Supplementary Information

New Allyldithiocarbimates: Synthesis, Structure and Antifungal Activity against *Phakopsora pachyrhizi* and *Hemileia vastatrix*

Antonio E. C. Vidigal,[a] Mayura M. M. Rubinger, *,[a] Lucas F. da Silva,[b] Laércio Zambolim,[b] Arthur B. D. Pereira,[a] Silvana Guilardi,[c] Rafael A. C. Souza[c] and Javier Ellena[d]

[a]Departamento de Química, Universidade Federal de Viçosa, 36570-977 Viçosa-MG, Brazil

[b]Departamento de Fitopatologia, Universidade Federal de Viçosa, 36570-977 Viçosa-MG, Brazil

[c]Instituto de Química, Universidade Federal de Uberlândia, 38400-902 Uberlândia-MG, Brazil

[d]Instituto de Física de São Carlos, Universidade de São Paulo, 13566-590 São Carlos-SP, Brazil

We include here (Figures S1-S36) the infrared and NMR spectra for the allyldithiocarbimate salts **1a-d**, **2a-d** and **3a-d**.

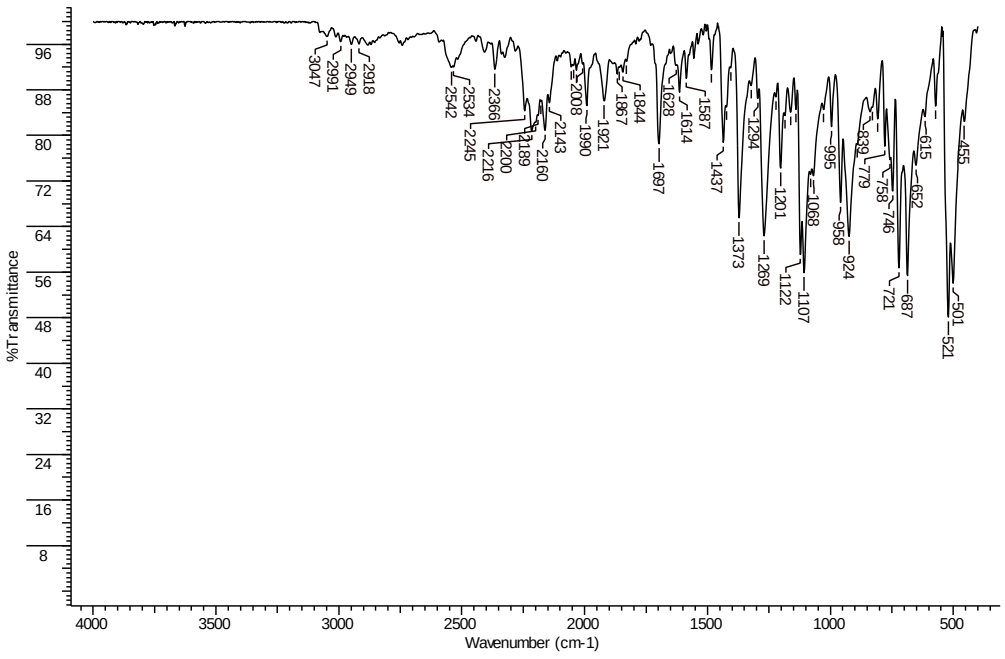

Figure S1. FTIR-ATR spectrum of compound **1a**.

*e-mail: mayura@ufv.br

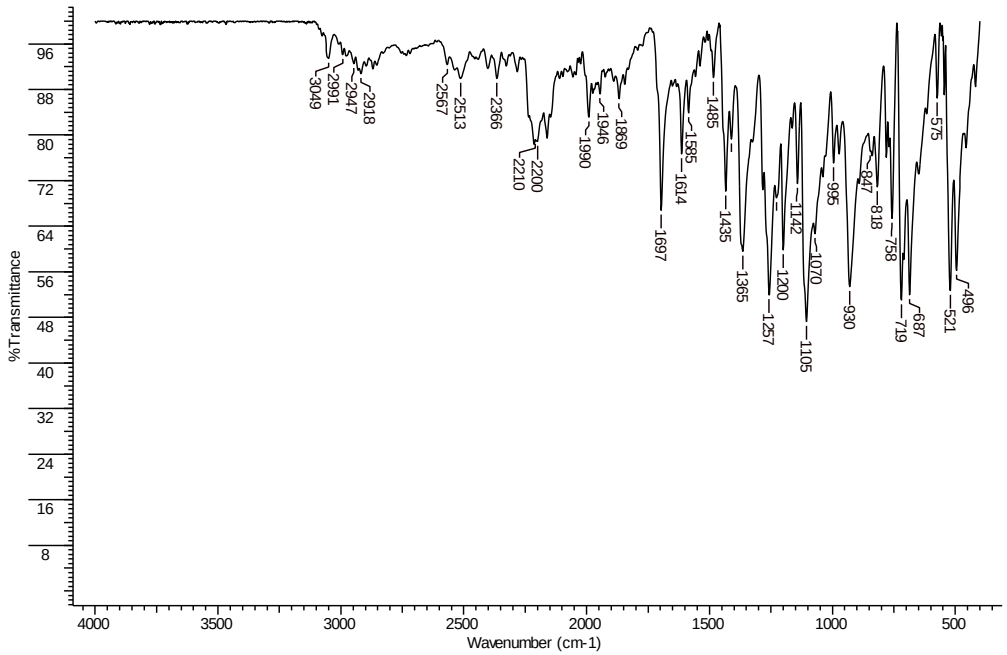

Figure S2. FTIR-ATR spectrum of compound **1b**.

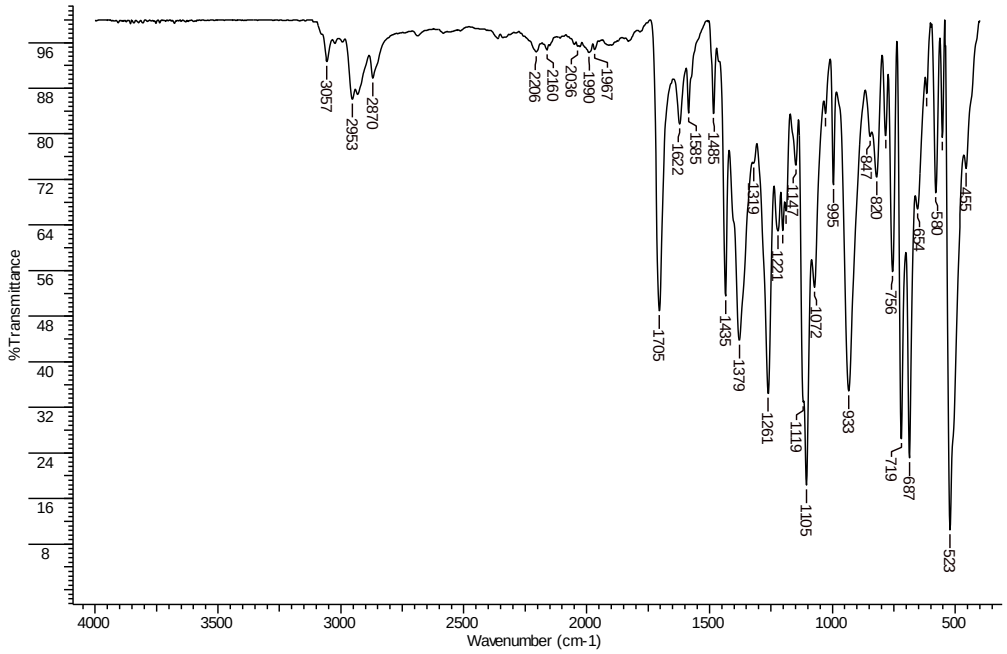

Figure S3. FTIR-ATR spectrum of compound **1c**.

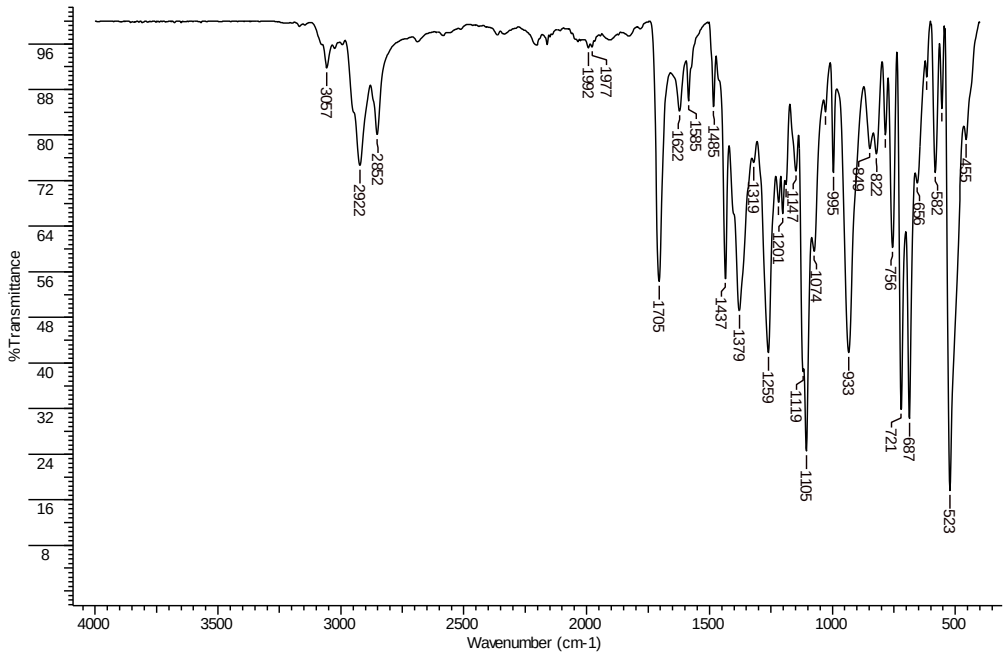

Figure S4. FTIR-ATR spectrum of compound **1d**.

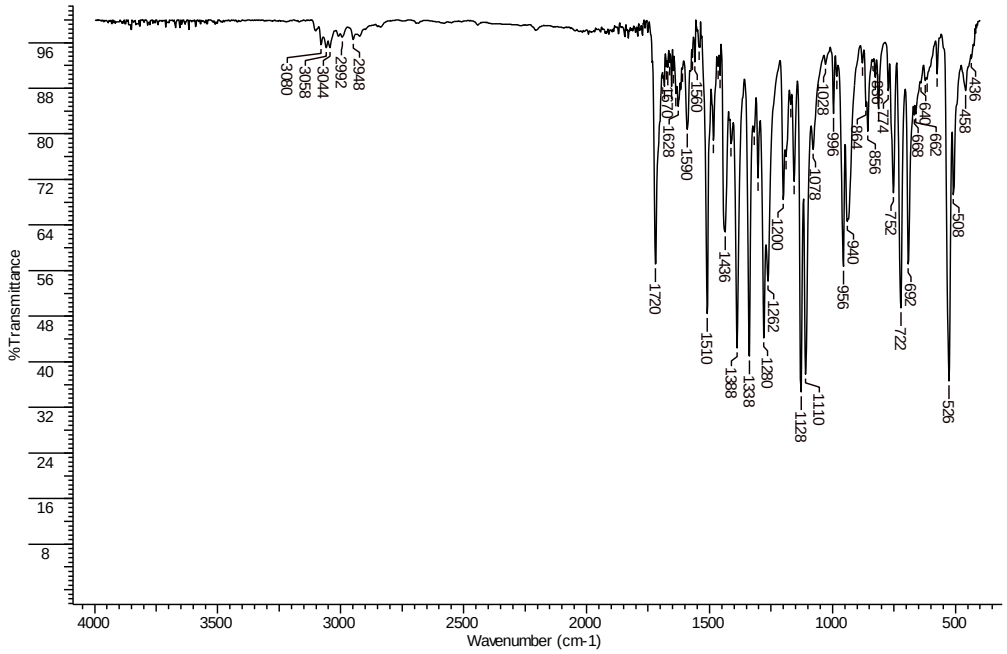

Figure S5. FTIR-ATR spectrum of compound **2a**.

