

New Journal of Chemistry

May 12th, 2016

Supplementary material for

Tricarbonylrhenium(I) complexes with 2-acetylpyridine-derived hydrazones are cytotoxic to NCI-H460 human large cell lung cancer

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Infrared spectra of complexes $[\text{ReCl}(\text{CO})_3(\text{H}_2\text{AcPh})]$ (**1**),

$[\text{ReCl}(\text{CO})_3(\text{H}_2\text{Ac}p\text{ClPh})] \cdot 0.5\text{C}_7\text{H}_8$ (**2**) and

$[\text{ReCl}(\text{CO})_3(\text{H}_2\text{Ac}p\text{NO}_2\text{Ph})] \cdot 0.5\text{C}_7\text{H}_8$ (**3**)

Fig. S1 IR spectrum of complex [ReCl(CO)₃(H₂AcPh)] (1)

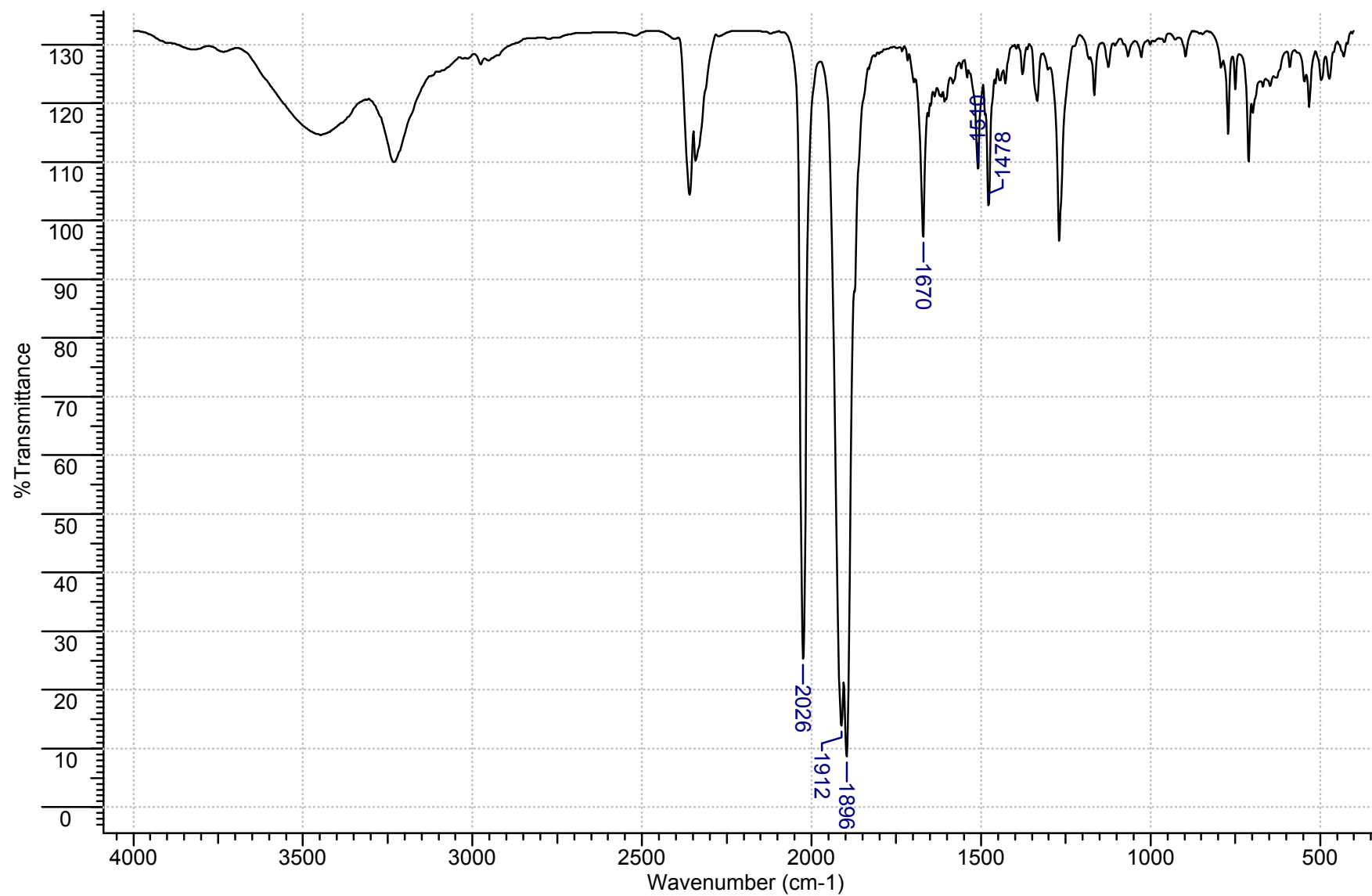


Fig. S2 IR spectrum of complex $[\text{ReCl}(\text{CO}_3)(\text{H}_2\text{AcpClPh})] \cdot 0.5\text{C}_7\text{H}_8$ (2)

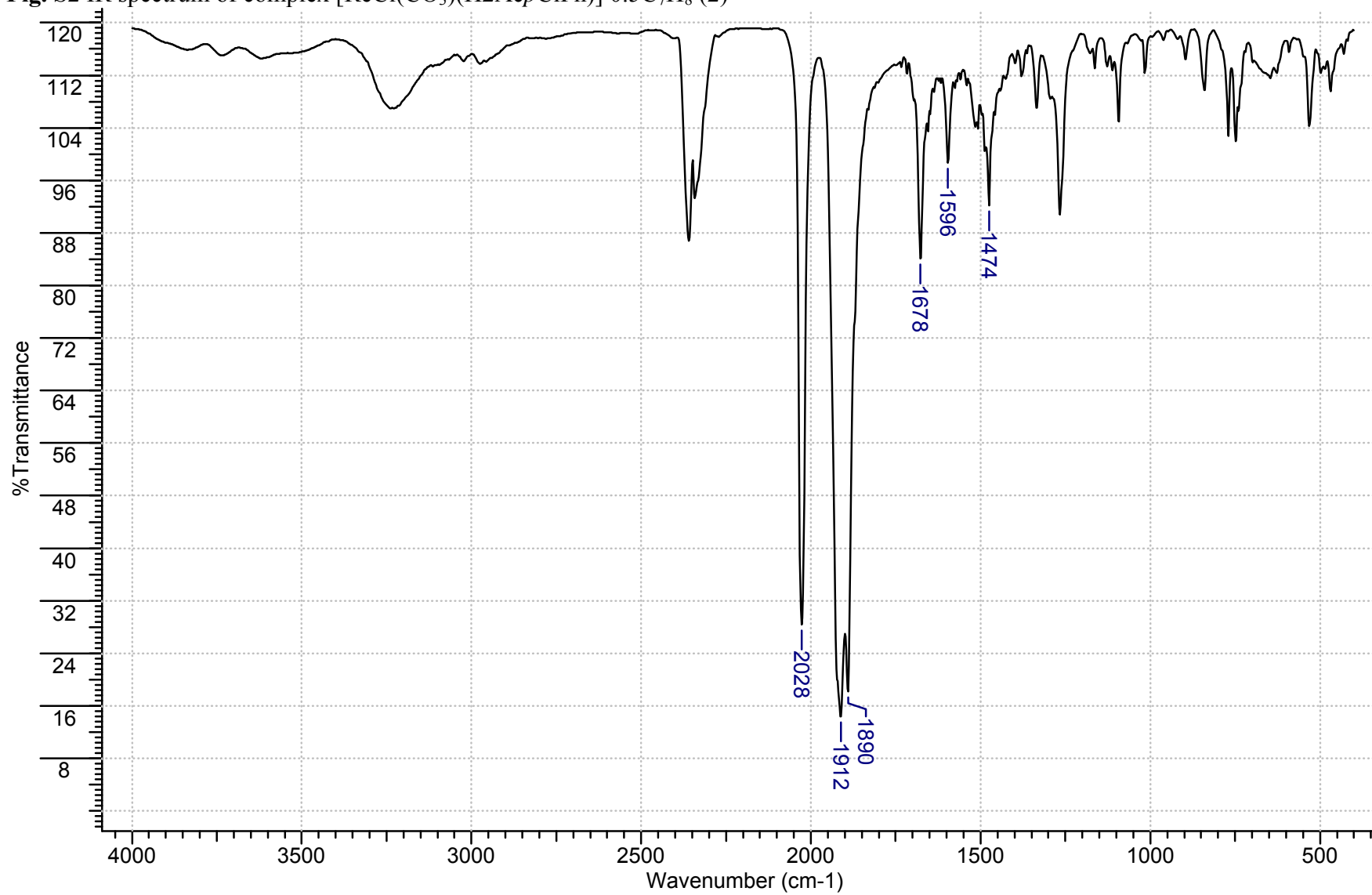
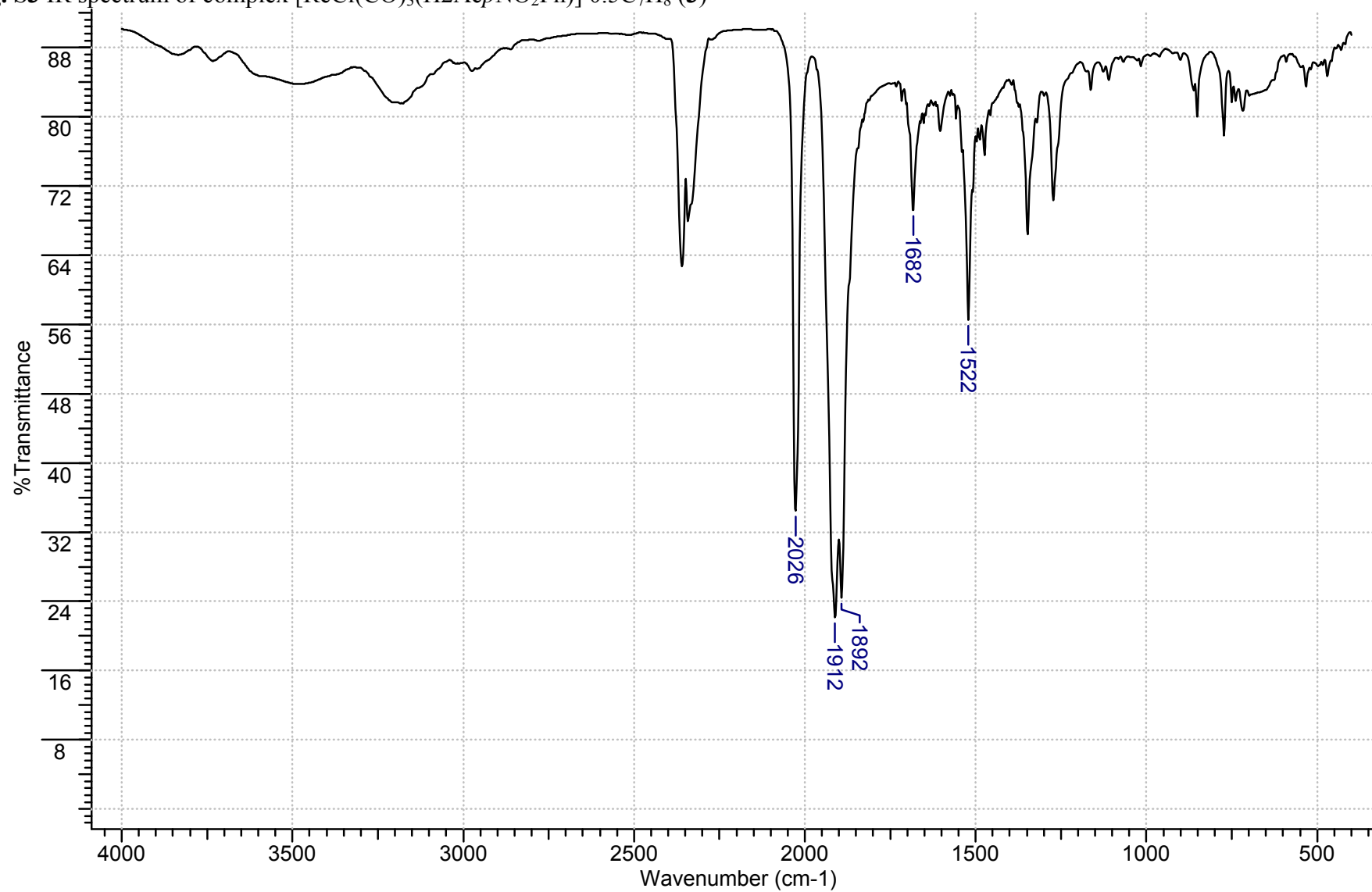


