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Data Article

NMR data of a Grubbs 2nd generation catalyst p-cresolate derivative

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A R T I C L E I N F O

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A B S T R A C T

The data presented in this article is related to the research article entitled “Spectroscopic characterisation of Grubbs 2nd generation catalyst and its *p*-cresol derivatives” (Swart *et al.* 2021). The 1D and 2D NMR characterisation data of the *p*-cresol derivative of the Grubbs 2nd generation catalyst, where one of the chloride ligands is replaced by the *p*-cresolate to form a Ru-O coordination compound (**3**) is reported. The characterization data include information obtained from ^{1}H, ^{13}C, Heteronuclear Single Quantum Coherence (HSQC), Heteronuclear Multiple Bond Correlation (HMBC), Homonuclear Correlation Spectroscopy (COSY), Nuclear Overhauser Effect (NOE) and Distortionless Enhancement by Polarization Transfer (DEPT) NMR spectroscopy.

Specifications Table

Subject	Chemistry
Specific subject area	Homogeneous catalysis, spectroscopic characterisation
Type of data	Table Figure

(*continued on next page*)

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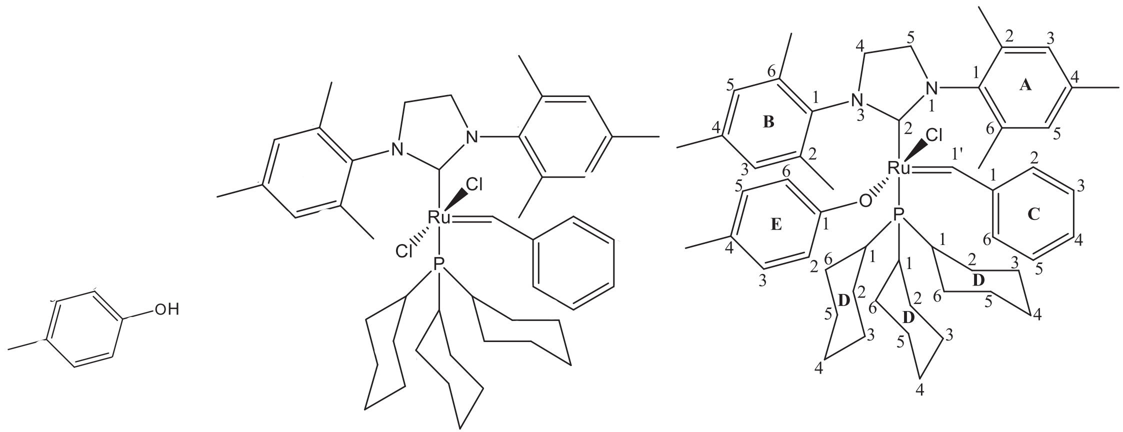
How data were acquired	Bruker AVANCE II 600 FT NMR spectrometer, 400 MHz AVANCE III spectrometer, Microsoft Excel 2016
Data format	Raw Analysed
Parameters for data collection	The NMR spectra were recorded using following parameters: Solvent: $CDCl_3$ Temperature (K): 278.15 Spectrometer frequency (MHz): 600.28 (^{1}H) & 150.95 (^{13}C) Number of scans: 8 (HSQC); 8 (HMBC); 8 (COSY); 8 (NOE) & 4 (DEPT) Relaxation delay(sec): 1.5 Acquisition time (sec): HSQC: AQ (^{1}H) = 0.0655; AQ (^{13}C) = 0.00486; HMBC: AQ (^{1}H) = 0.131; AQ (^{13}C) = 0.00486 COSY: AQ (^{1}H) = 00655; AQ (^{1}H) = 0.033 NOE: AQ (^{1}H) = 0.131; AQ (^{1}H) = 0.016 DEPT: AQ (^{13}C) = 0.612 Spectral width: HSQC: SW (^{1}H) = 26.0 ppm / 15 625 Hz; SW (^{13}C) = 348.63 ppm / 52 631 Hz HMBC: SW (^{1}H) = 26.0 ppm / 15 625 Hz SW (^{13}C) = 348.63 ppm / 52 631 Hz COSY: SW (^{1}H) = 26.0 ppm / 15 625 Hz SW (^{1}H) = 25.9 ppm / 15607 Hz NOE: SW (^{1}H) = 26.0 ppm / 15 625 Hz SW (^{1}H) = 25.9 ppm / 15607 Hz DEPT: SW (^{13}C) = 354.85 ppm / 53571 Hz
Description of data collection	NMR: Chemical shifts were shown as δ-values with reference to tetramethylsilane (TMS) as an internal standard.
Data source location	University of the Free State, Bloemfontein, South Africa Latitude: -29.110028° Longitude: 26185706°
Data accessibility	See Supplementary Information.
Related research article	M.R. Swart, Barend C.B. Bezuidenhoudt, C. Marais, E. Erasmus, Spectroscopic characterisation of Grubbs 2nd generation catalyst and its *p*-cresol derivatives, Inorganica Chimica Acta, 2021, 514, 120001, doi.org/10.1016/j.ica.2020.120001 [1].

