SUPPORTING INFORMATION

Copper nanoparticulates in guar-gum: a recyclable catalytic system for the Huisgen[3+2]-Cycloaddition of Azides and Alkynes without additives under ambient conditions

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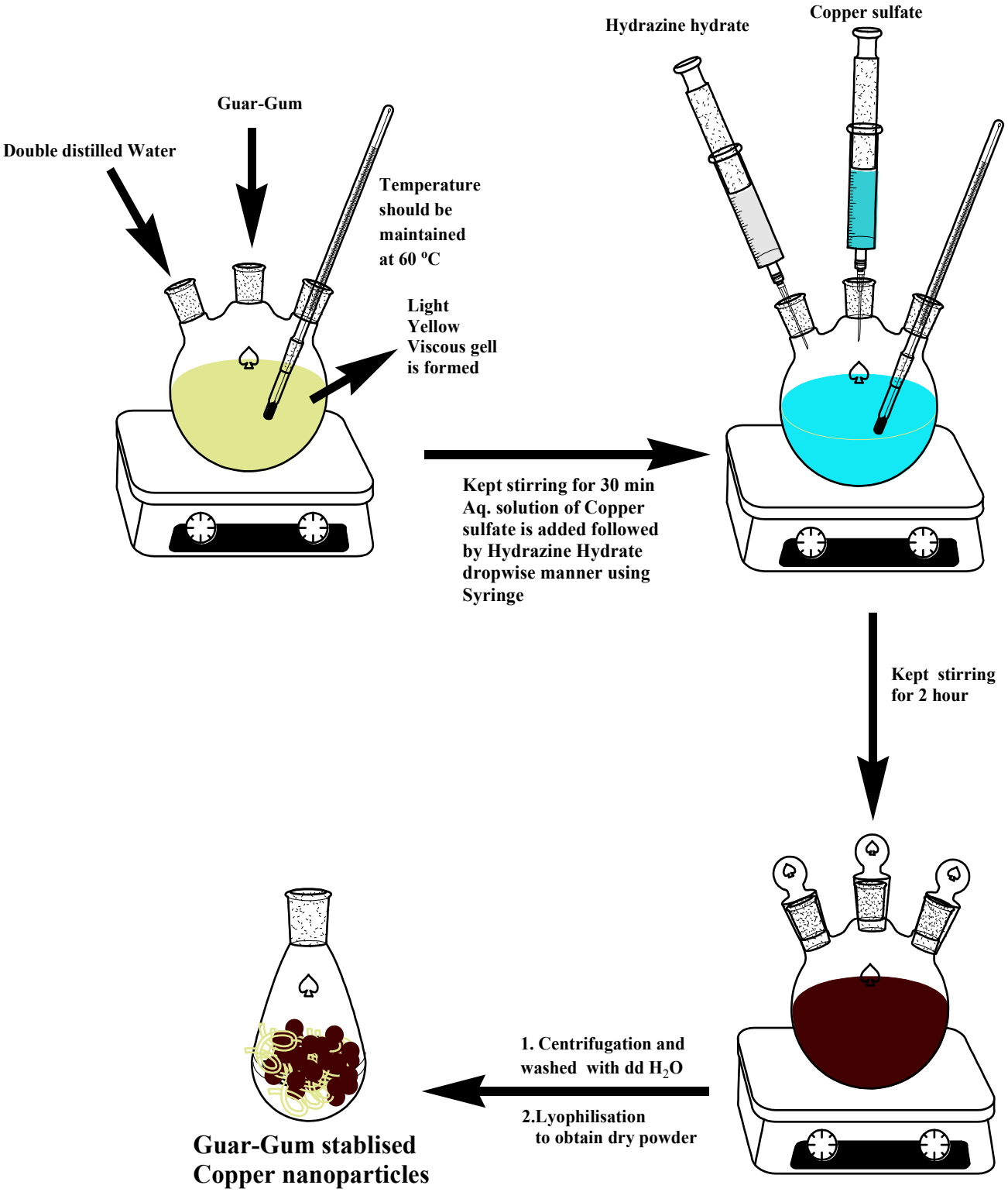
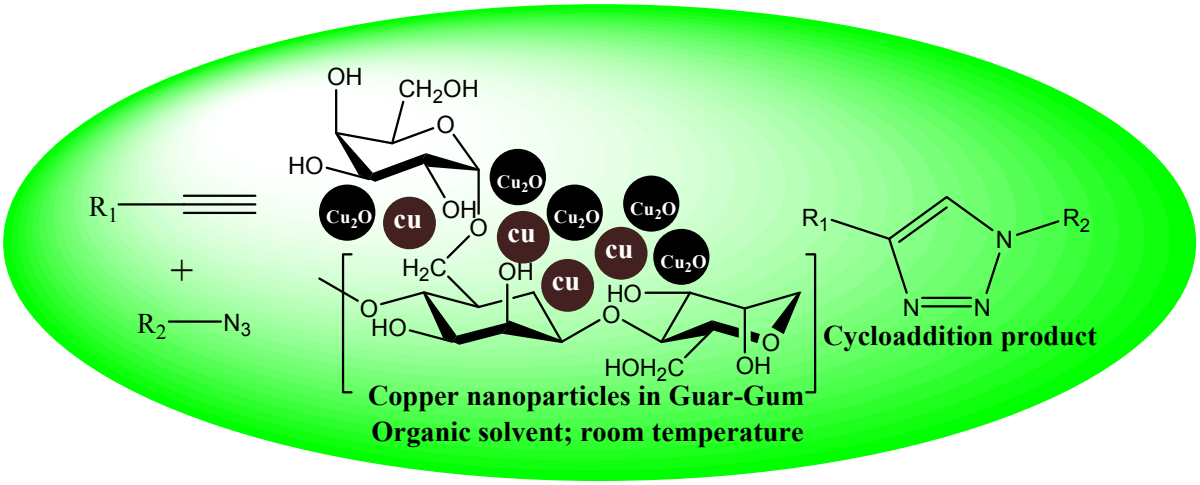
PREPARATION OF CATALYST (GUAR-GUM STABILIZED COPPER NANOPARTICLES FOR "CLICK REACTION")

The preparation of the catalyst for the "Click" reaction involves a very simple procedure described as follows (**Scheme 1**). In a 50 mL tri-neck round-bottom flask, 10 ml of double distilled water is added and kept on a magnetic stirrer maintaining the temperature at 60^{o}C. 50 mg Guar-gum powder is added slowly to form a viscous gel. By using a glass syringe, 1 mL (2% w/v) aqueous copper sulphate solution is added and the stirring is allowed until a viscous homogeneous solution is obtained. It is then purged with N_2 gas to eliminate the dissolved oxygen present in the solution (As the concentration of Guar-gum used has been very less, the direct purging of N_2 gas through the solution does not pose any problem). Aqueous solution of hydrazine hydrate (5% v/v) as reducing agent is then added to reduce the copper sulfate to copper which finally get embed in the highly viscous polymeric Guar mesh (**Scheme 1**). The reaction mixture is centrifuged at 10000 r.p.m for 10min and washed with double distilled water to remove the unentrapped particles and ions which is further subjected to lyophilisation so as to obtain a dry catalyst in powder form and this is kept in desiccators for further use. The synthesized catalyst can be characterized with various techniques such as TEM, UV-Visible spectroscopy, QELS, X-Ray Diffraction, Electron diffraction, EDAX, IR, etc.Copper nanoparticles Synthesized in the mesh of the viscous Guar-gum gel have been characterized to evaluate the morphology, shape and size of the nanoparticles. From the TEM analysis (**Figure 1**) it has been observed that particles are very small, less than 20 nm in diameter and are spherical in shape. Size of the nanoparticles has been further confirmed by dispersing the nanoparticles in the absolute ethanol and carrying out the QELS analysis (**Figure 2**). The results obtained have been in agreement with the results obtained with TEM analysis. To confirm the morphology of the synthesized nanoparticles, selected area electron diffraction (SAED) analysis of the samples was carried out (**Figure 3**) and it was found that the particles are crystalline in nature which is further confirmed by X-ray diffraction study (**Figure 4**). In order to confirm the entrapment of copper in the mesh of Guar gum, lyophilized particles have been analyzed using UV-Vis spectroscopy (**Figure 5**) and EDAX. The data obtained have revealed the presence of copper in the mesh of Guar-gum.



