

Scientific paper

Regioselective Synthesis of 1,2,3-Triazoles Catalyzed Over ZnO Supported Copper Oxide Nanocatalyst as a New and Efficient Recyclable Catalyst in Water

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Received: 14-11-2014

Abstract

The CuO/ZnO nanocatalysts are reported as efficient and recyclable catalysts for the regioselective synthesis of 1,2,3-triazoles from benzyl halides and terminal alkynes in water. The catalysts are synthesized by a co-precipitation method and characterized by BET surface area, XRD, SEM, TEM and EDS analysis. The effect of CuO loading, catalyst amount and solvent was investigated. The catalyst can be recovered by a simple filtration and applied in consecutive runs with no loss of activity.

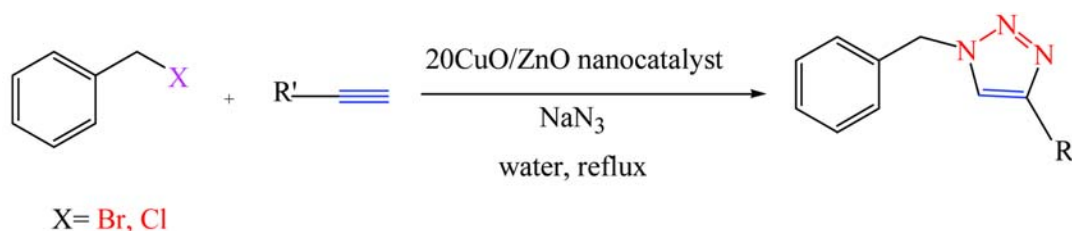
Keywords: CuO/ZnO nanocatalyst, Sodium azide, 1,2,3-triazole, Click chemistry.

1. Introduction

Nowadays, synthesis of 1,2,3-triazoles has received considerable attention because of their wide range of applications.¹ 1,2,3-Triazoles are five-membered nitrogen heterocyclic compounds that have been broadly applied in various research fields including synthetic organic, medicinal, materials, and biological chemistry.² These compounds are useful building blocks in the chemistry, resistant to moisture, oxygen, light, and also metabolism in the body.³ In addition, 1,2,3-triazoles are used as dyes, corrosion inhibition agents, photostabilizers, and photographic materials.⁴ The main method for the synthesis of

1,2,3-triazoles is the Huisgen 1,3-dipolar cycloaddition reaction of azides with alkynes.⁵ This reaction has been the model for click reactions which was introduced by Kolb and Sharpless in 2001. However, there are some catalytic reactions for the synthesis of 1,2,3-triazole derivatives. Copper-based catalysts can perform the cycloaddition reaction of azides with terminal alkynes by Cu(I) and/or Cu(II) species.^{6–21} Moreover, the performance of Ru-based catalysts on the cycloaddition of azides and terminal alkynes (RuAAC) for the regioselective synthesis of 1,2,3-triazoles has been reported.^{22–25}

Recently, nanocatalysts have been extensively studied as competitive substitutes to the usual catalysts due



Scheme 1. Synthesis of 1,2,3-triazoles catalyzed by 20CuO/ZnO nanocatalyst.

to their comparable performance.²⁶ Particularly, application of nano-metal oxides as catalysts in organic synthesis has been improved because of their significant characteristics. The recyclability of catalysts is an additional advantage of these catalysts.²⁷

We have been putting our efforts towards the design and development of nanocatalysts. In our previous research, we have reported the activity of (P_4 VPy-CuI), CuO-CeO₂ and Au supported nanocatalysts as efficient recyclable nanocatalysts in organic synthesis.^{29–31} Moreover, as a part of our current research on the development of new routes to prepare heterocyclic systems,^{32–35} the performance of CuO/ZnO nanocatalysts in the click synthesis of 1,2,3-triazoles from benzyl halides and terminal alkynes in water was investigated (Scheme 1).

2. Results and Discussion

The catalysts are synthesized by a co-precipitation method and characterized by BET surface area, XRD, SEM, TEM and EDS analysis. Figure 1, shows the XRD analysis of ZnO and 20CuO/ZnO samples.

There are several peaks attributed to ZnO at a wide range of $2\theta = 31.7, 34.3, 36.3, 47.3, 56.4, 62.8, 66.2, 67.8, 69.0, 72.5$ and 76.9 degrees which are characteristics of (100), (002), (101), (102), (110), (103), (200), (112),

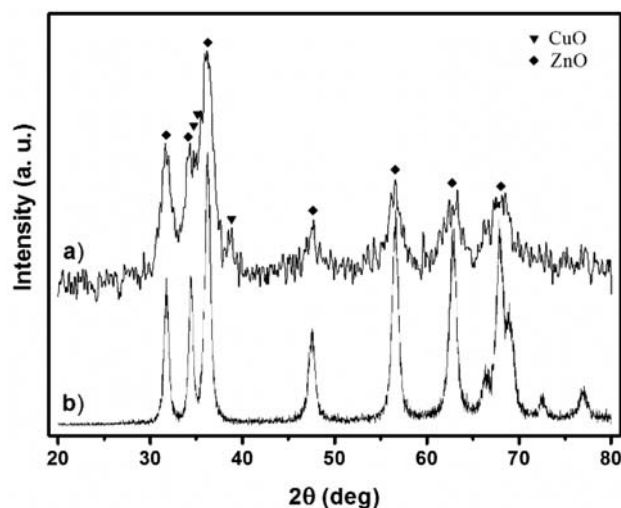


Figure 1. XRD patterns of (a) ZnO, and (b) 20CuO/ZnO nanocatalyst.

Table 1: Structural properties of prepared samples.

Sample	Crystallite Size ^a (nm)		S_{BET} (m ² /g)	Pore volume (cm ³ /g)	Pore Size (nm)
	D_{CuO}	D_{ZnO}			
ZnO	–	23	53	0.13	22.6
20CuO/ZnO	8	12	89	0.32	17.9

^a Average CuO crystal size calculated by XRD.

(201), (004) and (202) planes based on JCPDS card No. 89-1397. For the 20CuO/ZnO catalyst, the diffraction peaks at $35.5, 35.6, 38.7$ and 38.92° , are attributed to (002), (11 $\bar{1}$), (111) and (200) planes of cubic CuO, respectively (cod No. 05-0661). The peaks for ZnO crystals in 20CuO/ZnO have lower intensity and are broadened after addition of CuO to the ZnO. That indicates a lower crystallinity and smaller crystallite sizes of ZnO crystallites, maybe due to the incorporation of Cu in to the structure of ZnO. As shown in Table 1, the ZnO crystallite size is about 23 and 12 nm for ZnO support and 20CuO/ZnO catalyst, respectively. Also, the average crystallite size of CuO in the catalyst is about 8 nm. The structural properties of prepared samples are presented in Table 1.

The BET surface area of ZnO support is 53 m²/g, and as 20 wt% CuO is loaded on the support, the surface area increases to 89 m²/g. As shown in Table 1, after CuO loading on ZnO the pore volume of 20CuO/ZnO increases, while its pore size decreases, simultaneously. The N₂ adsorption/desorption profiles for ZnO and 20CuO/ZnO samples are shown in Figure 2a. According to the IUPAC classification, these isotherms can be categorized as type IV at the borderline with type II, with a type H3 hysteresis loop. This isotherm indicates the presence of mesopores with a pore size distribution continuing into the macropore domain³⁶ (Figure 2b). In addition, the type H3 hysteresis is usually observed on solids containing aggregates or agglomerates of some particles leading to slit-shaped pores, with nonuniform size and/or shape. Furthermore, the reflection point on ZnO occurs at around $P/P^0 = 0.86$, and by an increase of CuO it shifts to a lower value of around 0.8 for 20CuO/ZnO. These results suggested the smaller mesopores in the samples containing Cu content. Furthermore, higher amounts of N₂-adsorption on 20CuO/ZnO nanocatalyst can be related to mesopores with higher pore volume. These results are consistent with the average pore size and pore volume of the samples shown in Table 1.

Figure 3 shows the SEM analysis of ZnO and 20CuO/ZnO.

The ZnO micrograph (Figure 3a) exhibits an aggregation of nano-slit-like particles. However, the morphology of the catalyst after Cu loading is changed drastically. As can be seen, incorporation of Cu into the ZnO has a significant effect on the morphology of the catalyst. The 20CuO/ZnO sample exhibits an agglomerate of nanoparticles with a particle size of about 38 nm in diameter.

