10.1071/CH12321_AC

Australian Journal of Chemistry 2012, 65(12), 1594-1598

SUPPLEMENTARY MATERIAL

Direct One-Pot Cobalt(II) Phthalocyanine Catalyzed Synthesis of *N*-Substituted Isoindolinones

Vishal Kumar, Upendra Sharma, Bikram Singh, Neeraj Kumar*

CSIR-Institute of Himalayan Bioresource Technology (Council of Scientific & Industrial Research), Palampur, Himachal Pradesh 176 061, India

E-mail: neerajnpp@rediffmail.com

Table of Contents

General Experimental

Metal salts used were purchased from Merck, Germany. Cobalt phthalocyanines was synthesized by a reported procedure with some modification and characterized by FTIR and UV-VIS spectroscopy. Silica gel (60-120 mesh) used for column chromatography was purchased from Sisco Research Laboratories Pvt. Ltd. India and all other chemicals were purchased from Spectrochem, India, Merck, Germany, and Sigma-Aldrich, USA and were used without further purification. NMR spectra were recorded on a Bruker Avance-300 spectrometer. Mass spectra were recorded on QTOF-Micro of Waters Micromass and Maxis-Bruker. The GC-MS analysis was carried out on a Shimadzu (QP 2010) series Gas Chromatogram-Mass Spectrometer (Tokyo, Japan), AOC-20i auto-sampler coupled, and a DB-5MS capillary column, (30 m x 0.25 mm i.d., 0.25μm). The initial temperature of column was 70 °C held for 4 min. and was programmed to 230 °C at 4°C/min., then held for 15 min. at 230 °C; the sample injection volume was 2 μl in GC grade dichloromethane. Helium was used as carrier gas at a flow rate of 1.1 ml min^{-1} on split mode (1:50).

Synthesis of Cobalt (II) phthalocyanine

A mixture of phthalimide (26.28 g, 0.18 mol), urea (55.2 g, 0.92 mol), $CoCl_2.6H_2O$ (11.85 g, 0.05 mol) and ammonium molybdate (4.69 g, 0.0038 mol) was heated under microwave irradiation for 3 min. The reaction mixture was cooled to room temperature and in sequence washed with 5% NaOH, distilled water and 2% HCl and finally with distilled water again. After

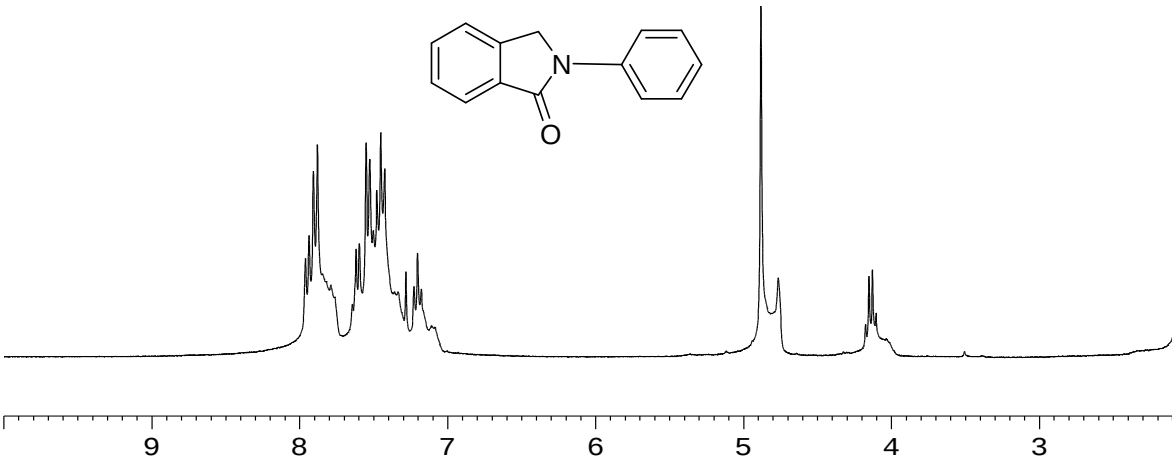
that the resulting solid was dissolved in minimum quantity of concentrated H_2SO_4 and poured in distilled water to precipitate the desired cobalt (II) phthalocyanine, which were then filtered to give 9.5 g (48.5% yield) of cobalt (II) phthalocyanine.

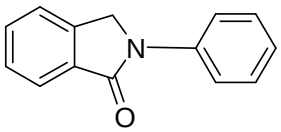
General procedure for the synthesis of *N*-substituted isoindolinones

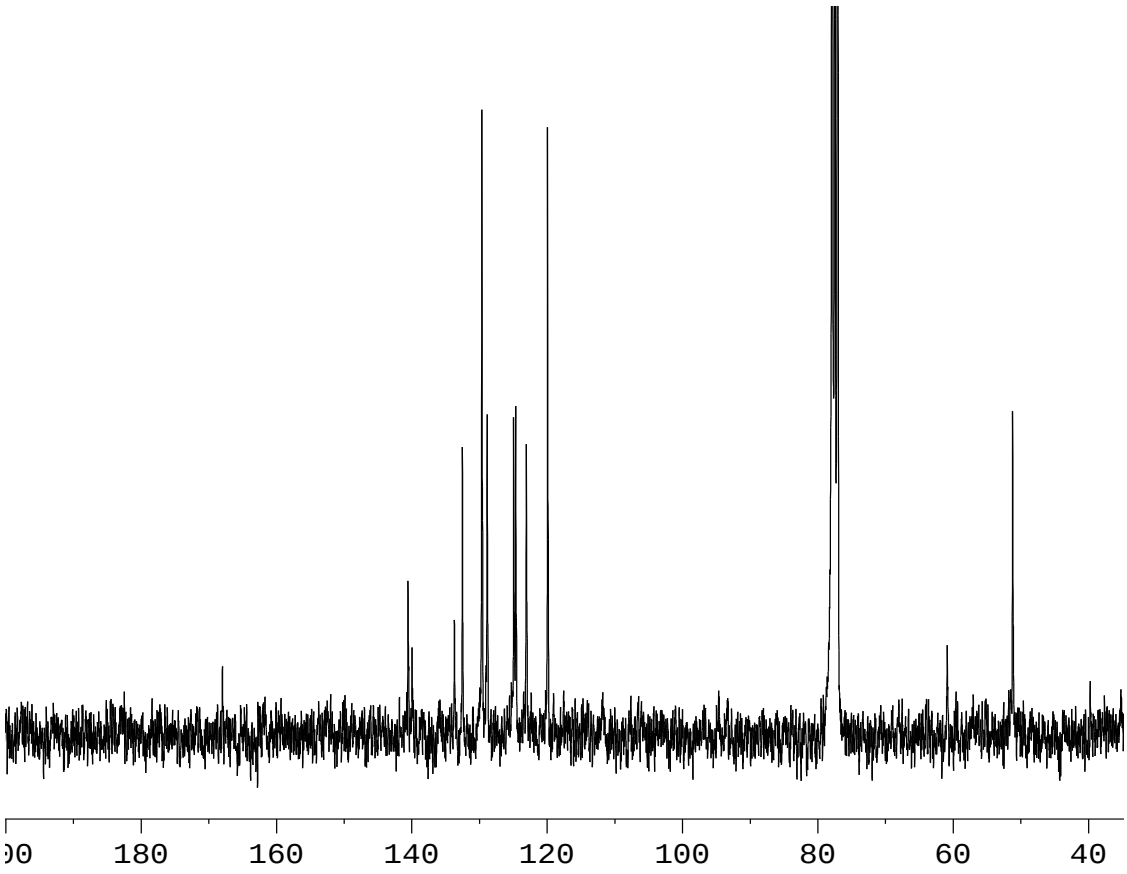
To a stirred suspension of CoPc (0.01 mmol) in ethanol were added 2-carboxybenzaldehyde (1.0 mmol), amine (1.0 mmol) and diphenylsilane (1.5 mmol) at room temperature and then the temperature was raised to 70 ^oC. On completion of the reaction (as monitored by TLC), reaction mixture was filtered and passed through anhydrous Na_2SO_4. The crude product was purified by column chromatography over silica-gel (60-120) mesh.