Scheme.1: Carton representation of general procedure for the synthesis of Guar-gum stabilized copper nanoparticles for "Click cycloaddition".



Scheme 2: Synthesis of 1, 2, 3-Triazole using Guar-gum capped nanoparticles.

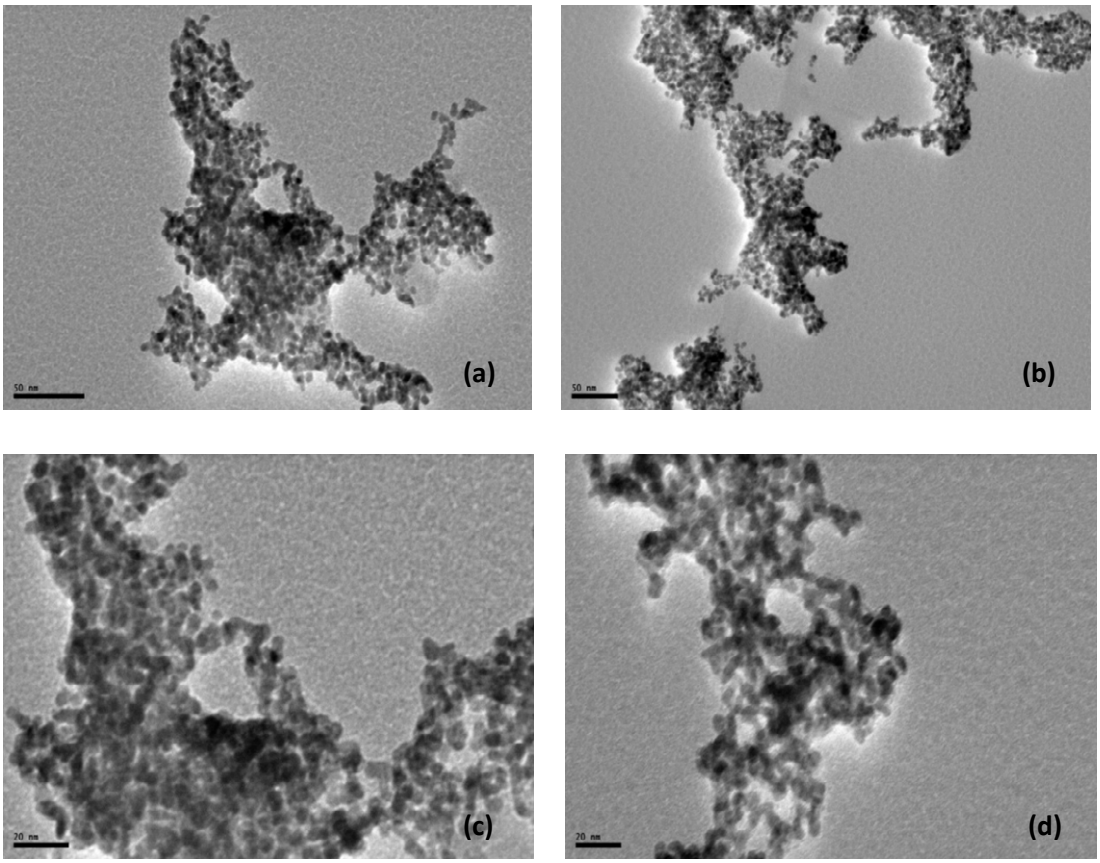
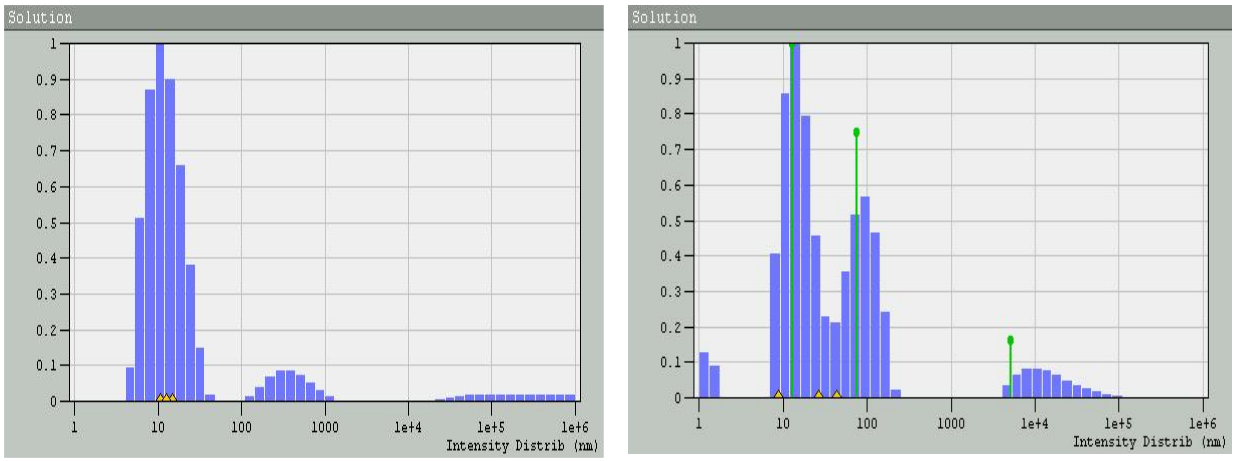


Figure 1: (a-d) TEM images of copper nanoparticles stabilized under the viscous mesh of Guar-gum gel.



[A] **[B]**

Figure 2: QELS data plot of population distribution in percentile versus size distribution in nanometres (nm)
[A] Before reaction [**B**]after reaction