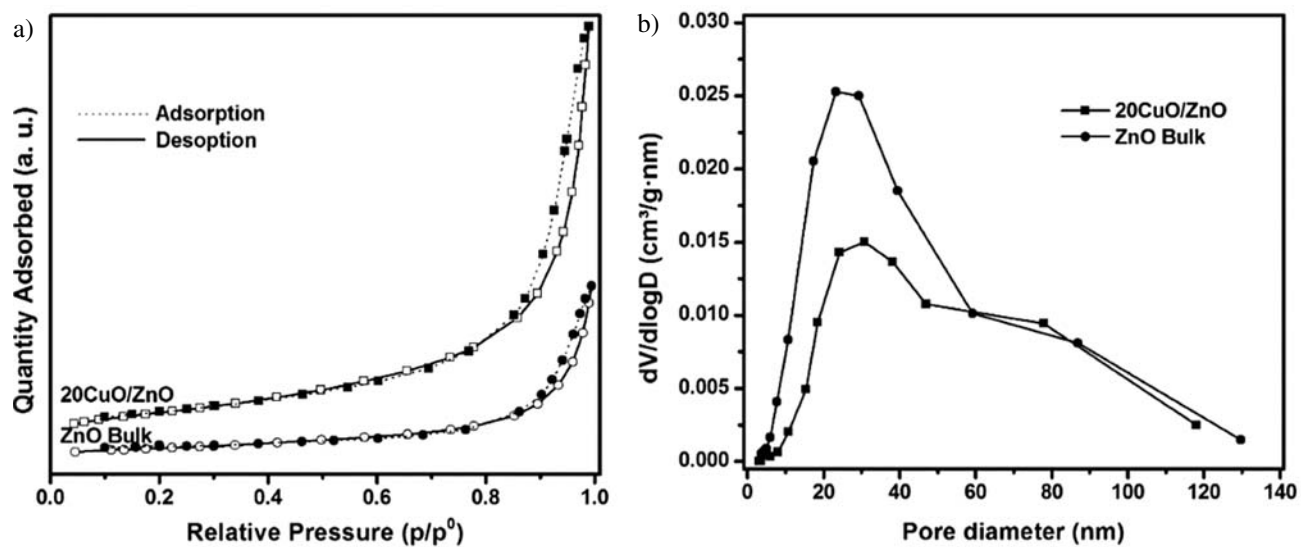


Figure 2. (a) N_2 adsorption/desorption isotherms, and (b) pore size distributions, of ZnO and 20CuO/ZnO samples.

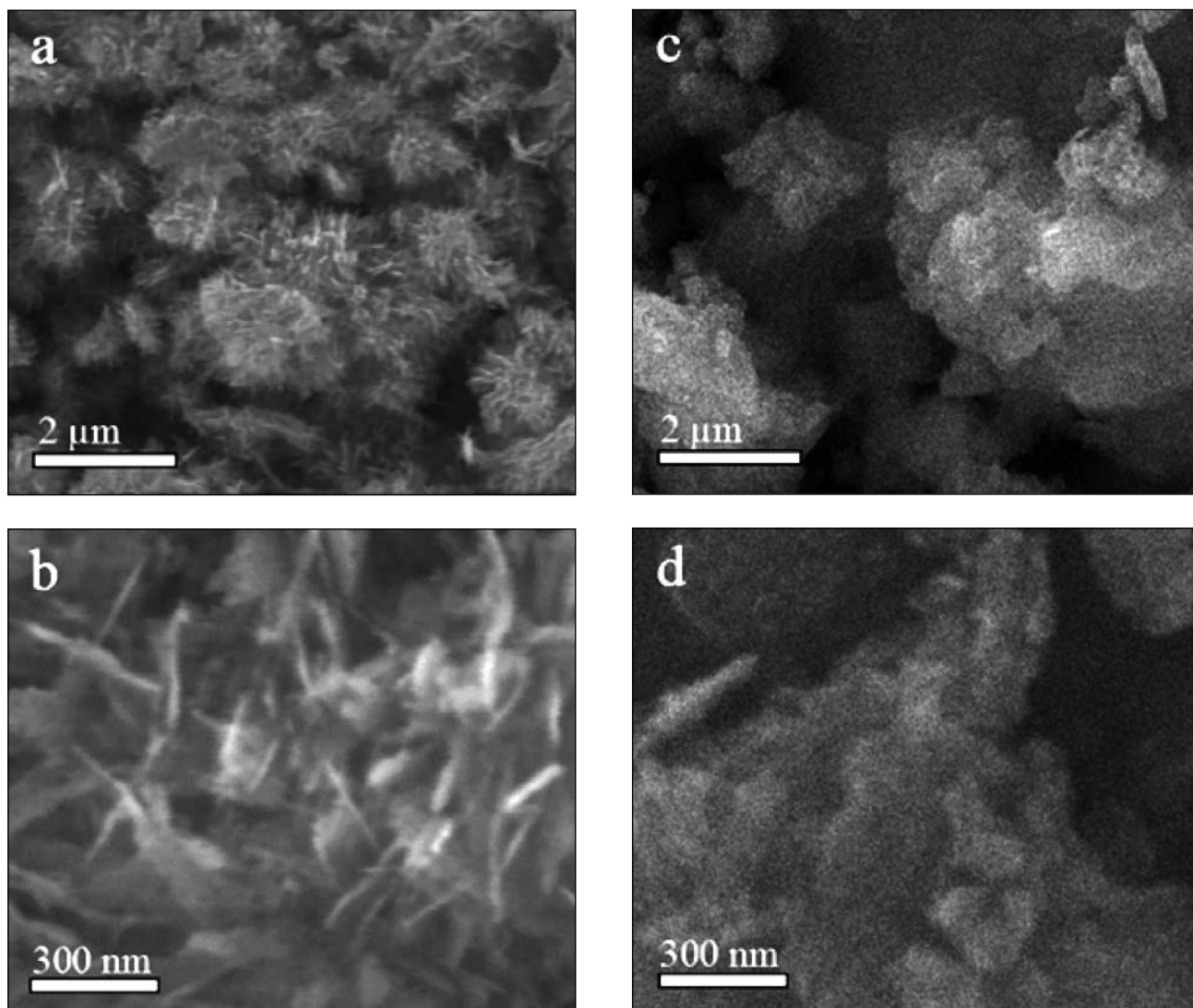


Figure 3. SEM micrographs of (a,b) ZnO, and (c,d) 20CuO/ZnO samples.

Figure 4a shows TEM image and CuO particle size distribution of 20CuO/ZnO. CuO nanoparticles are highly

Table 2. Effect of CuO loading on the yield of 1,2,3-triazoles.^a

Entry	CuO loading (wt.%)	Catalyst mass (g)	Time (min)	Yield (%) ^b
1	5	0.05	180	Trace
2	5	0.07	180	5
3	5	0.1	180	10
4	10	0.05	180	20
5	10	0.07	180	30
6	10	0.1	180	35
7	20	0.05	60	80
8	20	0.07	20	92

^a Reaction conditions: benzyl bromide (1 mmol), phenylacetylene (1 mmol), sodium azide (1.1 mmol) in water at reflux conditions. ^b Isolated pure products.

dispersed on the catalyst surface. Figure 4a-inset picture exhibits the CuO nanoparticle size distribution, by an average CuO particle size of about 4.4 nm. The HRTEM ima-

Table 3. Optimization of the reaction conditions.

Entry	Conditions ^a	Catalyst mass (g)	Time (min)	Yield (%) ^b
1	H ₂ O	–	120	–
2	H ₂ O	0.05	60	80
3	H ₂ O	0.07	20	92
4	CH ₃ CH ₂ OH	–	120	–
5	CH ₃ CH ₂ OH	0.05	90	50
6	CH ₃ CH ₂ OH	0.07	60	75
7	CH ₃ OH	0.07	90	75
8	CH ₂ Cl ₂	0.07	120	Trace
9	CH ₃ CN	0.07	120	Trace

^a Reflux conditions. ^b Isolated pure products.

