

The preparation and characterization of boehmite nanoparticles-TAPC: A tailored and reusable nanocatalyst for the synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones

Kiumars Bahrami,^{*a, b} Mohammad M. Khodaei,^{*a} and Mohsen Rostaie^a

Experimental

General

All reagents were purchased from Merck and used without further purification. Boehmite nanoparticles and TAPC were prepared according to reported procedure respectively.^{1,2} FT-IR spectra were obtained with potassium bromide pellets in the range 400–4000cm⁻¹ with a Perkin-Elmer 683 instrument. ¹H and ¹³C NMR spectra were recorded on a Bruker (200 MHz) spectrometer in CDCl₃ as solvent. The morphology of boehmite nanoparticles and BNP-TAPC was examined by scanning electron microscopy (SEM, TESCAN VEGA TS 5136 MM) and transmission electron microscopy (TEM, Philips CM10, high tension 100 kV). X-ray powder diffraction (XRD) patterns were recorded using an EQUINOX 3000 Inel diffractometer with CuK α radiation ($\lambda=1.5406 \text{ \AA}$).

General procedure for the preparation of BNPs-TAPC

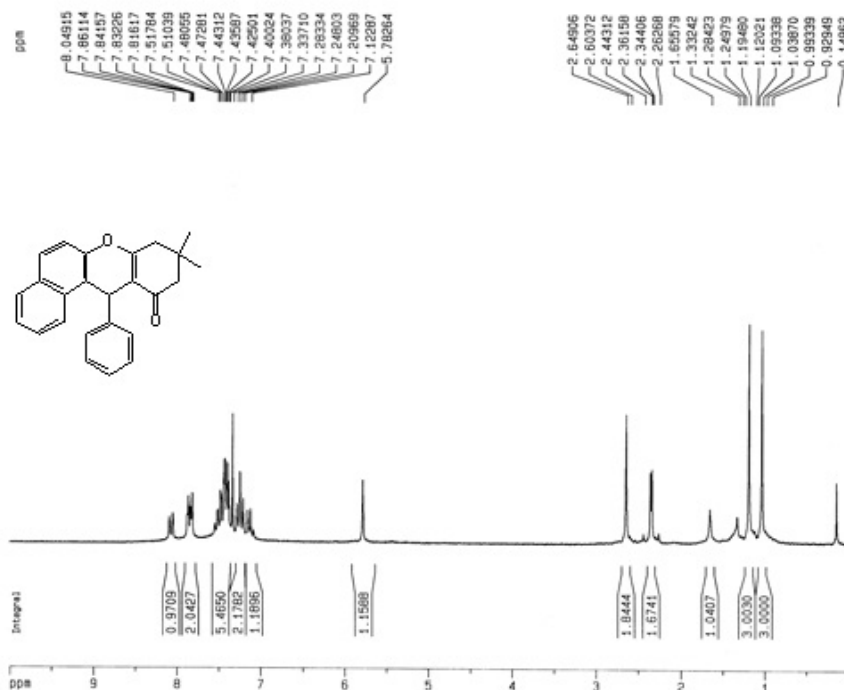
In a 100 ml round-bottom flask equipped and a magnetic stirrer bar, boehmite nanoparticles (50 mg), TAPC (50 mg) and CH₂Cl₂ (20 mL) were mixed and sonicated for 1 h at 30° C and then the reaction mixture was refluxed at 40 °C for 12 h. Then, the solids were collected by filtration, washed thoroughly with CH₂Cl₂ and dried at room temperature to afford a cream powder as the product.

General procedure for the synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one derivatives with BNPs-TAPC

To a mixture of aldehyde (1.0 mmol), β -naphthol (1 mmol, 0.144 g), and 5,5-dimethylcyclohexane-1,3-dione (1.0 mmol), was added BNPs-TAPC (10 mg) and heated at 80 °C for an appropriate time (Table 3). The reaction was monitored by TLC. After completion of the reaction, ethanol was added the reaction mixture, and it was stirred for 5 min at 25 °C. The reaction mixture was filtered to remove the catalyst and the filtrate was poured into cold water. The solid was suction filtered, washed with cold water (20 mL \times 2) to afford pure product. The spectral and physical properties of known products were compared with those reported in the literature. In every case excellent agreement was obtained. ¹H and ¹³C NMR spectra for some compound follow.