NMR spectra of isolated compounds

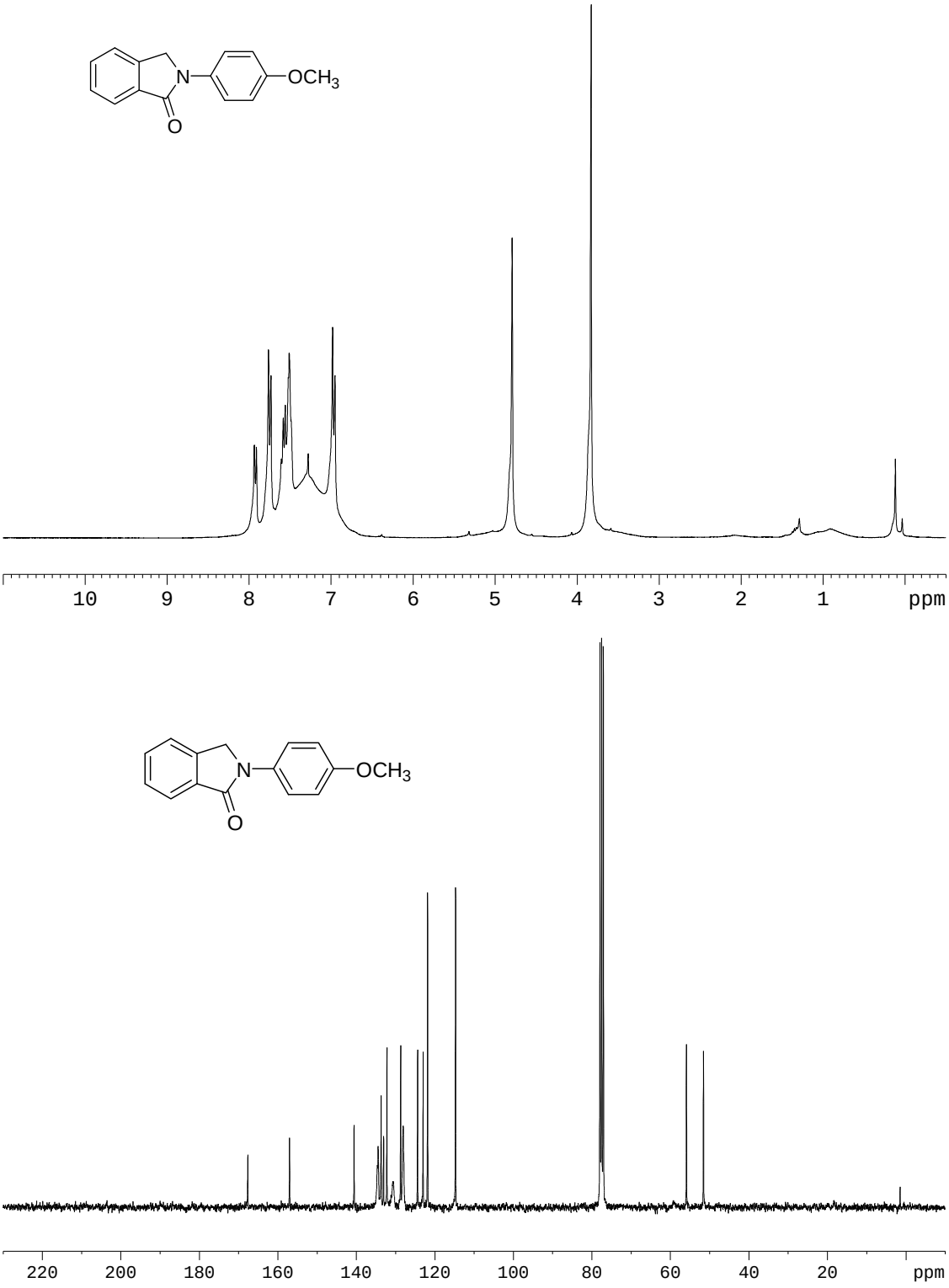
***N*-Phenylisoindolone 3a (Table 2, entry 1)**



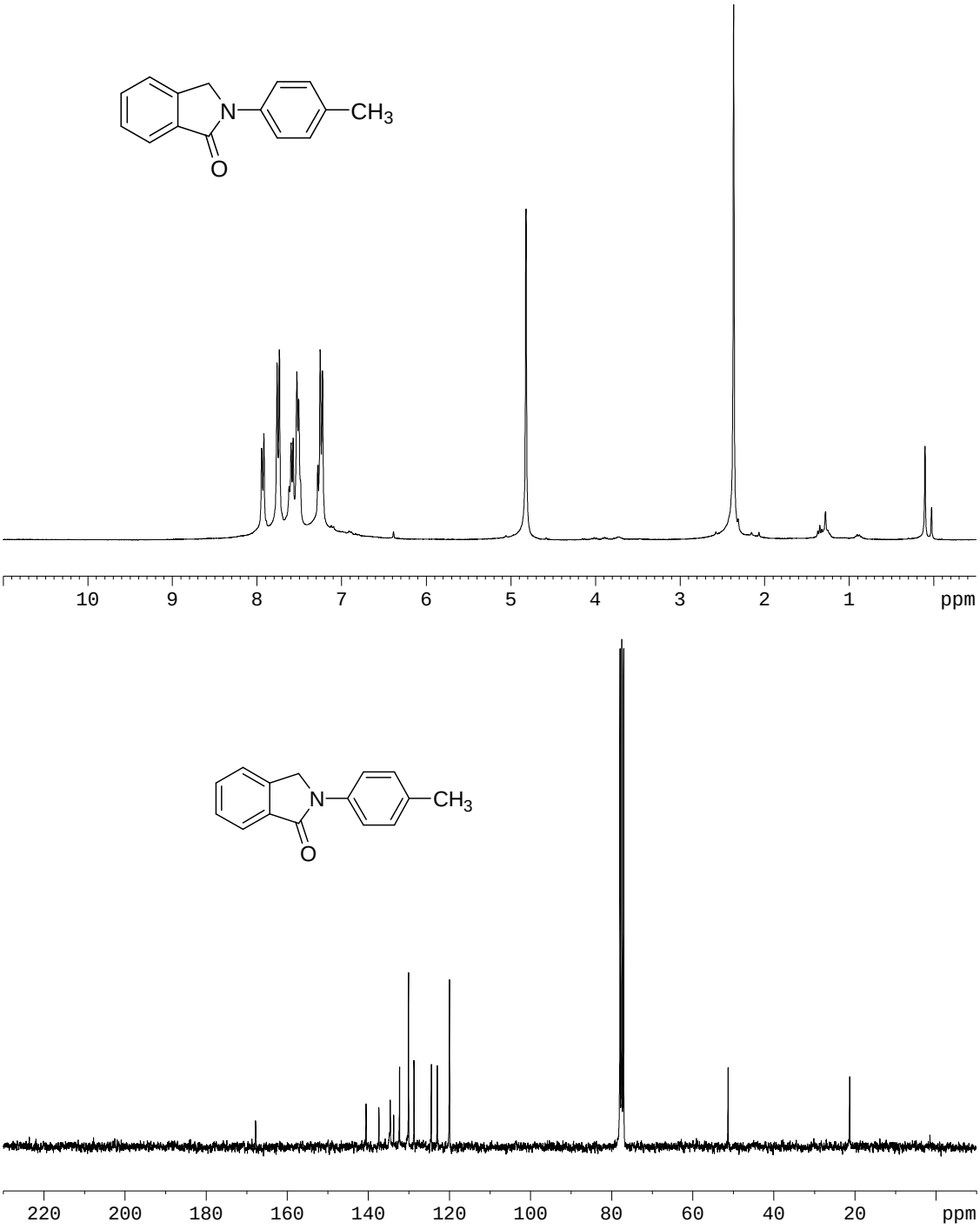




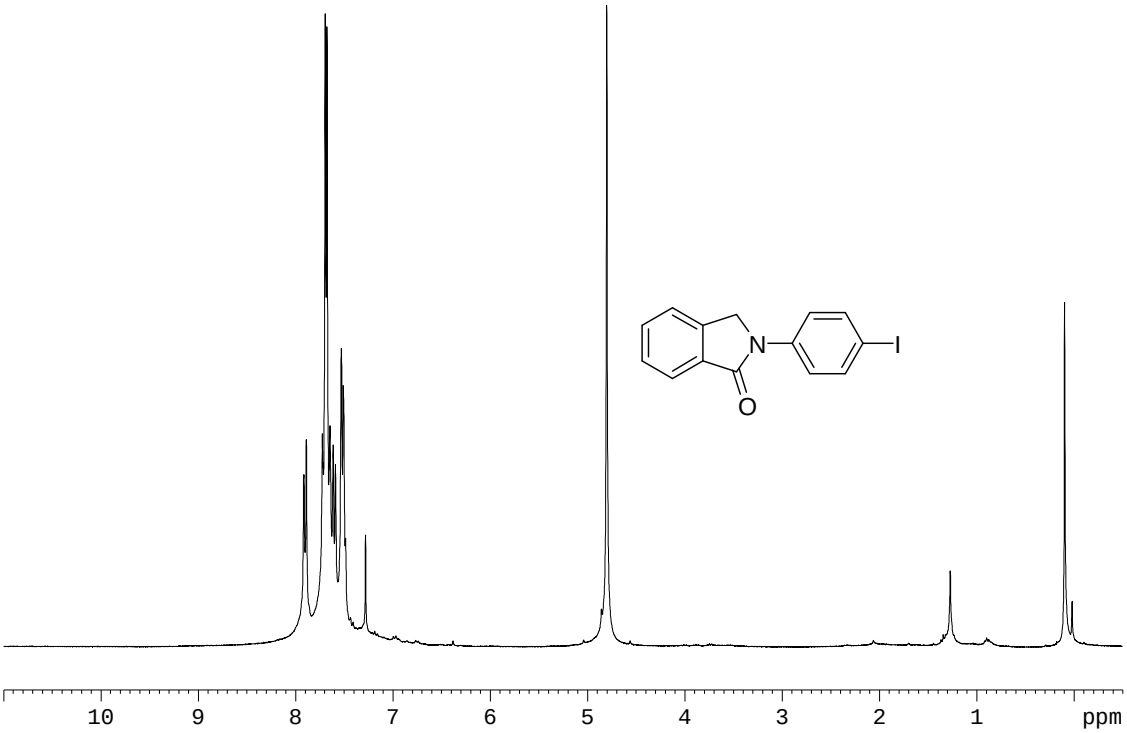
***N*-(4-methoxyphenyl)isoindolinone 3b (Table 2, entry 2)**