Fig. S3 IR spectrum of complex $[\text{ReCl}(\text{CO})_3(\text{H}_2\text{Ac}p\text{NO}_2\text{Ph})] \cdot 0.5\text{C}_7\text{H}_8$ (**3**)

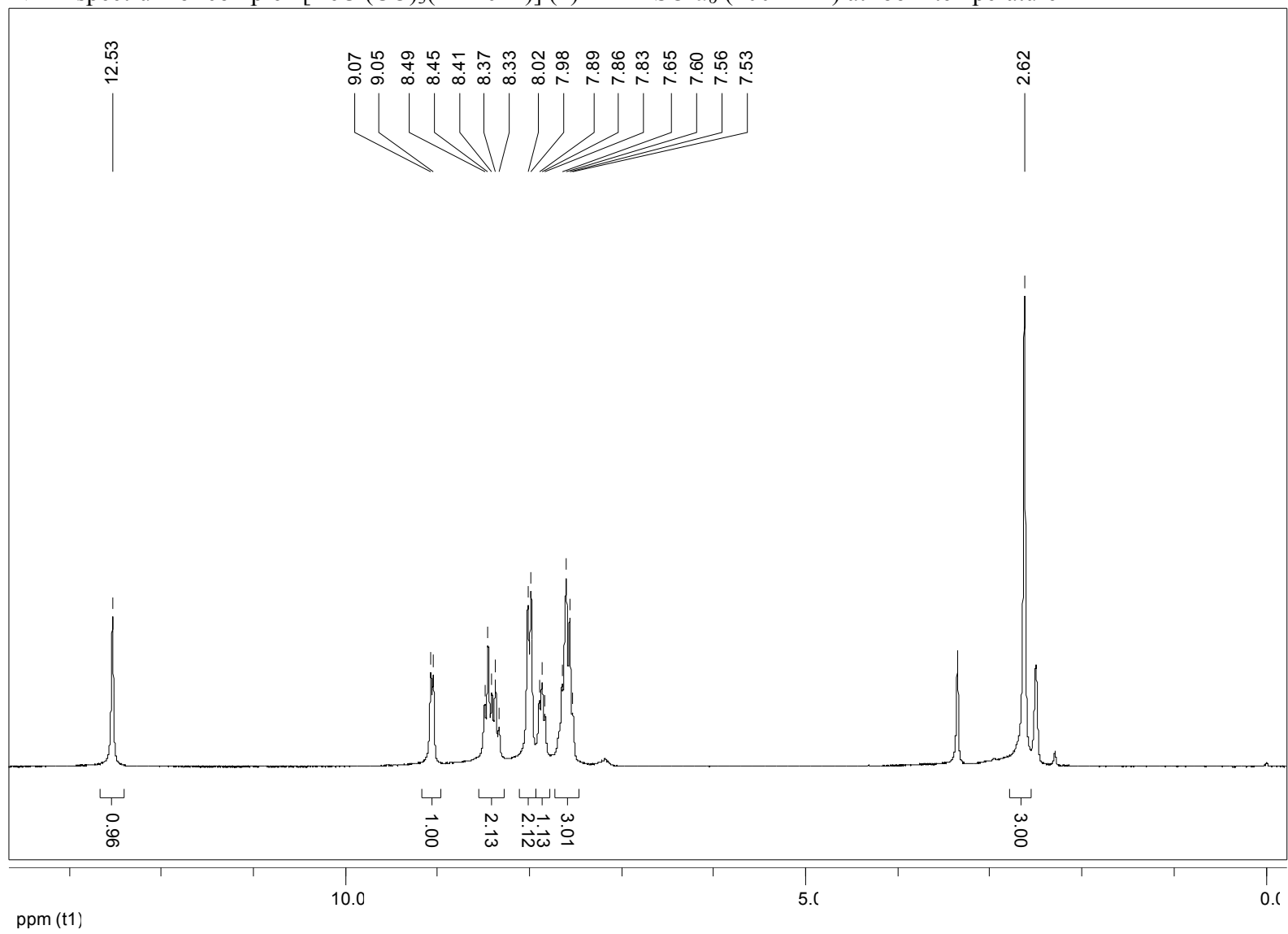


^1H and ^{13}C NMR spectra of complexes $[\text{ReCl}(\text{CO})_3(\text{H}_2\text{AcPh})]$ (**1**),

$[\text{ReCl}(\text{CO})_3(\text{H}_2\text{Ac}p\text{ClPh})] \cdot 0.5\text{C}_7\text{H}_8$ (**2**) and

$[\text{ReCl}(\text{CO})_3(\text{H}_2\text{Ac}p\text{NO}_2\text{Ph})] \cdot 0.5\text{C}_7\text{H}_8$ (**3**)

Fig. S4 ^1H NMR spectrum of complex $[\text{ReCl}(\text{CO})_3(\text{H}_2\text{AcPh})]$ (**1**) in $\text{DMSO-}d_6$ (200 MHz) at room temperature