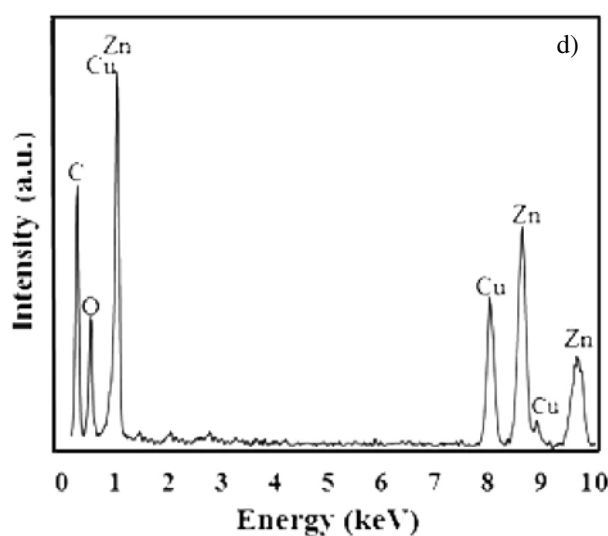
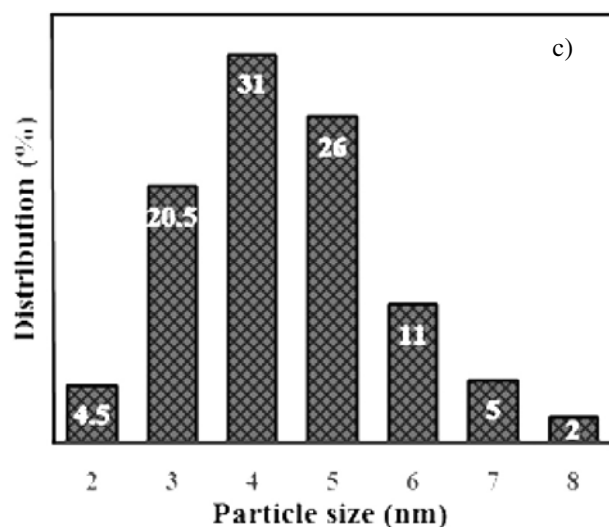
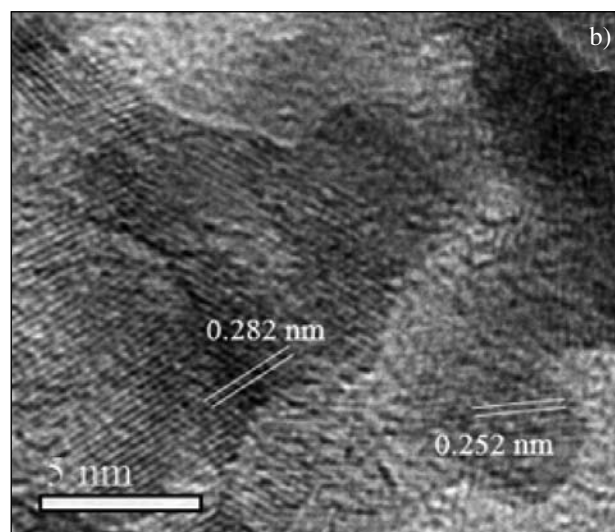
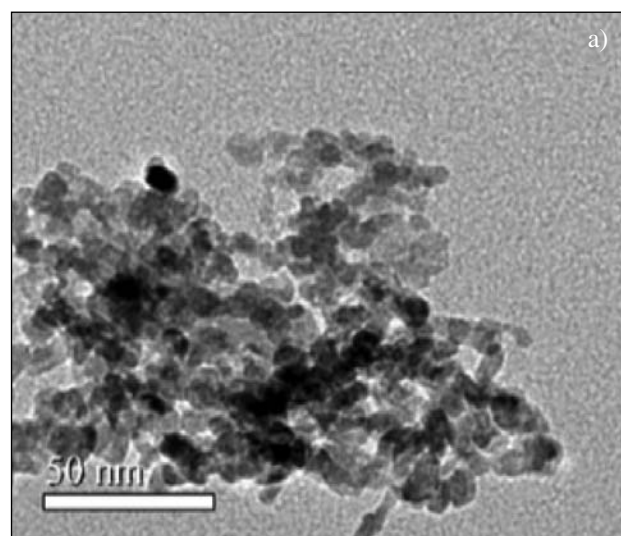
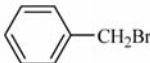
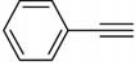
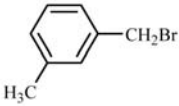
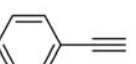

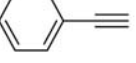
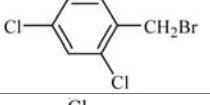
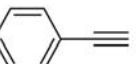
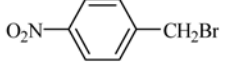
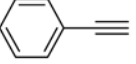
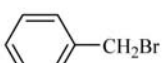
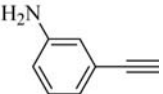

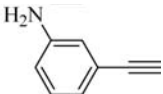
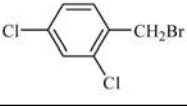
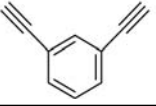
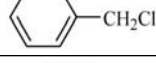
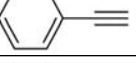
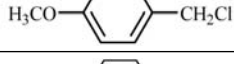
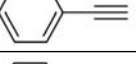
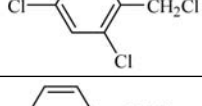
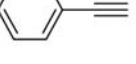
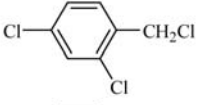
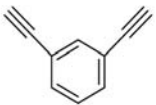
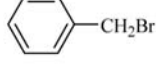

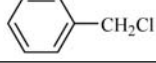



Figure 4. (a,b) TEM image, (c) particle size distribution, and (d) EDS analysis, of 20CuO-ZnO nanocatalyst.

Table 4. Synthesis of 1,2,3- triazoles catalyzed by 20CuO/ZnO.

Entry	Substrate	Alkyne	Time (min)	Yield (%) ^a	m.p. (°C) ^b
1			20	92	128–130
2			20	91	109–111
3			15	92	117–119
4			35	90	148–149
5			35	91	140–141
6			30	89	136–137
7			20	90	139–141
8			45	89	188–190
9			30	92	127–129
10			18	89	117–119
11			60	91	148–150
12			65	90	188–190
13			35	89	77–79
14			45	89	77–79

^a Isolated pure products. ^b Products were characterized by comparison of their spectroscopic data and melting points with those reported in the literature.^{19,35}

ge of 20CuO/ZnO is shown in Figure 4b. The reflections with *d*-spacing values of 0.282 and 0.252 nm correspond to ZnO (100) and CuO (11 $\bar{1}$) lattice planes.

For the optimization of reaction conditions, various parameters such as CuO loading, catalyst amount and solvent were investigated. Initially, the reaction between ben-

zyl bromide, phenylacetylene and sodium azide, in the presence of the catalyst as a model reaction was investigated (Scheme 1). The performance of various CuO/ZnO nanocatalysts containing 5, 10 and 20 wt.% CuO in the synthesis of 1,2,3-triazoles were investigated. As shown in Table 2, with an increase of CuO content, the activity of

the catalysts increases, and the 20CuO/ZnO showed the highest yield of reaction (about 92%), in a relatively short reaction time (20 min). Also, in the presence of ZnO-blank and CuO-bulk no products were obtained. These results demonstrated the effect of CuO nanoparticles on the synthesis of 1,2,3-triazoles. Also, it should be noted that no product was obtained in the absence of the catalyst (blank test) under these reaction condition.

Table 3 shows that the best results was achieved by the reaction of benzyl bromide, phenylacetylene and sodium azide (1: 1: 1.1 mol/ratio) in the presence of 0.07 g of the catalyst in water as the solvent under reflux condition (Table 3). The other solvents such as ethanol, methanol, CH₃CN, and CH₂Cl₂ resulted in lower yields.

The activity of various terminal alkynes and benzyl halides under optimized conditions is summarized in Table 4. It is clear that a substitution with electron withdrawing or electron donating groups on the phenyl rings did not have a significant effect on the result of the reaction. It should be noted that the corresponding triazoles were obtained with high yields and regioselectivities. Moreover, the obtained products can be isolated by a simple filtration and recrystallized from a mixture of ethanol/water (3:1 v/v).

Furthermore, the recyclability of the catalyst for the reaction of benzyl bromide and phenylacetylene under optimized conditions was investigated. After completion of each reaction, the spent catalyst was recovered by filtration. Then it was washed with hot ethanol, dried and used for the next run. The recovered catalyst showed a sustained performance after 5 consecutive runs with no loss of activity. Also, the results demonstrated high selectivity and high yields of corresponding products in the five consecutive runs (Table 5).

Table 5. Recyclability study of 20CuO/ZnO nanocatalyst.

Run	1	2	3	4	5
Time (min)	20	22	22	25	32
Yield (%) ^a	92	92	91	90	88

^a Isolated pure products.

3. Experimental

Chemicals were purchased from Merck. Products were characterized by comparison of their spectroscopic data (¹H NMR, ¹³C NMR and IR) and physical properties with those reported in the literature. NMR spectra were recorded in DMSO-*d*₆ or CDCl₃ on a Bruker Avance DPX 500 and 400 MHz spectrometers. IR spectra were recorded on a BOMEMMB-Series 1998 FT-IR spectrometer. All yields refer to isolated products. The Bruker AXS D8 advanced diffractometer carried out the XRD analysis of the catalyst. The sample was scanned over the range of

2θ = 10–80° using Cu Kα radiation. The specific surface area of samples was determined by nitrogen adsorption-desorption using BET method, using an automated gas adsorption analyzer (Tristar 3020, Micromeritics). The morphology of the catalyst was investigated comprehensively by scanning electron microscopy (SEM) and high resolution transmission electron microscopy (HRTEM) techniques using a HITACHI S-4160 instrument, as well as JEOL JEM-2100 (200 kV) microscope equipped with an EDS analytical system.

3. 1. Preparation of the Catalyst

The CuO/ZnO catalysts containing 5 to 20 wt.% CuO, were prepared by co-precipitation method. A Na₂CO₃·H₂O solution was added drop-wise into an appropriate mixture of 0.03 M Cu(NO₃)₂·3H₂O and Zn(NO₃)₂·6H₂O solutions under continuous mixing. The slurry was kept at pH 8.5 for 15 min at 65 °C, then filtered and washed with deionized water. The precipitates were dried for 12 h at 100 °C followed by calcination at 300 °C for 3 h. The final catalyst is denoted as *x*CuO/ZnO where *x* is the weight percent of the copper oxide in the catalyst.

3. 2. General Procedure

A mixture of benzyl halide (1 mmol), terminal alkyne (1 mmol), sodium azide (1.1 mmol), and proper amount of the catalyst was stirred in water under reflux condition for the appropriate time (Table 3). After reaction completion, the mixture was cooled, filtered and added to hot ethanol. The filtrate was evaporated and dried. Then, the obtained solid was recovered in a hot ethanol/water (3:1 v/v) and the catalyst was washed with hot ethanol, dried and applied for consecutive runs.

