# Supplementary Material
# Sulfated tungstate catalyzed hydration of alkynes

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## A) Experimental

### a) Reagents

All chemicals were purchased from Spectrochem Pvt. Ltd. India and were used without further purification.

### b) Preparation of sulfated tungstate

Anhydrous sodium tungstate (32.9 g, 0.1 mol) was added portionwise, maintaining the temperature between 0 to 5 °C, to a stirred solution of chlorosulfonic acid (23.2 g, 0.2 mol) in chloroform (150 mL) contained in a 250 mL round bottom flask fitted with $CaCl_2$ drying tube, placed in an ice bath. After completion of addition, the mixture was stirred further for 1 h. The pale yellow solid obtained was filtered, washed repeatedly with deionized water until filtrate was neutral and free from chloride ions (detected by $AgNO_3$ test) and dried in an oven for 2 h at 100 °C to get 33 g (86% w/w) of sulfated tungstate. (For detail characterization Ref 16)

### a) Typical procedure for hydration of phenylacetylene (Table 2 entry 1):

Sulfated tungstate (0.20 g, 20 wt%) was added to a mixture of phenylacetylene (1.00 g, 9.79 mmol) and water (0.74 g, 39.16 mmol) and the resulting suspension was stirred at 100 °C. The progress of the reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was cooled, diluted with EtOAc (10 mL) and filtered. The catalyst was washed with EtOAc (10 mL), and the organic extracts were combined and concentrated under reduced pressure to give crude product which was purified by chromatography on silica gel (#60-120) with *n*-hexane/EtOAc (90:10) as eluent to get pure acetophenone (1.12 g) as a colorless liquid.

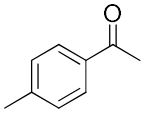
## B) Analytical Data of selected compounds:

**Acetophenone (Table 2, entry 1)[1]:**



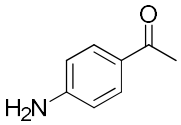
Colorless liquid; $R_f$ 0.47 (*n*-hexane/EtOAc, 90:10); **IR:** 1681, 1598, 1582, 1448, 1358, 1263, 1180, 1078, 1024 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$): $\delta_H$ 7.93-7.95 (m, 2H), 7.52-7.56 (m, 1H), 7.44-7.46 (m, 2H), 2.58 (s, 3H).

**4-Methylacetophenone (Table 2, entry 2)[1]:**



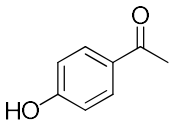
Colorless liquid; $R_f$ 0.47 (*n*-hexane/EtOAc, 90:10); **IR:** 1687, 1599, 1586, 1450, 1354, 1265, 1181, 1077, 1026 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$) $\delta_H$ 7.83 (d, *J* 8.1 Hz, 2H), 7.23 (d, *J* 8.1 Hz, 2H), 2.38 (s, 3H), 2.55 (s, 3H).

**4-Aminoacetophenone (Table 2, entry 3)[2]:**



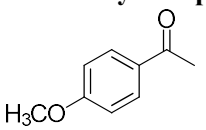
Brown solid; mp 102-104 °C; (lit.,[2] 104-106 °C); $R_f$ 0.13 (*n*-hexane/EtOAc, 90:10); **IR**(KBr): 3333, 2924, 1642 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$): $\delta_H$ 7.79 (d, *J* 8.6 Hz, 2H), 6.63 (d, *J* 8.6 Hz, 2H), 4.11 (br s, 2H), 2.48 (s, 3H).

**4-Hydroxyacetophenone (Table 2, entry 4)[3]:**



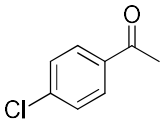
Colorless solid; mp 110-111 °C; (lit.,[3] 109-111 °C); $R_f$ 0.13 (*n*-hexane/EtOAc, 90:10); **IR** (KBr): 3311, 1663, 1614, 1507, 1239, 1072 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$): $\delta_H$ 7.59 (s, 1H), 7.89 (d, *J* 8.5 Hz, 2H), 6.92 (d, *J* 8.5 Hz, 2H), 2.56 (s, 3H).

**4-Methoxyacetophenone (Table 2, entry 5)[4]:**



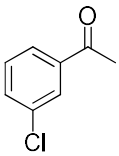
Colorless solid; mp 36-38 °C (lit.,[4] 37-38 °C); $R_f$ 0.31 (*n*-hexane/EtOAc, 90:10); **IR** (KBr): 1683, 1597, 1584, 1450 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$): $\delta_H$ 7.89 (d, *J* 9.0 Hz, 2H), 6.88 (d, *J* 9.0 Hz, 2H), 3.81 (s, 3H), 2.50 (s, 3H).

**4-Chloroacetophenone (Table 2, entry 6)[1]:**



Colorless liquid; $R_f$ 0.45 (*n*-hexane/EtOAc, 90:10); **IR:** 1696, 1590, 1588, 1454 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$): $\delta_H$ 7.86 (d, *J* 8.3 Hz, 2H), 7.39 (d, *J* 8.3 Hz, 2H), 2.55 (s, 3H).