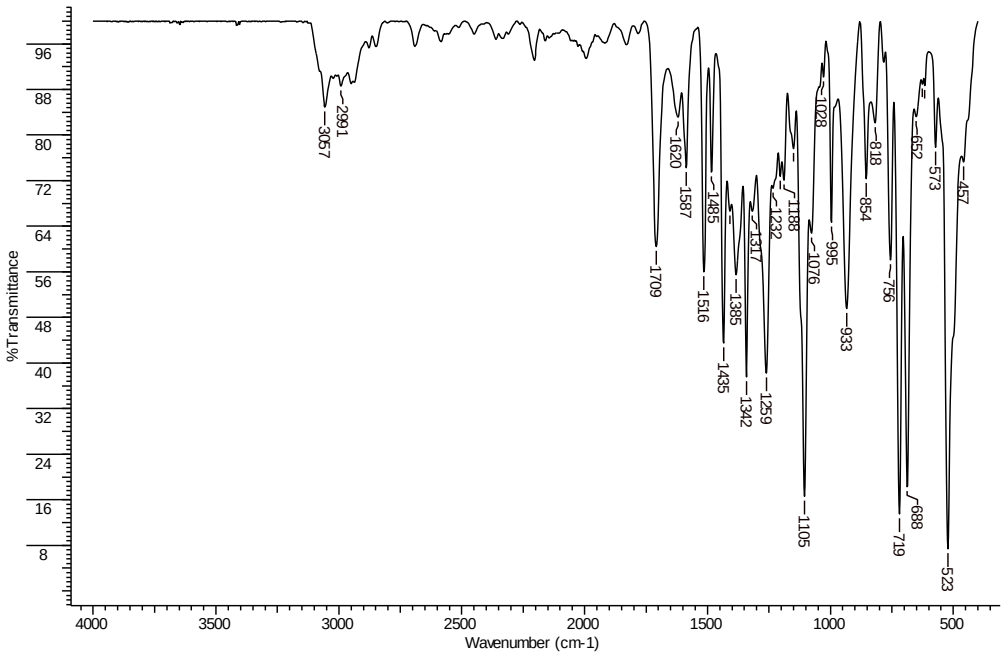

Figure S6. FTIR-ATR spectrum of compound **2b**.

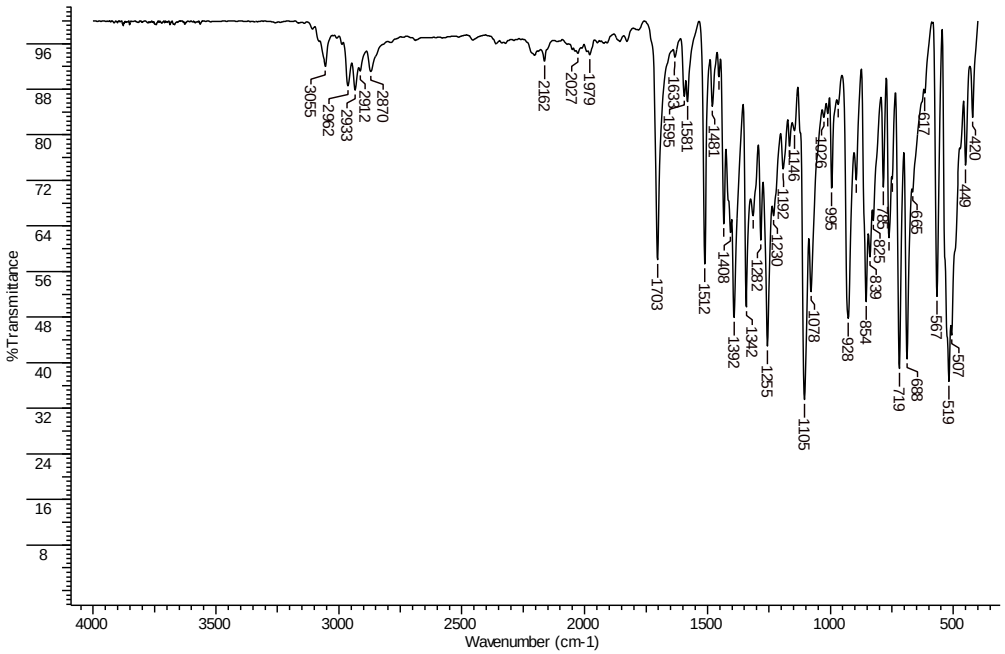

Figure S7. FTIR-ATR spectrum of compound **2c**.

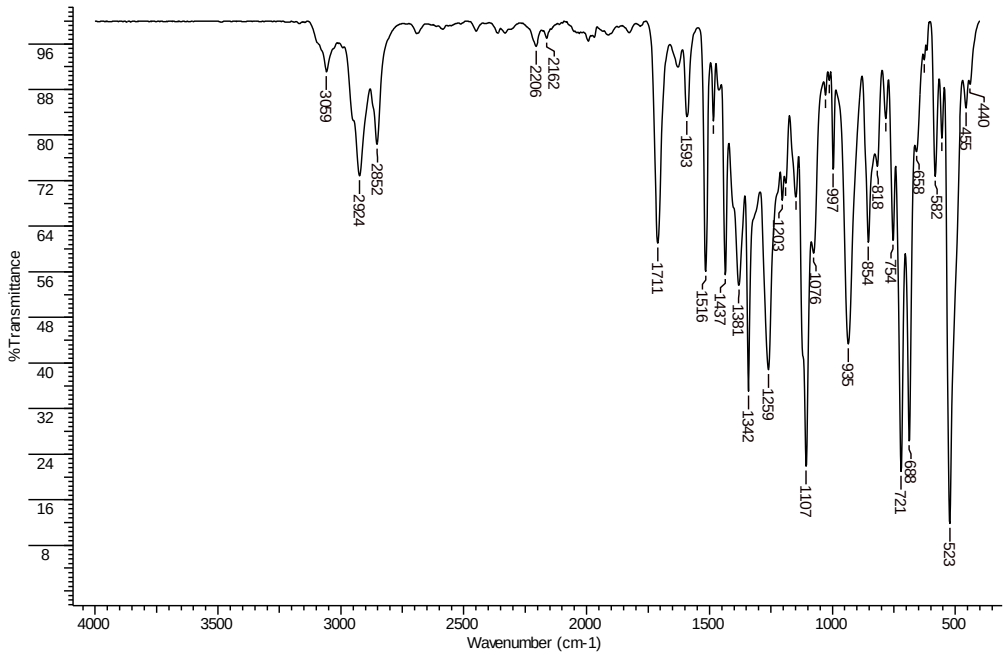

Figure S8. FTIR-ATR spectrum of compound **2d**.

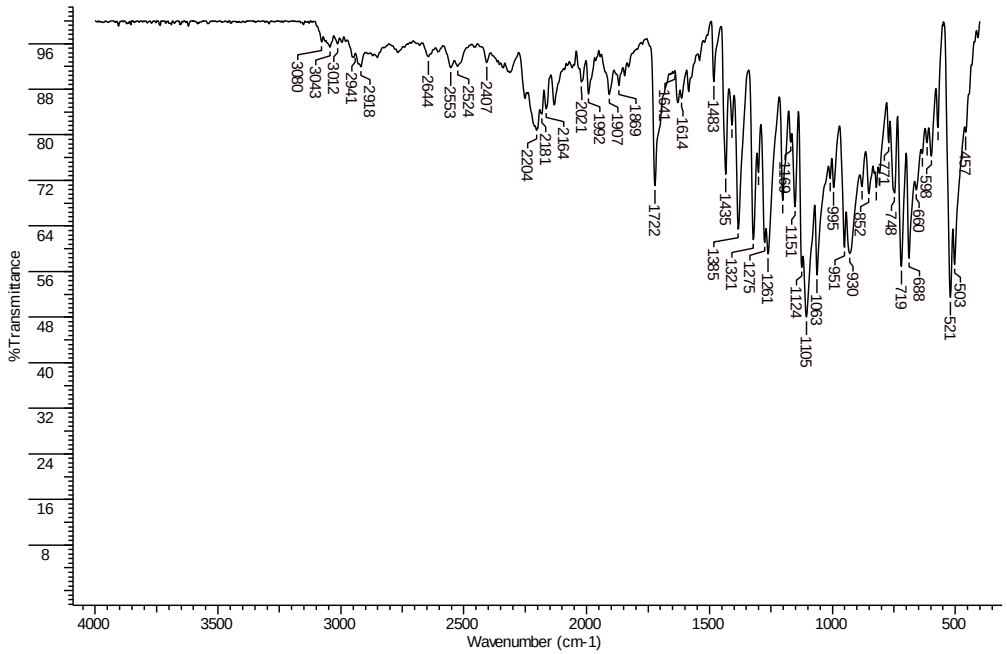

Figure S9. FTIR-ATR spectrum of compound **3a**.

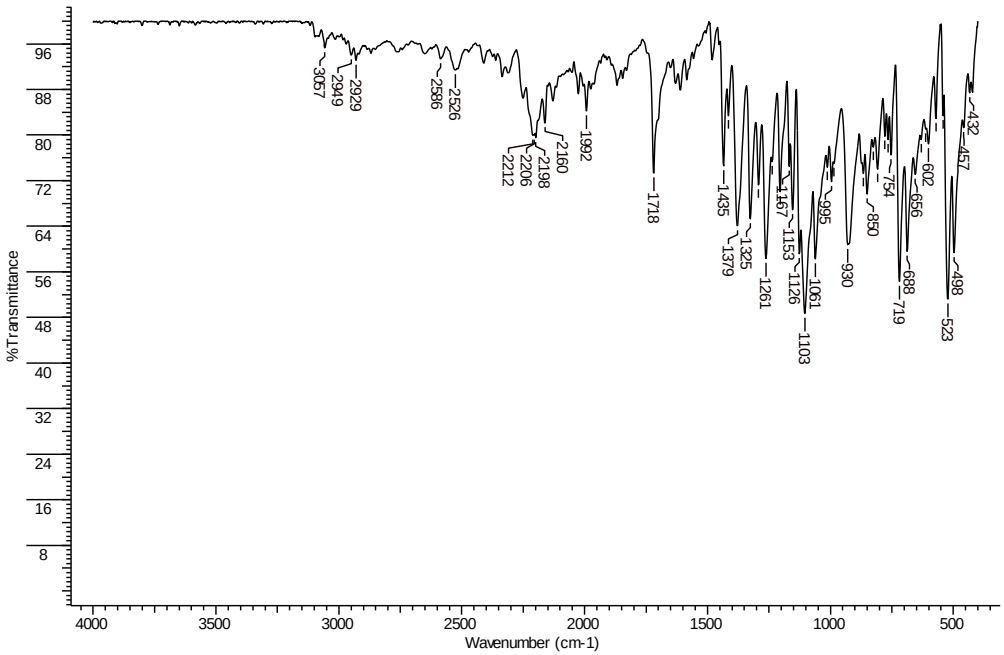

Figure S10. FTIR-ATR spectrum of compound **3b**.

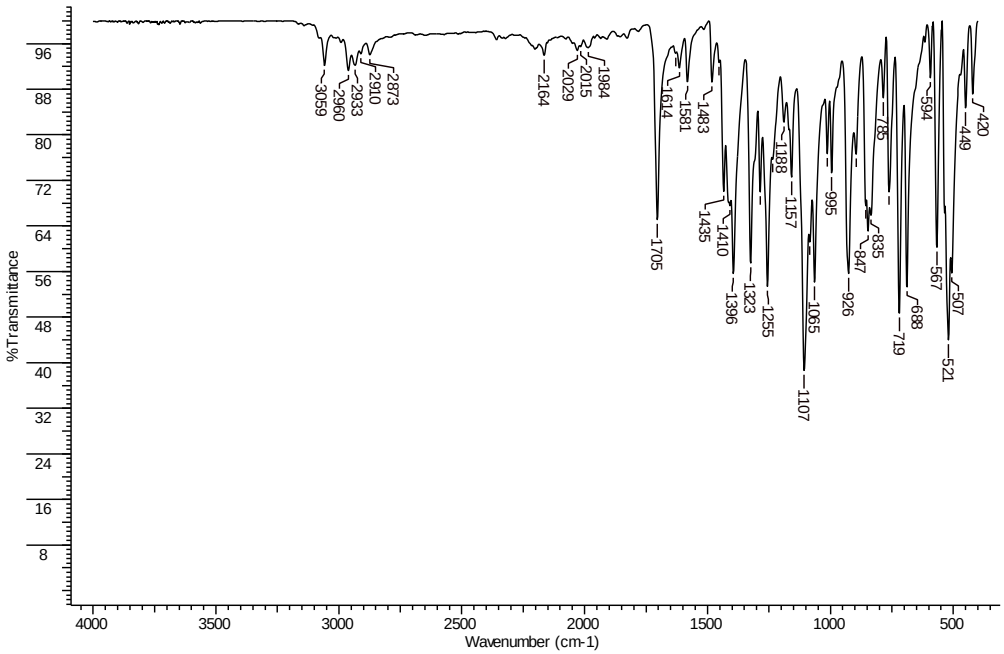

Figure S11. FTIR-ATR spectrum of compound **3c**.

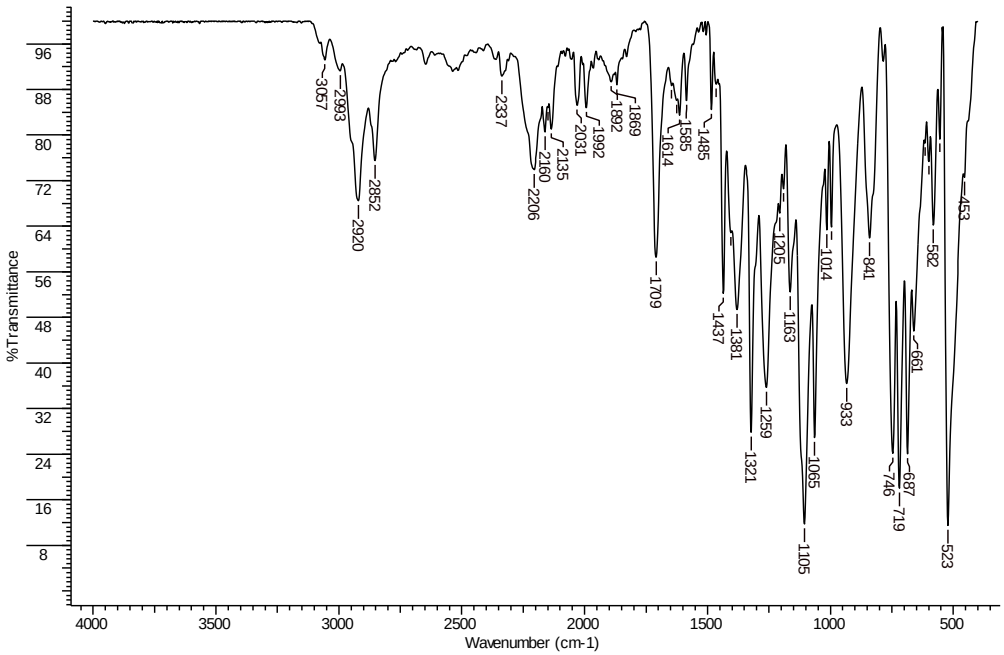

Figure S12. FTIR-ATR spectrum of compound **3d**.