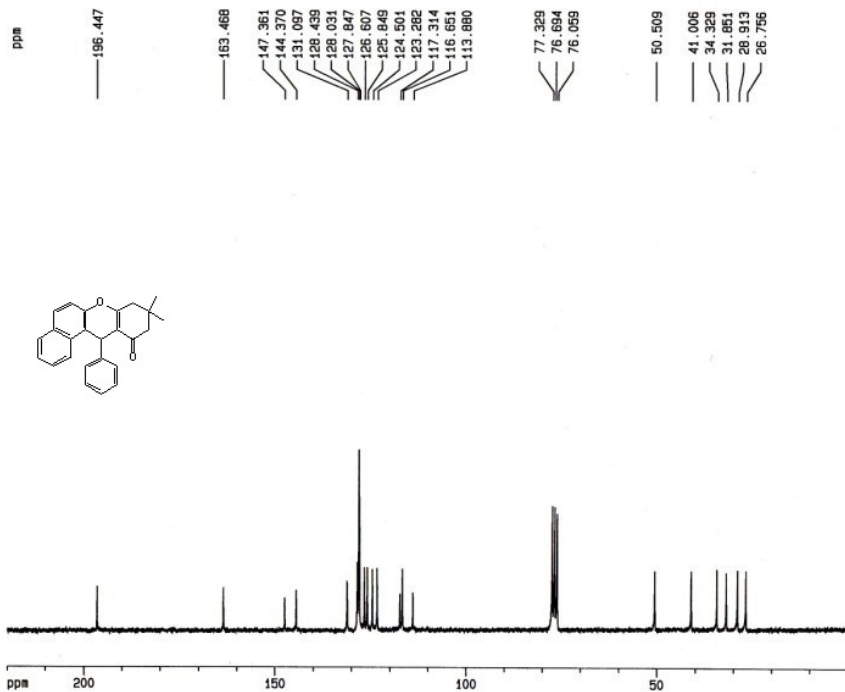
1. L. Rajabi and A. A. Derakhshan, *Sci. Adv. Mater.*, 2010, **2**, 163.

2. R. Steinman, F. B. Jr. Schirmer and L. F. Audrieth, *J. Am. Chem. Soc.*, 1942, **64**, 2377.



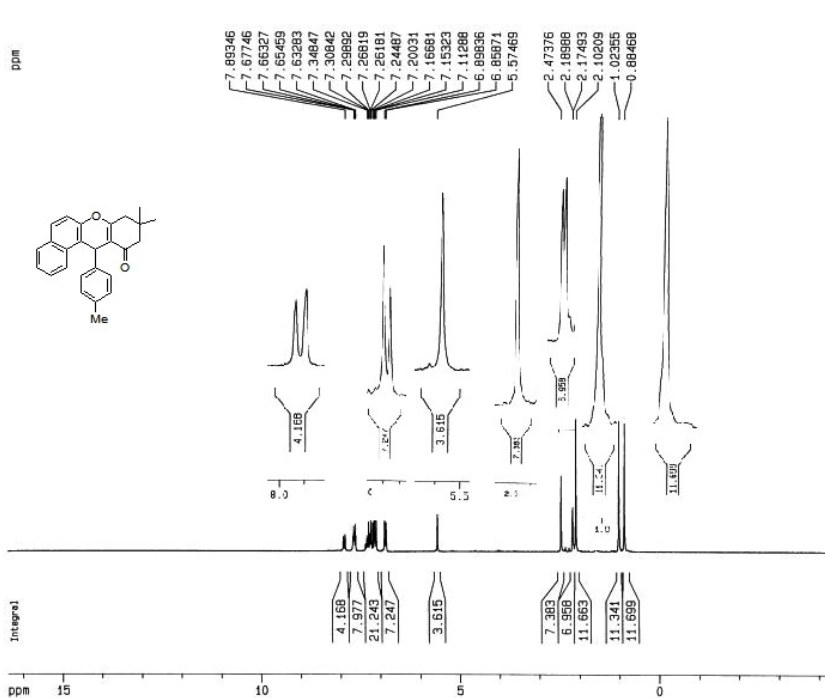
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 SOLVENT CDCl3
 NS 13
 DS 0
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 FIDRES 0.063157 Hz
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 RG 1448.0
 DM 120.800 usec
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 TE 300.0 K
 D1 1.00000000 sec