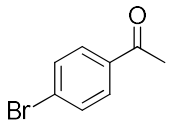
**3-Chloroacetophenone (Table 2, entry 7)[5]:**



Colorless liquid; $R_f$ 0.45 (*n*-hexane/EtOAc, 90:10); **IR**: 1688, 1572, 1422, 1358, 1251 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$): $\delta_H$ 7.52 (dd, *J* 2.0, 1.2 Hz, 1H), 7.31-740 (m, 2H), 7.27-7.29 (m, 1H), 2.62 (s, 3H).

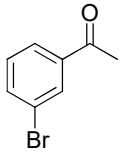

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**4-Bromoacetophenone (Table 2, entry 8)[2]:**



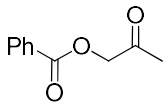

Colorless liquid; $R_f$ 0.43 (*n*-hexane/EtOAc, 90:10); **IR**: 1673, 1587, 1395, 1269, 1078 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$): $\delta_H$ 7.79 (d, *J* 8.7 Hz, 2H), 7.58 (d, *J* 8.7 Hz, 2H), 2.56 (s, 3H).

**3-Bromoacetophenone (Table 2, entry 9)[6]:**



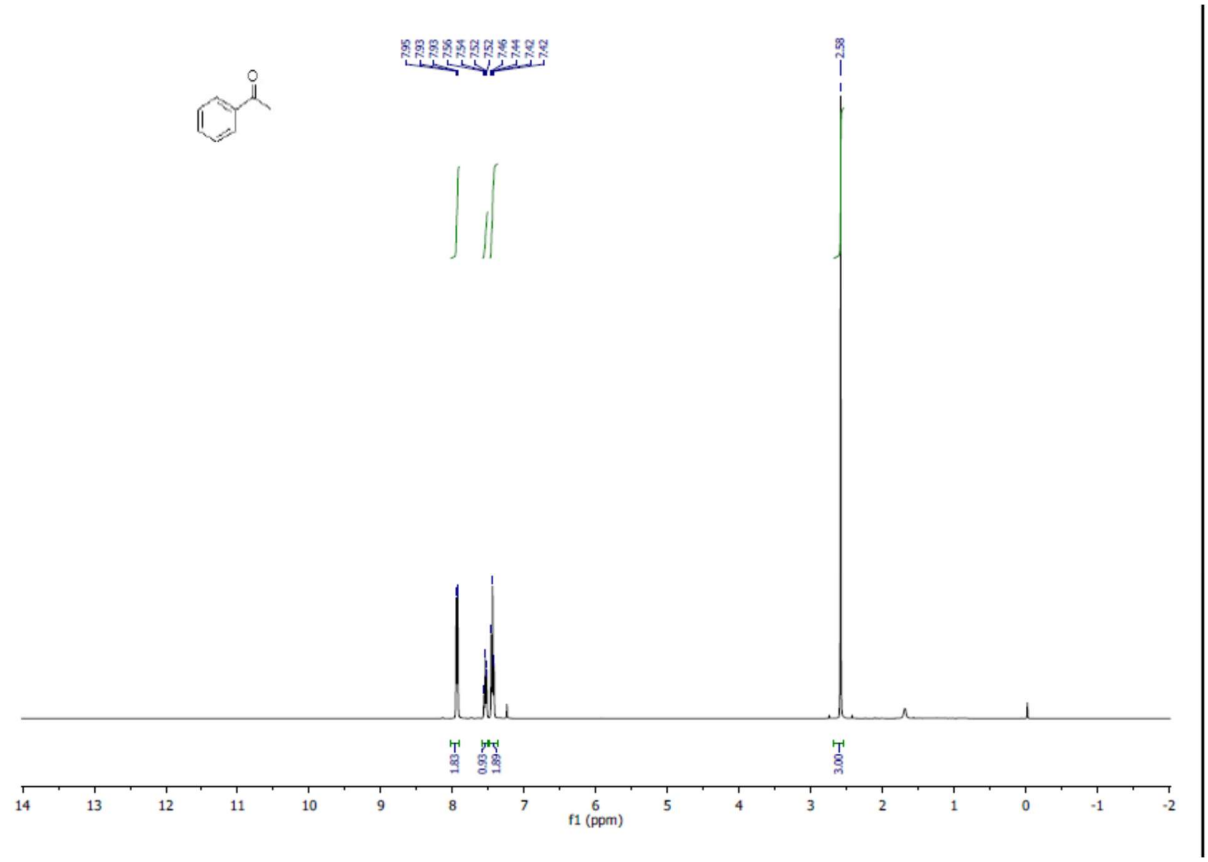

Colorless liquid; $R_f$ 0.43 (*n*-hexane/EtOAc, 90:10); I**R**: 1672, 1586, 1396, 1270, 1077 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$): $\delta_H$ 8.02 (dd, *J* 1.8 Hz, 1H), 7.82-7.84 (m, 1H), 7.63-7.65 (m, 1H), 7.29-7.30 (m, 1H), 2.54(s, 3H).