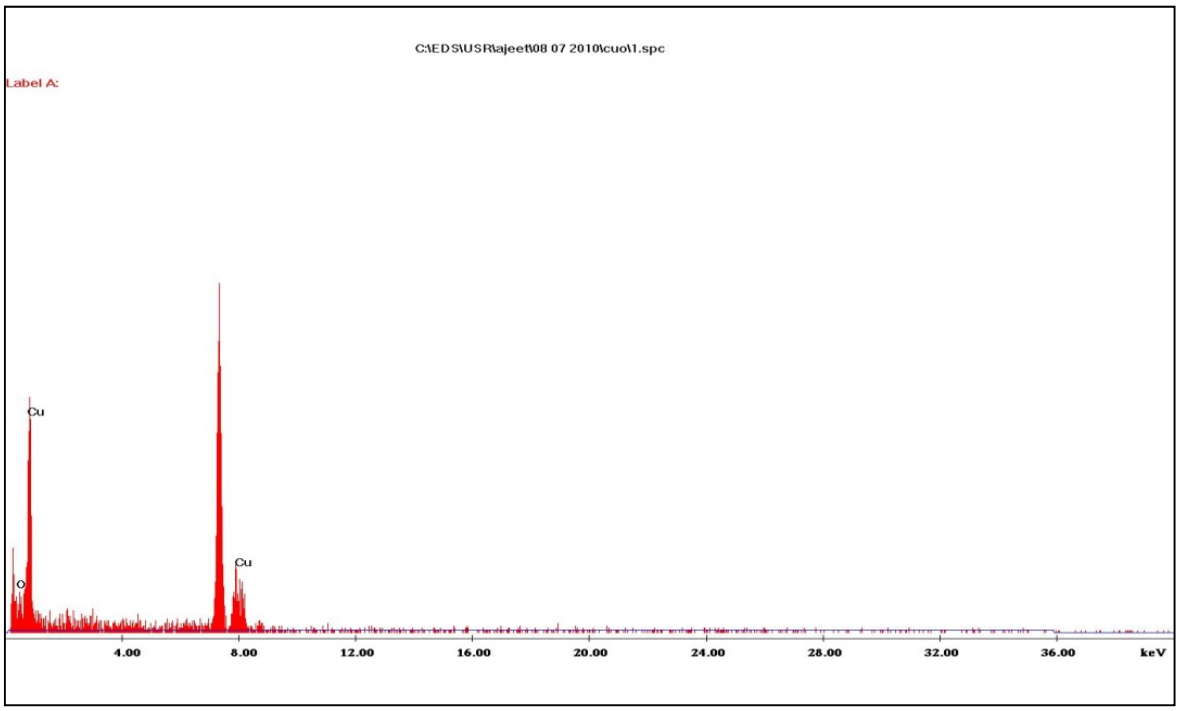


Figure 3: EDAX of copper nanoparticles stabilized under the viscous mesh of Guar-gum gel.

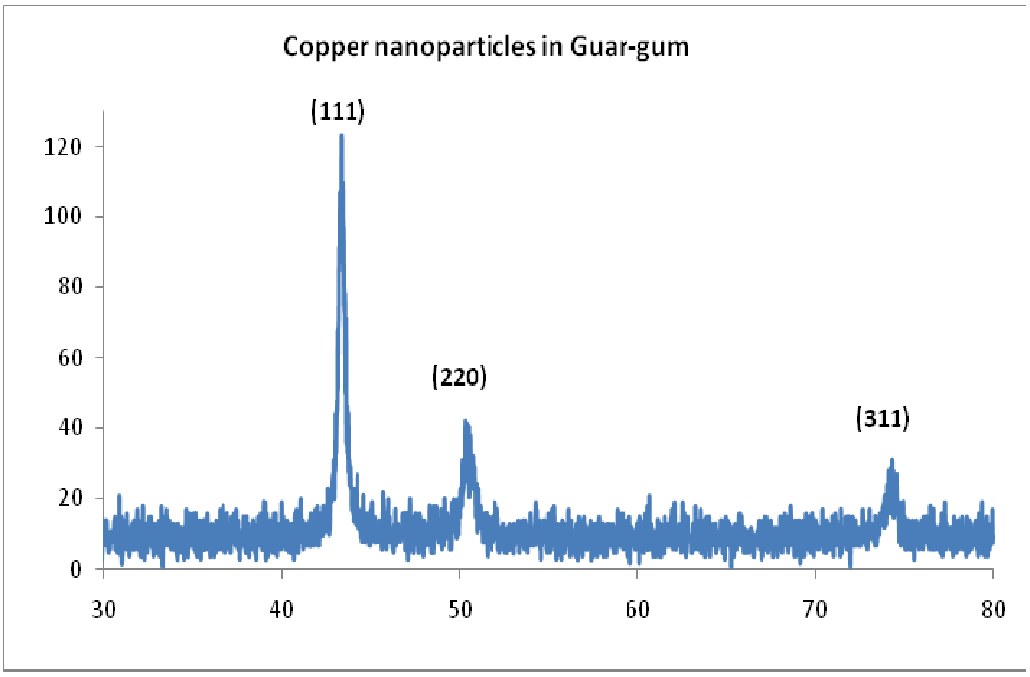


Figure 4: X-ray diffraction of copper nanoparticles stabilized under the viscous mesh of Guar-gum gel.

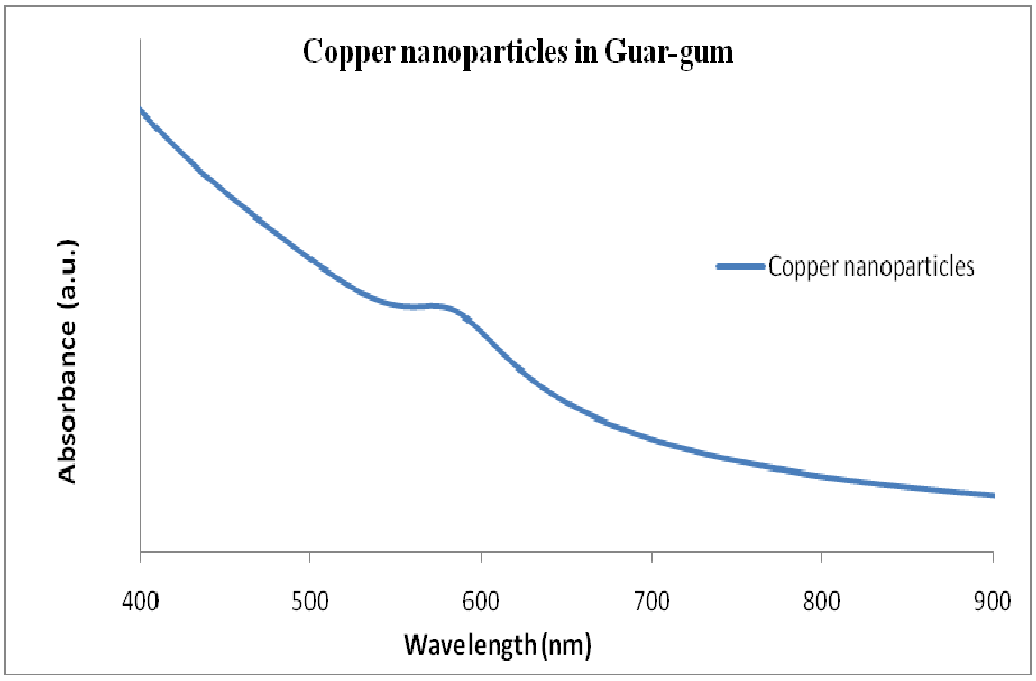


Figure 5: UV-absorption spectra of copper nanoparticles stabilized under the viscous mesh of Guar-gum gel.

