

## Supplementary Information

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### Ultrasound-Assisted Diversion of Nitrobenzene Derivatives to their Aniline Equivalents through Heterogeneous Magnetic Ag/Fe<sub>3</sub>O<sub>4</sub>-IT Nanocomposite Catalyst

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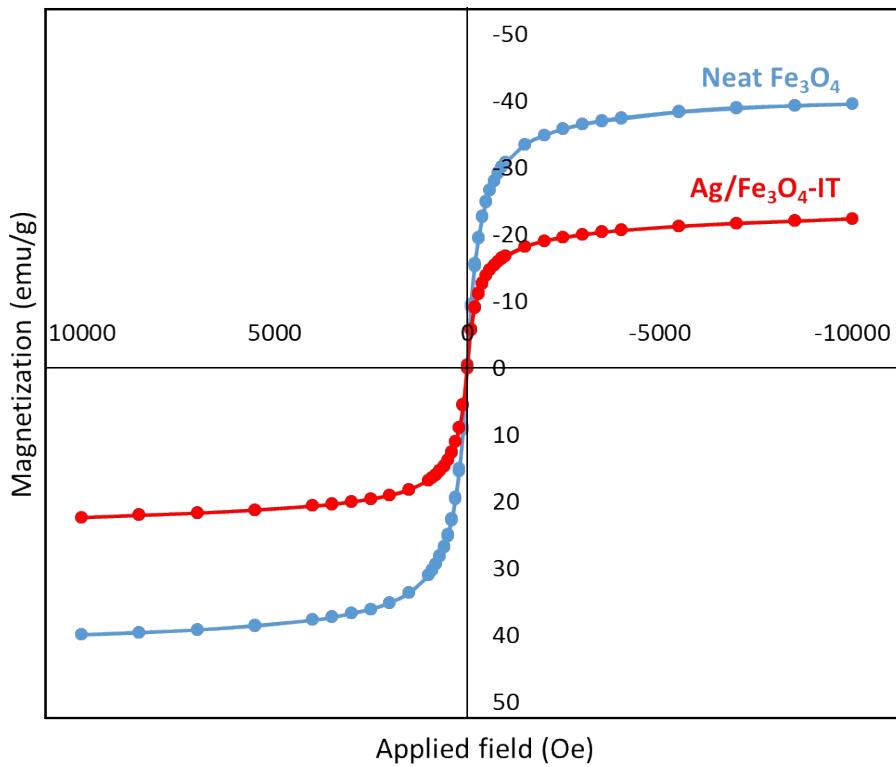
Ahmed Esmail Shalan: <https://orcid.org/0000-0002-3424-1609>

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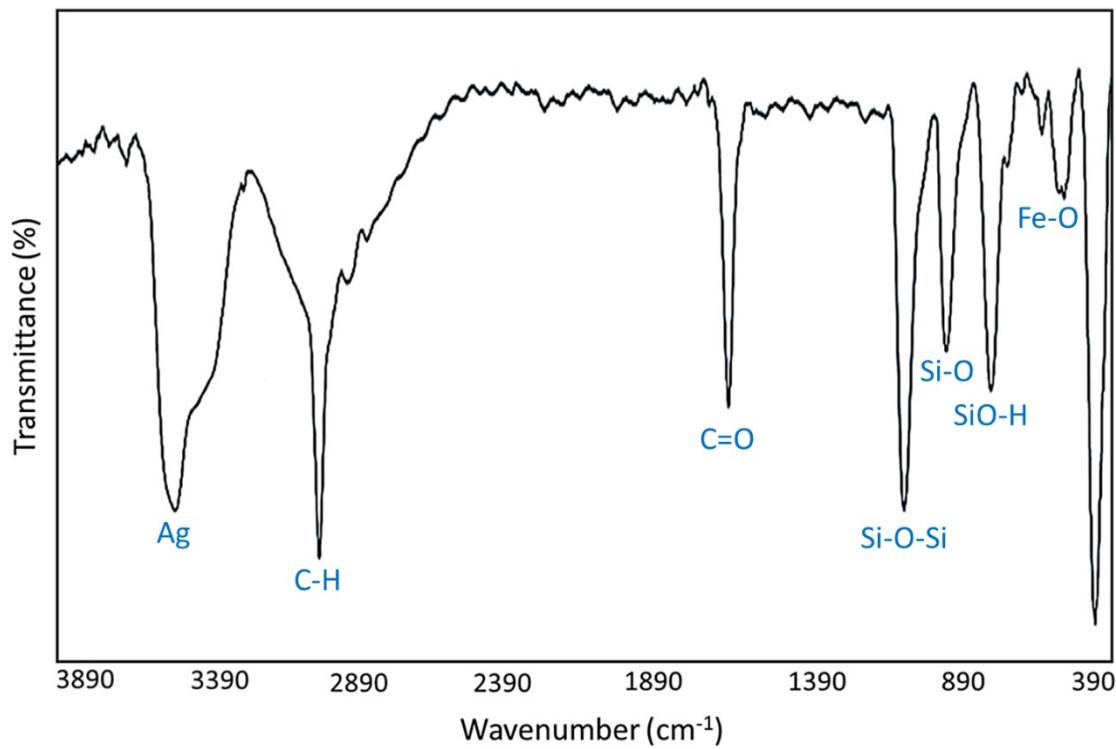
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**Table S1.** Applied materials and equipment in this project.

