Electronic Supplementary Material (ESI) for RSC Advances.

Use of CuO encapsulated in mesoporous silica SBA-15 as recycled catalyst for allylic C–H bond oxidation of cyclic olefins at room temperature

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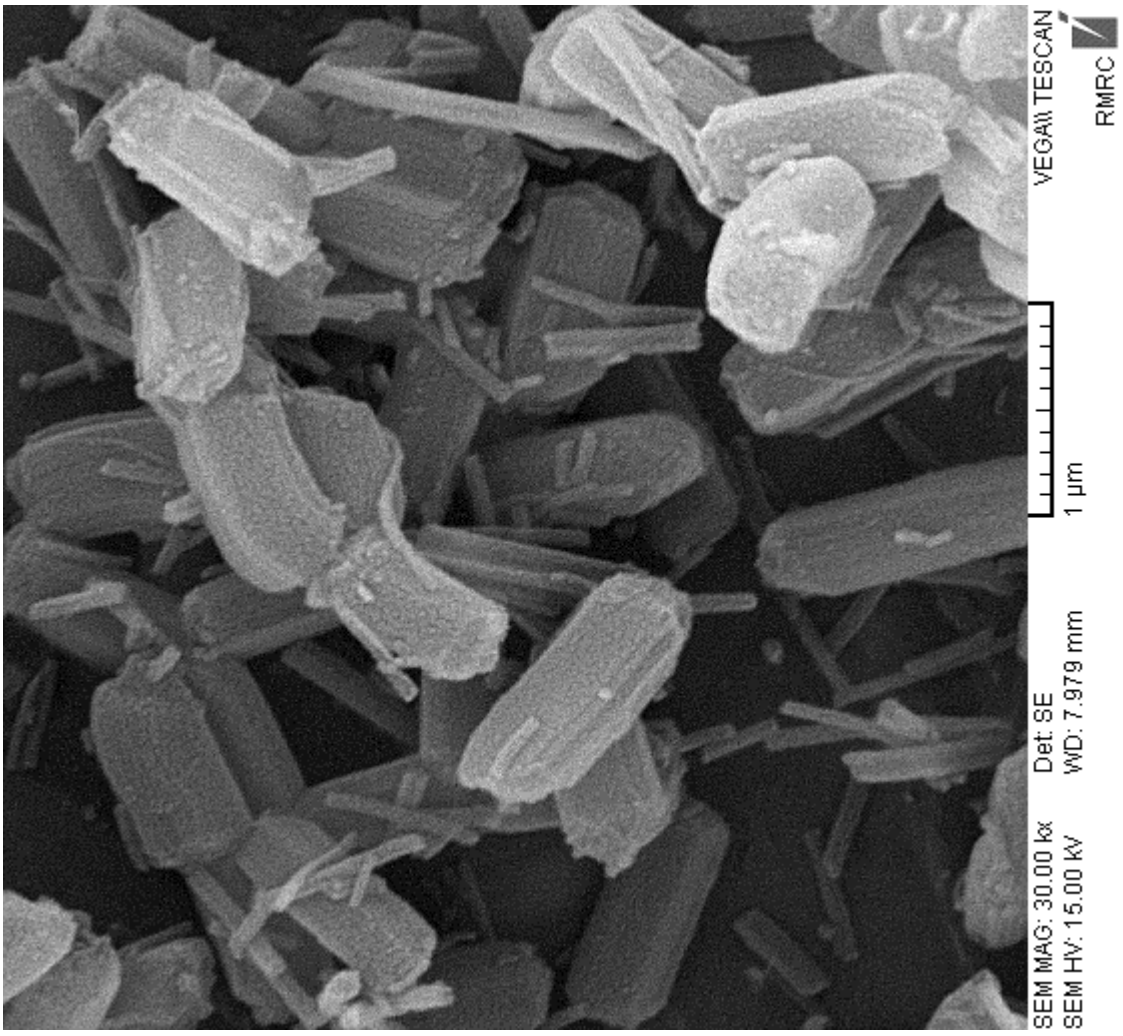
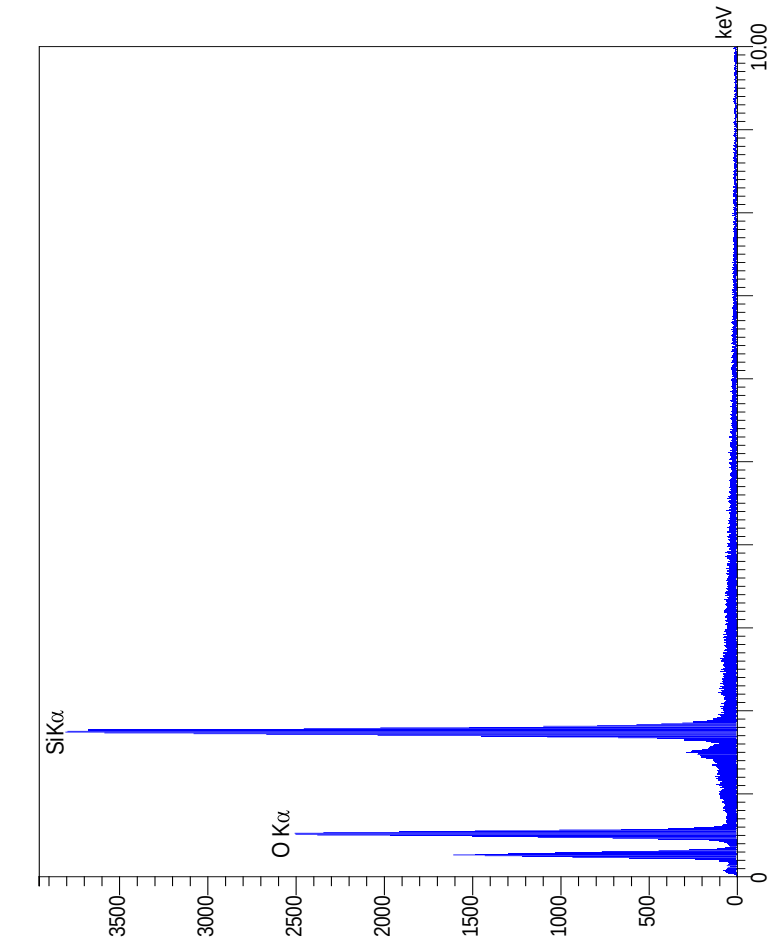
1. Typical procedure for preparation of SBA-15

Mesoporous silica SBA-15 was prepared by a method that described in the literature. 2.0 g Pluronic P123 (Poly(ethylene glycol)-block-Poly(ethylene glycol)-block-Poly(ethylene glycol)-block) copolymer as a surfactant was dissolved in 46 mL deionized water with 10ml concentrated HCl in a capped 100 mL Erlenmeyer flask at room temperature. After complete copolymer dissolution (0.5-1h), solution of TEOS as a source of Si (4.6 gr) in deionized water was added and the solution was stirred vigorously at 40 ˚C for 15 min, The solution was stirred for an additional 10 minutes and Finally the mixture transferred into a stainless steel jacketed Teflon vessel and heated at 100˚C for 48 h. the mixture was cooled to room temperature and solid product was separated by filtration followed by washing with deionized water until pH=7-8 was achieved. Finally dried at 60˚C . Template removal was performed by calcination at 823 K for 6h. the XRD, SEM, BET, EDX and FT-IR results clearly demonstrated that the mesoporous structure of SBA-15 [1].



