)-C(35)	106.97(8)
C(7)-P(3)-C(29)	102.33(8)
C(35)-P(3)-C(29)	101.89(8)
C(7)-P(3)-Cu	98.95(5)
C(35)-P(3)-Cu	124.09(6)
C(29)-P(3)-Cu	119.88(6)
C(7)-Fe-C(6)	41.97(6)
C(7)-Fe-C(5)	108.14(7)
C(6)-Fe-C(5)	136.12(7)
C(7)-Fe-C(8)	41.01(6)
C(6)-Fe-C(8)	69.26(7)
C(5)-Fe-C(8)	111.08(7)
C(7)-Fe-C(4)	135.09(7)
C(6)-Fe-C(4)	176.39(8)
C(5)-Fe-C(4)	40.61(7)
C(8)-Fe-C(4)	109.76(7)
C(7)-Fe-C(2)	141.86(7)
C(6)-Fe-C(2)	113.45(7)
C(5)-Fe-C(2)	67.99(7)
C(8)-Fe-C(2)	177.02(7)
C(4)-Fe-C(2)	67.61(8)
C(7)-Fe-C(3)	175.24(7)
C(6)-Fe-C(3)	142.77(7)
C(5)-Fe-C(3)	68.01(8)
C(8)-Fe-C(3)	136.87(7)
C(4)-Fe-C(3)	40.15(8)
C(2)-Fe-C(3)	40.19(7)
C(7)-Fe-C(1)	111.04(7)
C(6)-Fe-C(1)	110.14(7)
C(5)-Fe-C(1)	40.71(7)
C(8)-Fe-C(1)	140.46(7)
C(4)-Fe-C(1)	68.25(7)
C(2)-Fe-C(1)	40.51(7)
C(3)-Fe-C(1)	68.11(7)
C(7)-Fe-C(10)	68.71(7)
C(6)-Fe-C(10)	40.62(6)
C(5)-Fe-C(10)	176.60(7)
C(8)-Fe-C(10)	67.54(7)
C(4)-Fe-C(10)	142.60(7)
C(2)-Fe-C(10)	113.54(7)
C(3)-Fe-C(10)	115.20(7)
C(1)-Fe-C(10)	138.56(7)
C(7)-Fe-C(9)	68.64(7)
C(6)-Fe-C(9)	68.58(7)
C(5)-Fe-C(9)	140.80(7)
C(8)-Fe-C(9)	40.18(6)
C(4)-Fe-C(9)	113.08(7)
C(2)-Fe-C(9)	138.95(7)
C(3)-Fe-C(9)	112.34(7)
C(1)-Fe-C(9)	178.47(7)
C(10)-Fe-C(9)	39.92(7)
P(1)-Cu-P(2)	101.401(17)
P(1)-Cu-P(3)	108.109(17)
P(2)-Cu-P(3)	87.265(16)
P(1)-Cu-I	119.375(14)

P(2)-Cu-I	115.995(13)
P(3)-Cu-I	119.034(13)
C(2)-C(1)-C(5)	106.70(16)
C(2)-C(1)-P(1)	127.33(13)
C(5)-C(1)-P(1)	125.92(13)
C(2)-C(1)-Fe	69.40(10)
C(5)-C(1)-Fe	68.73(10)
P(1)-C(1)-Fe	124.99(9)
C(3)-C(2)-C(1)	108.78(16)
C(3)-C(2)-Fe	70.01(10)
C(1)-C(2)-Fe	70.09(10)
C(3)-C(2)-H(2)	125.6
C(1)-C(2)-H(2)	125.6
Fe-C(2)-H(2)	125.9
C(4)-C(3)-C(2)	108.09(17)
C(4)-C(3)-Fe	69.78(10)
C(2)-C(3)-Fe	69.80(10)
C(4)-C(3)-H(3)	126.0
C(2)-C(3)-H(3)	126.0
Fe-C(3)-H(3)	126.0
C(3)-C(4)-C(5)	108.02(16)
C(3)-C(4)-Fe	70.07(10)
C(5)-C(4)-Fe	69.16(10)
C(3)-C(4)-H(4)	126.0
C(5)-C(4)-H(4)	126.0
Fe-C(4)-H(4)	126.4
C(4)-C(5)-C(1)	108.40(16)
C(4)-C(5)-Fe	70.23(10)
C(1)-C(5)-Fe	70.57(10)
C(4)-C(5)-H(5)	125.8
C(1)-C(5)-H(5)	125.8
Fe-C(5)-H(5)	125.0
C(10)-C(6)-C(7)	106.91(14)
C(10)-C(6)-P(2)	135.42(13)
C(7)-C(6)-P(2)	117.63(12)
C(10)-C(6)-Fe	71.25(10)
C(7)-C(6)-Fe	68.71(9)
P(2)-C(6)-Fe	125.93(9)
C(8)-C(7)-C(6)	107.24(14)
C(8)-C(7)-P(3)	133.90(13)
C(6)-C(7)-P(3)	118.85(12)
C(8)-C(7)-Fe	70.33(10)
C(6)-C(7)-Fe	69.31(9)
P(3)-C(7)-Fe	126.16(9)
C(9)-C(8)-C(7)	109.20(15)
C(9)-C(8)-Fe	71.69(10)
C(7)-C(8)-Fe	68.66(9)
C(9)-C(8)-H(8)	125.4
C(7)-C(8)-H(8)	125.4
Fe-C(8)-H(8)	125.8
C(10)-C(9)-C(8)	107.21(15)
C(10)-C(9)-C(41)	125.95(15)
C(8)-C(9)-C(41)	126.28(16)
C(10)-C(9)-Fe	69.30(10)
C(8)-C(9)-Fe	68.13(10)
C(41)-C(9)-Fe	134.30(13)
C(9)-C(10)-C(6)	109.43(15)
C(9)-C(10)-Fe	70.78(10)

C(6)-C(10)-Fe	68.13(9)
C(9)-C(10)-H(10)	125.3
C(6)-C(10)-H(10)	125.3
Fe-C(10)-H(10)	127.4
C(12)-C(11)-C(13)	110.85(16)
C(12)-C(11)-P(1)	110.16(14)
C(13)-C(11)-P(1)	118.50(14)
C(12)-C(11)-H(11)	105.4
C(13)-C(11)-H(11)	105.4
P(1)-C(11)-H(11)	105.4
C(11)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-H(13A)	109.5
C(11)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(15)-C(14)-C(16)	112.30(17)
C(15)-C(14)-P(1)	111.52(14)
C(16)-C(14)-P(1)	116.36(14)
C(15)-C(14)-H(14)	105.2
C(16)-C(14)-H(14)	105.2
P(1)-C(14)-H(14)	105.2
C(14)-C(15)-H(15A)	109.5
C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(14)-C(16)-H(16A)	109.5
C(14)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(14)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(22)-C(17)-C(18)	118.46(16)
C(22)-C(17)-P(2)	127.16(13)
C(18)-C(17)-P(2)	114.30(14)
C(19)-C(18)-C(17)	120.95(19)
C(19)-C(18)-H(18)	119.5
C(17)-C(18)-H(18)	119.5
C(18)-C(19)-C(20)	120.2(2)
C(18)-C(19)-H(19)	119.9
C(20)-C(19)-H(19)	119.9
C(21)-C(20)-C(19)	119.38(18)
C(21)-C(20)-H(20)	120.3
C(19)-C(20)-H(20)	120.3
C(20)-C(21)-C(22)	120.60(18)
C(20)-C(21)-H(21)	119.7
C(22)-C(21)-H(21)	119.7
C(17)-C(22)-C(21)	120.38(17)
C(17)-C(22)-H(22)	119.8
C(21)-C(22)-H(22)	119.8

C(28)-C(23)-C(24)	119.63(16)
C(28)-C(23)-P(2)	118.85(13)
C(24)-C(23)-P(2)	121.45(13)
C(25)-C(24)-C(23)	119.91(17)
C(25)-C(24)-H(24)	120.0
C(23)-C(24)-H(24)	120.0
C(24)-C(25)-C(26)	120.14(17)
C(24)-C(25)-H(25)	119.9
C(26)-C(25)-H(25)	119.9
C(27)-C(26)-C(25)	120.00(17)
C(27)-C(26)-H(26)	120.0
C(25)-C(26)-H(26)	120.0
C(26)-C(27)-C(28)	120.17(16)
C(26)-C(27)-H(27)	119.9
C(28)-C(27)-H(27)	119.9
C(23)-C(28)-C(27)	120.10(16)
C(23)-C(28)-H(28)	120.0
C(27)-C(28)-H(28)	120.0
C(34)-C(29)-C(30)	119.02(17)
C(34)-C(29)-P(3)	123.50(14)
C(30)-C(29)-P(3)	117.39(13)
C(31)-C(30)-C(29)	120.47(18)
C(31)-C(30)-H(30)	119.8
C(29)-C(30)-H(30)	119.8
C(32)-C(31)-C(30)	120.11(19)
C(32)-C(31)-H(31)	119.9
C(30)-C(31)-H(31)	119.9
C(33)-C(32)-C(31)	119.89(18)
C(33)-C(32)-H(32)	120.1
C(31)-C(32)-H(32)	120.1
C(32)-C(33)-C(34)	120.40(19)
C(32)-C(33)-H(33)	119.8
C(34)-C(33)-H(33)	119.8
C(33)-C(34)-C(29)	120.10(18)
C(33)-C(34)-H(34)	119.9
C(29)-C(34)-H(34)	119.9
C(36)-C(35)-C(40)	118.97(16)
C(36)-C(35)-P(3)	117.86(13)
C(40)-C(35)-P(3)	123.08(13)
C(35)-C(36)-C(37)	120.46(18)
C(35)-C(36)-H(36)	119.8
C(37)-C(36)-H(36)	119.8
C(38)-C(37)-C(36)	119.94(18)
C(38)-C(37)-H(37)	120.0
C(36)-C(37)-H(37)	120.0
C(37)-C(38)-C(39)	120.16(18)
C(37)-C(38)-H(38)	119.9
C(39)-C(38)-H(38)	119.9
C(40)-C(39)-C(38)	119.96(18)
C(40)-C(39)-H(39)	120.0
C(38)-C(39)-H(39)	120.0
C(39)-C(40)-C(35)	120.47(17)
C(39)-C(40)-H(40)	119.8
C(35)-C(40)-H(40)	119.8
C(9)-C(41)-C(43)	111.53(16)
C(9)-C(41)-C(44)	111.05(16)
C(43)-C(41)-C(44)	108.19(18)
C(9)-C(41)-C(42)	106.86(16)

C(43)-C(41)-C(42)	110.8(2)
C(44)-C(41)-C(42)	108.41(18)
C(41)-C(42)-H(42A)	109.5
C(41)-C(42)-H(42B)	109.5
H(42A)-C(42)-H(42B)	109.5
C(41)-C(42)-H(42C)	109.5
H(42A)-C(42)-H(42C)	109.5
H(42B)-C(42)-H(42C)	109.5
C(41)-C(43)-H(43A)	109.5
C(41)-C(43)-H(43B)	109.5
H(43A)-C(43)-H(43B)	109.5
C(41)-C(43)-H(43C)	109.5
H(43A)-C(43)-H(43C)	109.5
H(43B)-C(43)-H(43C)	109.5
C(41)-C(44)-H(44A)	109.5
C(41)-C(44)-H(44B)	109.5
H(44A)-C(44)-H(44B)	109.5
C(41)-C(44)-H(44C)	109.5
H(44A)-C(44)-H(44C)	109.5
H(44B)-C(44)-H(44C)	109.5
C(50)-C(45)-C(46)	117.3(3)
C(50)-C(45)-C(51)	121.1(2)
C(46)-C(45)-C(51)	121.5(2)
C(47)-C(46)-C(45)	121.3(2)
C(47)-C(46)-H(46)	119.3
C(45)-C(46)-H(46)	119.3
C(48)-C(47)-C(46)	120.8(3)
C(48)-C(47)-H(47)	119.6
C(46)-C(47)-H(47)	119.6
C(47)-C(48)-C(49)	118.6(3)
C(47)-C(48)-H(48)	120.7
C(49)-C(48)-H(48)	120.7
C(50)-C(49)-C(48)	120.9(3)
C(50)-C(49)-H(49)	119.6
C(48)-C(49)-H(49)	119.6
C(49)-C(50)-C(45)	121.1(3)
C(49)-C(50)-H(50)	119.4
C(45)-C(50)-H(50)	119.4
C(45)-C(51)-H(51A)	109.5
C(45)-C(51)-H(51B)	109.5
H(51A)-C(51)-H(51B)	109.5
C(45)-C(51)-H(51C)	109.5
H(51A)-C(51)-H(51C)	109.5
H(51B)-C(51)-H(51C)	109.5

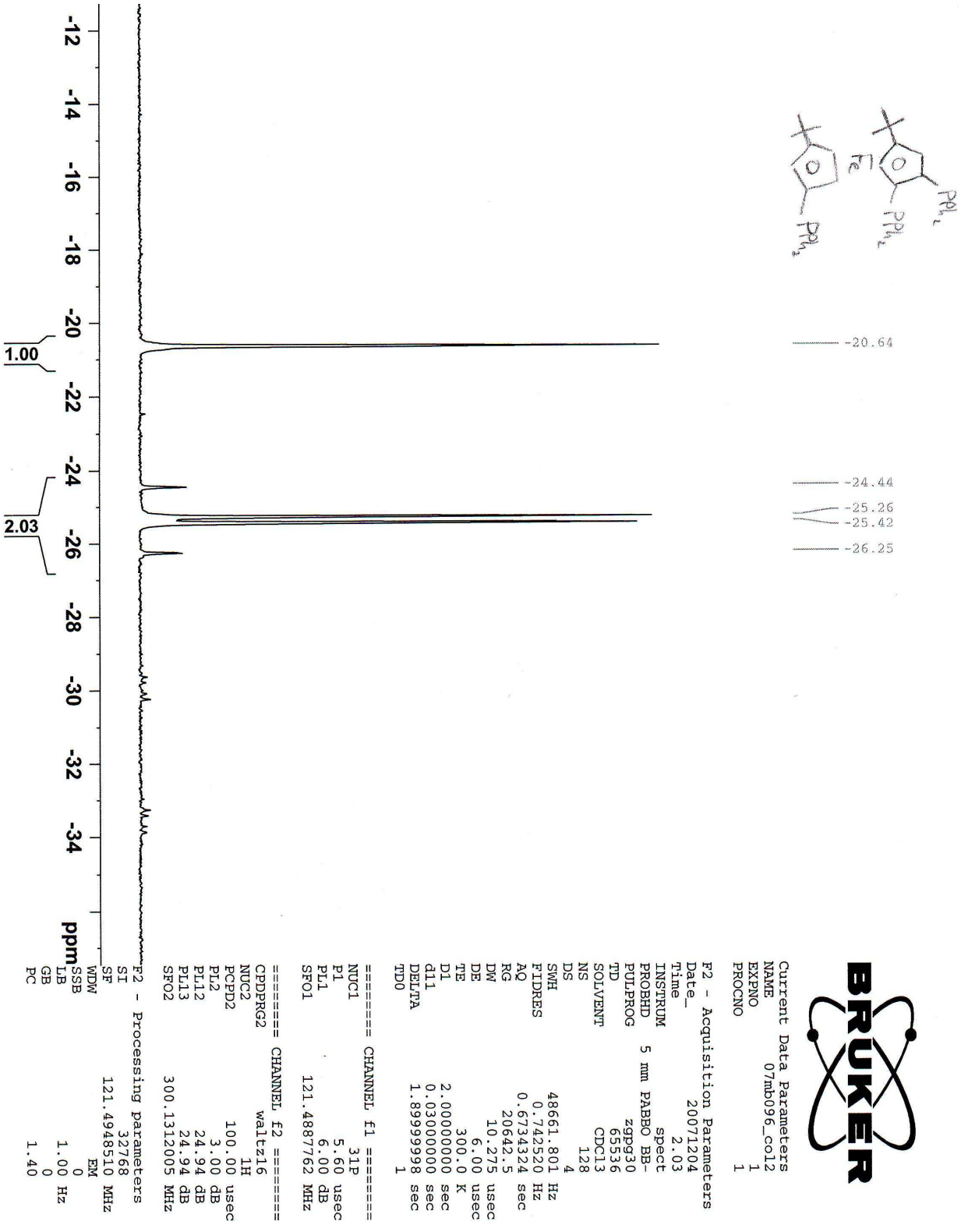
Symmetry transformations used to generate equivalent atoms