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 KW EM
 SSB 0
 LB 0.30 Hz
 GB 0
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 1D NMR plot parameters
 CX 20.00 cm
 FIP 10.000 ppm
 F1 2001.30 Hz
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 F2 0.00 Hz
 PPMCM 0.50000 ppm/cm
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Current Data Parameters
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 PROCNO 1
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 SOLVENT CDCl3
 NS 5000
 DS 0
 SMH 12562.814 Hz
 FIDRES 0.191693 Hz
 AQ 2.6083827 sec
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 PL1 0.00 dB
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 NUC2 1H
 P2P2 80.00 usec
 PL2 -3.00 dB
 PL12 20.00 dB
 PL13 20.00 dB
 SFO2 200.1308005 MHz
 F2 - Processing parameters
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 SF 50.3227509 MHz
 KW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40
 1D NMR plot parameters
 CX 20.00 cm
 FIP 220.000 ppm
 F1 11071.00 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 553.55023 Hz/cm



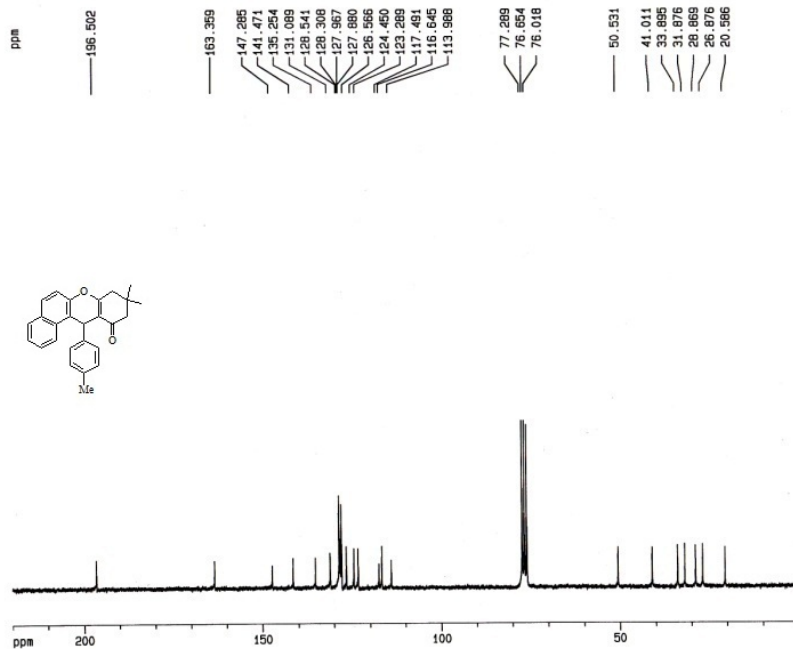
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 NS 12
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 FIDRES 0.063157 Hz
 AQ 7.9167585 sec
 RG 1024
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 P1 8.80 usec
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F2 - Processing parameters
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 SF 200.1300273 MHz
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 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 16.380 ppm
 F1 3278.11 Hz
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 FWHM 1.03410 ppm/cm
 HZCM 206.95363 Hz/cm



Current Data Parameters
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 PROCNO 1