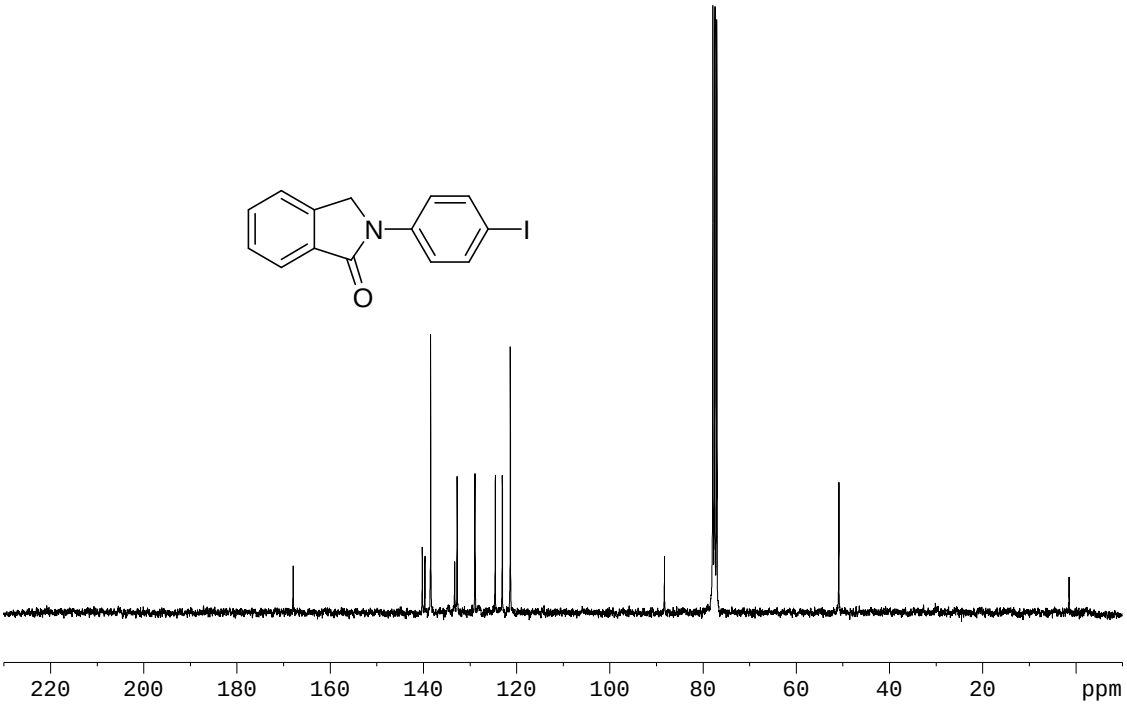


***N*-(4-methylphenyl)isoindolinone 3c (Table 2, entry 3)**

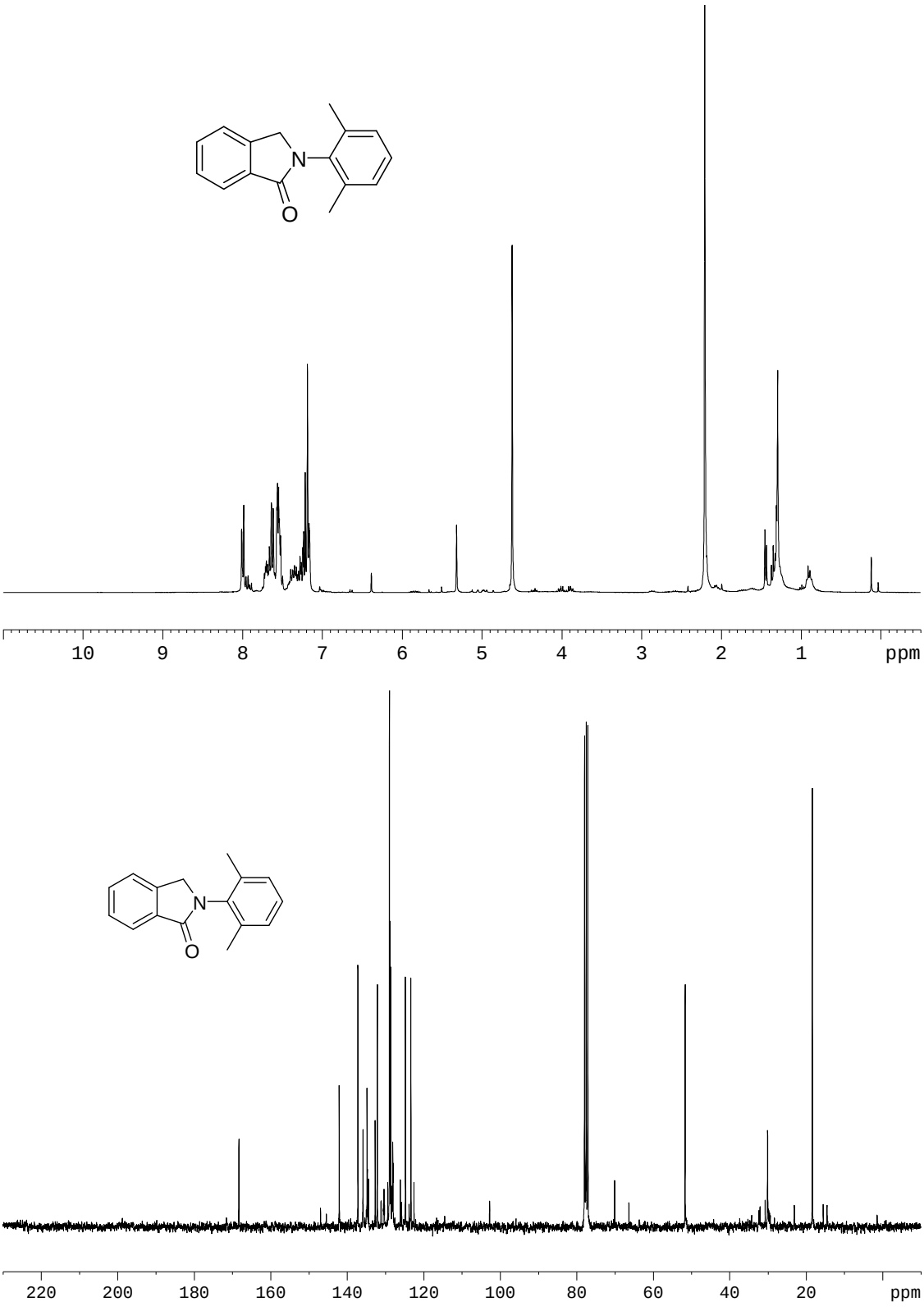


***N*-(4-Iodophenyl)isoindolinone 