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **0906jc20**.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

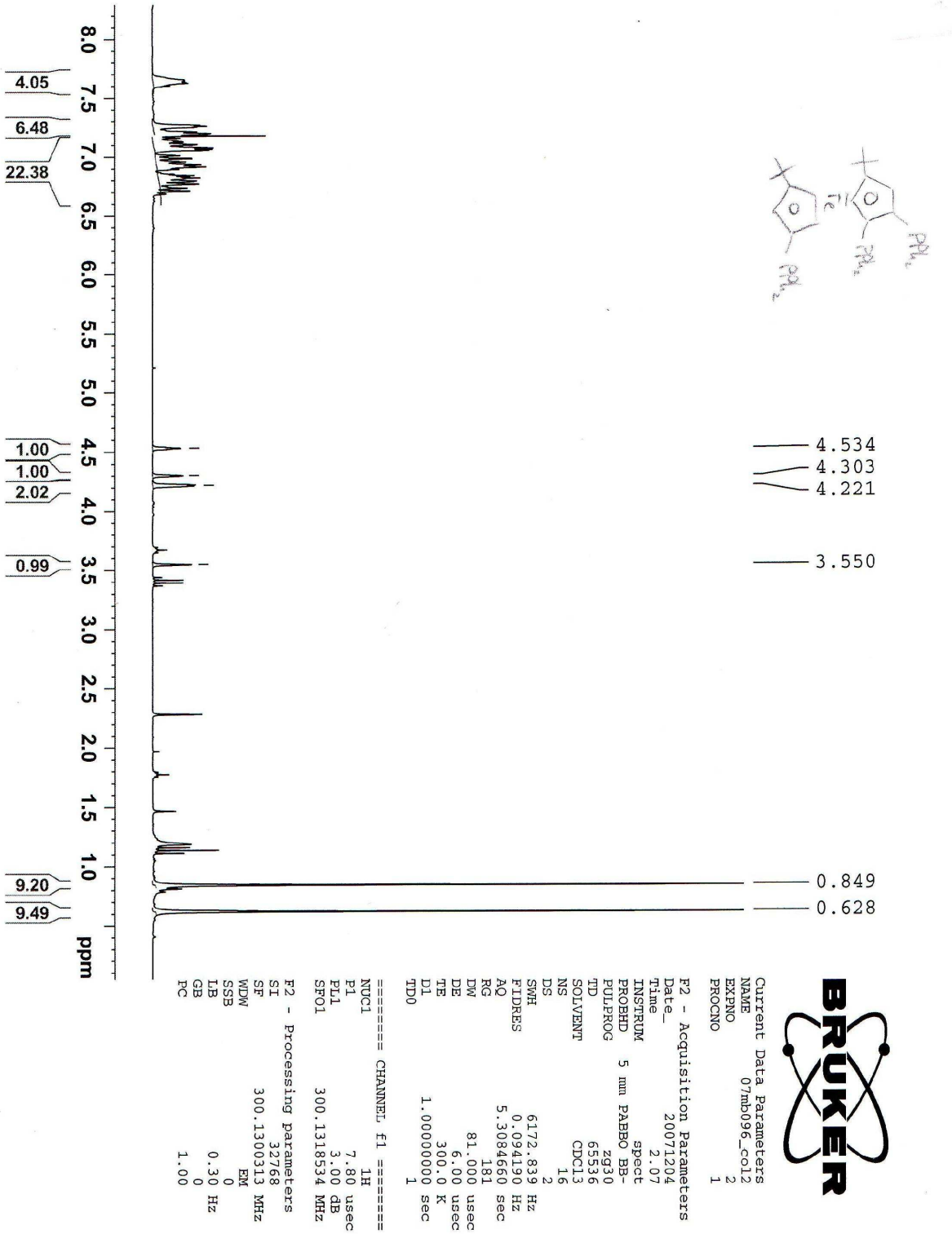
	U11	U22	U33	U23	U13	U12
C(106)	63(7)	89(10)	36(4)	12(4)	15(4)	56(7)
C(100)	103(12)	108(14)	47(7)	9(7)	-19(6)	65(11)
C(105)	46(3)	74(4)	38(3)	18(2)	7(2)	40(3)
C(104)	33(10)	56(9)	53(10)	24(8)	13(7)	26(8)
C(103)	47(3)	85(4)	32(2)	28(3)	11(2)	46(3)
C(102)	37(11)	88(13)	39(9)	28(9)	0(7)	21(10)
C(101)	51(3)	68(4)	68(4)	25(3)	-27(3)	6(3)
P(1)	24(1)	16(1)	20(1)	0(1)	0(1)	10(1)
P(2)	18(1)	14(1)	18(1)	-1(1)	0(1)	6(1)
P(3)	17(1)	19(1)	17(1)	1(1)	0(1)	6(1)
Fe	19(1)	17(1)	17(1)	-2(1)	1(1)	4(1)
Cu	19(1)	17(1)	18(1)	-1(1)	-1(1)	7(1)
I	27(1)	30(1)	18(1)	-1(1)	0(1)	15(1)
C(1)	24(1)	18(1)	24(1)	-3(1)	1(1)	10(1)
C(2)	22(1)	27(1)	25(1)	-1(1)	3(1)	10(1)
C(3)	33(1)	35(1)	22(1)	-2(1)	5(1)	18(1)
C(4)	37(1)	27(1)	25(1)	-12(1)	-5(1)	15(1)
C(5)	28(1)	16(1)	27(1)	-4(1)	-1(1)	6(1)
C(6)	20(1)	15(1)	21(1)	0(1)	1(1)	6(1)
C(7)	18(1)	18(1)	19(1)	1(1)	0(1)	5(1)
C(8)	20(1)	22(1)	20(1)	1(1)	-1(1)	6(1)
C(9)	24(1)	20(1)	18(1)	2(1)	1(1)	6(1)
C(10)	23(1)	18(1)	21(1)	2(1)	3(1)	5(1)
C(11)	24(1)	29(1)	26(1)	-4(1)	-4(1)	14(1)
C(12)	32(1)	68(2)	33(1)	-13(1)	-9(1)	24(1)
C(13)	30(1)	40(1)	40(1)	-2(1)	-1(1)	22(1)
C(14)	34(1)	21(1)	33(1)	2(1)	1(1)	12(1)
C(15)	55(1)	29(1)	36(1)	12(1)	9(1)	19(1)
C(16)	53(1)	22(1)	43(1)	2(1)	6(1)	21(1)
C(17)	19(1)	18(1)	25(1)	-2(1)	-1(1)	7(1)
C(18)	25(1)	57(1)	29(1)	-14(1)	-2(1)	15(1)
C(19)	24(1)	66(2)	40(1)	-25(1)	-9(1)	12(1)
C(20)	19(1)	40(1)	44(1)	-9(1)	-2(1)	7(1)
C(21)	23(1)	34(1)	32(1)	-2(1)	5(1)	7(1)
C(22)	21(1)	24(1)	26(1)	-4(1)	-1(1)	4(1)
C(23)	19(1)	16(1)	20(1)	-2(1)	-1(1)	6(1)
C(24)	33(1)	22(1)	34(1)	8(1)	15(1)	14(1)
C(25)	43(1)	23(1)	40(1)	12(1)	15(1)	17(1)
C(26)	33(1)	23(1)	34(1)	1(1)	6(1)	18(1)
C(27)	26(1)	25(1)	26(1)	-2(1)	7(1)	11(1)
C(28)	26(1)	18(1)	22(1)	1(1)	5(1)	7(1)
C(29)	18(1)	20(1)	23(1)	-1(1)	-1(1)	6(1)
C(30)	24(1)	30(1)	25(1)	1(1)	2(1)	11(1)
C(31)	29(1)	38(1)	33(1)	-1(1)	7(1)	16(1)
C(32)	30(1)	35(1)	45(1)	-5(1)	1(1)	20(1)
C(33)	37(1)	35(1)	43(1)	6(1)	-2(1)	22(1)
C(34)	30(1)	31(1)	26(1)	3(1)	0(1)	15(1)
C(35)	18(1)	19(1)	20(1)	2(1)	3(1)	5(1)
C(36)	24(1)	29(1)	28(1)	8(1)	2(1)	11(1)
C(37)	34(1)	23(1)	41(1)	11(1)	6(1)	10(1)
C(38)	32(1)	21(1)	38(1)	-1(1)	7(1)	4(1)
C(39)	24(1)	28(1)	24(1)	-2(1)	-1(1)	4(1)
C(40)	22(1)	24(1)	20(1)	4(1)	0(1)	7(1)

C(41)	31(1)	28(1)	18(1)	3(1)	0(1)	6(1)
C(42)	60(2)	75(2)	29(1)	15(1)	2(1)	42(1)
C(43)	62(2)	41(1)	21(1)	1(1)	−7(1)	−7(1)
C(44)	42(1)	47(1)	25(1)	10(1)	9(1)	11(1)
C(45)	35(1)	66(2)	37(1)	4(1)	5(1)	16(1)
C(46)	33(1)	63(2)	30(1)	6(1)	3(1)	−1(1)
C(47)	35(1)	56(2)	51(2)	22(1)	−2(1)	−3(1)
C(48)	36(1)	50(2)	62(2)	6(1)	−1(1)	5(1)
C(49)	43(1)	73(2)	45(1)	−3(1)	−1(1)	22(1)
C(50)	49(1)	86(2)	33(1)	4(1)	0(1)	35(1)
C(51)	71(2)	95(2)	52(2)	2(2)	12(2)	46(2)

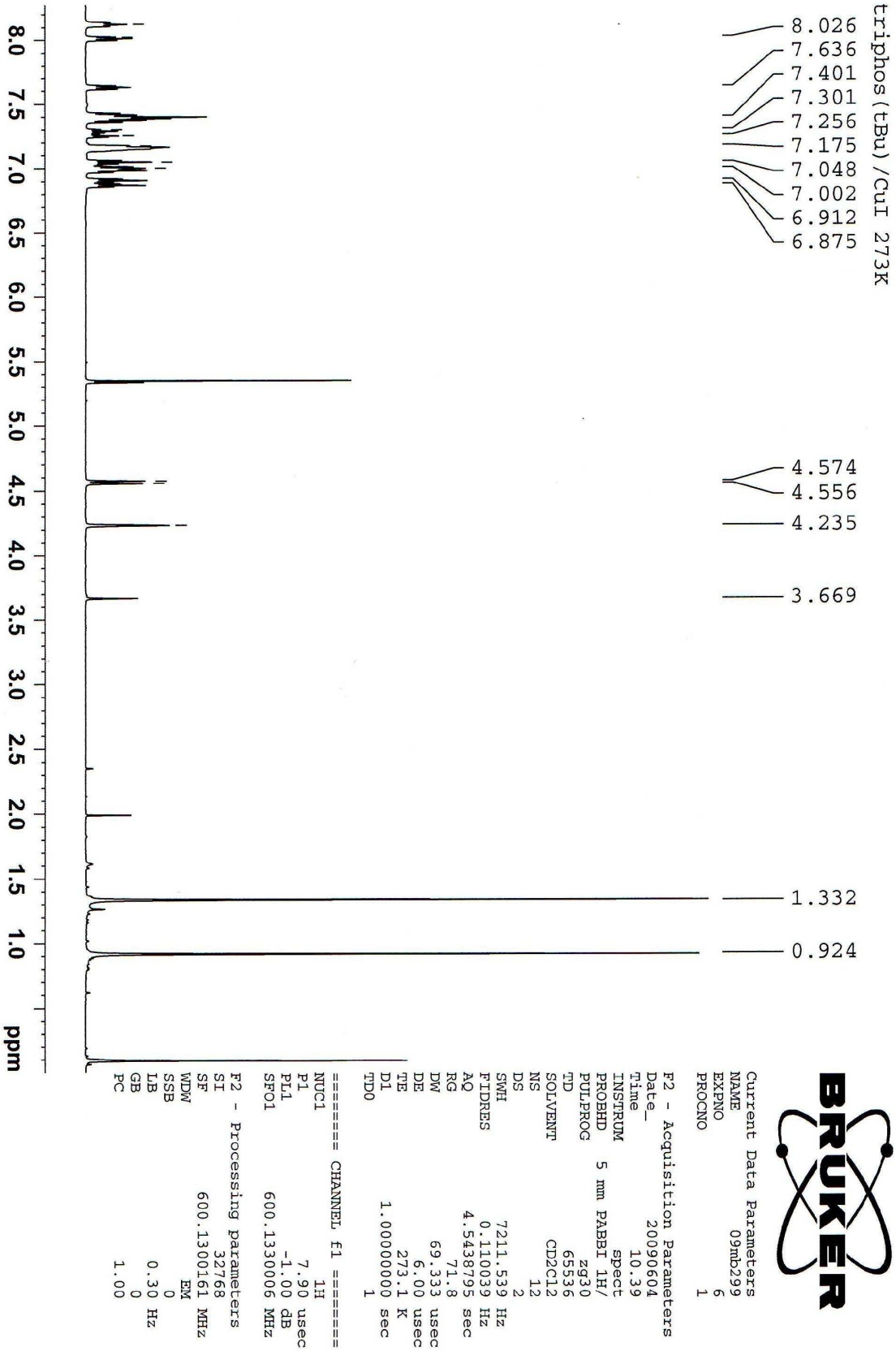
^{31}P NMR of the triphosphine ligand **3**