Value of the Data

- The data represents the NMR characterization of the Grubbs 2nd generation catalyst and *p*-cresol derivatives thereof.
- The data can be useful to researchers interested in improving Grubbs 2nd generation catalyst for a variety of metathesis reactions.
- Our data contributes to the characterization of the Grubbs 2nd generation catalyst and its *p*-cresol derivatives.
- The data will be useful for the modification of catalysts towards improved metathesis.

1. Data Description

A derivative between *p*-cresol (**1**) and Grubbs 2nd generation catalyst (**2**) was prepared. The adduct, **3**, is a Grubbs 2nd generation-*p*-cresolate derivative with a Ru-O coordination as a result of Cl - *p*-cresolate ligand exchange. The structure of **1-3** are presented in Fig. 1, with the structure of **3** showing the numerical labels used to assign the NMR data presented in Table 1. The ^{1}H and ^{13}C Nuclear Magnetic Resonance NMR resonances of **3** (see Figs. 2 and 3) were allocated in analogy to those of Grubbs second generation catalyst (**1**) [2,3] and by means of Heteronuclear single quantum coherence spectroscopy (HSQC) and Heteronuclear Multiple Bond Correlation (HMBC) experiments and are summarised in Table 1. The HSQC and HMBC spectra of complex (**3**) are presented in Fig. 4. Figs. 5–7 depicts the homonuclear correlation spectroscopy



1 **2** **3**

Fig. 1. The structures of p-cresol **(1)**, Grubbs 2nd generation catalyst **(2)**, and the modified Grubbs 2nd generation- p-cresolate catalyst, **3**. The structure of **3**, shows the numerical labels of the carbon atoms used to indicate their NMR positions reported in Table 1.

Table 1
^{1}H and ^{13}C NMR data of Ru(=CHC_6H_5)($OC_6H_4CH_3$)(Cl)(PCy_3)$_3$(H_2IMes) (**3**) in $CDCl_3$ at 25°C.