3d (Table 2, entry 4)**

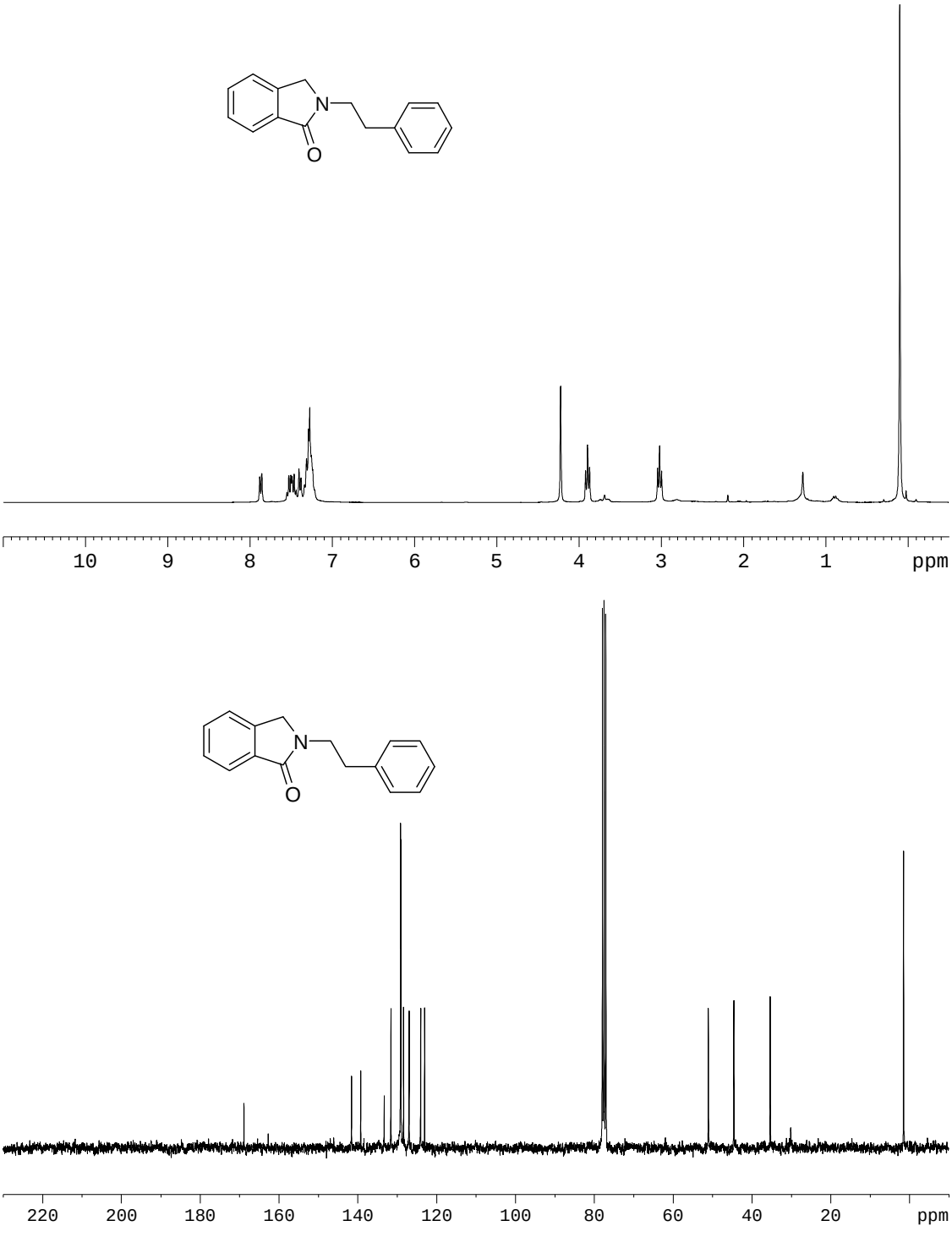




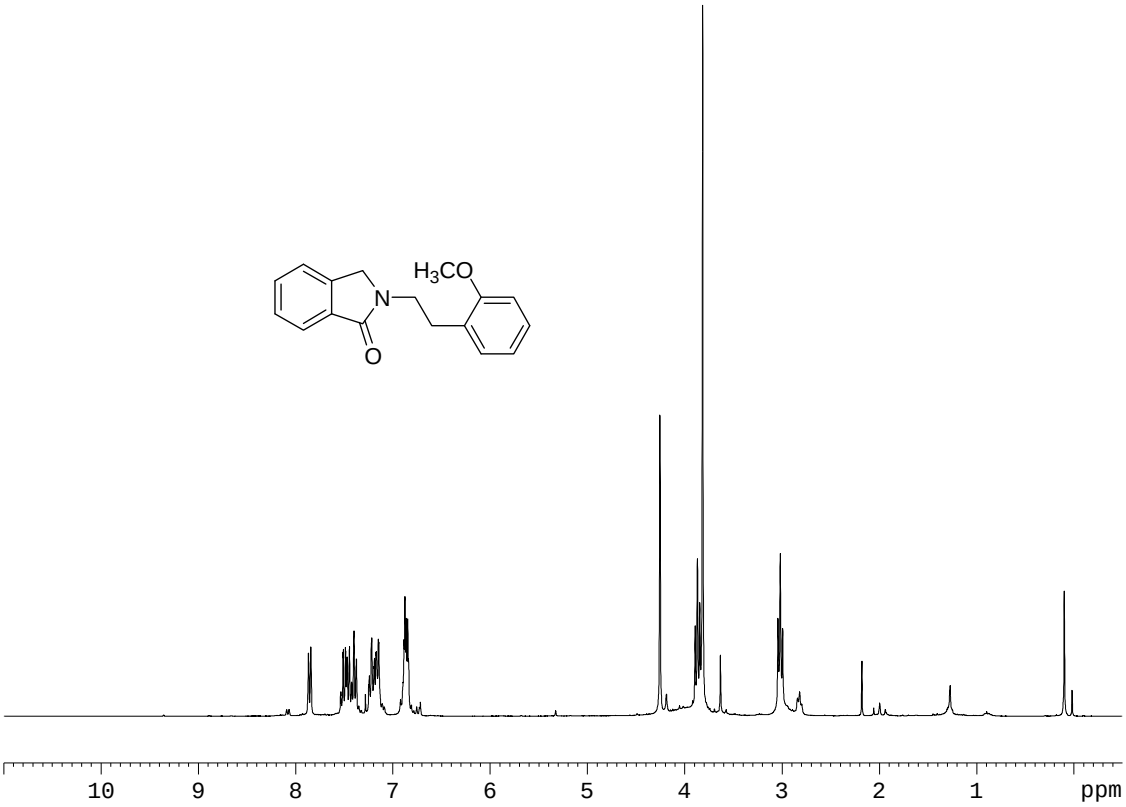
***N*-(2,6-dimethylphenyl)isoindolinone 3h (Table 2, entry 8)**

