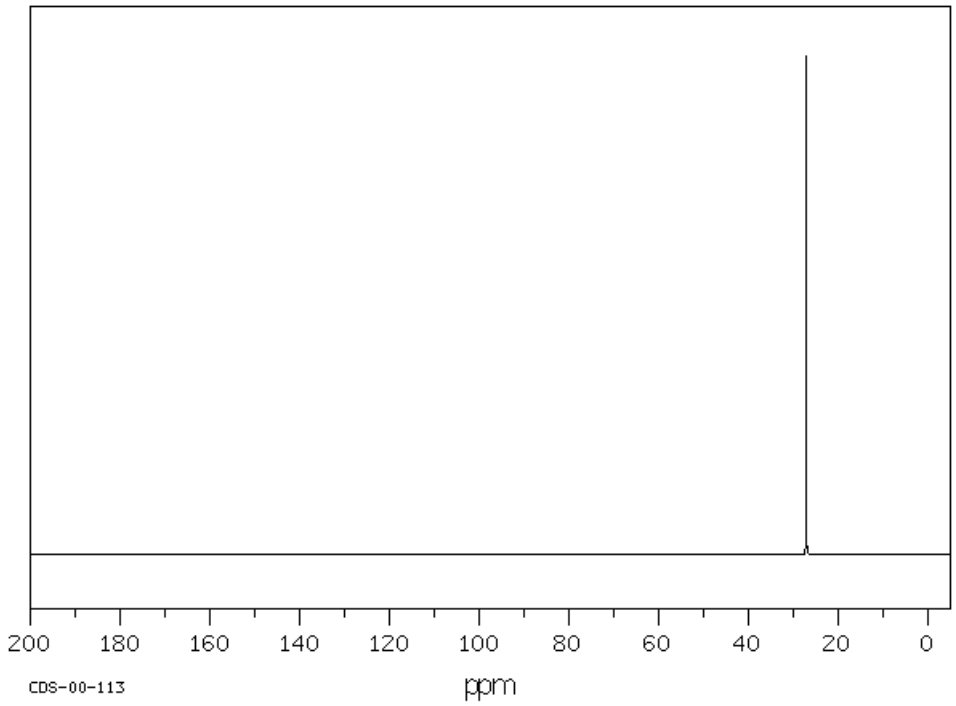
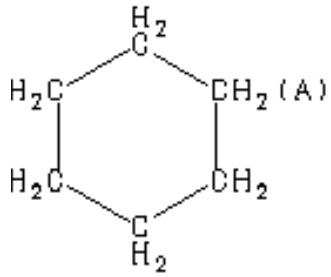
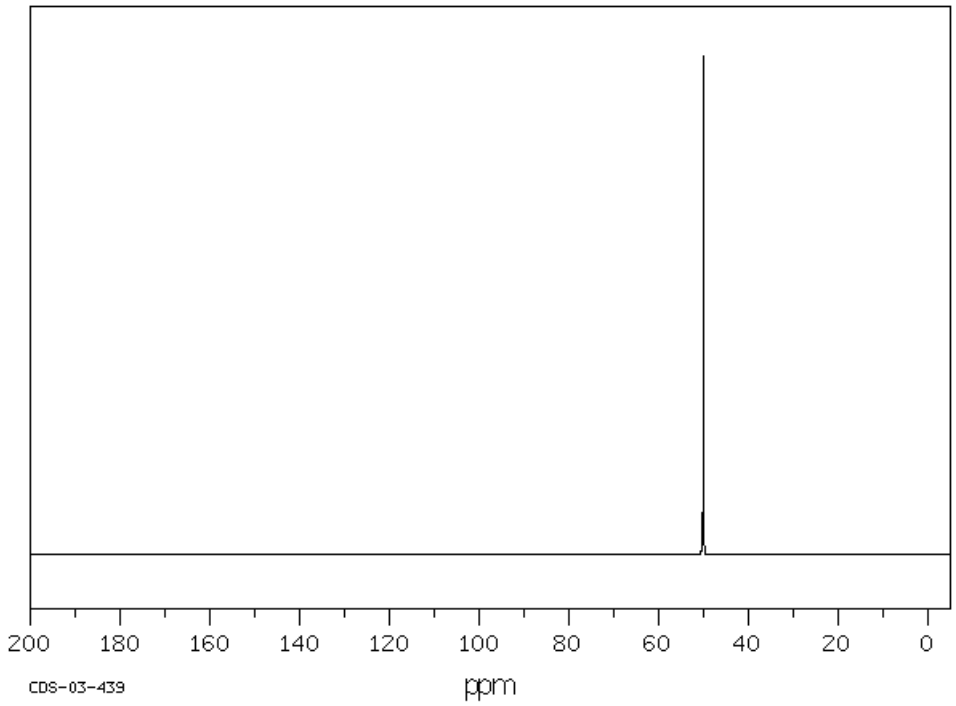
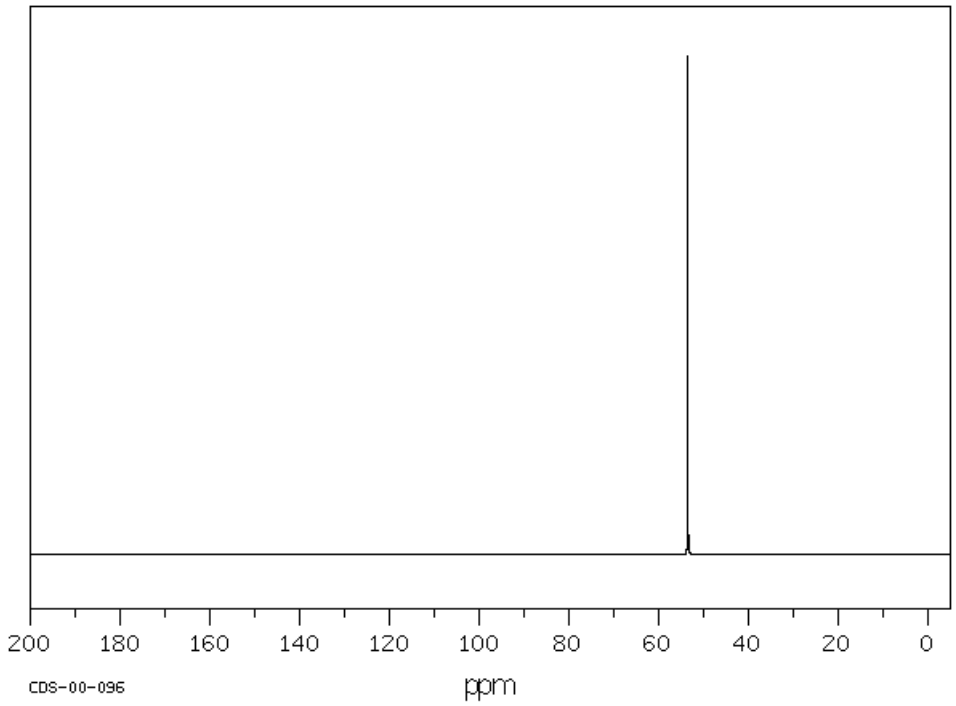
^{13}C NMR spectroscopy