**2-Oxopropyl benzoate (Table 2, entry 11)[7]:**




Colorless liquid; $R_f$ 0.47 (*n*-hexane/EtOAc, 90:10); **IR**: 1726, 1741 $cm^{-1}$; **$^1$H NMR** (300 MHz; $CDCl_3$; $Me_4Si$): $\delta_H$ 7.97-8.00 (m, 2H), 7.45-7.50 (m, 1H), 7.32-7.36 (m, 2H), 4.77 (s, 2H), 2.10 (s, 3H).

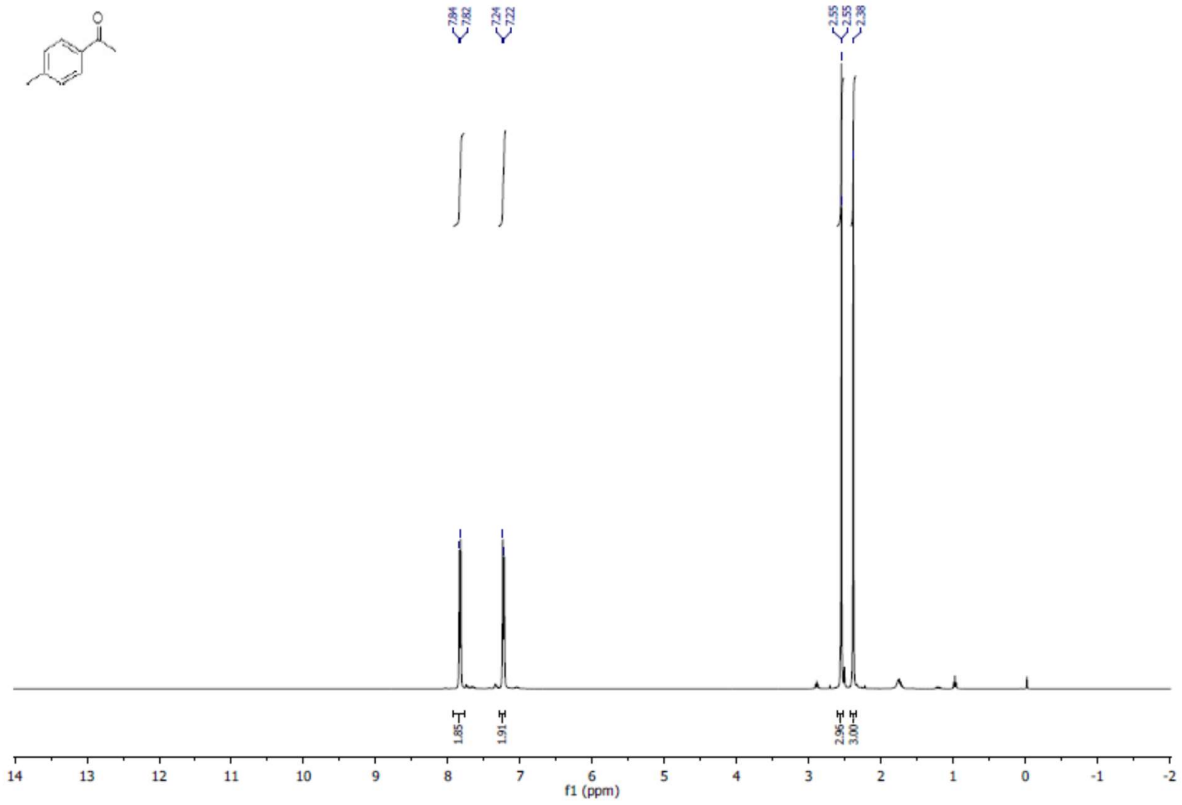
**1. Acetophenone (Table 2, entry 1) $^{1}$H NMR spectrum**