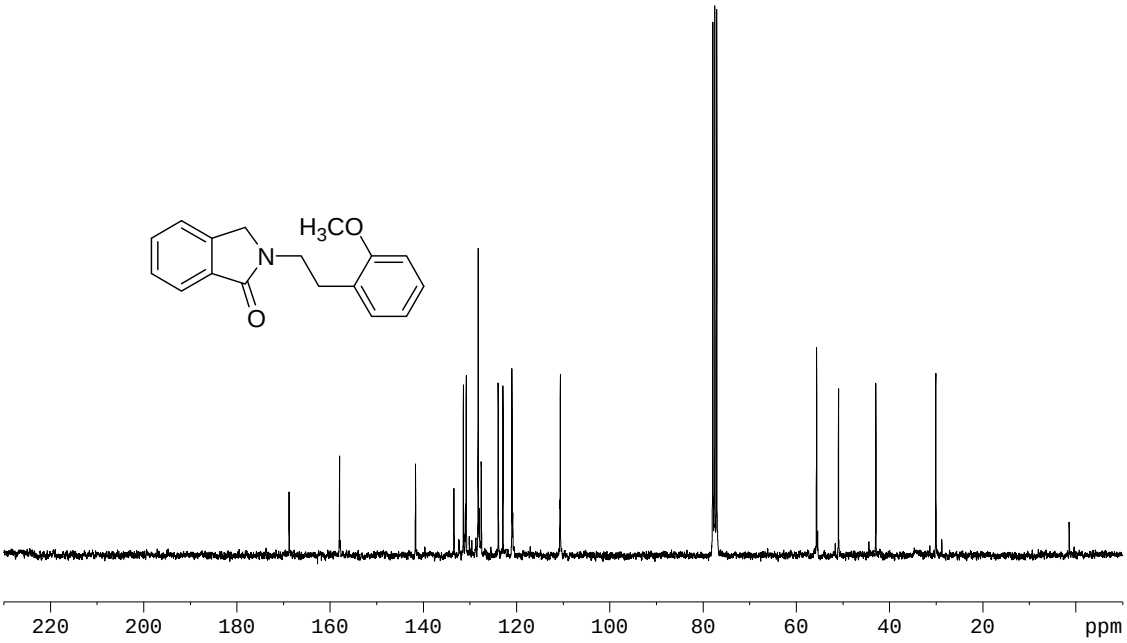


***N*-Phenethylisoindolinone 5b (Table 3, entry 2)**

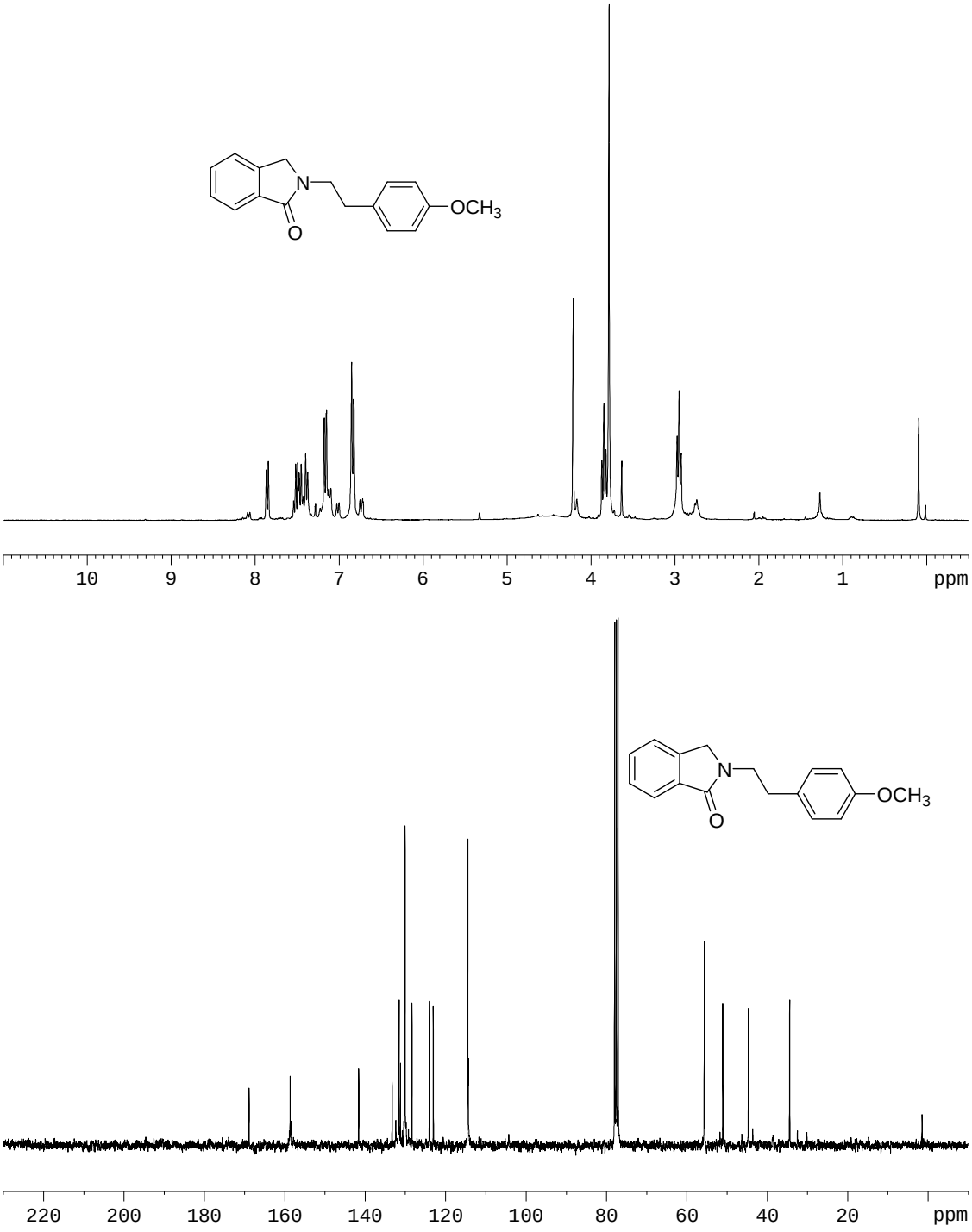


***N*-(2'-Methoxyphenethyl)isoindolinone 5c (Table 3, entry 3)**

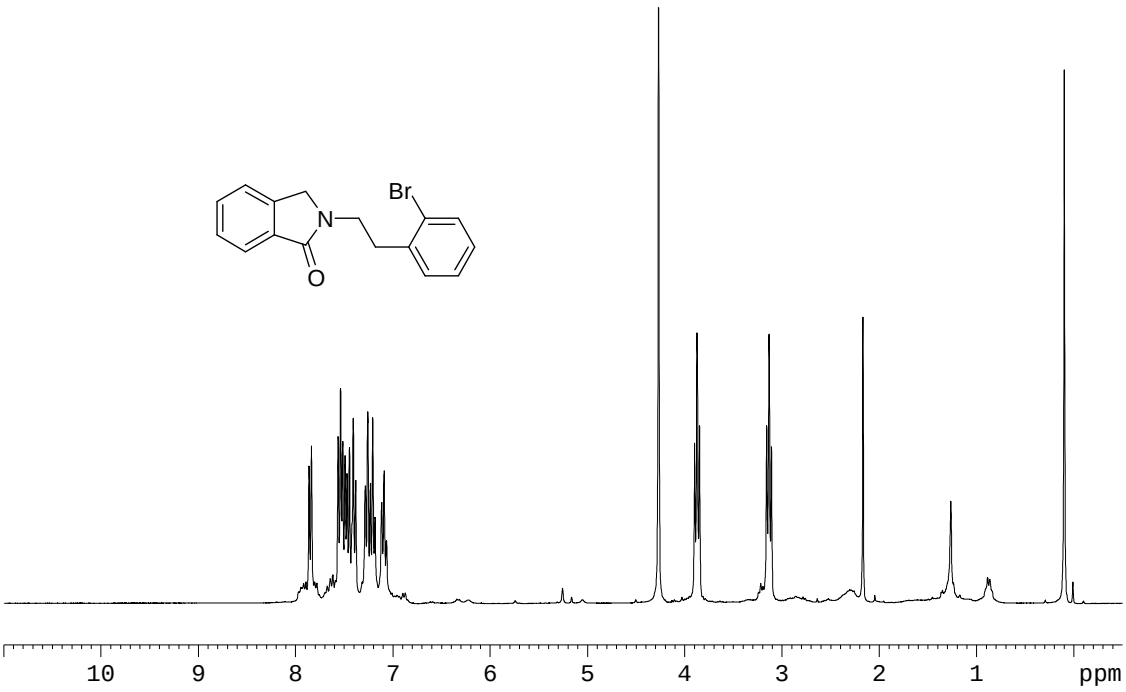