Material / equipment	Brand
Silver nitrate	Sigma Aldrich, ≥99.0%
Iron(II) chloride tetrahydrate	Sigma Aldrich, ≥99%
Iron(II) chloride hexahydrate	Sigma Aldrich, ≥99%
5-Chloro-2-methyl-4-isothiazolin-3-one	Santa Cruz Biotechnology
Ammonia	Merck, 25%
APTES	Sigma Aldrich, 99%
Solvents	Merck
Nitrobenzene derivatives	Sigma Aldrich
Potassium carbonate	Sigma Aldrich, ≥99%
Silica gel for Column chromatography	Sigma Aldrich, 60
Glass tube	13 by 100 mm, equipped with a threaded cap
Glassware	Iso-Lab
Ultrasound bath	Cleaning bath KQ-250 DE
SEM analysis	ZEISS SIGMA
TEM analysis	Philips CM200
HRTEM	Hitachi S-5200 and Philips CM200
XRD	X-ray diffractometer operating at 40 mA, 40 kV,
DLS analysis	Horiba (SZ-100)
FT-IR analysis	Shimadzu-8400s
EDX analysis	VEGA-TESCAN-XMU
XPS analysis	K-Alpha
VSM analysis	Meghnatis Kavir Kashan Co.
NMR analysis	Varian Unity Inova 500 MHz
Melting point apparatus	Electrothermal 9100
TLC plate (silica)	Merck silica gel GF254 plates

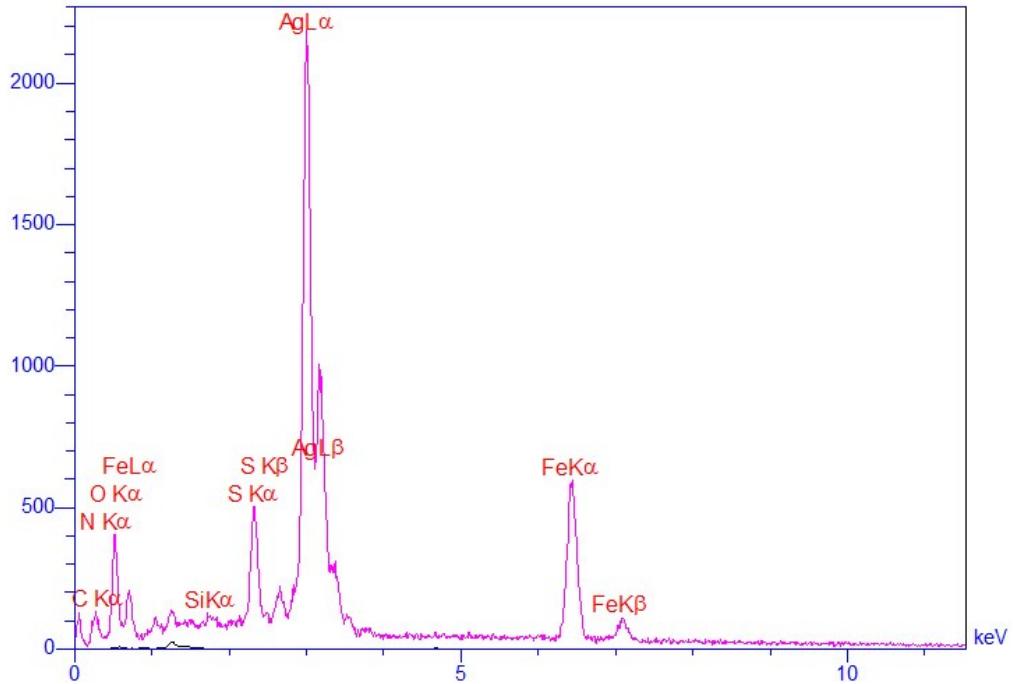


**Figure S1.** VSM M-H curves of IT-functionalized Ag/Fe<sub>3</sub>O<sub>4</sub> nanocatalyst and the neat Fe<sub>3</sub>O<sub>4</sub> NPs (recorded at room temperature).



**Figure S2.** FT-IR spectrum of recovered IT-functionalized Ag/Fe<sub>3</sub>O<sub>4</sub> nanocatalyst.

A sharp peak at 3500  $\text{cm}^{-1}$  is related to Ag NPs, a sharp peak at 2950  $\text{cm}^{-1}$  is related to C—H (sp<sup>3</sup>), a sharp peak at 1690  $\text{cm}^{-1}$  is related to C=O (present in the IT ring), a sharp peak at 1100  $\text{cm}^{-1}$  is related to Si—O—Si (stretching vibration), a peak at 1000  $\text{cm}^{-1}$  is related to Si—O (stretching vibration), a peak around 860  $\text{cm}^{-1}$  is related to SiO—H (stretching vibration), and the peak appeared at around 580  $\text{cm}^{-1}$  coming from Fe—O bond.