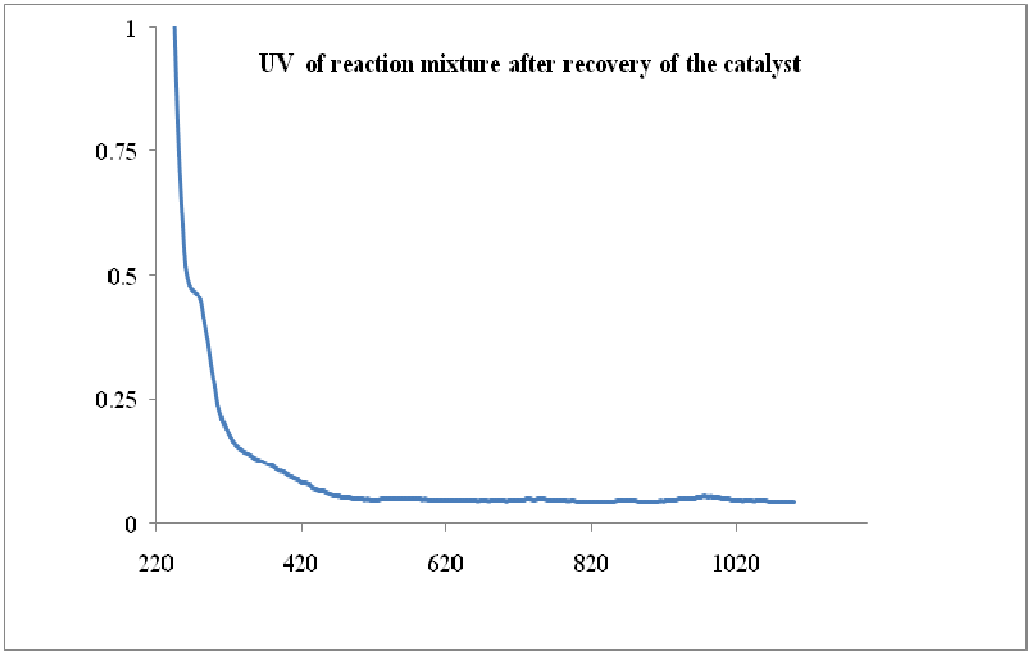


Figure 6: UV-absorption spectra of reaction mixture after recovery of catalyst

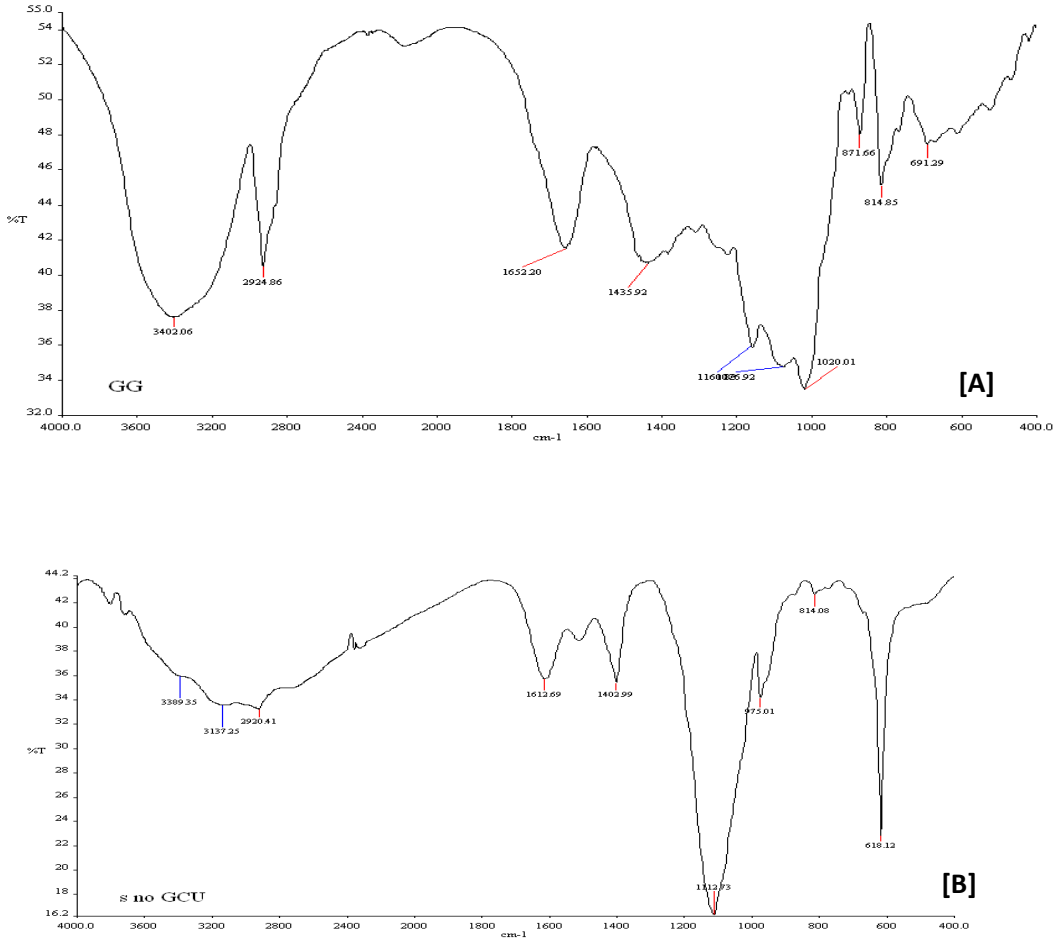