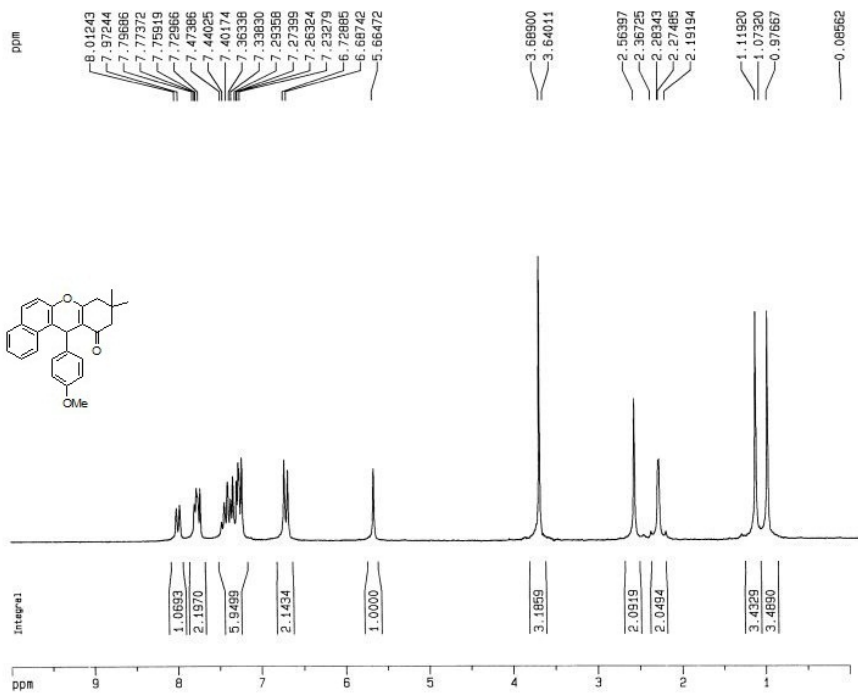
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 TE 300.0 K
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 D11 0.0300000 sec
 D12 0.0000000 sec

----- CHANNEL f1 -----
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 P1 10.30 usec
 PL1 0.00 dB
 SFO1 50.3282440 MHz

----- CHANNEL f2 -----
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 NUC2 1H
 P2 80.00 usec
 PL2 -3.00 dB
 PL12 20.00 dB
 PL13 20.00 dB
 SFO2 200.1300005 MHz

F2 - Processing parameters
 SI 32768
 SF 50.3227509 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
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 F1 11871.00 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 FWHM 11.00000 ppm/cm
 HZCM 563.59023 Hz/cm



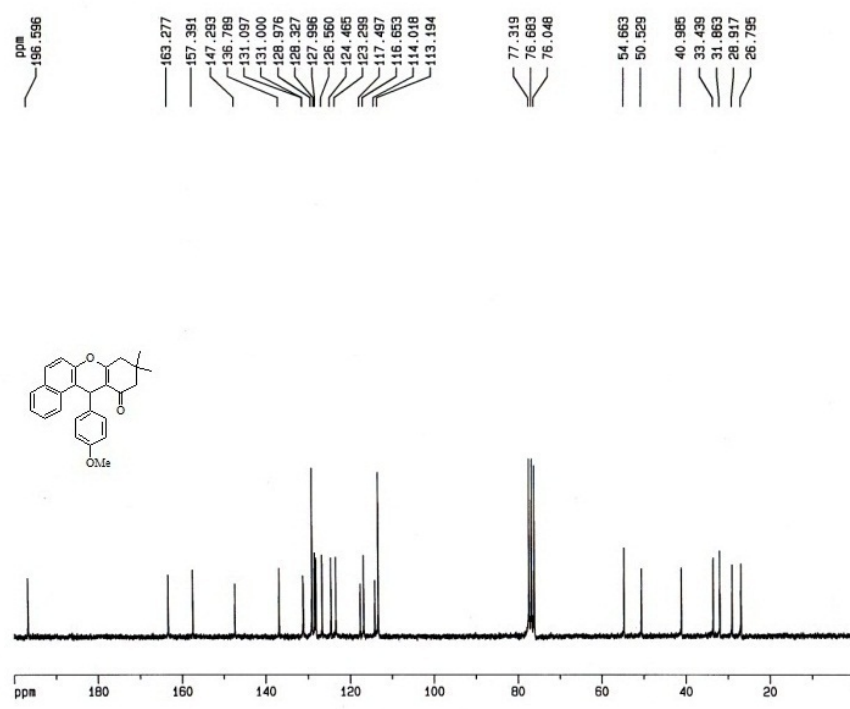
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 SOLVENT CDCl3
 NS 12
 DS 0
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 FIDRES 0.063157 Hz
 AQ 7.9167996 sec
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 TE 300.0 K
 D1 1.00000000 sec