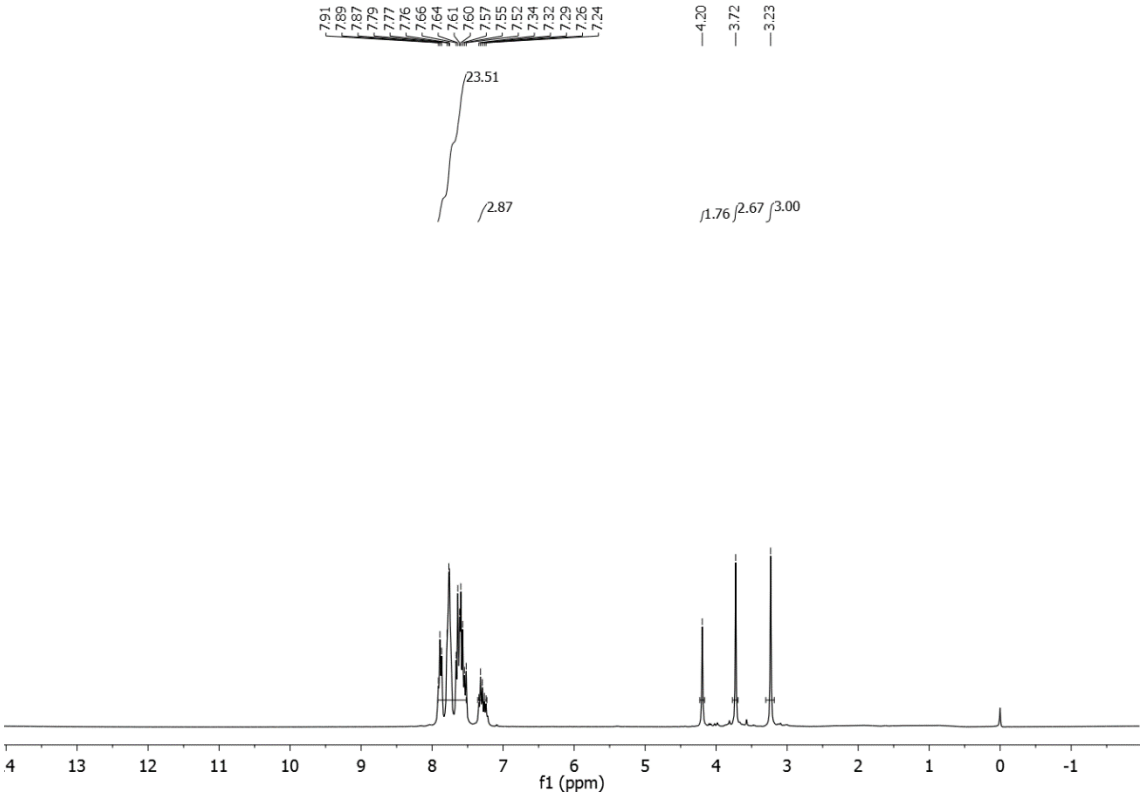

Figure S13. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **1a**.

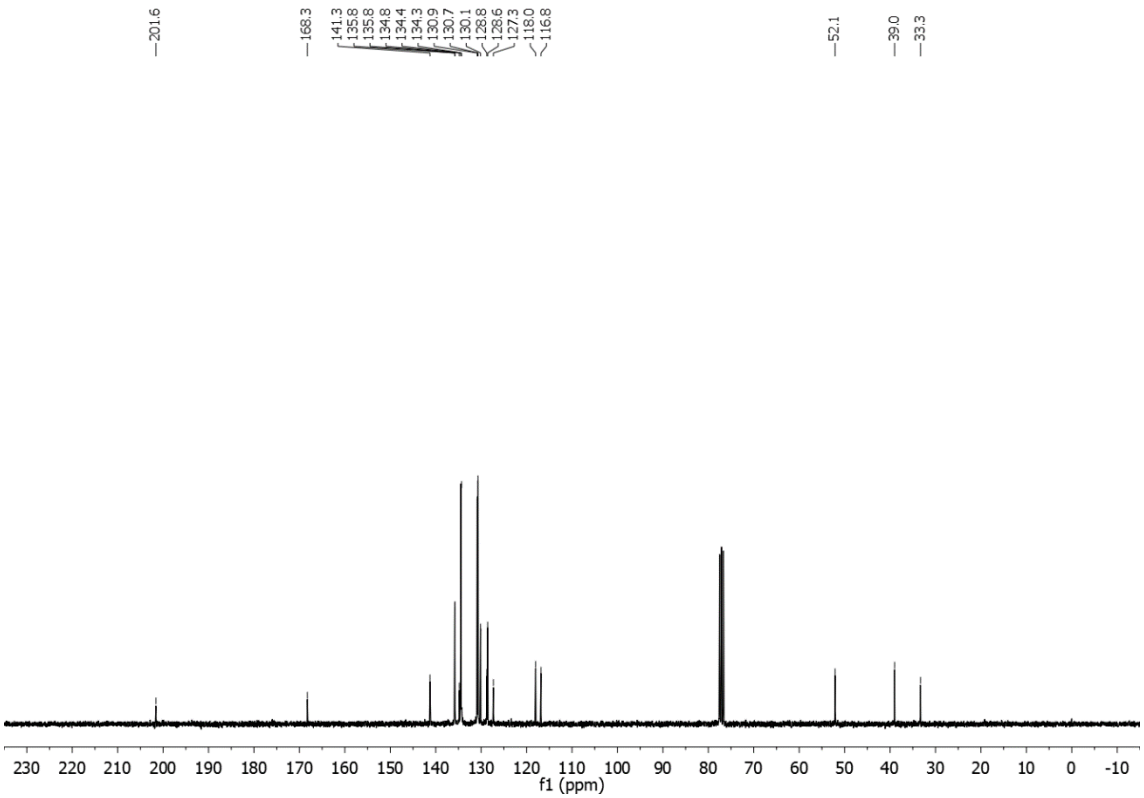

Figure S14. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **1a**.

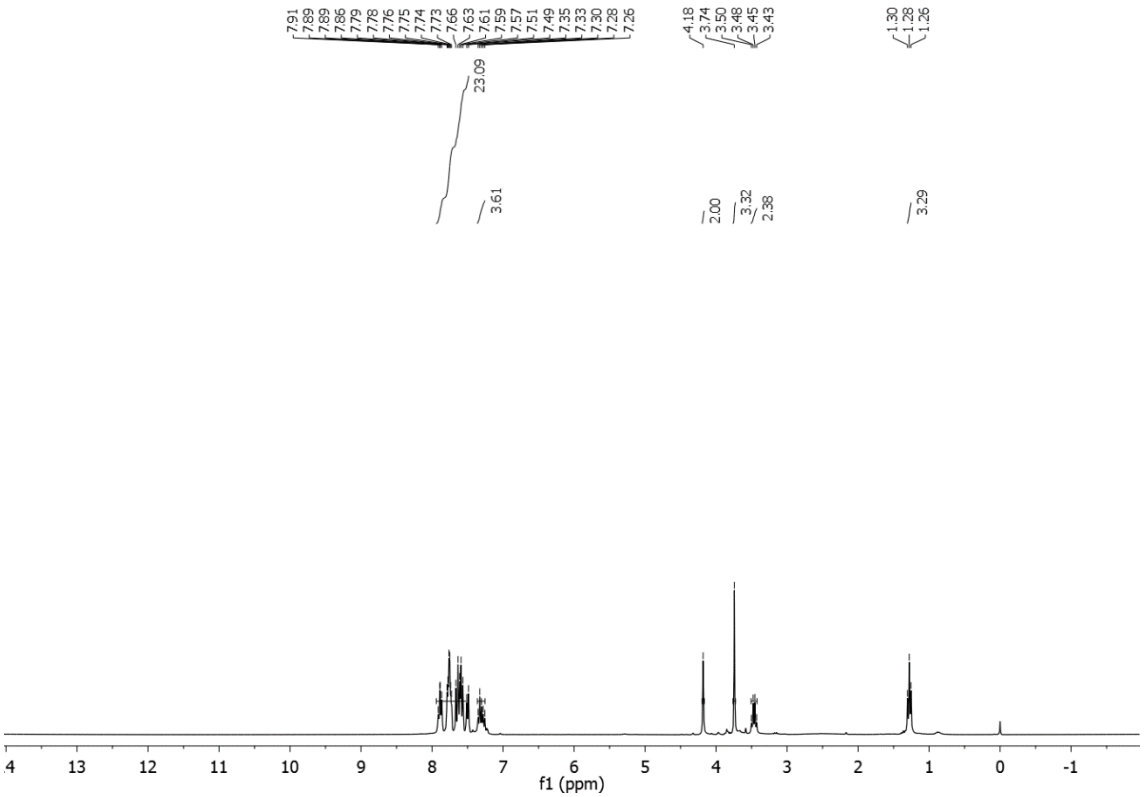

Figure S15. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **1b**.

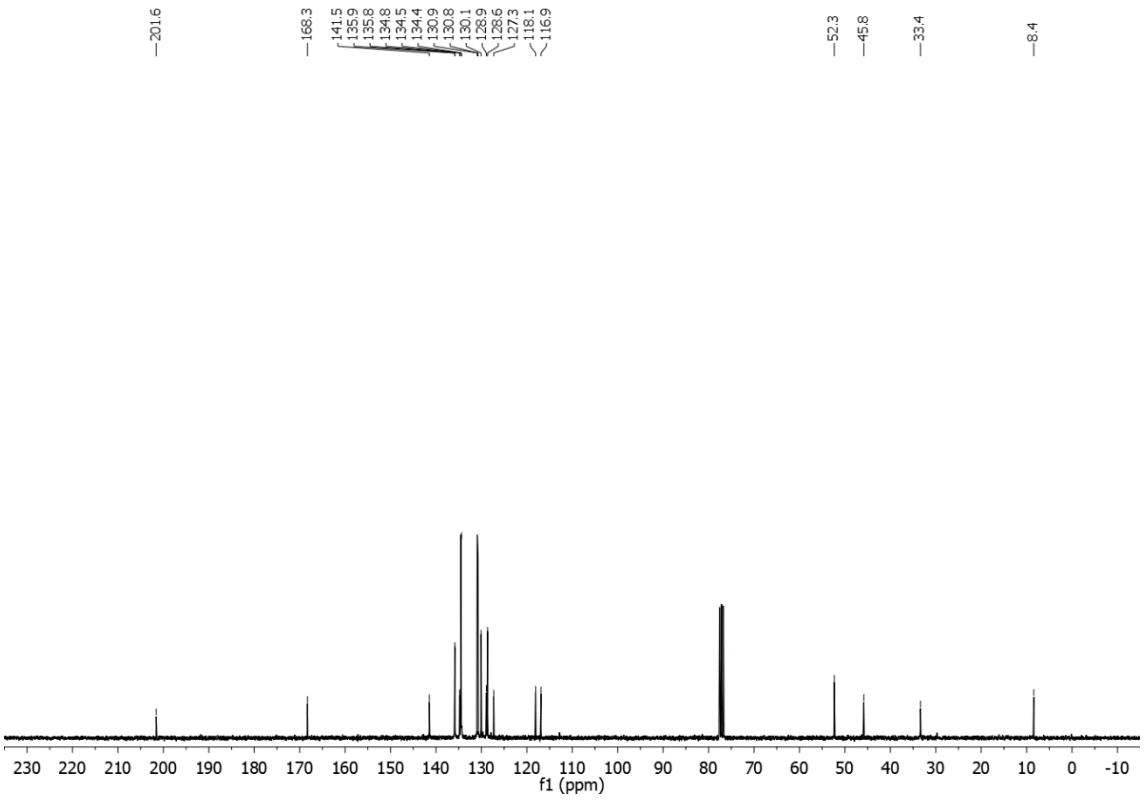

Figure S16. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **1b**.