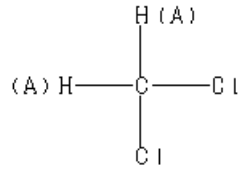


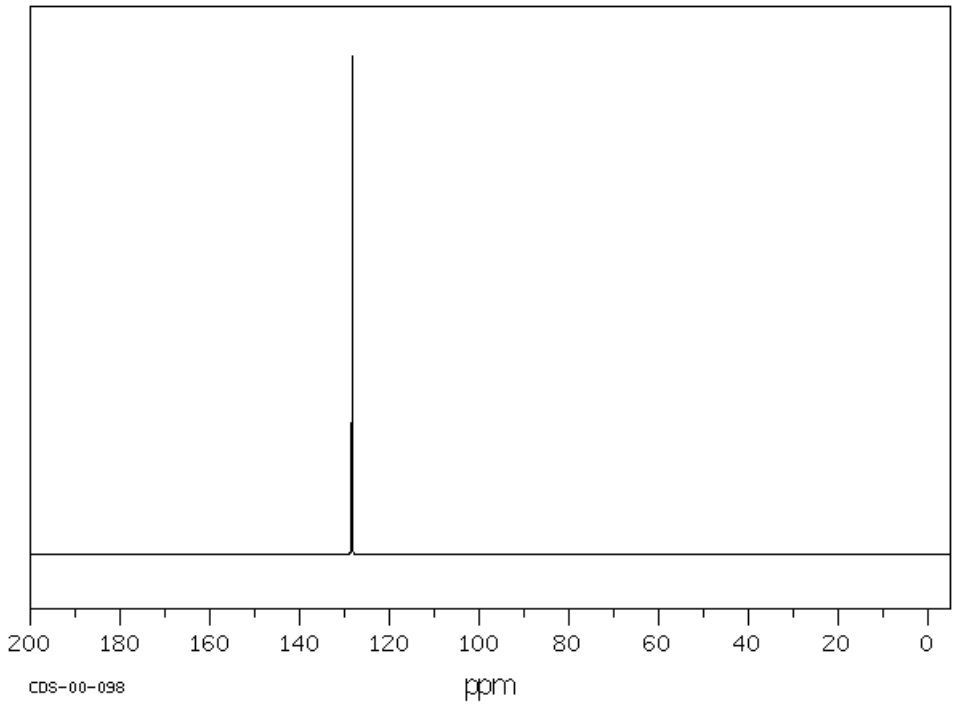


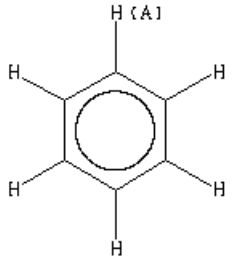


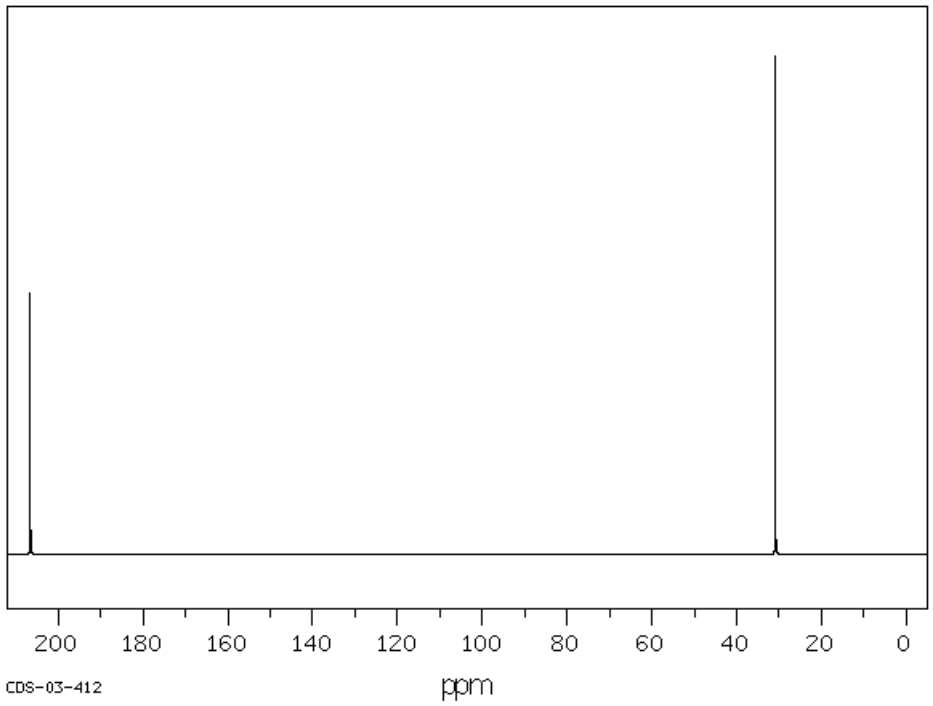
CH_3OH


200
180
160
140
120
100
80
60
40
20
0
CDS-00-096
ppm

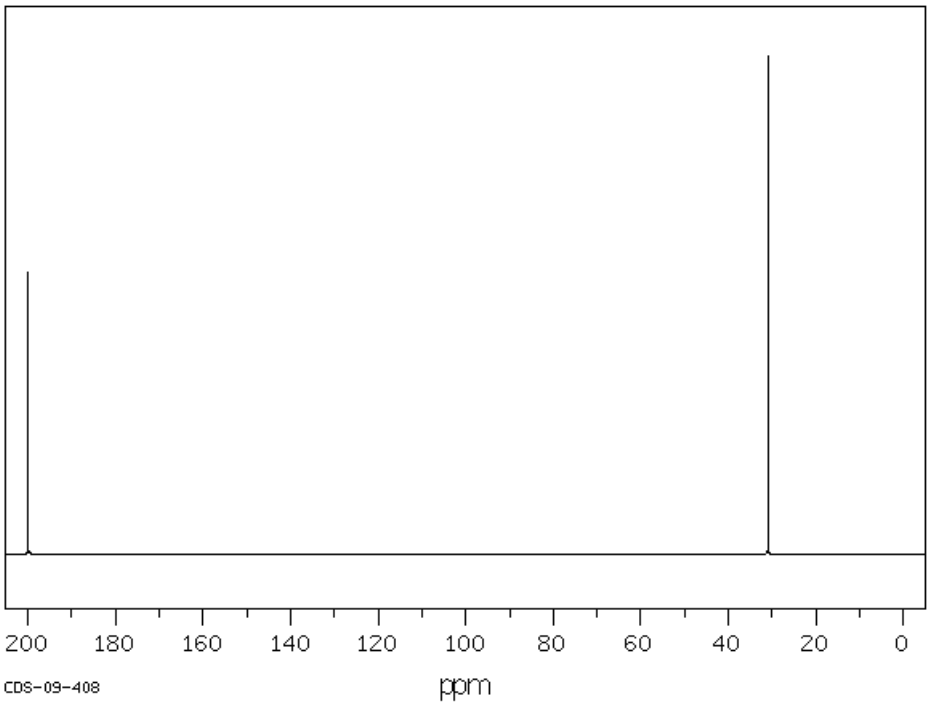

H (A)
(A) H
C
Cl
Cl


200
180
160
140
120
100
80
60
40
20
0
ppm
CDS-00-098