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 P1 8.80 usec
 PL1 -3.00 dB
 SFO1 200.1312359 MHz

F2 - Processing parameters
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 SF 200.1300080 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 2001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCH 0.50000 ppm/cm
 HZCM 100.06500 Hz/cm



Current Data Parameters
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 PROCNO 1

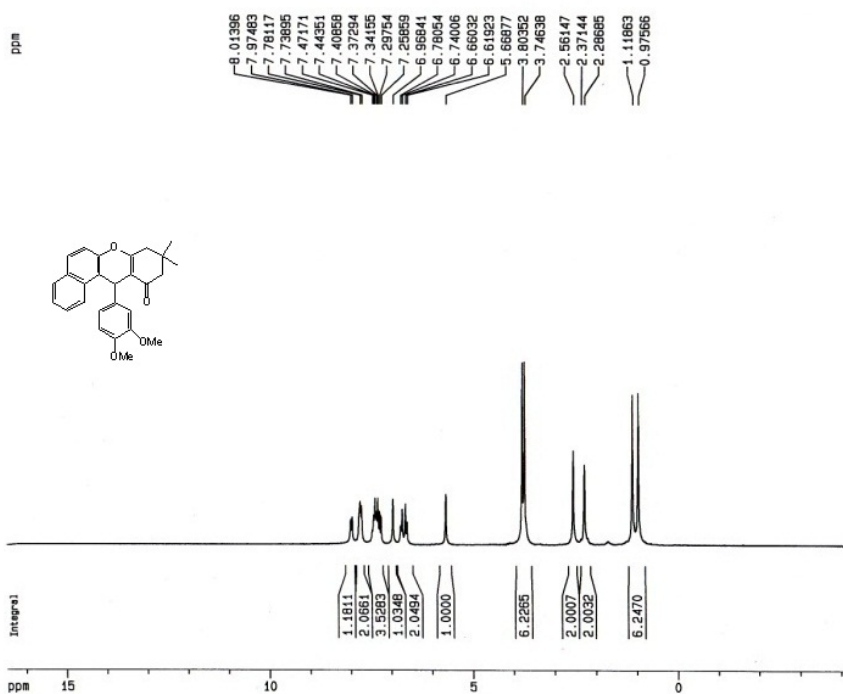
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2222
 DS 0
 SMH 12562.814 Hz
 FIDRES 0.191693 Hz
 AQ 2.6083827 sec
 RG 32000
 DM 39.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 D12 0.00012000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 10.30 usec
 PL1 0.00 dB
 SFO1 50.3282440 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 P2P2 80.00 usec
 PL2 -3.00 dB
 PL12 20.00 dB
 PL13 20.00 dB
 SFO2 200.1308005 MHz

F2 - Processing parameters
 SI 32768
 SF 50.3227509 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 10064.35 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCH 10.00000 ppm/cm
 HZCM 503.22751 Hz/cm