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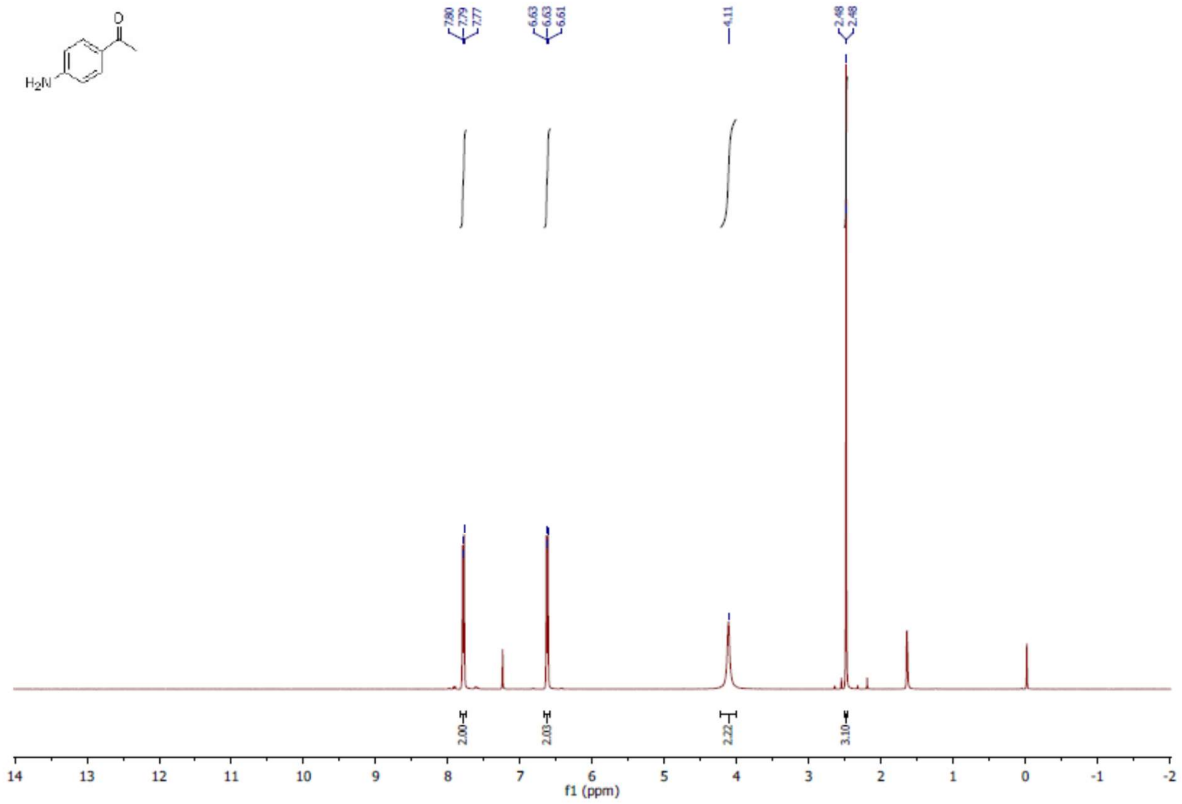
**2. 4-Methylacetophenone (Table 2, entry 2) $^{1}$H NMR spectrum**





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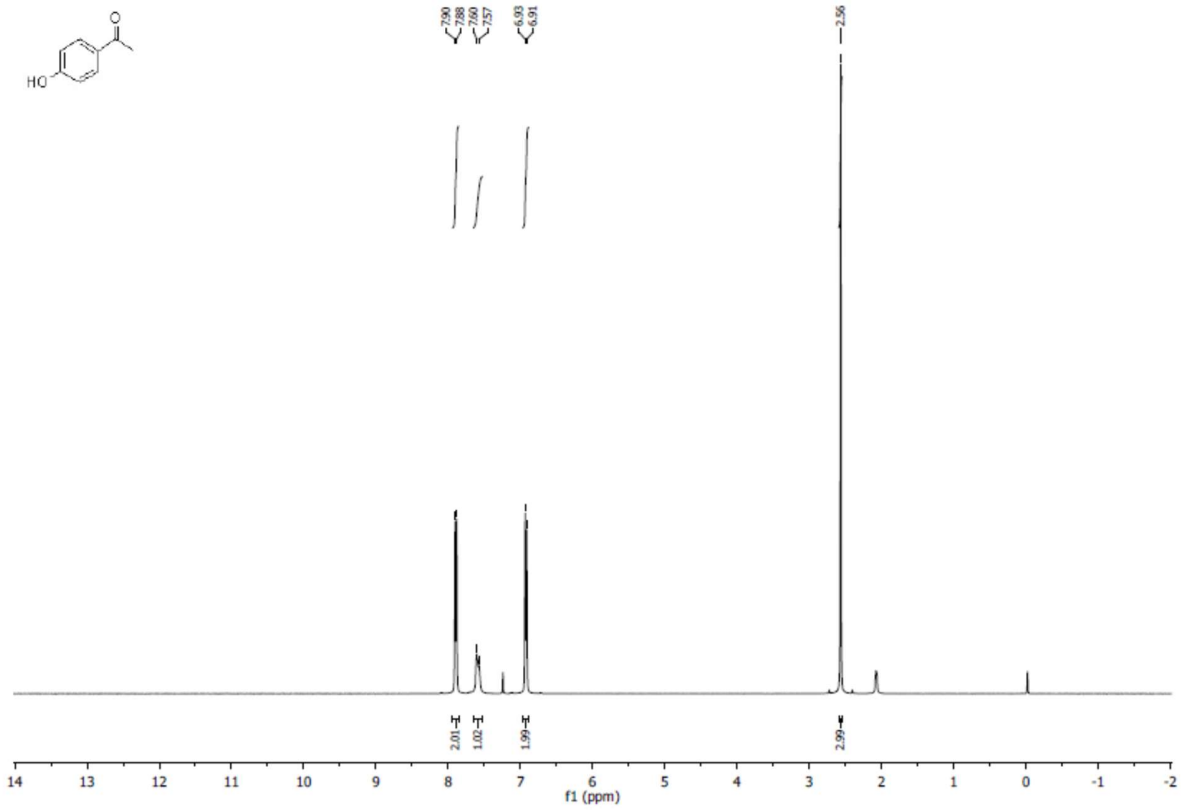
**3. 4-Aminoacetophenone (Table 2, entry 3) $^{1}$H NMR spectrum**