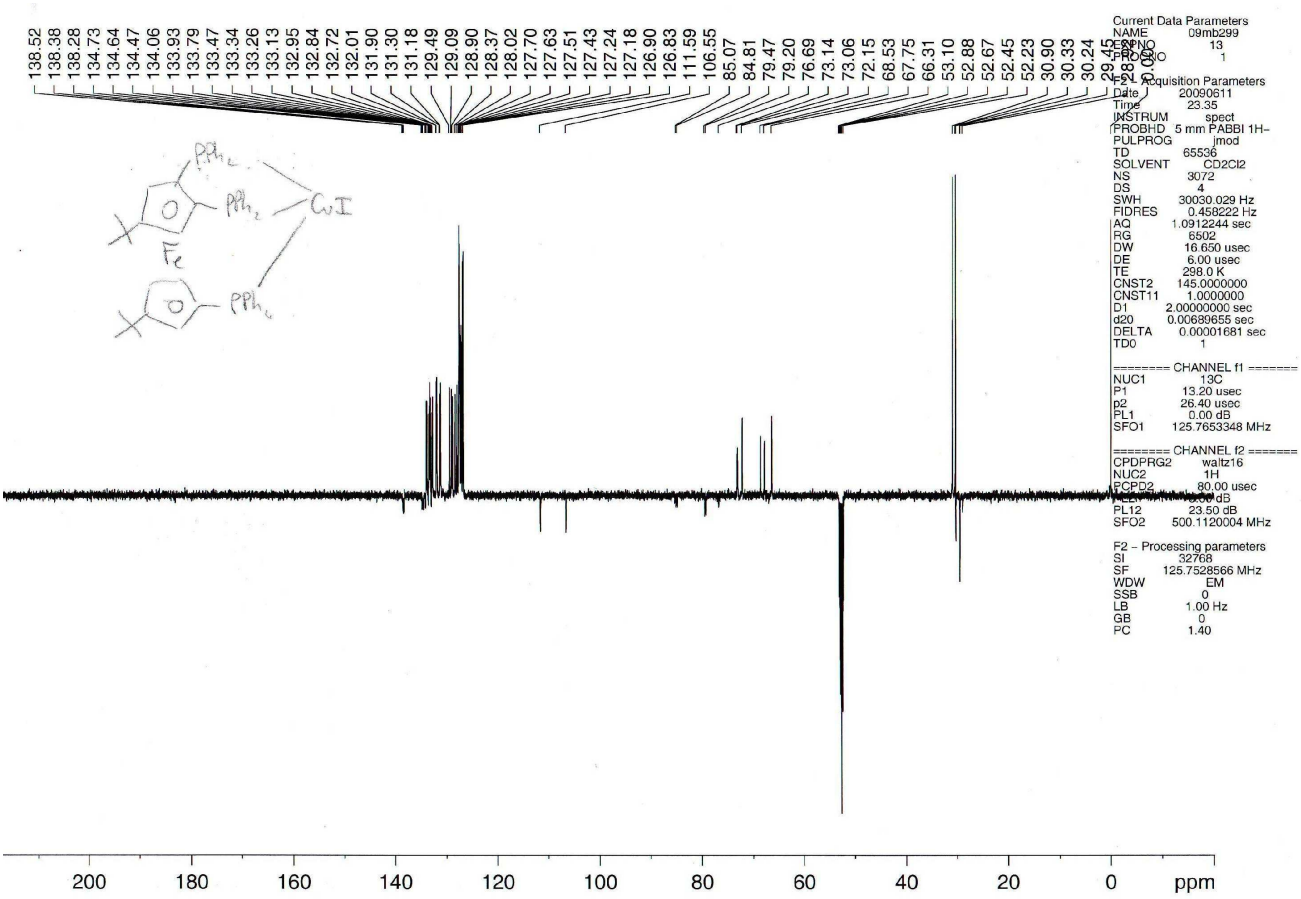
^{1}H NMR of the triphosphine ligand **3**

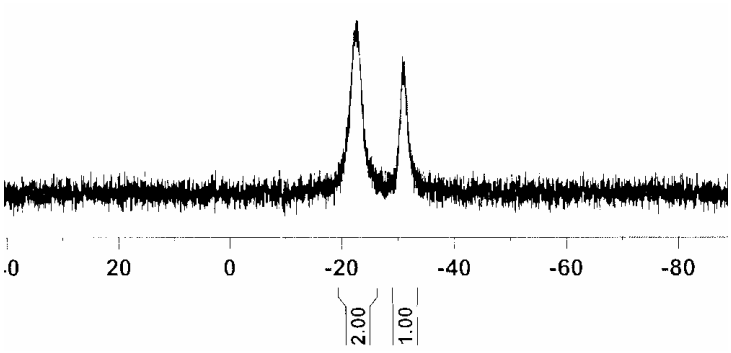


^{1}H NMR of the CuI-triphosphine adduct **4**



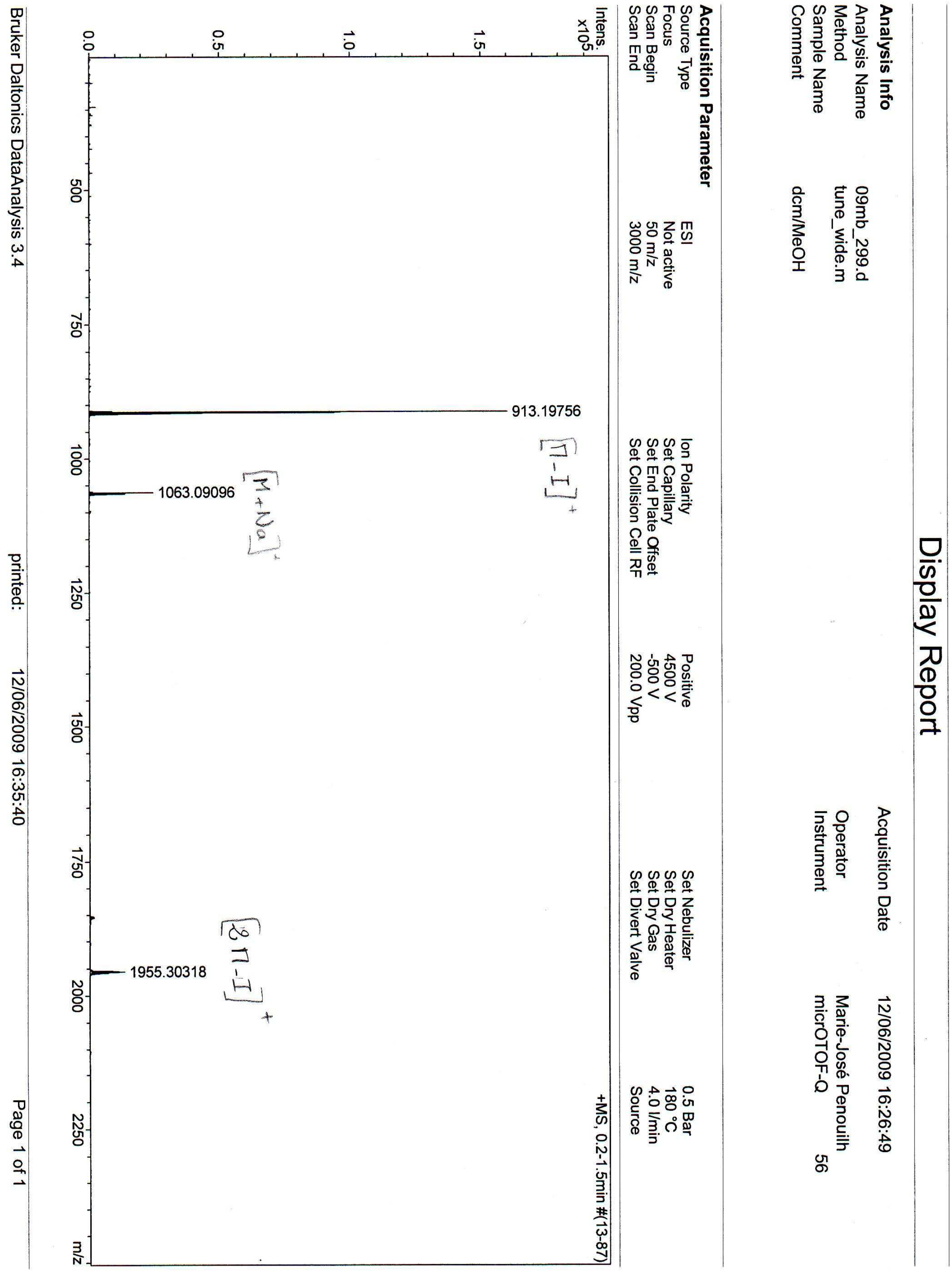
^{13}C NMR of the CuI-triphosphine adduct **4**





^{31}P NMR spectrum of **4** in CD_2Cl_2 at 298 K.

Exact-Mass of copper adduct **4**



<u>Exact-Mass of copper adduct **4** - Molecular peak m/z = 913.2001, σ = 0.030</u>

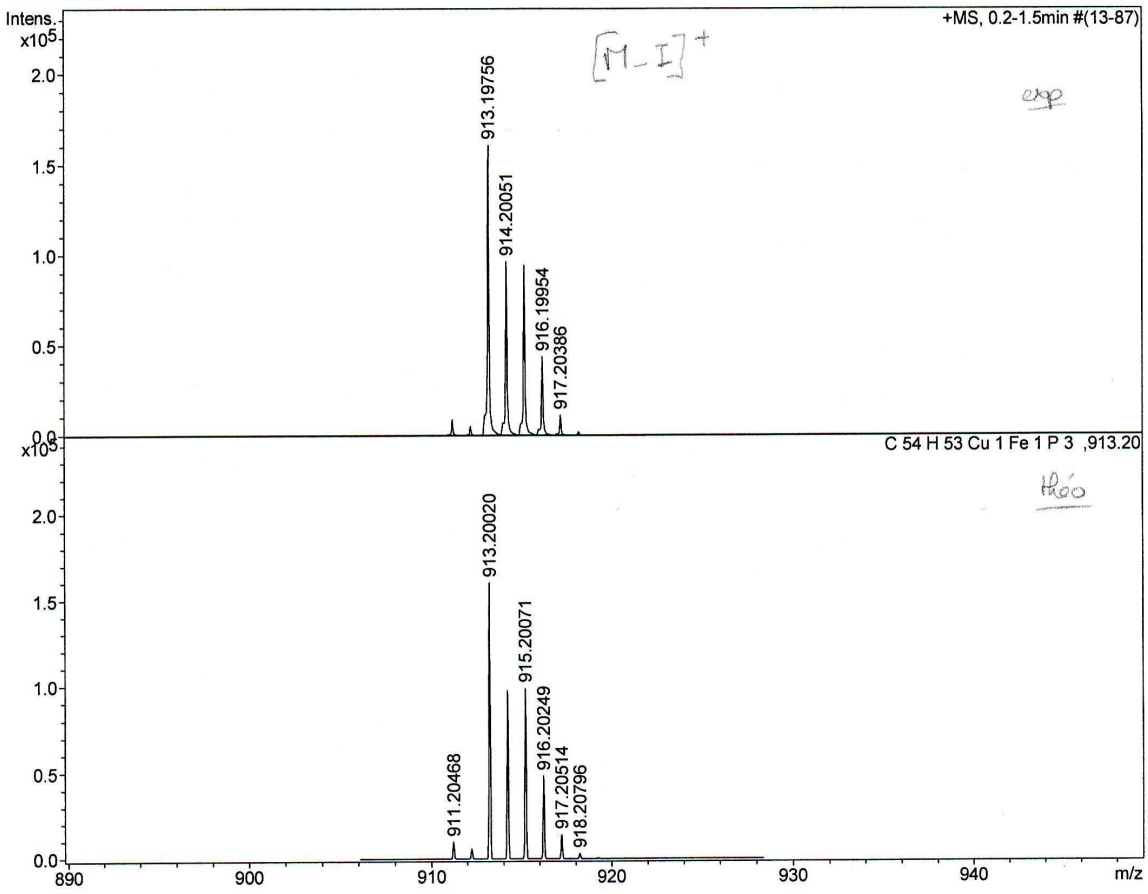
Display Report

Analysis Info

Analysis Name	09mb_299.d	Acquisition Date	12/06/2009 16:26:49
Method	tune_wide.m	Operator	Marie-José Penouilh
Sample Name		Instrument	micrOTOF-Q 56
Comment	dcm/MeOH		

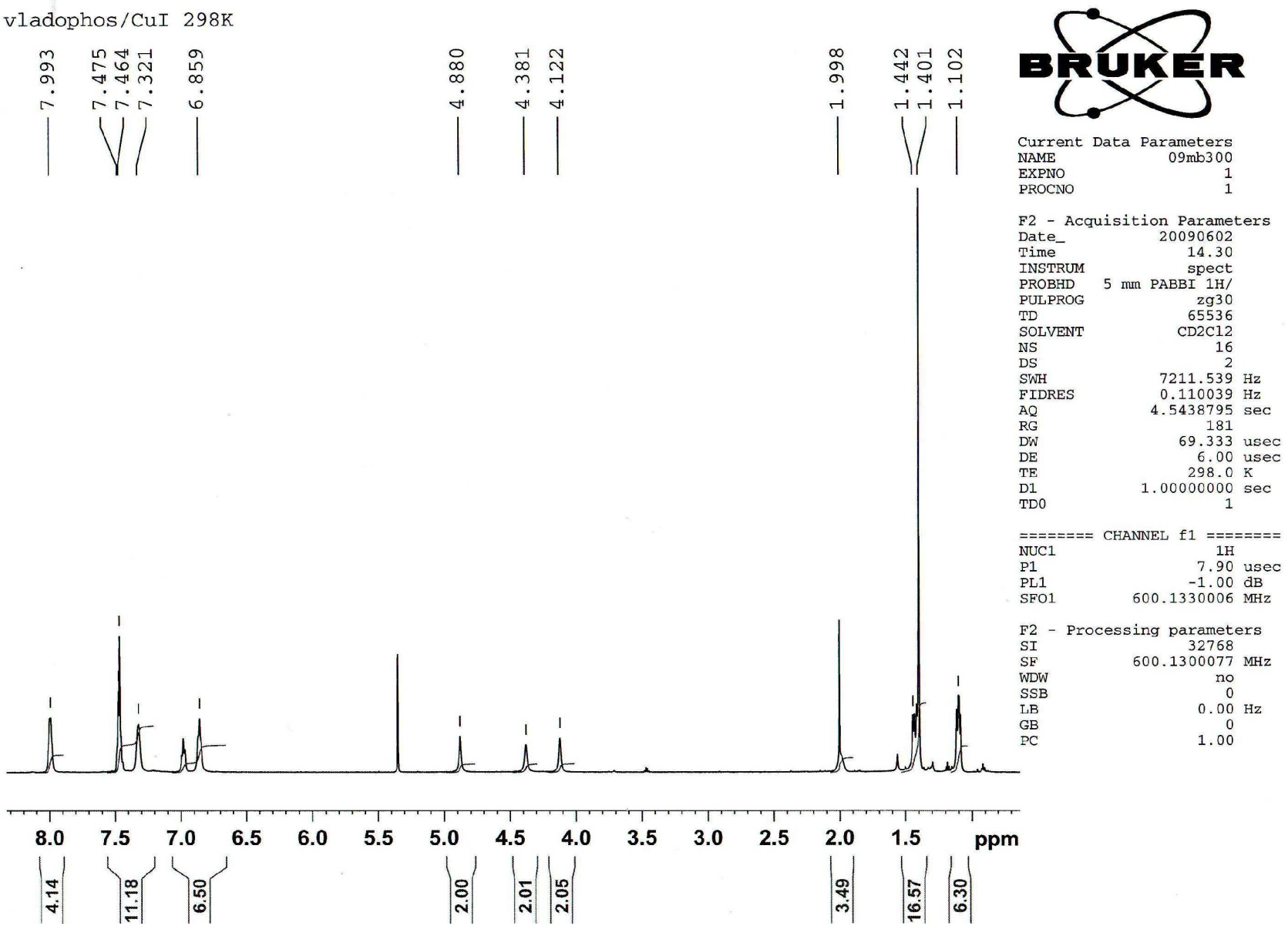
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Source