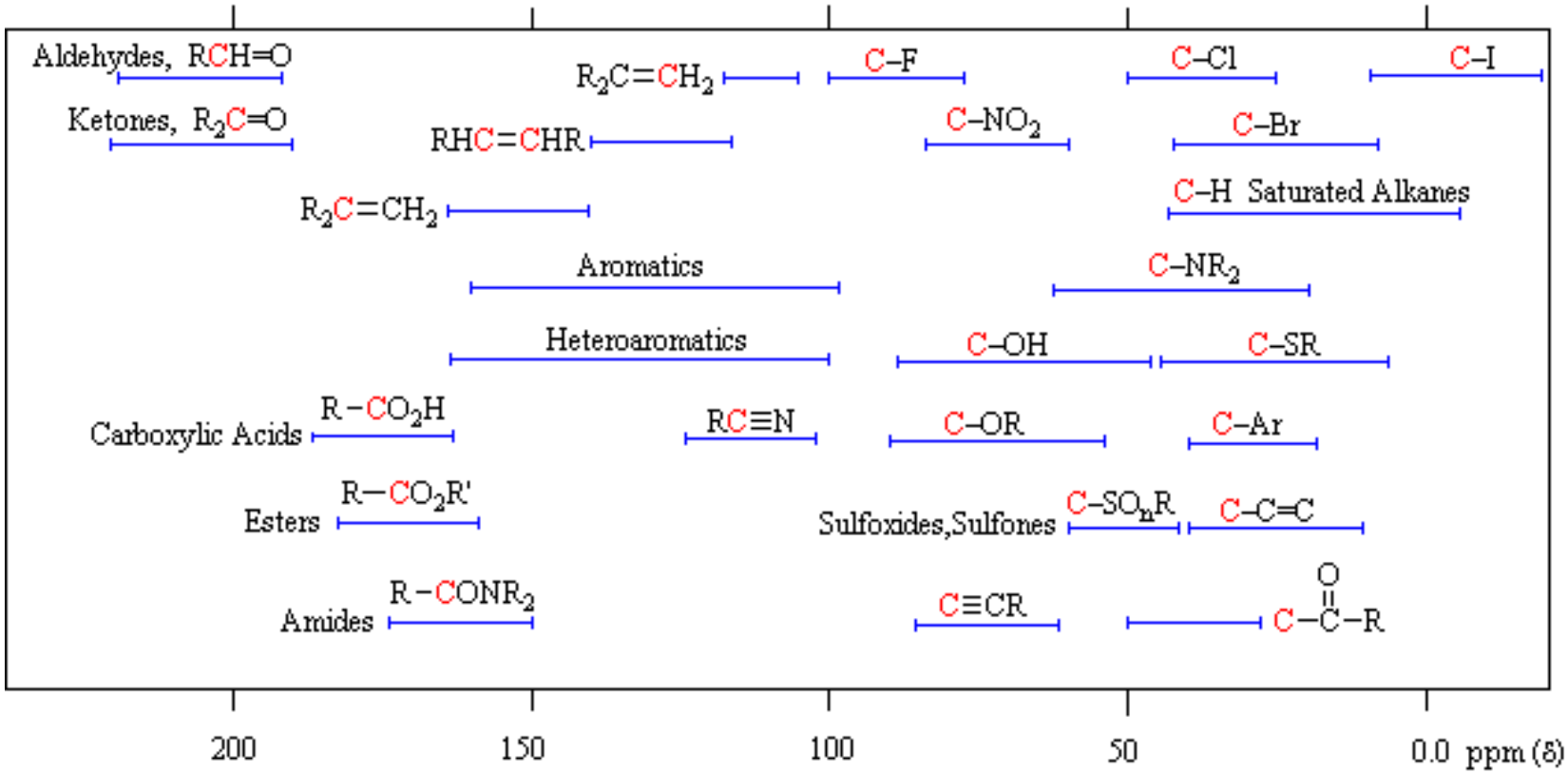

H (A)
H
H
H
H
H



$(CH_3)_2C=O$



CH_3-CHO


Aldehydes, RCH=O
Ketones, R2C=O
R2C=CH2
RHC=CHR
R2C=CH2
Aromatics
Heteroaromatics
R−CO2H
Carboxylic Acids
R−CO2R'
Esters
R−CONR2
Amides
RC≡N
C≡CR
C–F
C–NO2
C–OH
C–OR
C–SOnR
Sulfoxides,Sulfones
C–Cl
C–Br
C–NR2
C–SR
C–Ar
C–C=C
C–I
C–H Saturated Alkanes
200
150
100
50
0.0 ppm (δ)