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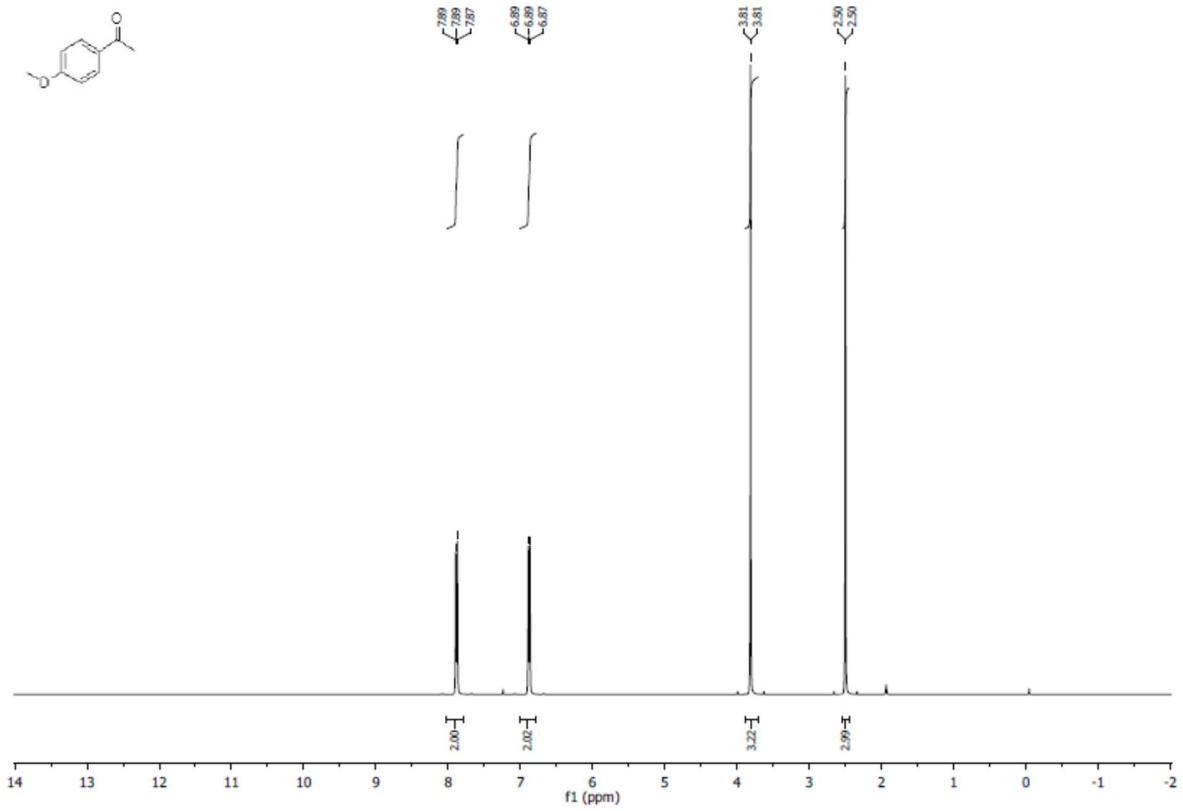
**4. 4-Hydroxyacetophenone (Table 2, entry 4) $^{1}$H NMR spectrum**