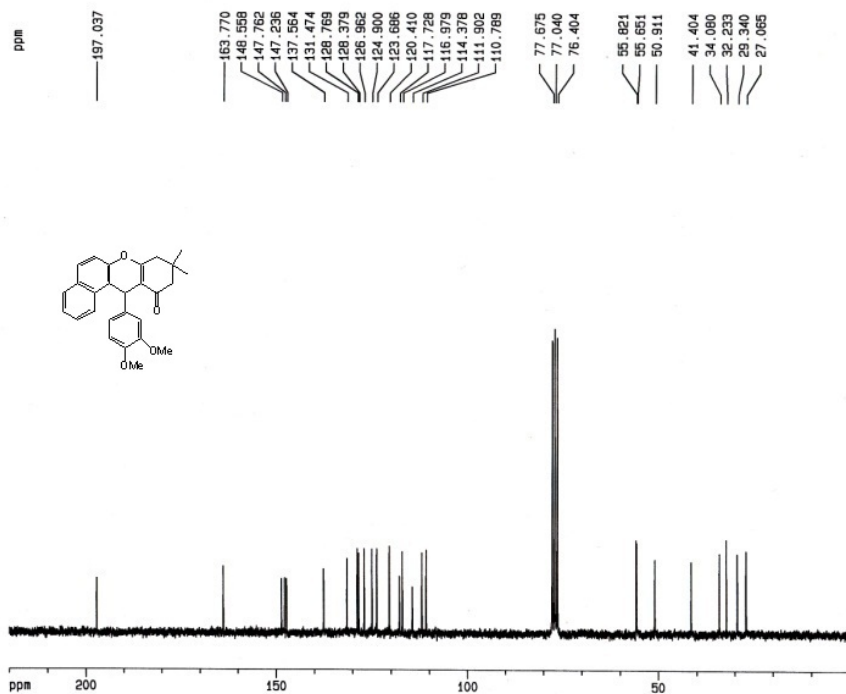
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 PULPROG zg30
 TD 65636
 SOLVENT CDCl3
 NS 12
 DS 0
 SMH 4139.073 Hz
 FIDRES 0.063157 Hz
 AQ 7.9167986 sec
 RG 574.7
 DM 120.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

CHANNEL f1
 NUC1 1H
 P1 8.80 usec
 PL1 -3.00 dB
 SFO1 200.1312359 MHz

F2 - Processing parameters
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 SF 200.1300064 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
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 F1 3297.01 Hz
 F2P -4.208 ppm
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 PPMCH 1.03410 ppm/cm
 HZCM 206.95364 Hz/cm



Current Data Parameters
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 EXPNO 28
 PROCNO 1

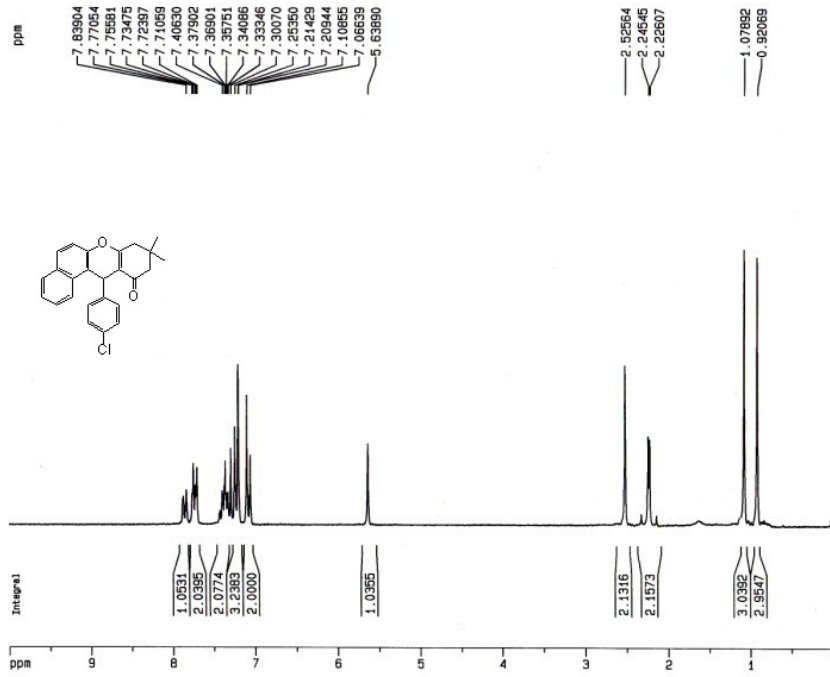
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 SOLVENT CDCl3
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 DS 0
 SMH 12562.814 Hz
 FIDRES 0.191693 Hz
 AQ 2.6083827 sec
 RG 32000
 DM 39.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec

CHANNEL f1
 NUC1 13C
 P1 10.30 usec
 PL1 0.00 dB
 SFO1 50.3282440 MHz

CHANNEL f2
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 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 20.00 dB
 PL13 20.00 dB
 SFO2 200.1308005 MHz

F2 - Processing parameters
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 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 11071.00 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCH 11.00000 ppm/cm
 HZCM 553.55005 Hz/cm