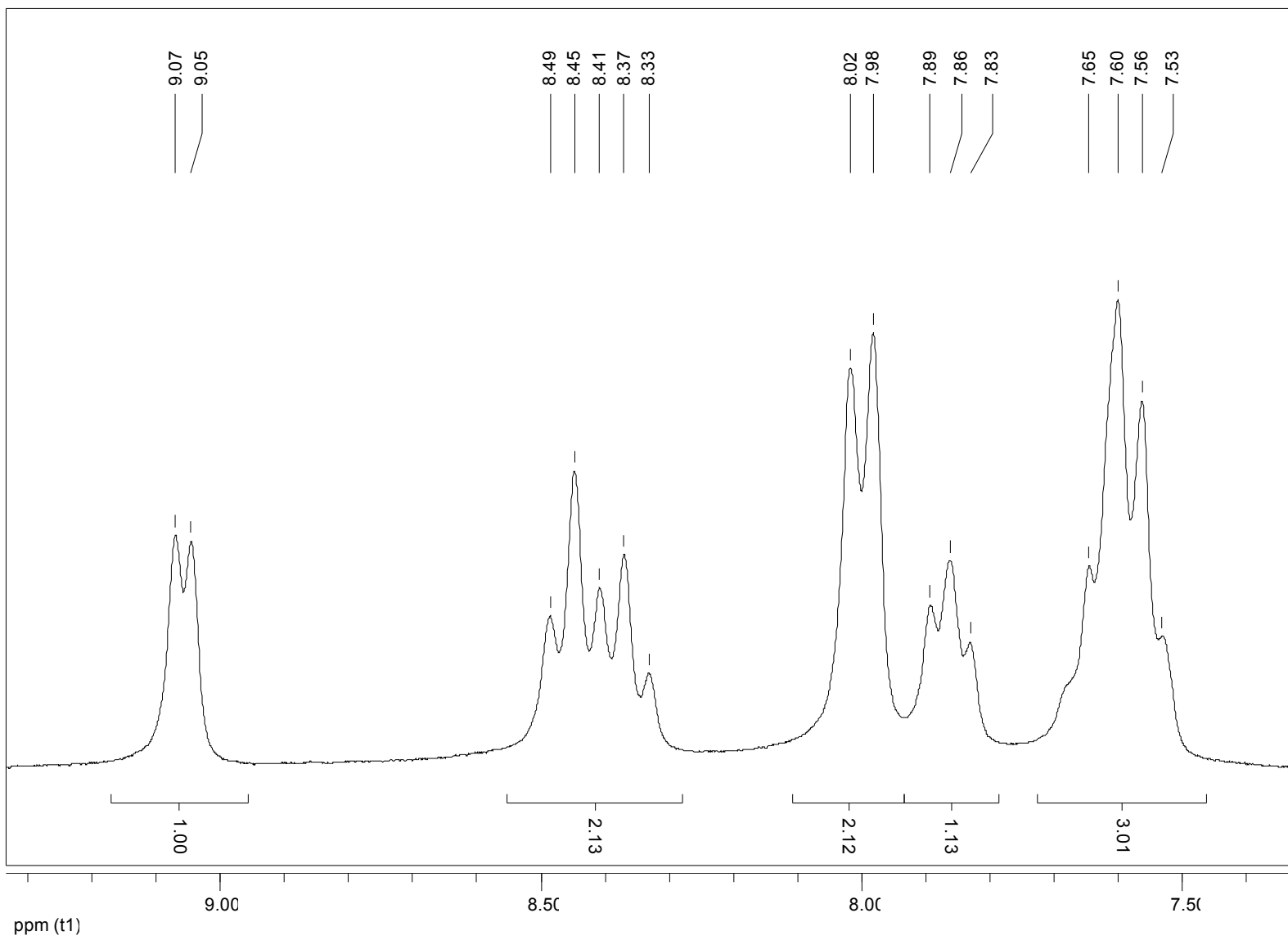
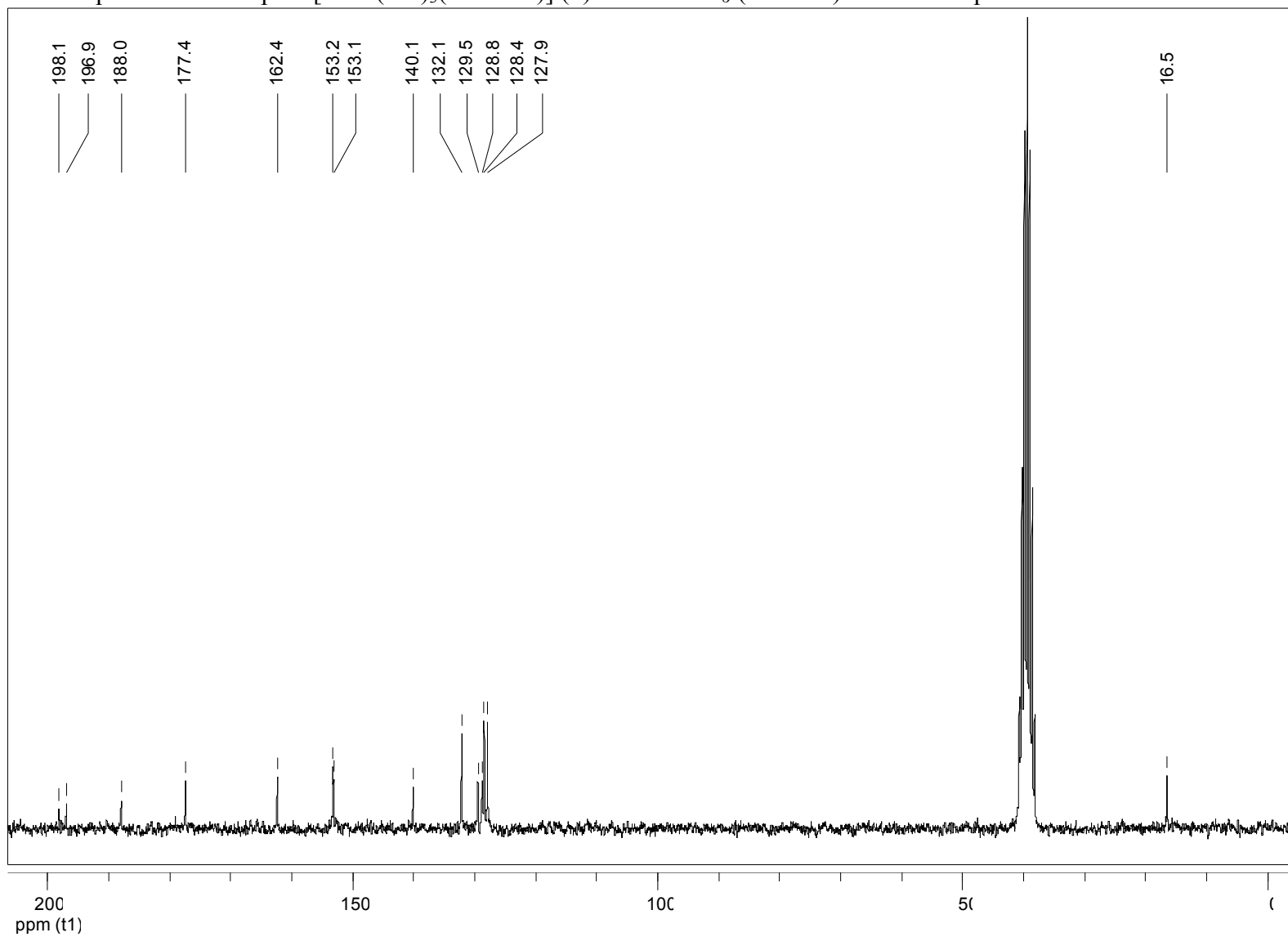
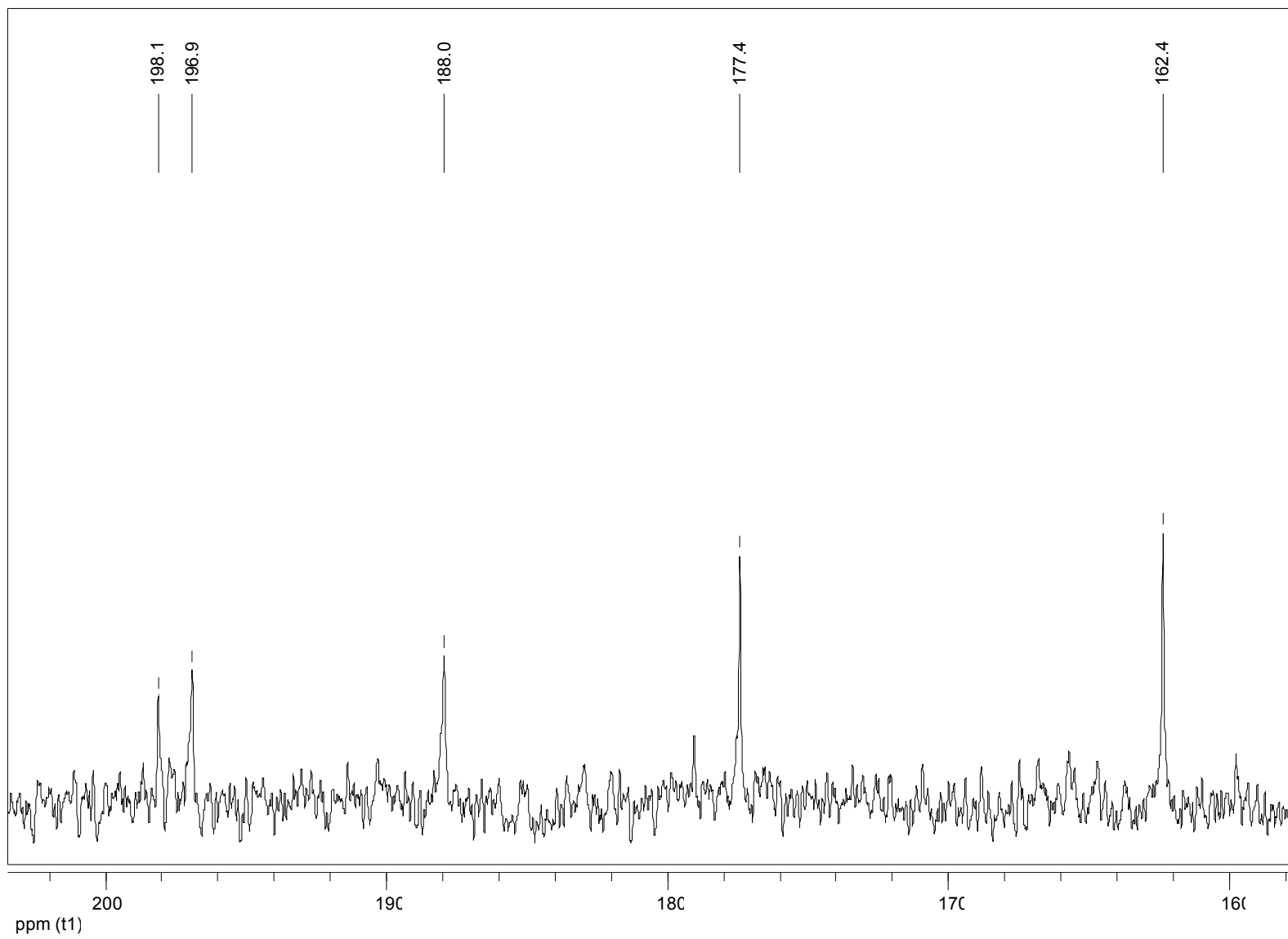
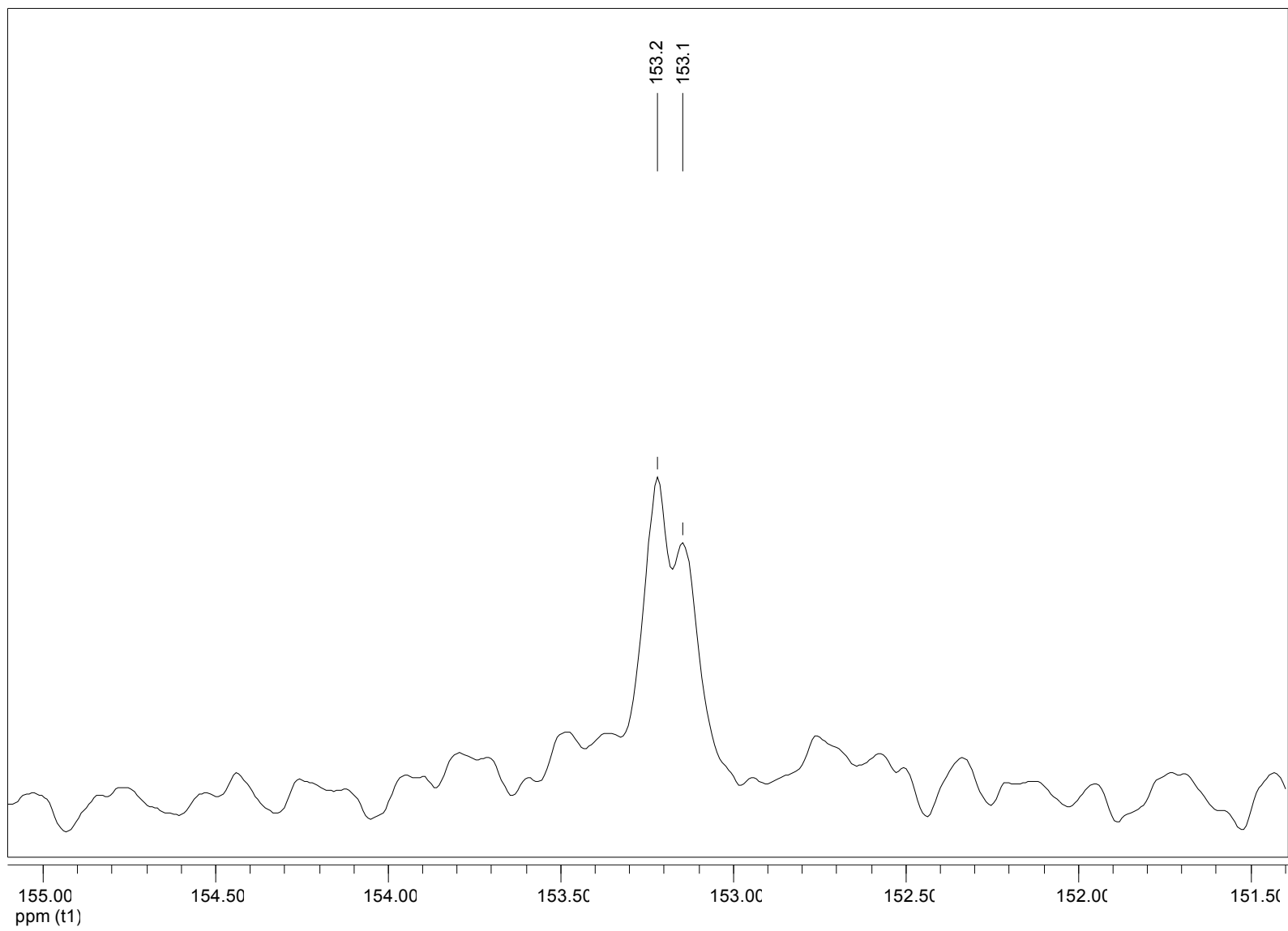


Fig. S5 ^{13}C NMR spectrum of complex $[\text{ReCl}(\text{CO})_3(\text{H}_2\text{AcPh})]$ (**1**) in $\text{DMSO-}d_6$ (50 MHz) at room temperature