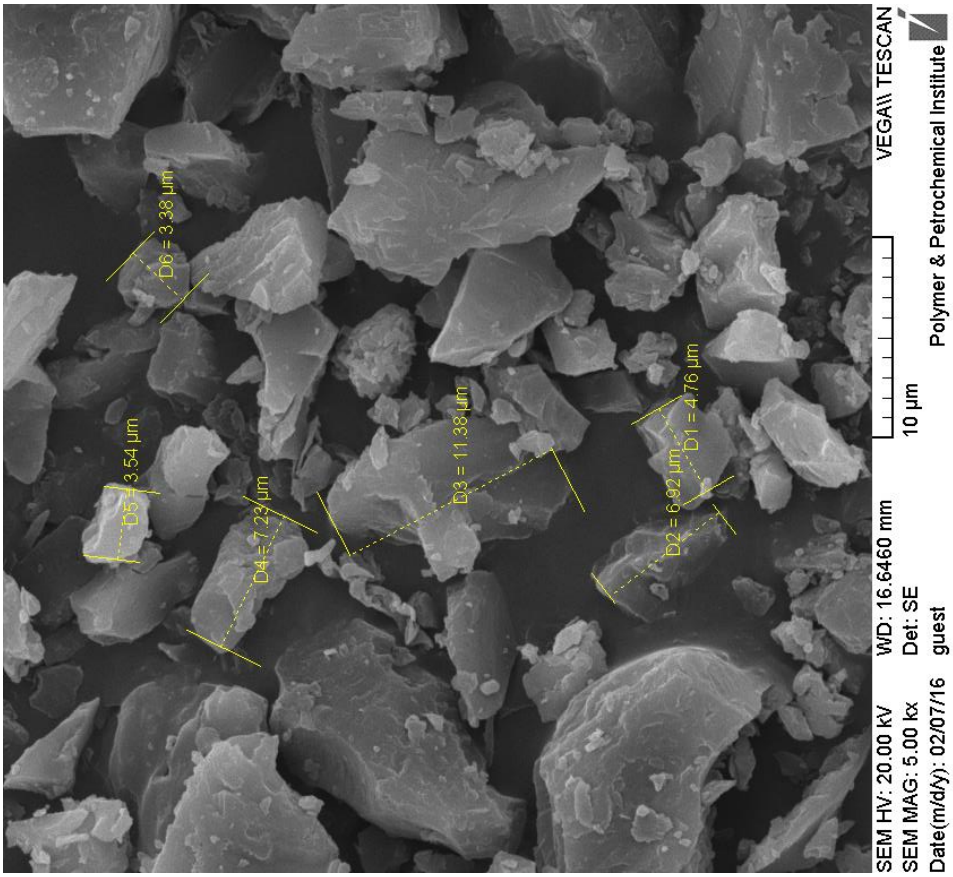
Scanning electron micrograph of SBA-15



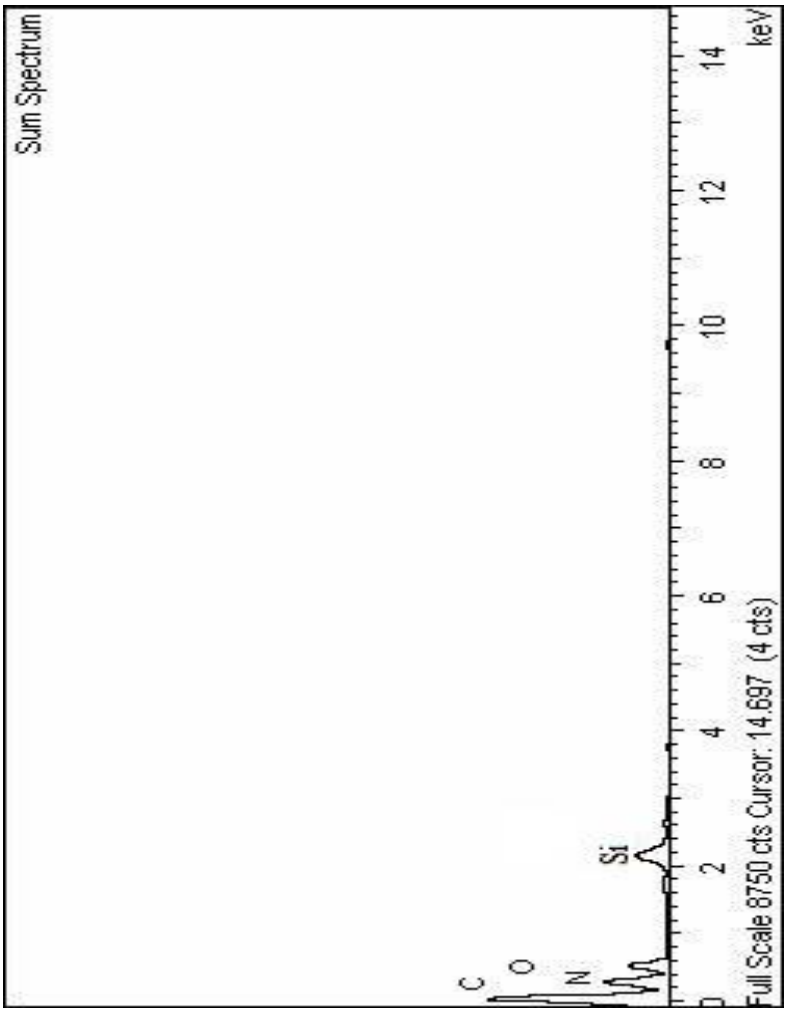
EDX spectrum of the SBA-15

2. Typical procedure for Functionalization of SBA-15 by 3-Aminopropyltrimethoxysilane

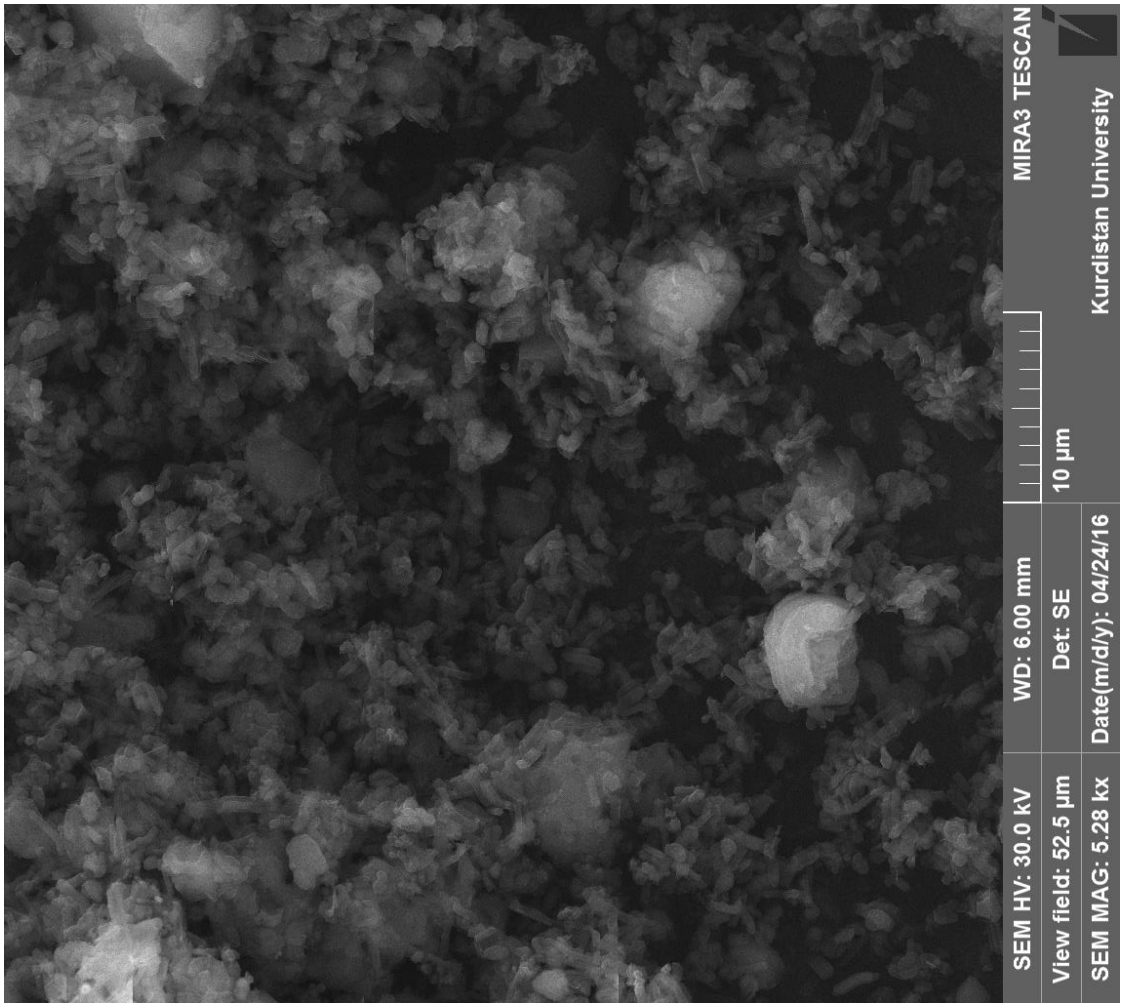
Surface modification of SBA-15 was performed corresponding to the literature. Briefly, the dry mesoporous silica materials (1g) were dispersed in 50 mL of dry toluene, then 2 mL of 3-Aminopropyltrimethoxysilane was added. The resulting mixture was refluxed under nitrogen atmosphere for 24 h. The modified nonporous were collected by filtration and washed thoroughly with CH_2Cl_2/CH_3OH. Then soxhlet for 24h with CH_2Cl_2/CH_3OH (1:1) to remove any untethered species then the white solid NH2- SBA-15 dried at room temperature [2].