Position	^{1}H δ (ppm)	m, J (Hz)	^{13}C δ (ppm)	m, J (Hz)	HMBC correlations and other support
2	-		220.4	d, 77.6	
4	3.99	br s	52.2	d, 3.79	
5	3.80	br s	51.3	br s	
	3.99	br s	52.2	d, 3.79	
1′	19.14	s	294.5		1C, 2C, 6C
	16.84(trace)		288.8 (trace)		
	16.80 (trace)				
1 (A)			134.9 or 135.1		
2&6 (A)	-		139.5 – 138.5 and/or 137.0 – 136.5	m, m	
3 (A)	5.82	br s	128.9 & 128.4		4-$\underline{C}H_3$(A)
4 (A)	-		137.7 & 137.6		4-$\underline{C}H_3$(A)
5 (A)	6.72	br s	128.9 & 128.4		4-$\underline{C}H_3$(A)
2-Me (A)	2.15 – 1.96	m	19.0 – 18.2		Direct coupling
4-Me (A)	1.91	s	20.9		Direct coupling; C-3(A), C-5(A), C-4(A)
6-Me (A)	2.64 – 2.45	m	19.0 – 18.2		Direct coupling
1 (B)			135.1 or 134.9		
2&6 (B)	-		139.5 – 138.5 and/or 137.0 – 136.5	m, m	
3 (B)	7.01 (2H)	br s	129.9		2-CH_3(B), 4-CH_3(B)
4 (B)	-		138.4		4-CH_3(B)
5 (B)	6.92	s	127.1		4-CH_3(B)
2-Me (B)	2.76 & 2.37	br s	20.0		Direct coupling; C-1(B)
4-Me (B)	2.31	s	21.2		Direct coupling, C-4(B)
6-Me (B)	2.64 – 2.45	m	20.0		Direct coupling
1 (C)			151.2		
2 (C)	9.05 – 8.88	m	132.5 – 131.1	m	C-4(C)
3 (C)	7.15 – 7.05 [a]	m	126.5		
4 (C)	7.38 – 7.33[a]	m	128.4		C-3(C), C-5(C), C-6(C) and DEPT
5 (C)	7.15 – 7.05 [a]	m	130.1		
6 (C)	7.15 – 7.05 [a]	m	129.5		
1 (D)	2.22 – 2.14	m	29.4 – 28.7	m	
2,	1.95 – 0.6	Various m	35.2	d, 40.1	
3,			31.4	d, 16.4	
5,			29.0	br d	
6(D)			27.7	d, 10.0	
			26.8	d, 11.6	
			26.3	d, 3.1	
4D			26.1	br s	
1 (E)	-		154.7		
2,6 (E)	6.84 – 6.69	m	115.4		C-1(E)
3,5 (E)	6.97	d, 8.0	129.8, 128.4		C-1(E)
	6.99 – 6.93	m			
4 (E)	-		129.5 - 130.1		DEPT
4-Me (E)	2.30 – 2.24	m	20.5 & 18.3 – 17.2	s and m	C-3(E), C-5(E), C-4(E)
1 (E’)			137.1		
2 (E’)	7.52	d, 7.4	126.5		
3 (E’)	7.15 – 7.05 [a]	m	127.1		C-1(E’), C-2(E’)
4 (E’)			128.7		4-CH_3(E’) (and DEPT)
5 (E’)	7.38 – 7.33[a]	m	127.9		C-1(E’), C-4(E’)
6 (E’)	7.28 – 7.23[b]	m	127.6		C-2(E’)
4-Me (E’)	2.30 – 2.24	m	20.5 & 18.3 – 17.2	s and m	

[a] Multiple peaks overlapping;

[b] Overlaps with $CHCl_3$. A: mesityl ring *pi*-stacked with the benzylidene ring; B: second mesityl ring; C: benzylidene aromatic ring; D: cyclohexyl rings; E and E’: *p*-cresolate moieties; 1′: benzylidene; 2, 4 and 5: *N*-heterocyclic carbene ring

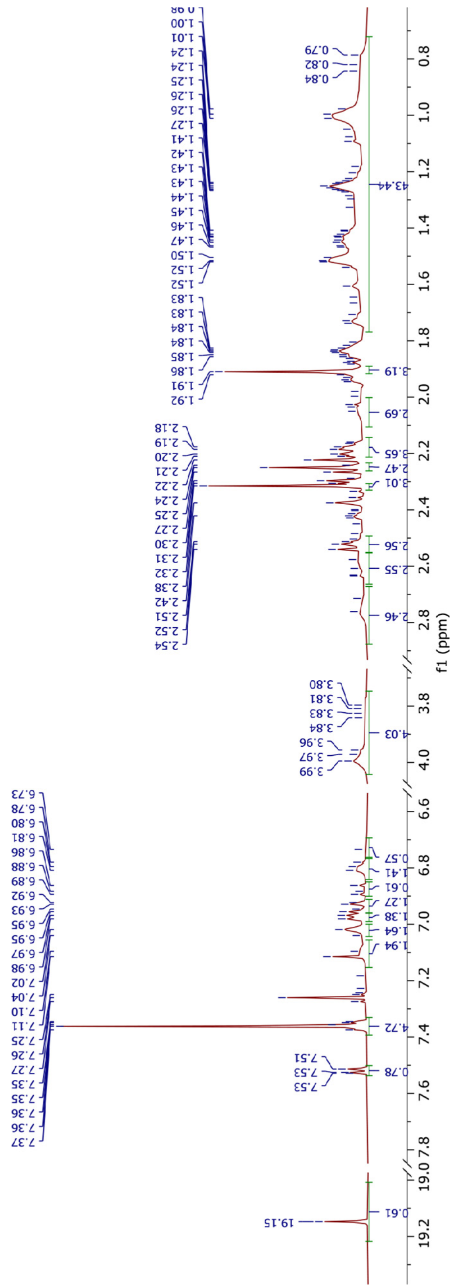


Fig. 2. The ^{1}H NMR spectra of **3** measured in $CDCl_3$.