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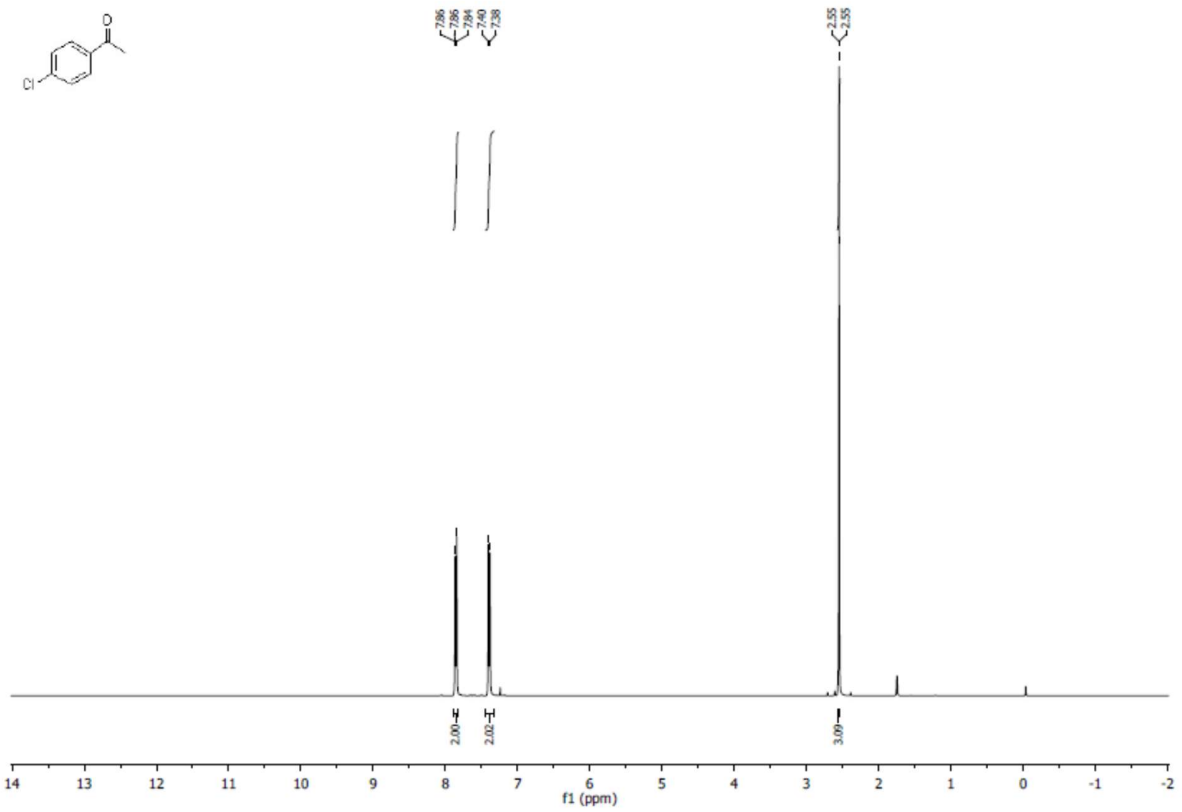
**5. 4-Methoxyacetophenone (Table 2, entry 5) $^{1}$H NMR spectrum**





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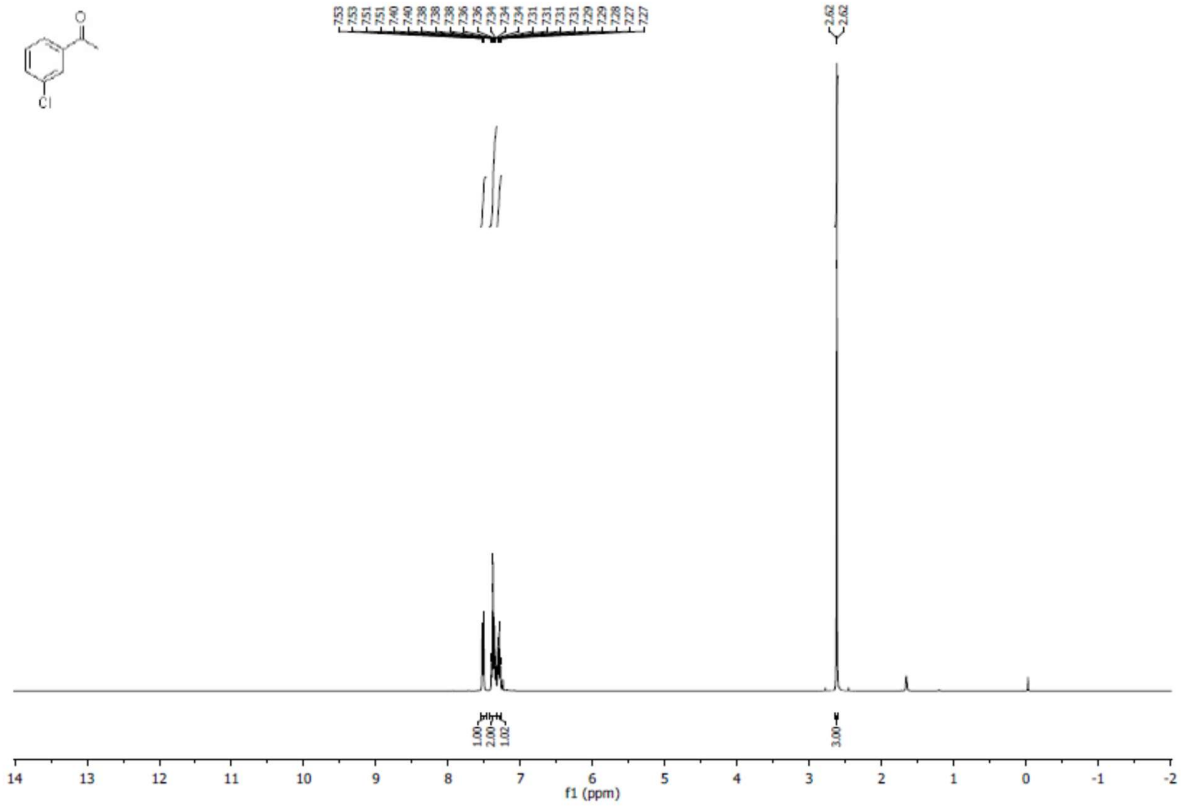
6. 4-Chloroacetophenone (Table 2, entry 6) $^{1}$H NMR spectrum