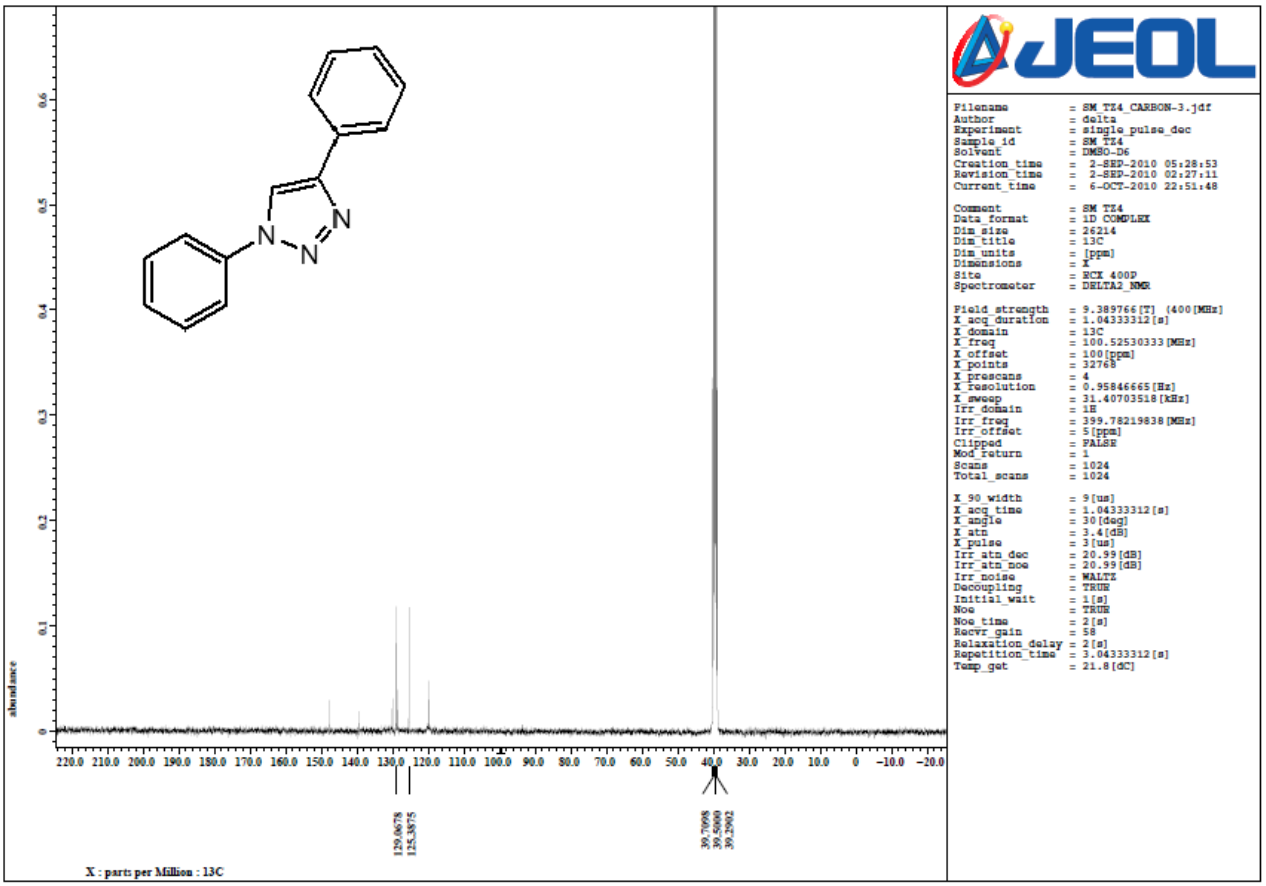
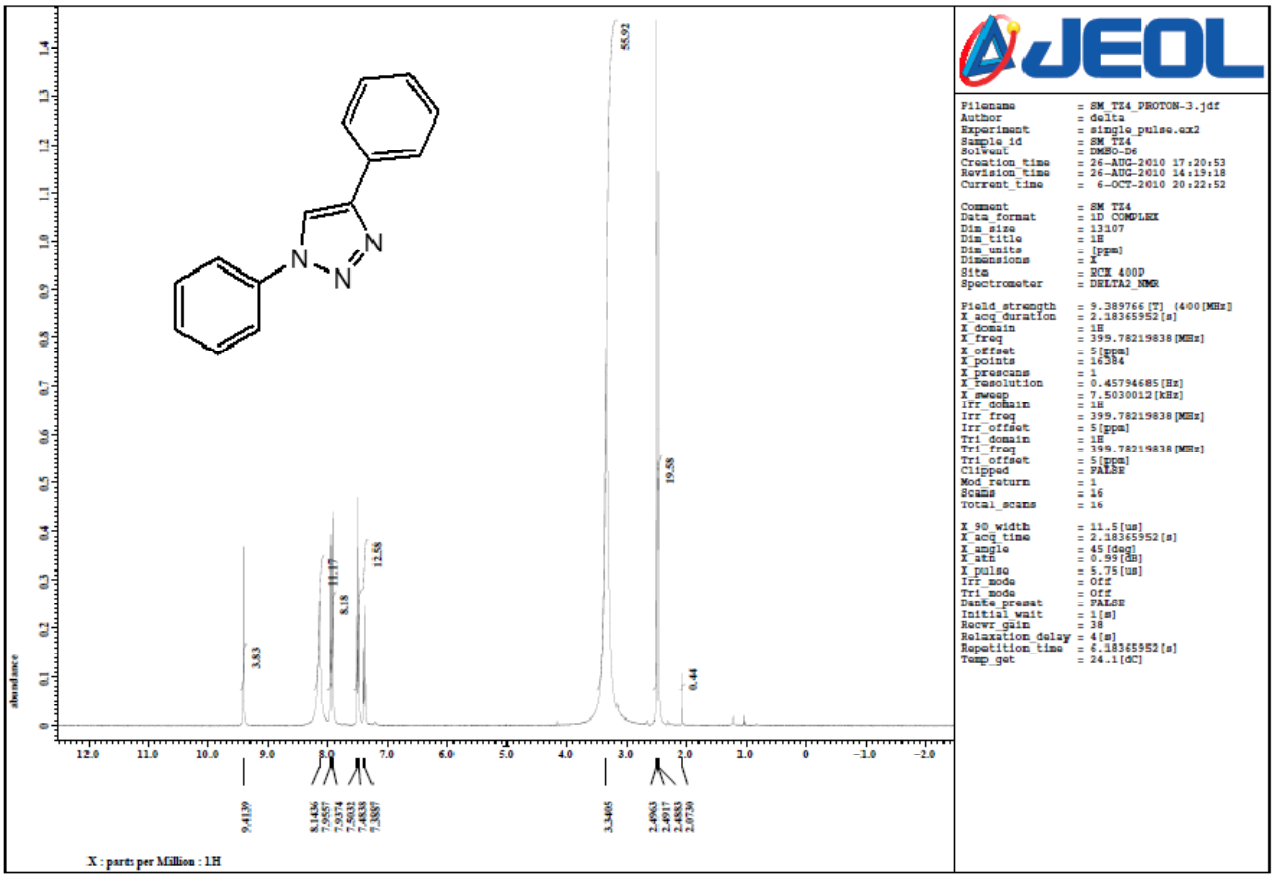
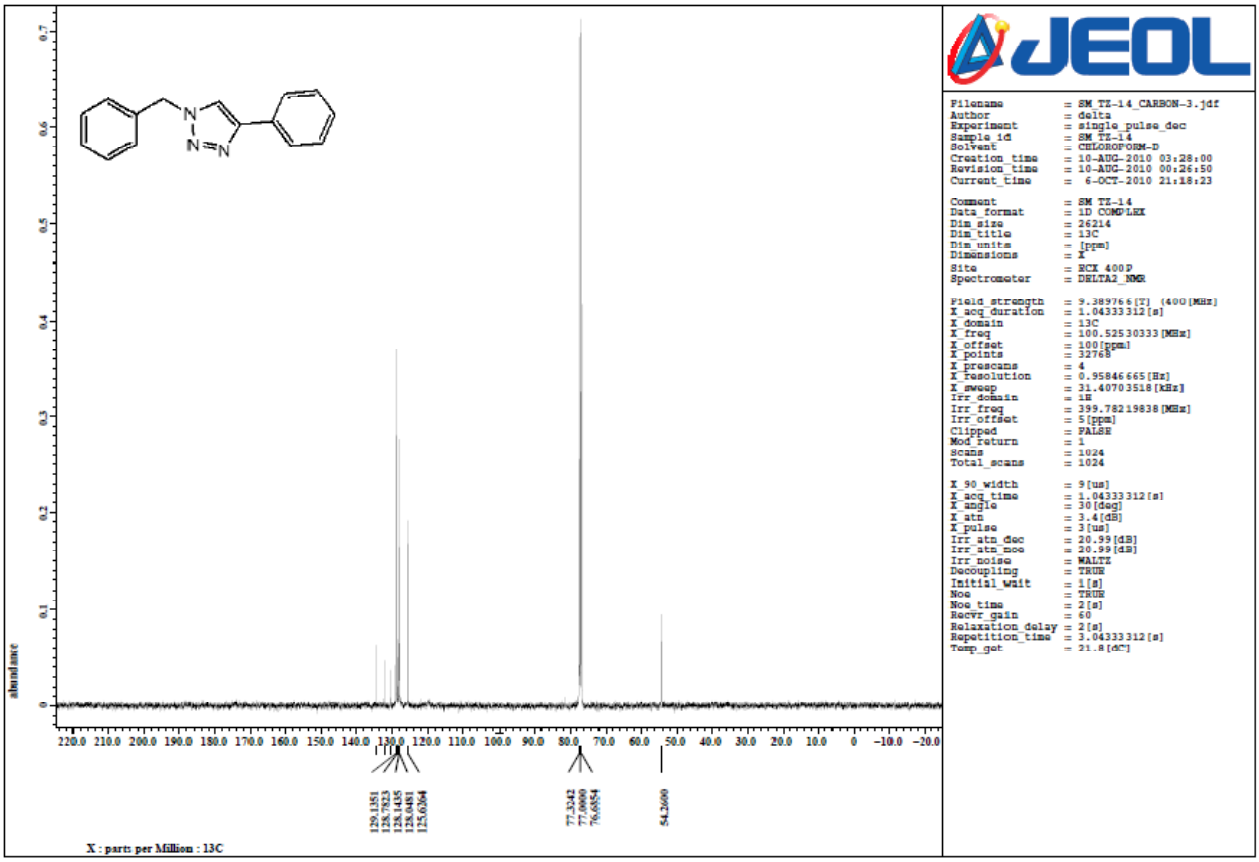