**Figure S3.** EDX spectrum of recovered IT-functionalized Ag/Fe<sub>3</sub>O<sub>4</sub> nanocatalyst.

C: 10.6 wt%

N: 5.4 wt%

O: 20.3 wt%

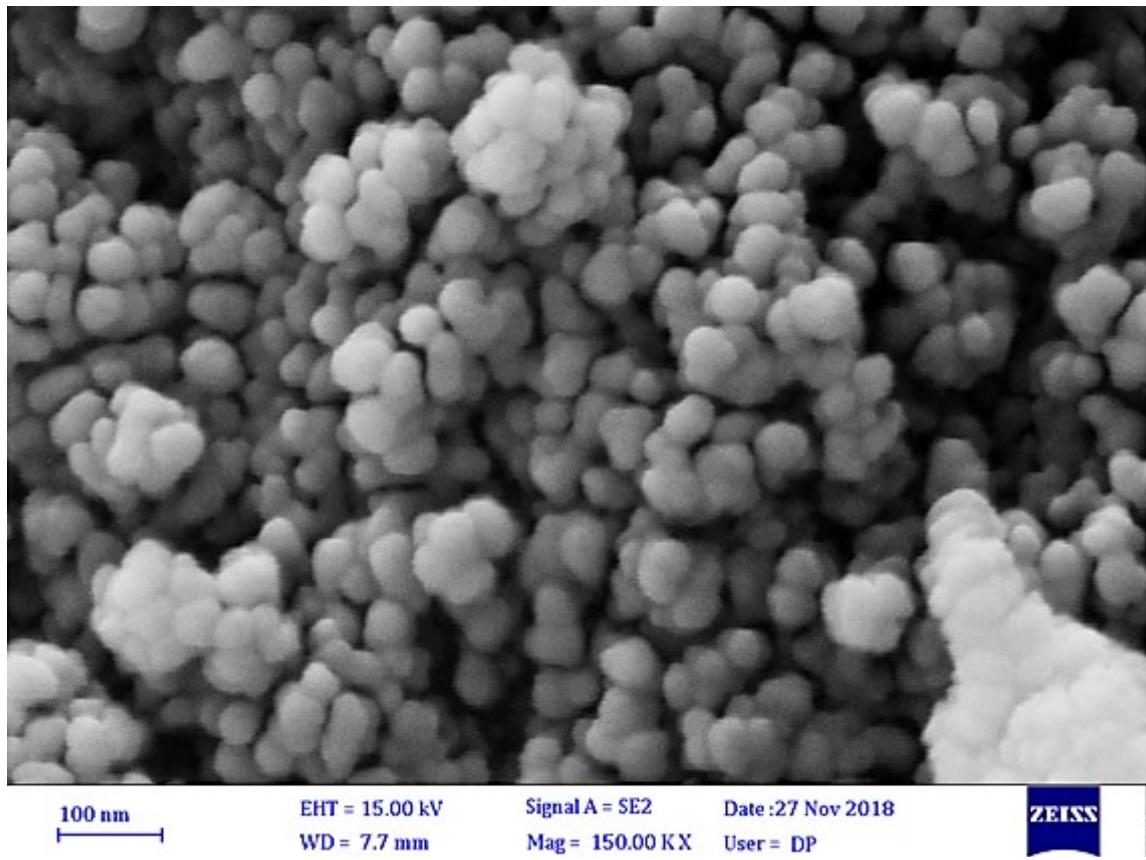
Si: 0.3 wt%

S: 3.5 wt%

Fe: 14.4 wt%

Ag: 45.5 wt%

Per 100 g of catalyst, there are 0.26 mol Fe and 0.42 mol of Ag elements. It means that the molar ratio of Fe/Ag in 100 g of catalyst is: **0.61**