1.2.1
Natural Abundance ^{13}C Spectrum of Compound 1

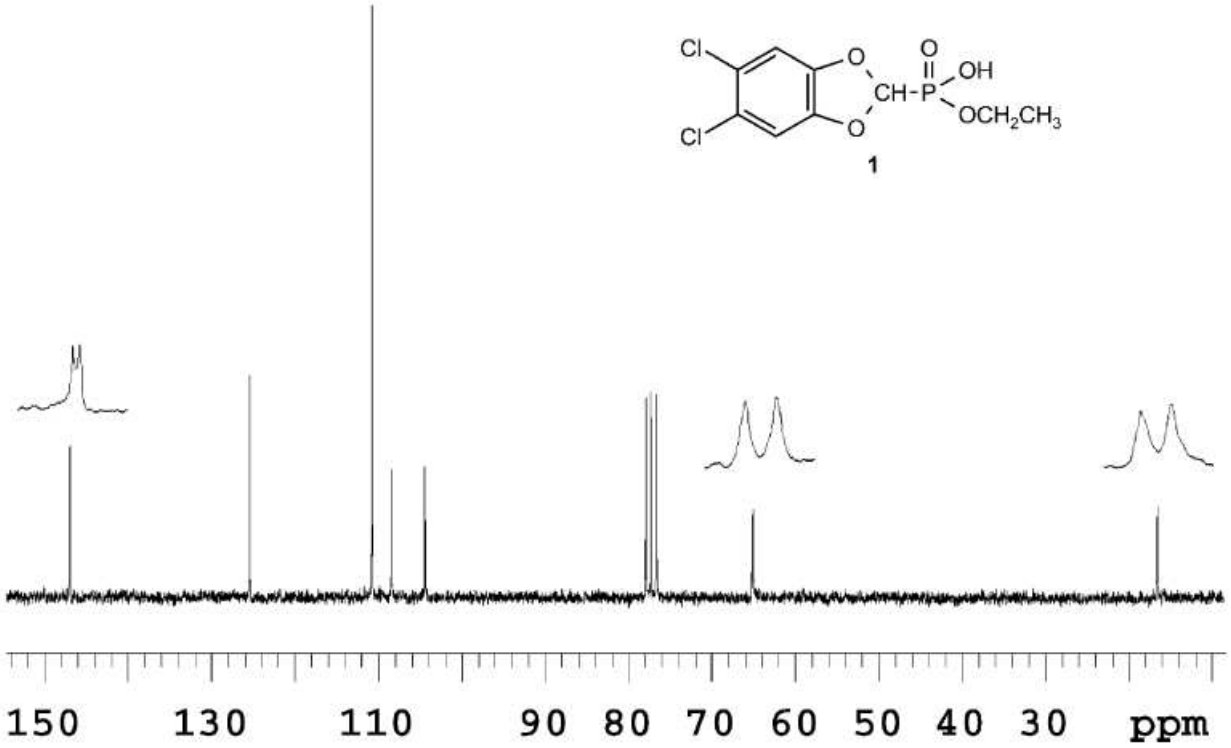


Fig. 14 Natural abundance carbon-13 spectrum of 1 (50 MHz) with expansion where necessary to show doublet structure. The assignments are as follows (*from left to right*): aromatic C bonded to oxygen (doublet) ; aromatic C bonded to chlorine (singlet); aromatic CH (singlet); methine (doublet); $CDCl_3$; OCH_2 (doublet); CH_3 (doublet). Multiplet splittings are due to coupling with phosphorus and are (except for $^1J_{PC}$) small