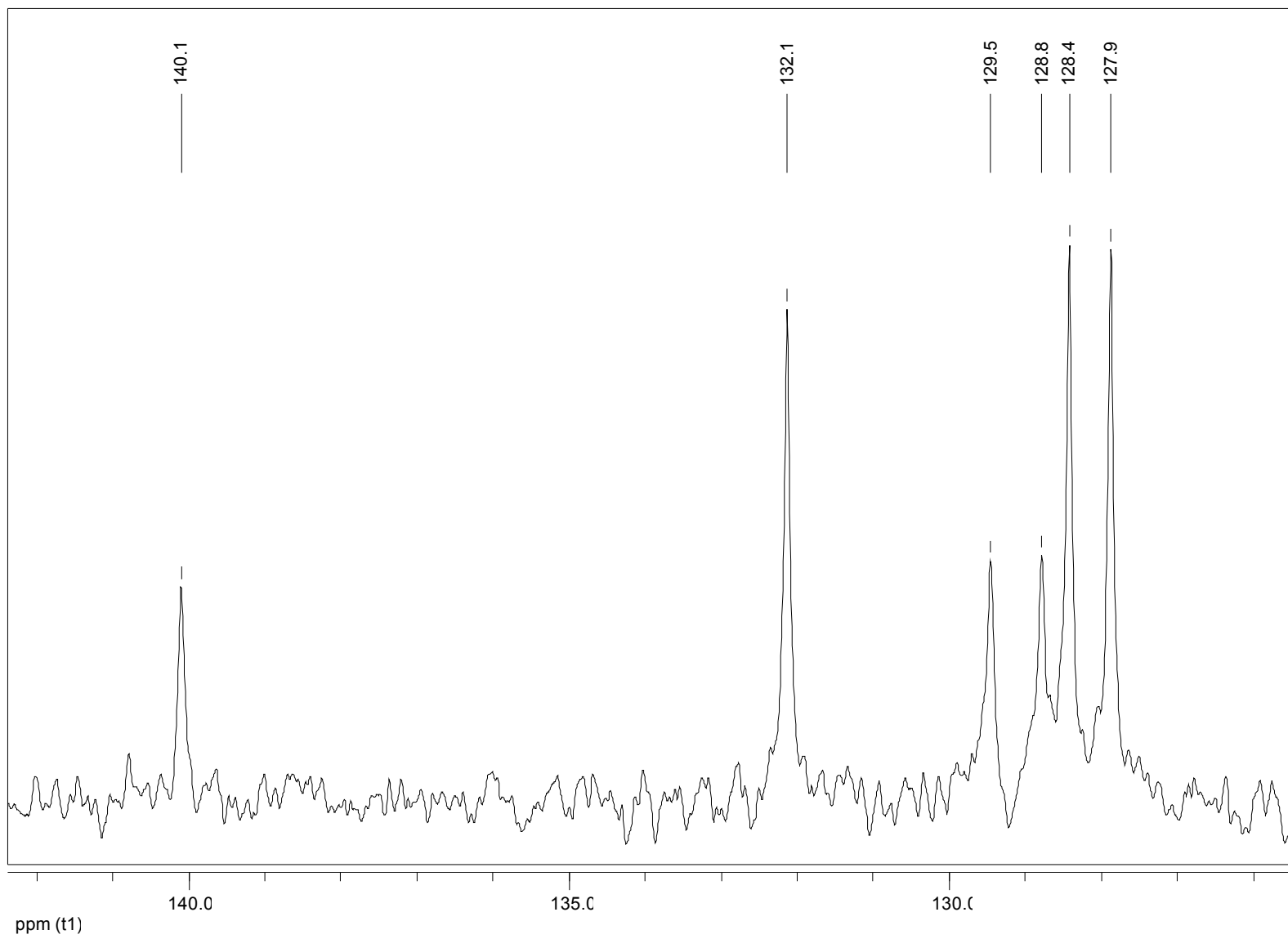
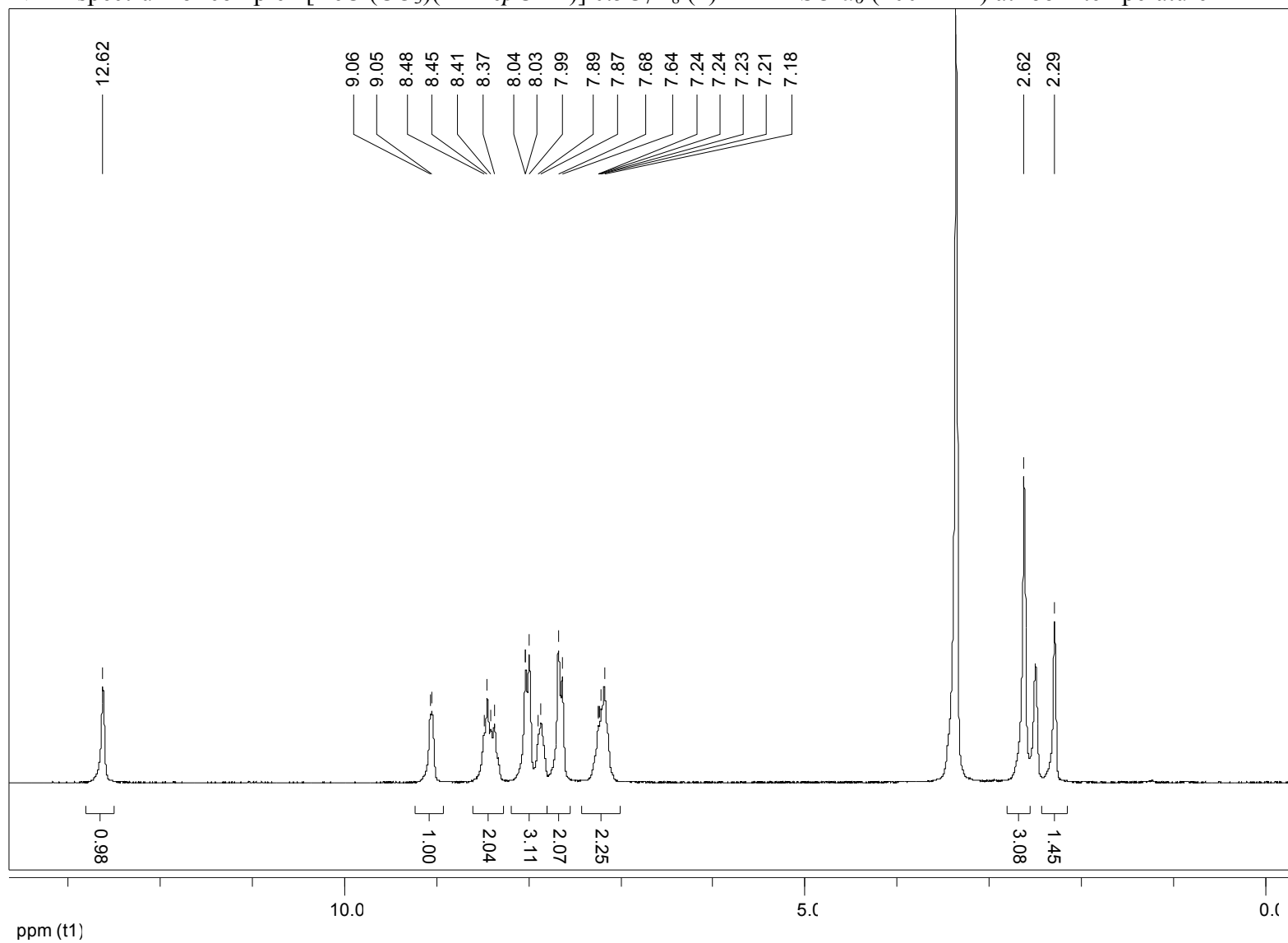


Fig. S6 ^1H NMR spectrum of complex $[\text{ReCl}(\text{CO}_3)(\text{H}_2\text{AcpClPh})] \cdot 0.5\text{C}_7\text{H}_8$ (**2**) in $\text{DMSO-}d_6$ (200 MHz) at room temperature