100 nm

EHT = 15.00 kV  
WD = 7.7 mm

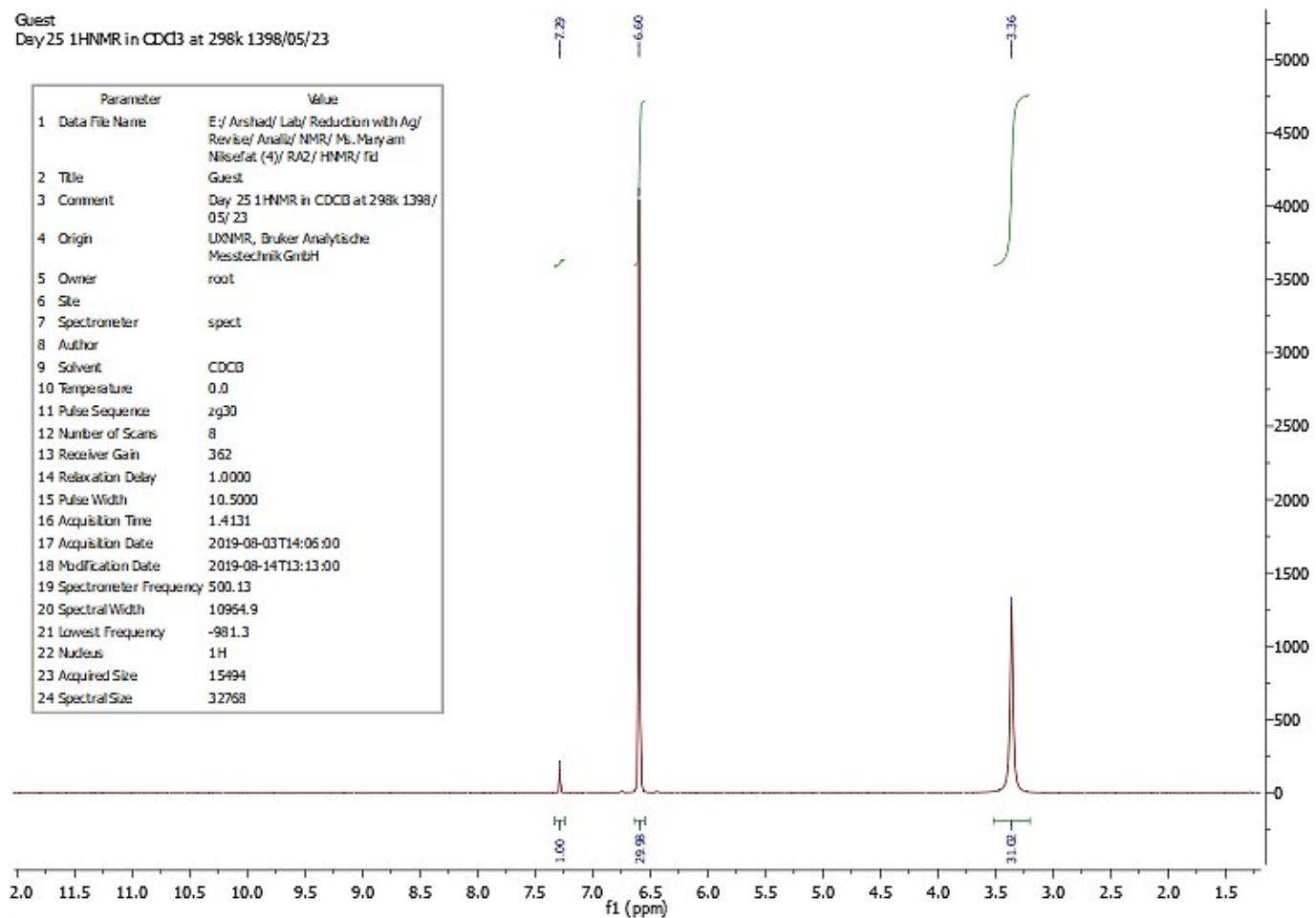
Signal A = SE2  
Mag = 150.00 KX

Date : 27 Nov 2018  
User = DP

ZEISS

**Figure S4.** SEM image of recovered IT-functionalized Ag/Fe<sub>3</sub>O<sub>4</sub> nanocatalyst.

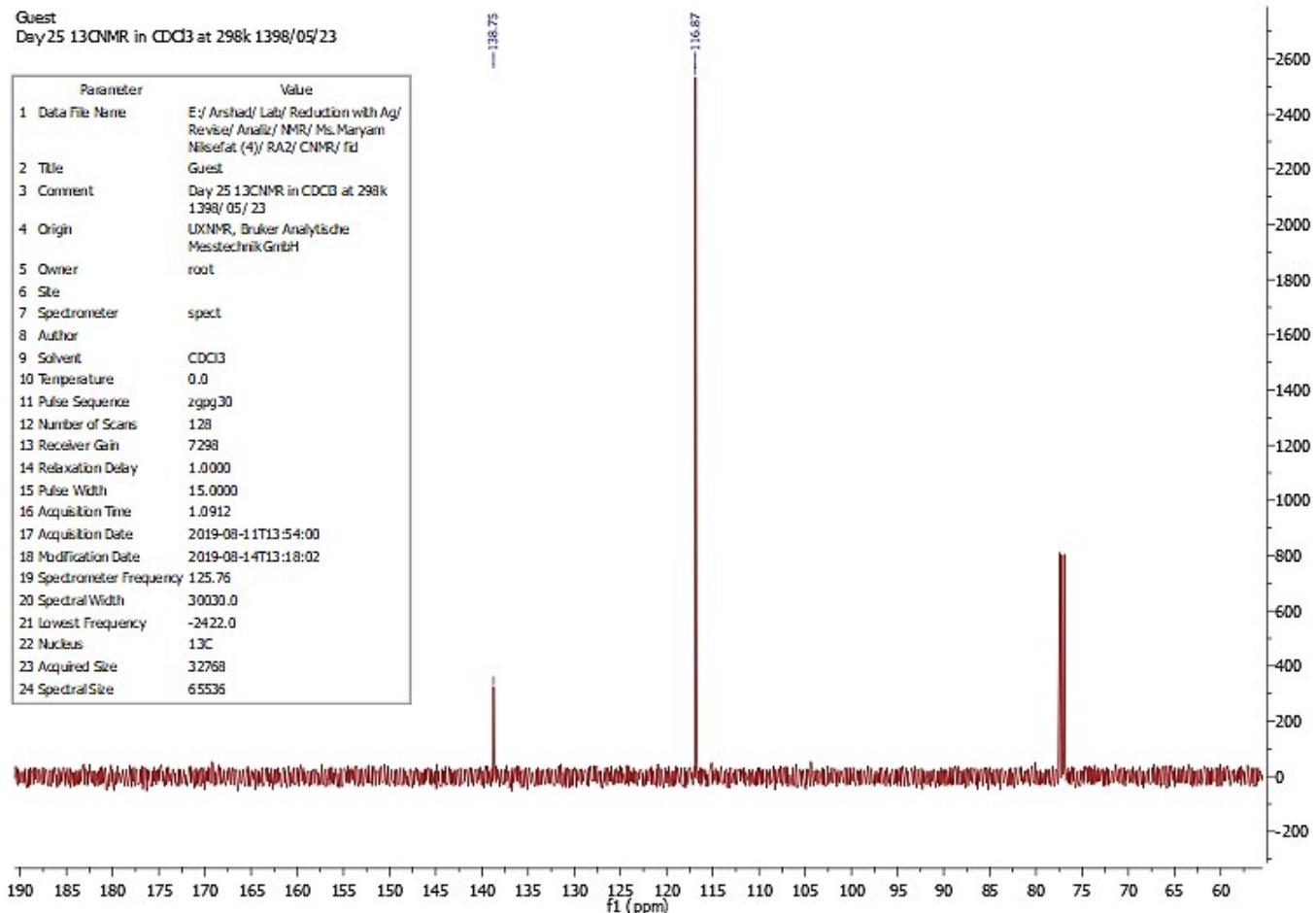
*Benzene-1,4-diamine (Table 2 – Entry 2):*  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.59 (s, 4 H), 3.36 (s, 4 H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  138.75, 116.87.