4. Conclusion

In this research CuO/ZnO nanocatalysts as efficient recyclable catalysts were introduced for the click synthesis of 1,2,3-triazoles under reflux conditions in water. The catalysts demonstrated a satisfactory yield of about 88% after 5 consecutive runs, with sustained activity. Clean procedure, easy work-up, short reaction time and high yields of corresponding products make this method an attractive procedure for the synthesis of 1,2,3-triazoles.

5. Acknowledgement

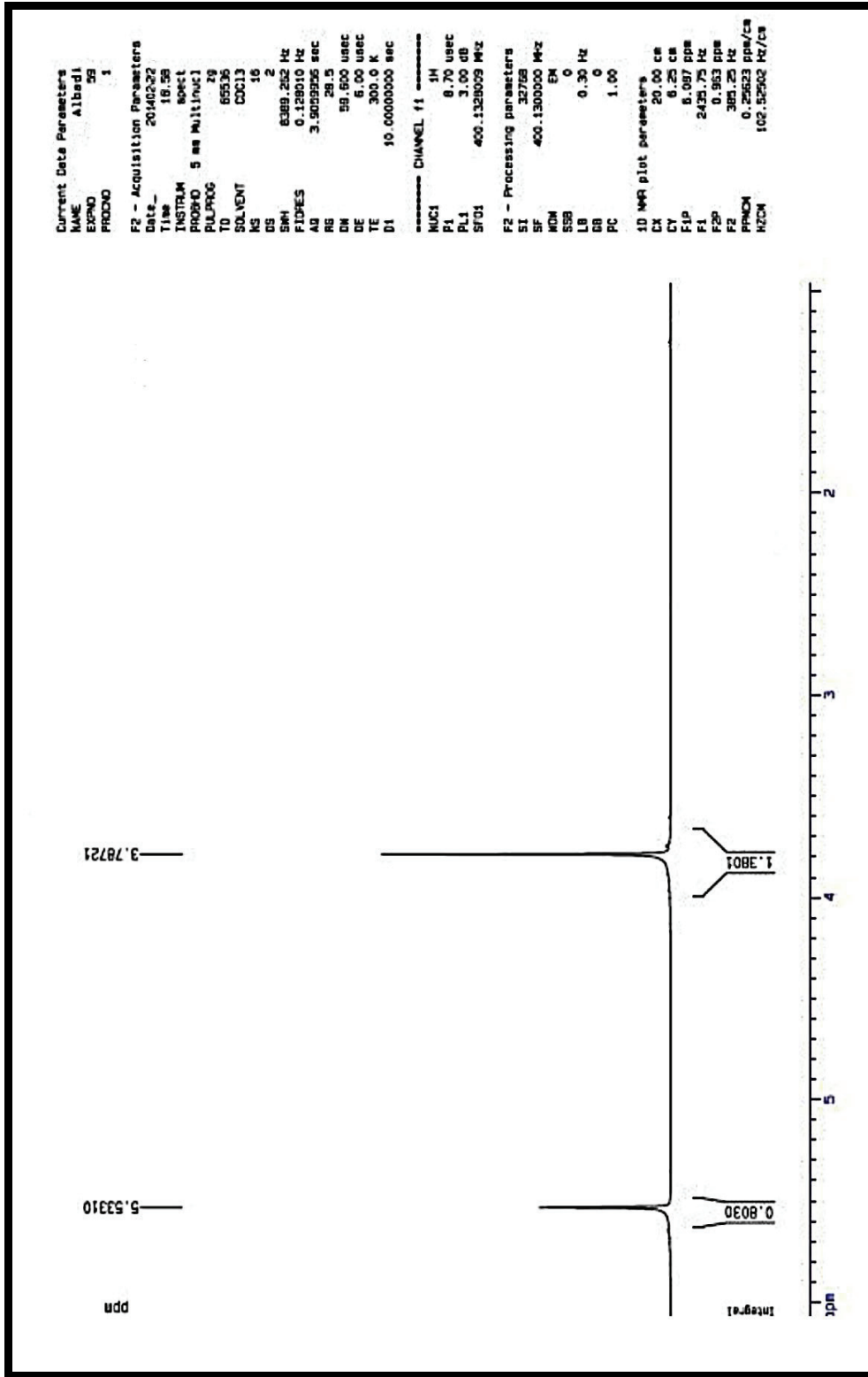
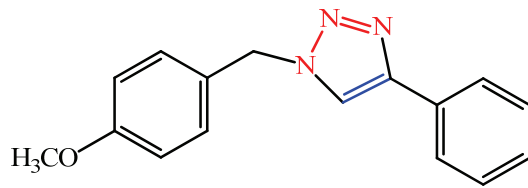
We are thankful to research council of Behbahan Khatam Alanbia University of Technology for the support of this work.

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Povzetek

Poročamo o CuO/ZnO nanokatalizatorjih kot učinkovitih katalizatorjih za regioselektivno sintezo 1,2,3-triazolov iz benzil halidov in terminalnih alkinov v vodi. Ti katalizatorji so zelo učinkoviti poleg tega pa jih je mogoče tudi enostavno reciklirati. Pripravili smo jih z metodo so-obarjanja ter karakterizirali z določevanjem površine z BET metodo ter z XRD, SEM, TEM in EDS analizami. Raziskali smo učinek deleža CuO v katalizatorju, mase katalizatorja in lastnosti uporabljenega topila. Pokazali smo, da lahko katalizatorje recikliramo z enostavno filtracijo in uporabimo v ponovljenih reakcijah brez zmanjšane aktivnosti.



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 RG 28.5
 DM 59.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 10.0000000 sec