^1H decoupling
C-H NOE
T_1 relaxation

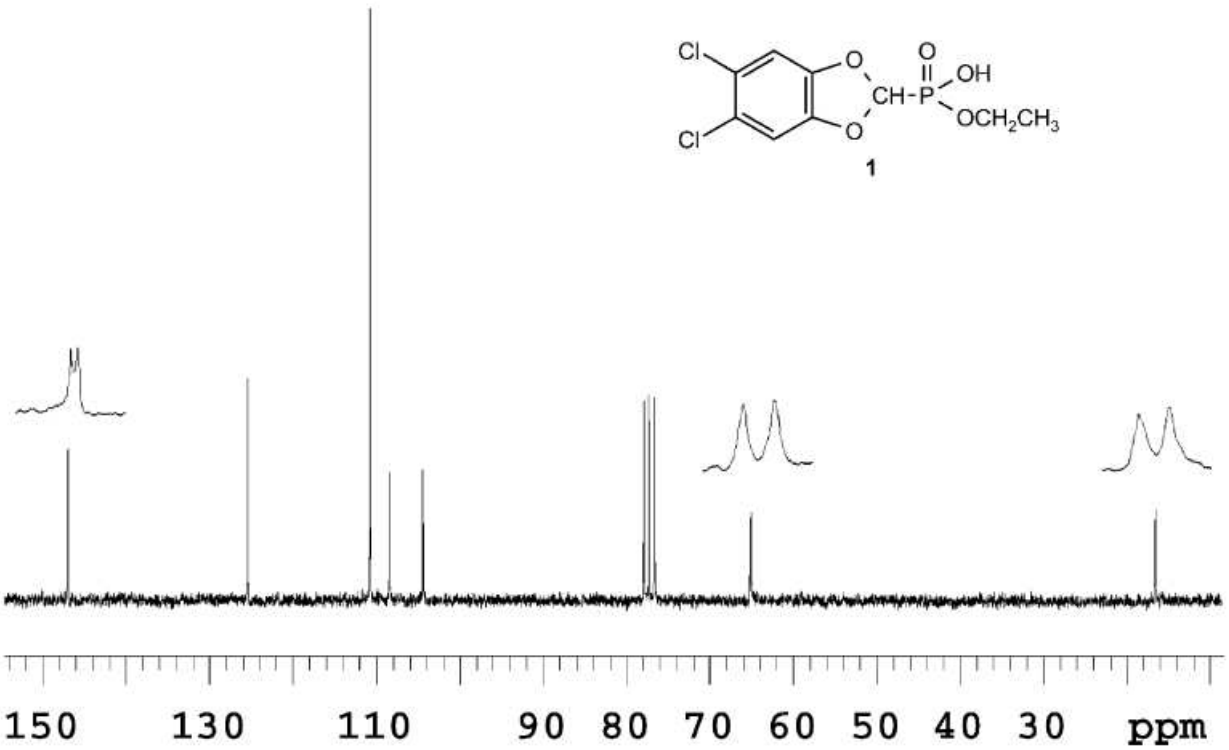


Table 4 Result of a prediction compared with the actual values

Chemical shift (ppm)	J_{CP} (Hz)	Calculated shift	J_{CP} (calc.)	Assignment
147.1 (d)	2.3	152.0	8.0	C_{arom}–O
125.5 (s)	0	128.4	0	C_{arom}–Cl
110.9 (s)	0	115.5	4.8	C_{arom}–H
106.5 (d)	201.3	102.6	207.2	CH–P
65.2 (d)	7.2	61.9	6.0	OCH_2
16.7 (d)	5.5	15.5	8.0	CH_3

1.2.2
Coupled Spectrum (Gated Decoupling)

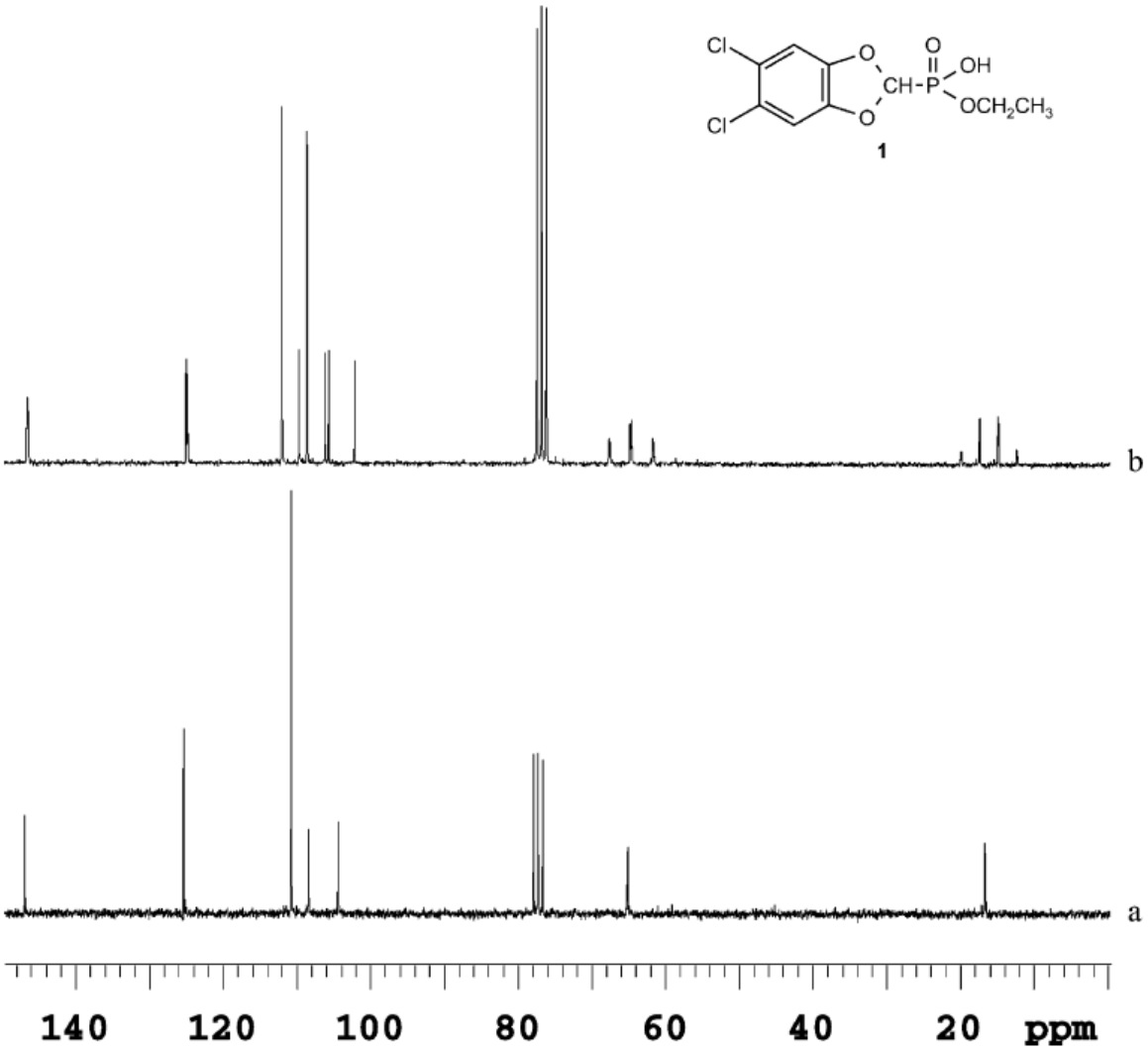
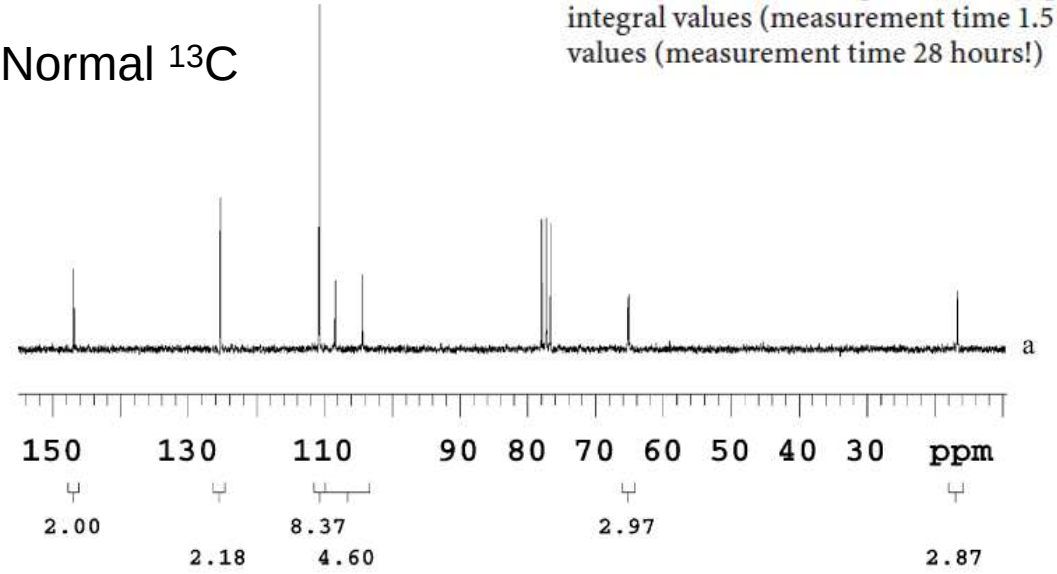
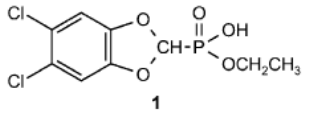