Figure 7: IR spectra of [a] Guar-gum [b] Cu particles in Guar-gum







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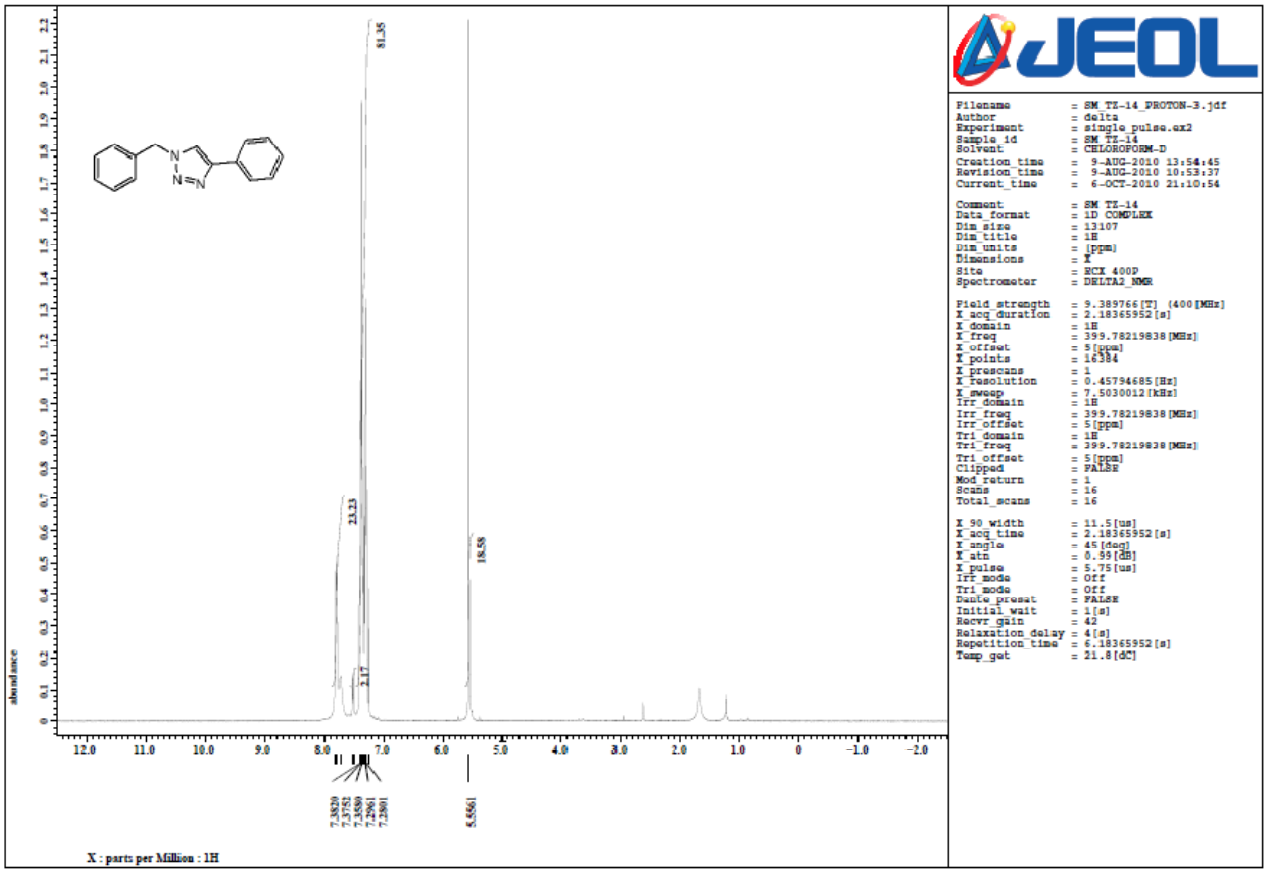

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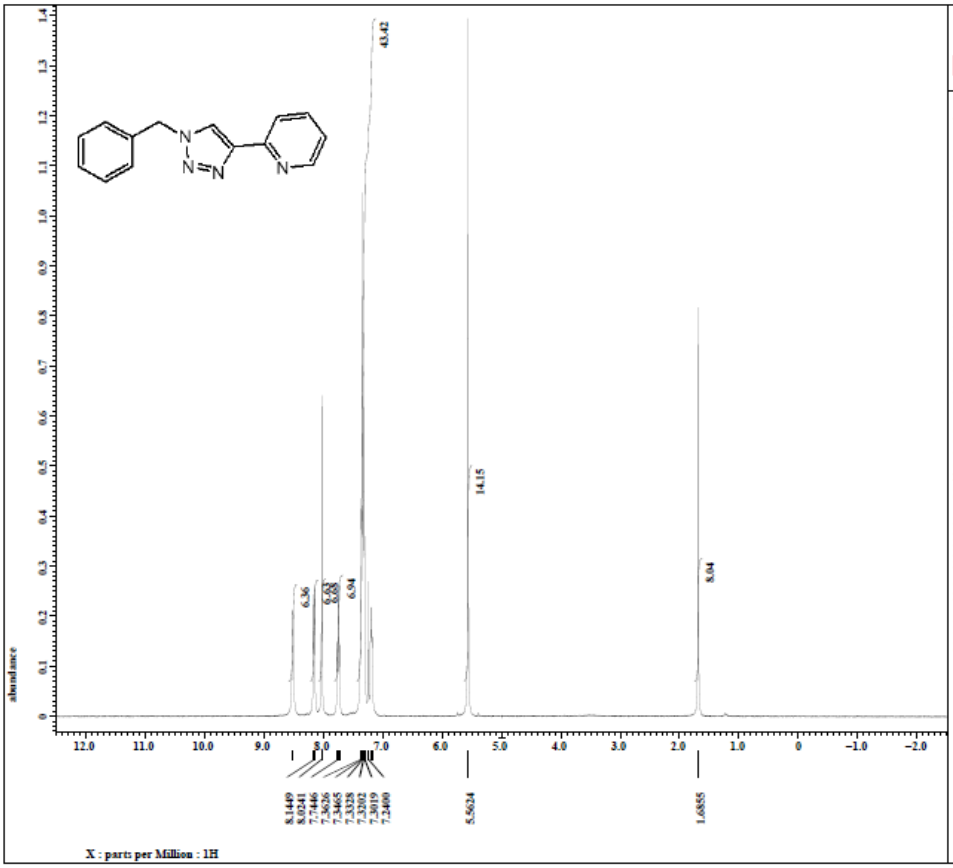


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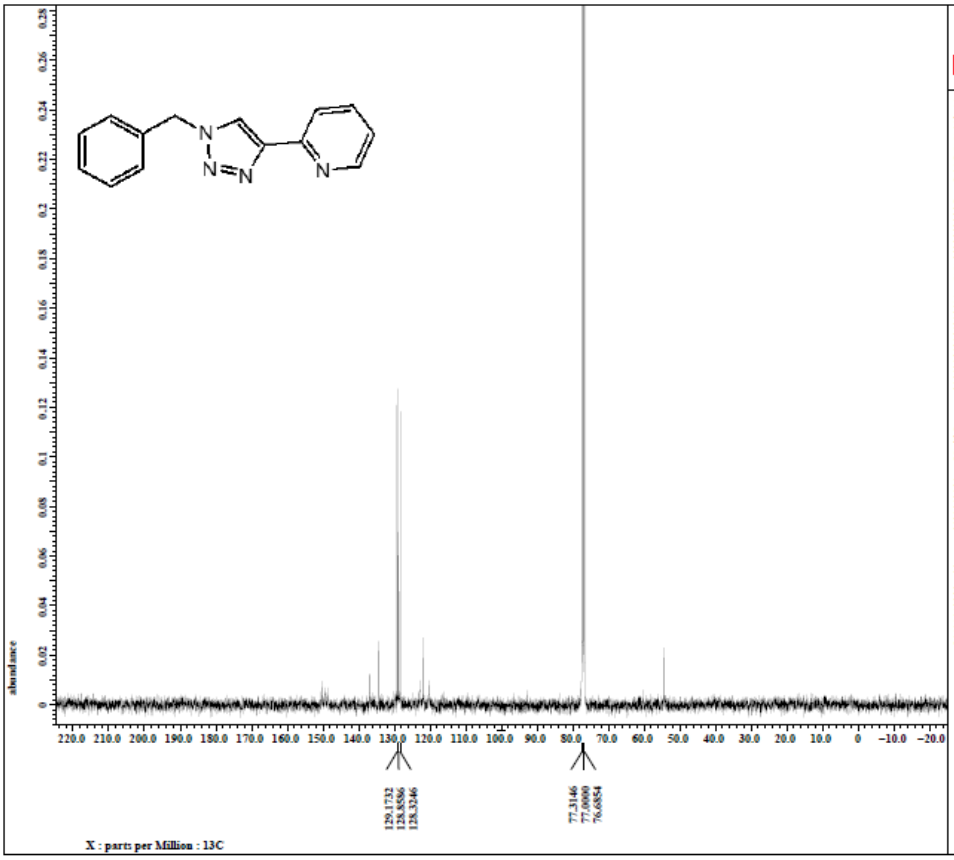