**Figure S5.** Spectral data and  $^1\text{H}$ -NMR spectrum of benzene-1,4-diamine.

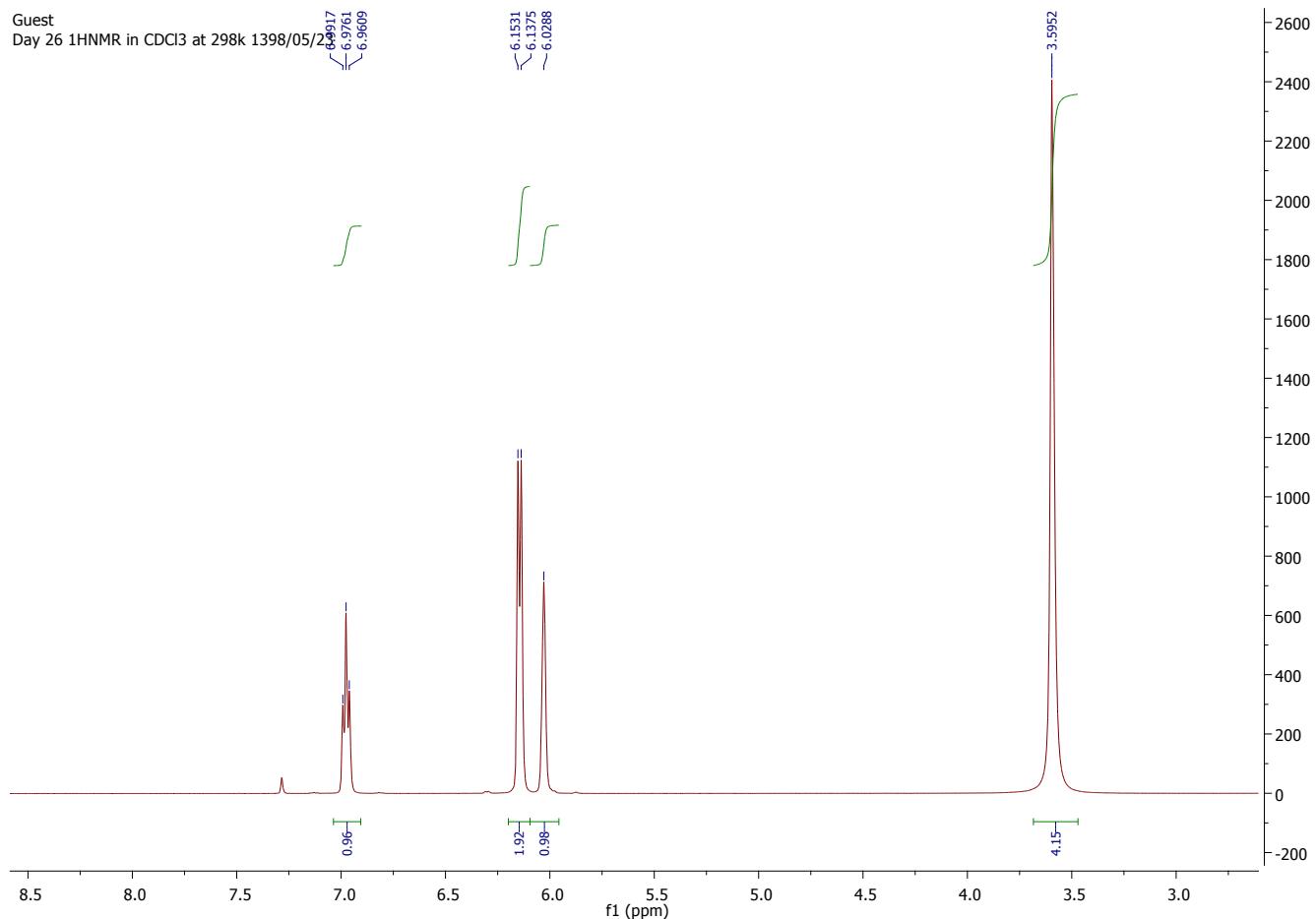
Guest  
Day 25  $^{13}\text{C}$ NMR in  $\text{CDCl}_3$  at 298k 1398/05/23

Parameter	Value
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2 Title	Guest
3 Comment	Day 25 $^{13}\text{C}$ NMR in $\text{CDCl}_3$ at 298k 1398/05/23
4 Origin	UXNMR, Bruker Analytische Messtechnik GmbH
5 Owner	root
6 Site	
7 Spectrometer	spec1
8 Author	
9 Solvent	$\text{CDCl}_3$
10 Temperature	0.0
11 Pulse Sequence	zgpg30
12 Number of Scans	128
13 Receiver Gain	7298
14 Relaxation Delay	1.0000
15 Pulse Width	15.0000
16 Acquisition Time	1.0912
17 Acquisition Date	2019-08-11T13:54:00
18 Modification Date	2019-08-14T13:18:02
19 Spectrometer Frequency	125.76
20 Spectral Width	30030.0
21 Lowest Frequency	-2422.0
22 Nucleus	$^{13}\text{C}$
23 Acquired Size	32768
24 SpectralSize	65536



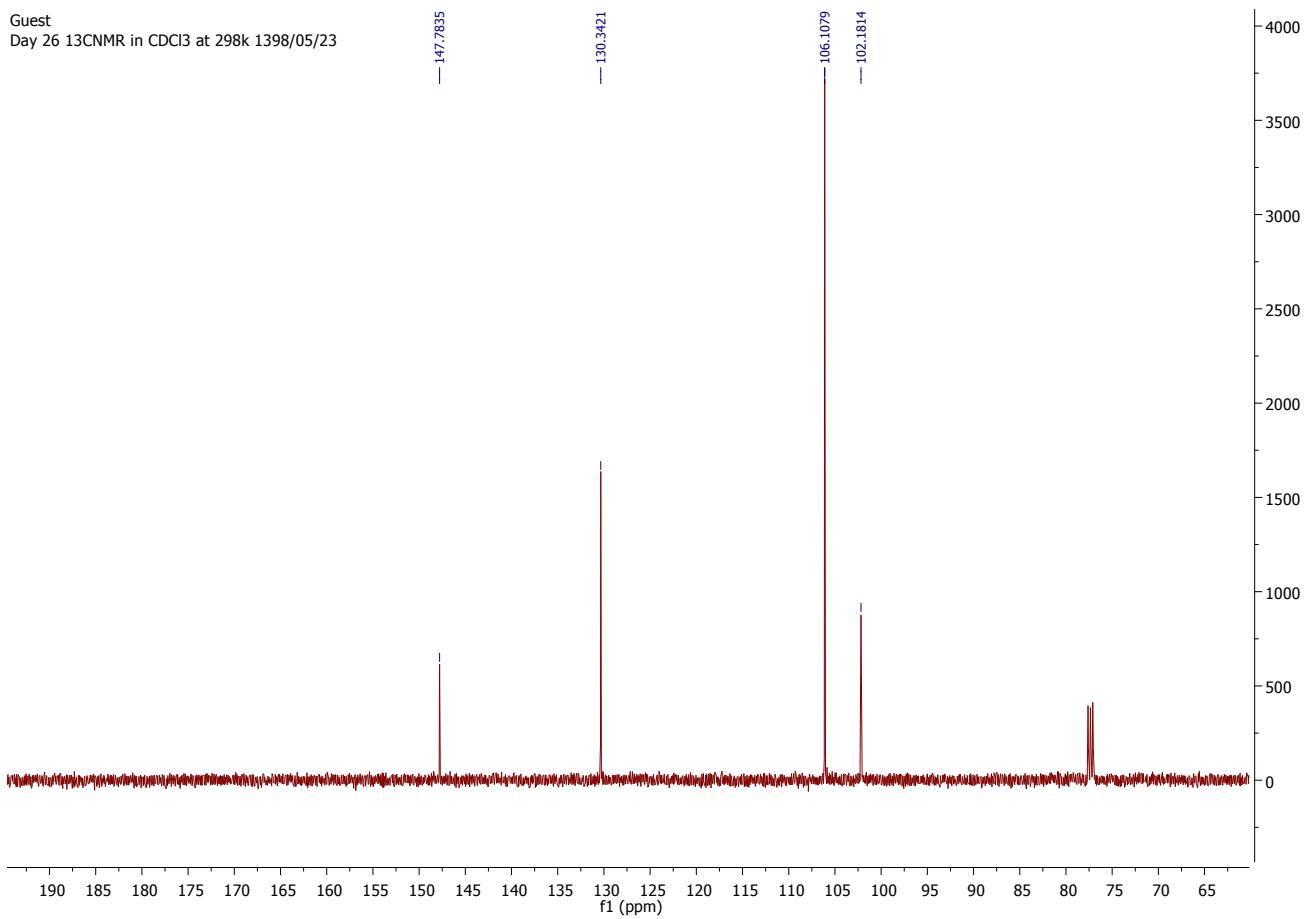
**Figure S6.**  $^{13}\text{C}$ -NMR spectrum of benzene-1,4-diamine.