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 54 H 53 Cu 1 Fe 1 P 3	0.030	913.20001	2.68	2.87	2.45	29.50	ok	even

^{1}H NMR of the CuI-triphosphine adduct **6**



BRUKER

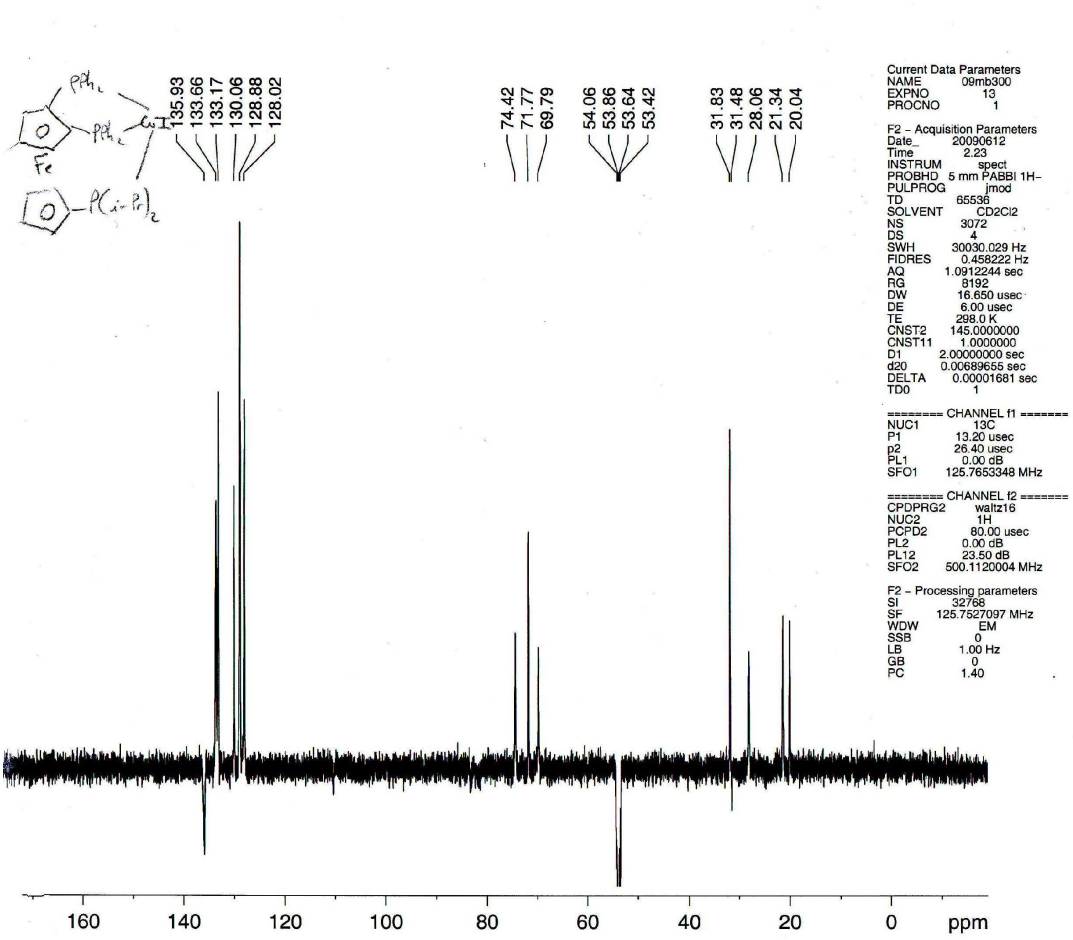
Current Data Parameters
NAME 09mb300
EXPNO 1
PROCNO 1

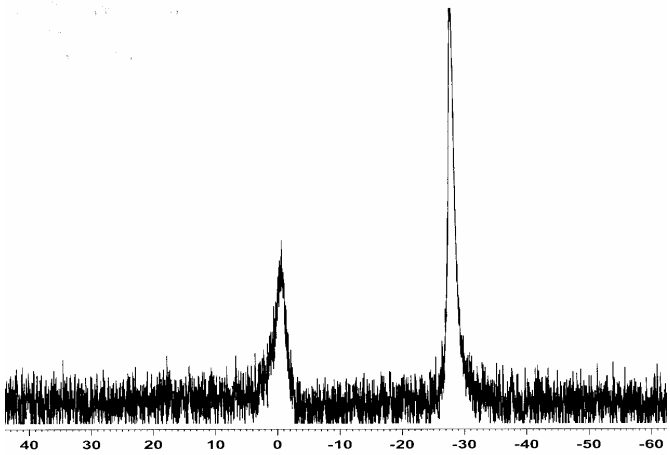
F2 - Acquisition Parameters
Date_ 20090602
Time 14.30
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CD2C12
NS 16
DS 2
SWH 7211.539 Hz
FIDRES 0.110039 Hz
AQ 4.5438795 sec
RG 181
DW 69.333 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -1.00 dB
SFO1 600.1330006 MHz

F2 - Processing parameters
SI 32768
SF 600.1300077 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

^{13}C NMR of the CuI-triphosphine adduct **6**





^{31}P NMR spectrum of **6** in CD_2Cl_2 at 298 K.

Exact-Mass of copper adduct **6**

Display Report

Analysis Info

Analysis Name	09mb_300.d	Acquisition Date	12/06/2009 16:29:25
Method	tune_wide.m		
Sample Name		Operator	Marie-José Penouilh
Comment	dcm/MeOH	Instrument	micrOTOF-Q 56

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Source

Bruker Daltonics DataAnalysis 3.4 printed: 12/06/2009 16:34:07 Page 1 of 1

Exact-Mass of copper adduct **6** - Molecular peak m/z = 789.16871, σ = 0.033

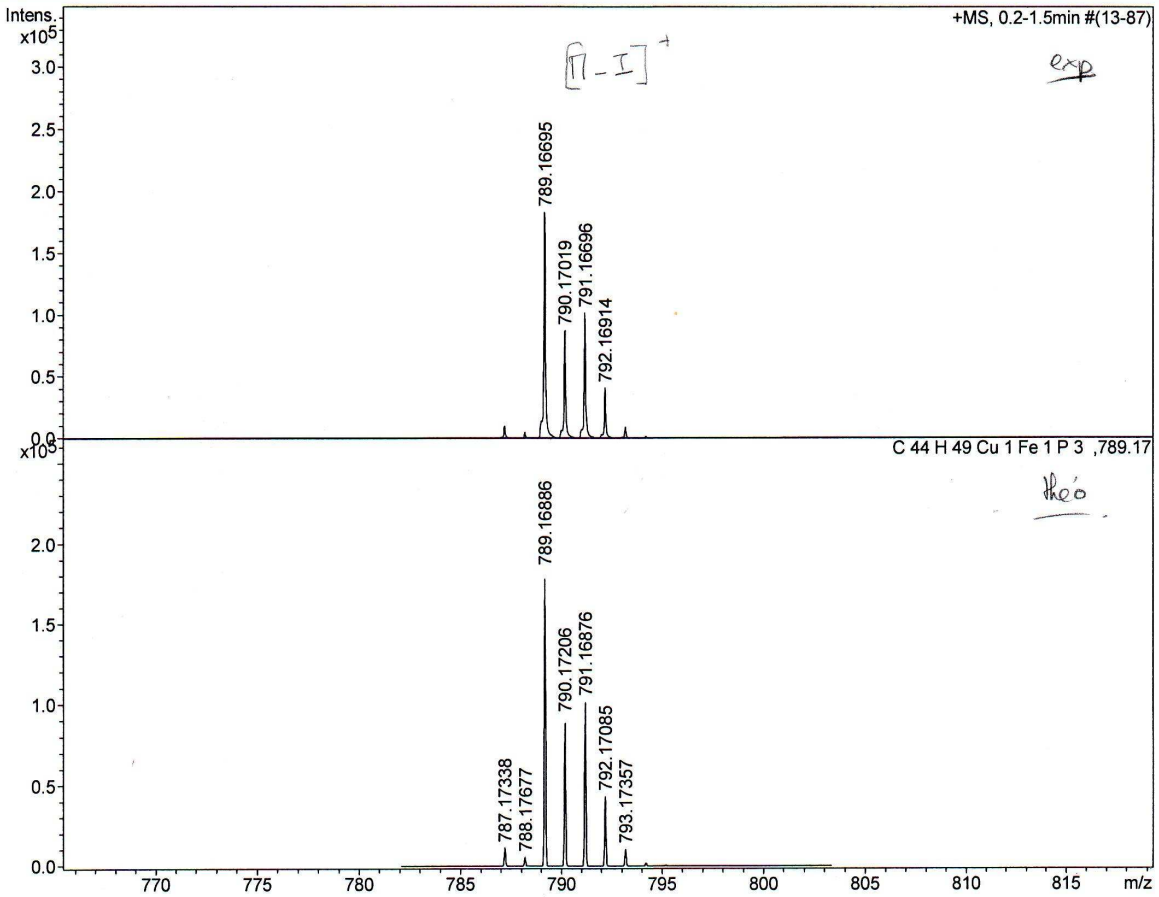
Display Report

Analysis Info

Analysis Name	09mb_300.d	Acquisition Date	12/06/2009 16:29:25
Method	tune_wide.m	Operator	Marie-José Penouilh
Sample Name		Instrument	micrOTOF-Q 56
Comment	dcm/MeOH		

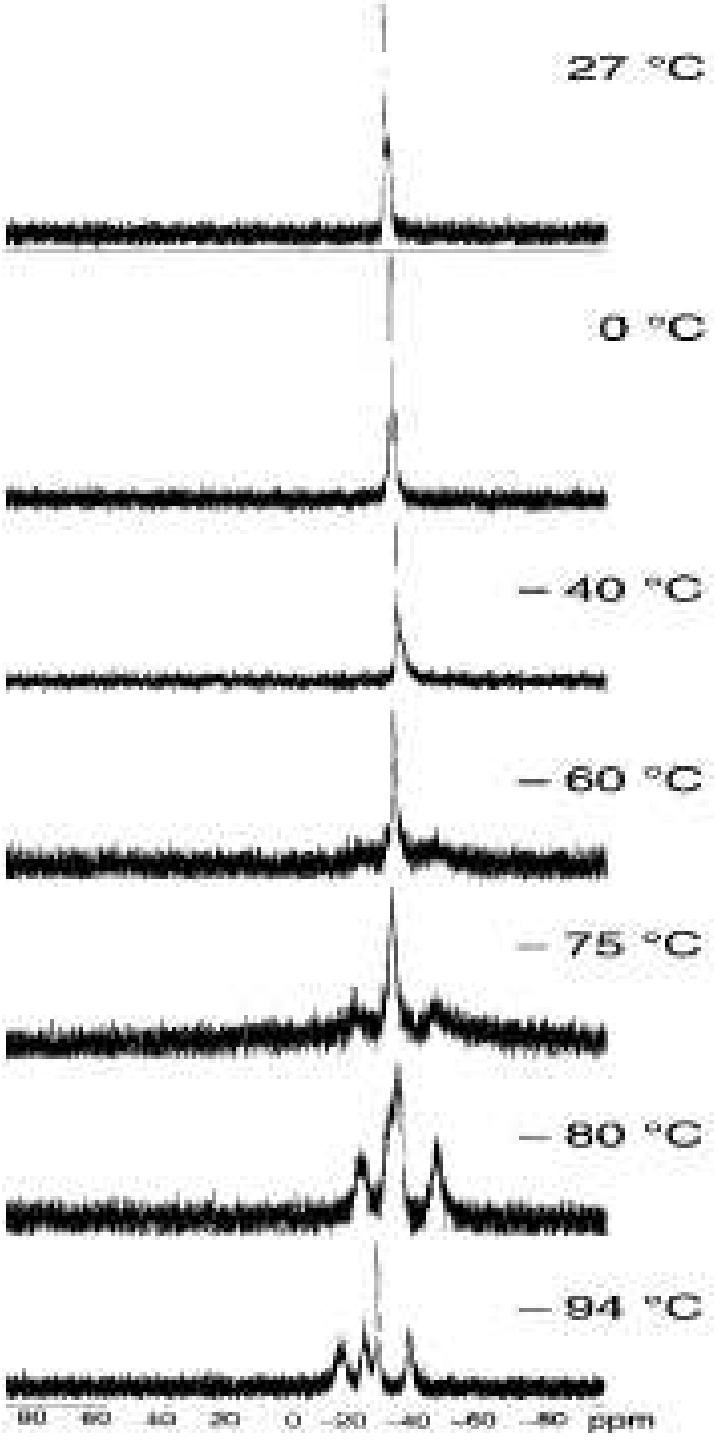
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Source