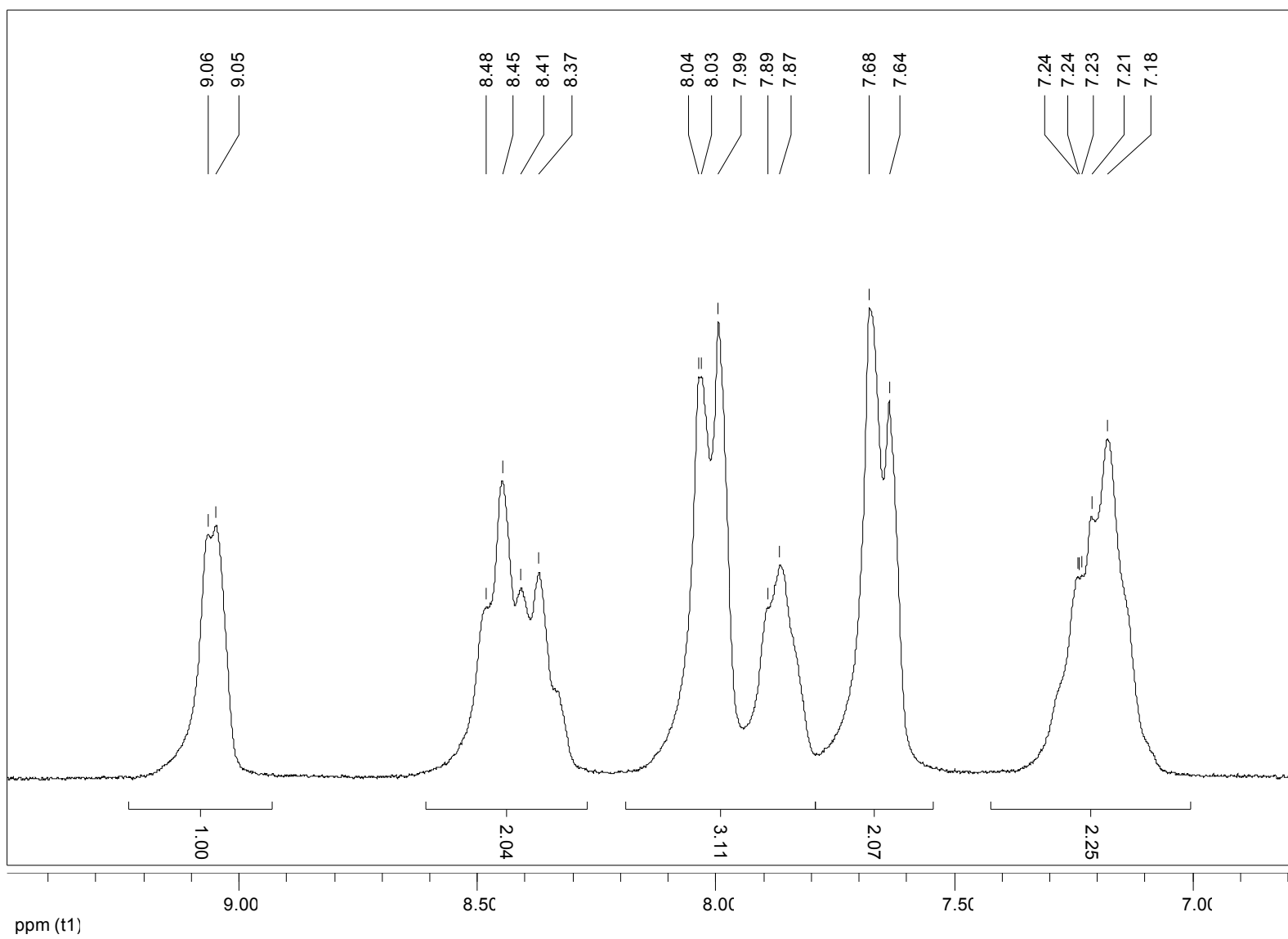
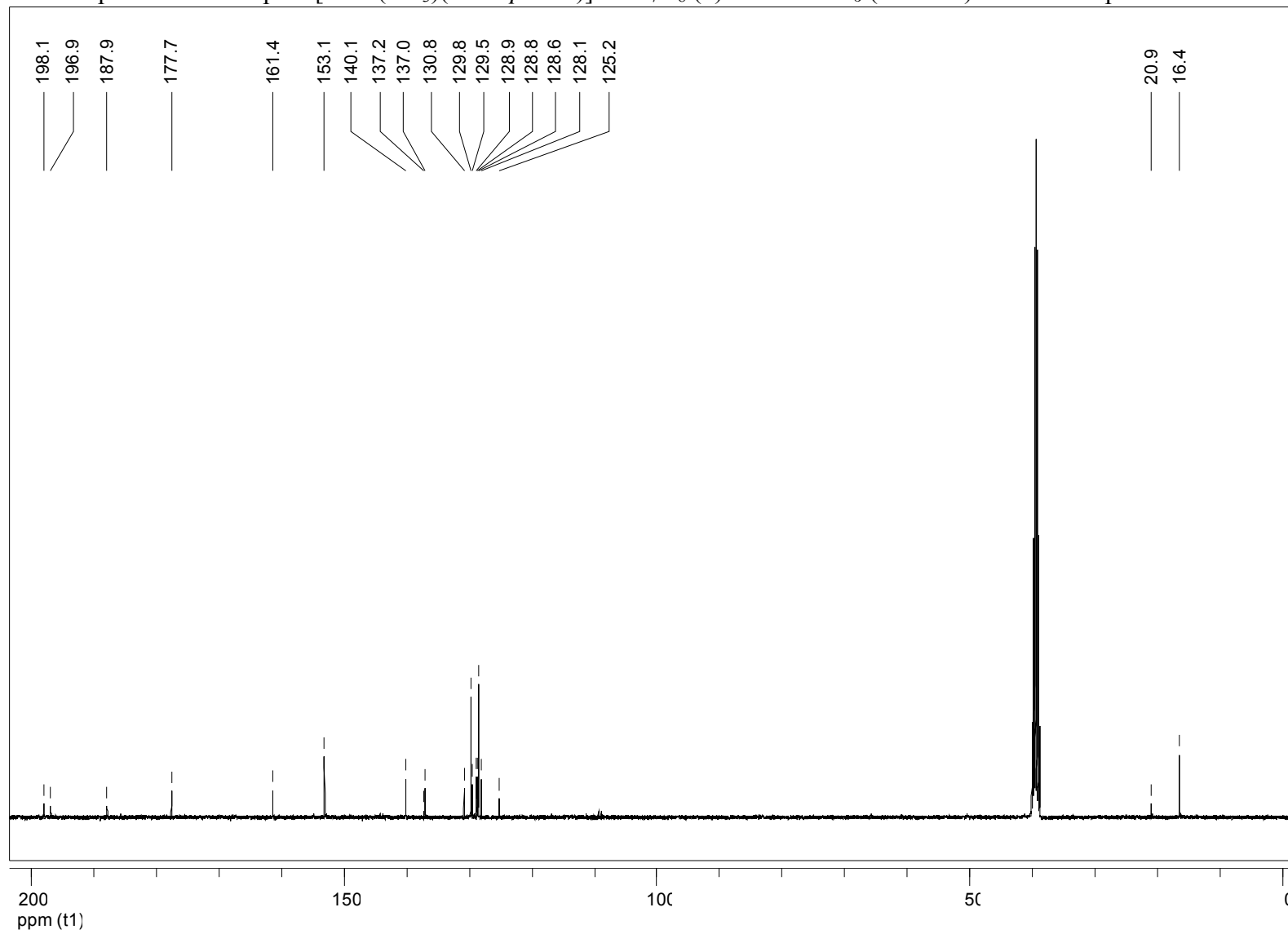
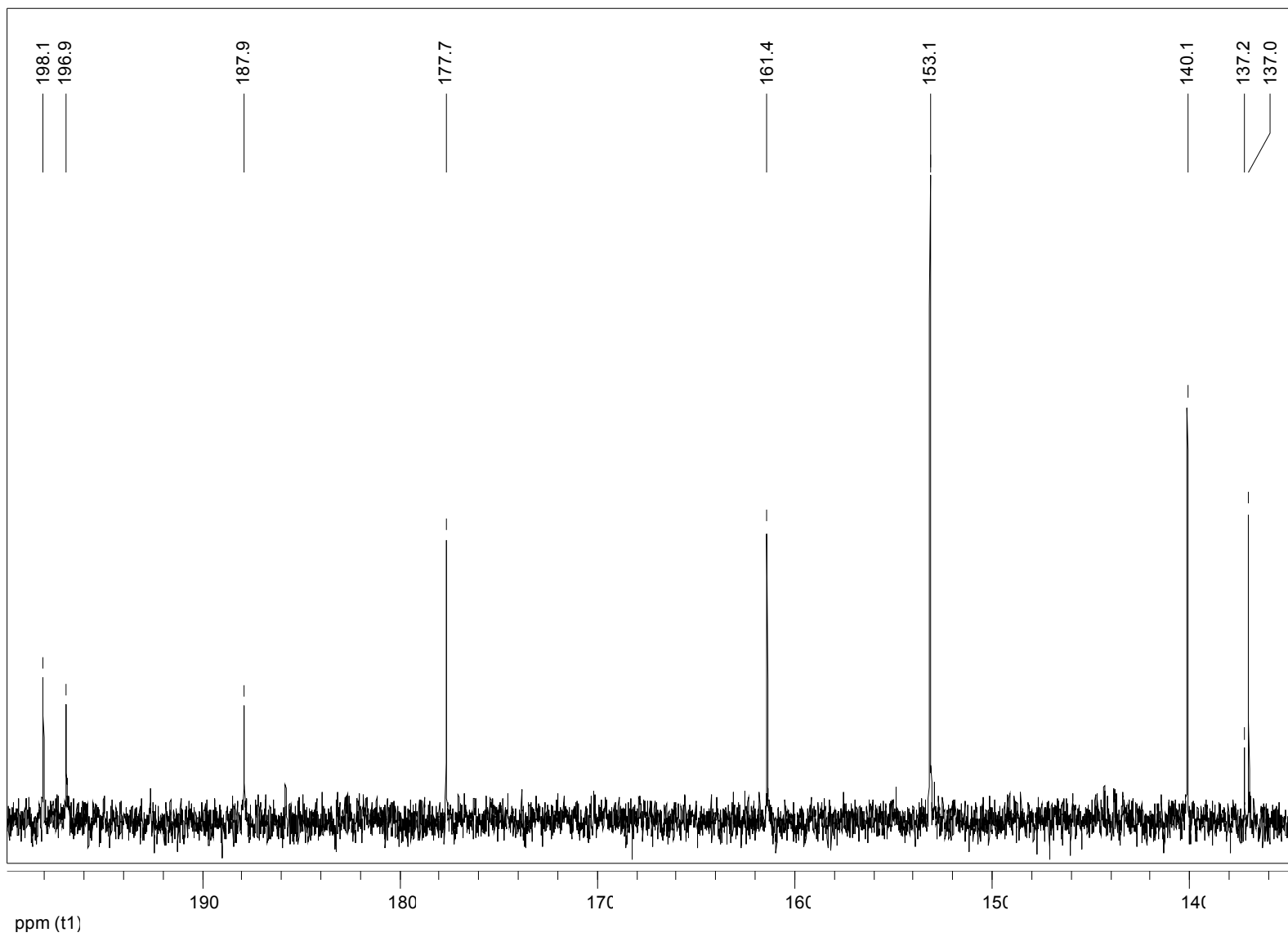


Fig. S7 ^{13}C NMR spectrum of complex $[\text{ReCl}(\text{CO}_3)(\text{H}_2\text{Ac}_p\text{ClPh})]\cdot 