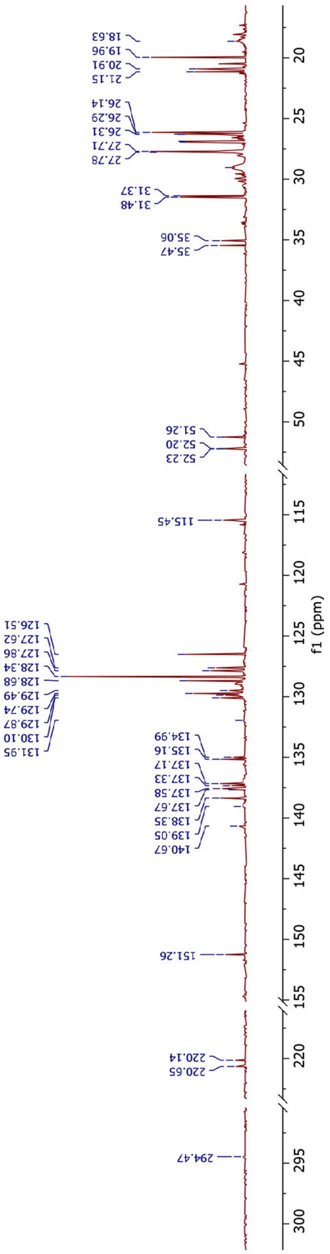


Fig. 3. The ^{13}C NMR spectra of **3** measured in $CDCl_3$.