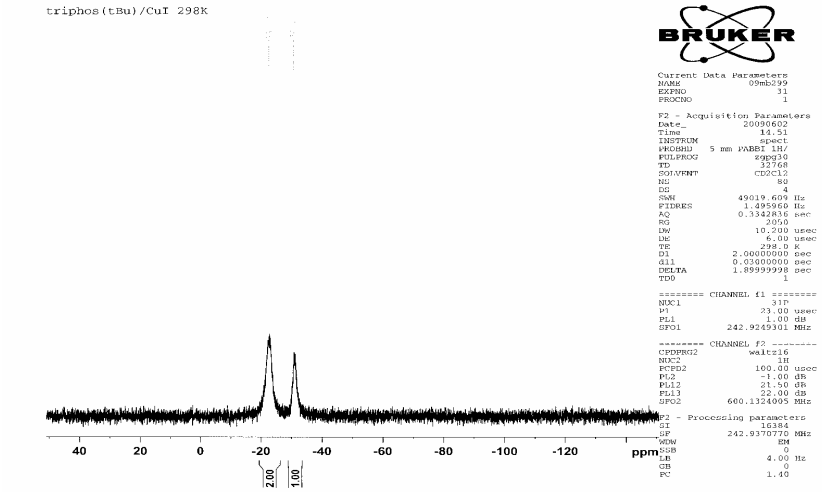
Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 44 H 49 Cu 1 Fe 1 P 3	0.033	789.16871	2.22	2.33	1.75	21.50	ok	even

VT-31P NMR of crystals of **2** solubilised in CD_2Cl_2 (from ref. [12]): *fluxional behaviour of copper on tetraphosphine.*

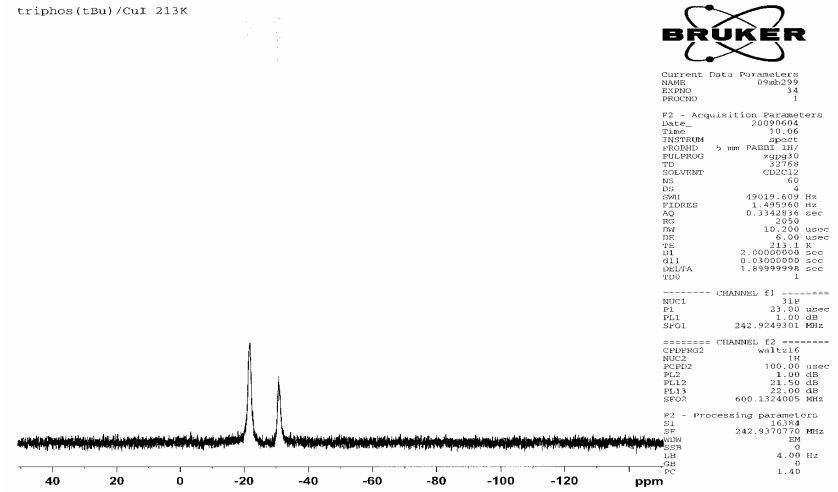


VT-31P NMR of crystals of **4** solubilised in CD_2Cl_2:
No fluxional behaviour of copper-triphosphane detected

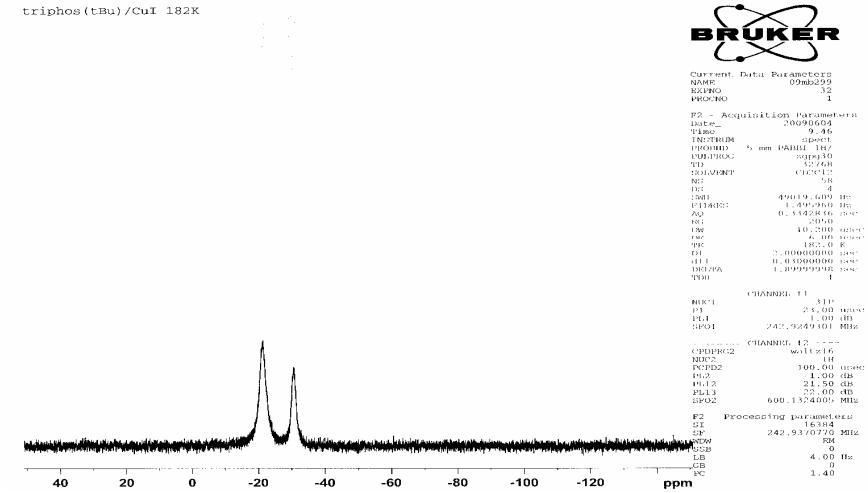
298 K (25 °C)



213 K (−60 °C)

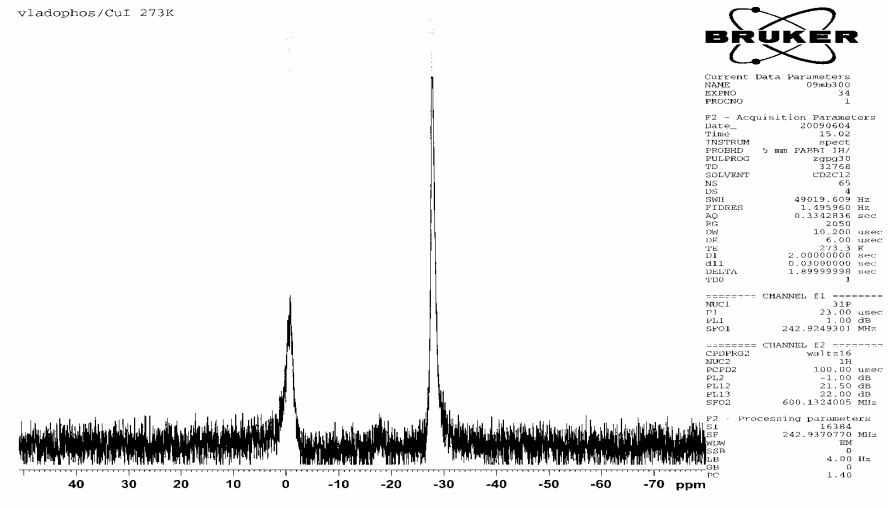


183 K (−90 °C)

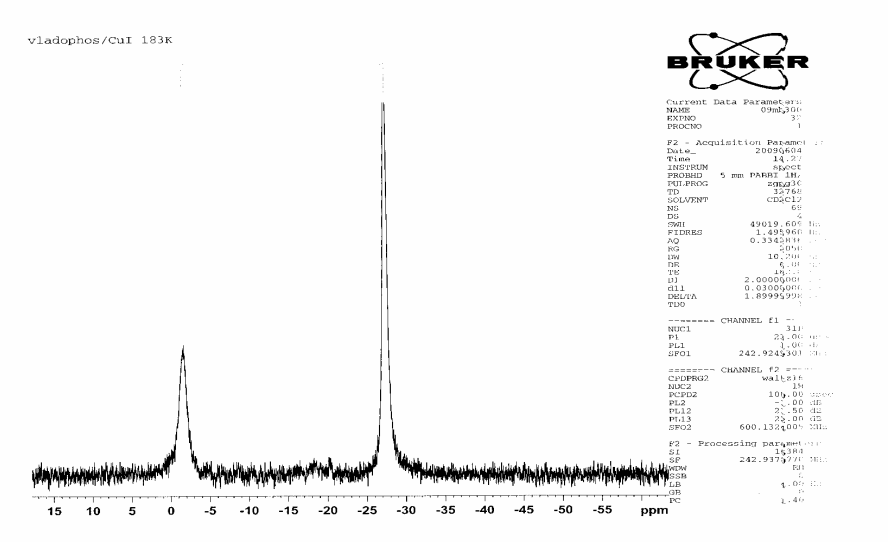


VT-31P NMR of crystals of **6** solubilised in CD_2Cl_2:
No fluxional behaviour of copper-triphosphane detected

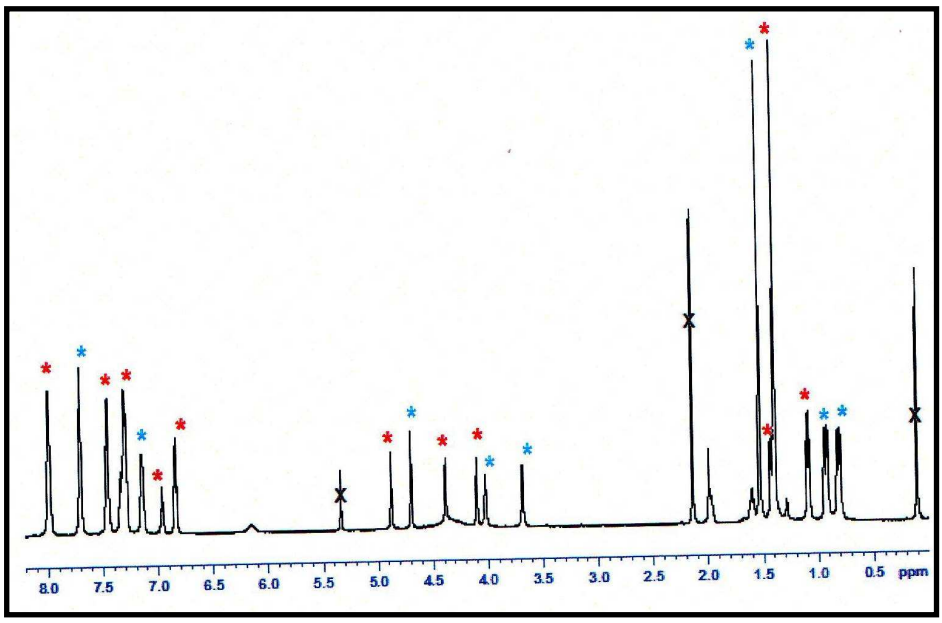
273 K (0 °C)



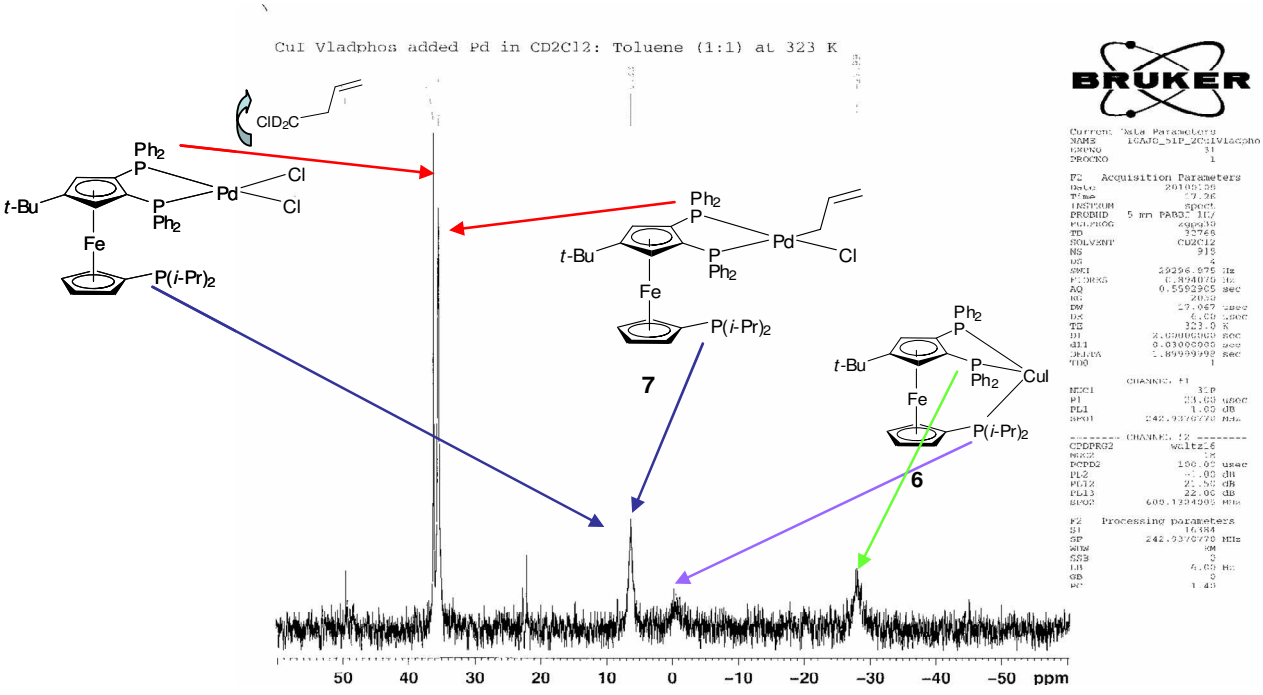
183 K (−90 °C)



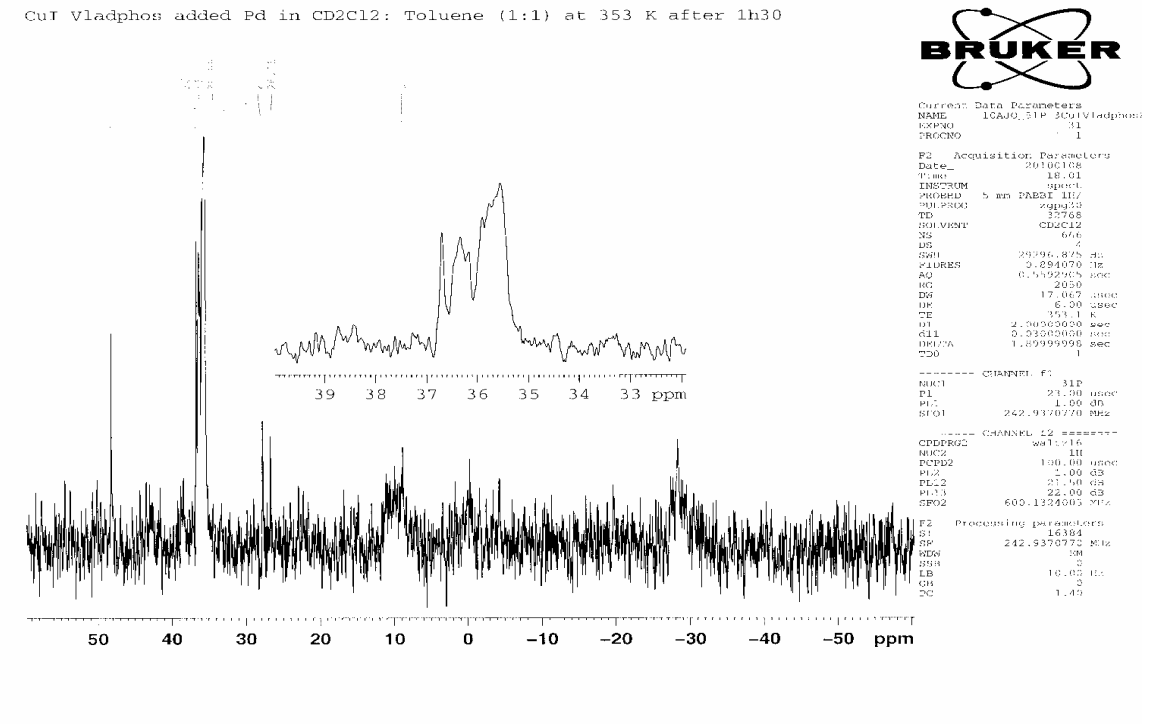
^{1}H NMR of a mixture of **6** with [Pd(allyl)Cl]$_2$ in CD_2Cl_2 after 30 min: *An equilibrium about 50:50 is obtained between copper-adduct **6** (red stars) and Pd complex **7** (blue stars)*