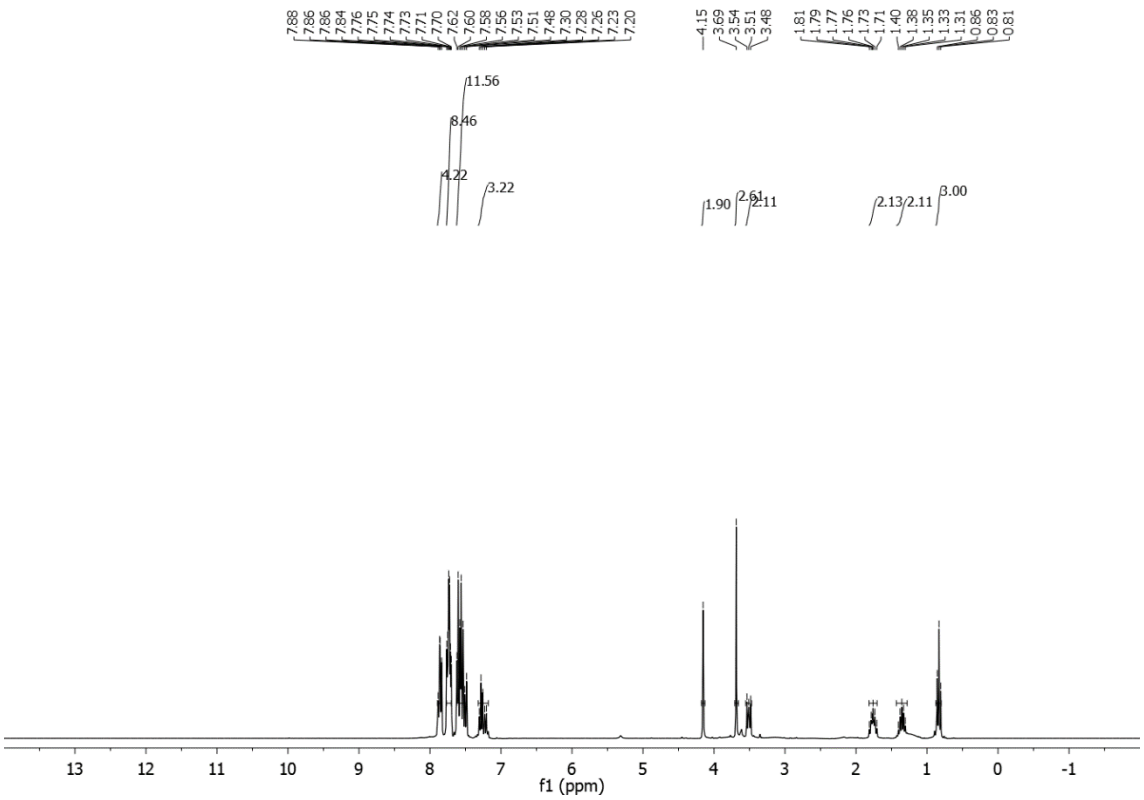

Figure S17. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **1c**.

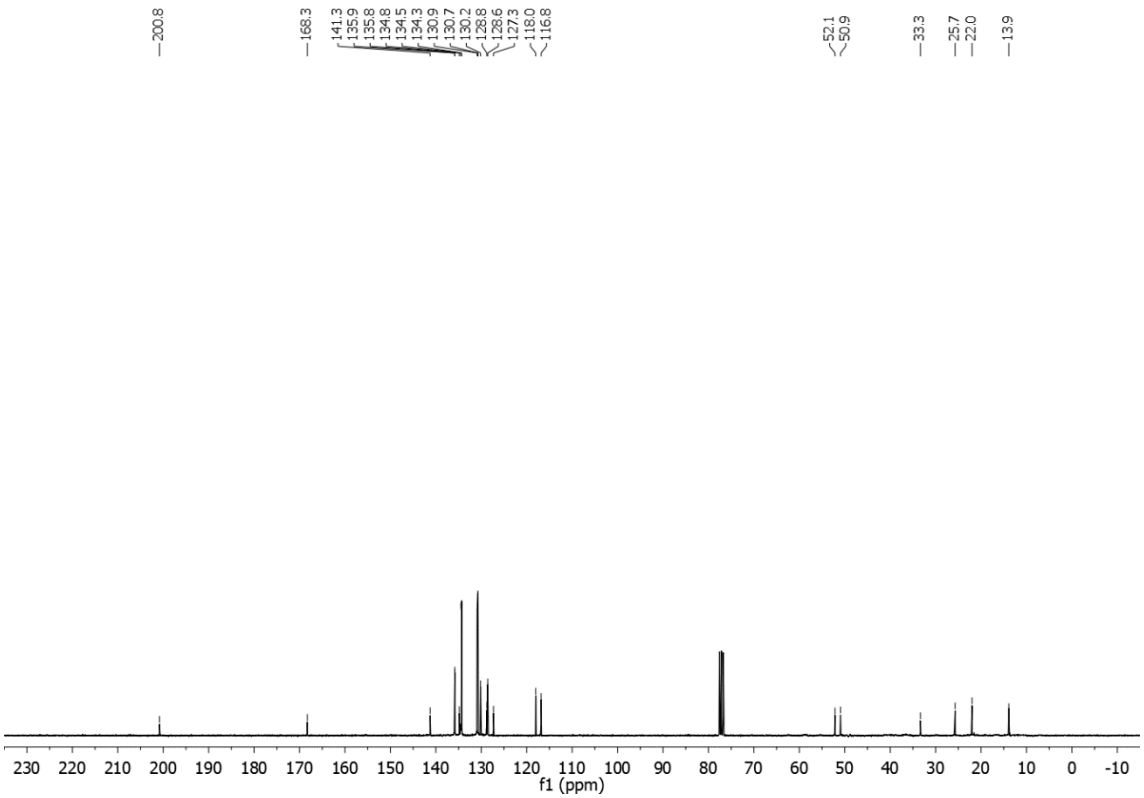

Figure S18. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **1c**.

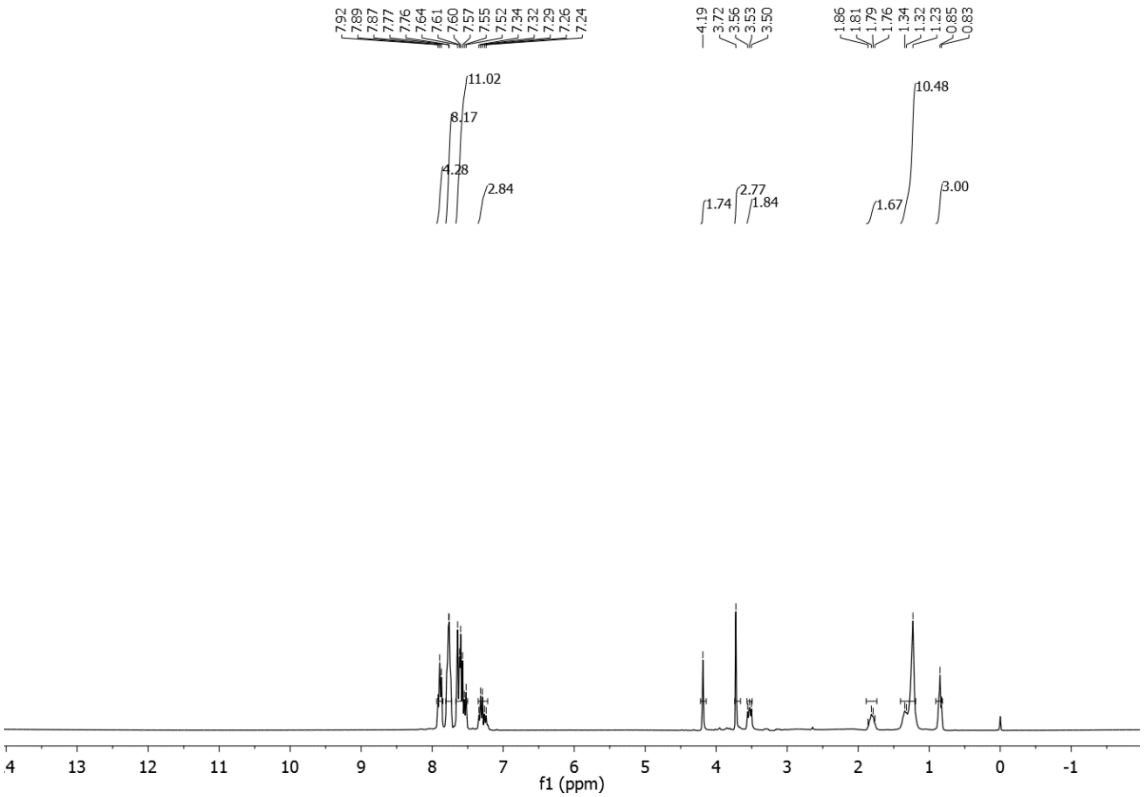

Figure S19. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **1d**.

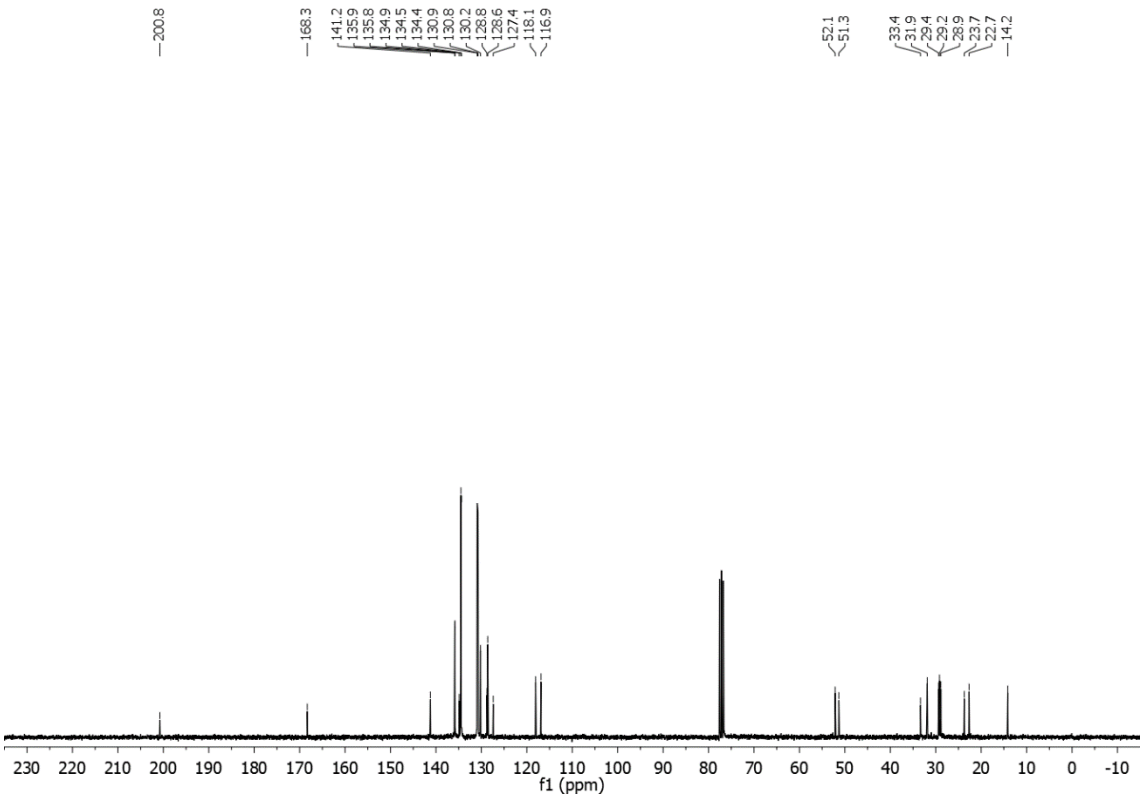

Figure S20. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **1d**.

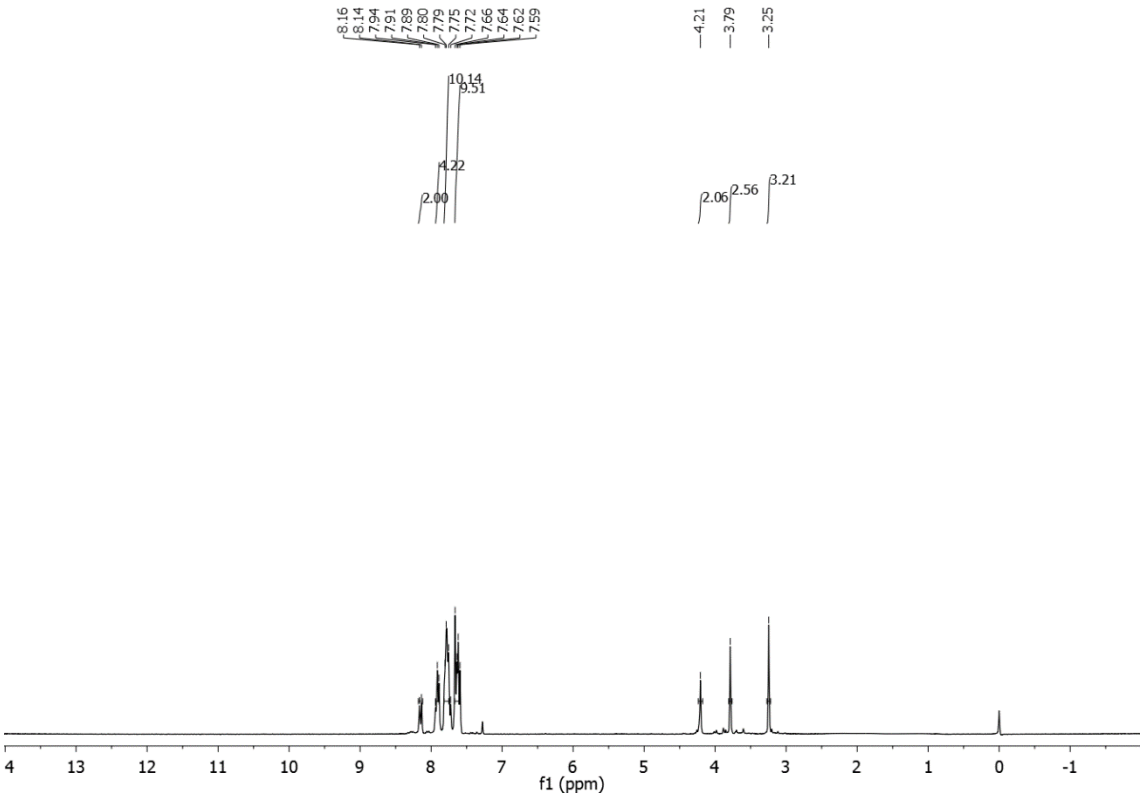

Figure S21. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **2a**.