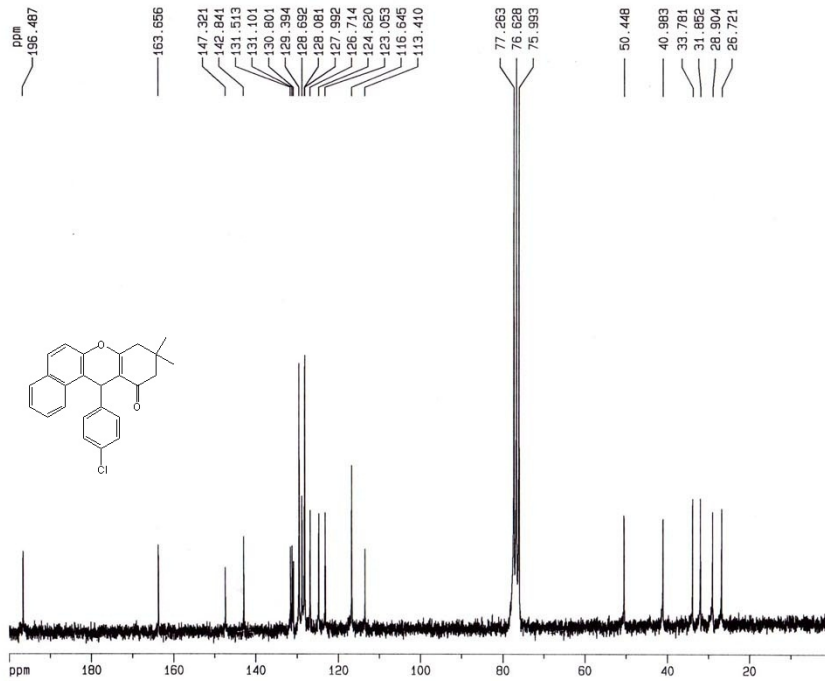
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 PROCNO 1

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 Time 5.16
 INSTRUM spect
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 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 12
 DS 0
 SMH 4139.073 Hz
 FIDRES 0.063157 Hz
 AQ 7.9167996 sec
 RG 1149.4
 DM 120.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 8.80 usec
 PL1 -3.00 dB
 SFO1 200.1312359 MHz

F2 - Processing parameters
 SI 32768
 SF 200.1300173 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 2001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PRNCH 0.50000 ppm/cm
 HZCM 100.05501 Hz/cm



Current Data Parameters
 NAME 13C
 EXPNO 5
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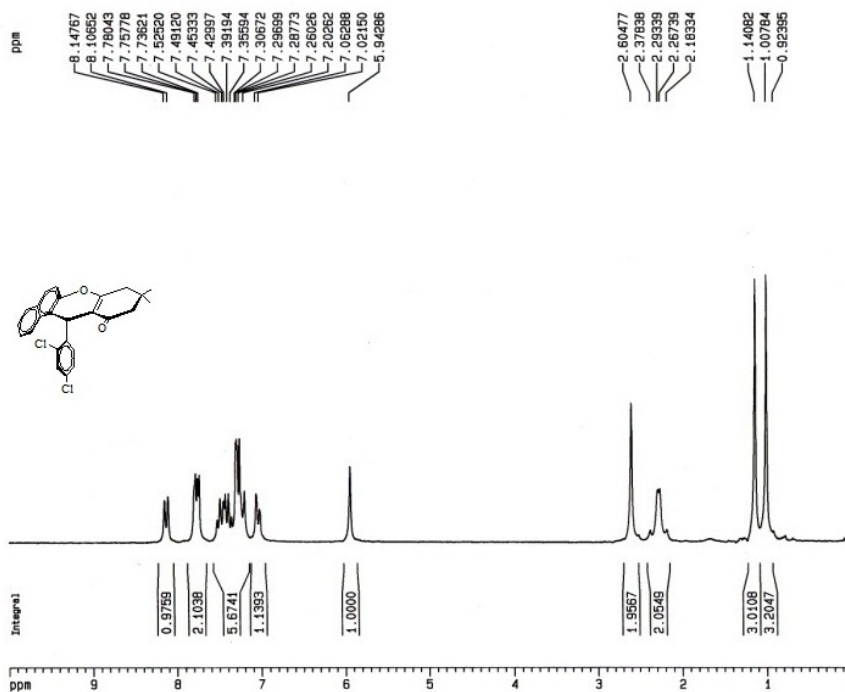
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 FIDRES 0.191693 Hz
 AQ 2.6083827 sec
 RG 32000
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 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.33000000 sec
 D12 0.00020000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 10.30 usec
 PL1 0.00 dB
 SFO1 50.3282440 MHz