Fig. 15a,b Carbon-13 spectra of compound 1. a Protons broad-band decoupled; b carbon–proton coupling present (gated decoupling)

Normal ^{13}C

Fig. 16a,b Carbon-13 spectra of compound 1 recorded at 50 MHz. a Standard spectrum with integral values (measurement time 1.5 hours); b inverse gated decoupled spectrum with integral values (measurement time 28 hours!)





Inverse gated decoupling
Quantitative spectrum

1.2.4
Decoupled Spectrum: Proton Decoupling

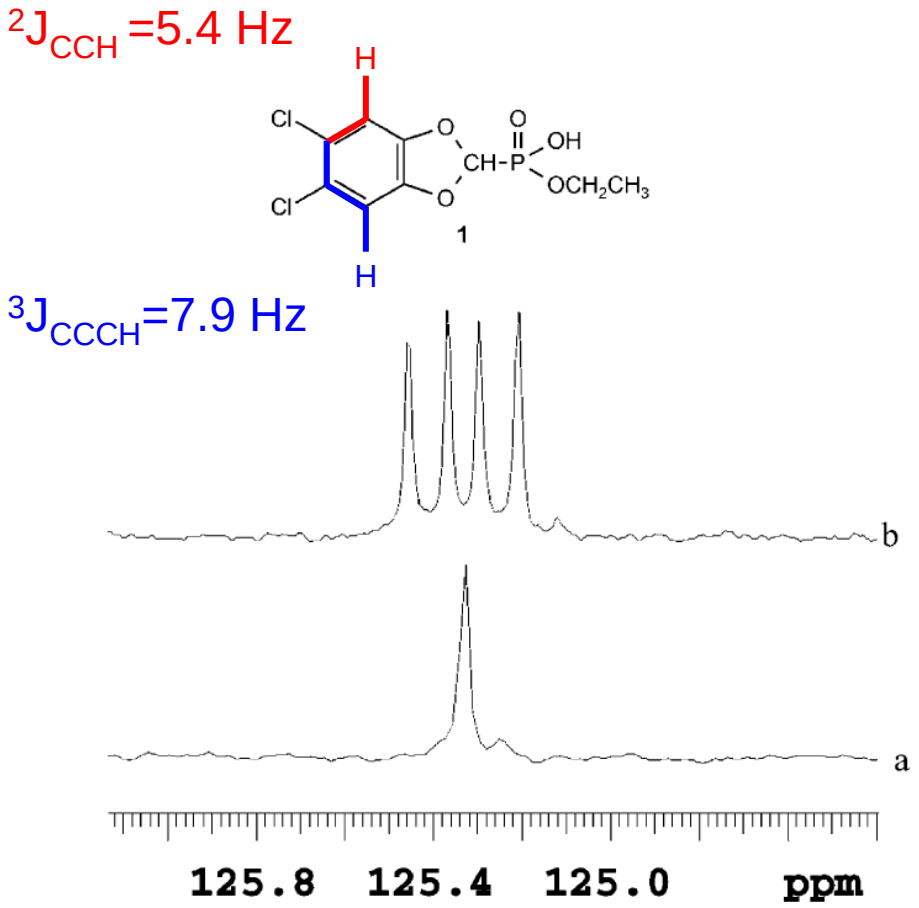
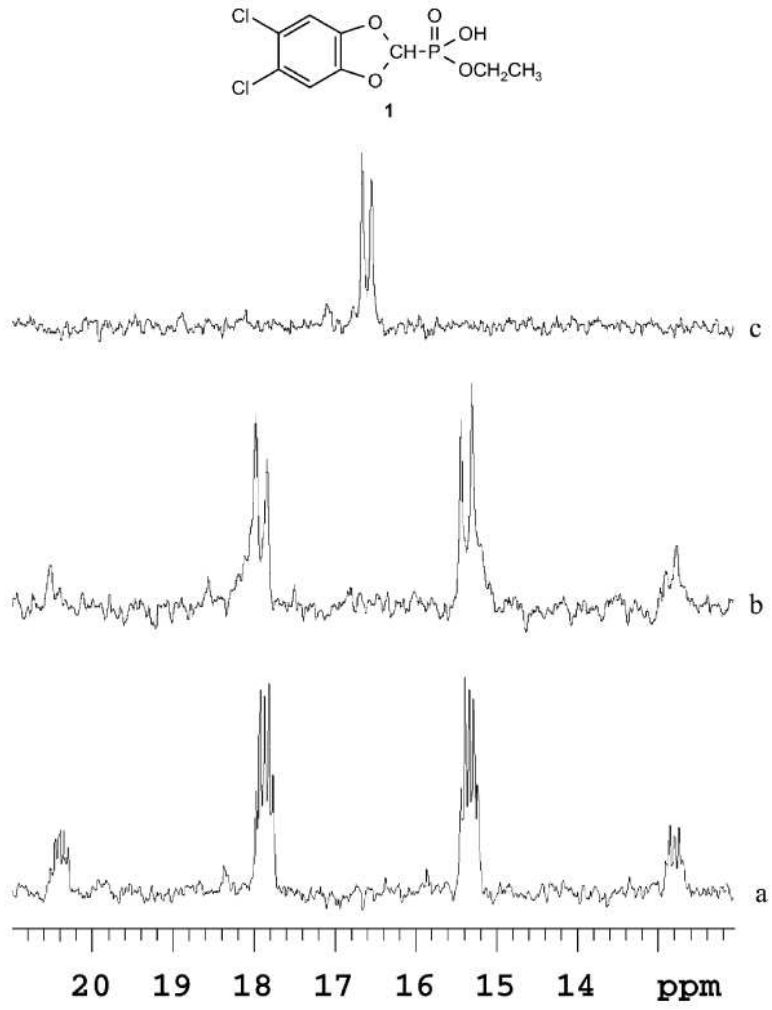