*Benzene-1,3-diamine (Table 2 – Entry 3):*  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.96–6.99 (m, 1 H), 6.15–6.13 (d,  $J$  = 7.8 Hz, 2 H), 6.03 (s, 1 H), 3.59 (s, 4 H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  147.78, 130.34, 106.25, 102.18.



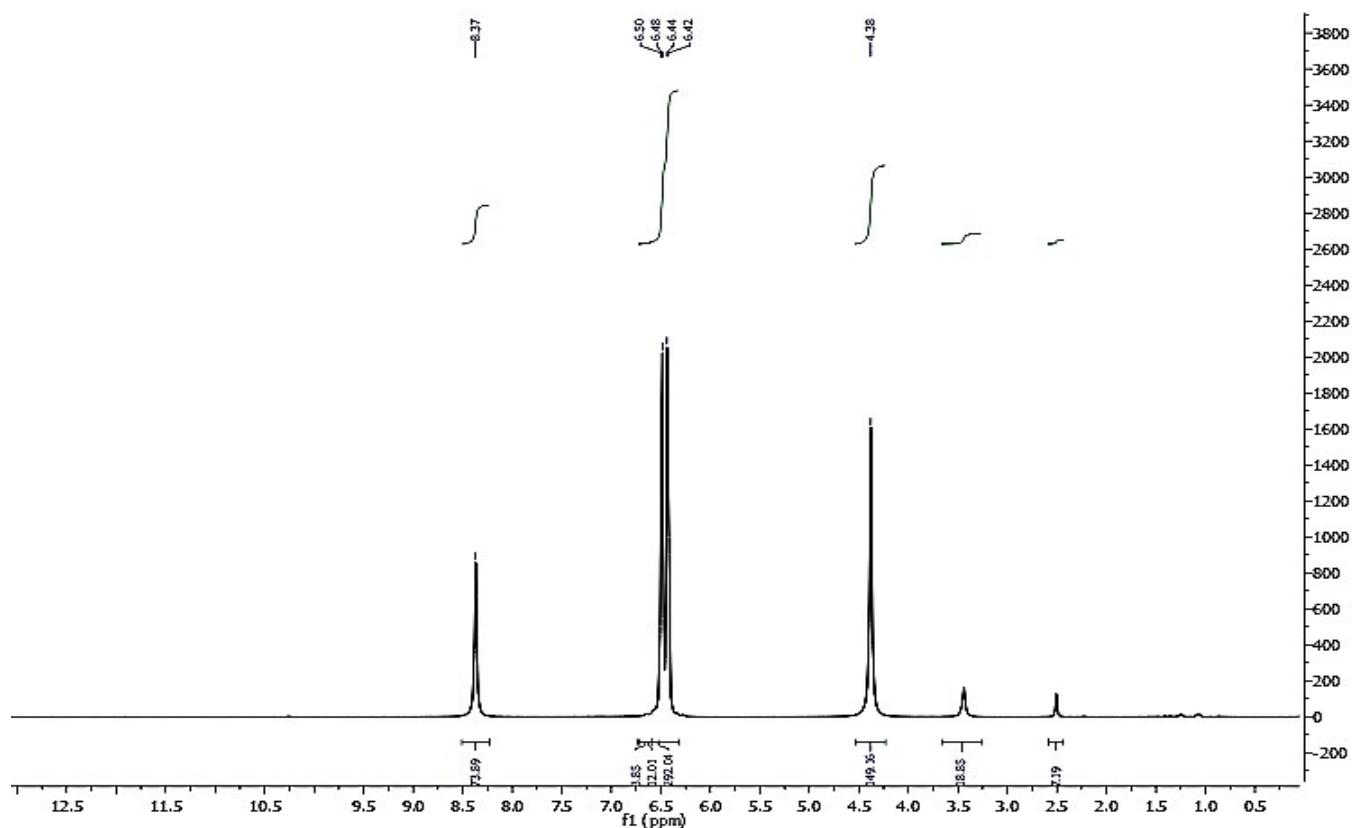
**Figure S7.** Spectral data and  $^1\text{H}$ -NMR spectrum of benzene-1,3-diamine.