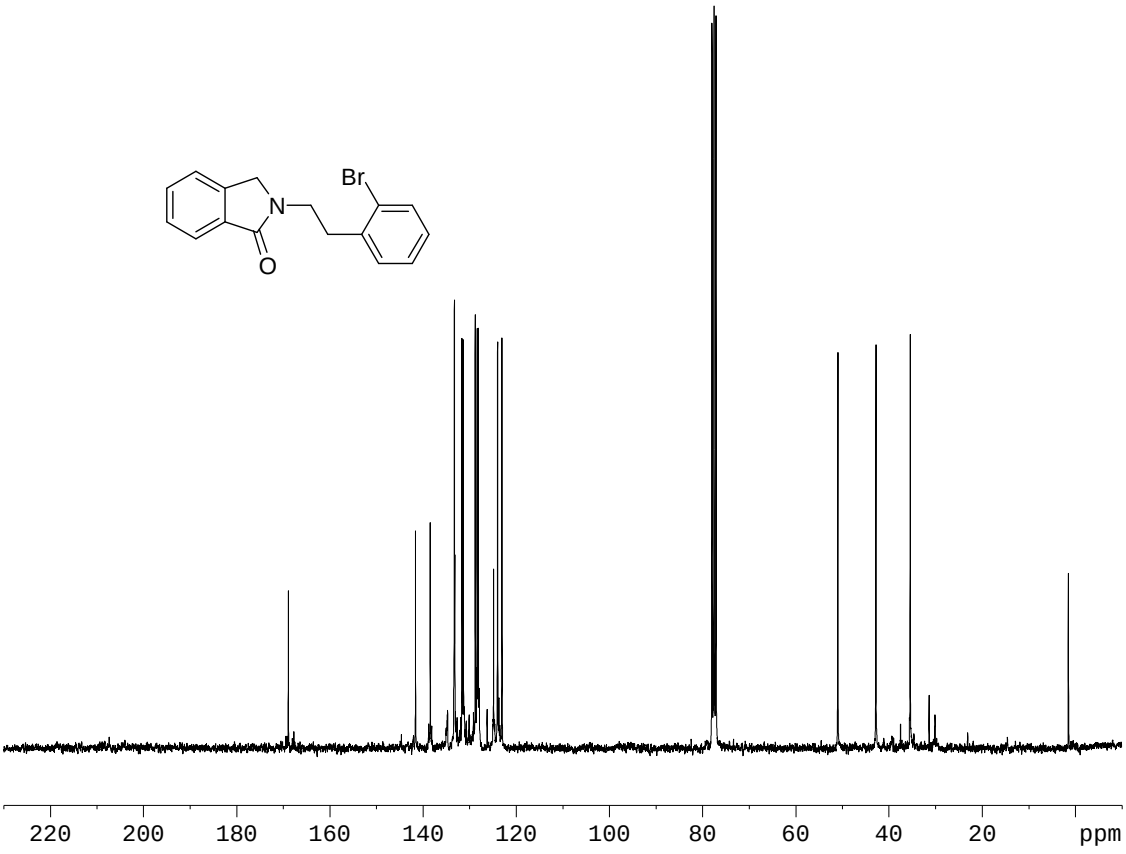


***N*-(4'-Methoxyphenethyl)isoindolinone 5d (Table 3, entry 4)**

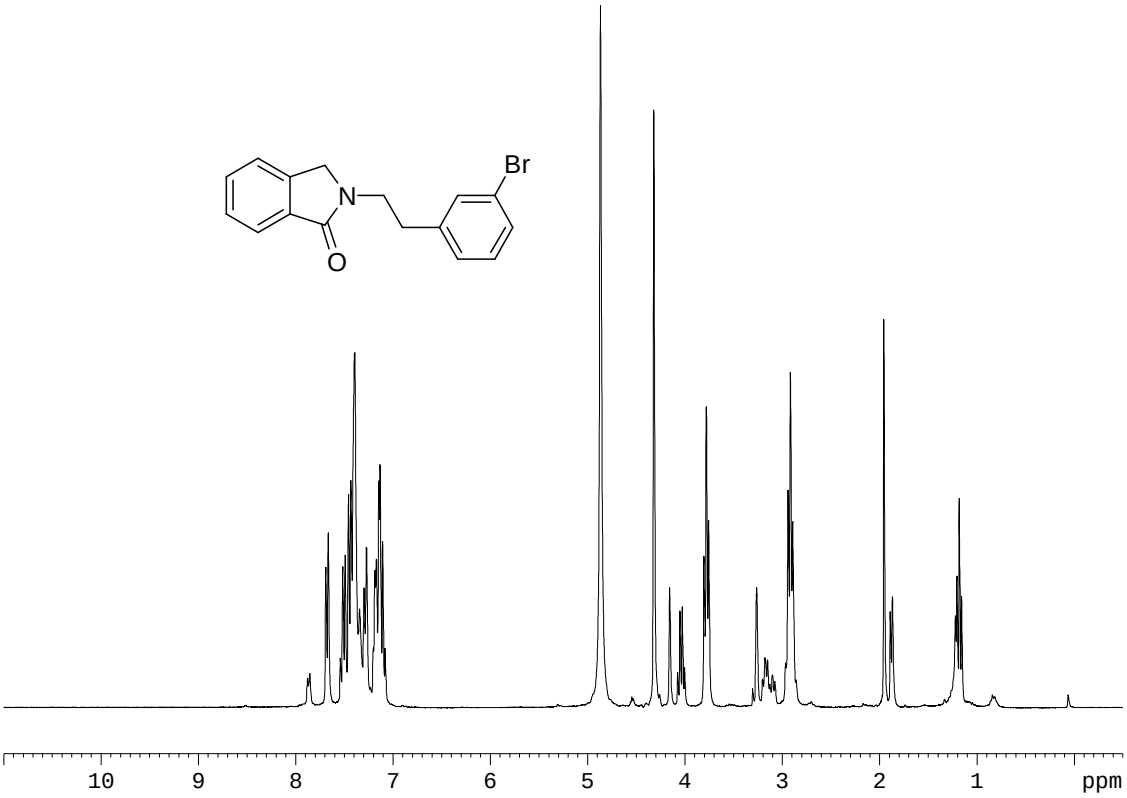


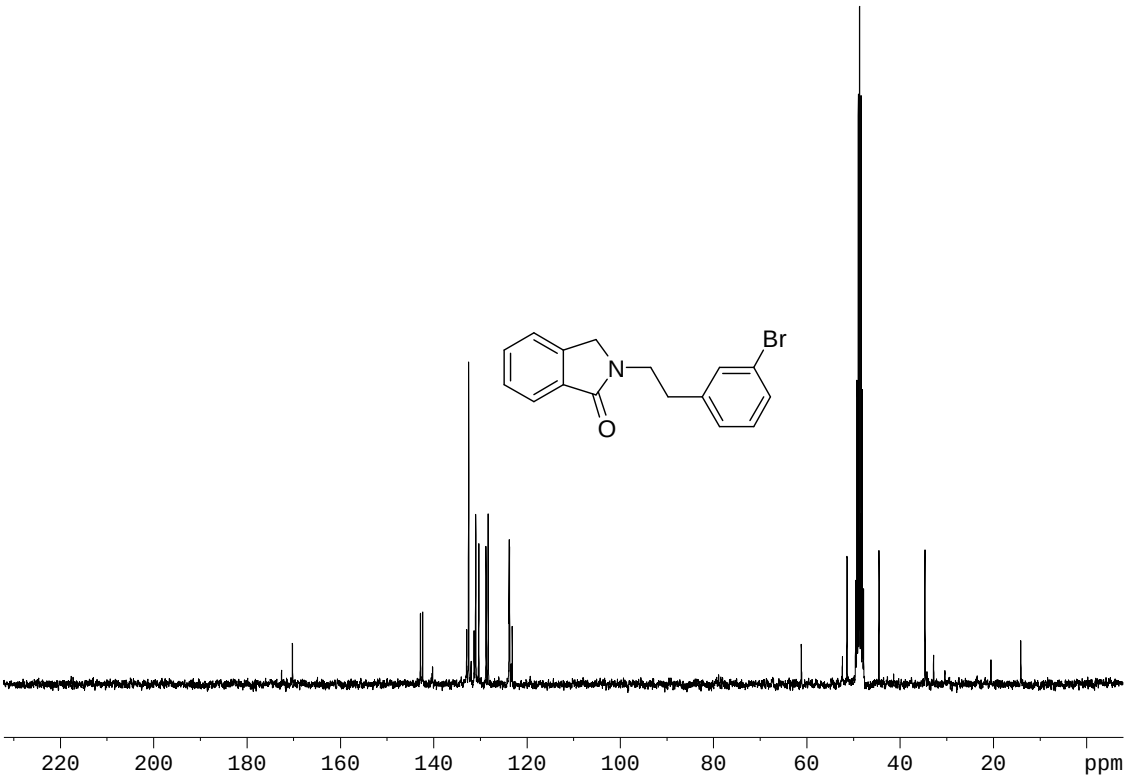
***N*-(2'-Bromophenethyl)isoindolinone 5e (Table 3, entry 5)**