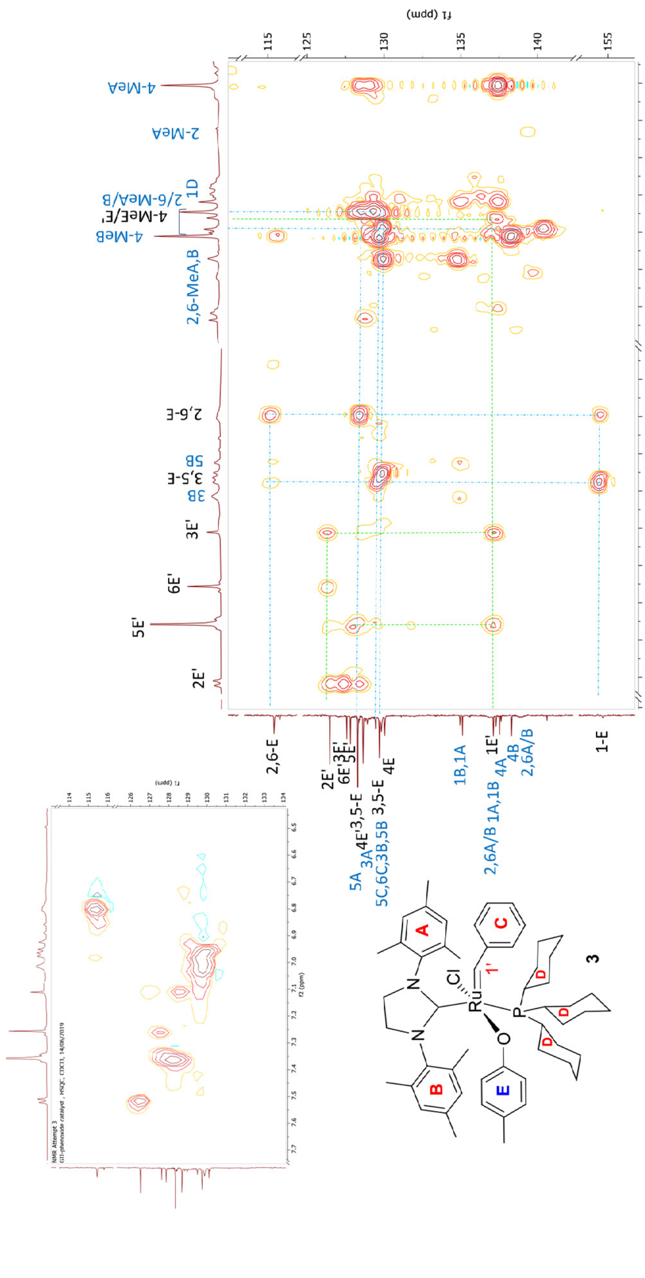


Fig. 4. HSQC (insert) and HMBC spectra of complex (**3**) measured in $CDCl_3$.

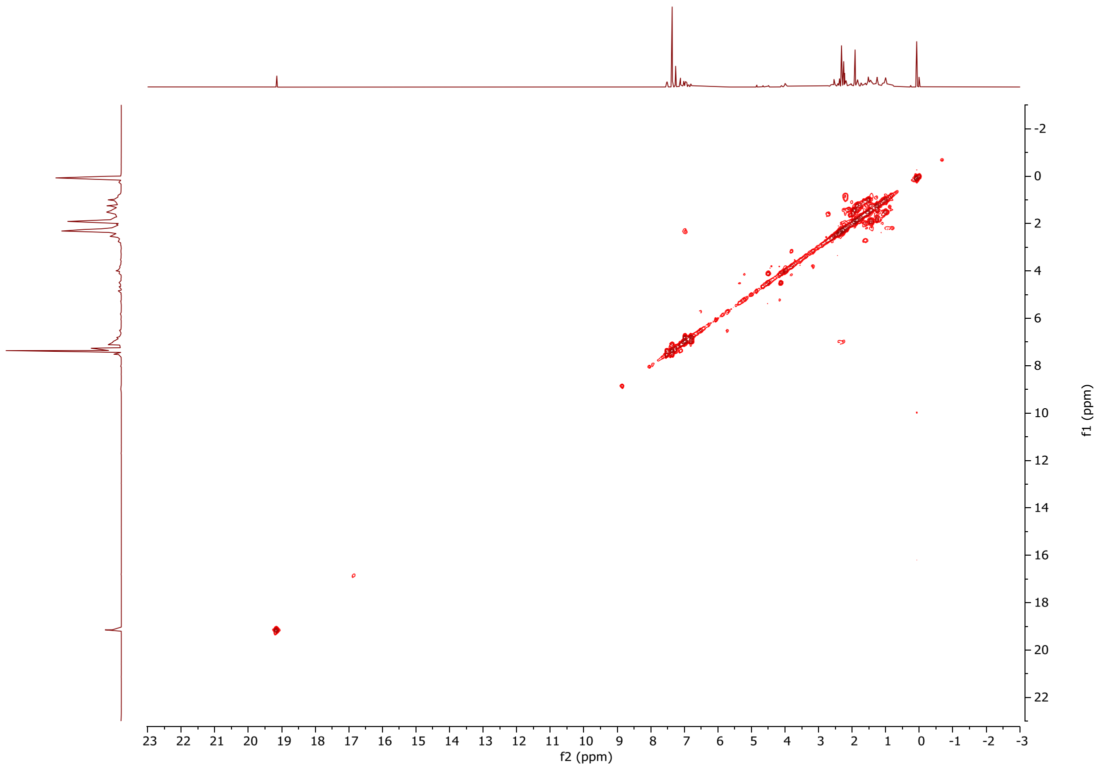


Fig. 5. COSY spectra of complex (**3**) measured in $CDCl_3$.