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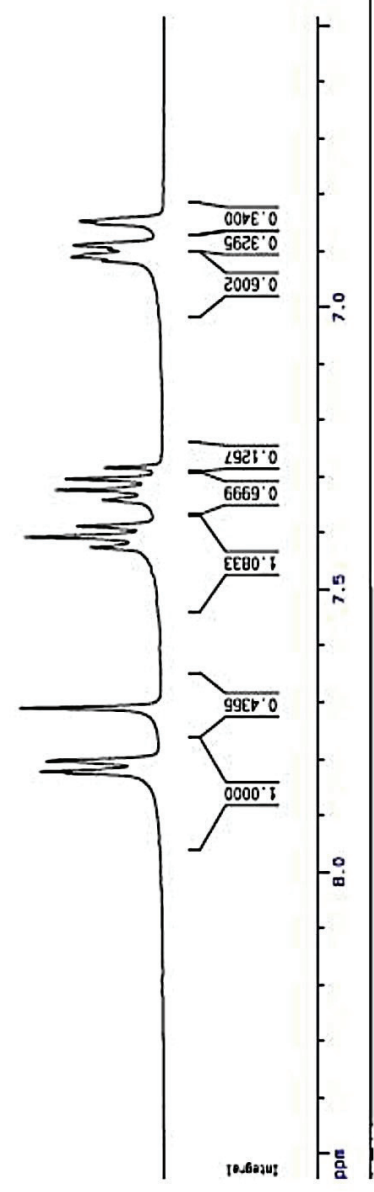
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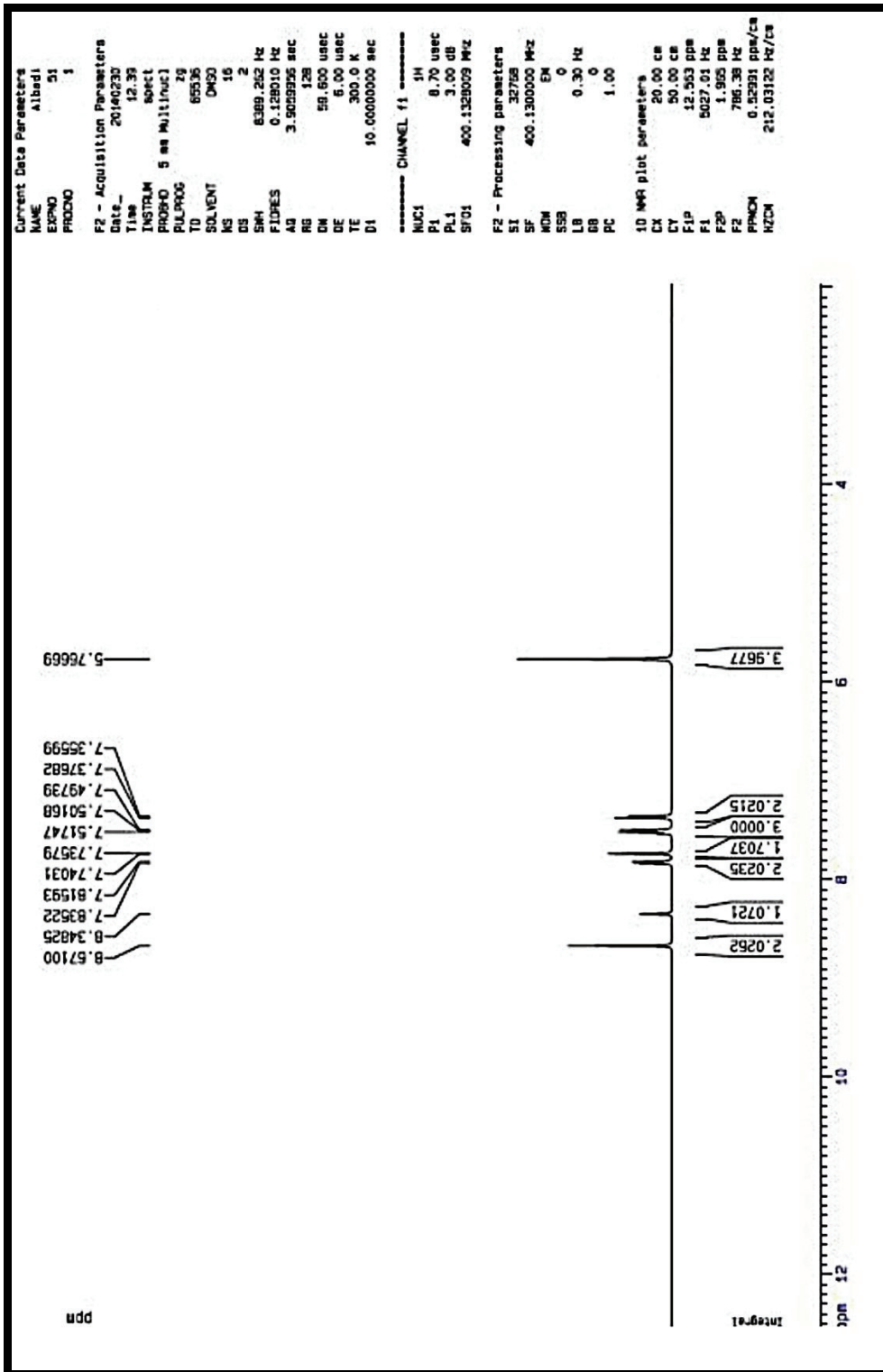
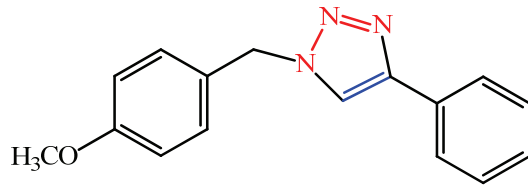
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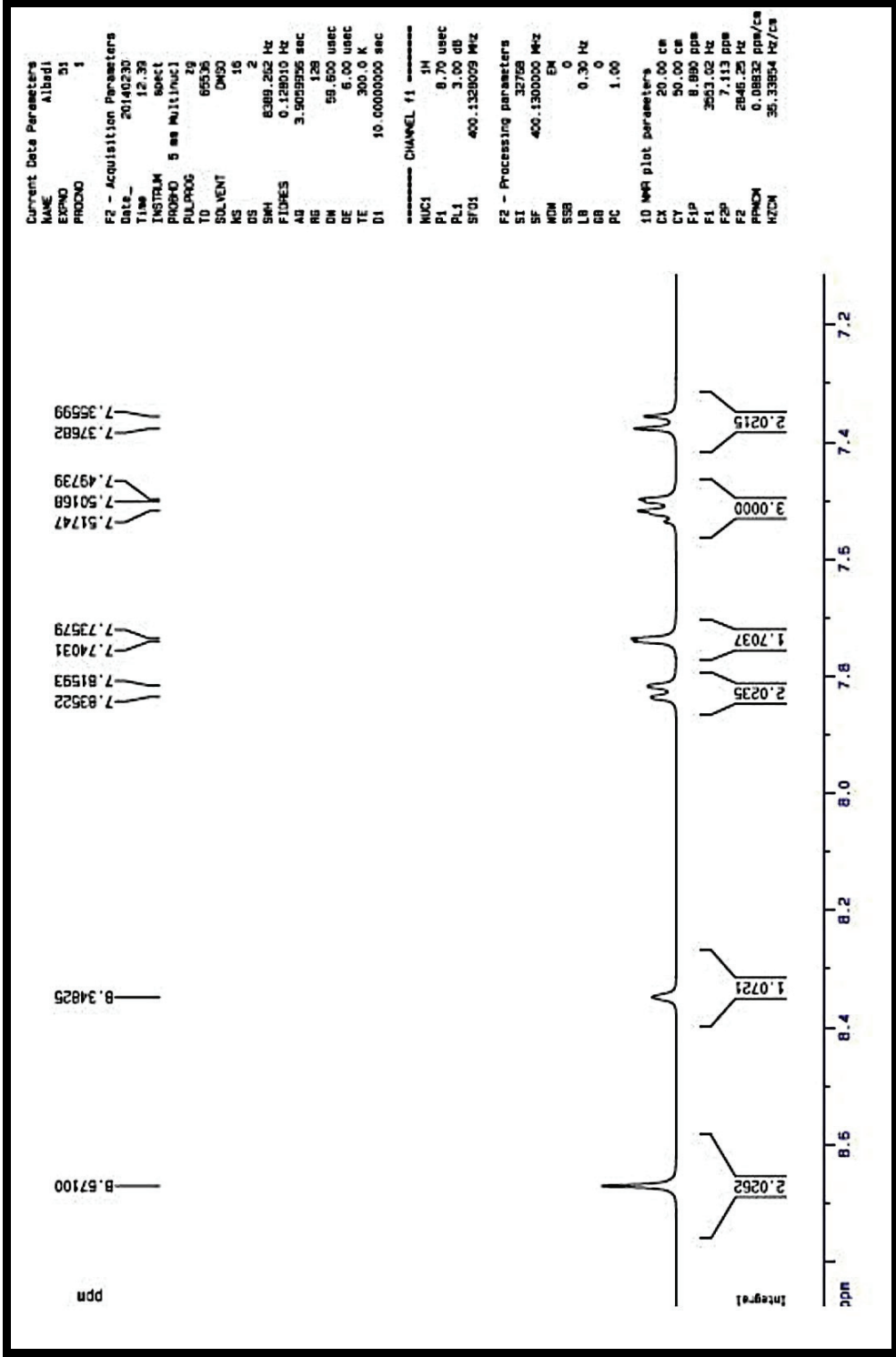
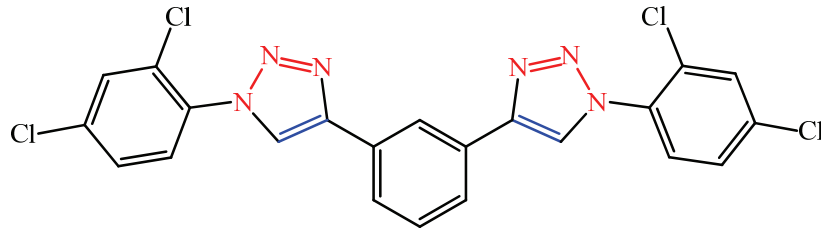
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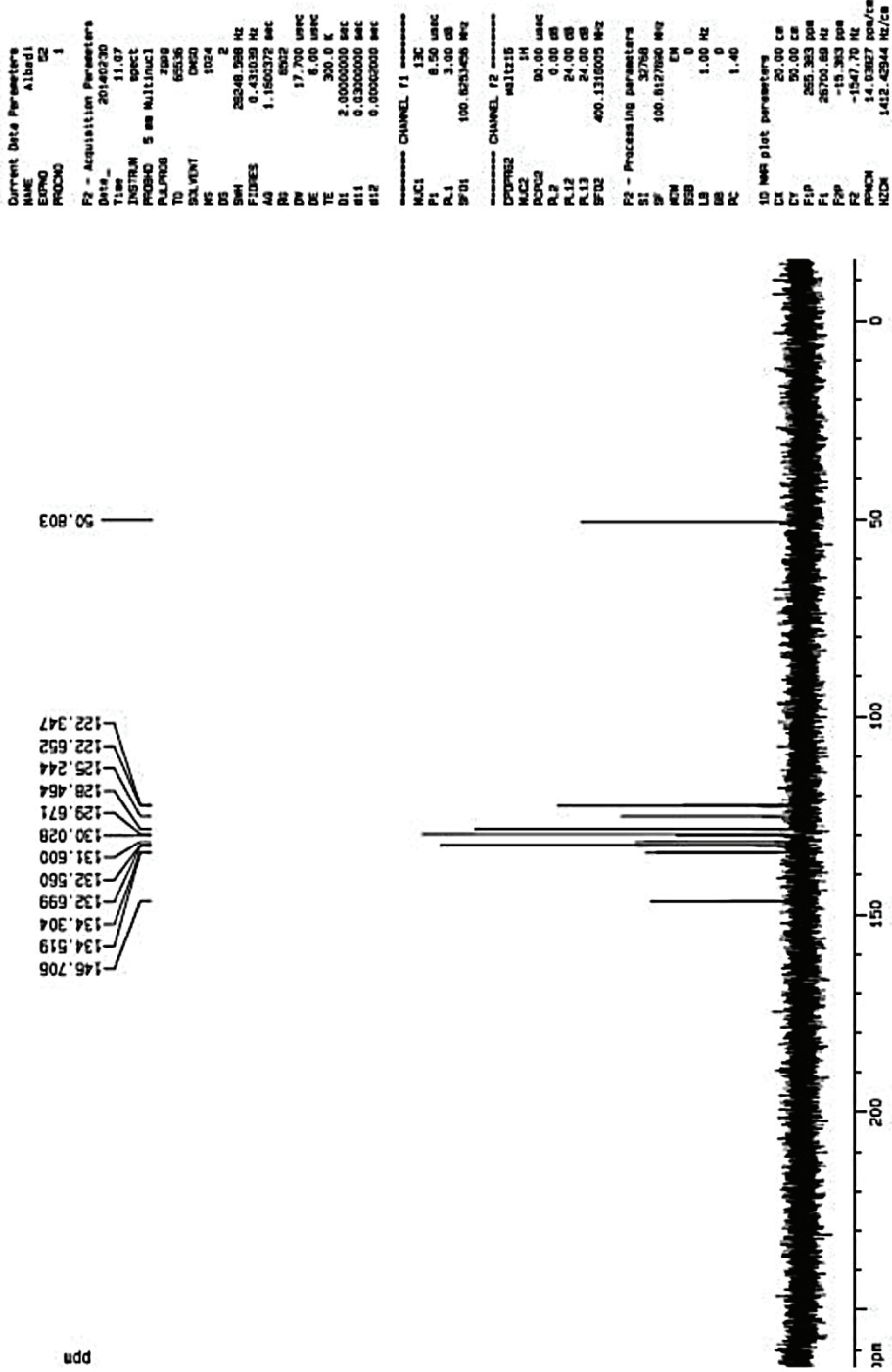
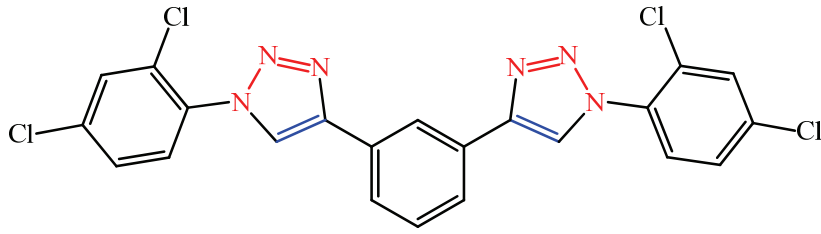
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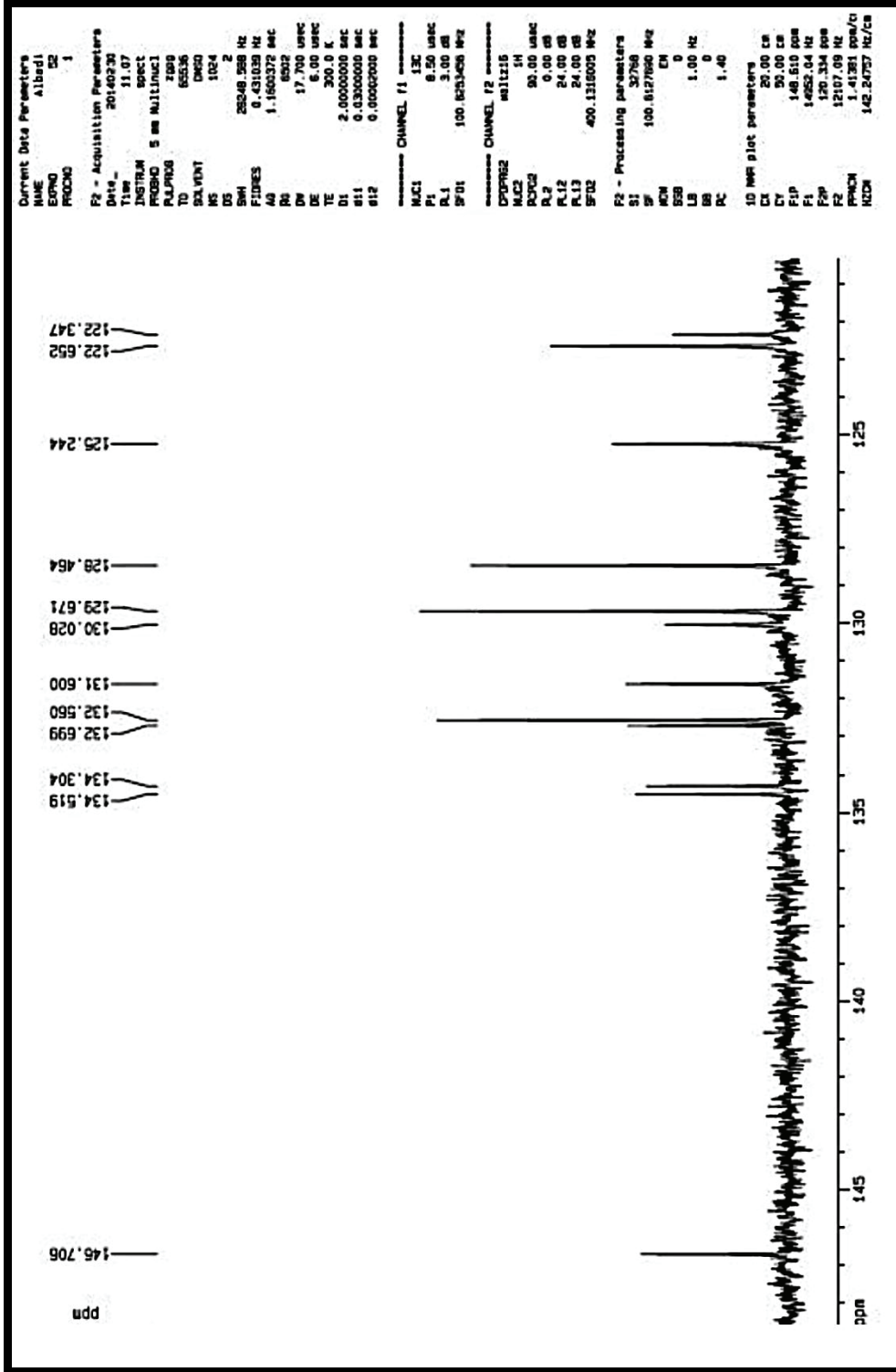
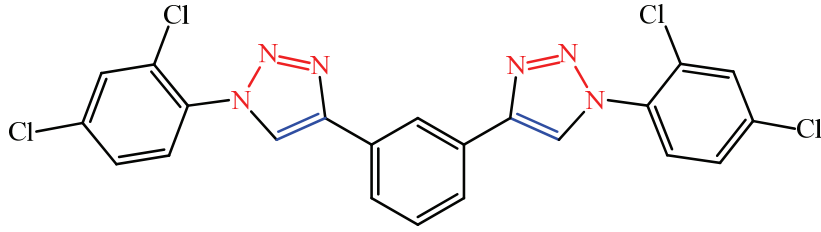