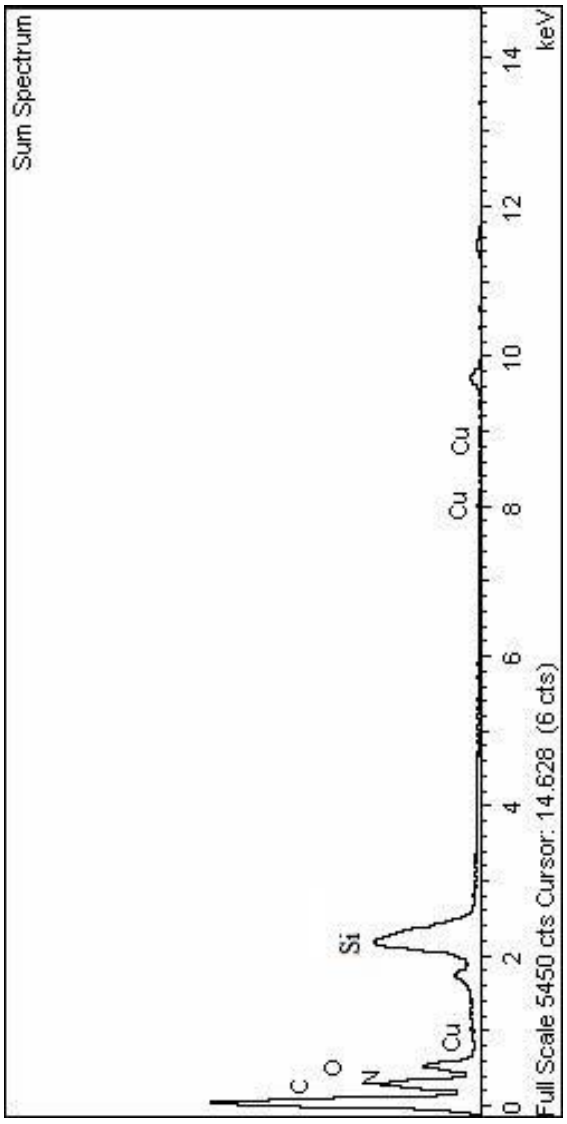
Scanning electron micrograph of NH_2-SBA-15



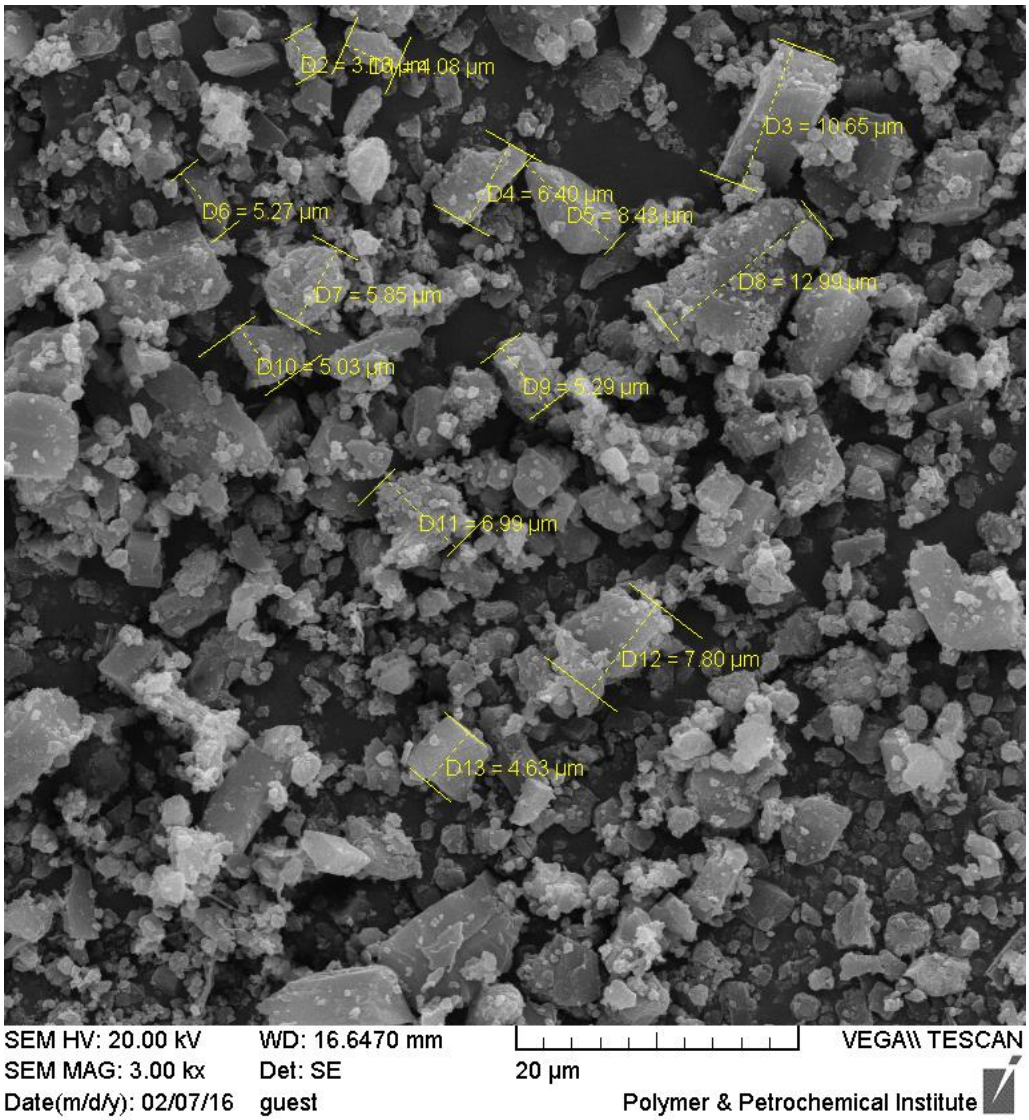
EDX spectrum of NH_2-SBA-15



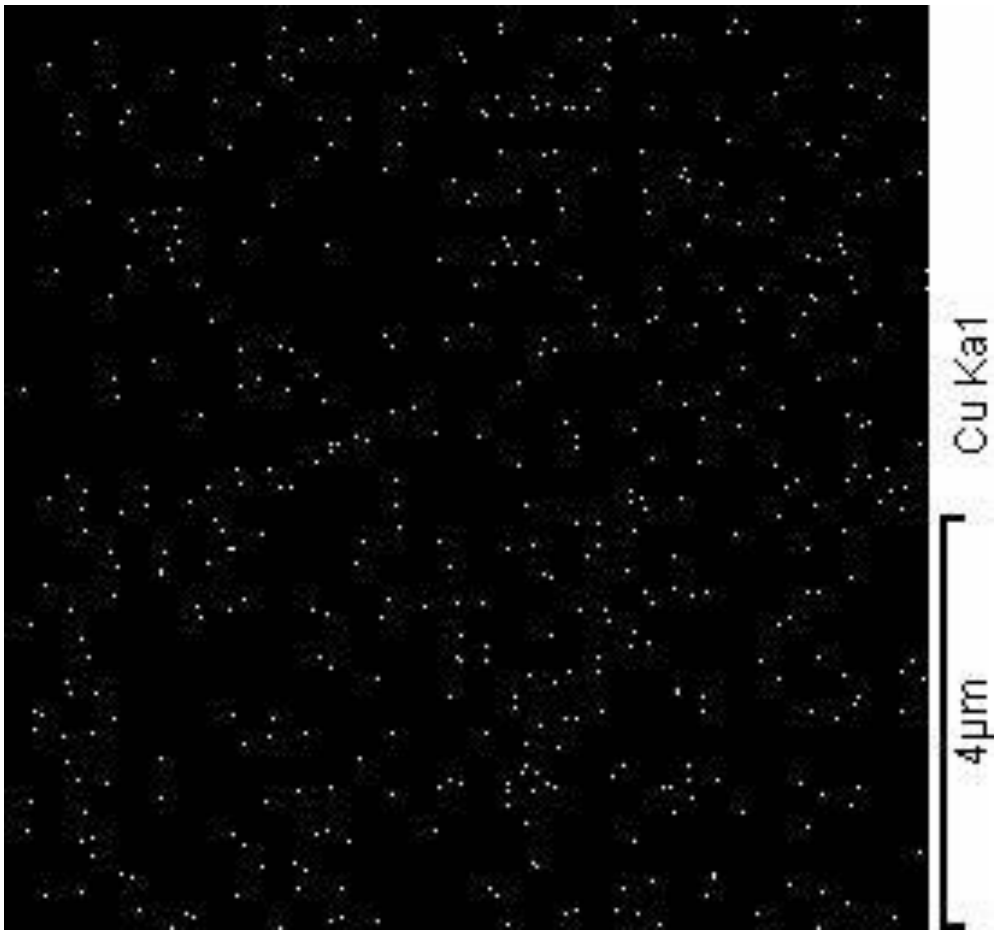
Scanning electron micrograph of Cu-SBA-15