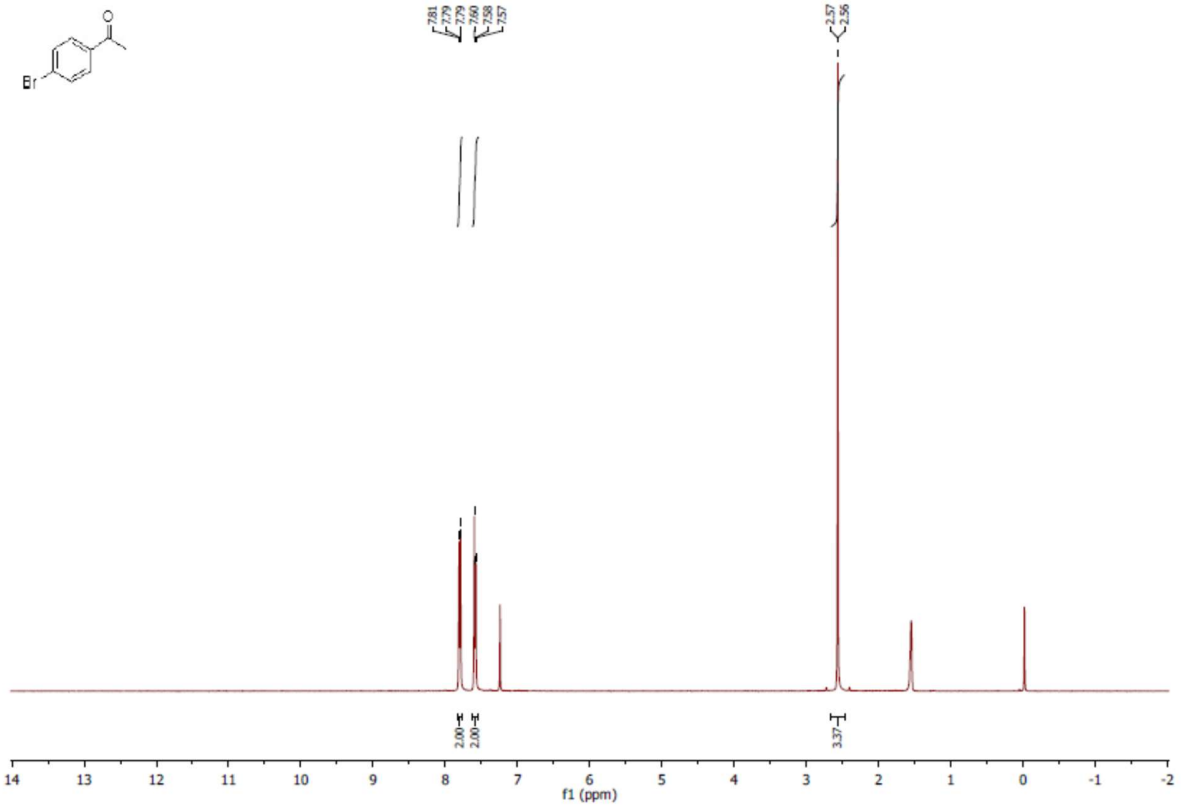


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**7. 3-Chloroacetophenone (Table 2, entry 7) $^{1}$H NMR spectrum**