----- CHANNEL f2 -----
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 NUC2 1H
 P2P2 80.00 usec
 PL2 -3.00 dB
 PL12 20.00 dB
 PL13 20.00 dB
 SFO2 200.1309005 MHz

F2 - Processing parameters
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 SF 50.3227509 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

ID NMR plot parameters
 CX 20.00 cm
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 F1 10064.55 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PRNCH 10.00000 ppm/cm
 HZCM 503.22751 Hz/cm



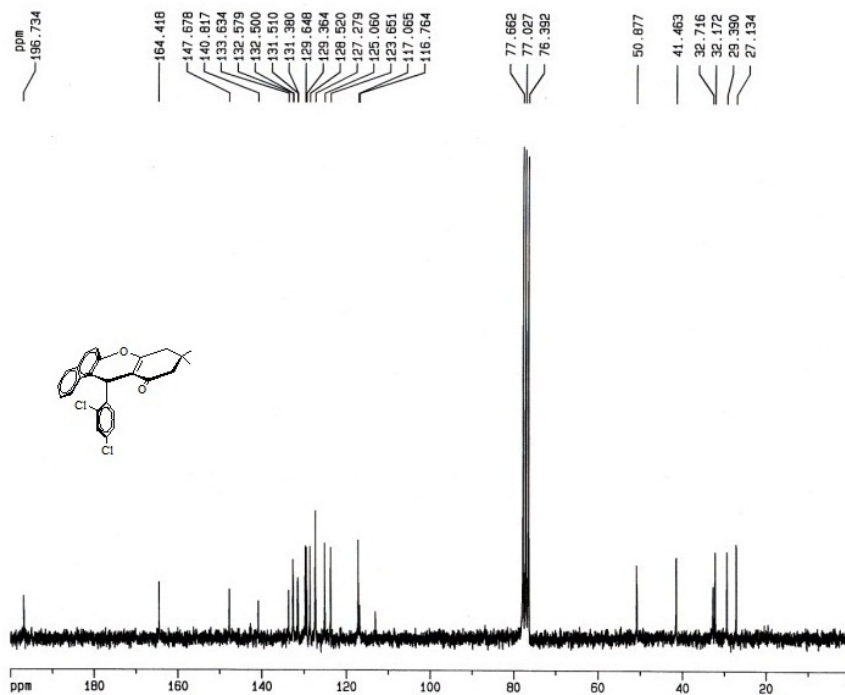
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 SOLVENT CDCl3
 NS 12
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 FIDRES 0.083157 Hz
 AQ 7.9167986 sec
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CHANNEL f1
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 PL1 -3.00 dB
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F2 - Processing parameters
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 SF 200.1300064 MHz
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 LB 0.30 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
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 F1 2001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
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 HZCH 100.05500 Hz/cm



Current Data Parameters
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 PROCNO 1

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 SOLVENT CDCl3
 NS 1988
 DS 0
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 FIDRES 0.191693 Hz
 AQ 2.5083827 sec
 RG 32000
 DM 39.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec

CHANNEL f1
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 P1 10.30 usec
 PL1 0.00 dB
 SFO1 50.3282440 MHz

CHANNEL f2
 CPDPRG2 waltz16
 NUC2 1H
 PCDPRG2 80.00 usec
 PL2 -3.00 dB
 PL12 20.00 dB
 PL13 20.00 dB
 SFO2 200.1308005 MHz

F2 - Processing parameters
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 MH EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

ID NMR plot parameters
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 F1P 200.000 ppm
 F1 10054.95 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PRNCH 10.00000 ppm/cm
 HZCH 503.22729 Hz/cm