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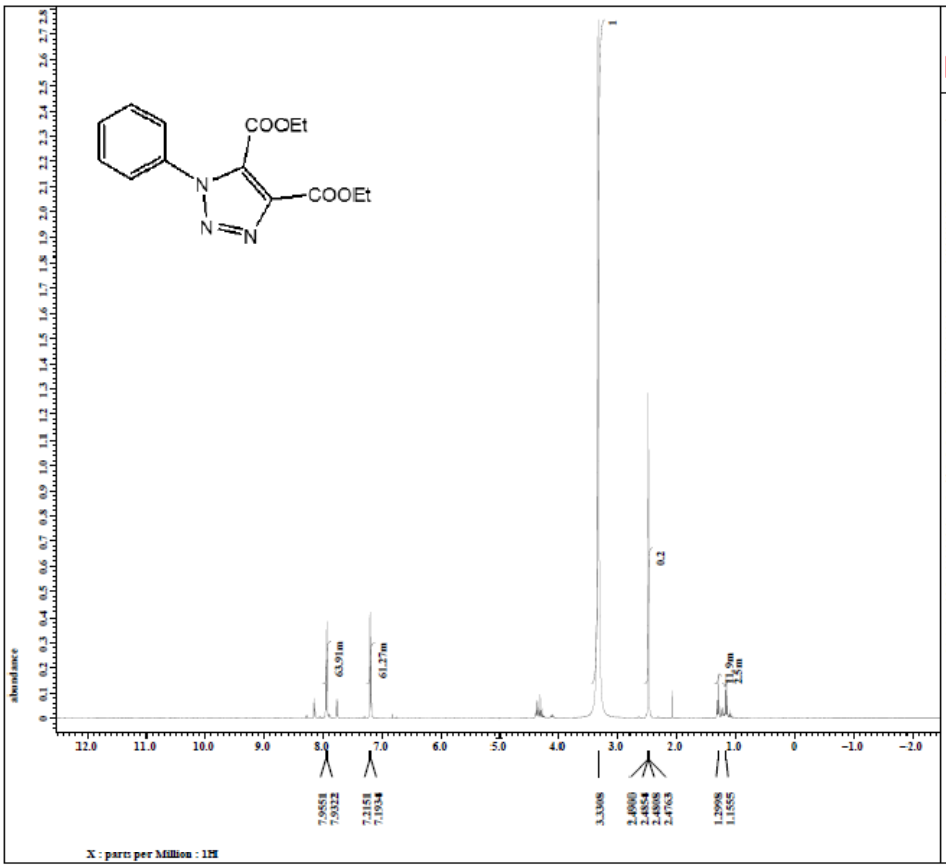