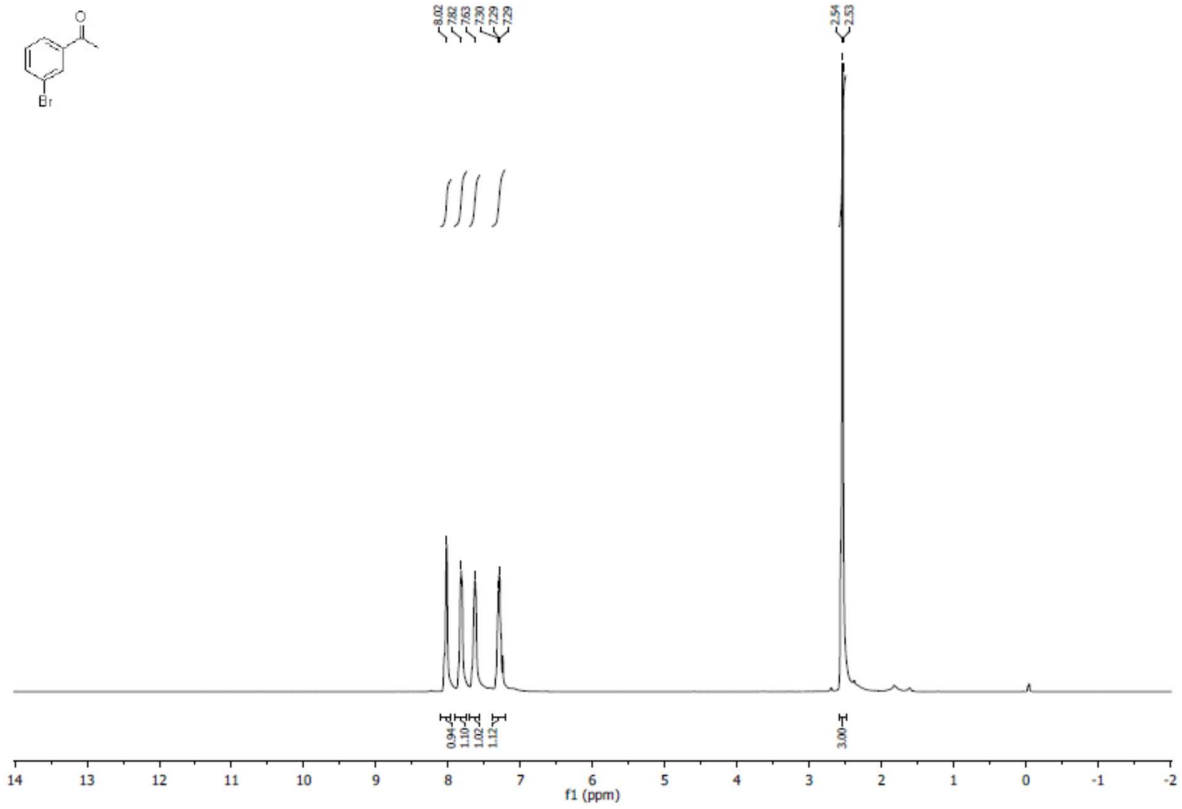
**8. 4-Bromoacetophenone (Table 2, entry 8) $^{1}$H NMR spectrum**





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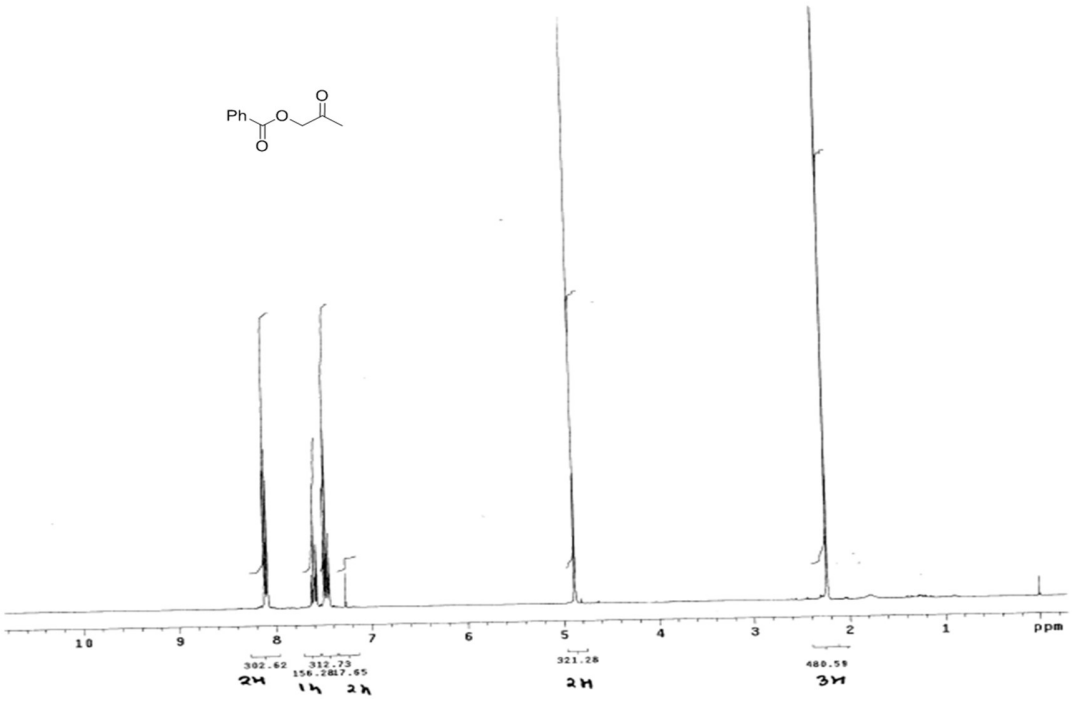
**9. 3-Bromoacetophenone (Table 2, entry 9) $^{1}$H NMR spectrum**





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**3. 2-Oxopropyl benzoate (Table 2, entry 11) $^1$H NMR spectrum**




**C) Reference:**


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