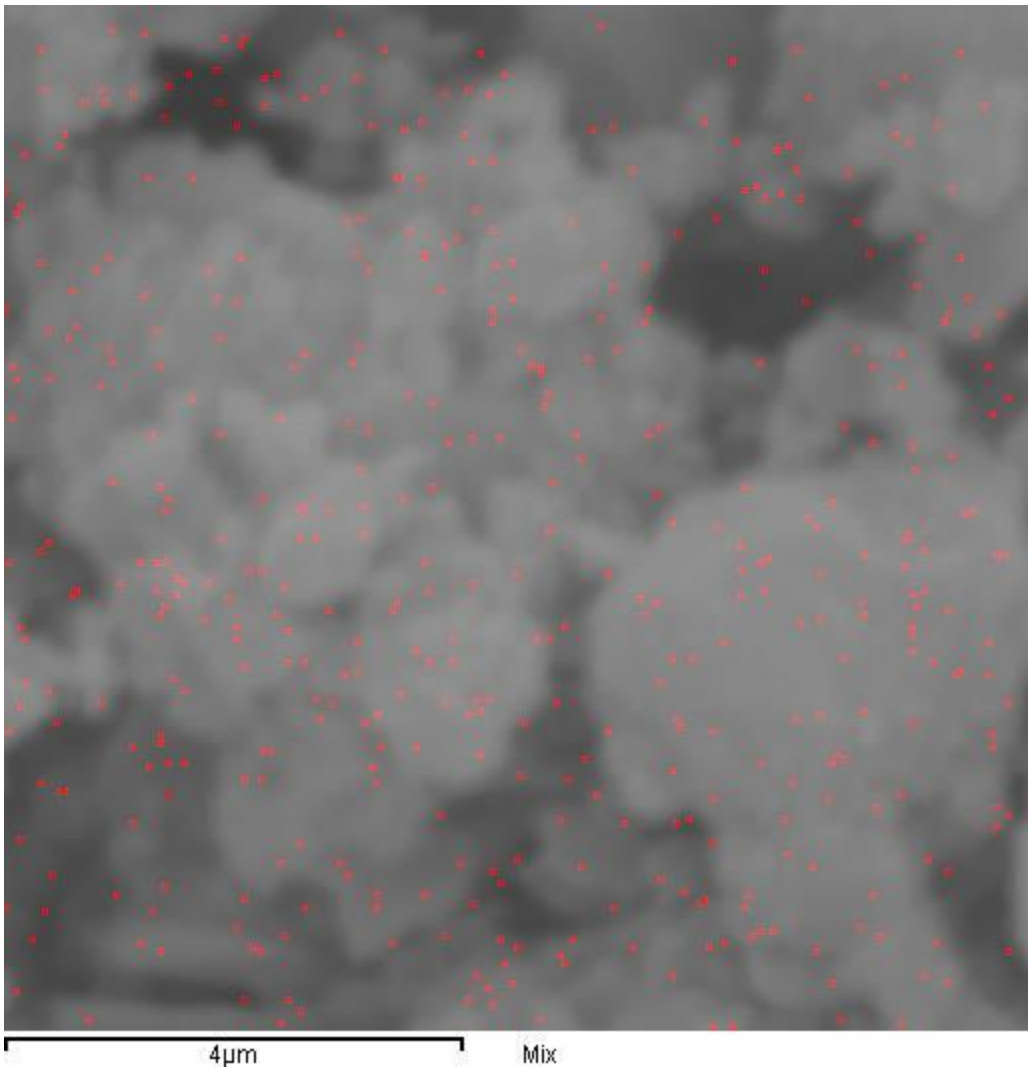
EDX spectrum of the Cu-SBA-15



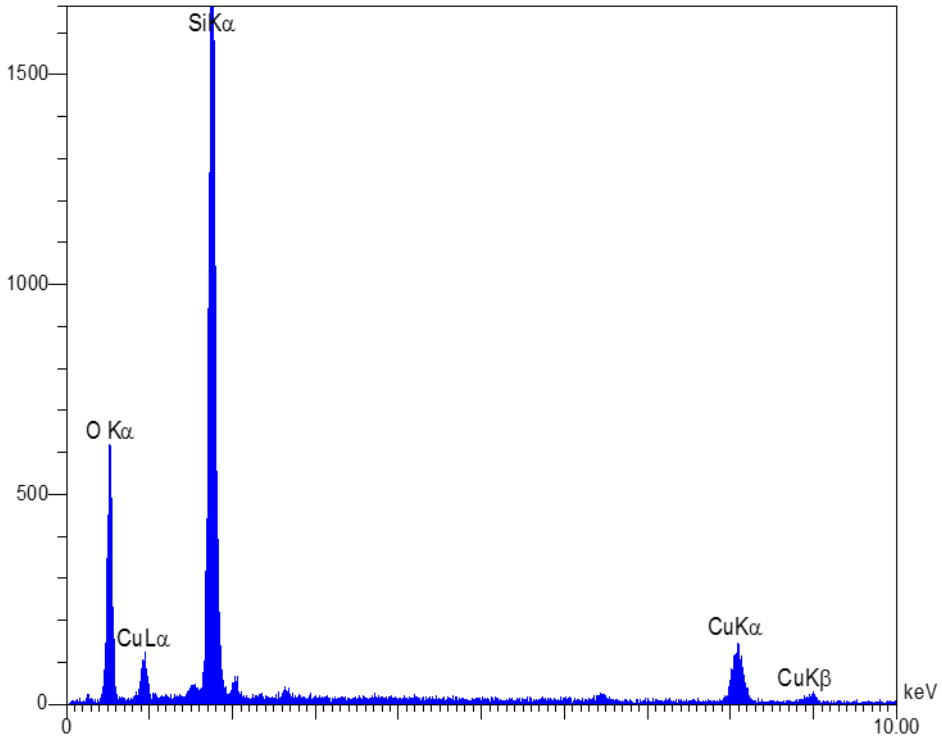
Scanning electron micrograph of Cu-N- SBA-15