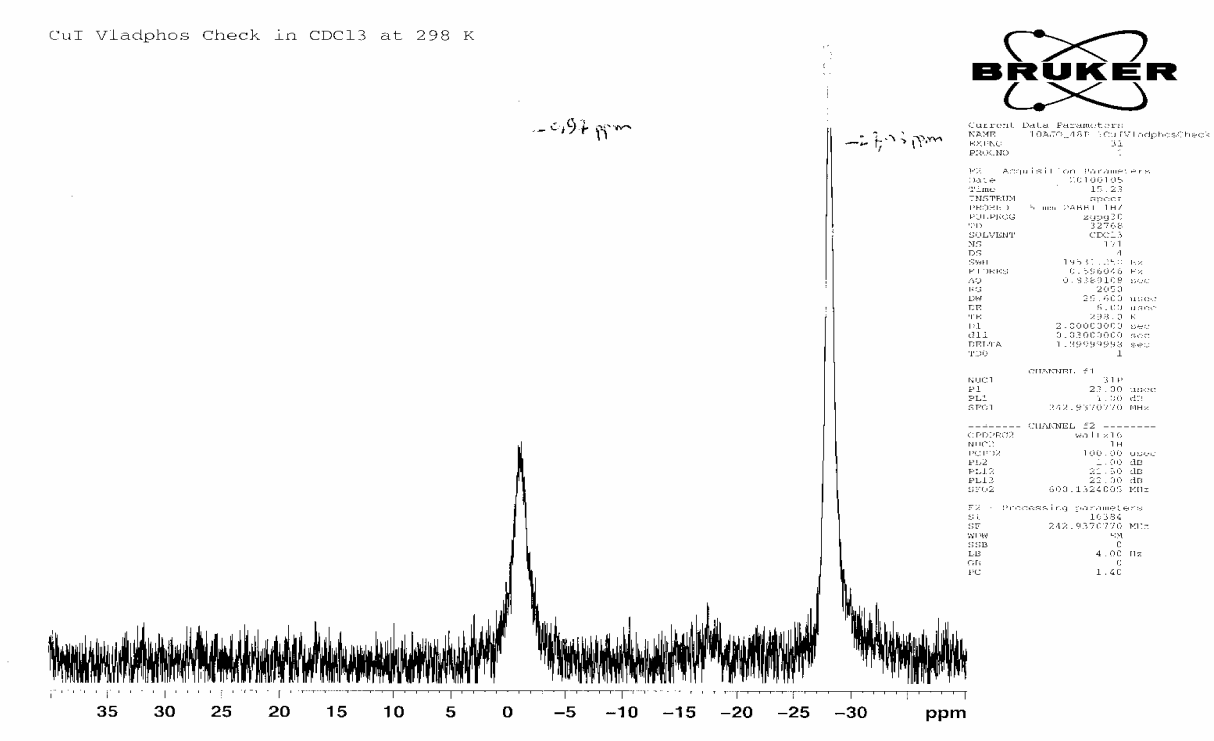
VT-31P NMR of a mixture **6** with [Pd(allyl)Cl]$_2$ in CD_2Cl_2:Toluene (1:1) after 1h at 323 K: *the equilibrium found between copper and palladium species (see Figure 6) is not significantly modified.*



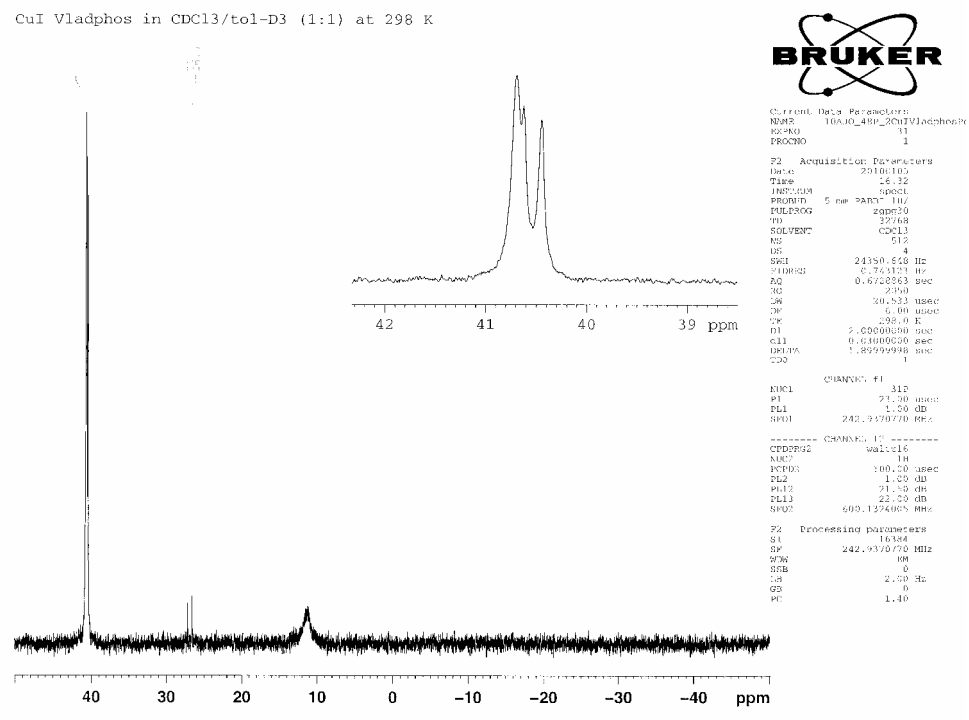
VT-31P NMR of a mixture **6** with $[Pd(allyl)Cl)]_2$ in CD_2Cl_2 : toluene (1:1) after 1h at 353 K: *copper-phosphine interactions still detectable but very difficult to estimate at -28.3 ppm.*



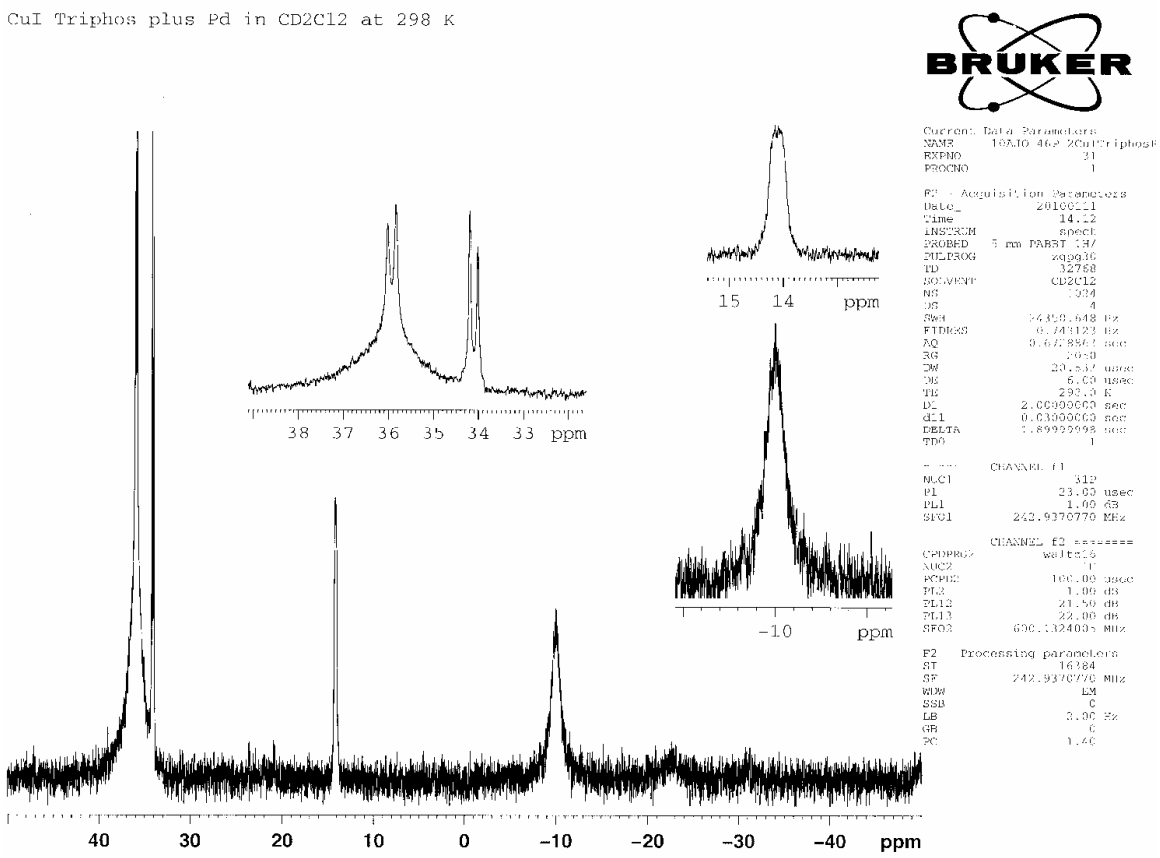
^{31}P NMR of **6** solubilised in $CDCl_3$ before Pd^{II} addition



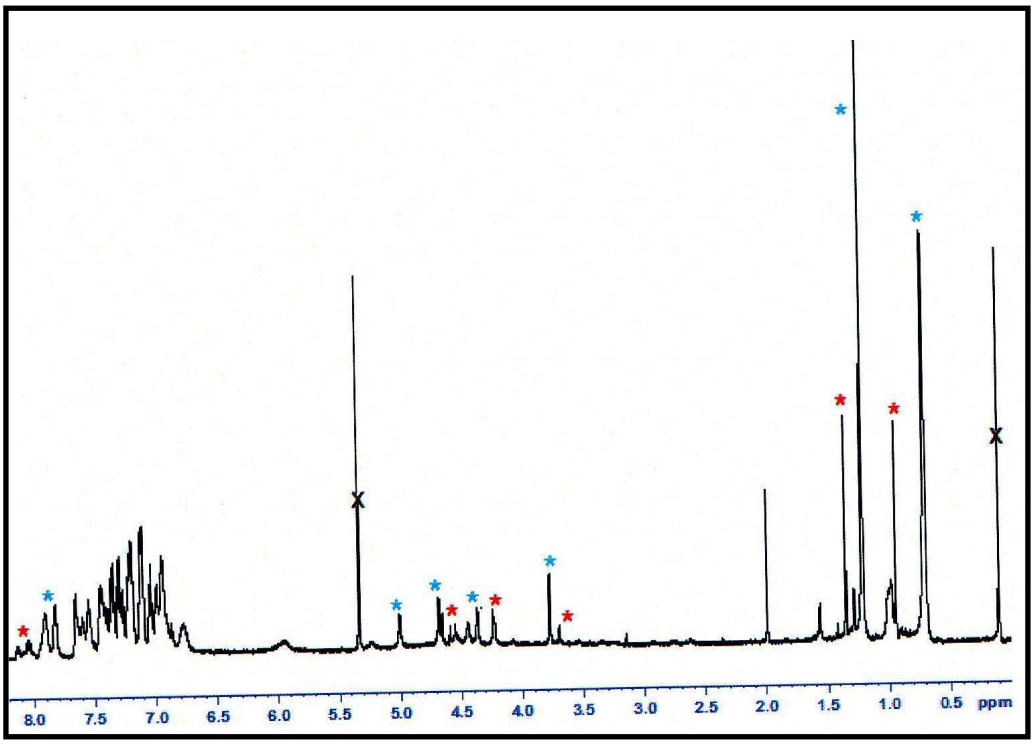
^{31}P NMR of a mixture of **6** with $[Pd(allyl)Cl)]_2$ ***in $CDCl_3$*** after 10 min at room temperature: *total transfer of the ligand from copper to palladium*



^{31}P NMR of a 1:1 mixture of **4** to with $[Pd(allyl)Cl)]_2$ in CD_2Cl_2 at 298 K (for comparison with Fig. 7, copper-bonded species are less discernible)

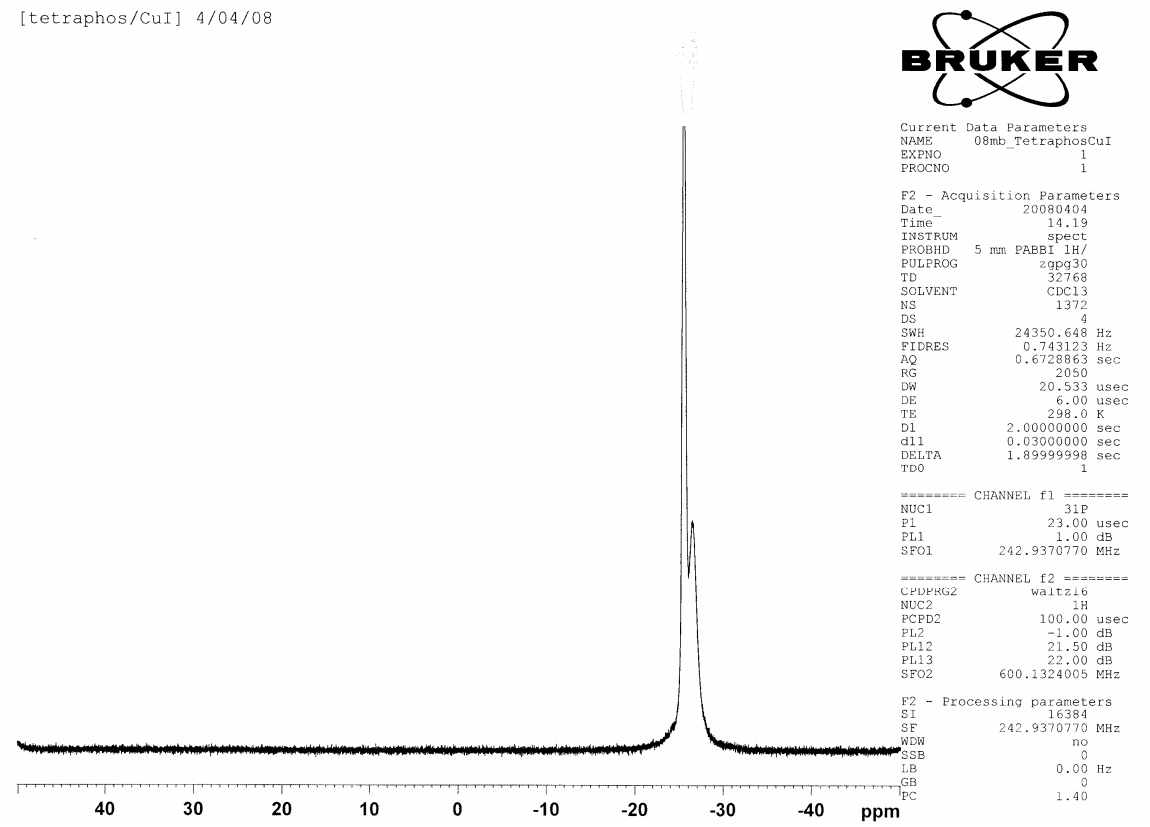


^{1}H NMR of a mixture of **4** with $[Pd(allyl)Cl)]_2$ in CD_2Cl_2 after 10 min:
*An equilibrium about 80:20 is obtained between copper-adduct **4** (red stars) and new Pd species formed (blue stars)*