Guest  
Day 26 13CNMR in CDCl<sub>3</sub> at 298k 1398/05/23

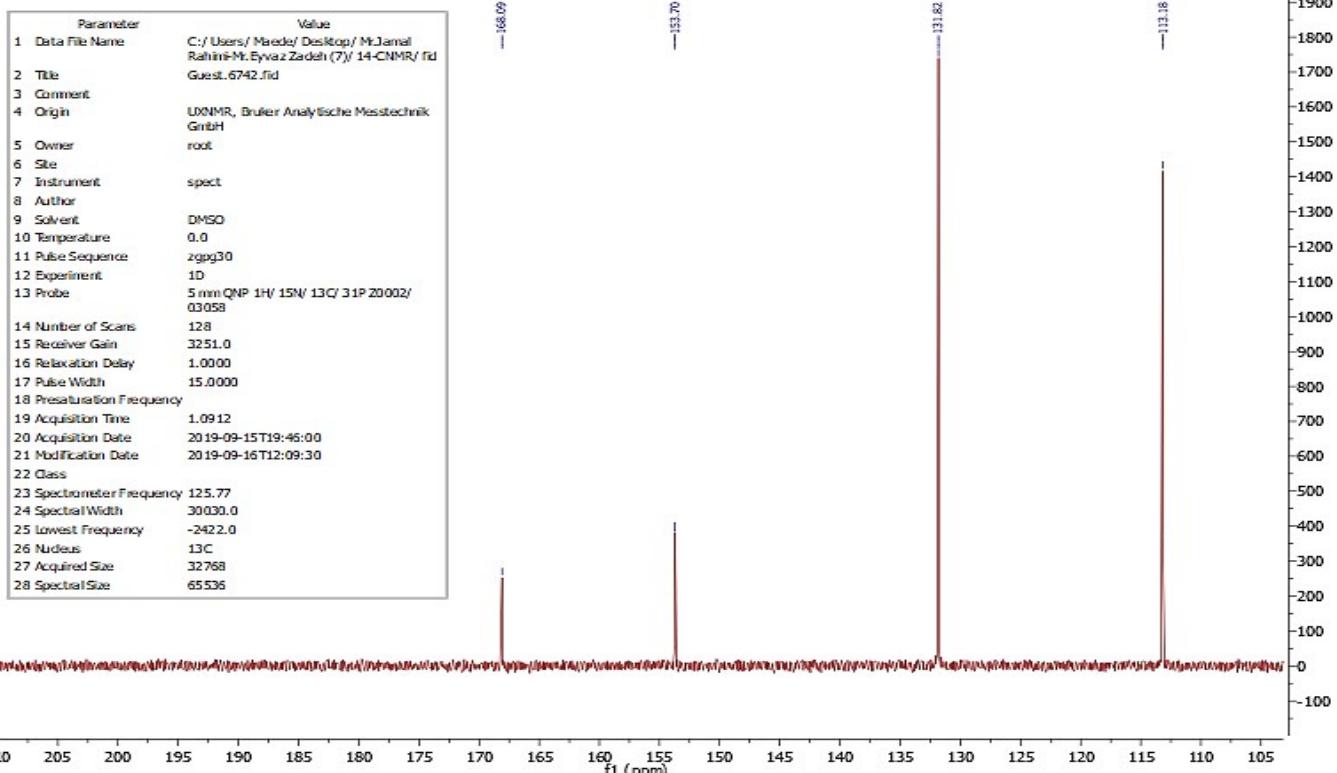


**Figure S8.** <sup>13</sup>C-NMR spectrum of benzene-1,3-diamine.

**4-Aminophenol (Table 3, Entry 6):** white solid,  $^1\text{H}$  NMR (500 MHz, DMSO):  $\delta$  (ppm) = 4.38 (2H, s, NH<sub>2</sub>), 6.42–6.44 (2H, d, J=10 Hz, H–Ar), 6.48–6.50 (2H, d, J=10 Hz, H–Ar), 8.37 (1H, s, OH).  $^{13}\text{C}$  NMR (500 MHz, DMSO)  $\delta$  168.09, 153.70, 131.82, 113.18.