Elemental mapping of the Cu-N-SBA-15 catalyst



Mix elemental mapping of the Cu-N-SBA-15 catalyst

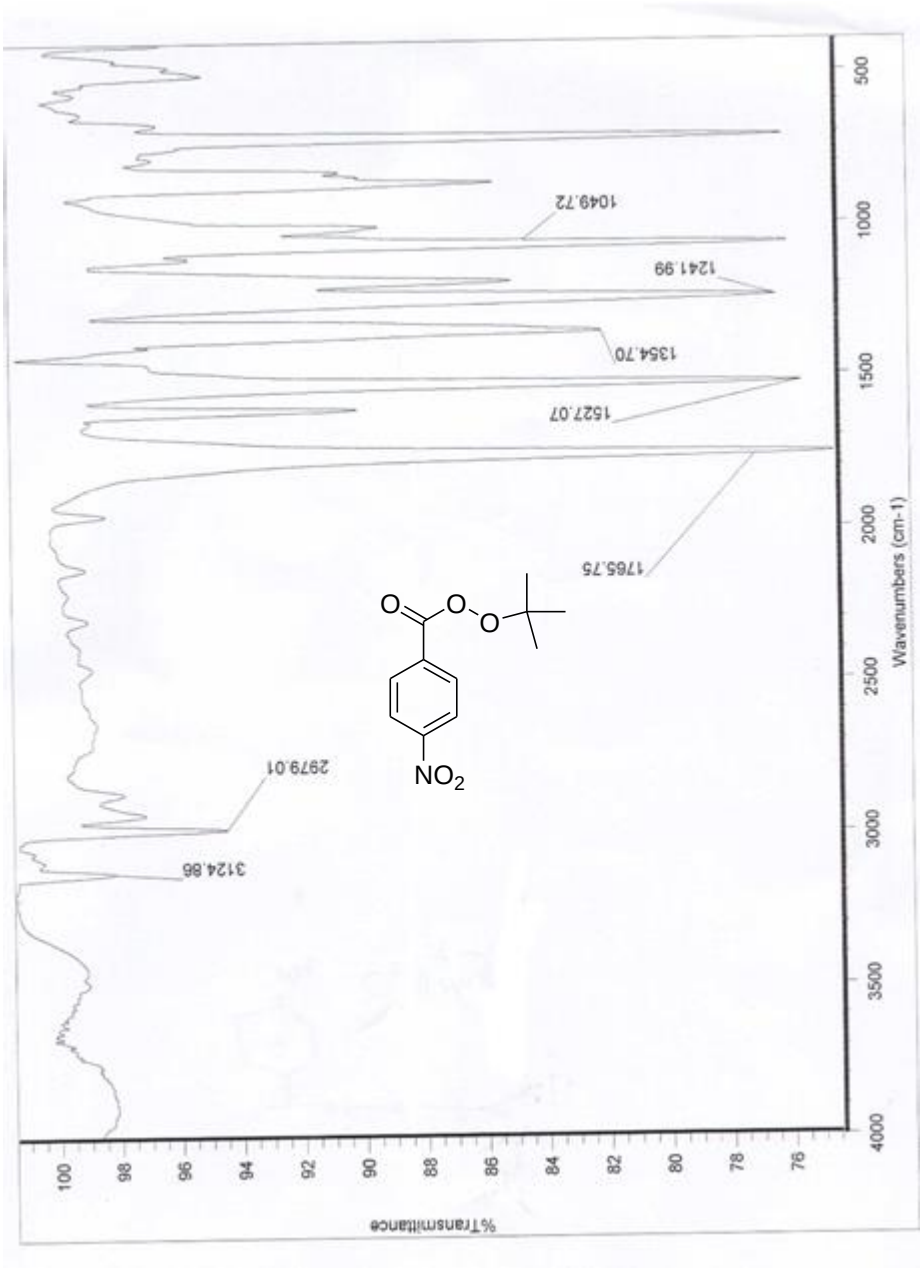


EDX spectrum of the N- Cu-SBA-15

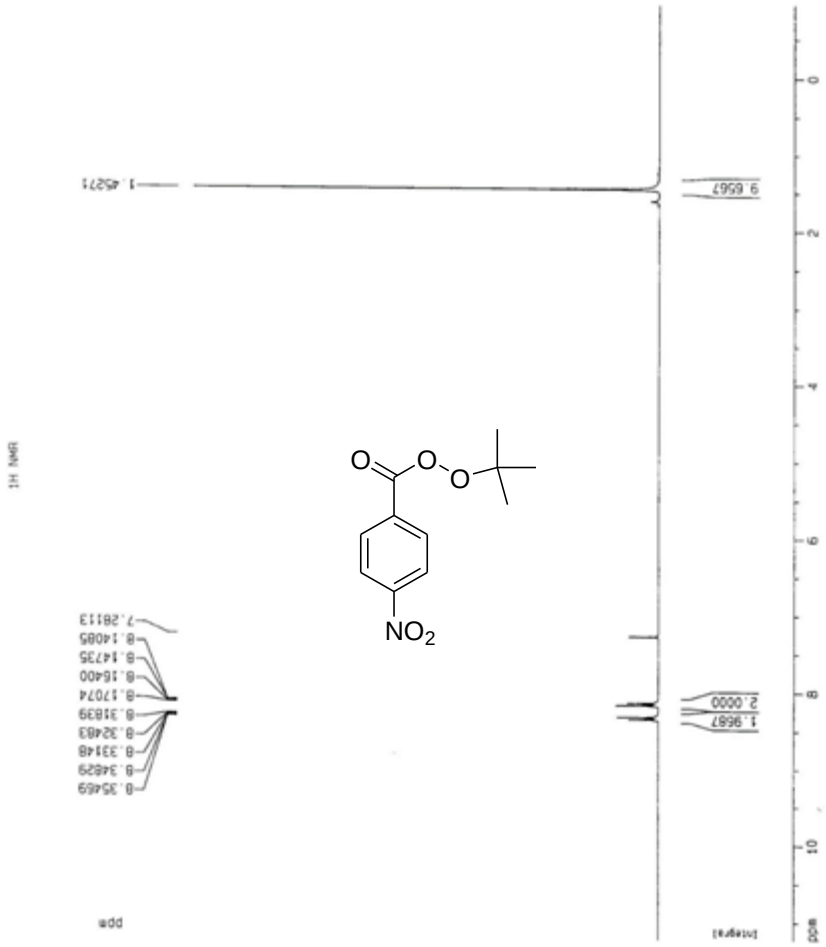
3. Typical procedure for Synthesis of tert-butyl 4-nitrobenzoperoxoate 1:

p-nitrobenzoyl chloride (3.2 g, 17.2 mmol) was dissolved in a 100 mL round bottom flask containing CH_2Cl_2 (35 mL). The solution was cooled to –20 ˚C and stirred under nitrogen for 15 min. Pyridine (1.7 mL, 20.0 mmol) was added and the reaction mixture was stirred for 10 min. Then, *tert*-butyl hydroperoxide (3.5 mL, 20.0 mmol) was added dropwise to the reaction at -20 ˚C, and stirred for 4 h. Then the reaction solution was diluted with CH_2Cl_2 (20 mL), and washed with water. The organic layer was separated, dried over $MgSO_4$, and evaporated to obtained crude yellow solid product. Purification using flash chromatography (90:10; n-hexane: EtOAc) afforded the light yellow solid product **1**. (3.9 g, 98% yield) [3, 4].

*Tert-butyl 4-nitrobenzoperoxoate **(1)*****:** Mp: 74-76˚C (lit.75-78°C (ref. 3)); ^{1}H NMR (300 MHz, $CDCl_3$): δ_H (ppm) = 1.45 (9H, s, Me), 8.17-8.40 (4H, m, Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C (ppm) = 26.7, 84.5, 124.1, 131.3, 133.9, 150.6, 163.0.