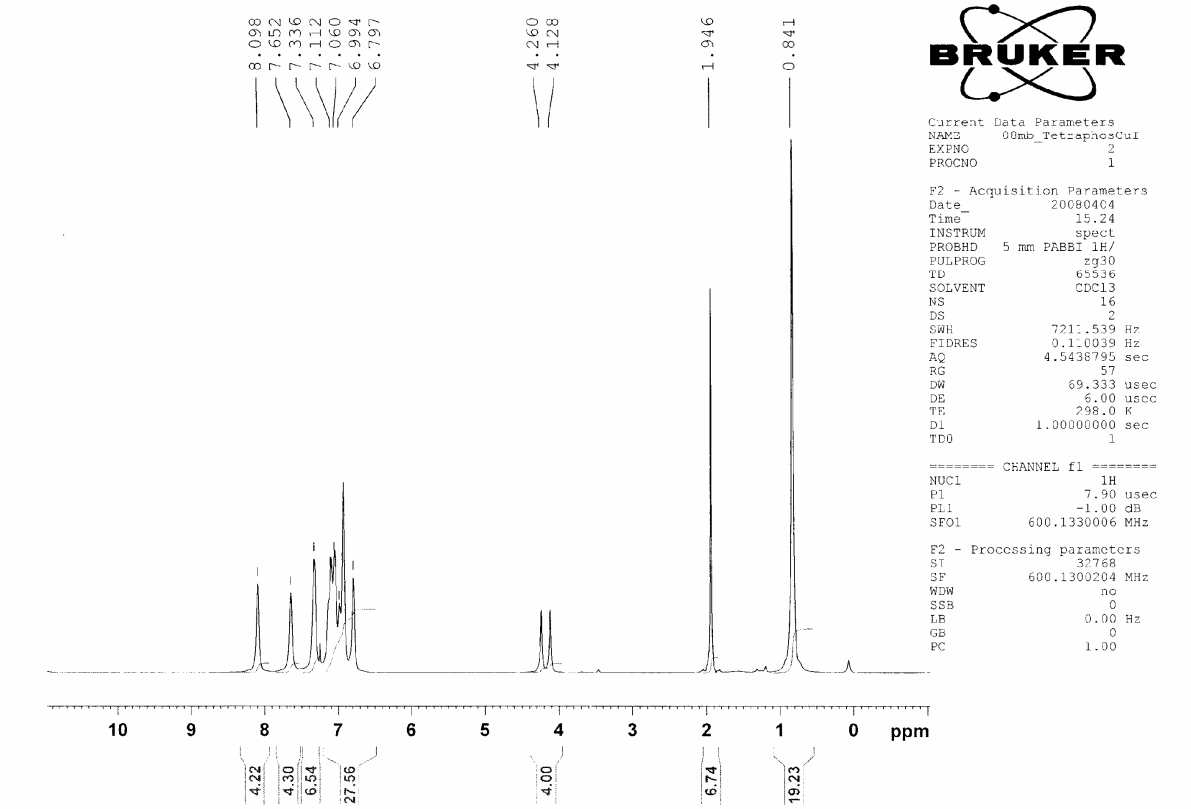


^{31}P NMR of **2** in $CDCl_3$ before palladium addition

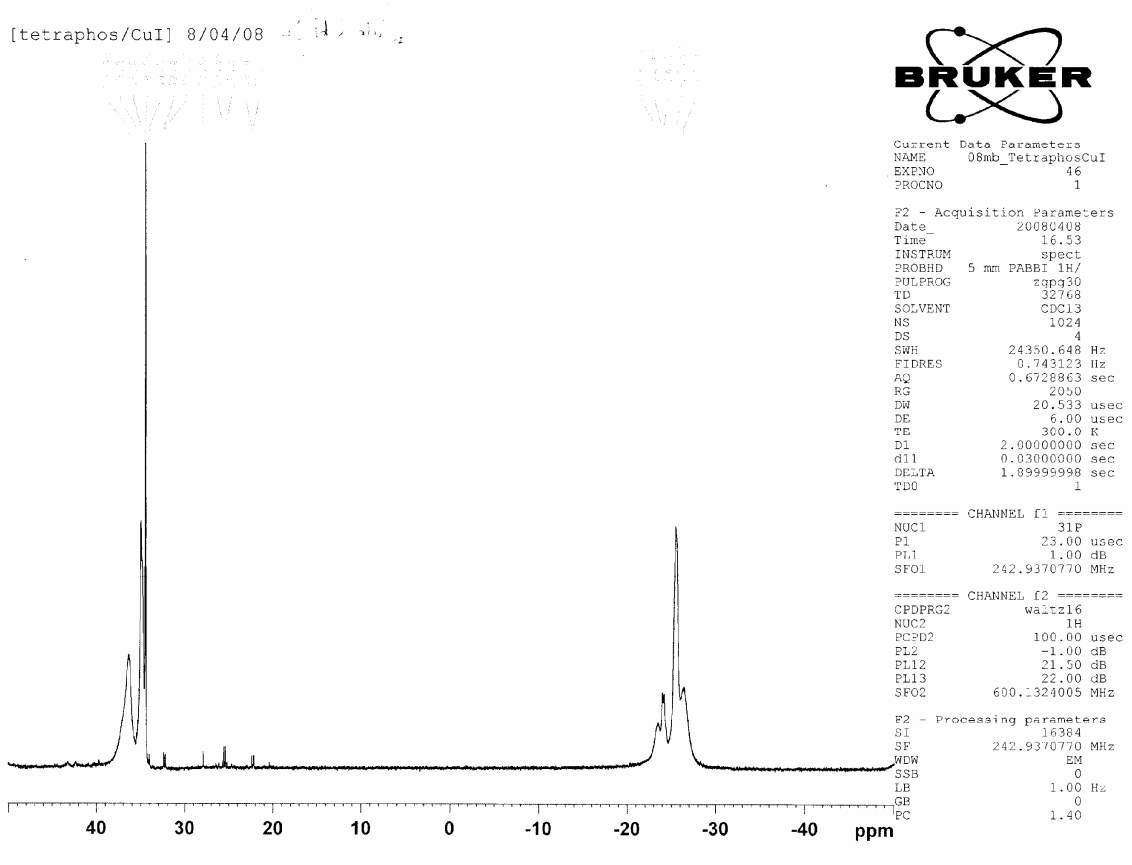
[tetraphos/CuI] 4/04/08



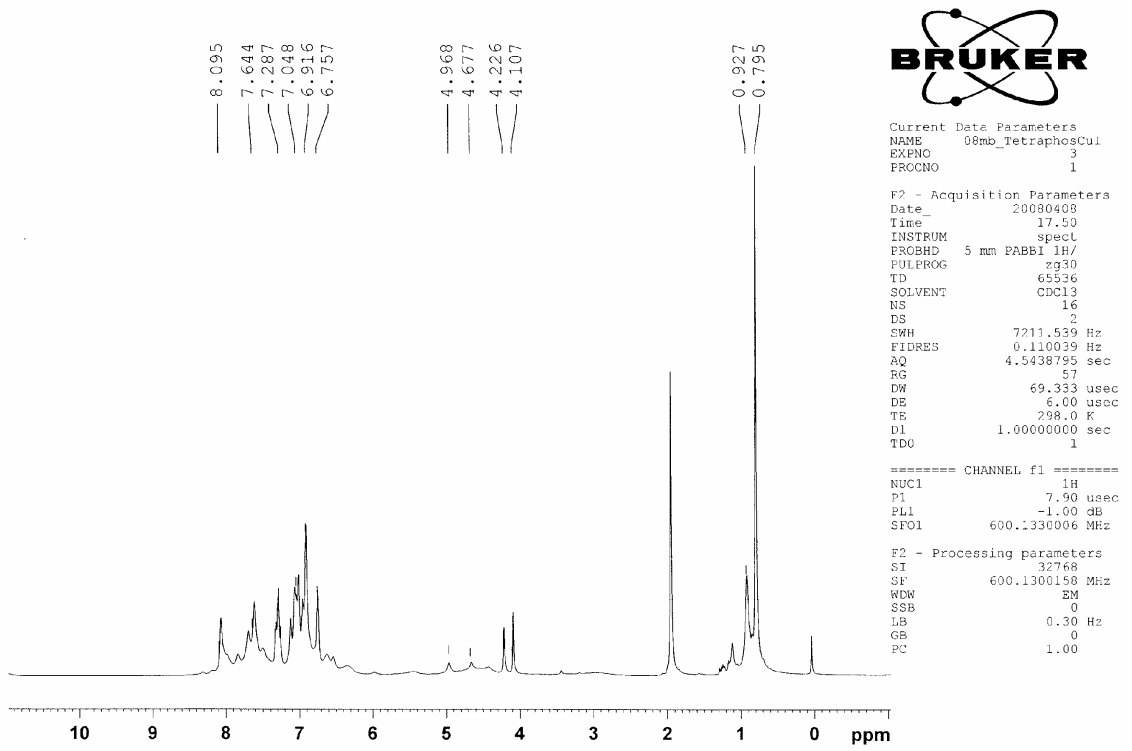
^{1}H NMR of **2** in $CDCl_3$ before palladium addition



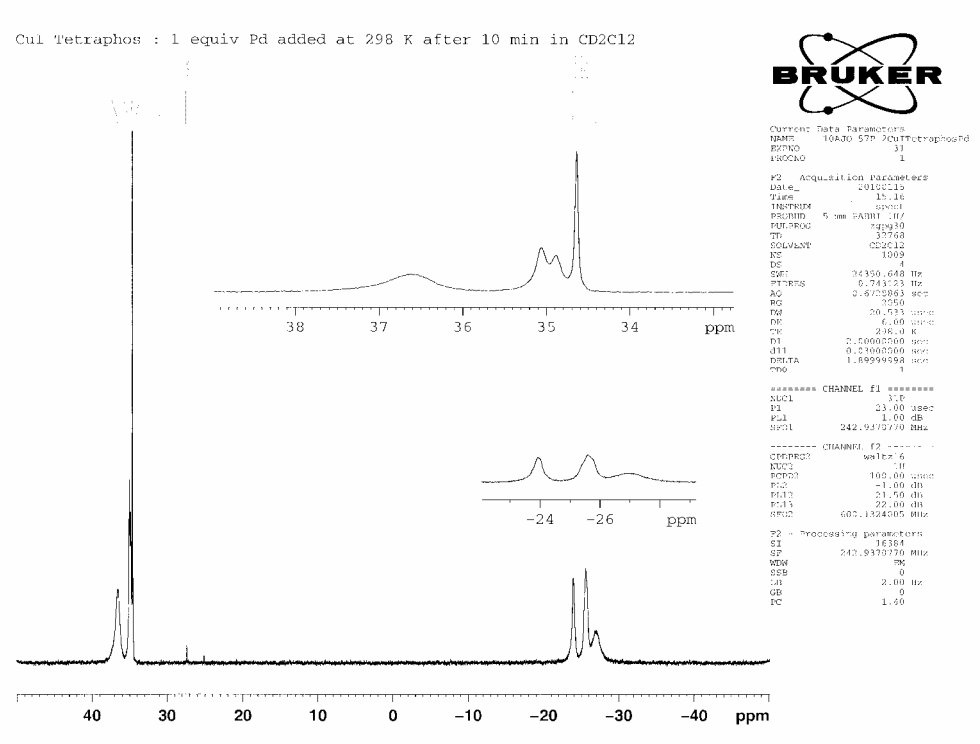
^{31}P NMR of a mixture of **2** with 1/2[Pd(allyl)Cl)]$_2$ in $CDCl_3$ after 1 hr



^{1}H NMR of a mixture of **2** with 1/2[Pd(allyl)Cl)]$_2$ in $CDCl_3$ after 1 hr



^{31}P NMR of a mixture of **2** with $[Pd(allyl)Cl]_2$ in CD_2Cl_2



Exact-Mass of ligand **3** - Molecular peak m/z = 850.27040, σ = 0.033

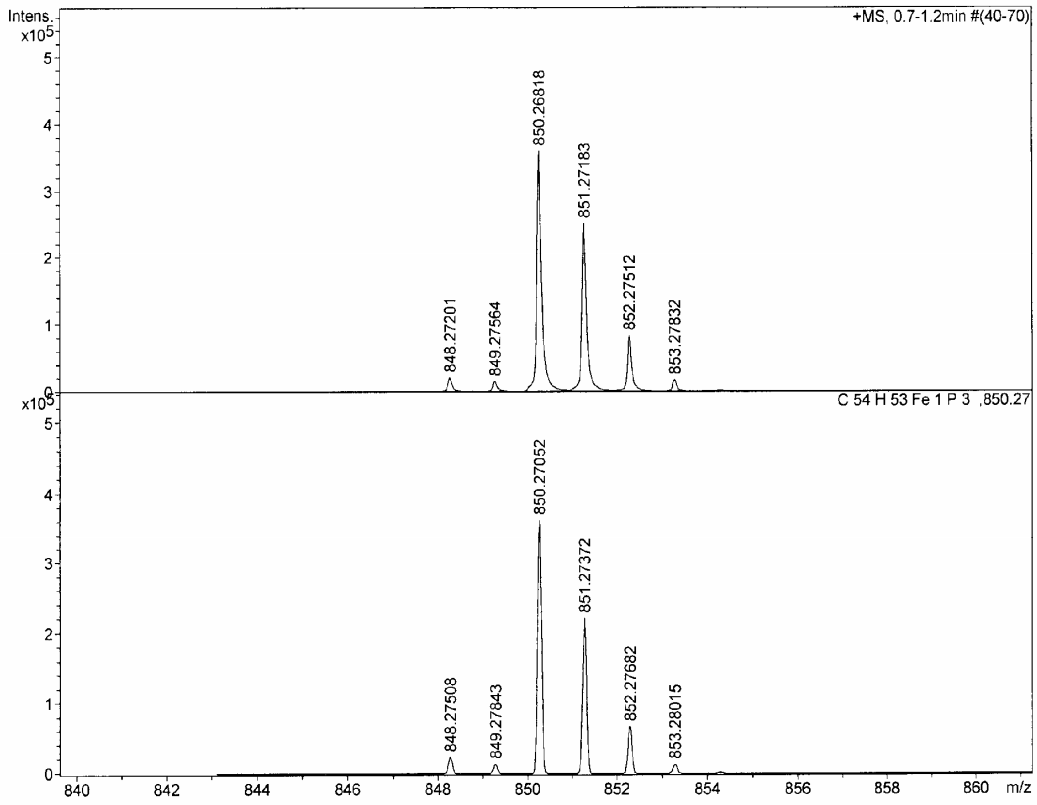
Display Report

Analysis Info

Analysis Name	07mb_096d.d	Acquisition Date	12/12/2007 2:29:48 PM
Method	tune_wide.m	Operator	Marie-José Penouilh
Sample Name		Instrument	micrOTOF-Q 56
Comment	dcm/meoh		