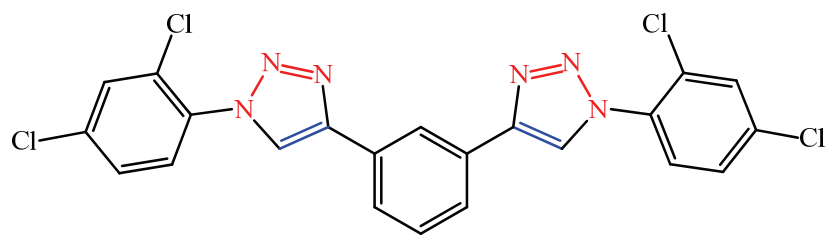
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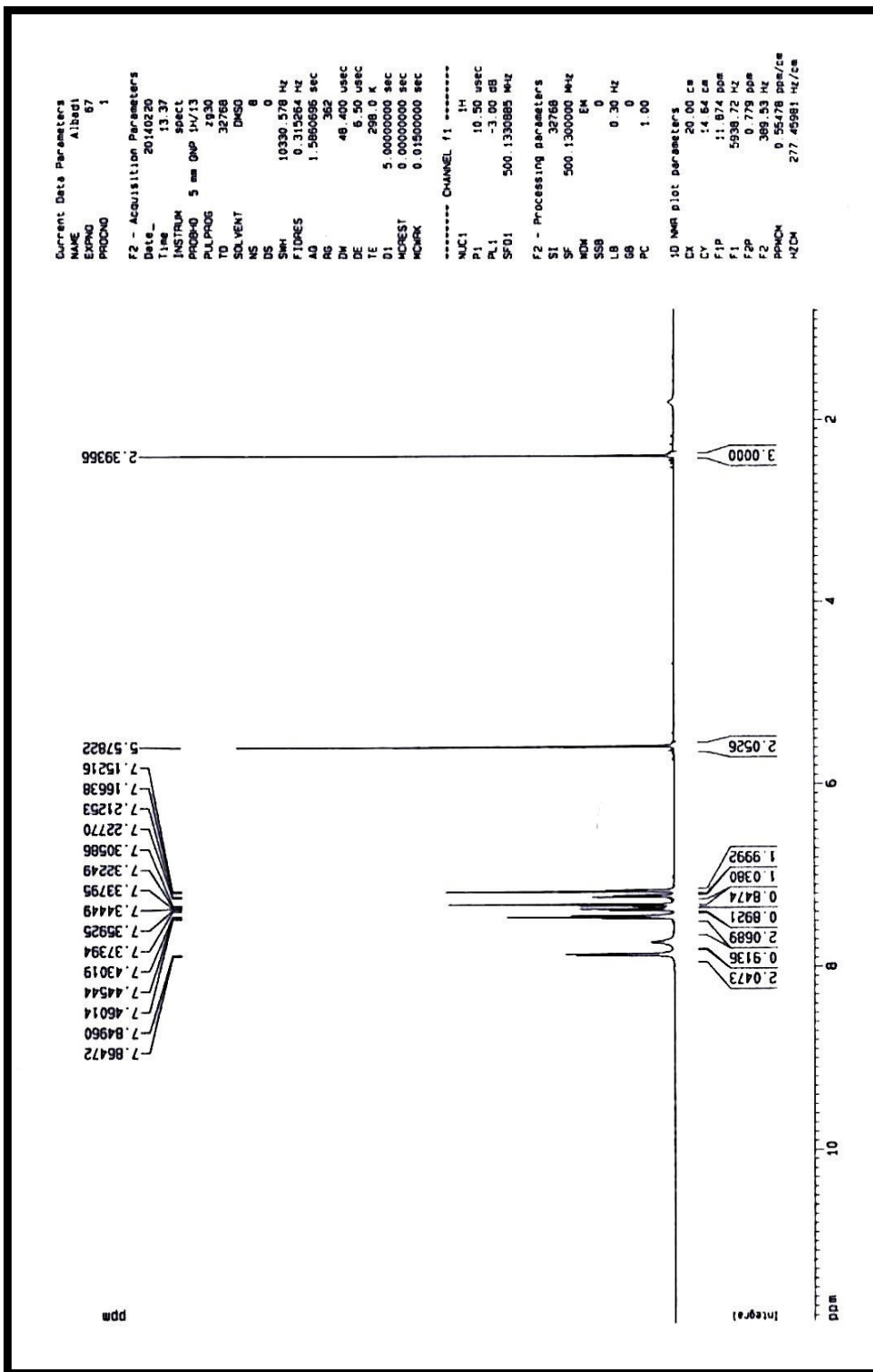
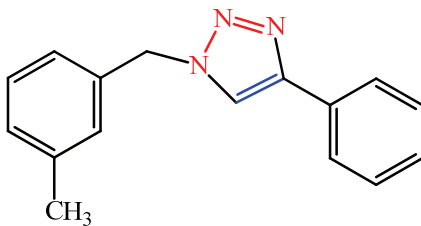