FT-IR of perester (1)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O
O
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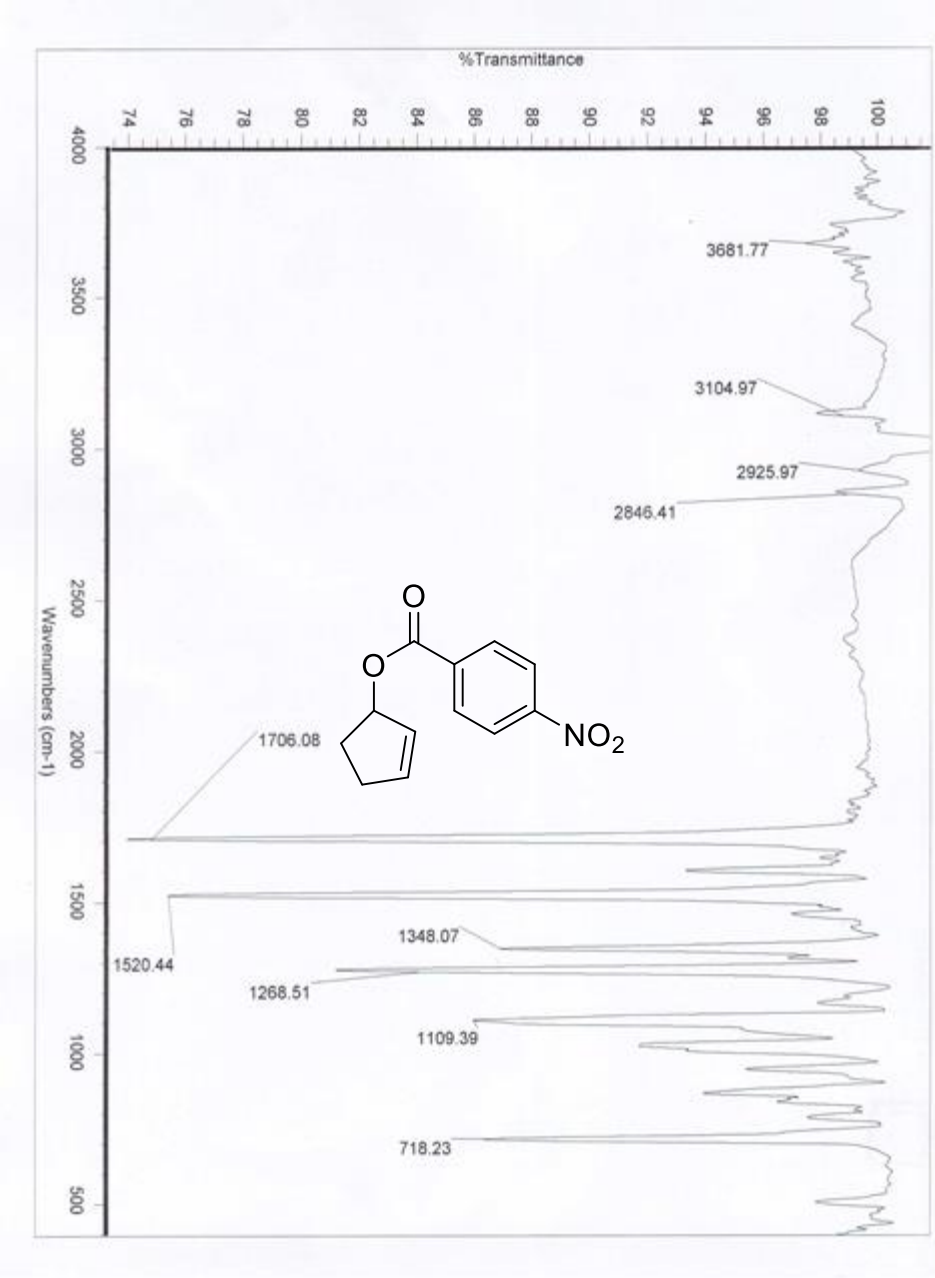
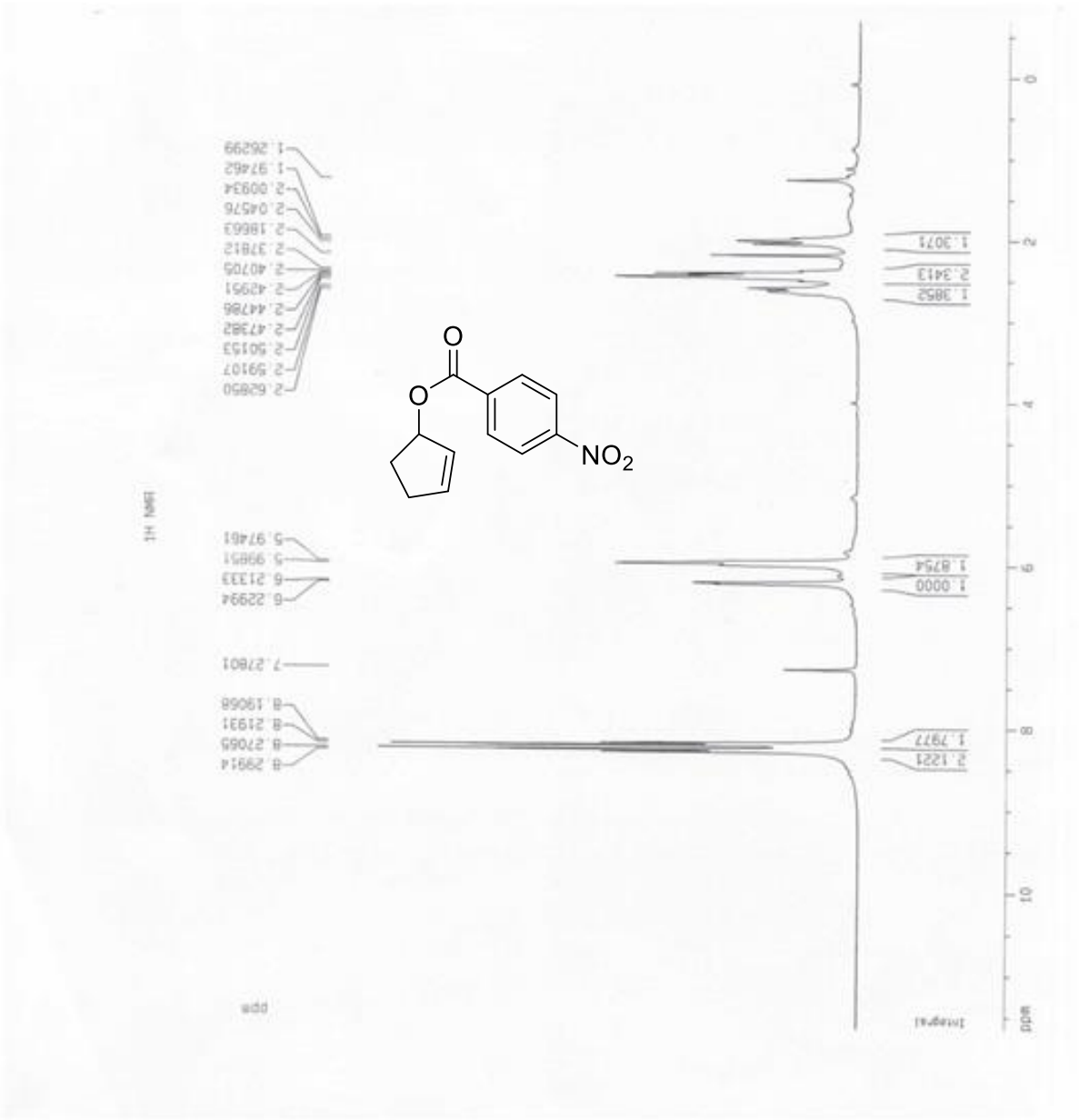
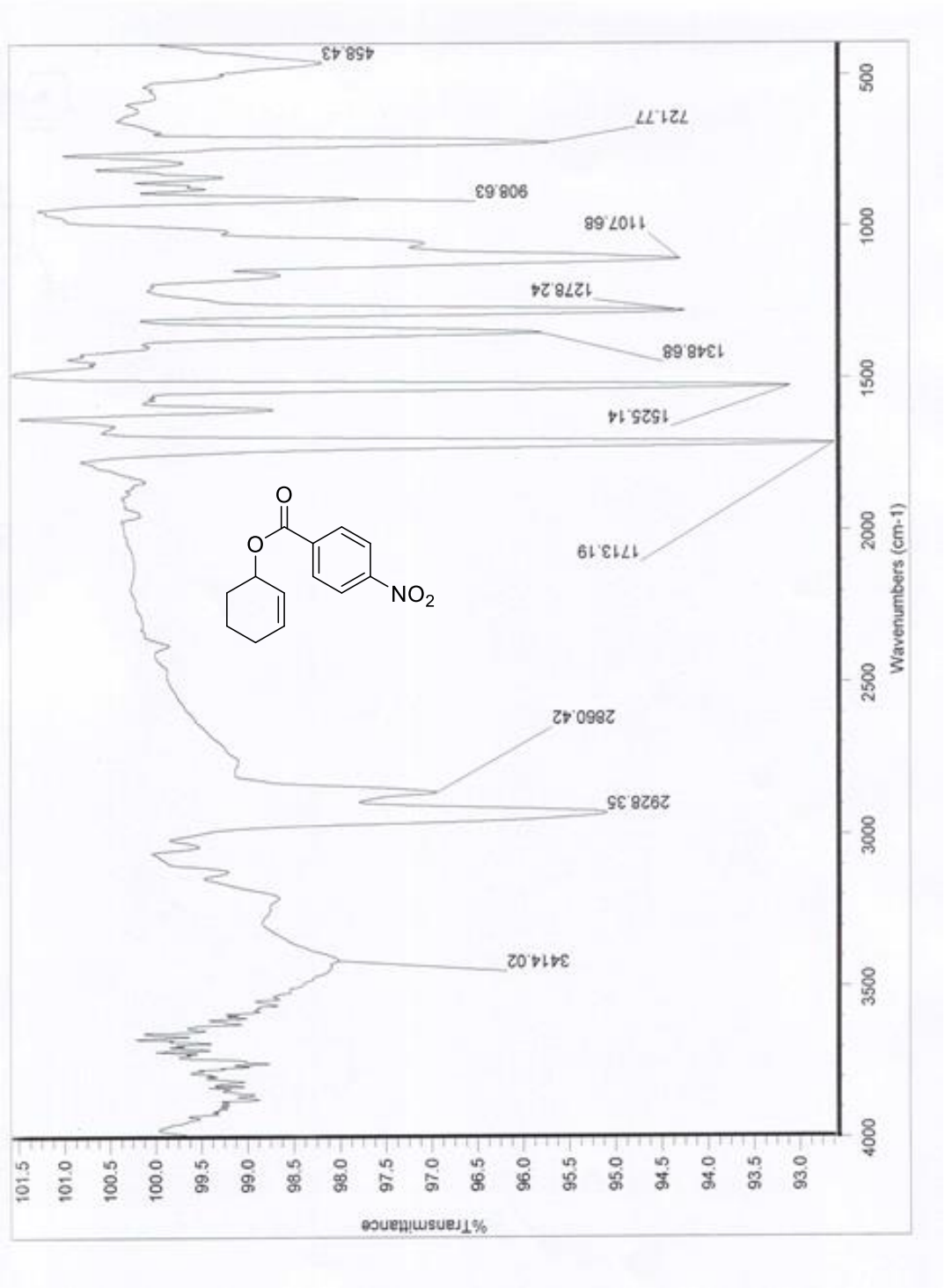
8.17074
8.16400
8.14735
8.14085
ppm