Fig. 17a,b Carbon-13 signals for the chlorine-bearing aromatic carbons in **1**. **a** Proton decoupled; **b** no proton decoupling

1.2.4
Decoupled Spectrum: Proton and Phosphorus Decoupling



$^{3}J_{COOP}$

$^{1}J_{CH} + {}^{3}J_{COOP}$

$^{1}J_{CH} + {}^{2}J_{CCH} + {}^{3}J_{COOP}$

Fig. 18a–c Carbon-13 signals for the methyl carbon in 1. a Complete carbon–proton coupling present; b selective decoupling of methylene protons; c broad-band decoupled

1.2.5
APT, DEPT, INEPT

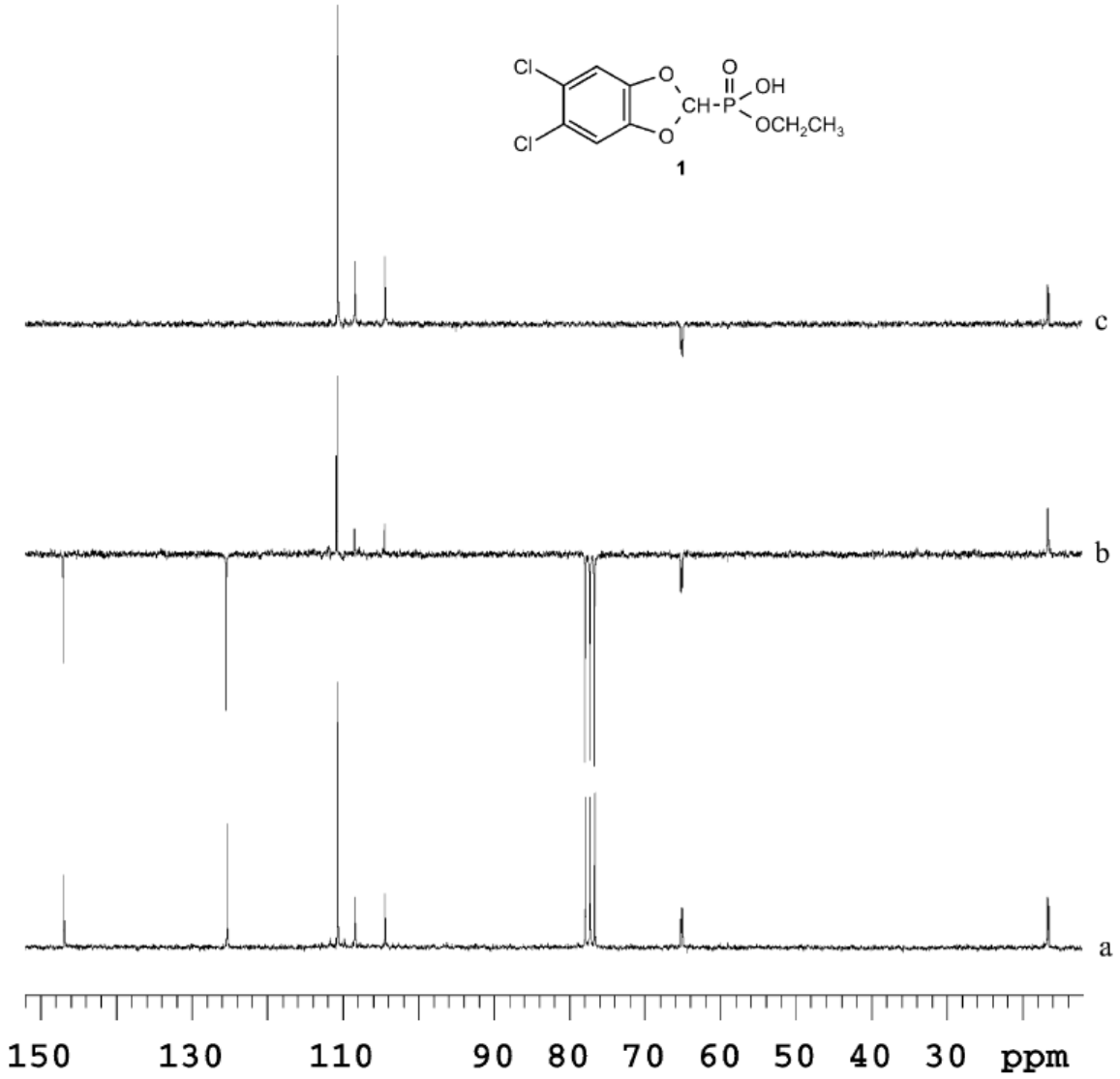
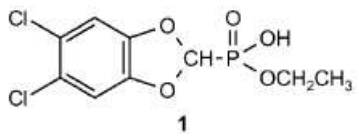


Fig. 19a–c Carbon-13 spectra of compound 1. a Standard spectrum (broad band decoupling); b APT spectrum; c DEPT-135 spectrum