Acquisition Parameter

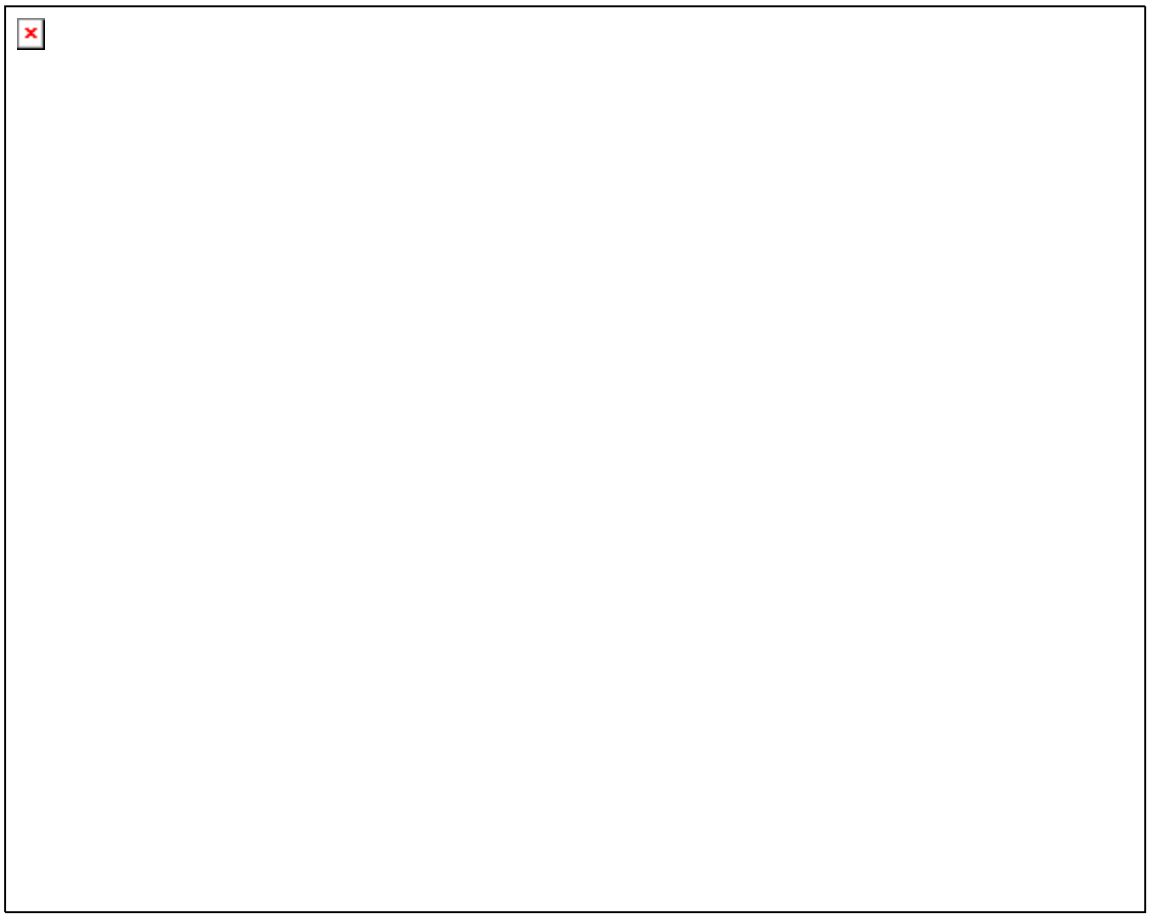
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Source



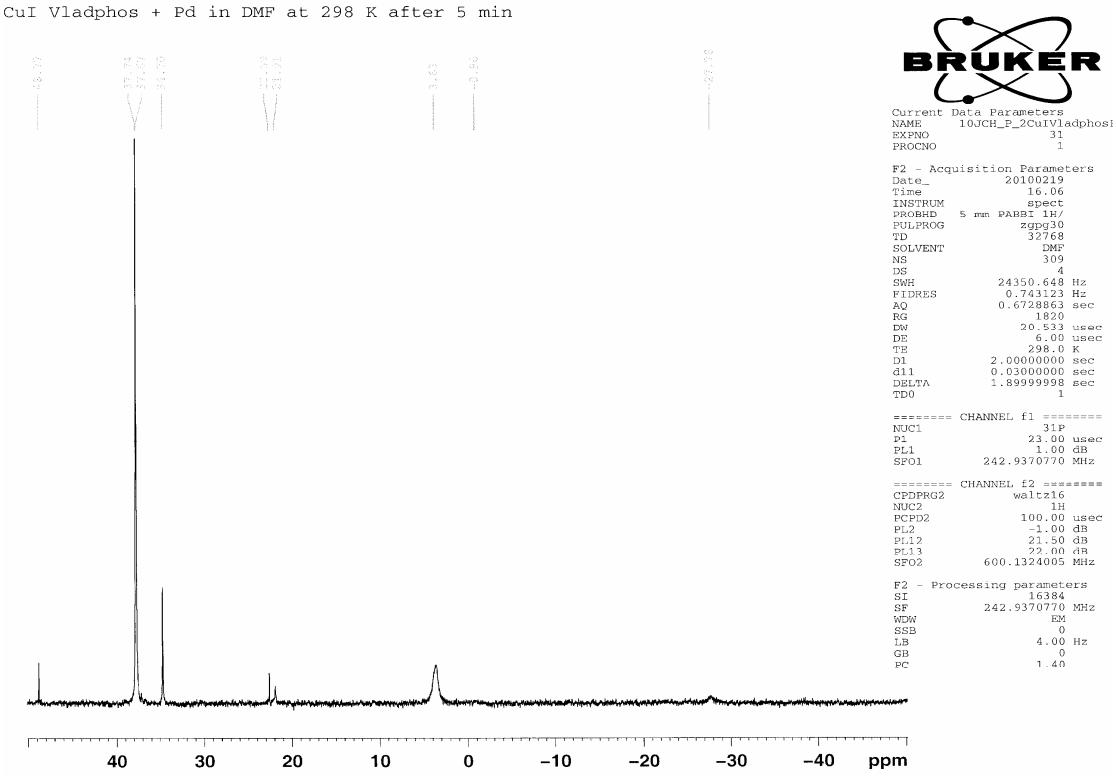
Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 54 H 53 Fe 1 P 3	0.043	850.27040	2.61	2.50	2.22	30.00	-	odd

Bruker Daltonics DataAnalysis 3.4 printed: 12/12/2007 2:32:04 PM Page 1 of 1

^{31}P NMR of **6** solubilised in DMF before Pd^{II} addition



^{31}P NMR of a mixture of **6** with $[Pd(allyl)Cl)]_2$ ***in DMF*** after 5 min at room temperature: *total transfer of the ligand from copper to palladium*



GC and GC-MS experimental conditions.

GC yields are based upon either external standard calibration from pure starting products (for instance, an injection containing 4-(phenylethynyl)acetophenone at a concentration of 2,000 μg/mL is made and results in a peak area of 100,000; the response factor rf using the equation [response factor = peak area/sample amount] is calculated rf = 100,000/2,000 = 50; An injection of the sample with unknown concentration of 4-(phenylethynyl)acetophenone has a peak area of 60,000, the calculation of the amount of product is given by [peak area/response factor] = 1,200 μg/mL of coupling product, assuming analyte response to be linear over this range of concentration) or by internal standard (analogous calibration methods with 0.05 mL of neat *n*-decane internal standard, retention time 6.5 min, to each sample solution after reaction mixture is quenched with water, 3 mL, and extracted with diethyl ether 8 mL). The retention time obtained for the reagents were obtained from a Supelco equity-5 capillary column (30 m) on a Shimadzu GC-2014 for GC, or from a hp-5 capillary column (30m) for GC-MS; the following program was used in GC-MS and GC for experiments reported in Tables: carrier gas He, gas flow 1.3 ml/min (GC-MS); Injector (300°C), oven temperature program: 1 min at 60°C, 20°C/min till 280°C, 5, 10 or 15 min at 280°C, detector FID (300°C) or MSD.

^{1}H NMR, elemental analysis or GC-MS coupling products characterization.

NMR spectra were recorded using TMS as internal standard (^{1}H 300.13 MHz). NMR spectroscopic data of the coupling products are identical to those reported in the literature, see for instance ref [1-12].

Table 3, Entry 1 : ***4-(Phenylethynyl)anisole.***
Isolated yield: 0.90 mmol, 0.187 g, 90%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.50-7.48 (m, 2H), 7.45 (d, *J* = 8.8 Hz, 2H), 7.32 (m, 3H), 6.86 (d, *J* = 8.8 Hz, 2H), 3.80 (s, 3H). Anal. Calcd for $C_{15}H_{12}O$: C, 86.51; H, 5.81. Found: C, 86.55; H, 5.67.

Table 3, Entry 2 : ***3-(Phenylethynyl)anisole.***
Isolated yield: 0.88 mmol, 0.180 g, 88%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.56-7.53 (m, 2H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.35-7.32 (m, 4H), 6.95-6.87 (m, 2H), 3.91 (s, 3H). Anal. Calcd for $C_{15}H_{12}O$: C, 86.51; H, 5.81. Found: C, 86.50; H, 5.62.

Table 3, Entry 3 : ***3-(Phenylethynyl)toluene.***
Isolated yield: 0.90 mmol, 0.173 g, 90%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.80-7.65 (m, 2H), 7.56-7.20 (m, 7H), 2.50 (s, 3H). MS (70eV); m/z (%): 192 ($M^{+\cdot}$,100), 191 (40), 190 (25), 189 (30), 176 (8), 165 (30), 152 (10), 139 (12), 115 (18), 95 (20), 82 (18), 74 (8), 63(10), 51(8).

Table 3, Entry 4 : ***2-(Phenylethynyl)toluene.***
Isolated yield: 0.75 mmol, 0.134 g, 75%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.63-7.57 (m, 3H), 7.45-7.38 (m, 3H), 7.30-7.20 (m, 3H), 2.60 (s, 3H). MS (70eV); m/z (%): 192 ($M^{+\cdot}$,100), 191 (96), 190 (18), 187 (42), 165 (33), 135 (13), 115 (18), 94 (15), 82 (14), 63(10), 51(8).

Table 3, Entry 5 : ***2-(Phenylethynyl)naphthalene.***
Isolated yield: 0.95 mmol, 0.170 g, 95%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.45 (d, *J* = 8.0 Hz, 1H), 7.85-7.70 (m, 3H), 7.69-7.60 (m, 2H), 7.60-7.45 (m, 2H), 7.45-7.29 (m, 4H). MS (70eV); m/z (%): 228 ($M^{+\cdot}$,100), 227 (8), 226 (30), 202 (4), 200 (4), 188 (2), 176 (2), 163 (2), 150 (2), 138 (1), 126 (2), 114 (16), 113 (15), 101 (10), 88 (5), 75 (2), 63 (2), 51 (2).

Table 3, Entry 6 : ***1,2-Bis(phenylethynyl)benzene.***
Isolated yield: 0.94 mmol, 0.260 g, 94%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.38-7.35 (m, 7H), 7.55-7.50 (m, 7H). MS (70eV); m/z (%): 278 ($M^{+\cdot}$,18), 277 (33), 276 (75), 274 (25), 250 (8), 237 (4), 224 (6), 137 (16), 138 (35), 125 (15), 112 (8), 111 (6), 87 (4), 63 (4), 50 (3).

Table 3, Entry 10: ***4-(Phenylethynyl)benzonitrile.***
Isolated yield: 0.54 mmol, 0.110 g, 54%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.62-7.58 (m, 4H), 7.55-7.52 (m, 2H), 7.39-7.34 (m, 3H). Anal. Calcd for $C_{15}H_9N$: C, 88.64; H, 4.46. Found: C, 88.92; H, 4.38.

Table 3, Entry 11: ***4-(Phenylethynyl)acetophenone.***
GC yield 36%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.92 (d, *J* = 8.1 Hz, 2H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.53 (m, 2H), 7.35 (m, 3H), 2.58 (s, 3H).

Table 3, Entry 14: ***4-(Phenylethynyl)toluene.***
Isolated yield: 0.90 mmol, 0.173 g, 90%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.50 (d, *J* = 7.7 Hz, 3H), 7.41 (d, *J* = 7.9 Hz, 2H), 7.30-7.25 (m, 3H), 7.13 (d, *J* = 7.7 Hz, 2H), 2.43 (s, 3H). Anal. Calcd for $C_{15}H_{12}$: C, 93.75; H, 6.25. Found: C, 93.88; H, 6.22.

References

[1] Boehm, V. P. W.; Herrmann, W. A. *Eur. J. Org. Chem.* **2000**, 3679.
[2] Mori, A.; Ahmed, M. S. M.; Sekiguchi, A.; Masui, K.; Koike, T. *Chem. Lett.* **2002**, 756.
[3] De la Rosa, M. A.; Velarde, E.; Guzman, A. *Synth. Commun.* **1990**, *20*, 2059.
[4] Koellhofer, A.; Plenio, H. *Chem. Eur. J.* **2003**, *9*, 1416 and references therein.
[5] Shirakawa, E.; Yoshida, H.; Takaya, H. *Tetrahedron Lett.* **1997**, *38*, 3759 and references therein.
[6] Alonso, D.; Najera, C.; Pacheco, C. *Tetrahedron Lett.* **2002**, *43*, 9365 and references therein.
[7] Koellhofer, A.; Pullmann, T.; Plenio, H. *Angew. Chem., Int. Ed.* **2003**, *42*, 1056 and references therein.
[8] Pal, M.; Parasuraman, K.; Gupta, S.; Yeleswarapu, K.; Koteswar, R. *Synlett* **2002**, 1976.
[9] Hierso, J. C.; Fihri, A. ; Meunier, P.; Doucet, H.; Santelli, M.; Ivanov, V. V. *Org. Lett.* **2004**, *6*, 3473.
[10] John, J. A.; Tour, J. M. *Tetrahedron*, **1997**, 155515.
[11] Kamikawa, T.; Hayashi, T. *J. Org. Chem.* **1998**, *63*, 8922.
[12] Mino, T.; Shirae, Y.; Saityo, T.; Sakamoto, M.; Fujita, T. *J. Org. Chem.* **2006**, *71*, 9499.