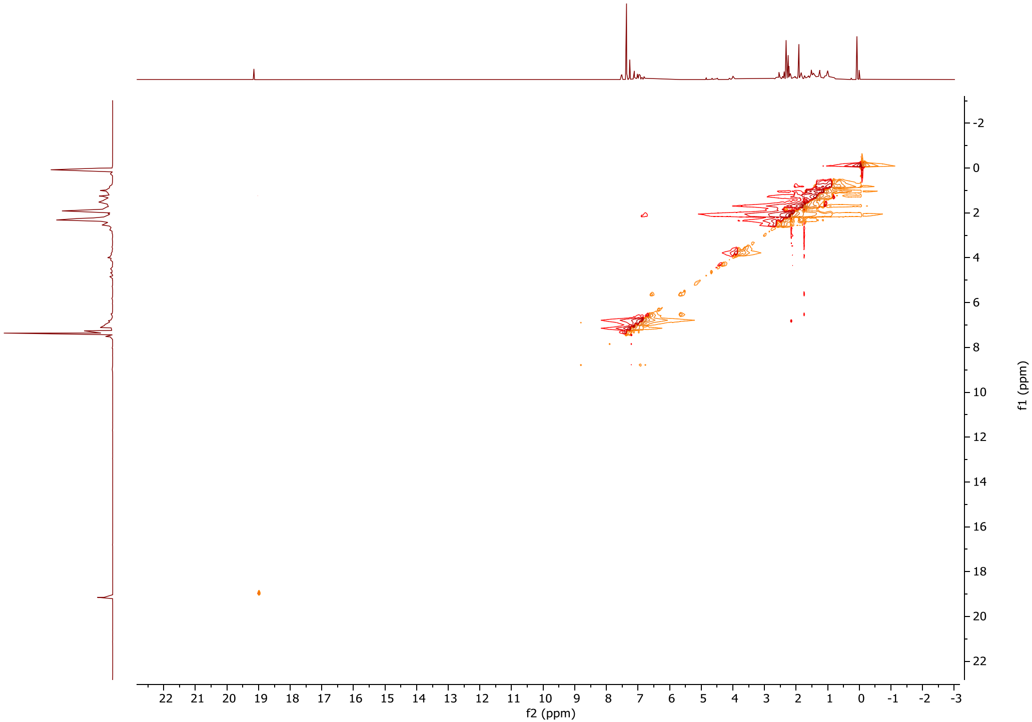


Fig. 6. NOE spectra of complex (**3**) measured in $CDCl_3$.