0.5\text{C}_7\text{H}_8$ (**2**) in $\text{DMSO-}d_6$ (50 MHz) at room temperature





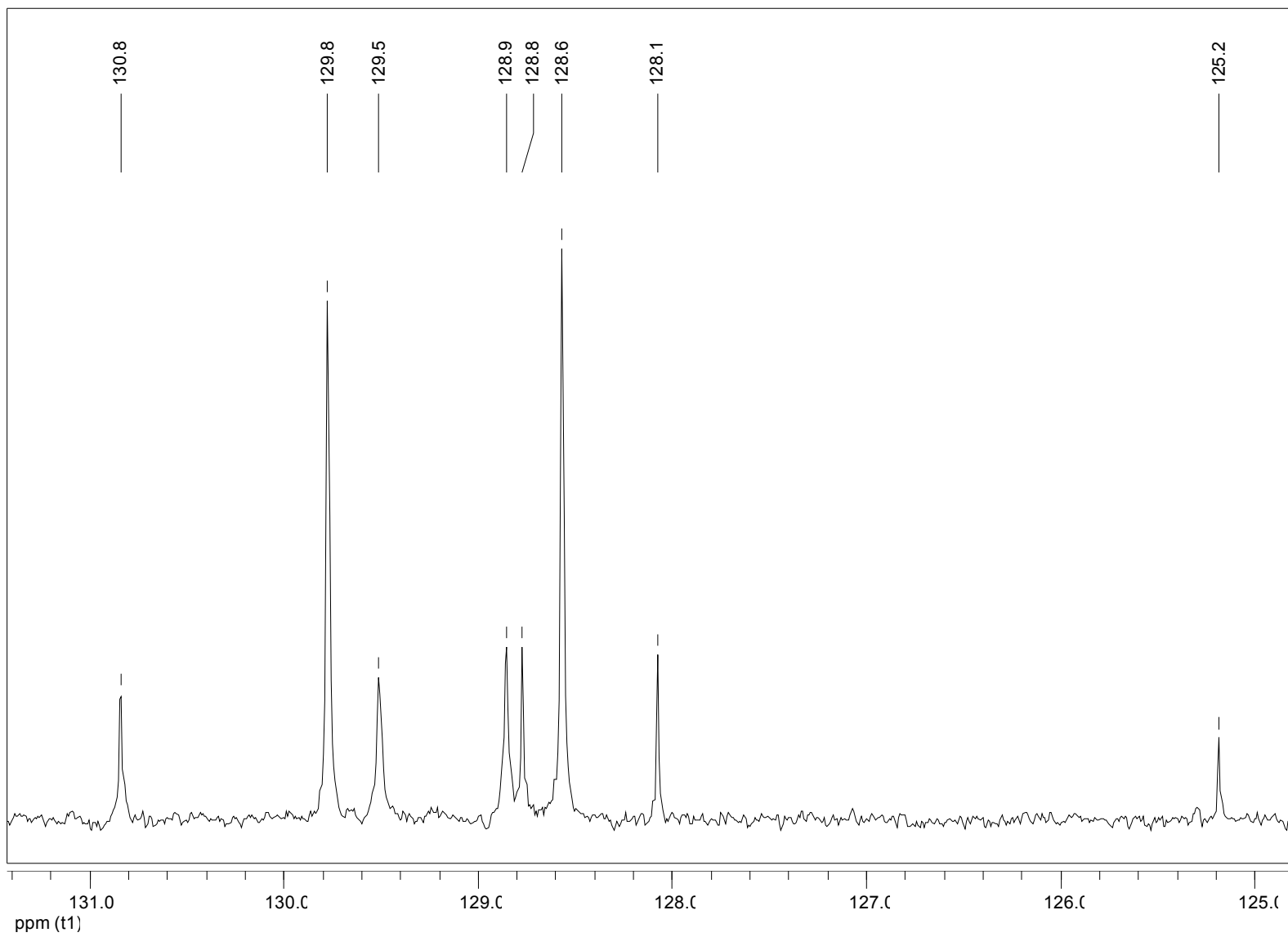
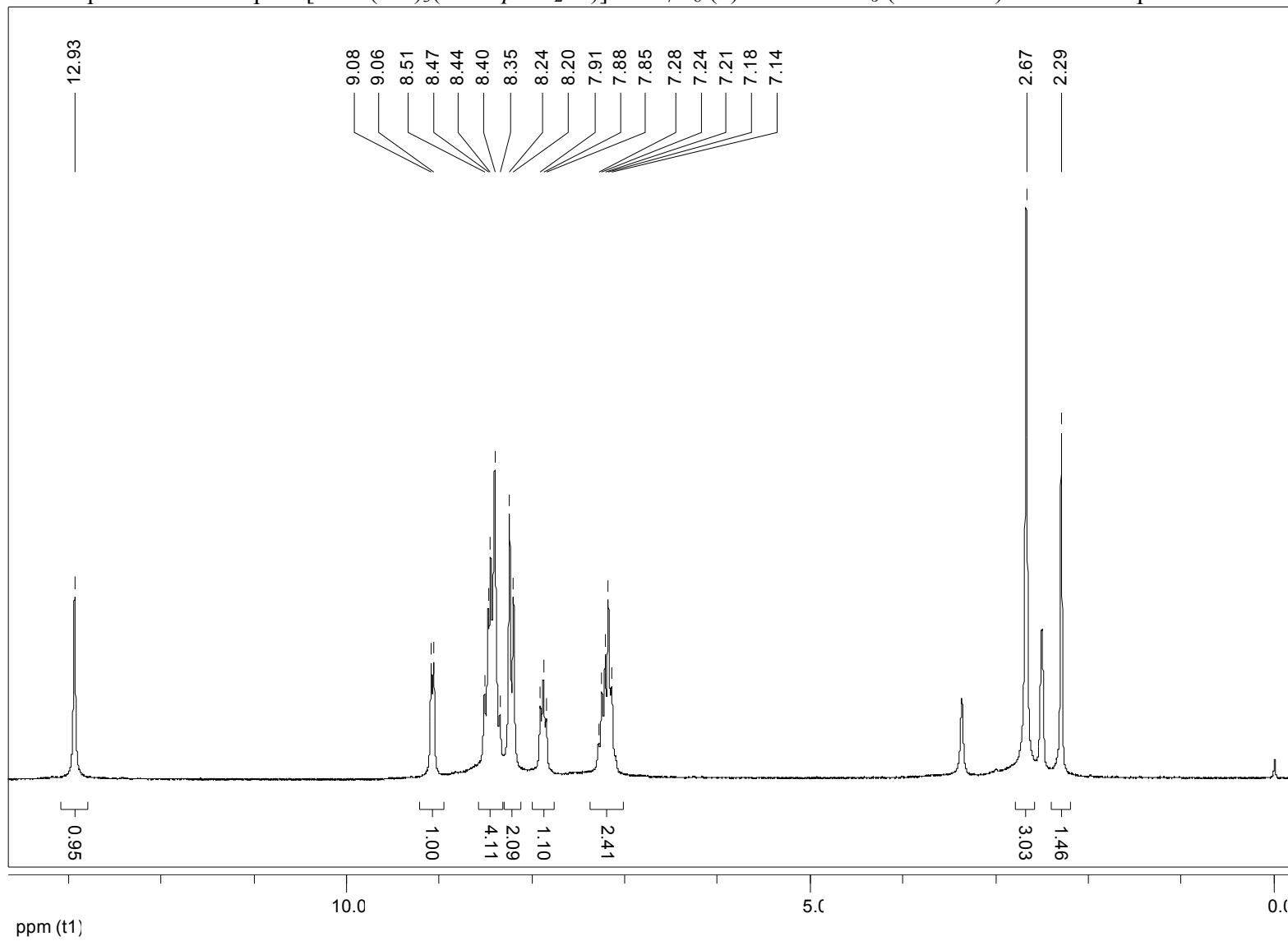