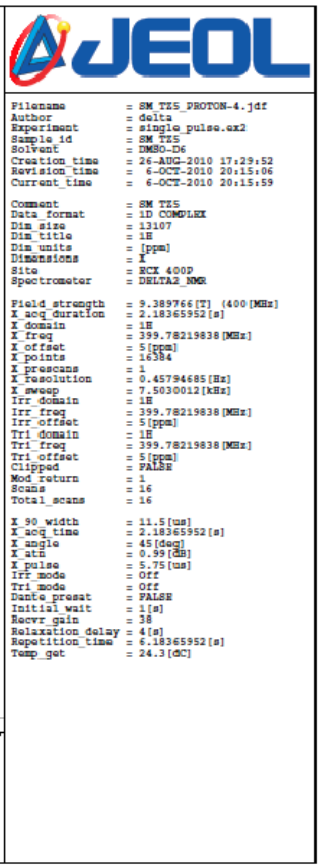

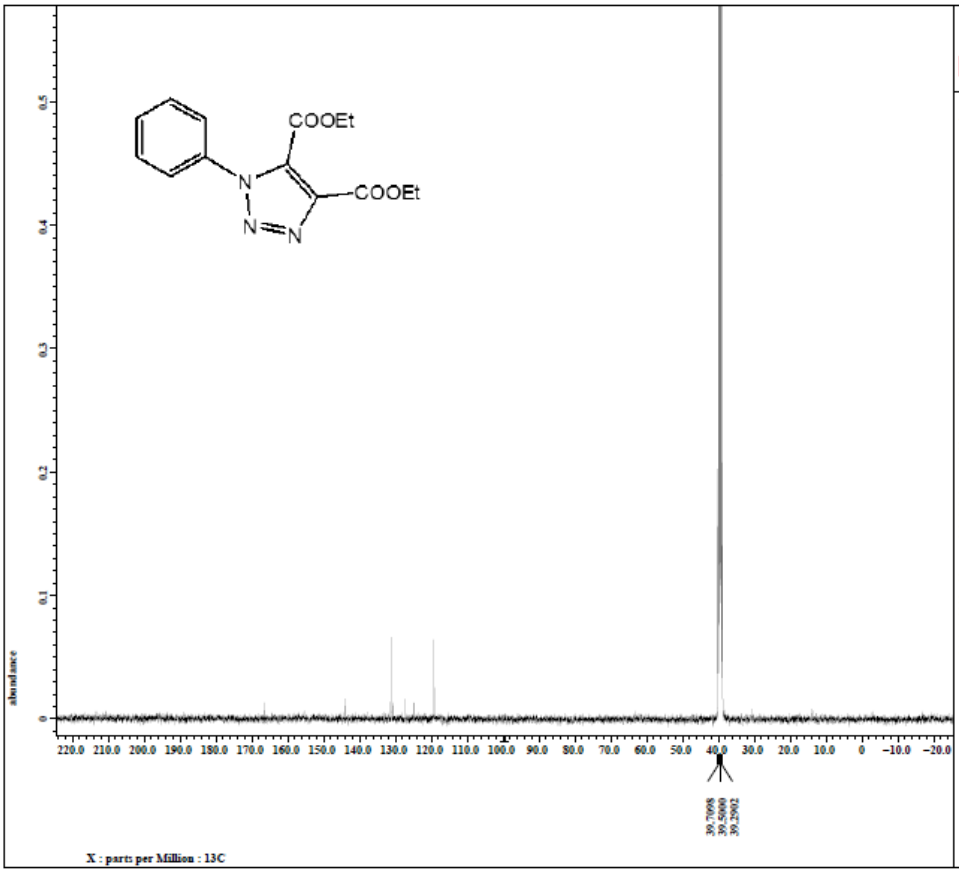

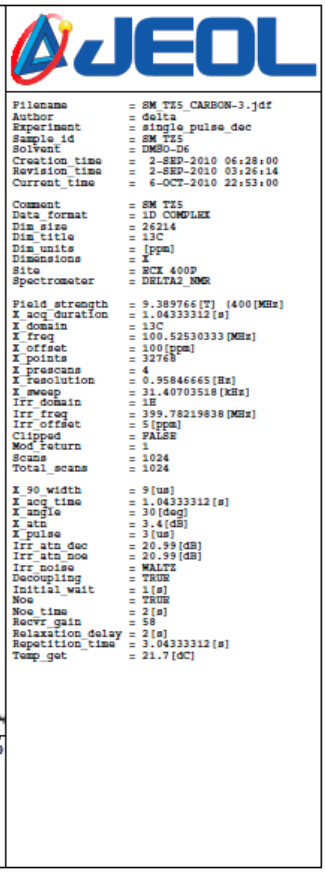

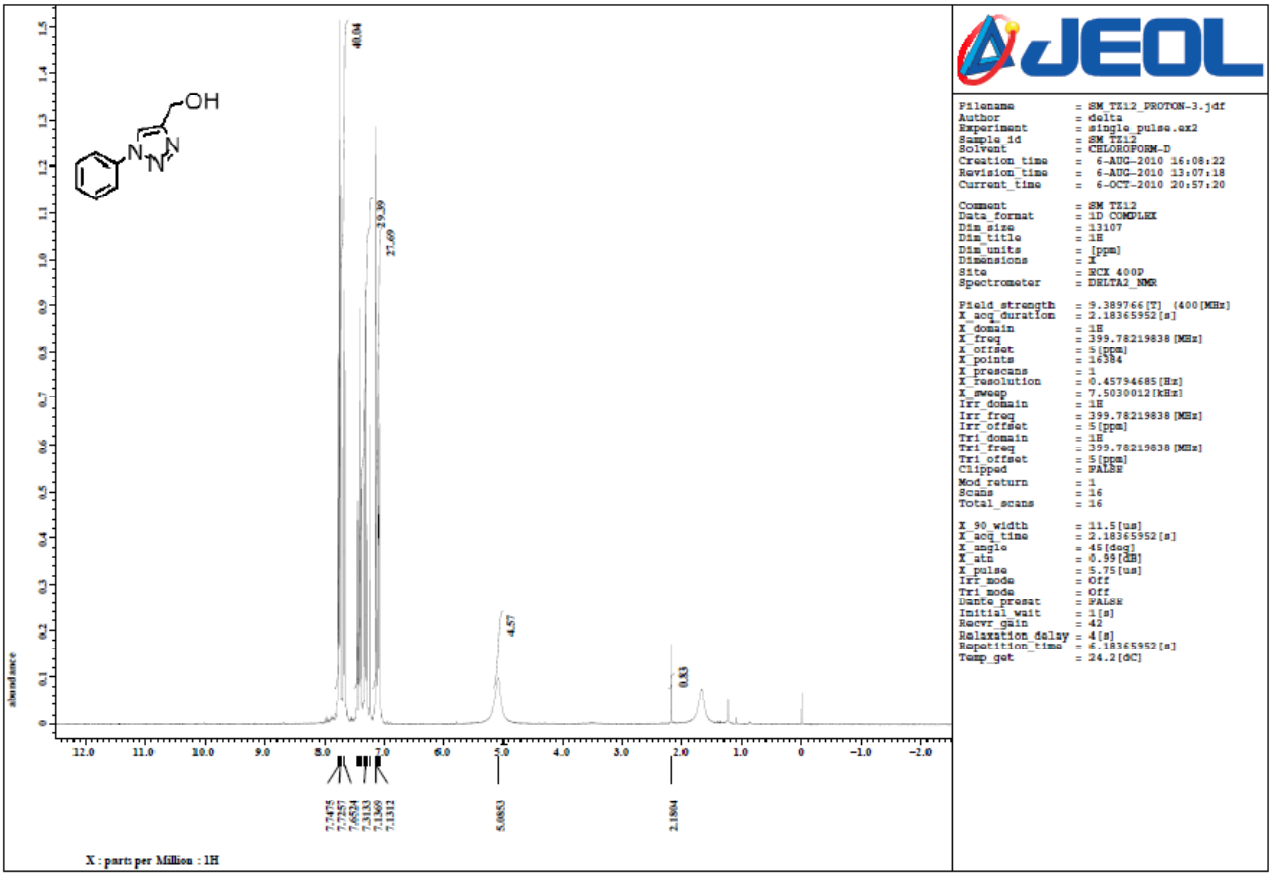

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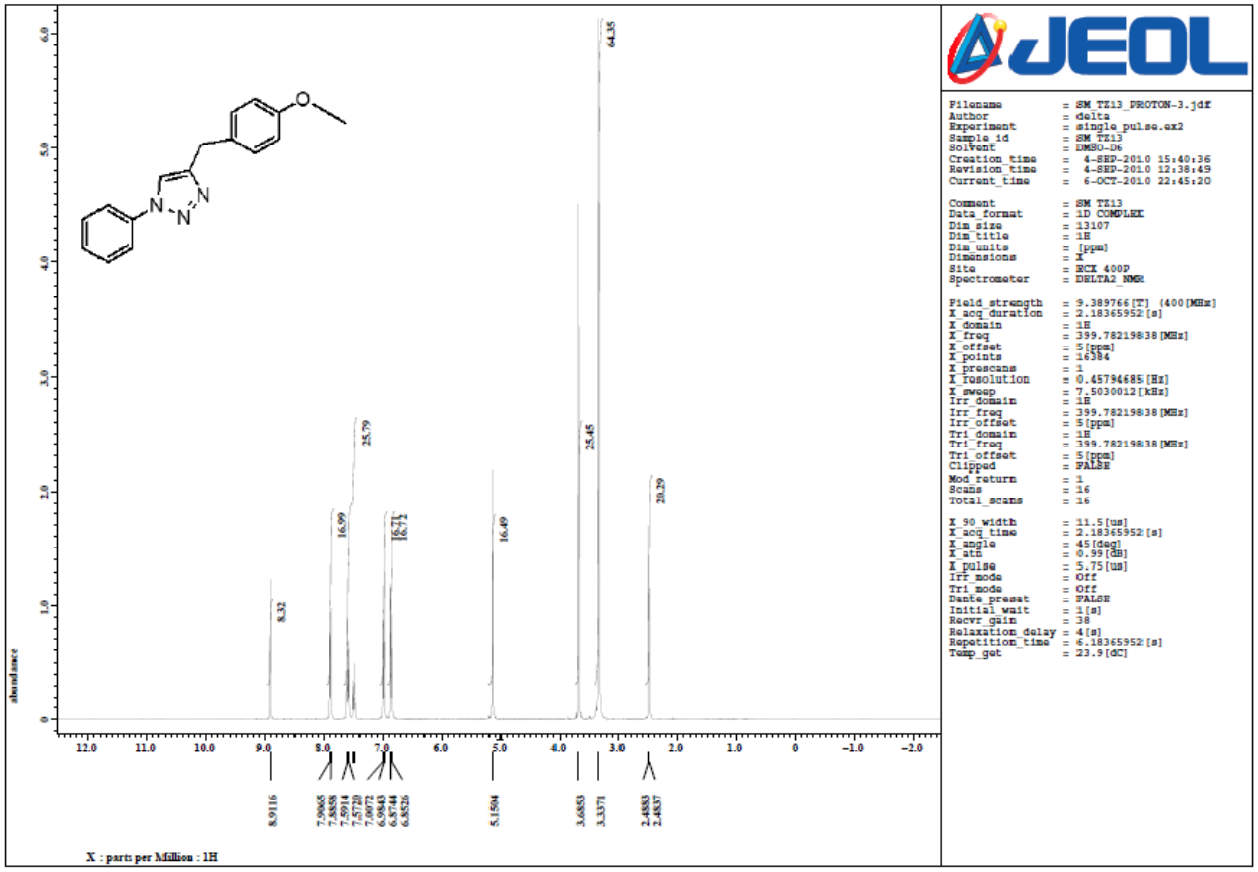

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