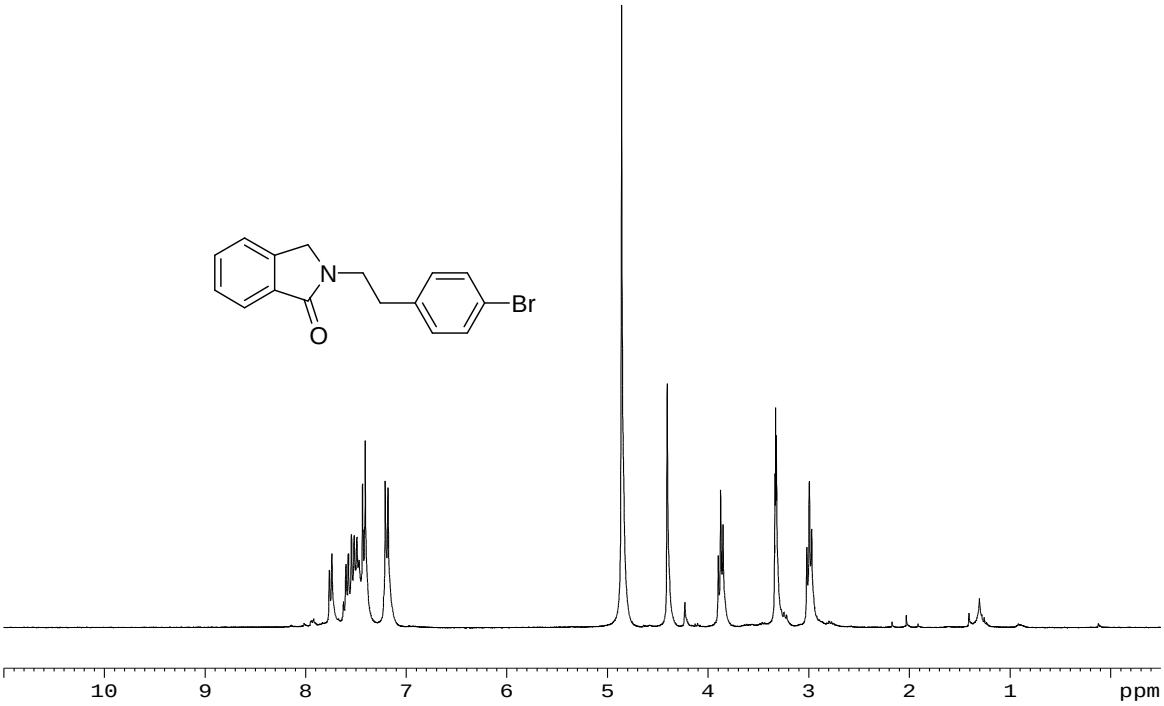


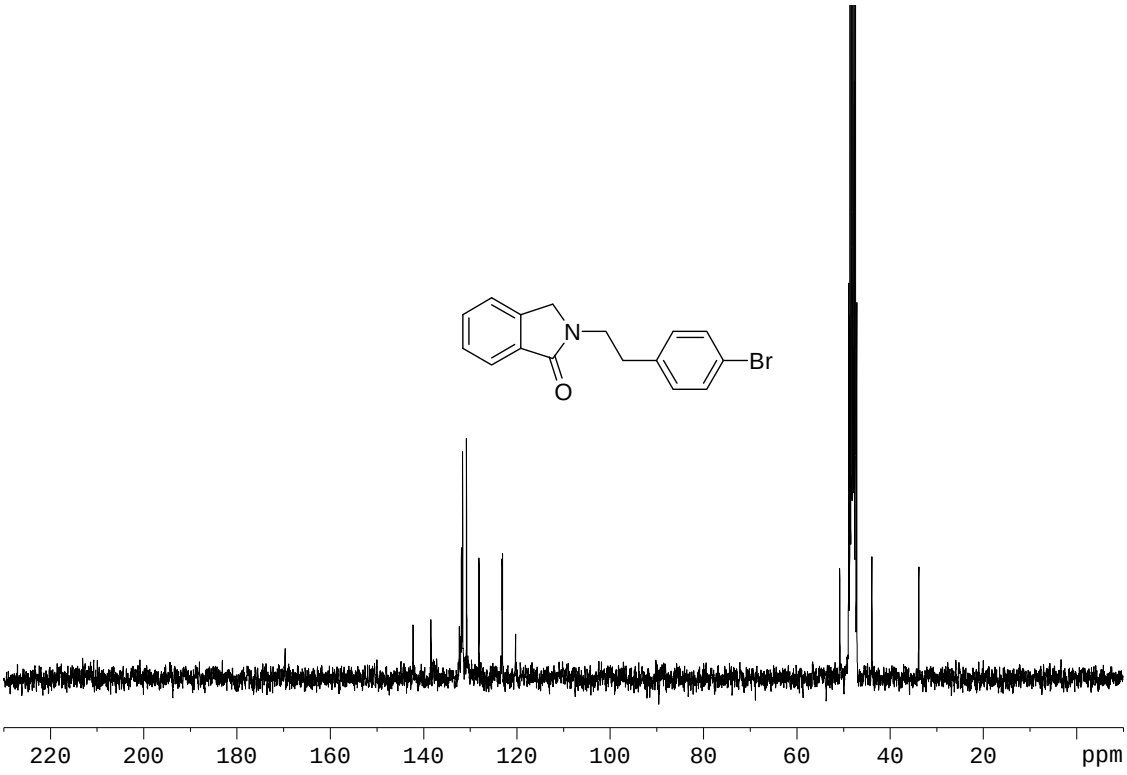
***N*-(3'-Bromophenethyl)isoindolinone 5f (Table 3, entry 6)**



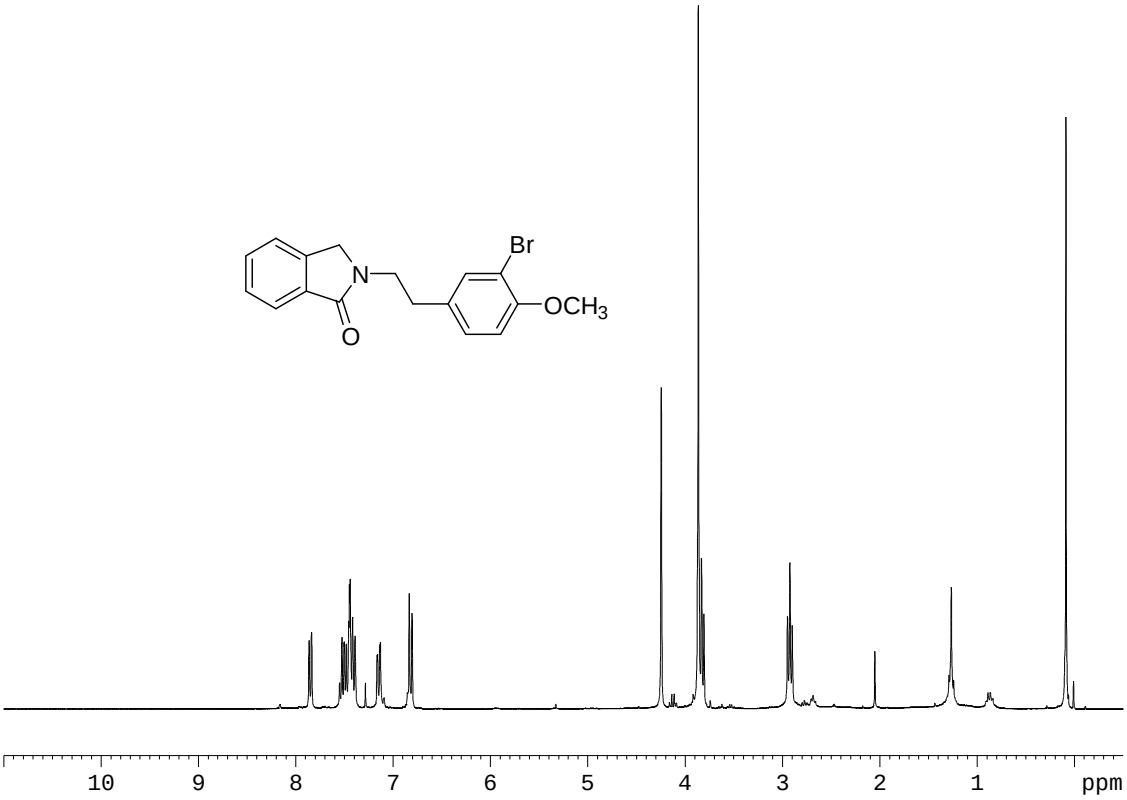


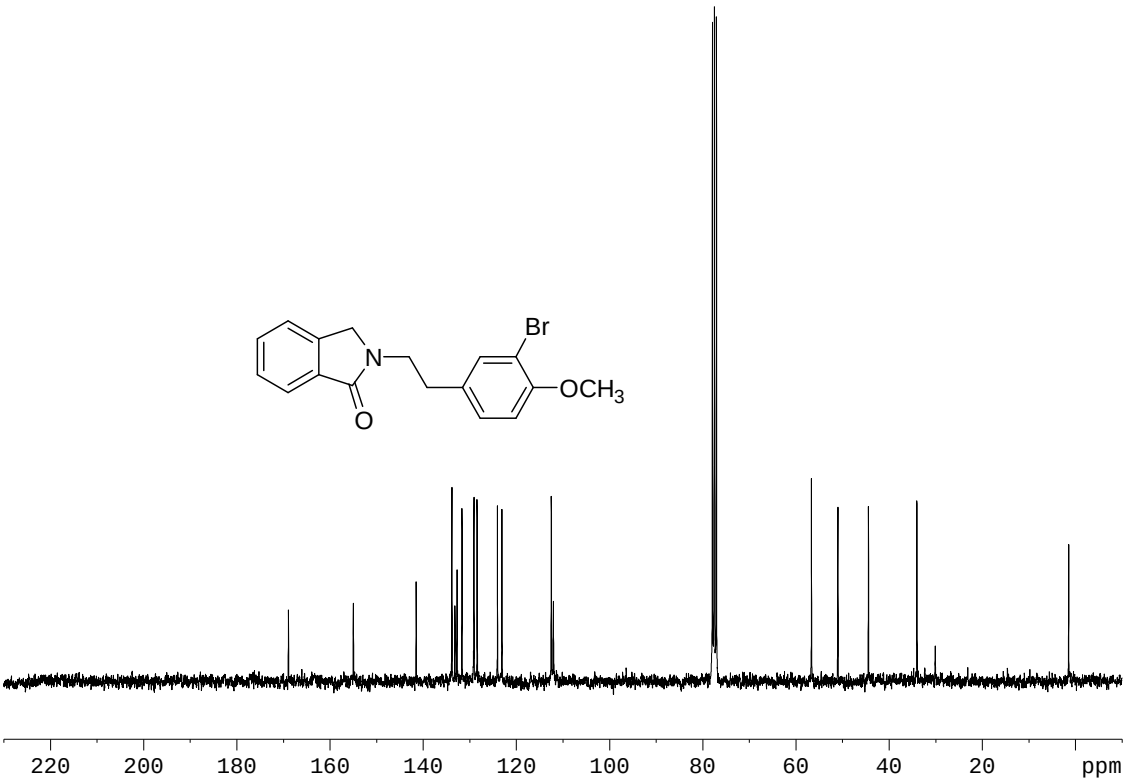
***N*-(4'-Bromophenethyl)isoindolinone 5g (Table 3, entry 7)**