Fig. S8 ^1H NMR spectrum of complex $[\text{ReCl}(\text{CO})_3(\text{H}_2\text{Ac}p\text{NO}_2\text{Ph})]\cdot 0.5\text{C}_7\text{H}_8$ (**3**) in $\text{DMSO-}d_6$ (200 MHz) at room temperature



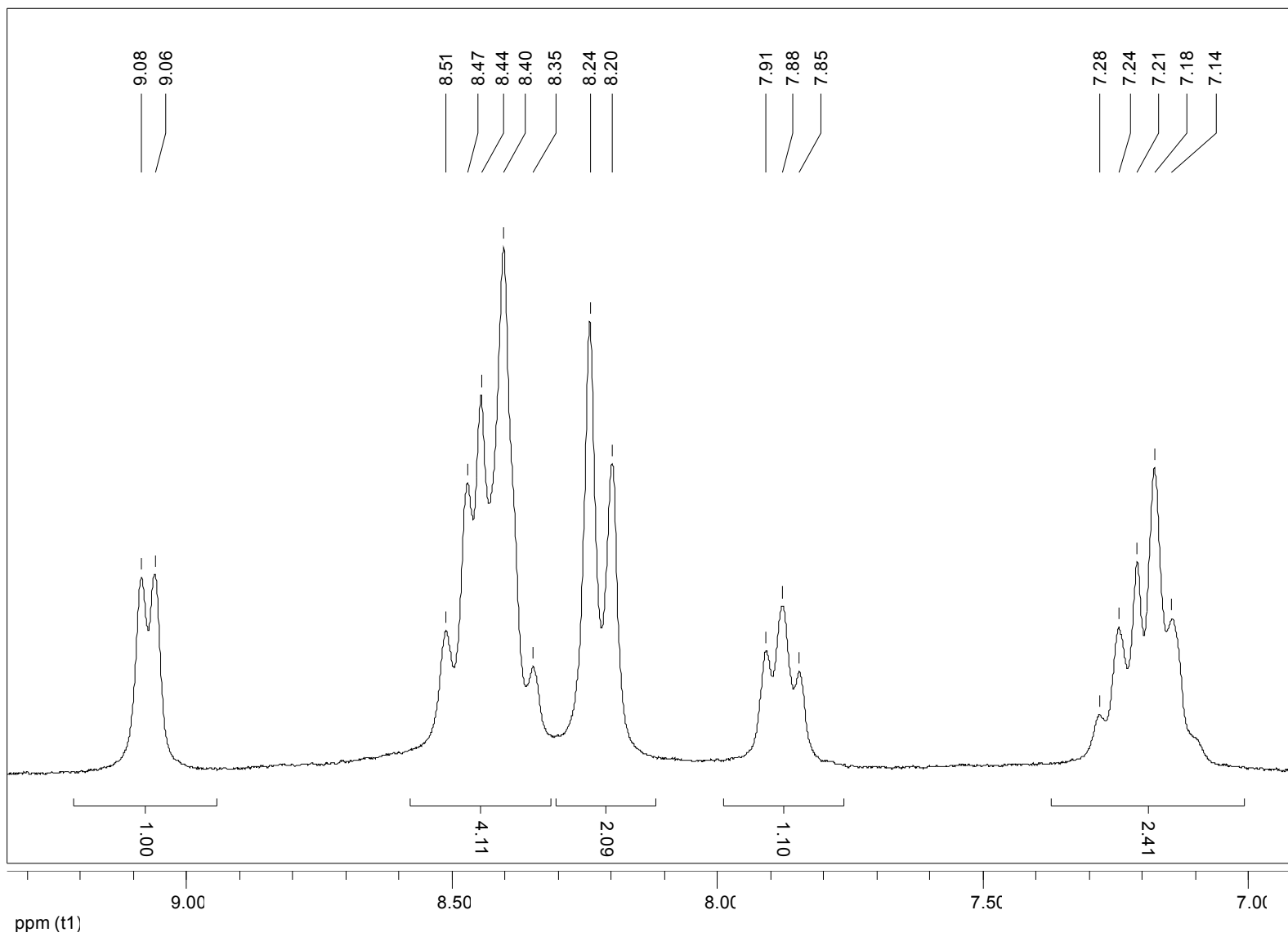
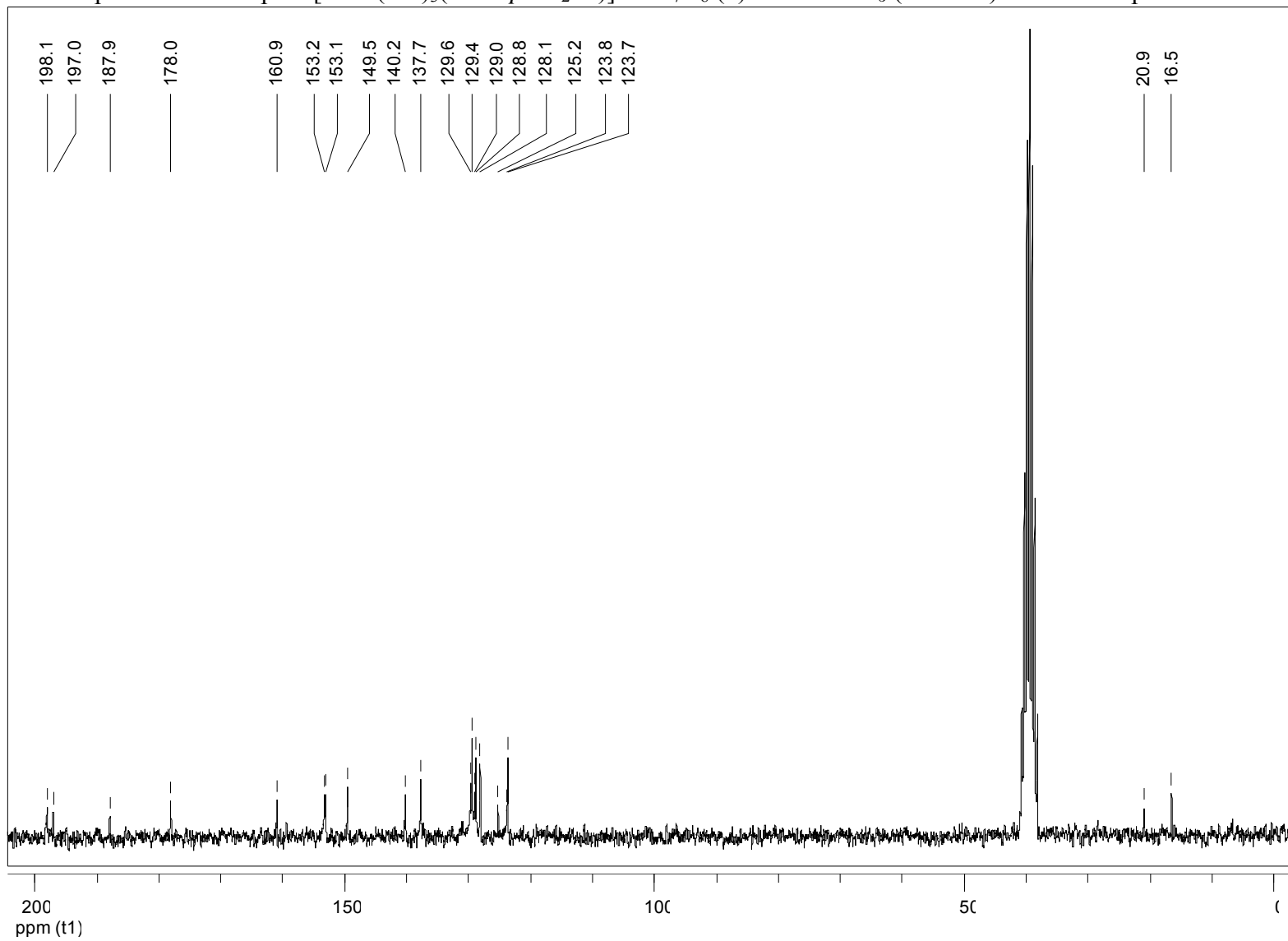
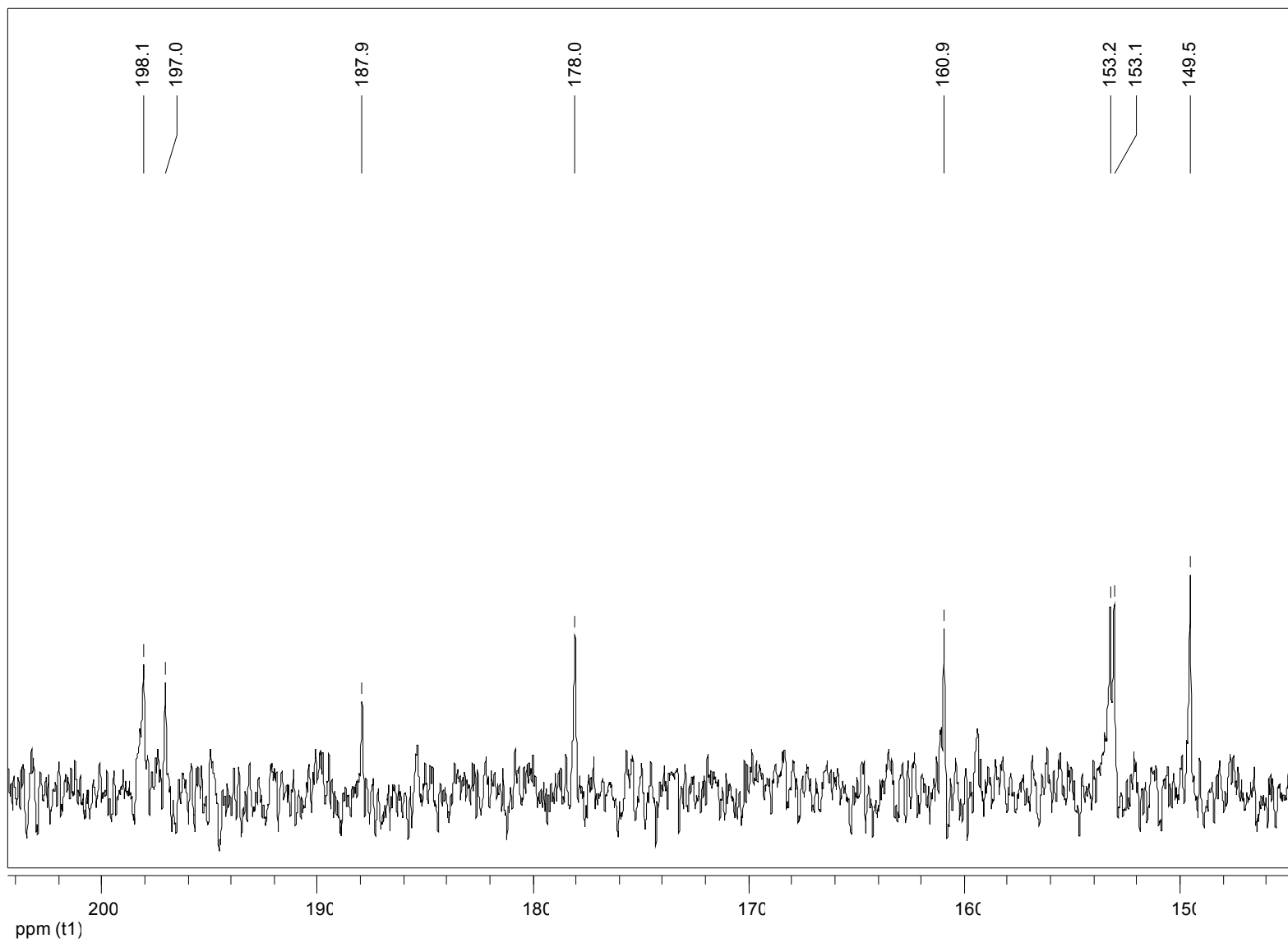
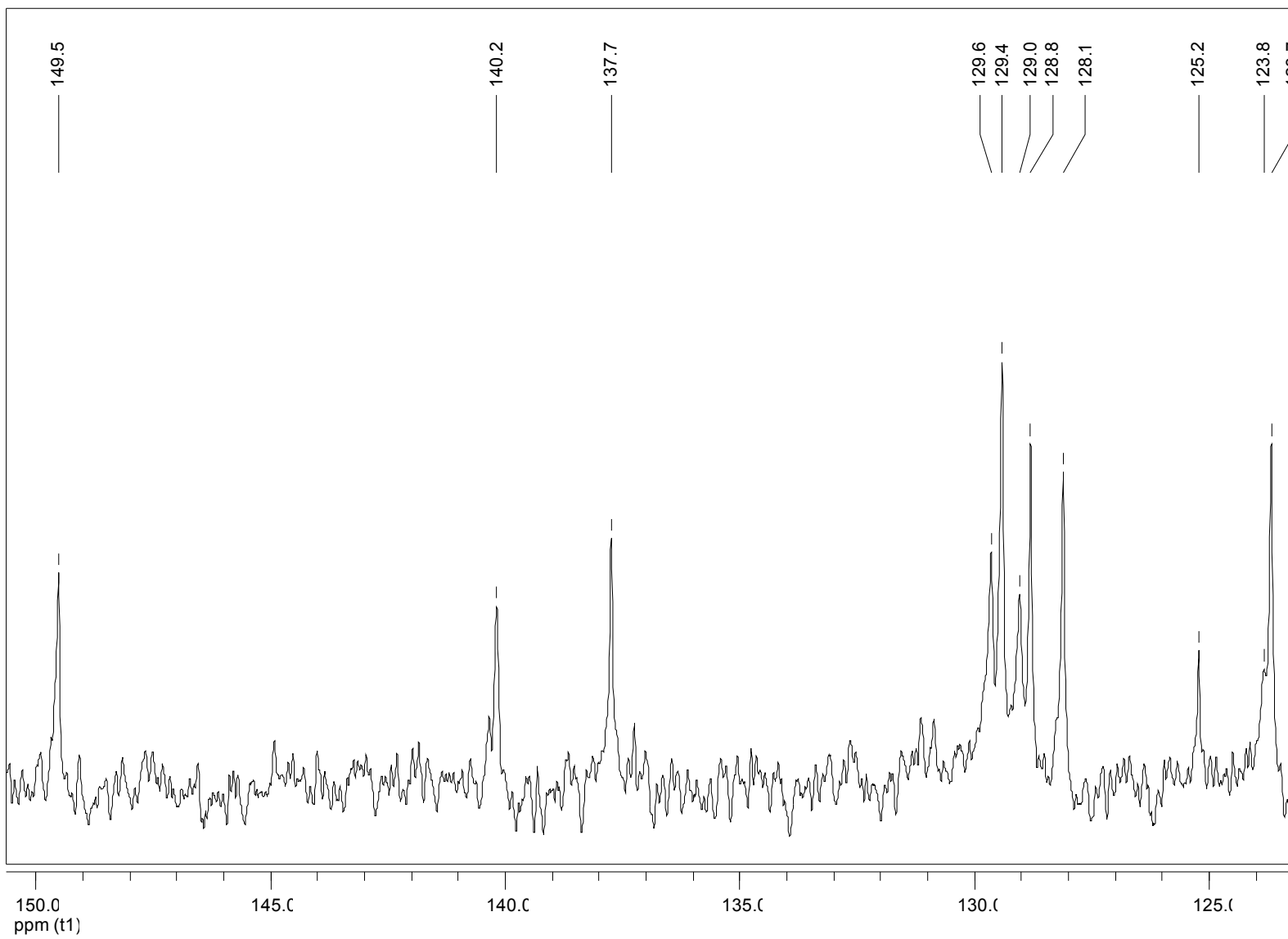


Fig. S9 ^{13}C NMR spectrum of complex $[\text{ReCl}(\text{CO})_3(\text{H}_2\text{Ac}_p\text{NO}_2\text{Ph})]\cdot 0.5\text{C}_7\text{H}_8$ (**3**) in $\text{DMSO-}d_6$ (50 MHz) at room temperature







CHECKCIFs files of complexes $[\text{ReCl}(\text{CO})_3(\text{H}_2\text{AcPh})]$ (**1**),

$[\text{ReCl}(\text{CO})_3(\text{H}_2\text{Ac}p\text{ClPh})]\cdot\text{DMSO}$ (**2a**) and

$[\text{ReCl}(\text{CO})_3(\text{H}_2\text{Ac}p\text{NO}_2\text{Ph})]\cdot\text{DMSO}$ (**3a**)

checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: RePh Complex (1)

Bond precision:	C-C = 0.0048 A	Wavelength=0.71073
Cell:	a=12.6872(2) b=9.5720(2) c=14.9830(2)	alpha=90 beta=98.300(2) gamma=90
Temperature:	293 K	
	Calculated	Reported
Volume	1800.51(5)	1800.51(5)
Space group	P 21/n	P_21/n
Hall group	-P 2yn	-P_2yn
Moiety formula	C17 H13 Cl N3 O4 Re	?
Sum formula	C17 H13 Cl N3 O4 Re	C17 H13 Cl N3 O4 Re
Mr	544.96	544.95
Dx,g cm-3	2.010	2.010
Z	4	4
Mu (mm-1)	6.925	6.925
F000	1040.0	1040.0
F000'	1037.09	
h,k,lmax	19,14,22	19,14,22
Nref	6806	6375
Tmin,Tmax	0.667,0.871	0.534,1.000
Tmin'	0.495	

Correction method= # Reported T Limits: Tmin=0.534 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.937 Theta(max)= 33.010

R(reflections)= 0.0222(5218) wR2(reflections)= 0.0650(6375)

S = 1.189 Npar= 235

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

● Alert level G

PLAT005_ALERT_5_G	No Embedded Refinement Details found in the CIF	Please Do !
PLAT007_ALERT_5_G	Number of Unrefined Donor-H Atoms	1 Report
PLAT093_ALERT_1_G	No s.u.'s on H-positions, Refinement Reported as	mixed Check
PLAT153_ALERT_1_G	The s.u.'s on the Cell Axes are Equal ..(Note)	0.0002 Ang.
PLAT180_ALERT_4_G	Check Cell Rounding: # of Values Ending with 0 =	3 Note
PLAT199_ALERT_1_G	Reported _cell_measurement_temperature (K)	293 Check
PLAT200_ALERT_1_G	Reported _diffrn_ambient_temperature (K)	293 Check
PLAT232_ALERT_2_G	Hirshfeld Test Diff (M-X) Rel -- C11 ..	6.5 s.u.
PLAT232_ALERT_2_G	Hirshfeld Test Diff (M-X) Rel -- C16 ..	6.8 s.u.
PLAT232_ALERT_2_G	Hirshfeld Test Diff (M-X) Rel -- C17 ..	8.1 s.u.
PLAT720_ALERT_4_G	Number of Unusual/Non-Standard Labels	1 Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
0 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
11 **ALERT level G** = General information/check it is not something unexpected

4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
0 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
2 ALERT type 5 Informative message, check