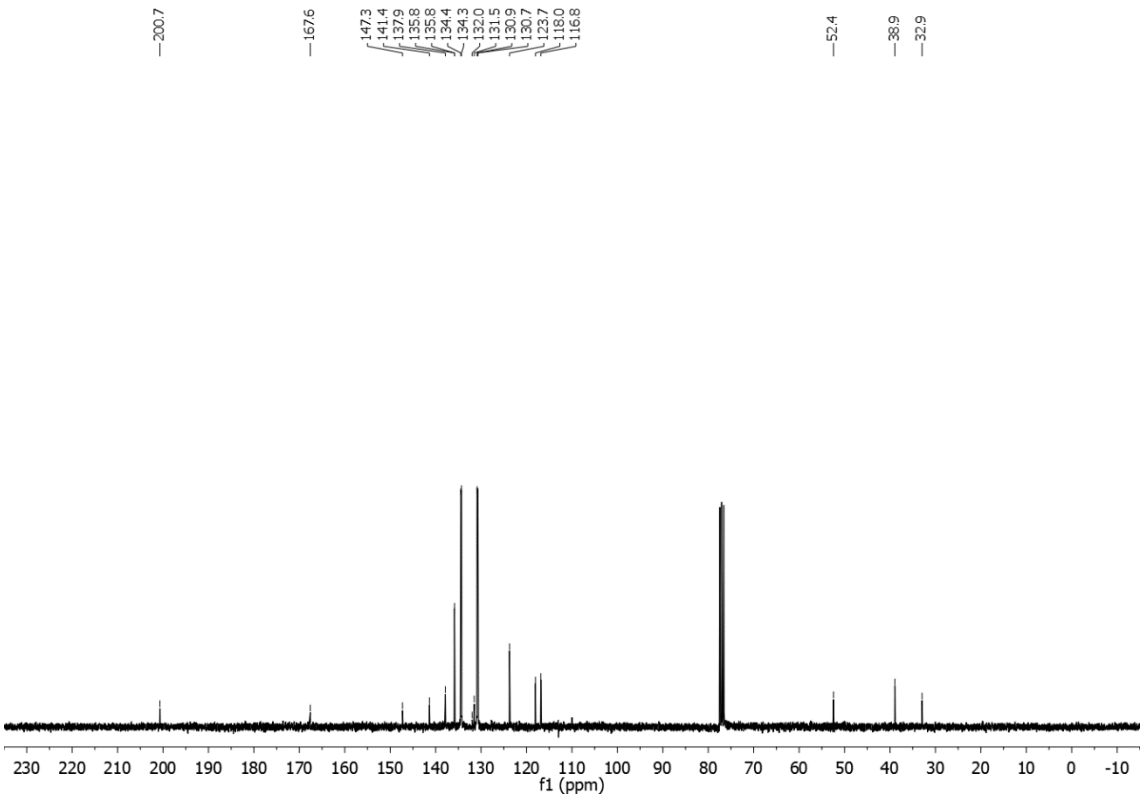

Figure S22. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **2a**.

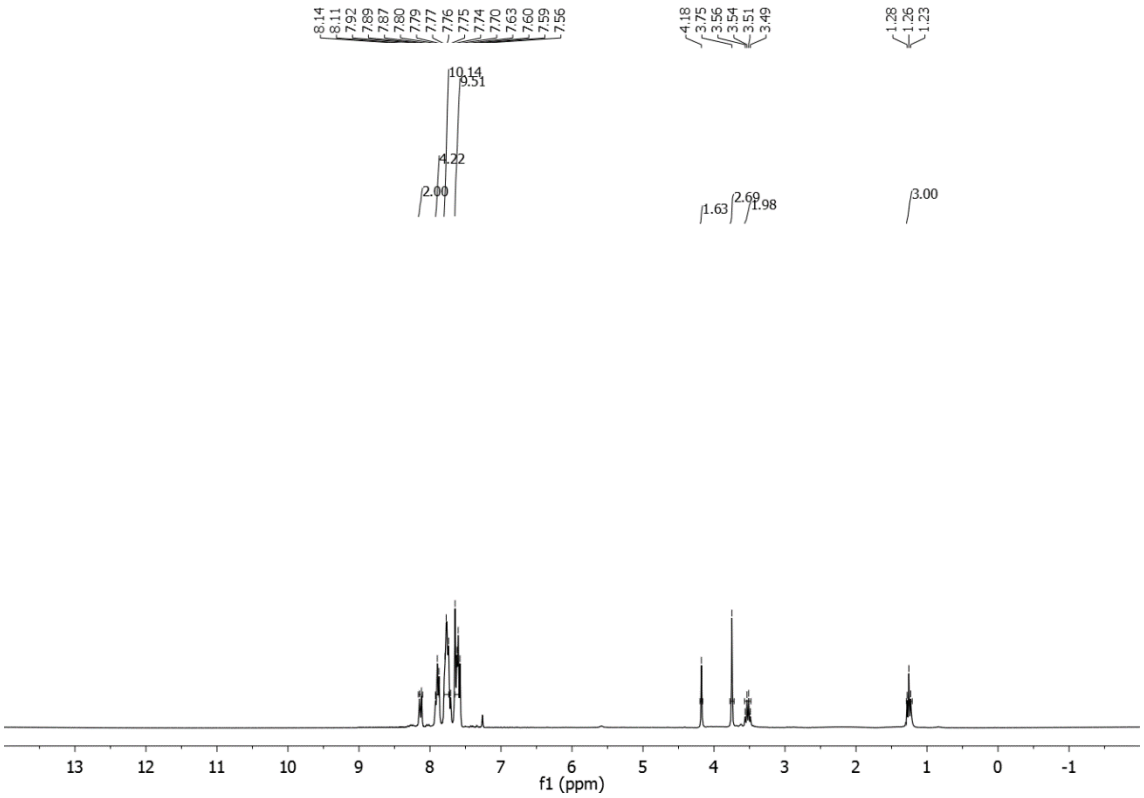

Figure S23. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **2b**.

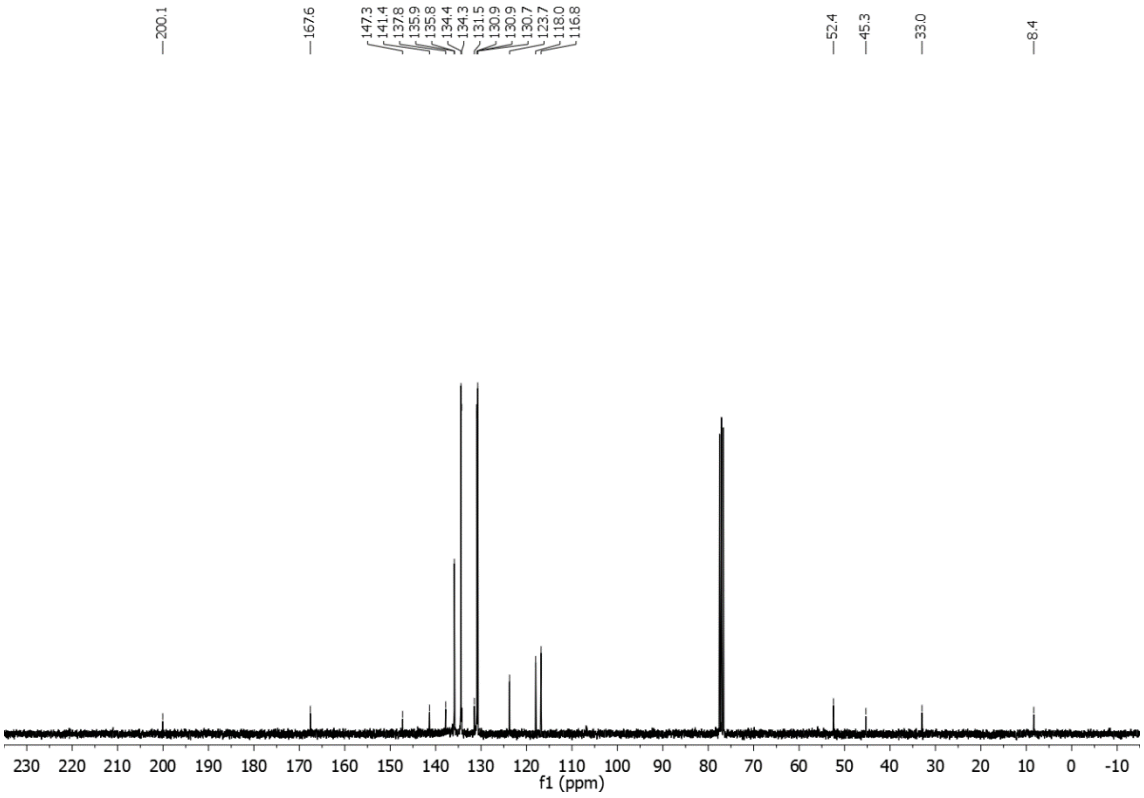

Figure S24. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **2b**.

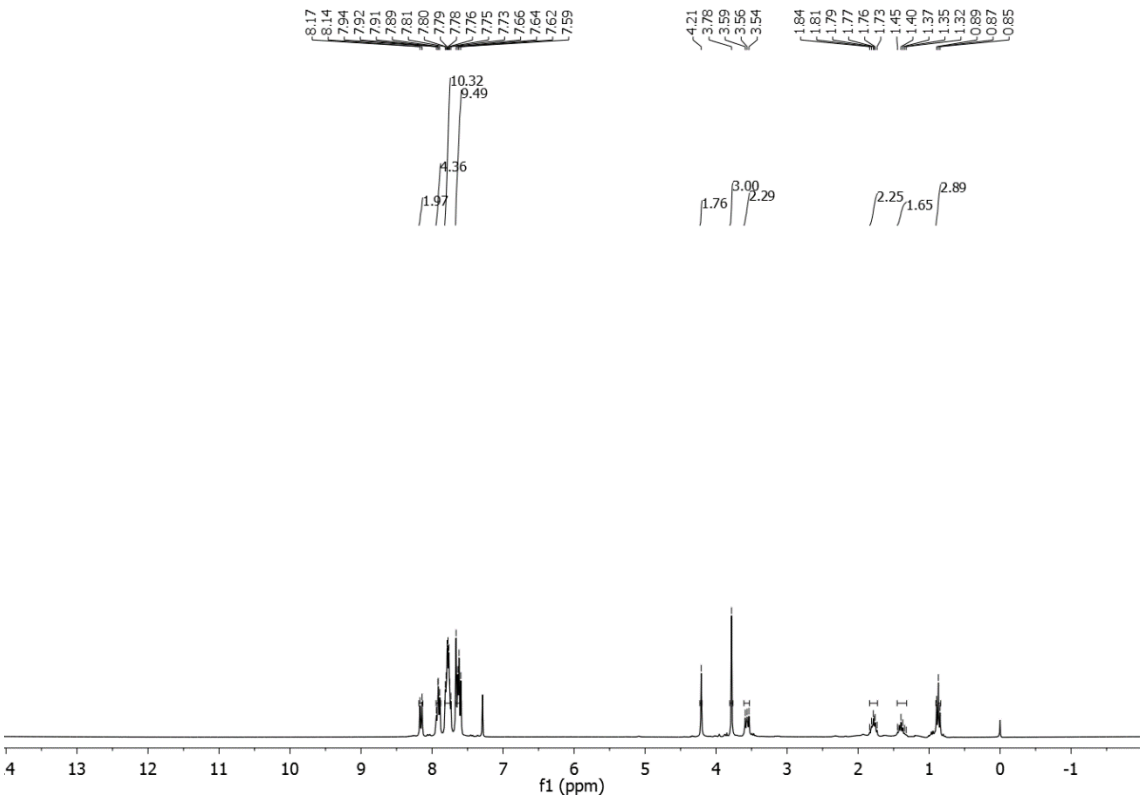

Figure S25. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **2c**.