1.3
^{31}P



1

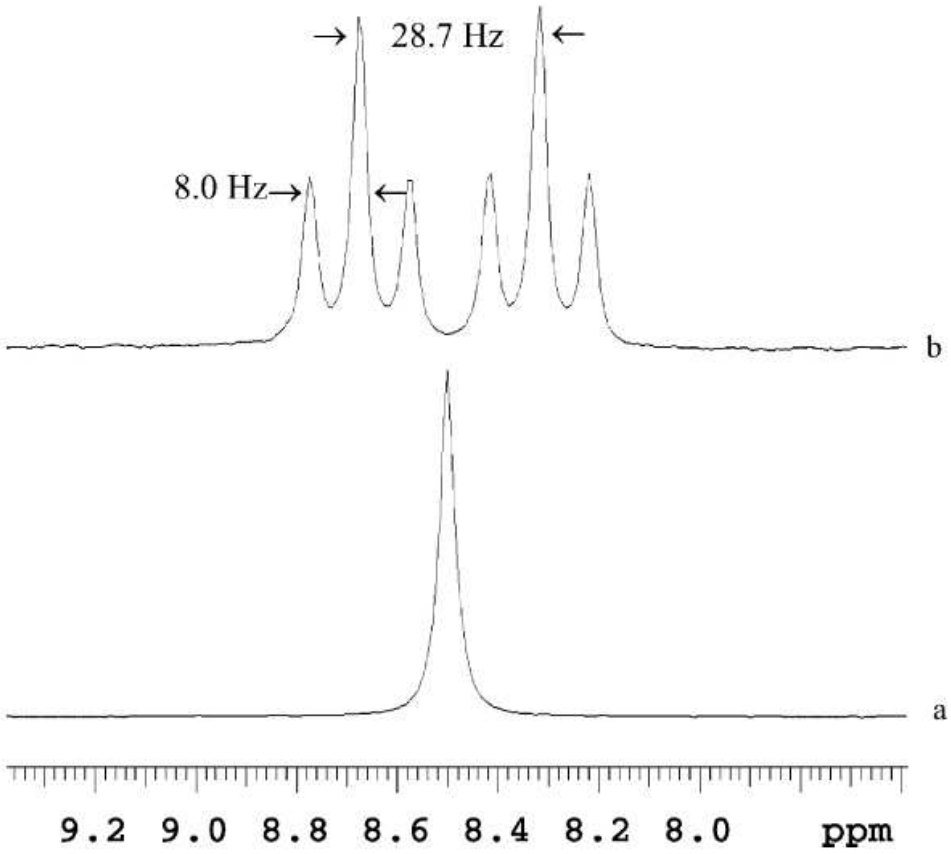


Fig. 22a,b Phosphorus-31 spectra of compound 1. a Protons decoupled; b proton–phosphorus coupling present

1.3.3
Coupled Spectrum (P–P Coupling)

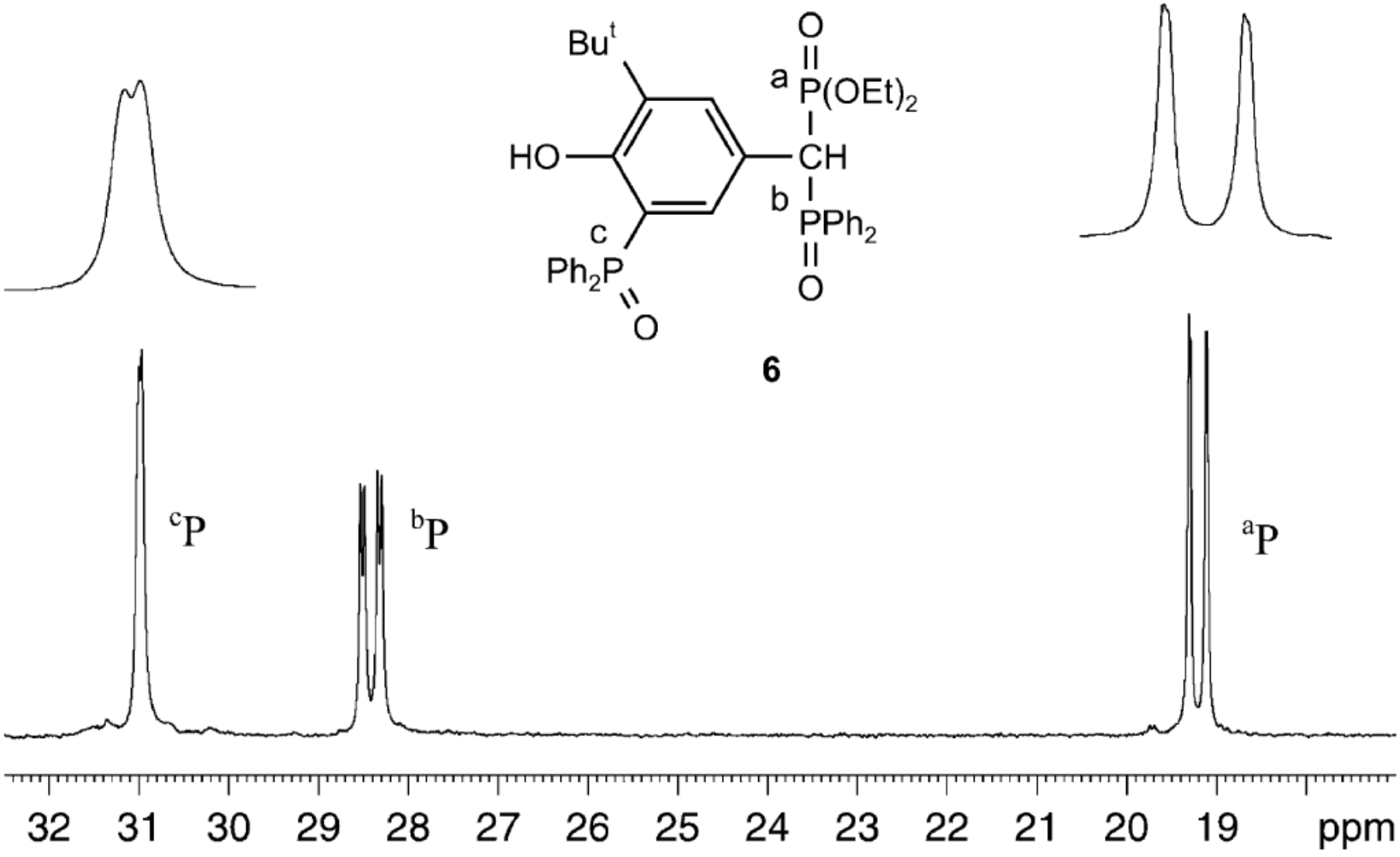


Fig. 23 Phosphorus-31 spectrum (202 MHz) of compound **6**, measurement time 2 min