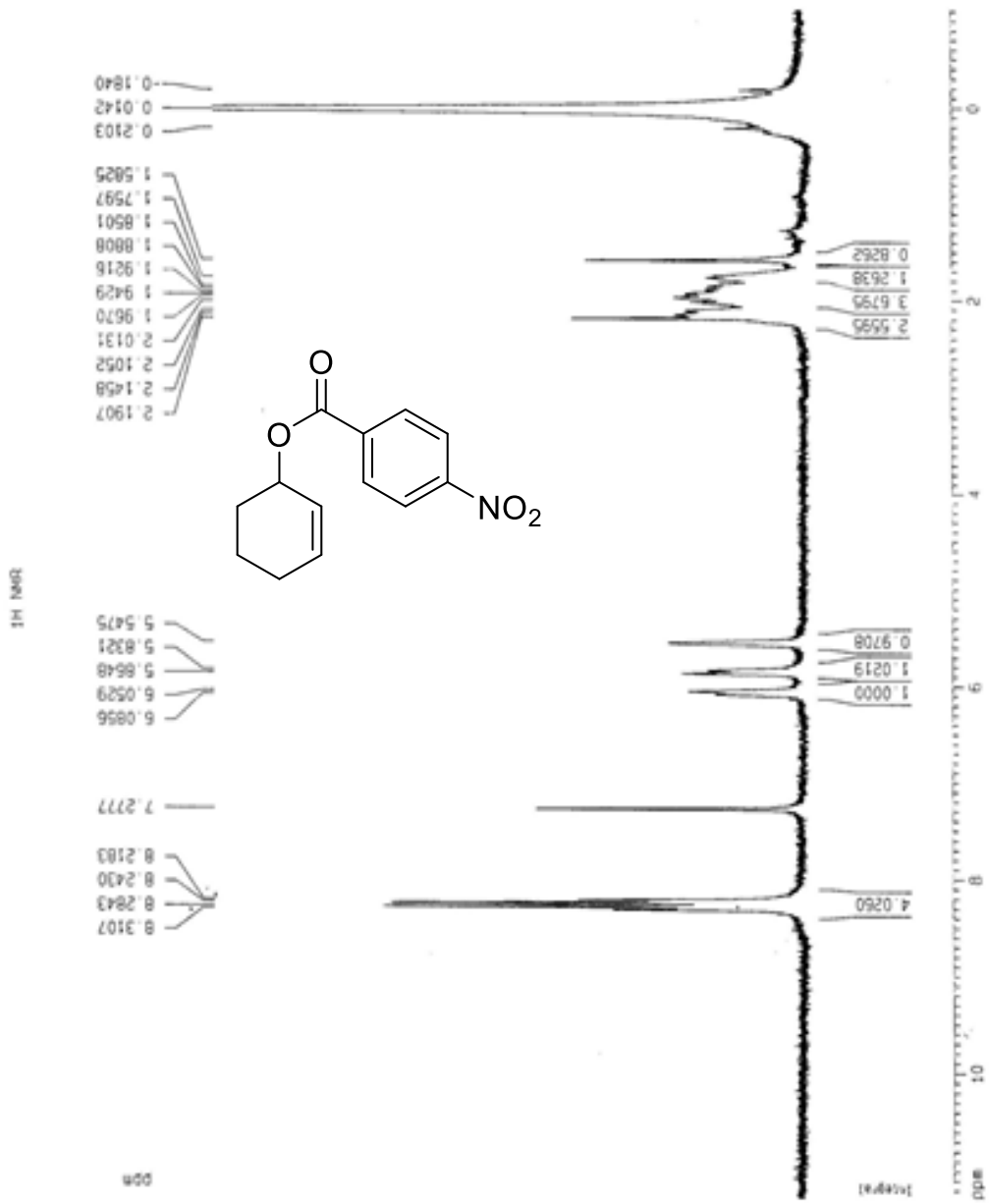
FT-IR of compound 2



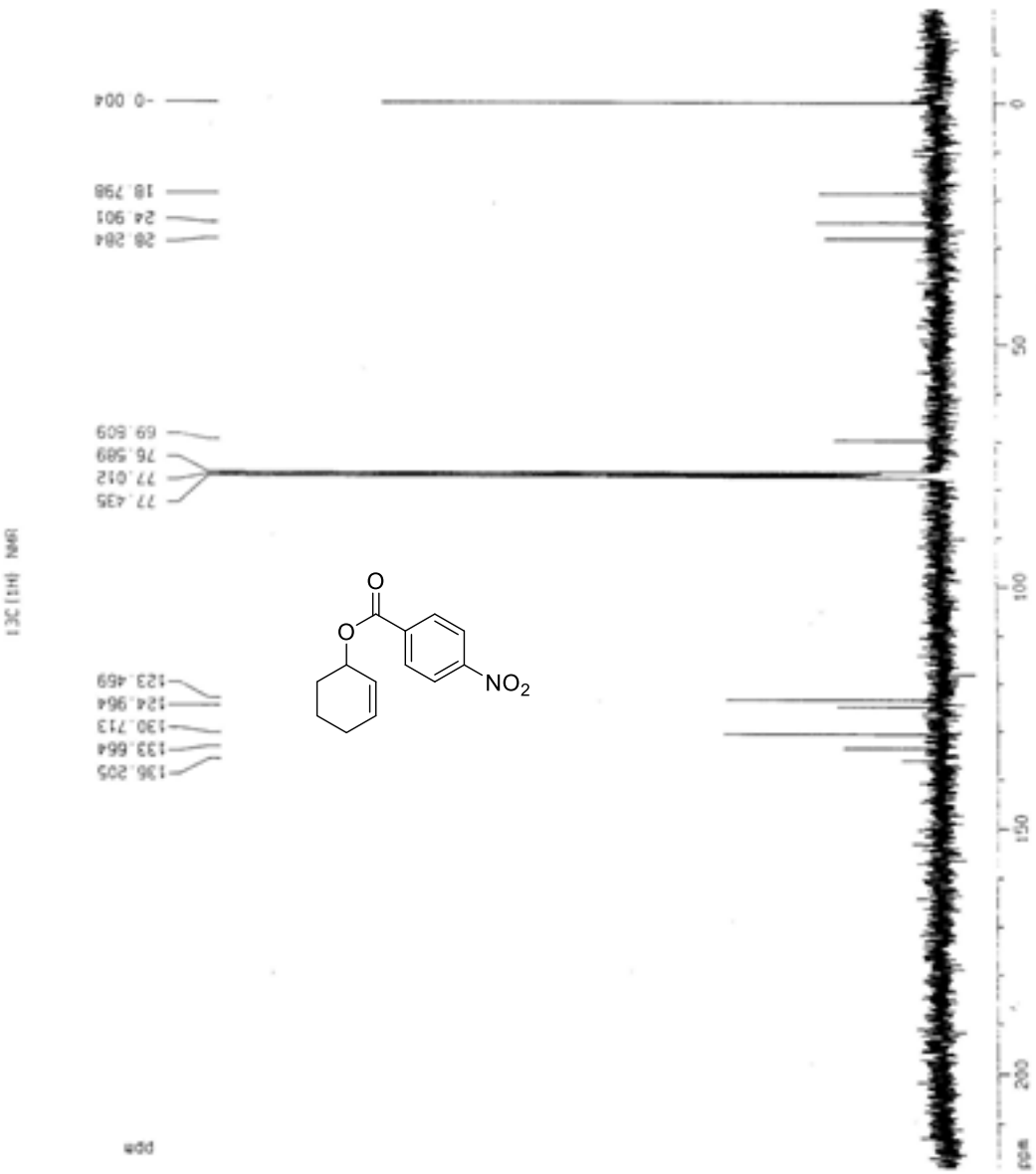
^{1}H NMR of compound 2



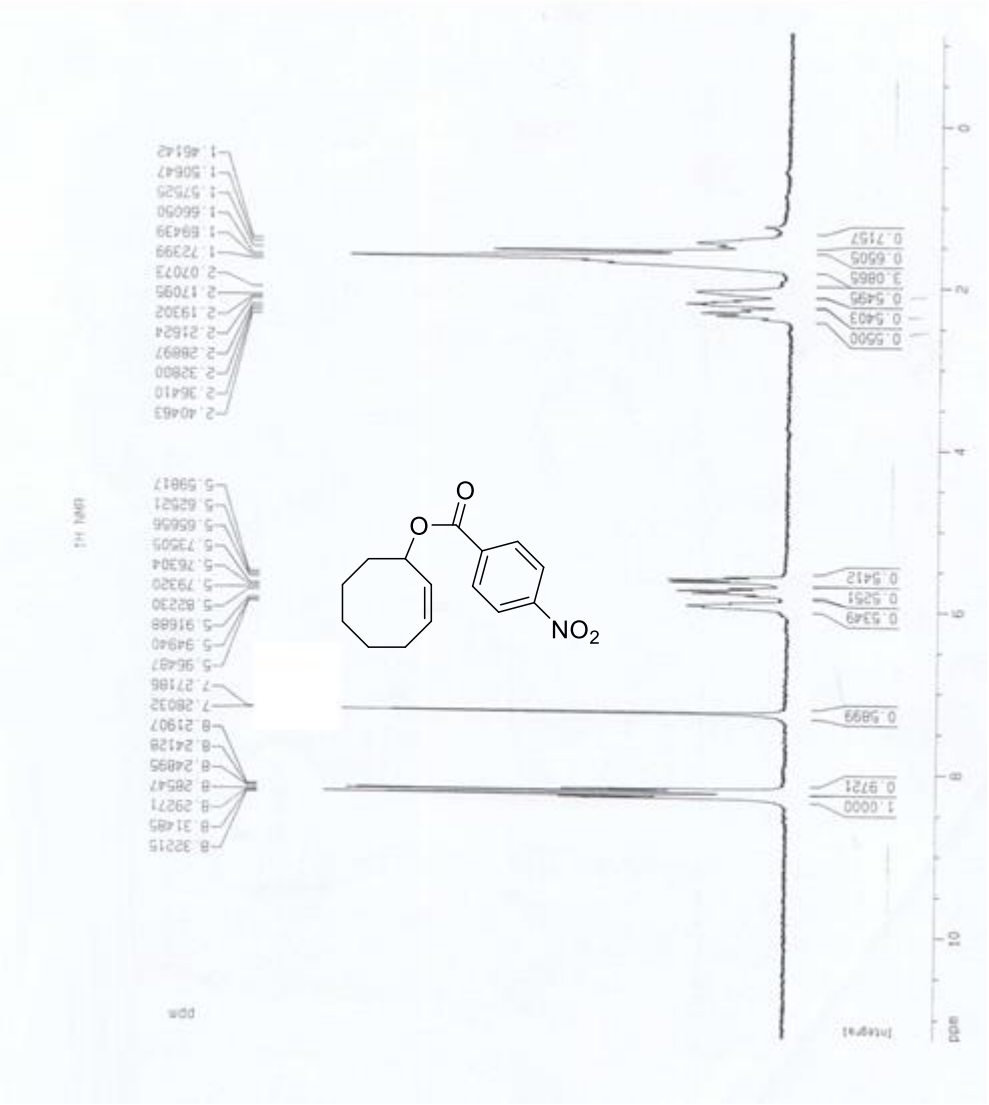
FT-IR of compound 3



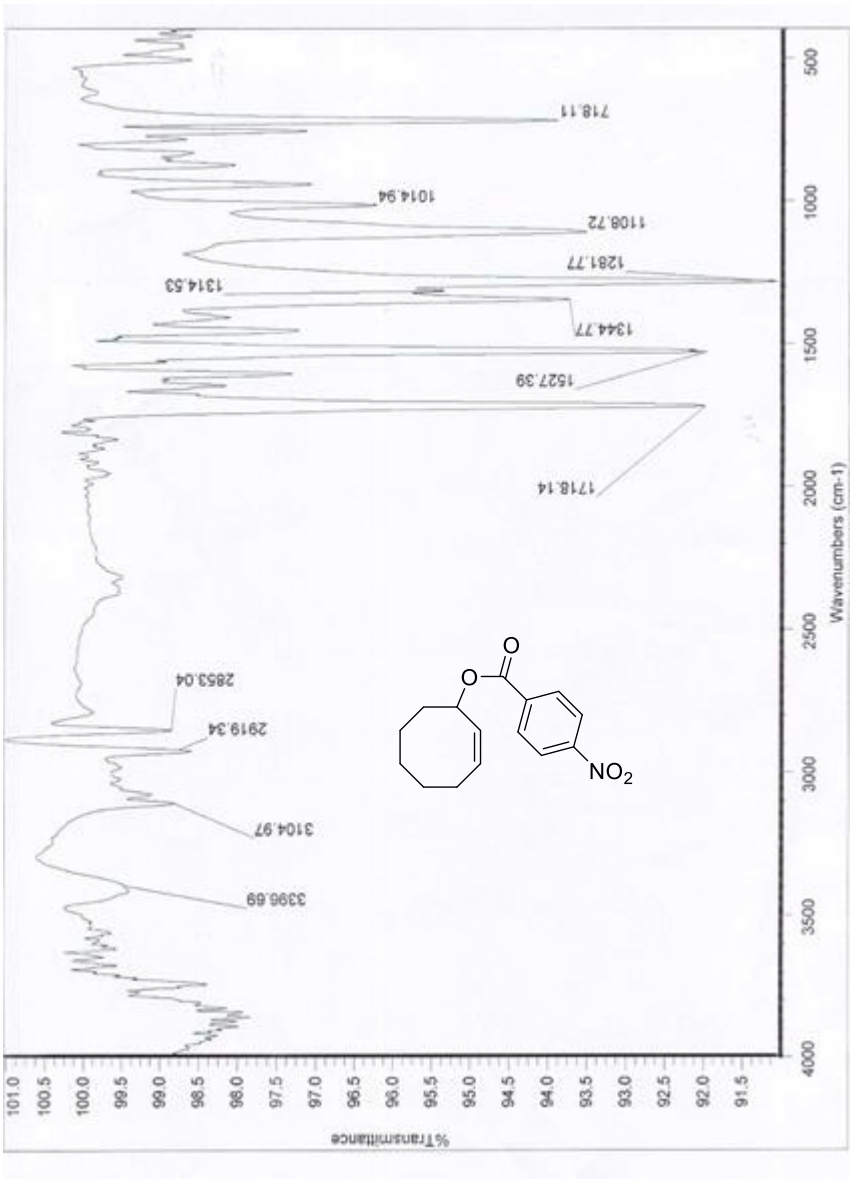
^{1}H NMR of compound 3