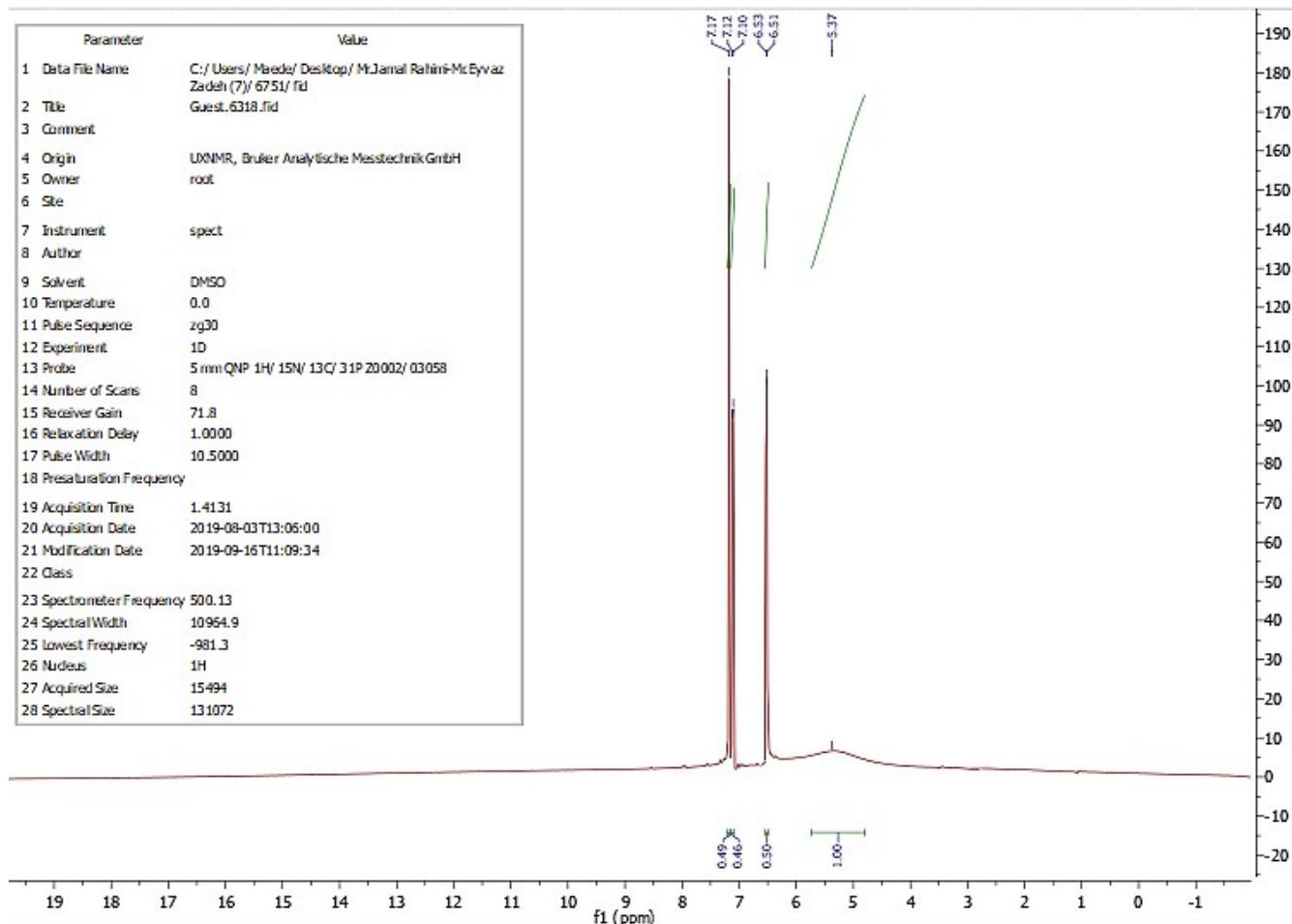


**Figure S9.** Spectral data and  $^1\text{H}$ -NMR spectrum of 4-aminophenol.

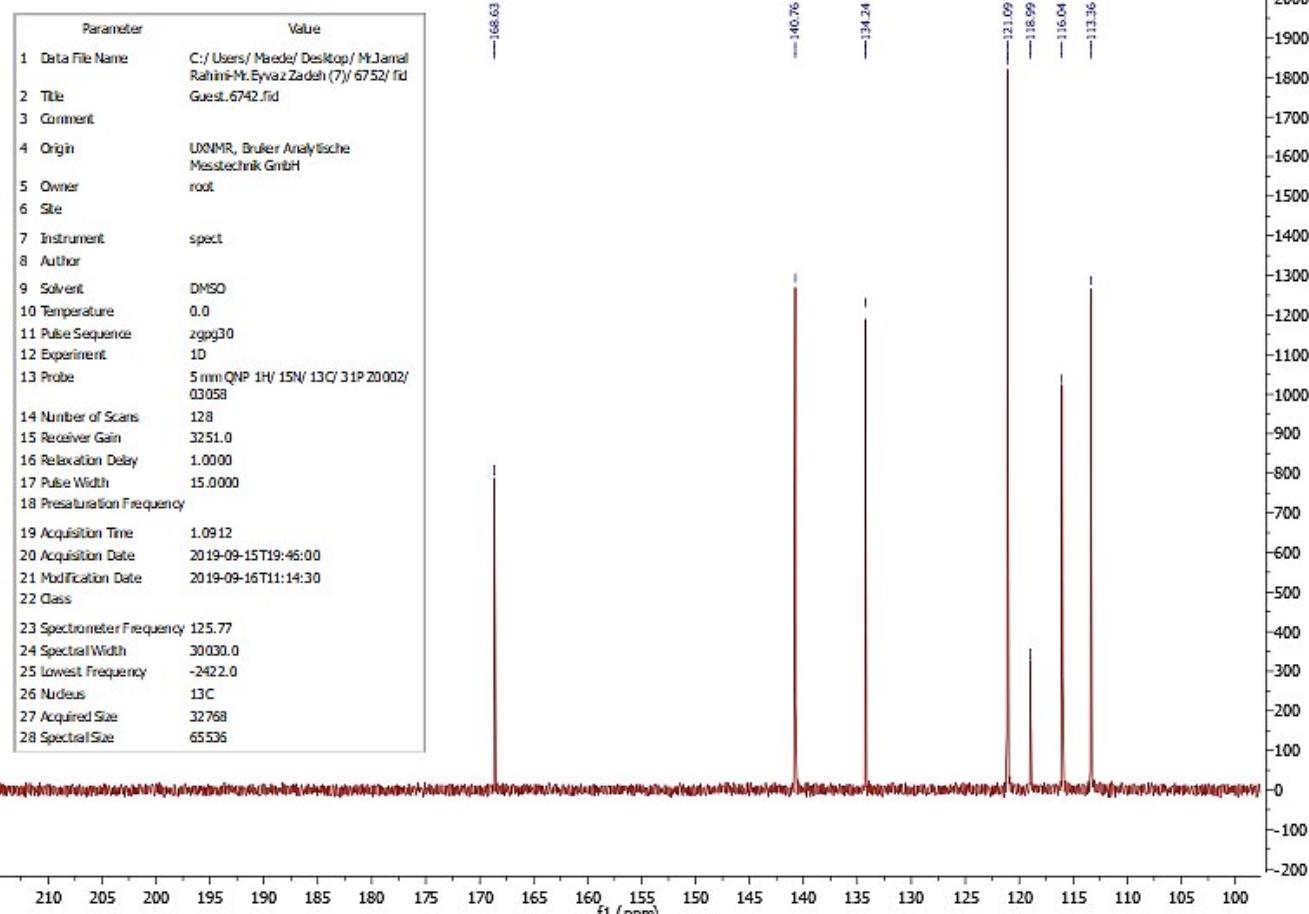


**Figure S10.**  $^{13}\text{C}$ -NMR spectrum of 4-aminophenol.

**3,4-Diaminobenzoic acid (Table 2 – Entry 9):**  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  5.37 (s, 4H), 6.51-6.53 (d,  $J$  = 10 Hz, 1H), 7.10-7.12 (d,  $J$  = 10 Hz, 1H), 7.17 (s, 1H).  $^{13}\text{C}$  NMR (500 MHz, DMSO)  $\delta$  168.63, 140.76, 134.24, 121.09, 118.99, 116.04, 113.36.



**Figure S11.** Spectral data and  $^1\text{H}$ -NMR spectrum of 3,4-diaminobenzoic acid.



**Figure S12.** <sup>13</sup>C-NMR spectrum of 3,4-diaminobenzoic acid.