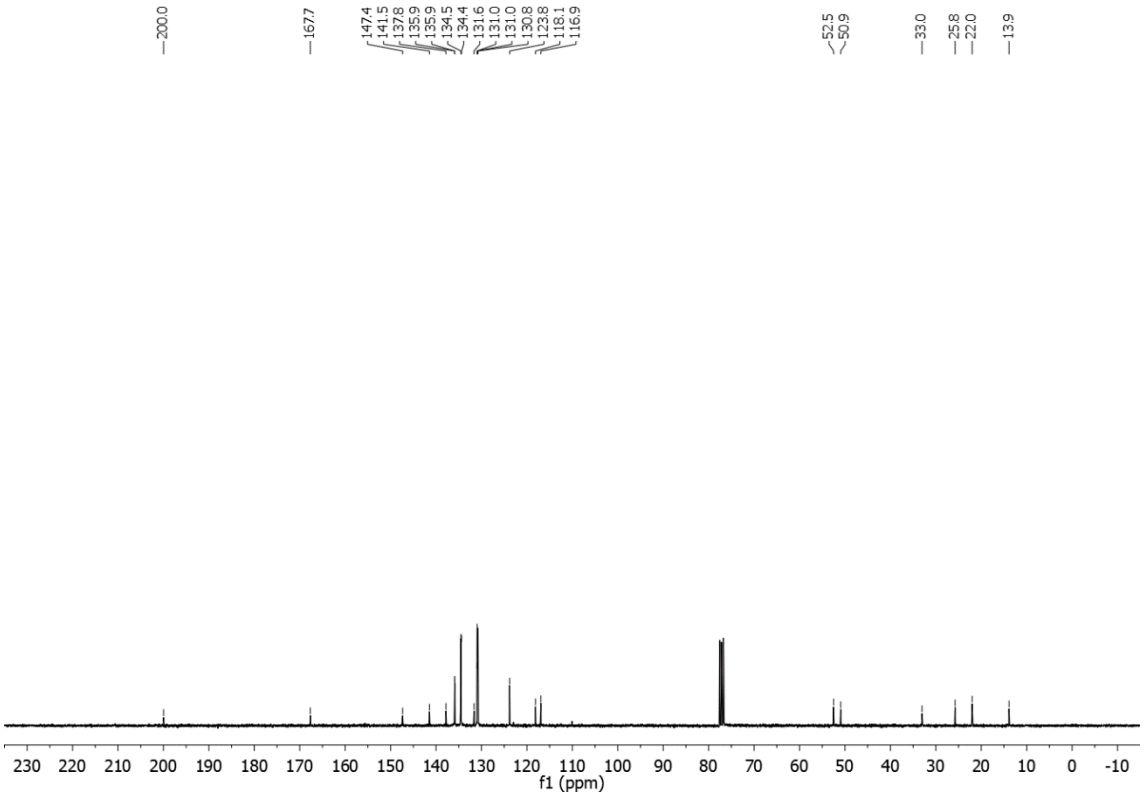

Figure S26. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **2c**.

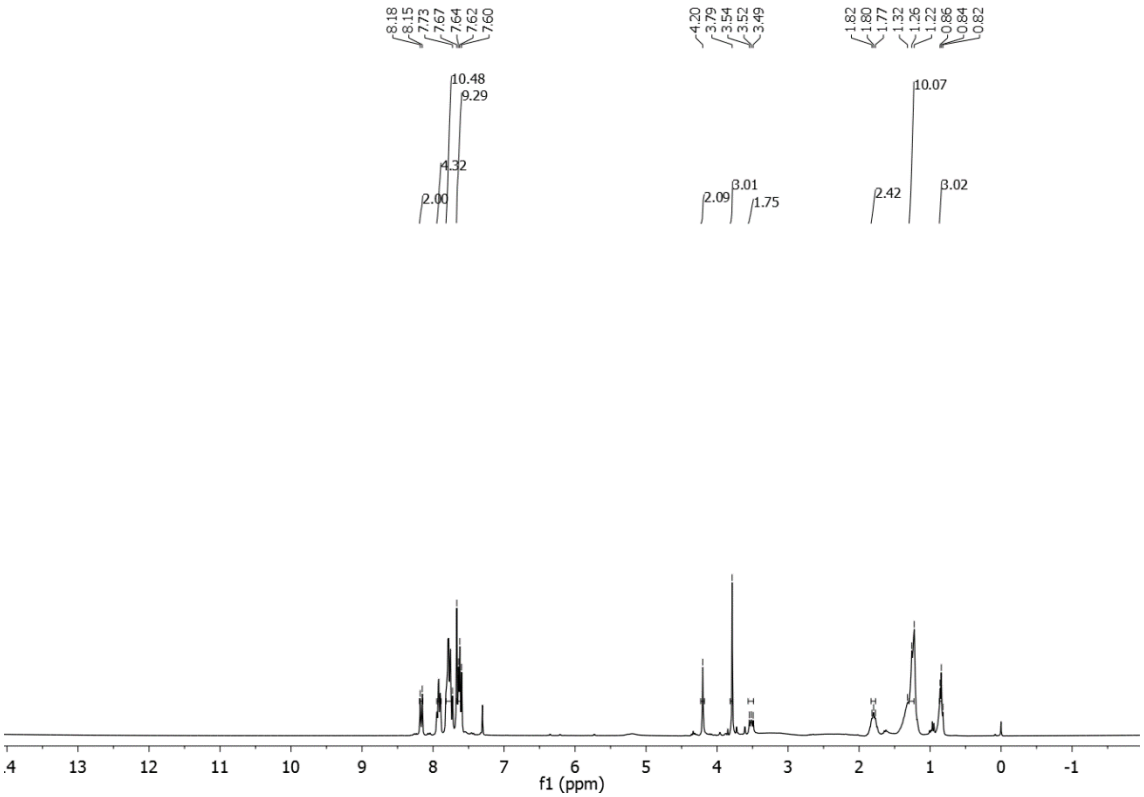

Figure S27. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **2d**.

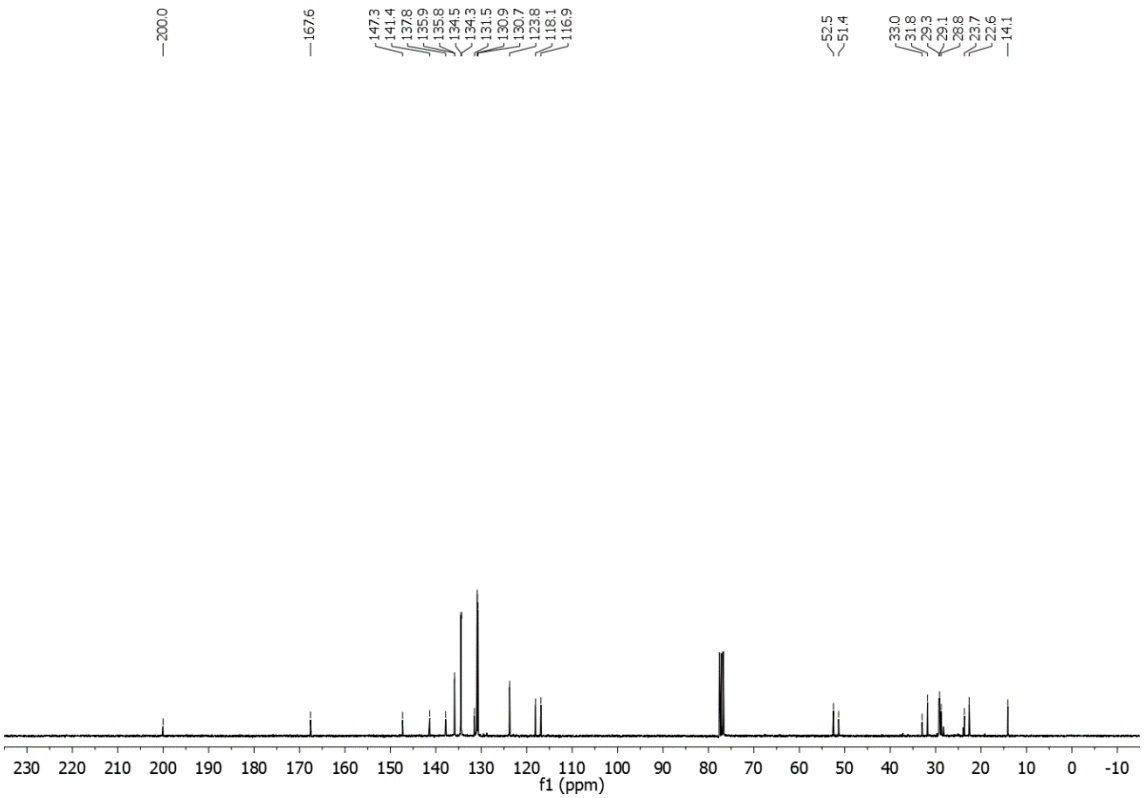

Figure S28. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **2d**.

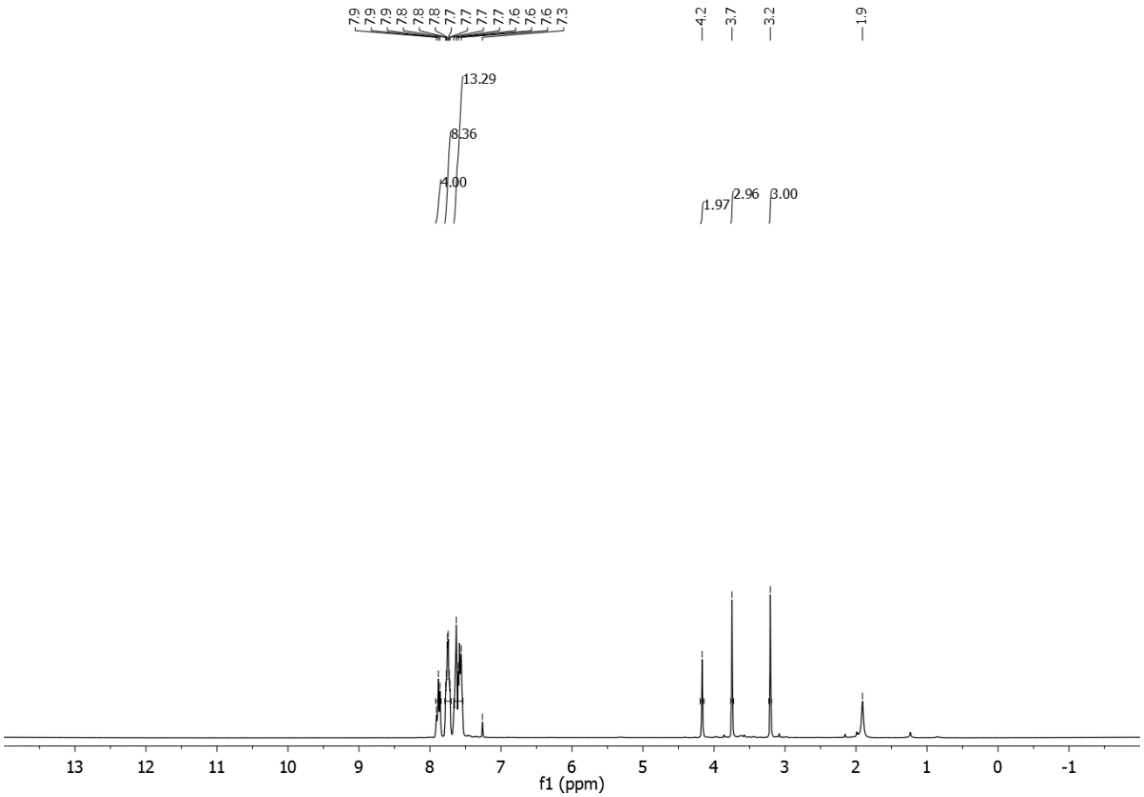

Figure S29. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **3a**.