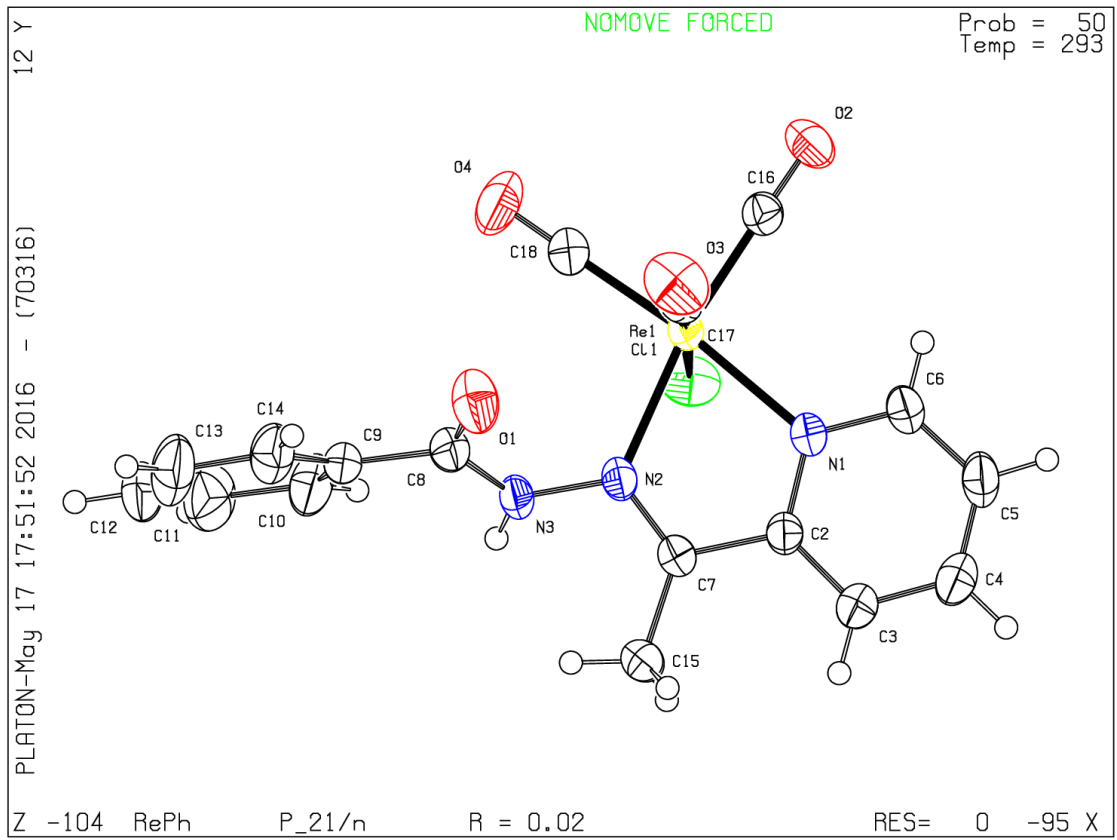
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You have not supplied any structure factors. As a result the full set of tests cannot be run.

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: Re_pCl complex (2a)

Bond precision: C-C = 0.0039 A

Wavelength=0.71073

Cell: a=8.7687(4) b=11.1002(5) c=12.9282(5)
 alpha=113.002(4) beta=97.163(4) gamma=93.603(4)
Temperature: 200 K

	Calculated	Reported
Volume	1140.66(9)	1140.66(9)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C17 H12 Cl2 N3 O4 Re, C2 H6 O S	C19 H18 Cl2 N3 O5 Re S
Sum formula	C19 H18 Cl2 N3 O5 Re S	C19 H18 Cl2 N3 O5 Re S
Mr	657.53	657.52
Dx,g cm-3	1.914	1.914
Z	2	2
Mu (mm-1)	5.688	5.688
F000	636.0	636.0
F000'	635.10	
h,k,lmax	13,16,19	13,16,19
Nref	8528	7953
Tmin,Tmax	0.626,0.892	0.555,1.000
Tmin'	0.422	

Correction method= # Reported T Limits: Tmin=0.555 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.933

Theta(max)= 32.880

R(reflections)= 0.0255(7050)

wR2(reflections)= 0.0508(7953)

S = 1.061

Npar= 283

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

● **Alert level G**

PLAT005_ALERT_5_G	No Embedded Refinement Details found in the CIF	Please Do !
PLAT007_ALERT_5_G	Number of Unrefined Donor-H Atoms	1 Report
PLAT042_ALERT_1_G	Calc. and Reported MoietyFormula Strings Differ	Please Check
PLAT093_ALERT_1_G	No s.u.'s on H-positions, Refinement Reported as	mixed Check
PLAT154_ALERT_1_G	The s.u.'s on the Cell Angles are Equal ..(Note)	0.004 Degree
PLAT232_ALERT_2_G	Hirshfeld Test Diff (M-X) Re1 -- C16 ..	6.9 s.u.
PLAT232_ALERT_2_G	Hirshfeld Test Diff (M-X) Re1 -- C17 ..	6.0 s.u.
PLAT232_ALERT_2_G	Hirshfeld Test Diff (M-X) Re1 -- C18 ..	7.3 s.u.
PLAT720_ALERT_4_G	Number of Unusual/Non-Standard Labels	1 Note
PLAT721_ALERT_1_G	Bond Calc 4.95000, Rep 0.86000 Dev...	4.09 Ang.
	N3 -H3 1.555 1.555	Bond # 20 Check
PLAT722_ALERT_1_G	Angle Calc 144.00, Rep 120.60 Dev...	23.40 Degree
	C8 -N3 -H3 1.555 1.555 1.555 #	26
PLAT722_ALERT_1_G	Angle Calc 38.00, Rep 120.60 Dev...	82.60 Degree
	N2 -N3 -H3 1.555 1.555 1.555 #	27

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
0 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
12 **ALERT level G** = General information/check it is not something unexpected

6 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
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1 ALERT type 4 Improvement, methodology, query or suggestion
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It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

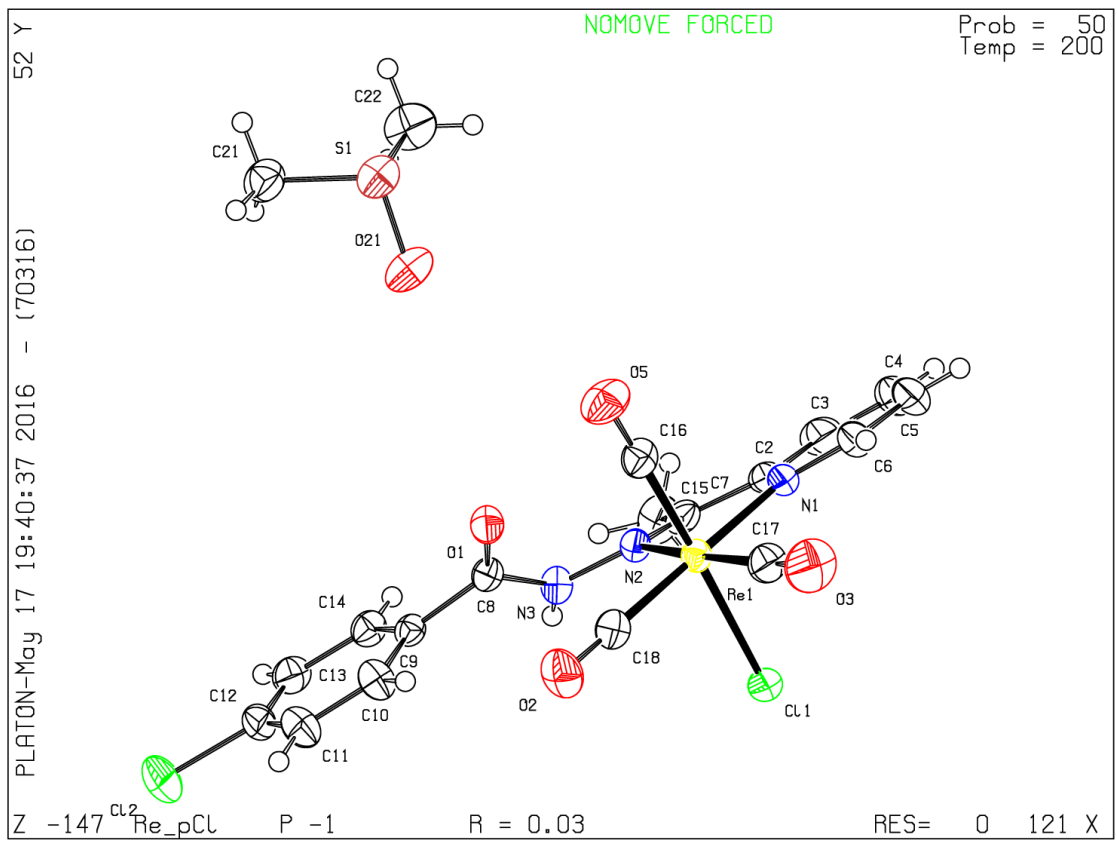
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A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

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PLATON version of 06/05/2016; check.def file version of 05/05/2016



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No syntax errors found. CIF dictionary Interpreting this report

Datablock: RepNO2 complex (3a)

Bond precision: C-C = 0.0072 A

Wavelength=0.71073

Cell: a=8.8972(5) b=11.1200(6) c=12.9995(6)
 alpha=112.409(4) beta=96.989(4) gamma=93.490(4)
Temperature: 293 K

	Calculated	Reported
Volume	1172.08(11)	1172.08(11)
Space group	P -1	P-1
Hall group	-P 1	-P1
Moiety formula	C17 H12 Cl N4 O6 Re, C2 H6 O S	?
Sum formula	C19 H18 Cl N4 O7 Re S	C19 H18 Cl N4 O7 Re S
Mr	668.09	668.08
Dx, g cm-3	1.893	1.893
Z	2	2
Mu (mm-1)	5.434	5.434
F000	648.0	648.0
F000'	646.83	
h,k,lmax	12,15,18	11,15,17
Nref	6568	5446
Tmin,Tmax	0.729,0.897	0.669,1.000
Tmin'	0.575	

Correction method= # Reported T Limits: Tmin=0.669 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.829

Theta(max)= 29.550

R(reflections)= 0.0297(4833)

wR2(reflections)= 0.0715(5446)

S = 1.082

Npar= 300

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.



Alert level C

PLAT242_ALERT_2_C	Low	'MainMol'	Ueq as Compared to Neighbors of	N4	Check
PLAT751_ALERT_4_C	Bond	Calc	0.93000, Rep 0.927(4)	Senseless	s.u.
	N3 -HN2	1.555	1.555	Bond #	21 Check
PLAT752_ALERT_4_C	Angle	Calc	117.00, Rep 117.3(3)	Senseless	s.u.
	C8 -N3 -HN2	1.555	1.555 1.555	#	26
PLAT752_ALERT_4_C	Angle	Calc	123.00, Rep 122.5(3)	Senseless	s.u.
	N2 -N3 -HN2	1.555	1.555 1.555	#	27



Alert level G

PLAT005_ALERT_5_G	No Embedded Refinement Details found	in the CIF	Please Do !
PLAT007_ALERT_5_G	Number of Unrefined Donor-H Atoms		1 Report
PLAT093_ALERT_1_G	No s.u.'s on H-positions, Refinement Reported as		mixed Check
PLAT154_ALERT_1_G	The s.u.'s on the Cell Angles are Equal ..(Note)		0.004 Degree
PLAT199_ALERT_1_G	Reported _cell_measurement_temperature	(K)	293 Check
PLAT200_ALERT_1_G	Reported _diffrn_ambient_temperature	(K)	293 Check
PLAT380_ALERT_4_G	Incorrectly? Oriented X(sp2)-Methyl Moiety		C15 Check
PLAT720_ALERT_4_G	Number of Unusual/Non-Standard Labels		1 Note

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