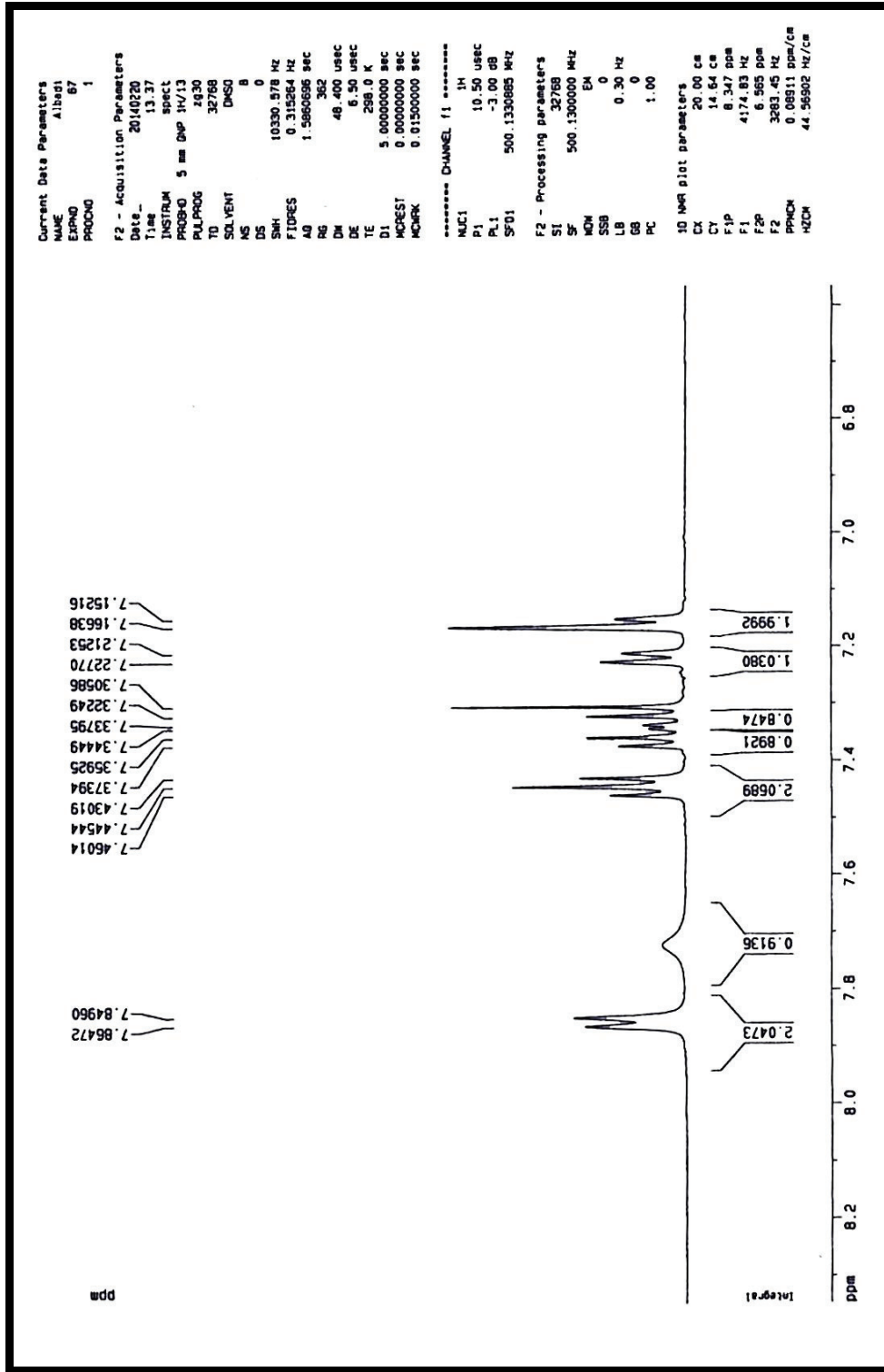
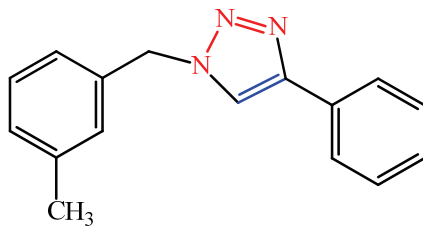


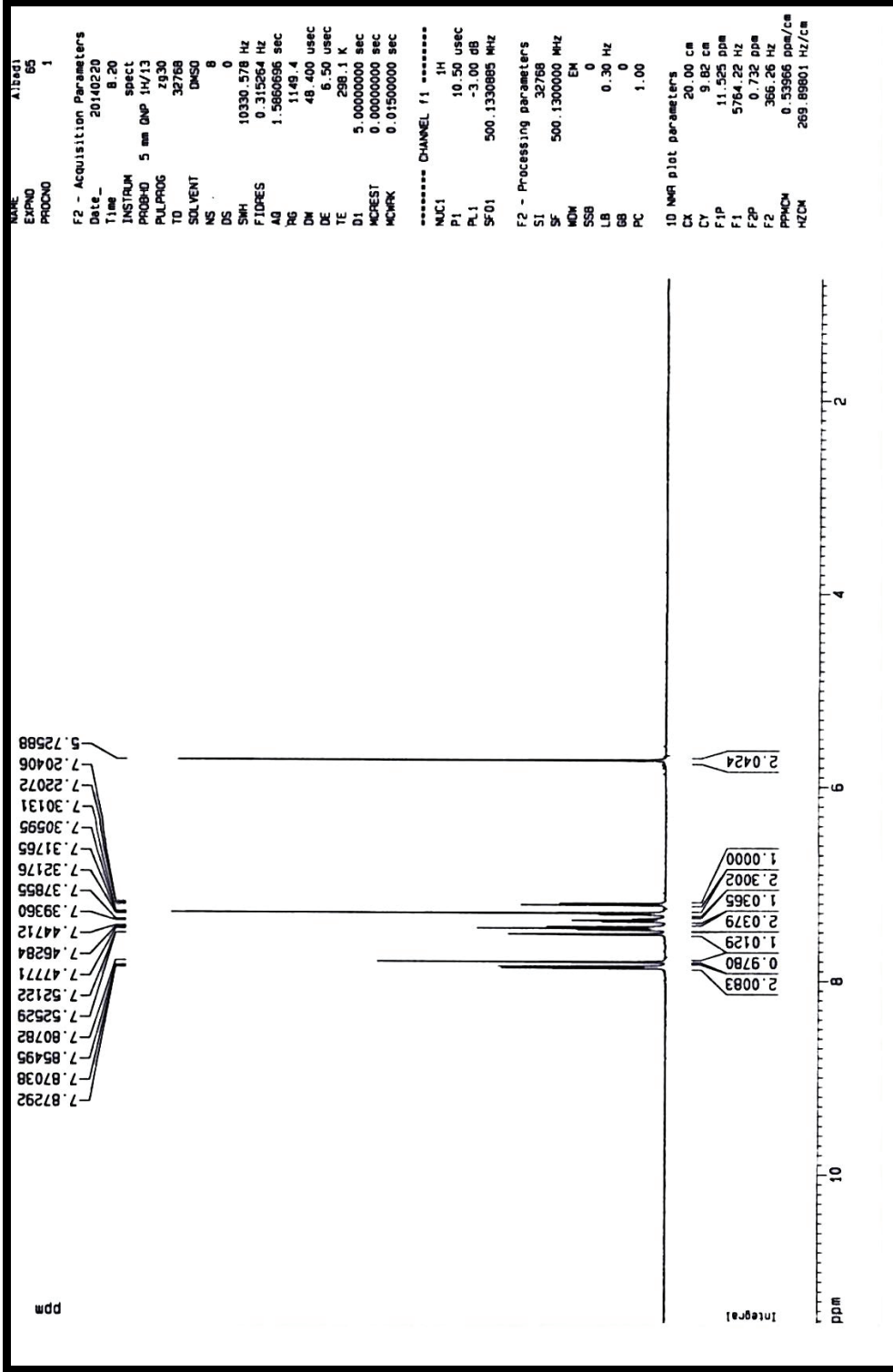
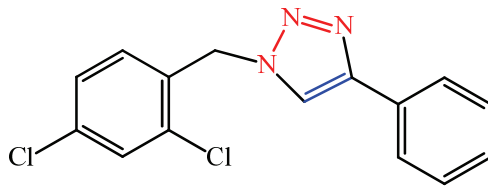