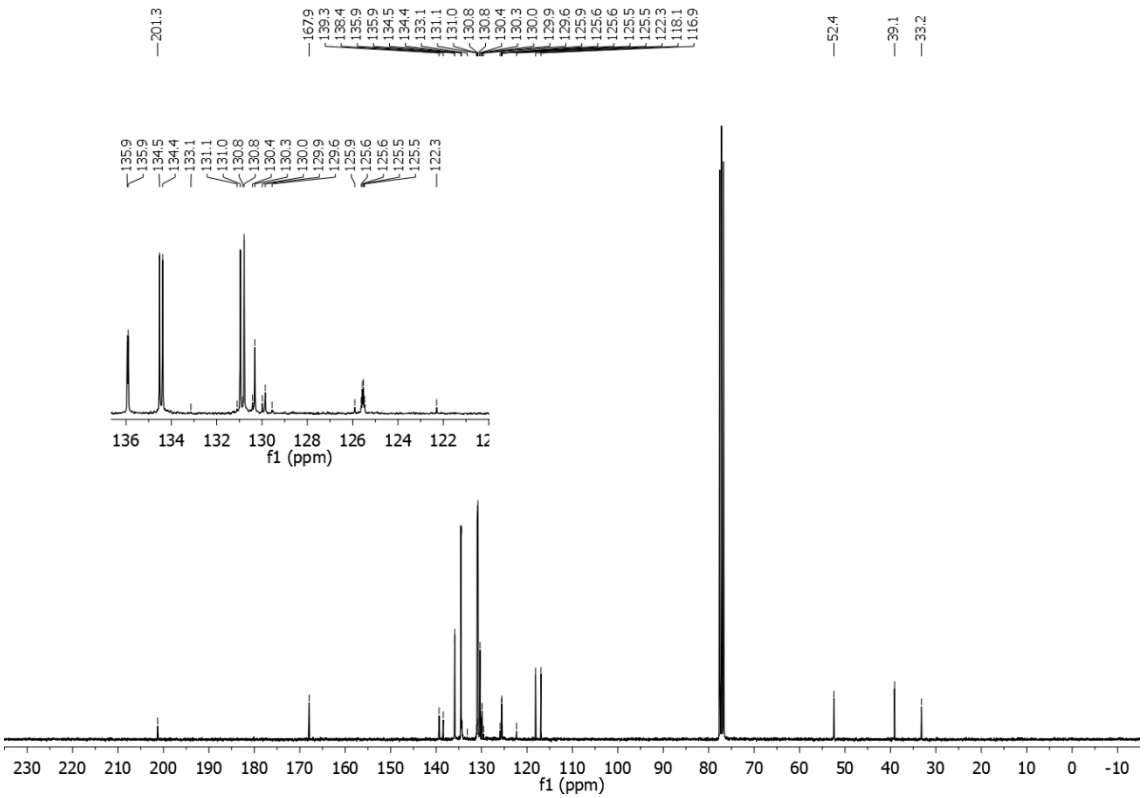

Figure S30. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **3a**.

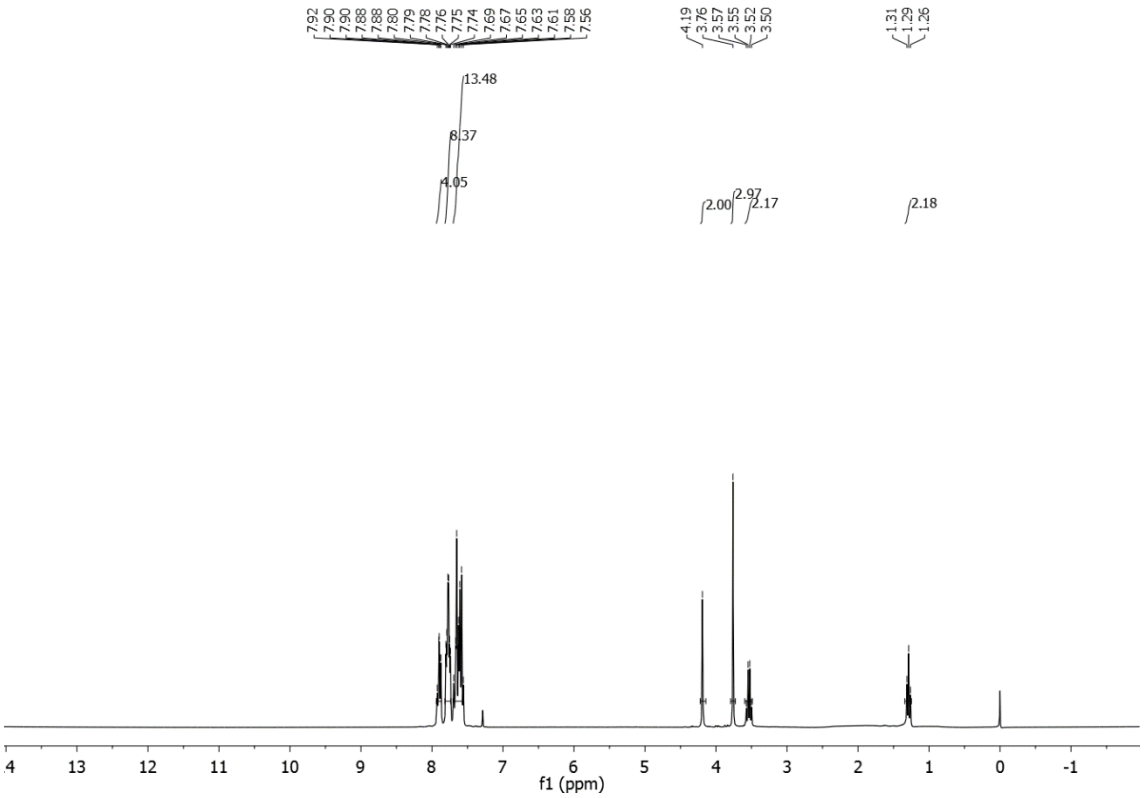

Figure S31. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **3b**.

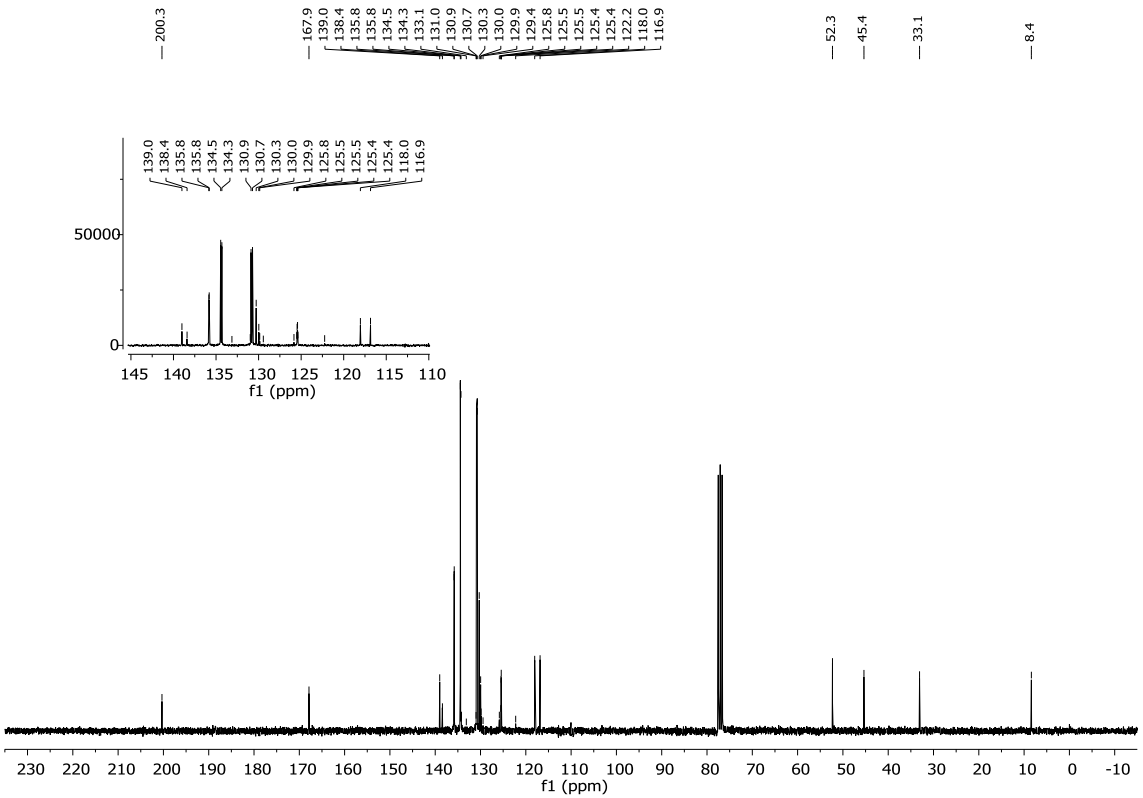

Figure S32. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **3b**.

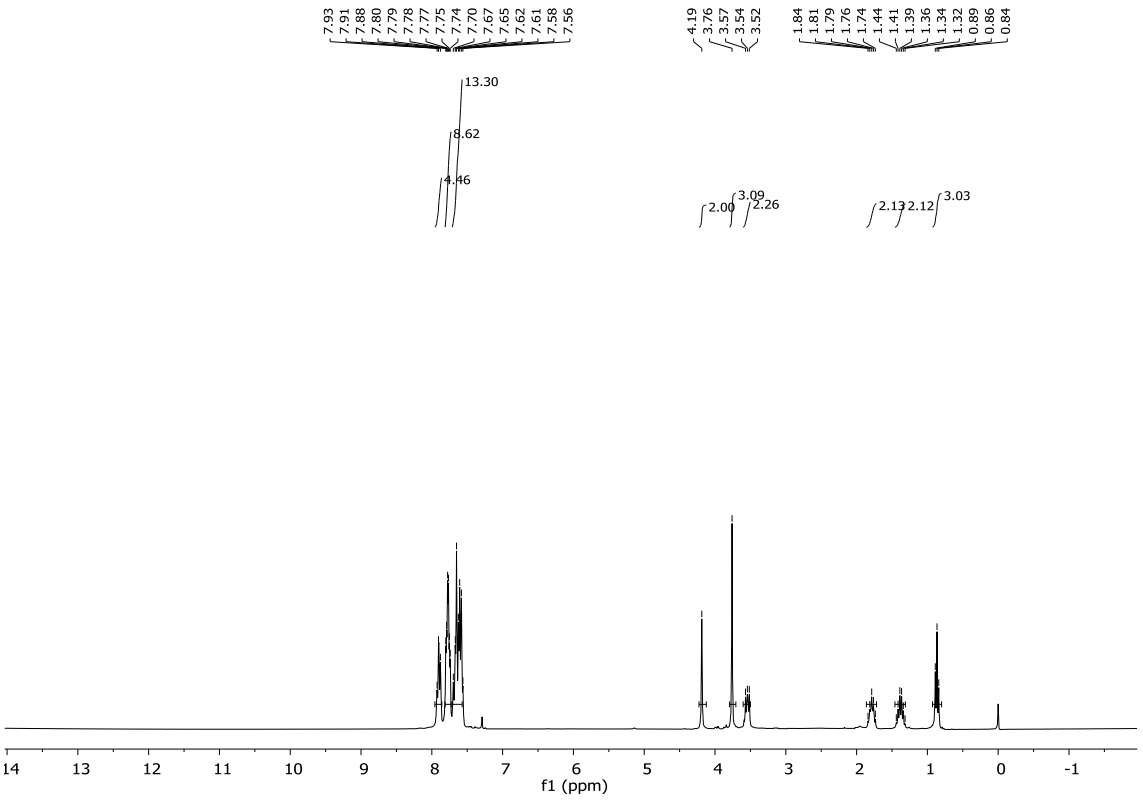

Figure S33. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **3c**.

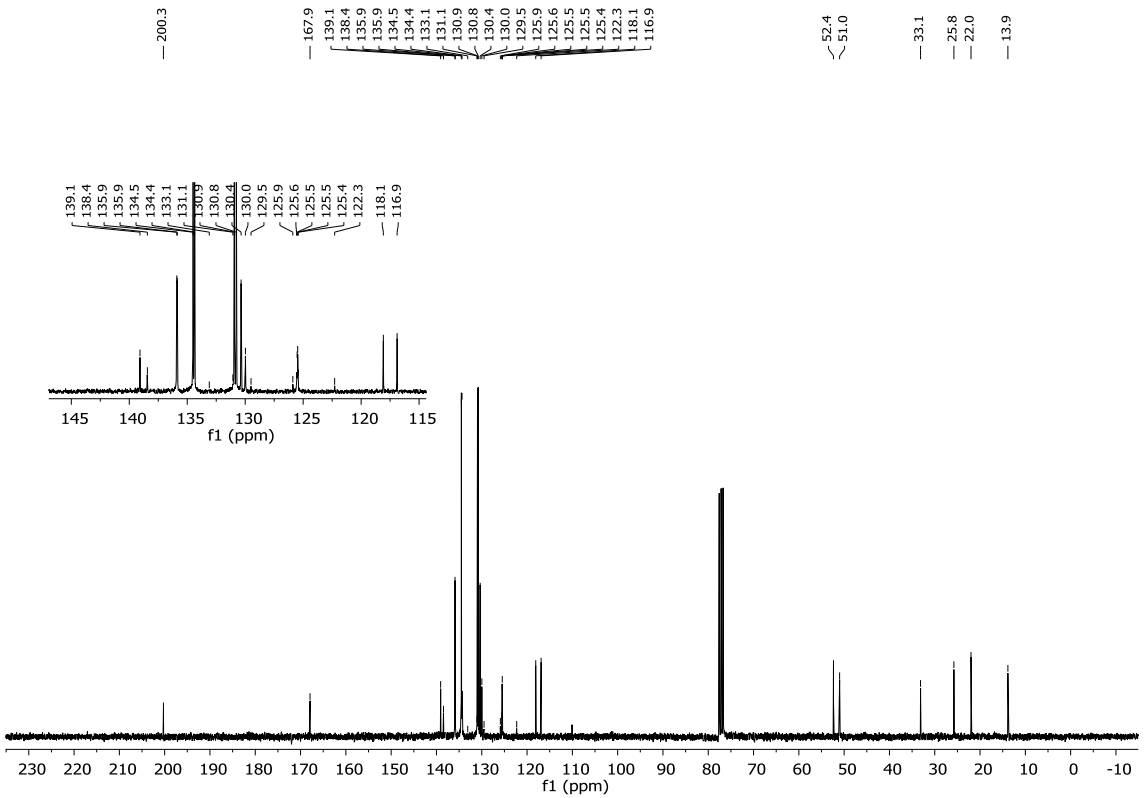

Figure S34. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **3c**.