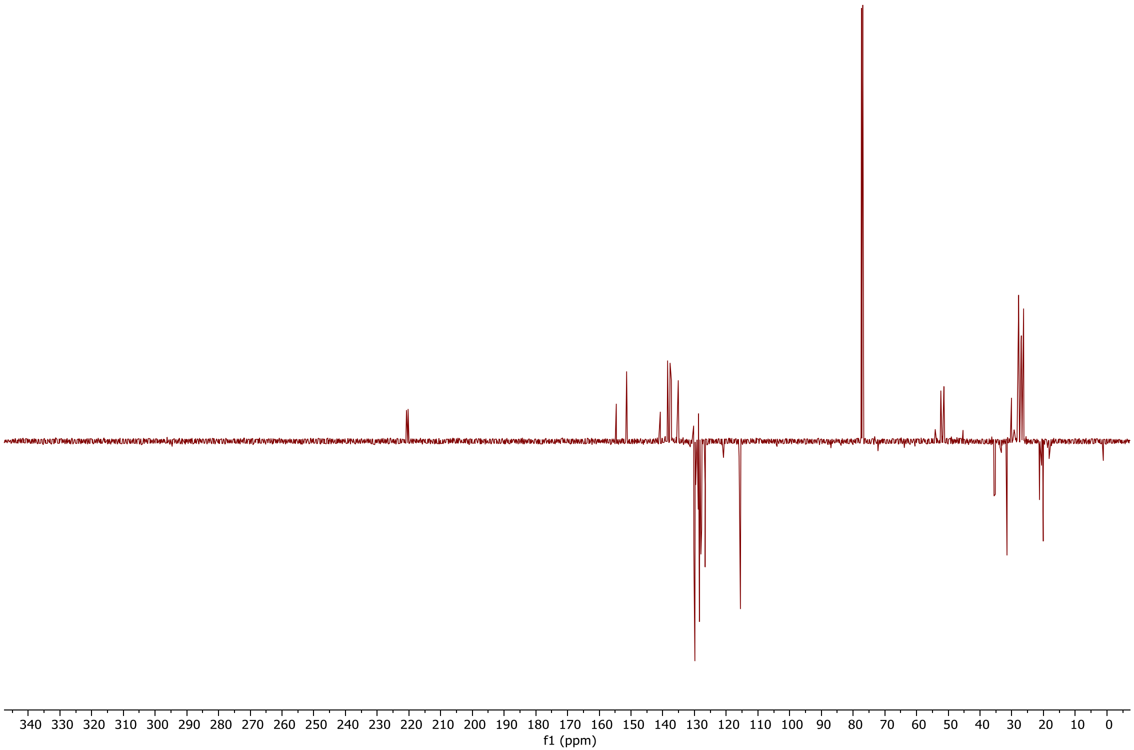


Fig. 7. DEPT spectra of complex (**3**) measured in $CDCl_3$.

(COSY), Nuclear Overhauser effect (NOE) and Distortionless enhancement by polarization transfer (DEPTH) spectra of **3** measured in $CDCl_3$. The raw data obtained from NMR instrument (^1H, ^{13}C, HSQC, HMBC, COSY, NOE and DEPT) was in the form of FID files which are difficult to understand without plotting. The raw data in the form of FID files were plotted using BRUKER TOPSPIN software which is presented in the form of images, shown in Figs. 2-7. The raw NMR data are shared as supplemental files in the form of FID files and in Microsoft Excel Worksheet format.

2. Experimental Design, Materials and Methods

Materials and methods to prepare the Grubbs 2nd generation derivative **3**, which allowed the data to be presented here are describes in Ref [1]. In this article only the protocol used to record the NMR and UV-Vis data are provided.

2.1. Spectroscopic measurements

After removal of the solvent from the reaction mixture (in which **3** were prepared) under vacuo. The residue was dissolved in $CDCl_3$ (0.6 mL) for NMR spectral analysis. ^1H and ^{13}C NMR measurements were recorded on a Bruker AVANCE II 600 FT NMR spectrometer at 278.15 K. The chemical shifts are reported relative to $SiMe_4$ at 0.00 ppm for ^1H and ^{13}C. The ^1H NMR spectra were recorded at 600.26 MHz and ^{13}C NMR spectra at 150.95 MHz. HMBC and HSQC was used to assign the NMR signals. 8 Scan (TD1 = 512, TD2 = 2048) were recorded for both HMBC and HSQC with a relaxation time delay of 1.5 s. The acquisition time for the HSQC was 0.0655 s for the ^1H and 0.00486 s for the ^{13}C. The spectral width of the HSQC for the ^1H is 26.0 ppm / 15 625 Hz and 348.63 ppm / 52 631 Hz for ^{13}C. The acquisition time for the HMBC was 0.131 s for the ^1H and 0.00486 s for the ^{13}C. The spectral width of the HMBC for the ^1H is 26.0 ppm / 15 625 Hz and 348.63 ppm / 52 631 Hz for ^{13}C. For the COSY, NOE and DEPTH NMR a relaxation time delay of 1.5 s was used. The acquisition time for the COSY was 0.0655 and 0.033 s for the ^1H. The spectral width of the COSY for the ^1H is 26.0 ppm / 15 625 Hz and 25.9 ppm / 15607 Hz. The acquisition time for the NOE was 0.131 and 0.016 s for the ^1H. The spectral width of the NOE for the ^1H is 26.0 ppm / 15 625 Hz and 25.9 ppm / 15607 Hz. The acquisition time for the DEPT was 0.612 s for the ^{13}C. The spectral width of the DEPT for the ^{13}C is 354.85 ppm / 53571 Hz.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Supplementary Materials

Supplementary material associated with this article can be found in the online version at doi:10.1016/j.dib.2020.106634.

References

[1] M.R. Swart, Barend C.B. Bezuidenhoudt, C. Marais, E. Erasmus, Spectroscopic characterisation of Grubbs 2nd generation catalyst and its *p*-cresol derivatives, Inorganica Chimica Acta 514 (2021) 120001 doi.org/, doi:10.1016/j.ica.2020.120001.
[2] M.R. Swart, L. Twigge, C. Marais, B.C.B. Bezuidenhoudt, Polyhedron 152 (2018) 31–36.
[3] M.M. Gallagher, A.D. Rooney, J.J. Rooney, J. Organomet. Chem. 693 (2008) 1252–1260.