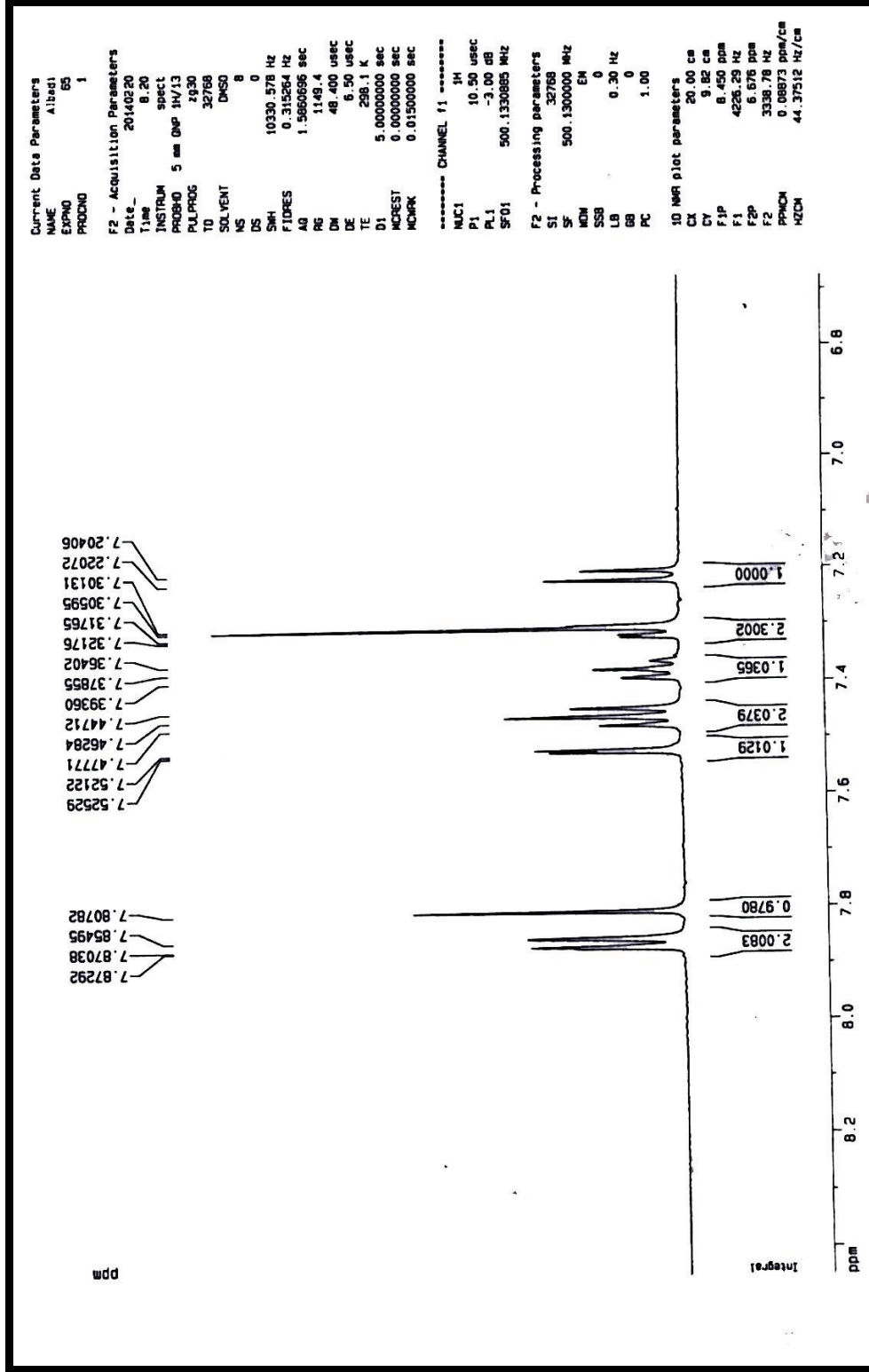
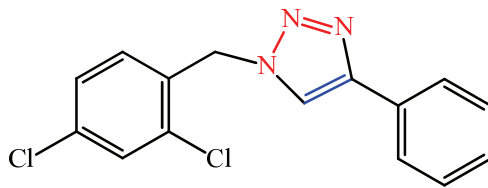


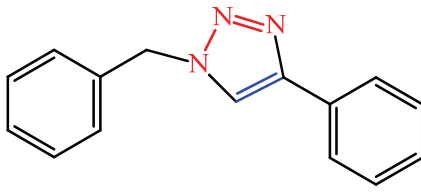












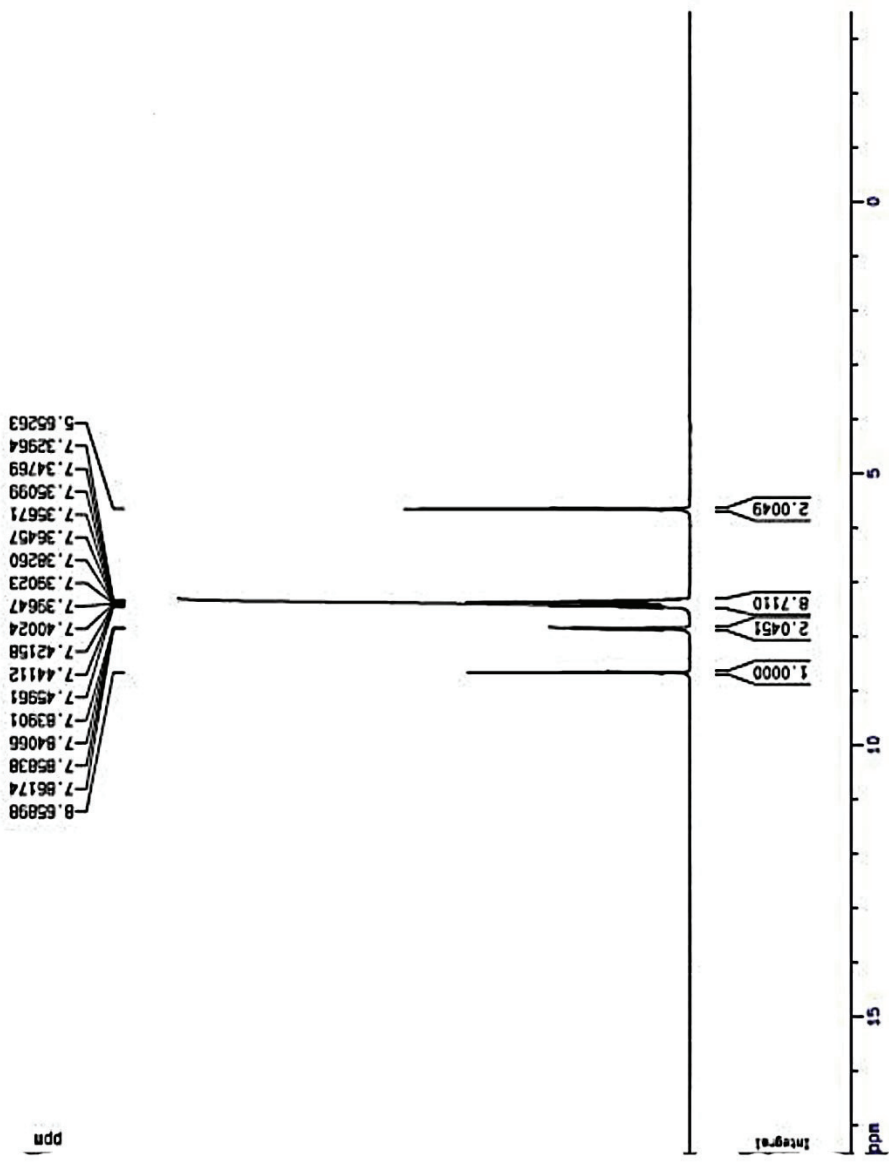
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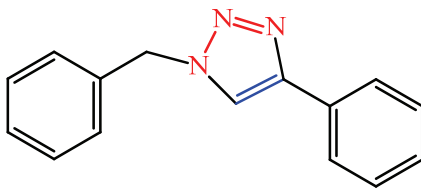
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 D1 10.0000000 sec

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Current Data Parameters
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 PROCNO 1

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