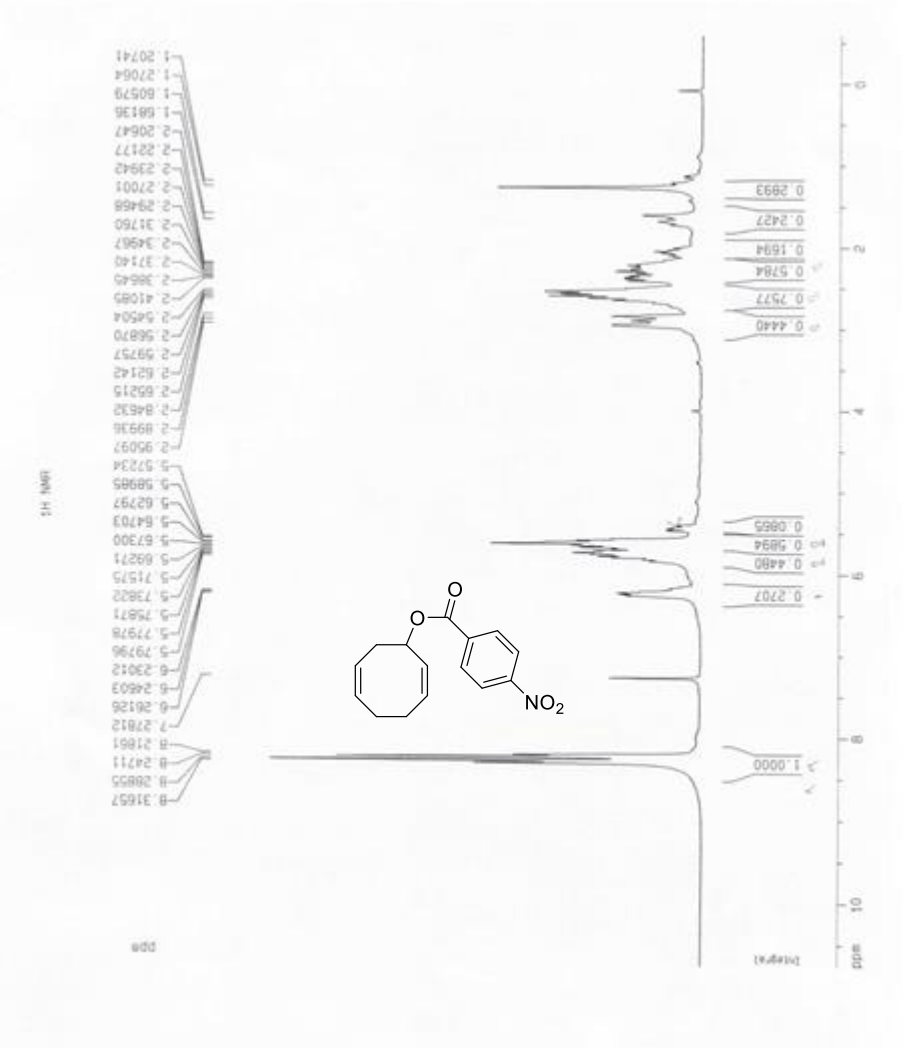
^{13}C NMR of compound 3



^{13}C NMR of compound 4



FT-IR of compound 4



^{1}H NMR of compound 5

References

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4 S. Samadi, K.Jadidi, B. Khanmohammadi N. Tavakoli, *J. Catal.* 2016, **340**, 344-353.