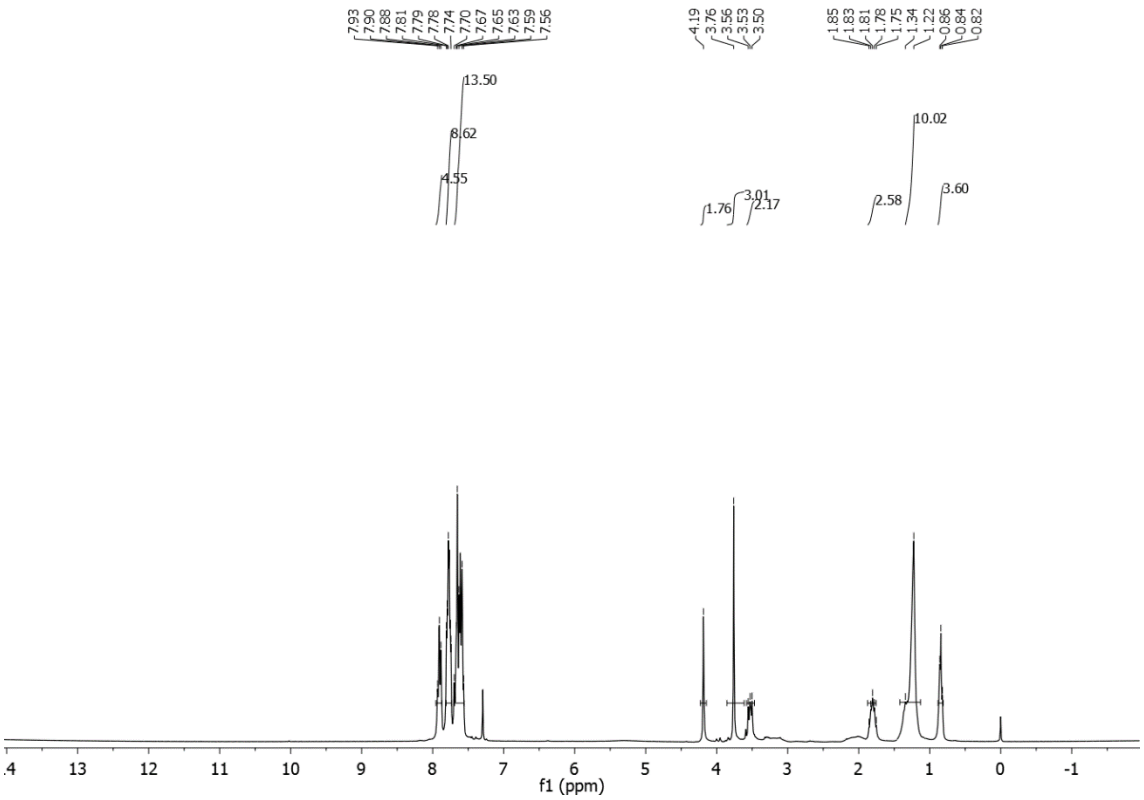

Figure S35. ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of compound **3d**.

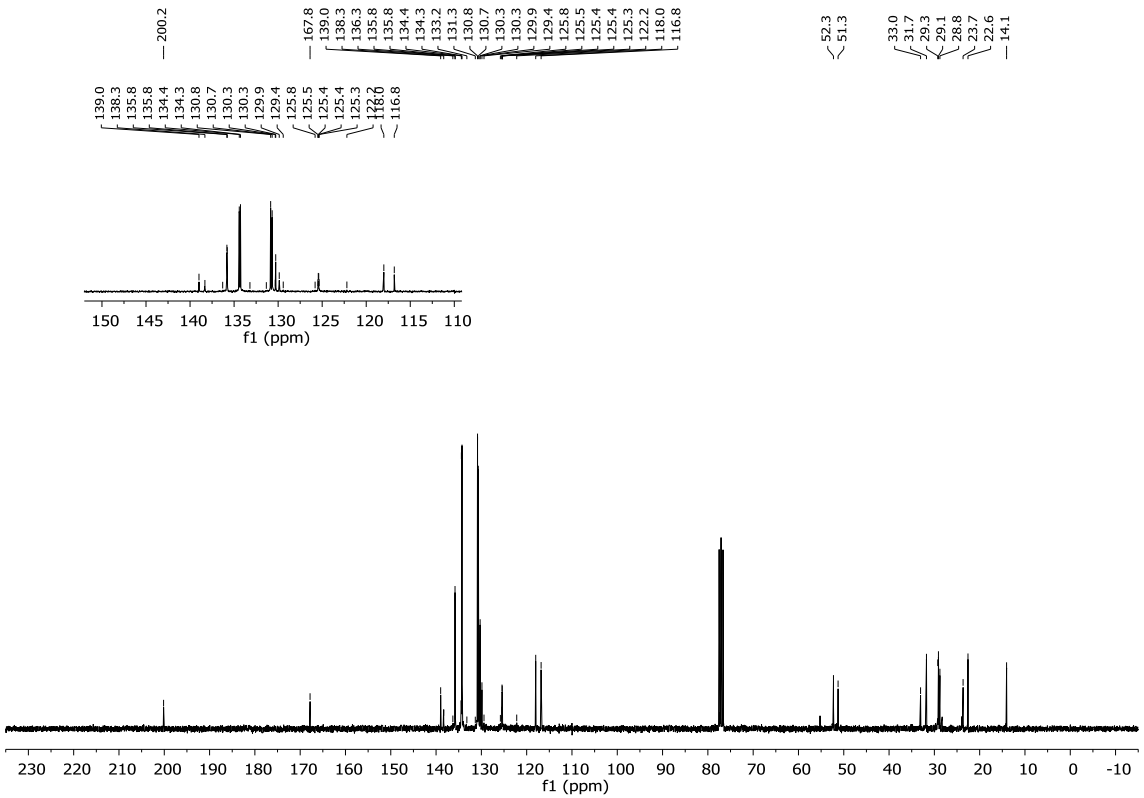

Figure S36. ^{13}C {^{1}H} NMR spectrum (75 MHz, $CDCl_3$) of compound **3d**.

We include here (**Tables S1**, **S2** and **S3**) the data obtained from the second experiment for the confirmation of the antifungal activity results.

Table S1. Regression equations for the inhibition of *P. pachyrhizi* spore germination *versus* the log of the allyldithiocarbimate salts concentrations, equation parameters and statistical data (second experiment with three replicates *per* treatment)

Compound	Equation: $y = A1 + \frac{(A2 - A1)}{(1 + 10^{((LOGx0 - x)p)})}$				Statistical data		
	A1	A2	LOGx0	p	r^2	*F*	Prob > *F*
1a	14.23	100.63	−0.99	2.36	0.9906	836.88	< 0.0001
1b	9.37	99.49	−1.14	2.35	0.9963	2275.81	< 0.0001
1c	7.85	105.54	−1.40	0.99	0.9819	1181.61	< 0.0001
1d	1.69	103.96	−1.48	1.03	0.9775	930.19	< 0.0001
2a	19.1	101.69	−1.04	2.08	0.9921	1286.99	< 0.0001
2b	7.88	109.62	−1.16	0.97	0.9724	517.34	< 0.0001
2c	8.27	100.24	−1.30	1.87	0.9916	1431.39	< 0.0001
2d	−8.73	83.65	−1.29	1.52	0.9915	1007.81	< 0.0001
3a	20.35	103.88	−1.03	1.47	0.9886	1093.87	< 0.0001
3b	18.58	106.13	−1.00	1.31	0.9880	1041.49	< 0.0001
3c	27.79	103.13	−1.05	1.59	0.9952	3197.32	< 0.0001
3d	3.00	107.02	−1.23	1.04	0.9841	857.97	< 0.0001

y: inhibition percentage; A1: bottom asymptote; A2: top asymptote; LOGx0: center; x: $\log_{10}$[concentration]; p: hill slope; r^2: coefficient of determination; *F*: value obtained by the *F*-test; Prob > F: probability value (*p* model, $F < 0.0001$).

Table S2. Regression equations for the inhibition of *H. vastatrix* spore germination *versus* the log of the allyldithiocarbimate salts concentrations, equation parameters and statistical data (second experiment with three replicates *per* treatment)

Compound	Equation: $y = A1 + \frac{(A2 - A1)}{(1 + 10^{((LOGx0 - x)p)})}$				Statistical data		
	A1	A2	LOGx0	p	r^2	*F*	Prob > *F*
1a	7.52	103.65	−0.82	2.43	0.9924	709.56	< 0.0001
1b	4.23	102.36	−0.74	2.92	0.9977	1931.19	< 0.0001
1c	11.44	104.41	−0.86	2.09	0.9931	907.90	< 0.0001
1d	5.45	111.99	−0.74	1.45	0.9775	260.04	< 0.0001
2a	15.41	102.44	−0.79	2.35	0.9819	366.23	< 0.0001
2b	11.50	115.84	−0.66	1.23	0.9896	719.19	< 0.0001
2c	22.81	102.68	−0.93	2.20	0.9946	1803.72	< 0.0001
2d	17.27	101.79	−0.84	2.77	0.9894	655.81	< 0.0001
3a	16.70	100.29	−0.79	2.75	0.9920	823.89	< 0.0001
3b	10.33	118.34	−0.64	1.24	0.9701	231.69	< 0.0001
3c	20.52	102.03	−0.98	2.00	0.9988	8121.37	< 0.0001
3d	16.44	103.89	−0.82	2.38	0.9905	732.78	< 0.0001

y: inhibition percentage; A1: bottom asymptote; A2: top asymptote; LOGx0: center; x: $\log_{10}$[concentration]; p: hill slope; r^2: coefficient of determination; *F*: value obtained by the *F*-test; Prob > F: probability value (*p* model, $F < 0.0001$).

Table S3. Concentrations of the allyldithiocarbimate salts to inhibit 50% (IC_{50}) and 90% (IC_{90}) spore germination of *P. pachyrhizi* and *H. vastatrix* (second experiment with three replicates *per* treatment)

Compound	Fungi			
	P. pachyrhizi		*H. vastatrix*	
	IC_{50} / (µmol L^{-1})	IC_{90} / (µmol L^{-1})	IC_{50} / (µmol L^{-1})	IC_{90} / (µmol L^{-1})
1a	87	232	139	320
1b	66	178	172	349
1c	30	215	117	311
1d	30	199	146	462
2a	70	213	134	344
2b	49	304	141	538
2c	45	151	87	251
2d	58	221	122	276
3a	62	278	141	333
3b	63	304	147	524
3c	52	237	79	252
3d	48	279	125	306

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: shelx

Bond precision: C-C = 0.0094 A Wavelength=0.71073

Cell: a=7.8085(4) b=14.1824(7) c=32.5064(19)
alpha=90 beta=93.209(2) gamma=90
Temperature: 296 K

	Calculated	Reported
Volume	3594.2(3)	3594.2(3)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C24 H20 P, C13 H13 N2 O6 S3	C24 H20 P, C13 H13 N2 O6 S3
Sum formula	C37 H33 N2 O6 P S3	C37 H33 N2 O6 P S3
Mr	728.80	728.80
Dx,g cm-3	1.347	1.347
Z	4	4
Mu (mm-1)	0.299	0.299
F000	1520.0	1520.0
F000'	1522.51	
h,k,lmax	9,17,40	9,17,40
Nref	7377	7377
Tmin,Tmax	0.902,0.926	0.902,0.926
Tmin'	0.891	

Correction method= # Reported T Limits: Tmin=0.902 Tmax=0.926
AbsCorr = MULTI-SCAN

Data completeness= 1.000 Theta(max)= 26.403

R(reflections)= 0.0853(5872) wR2(reflections)= 0.3057(7377)

S = 1.087 Npar= 444

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 B

PLAT930_ALERT_2_B Check Twin Law (0 0 1)[1 0 4] Estimated BASF 0.13

Author Response: strucutre refined as a Non-merohedral twin 2-axis (0 0 1) [1 0 4] with a basf = 0.04767

Alert level C

PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25)	0.31	Report
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of	S1	Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of	N1'	Check
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds	0.00938	Ang.
PLAT906_ALERT_3_C Large K value in the Analysis of Variance	2.150	Check
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.600	43	Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) .	5	Check
PLAT939_ALERT_3_C Large Value of Not (SHELXL) Weight Optimized S .	15.43	Check

---

Alert level G

PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical	?	Check
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large	0.17	Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large	9.14	Why ?
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels	2	Note
PLAT802_ALERT_4_G CIF Input Record(s) with more than 80 Characters	1	Info
PLAT870_ALERT_4_G ALERTS Related to Twinning Effects Suppressed ..	!	Info
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min)	1	Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600	37	Note
PLAT931_ALERT_5_G Found Twin Law ( )[1 0 4] Estimated BASF	0.16	Check

---

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

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

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.

Publication of your CIF in IUCr journals

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.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 27/03/2017; check.def file version of 24/03/2017

Datablock shelx - ellipsoid plot