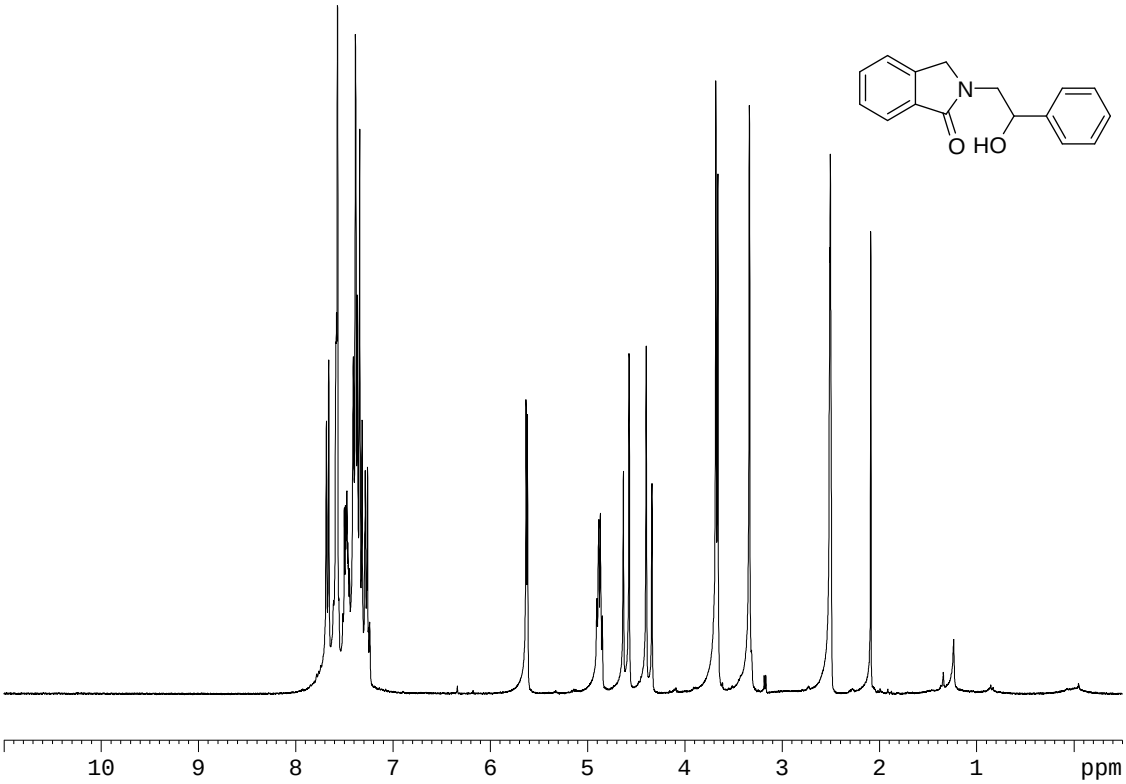


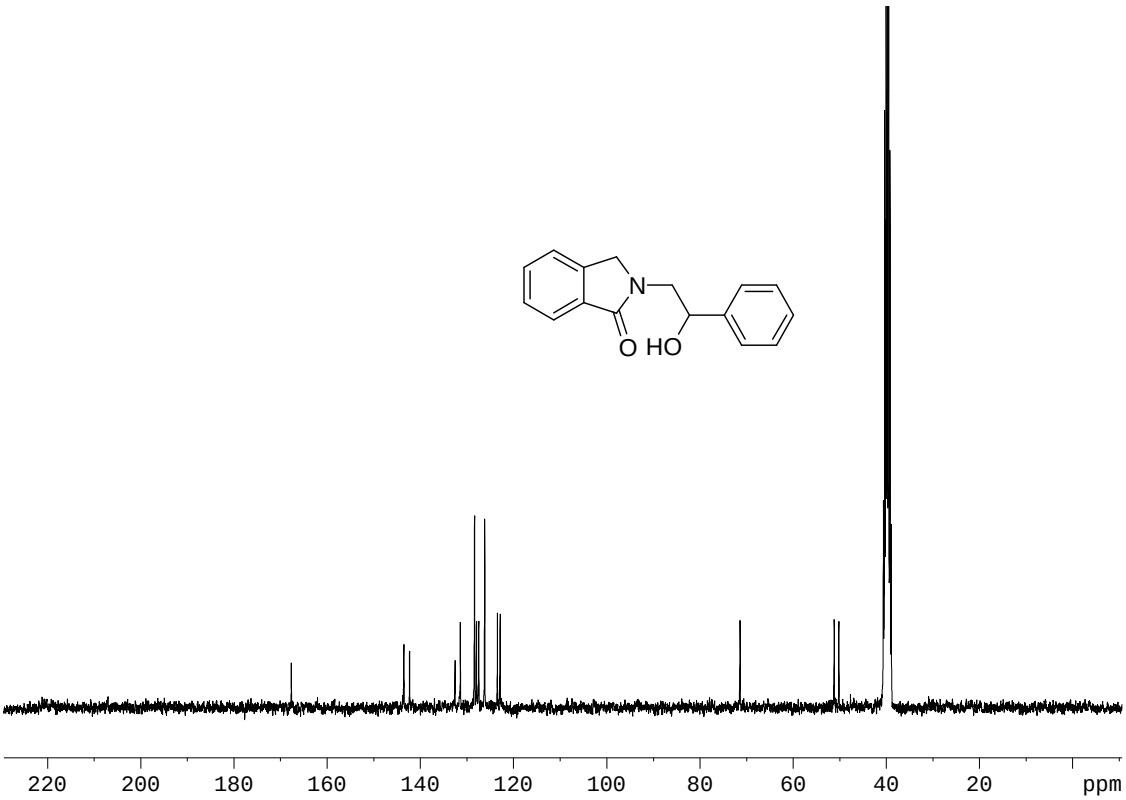
***N*-(3'-Bromo-4'-methoxyphenethyl)isoindolinone 5h (Table 3, entry 8)**



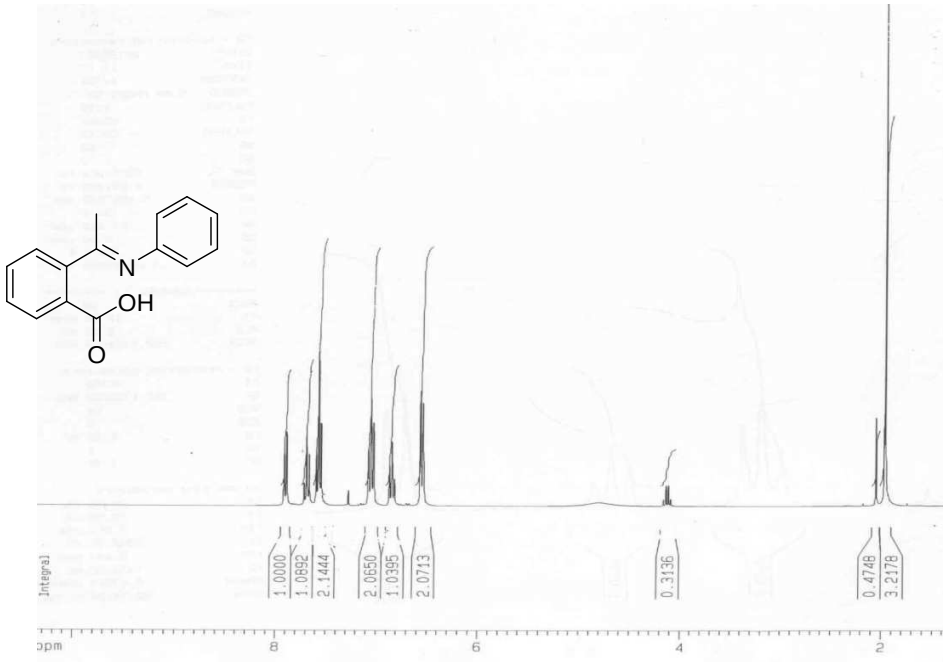


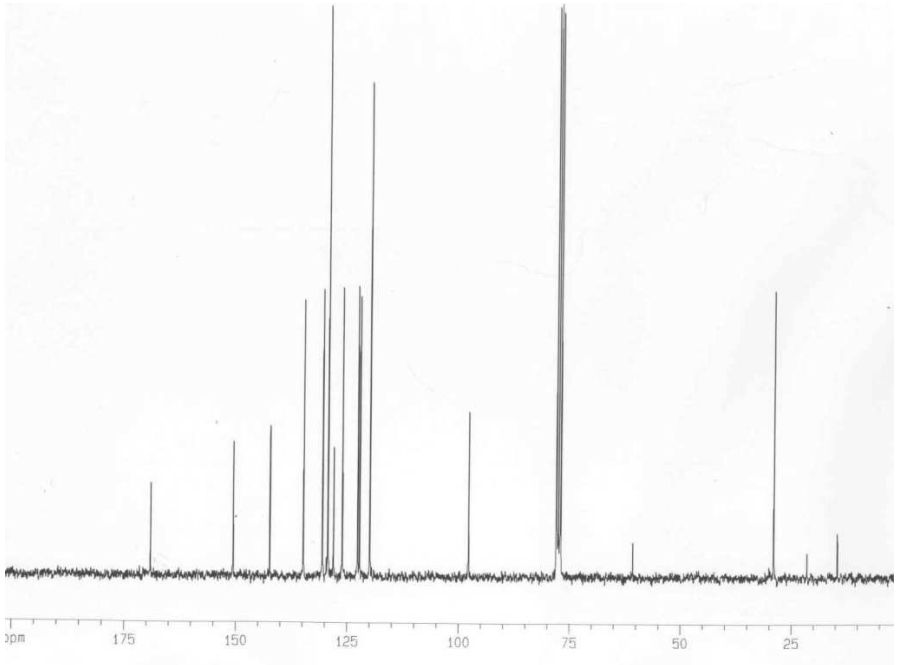
***N*-(1-Hydroxyphenethyl)isoindolinone 5i (Table 3, entry 9)**



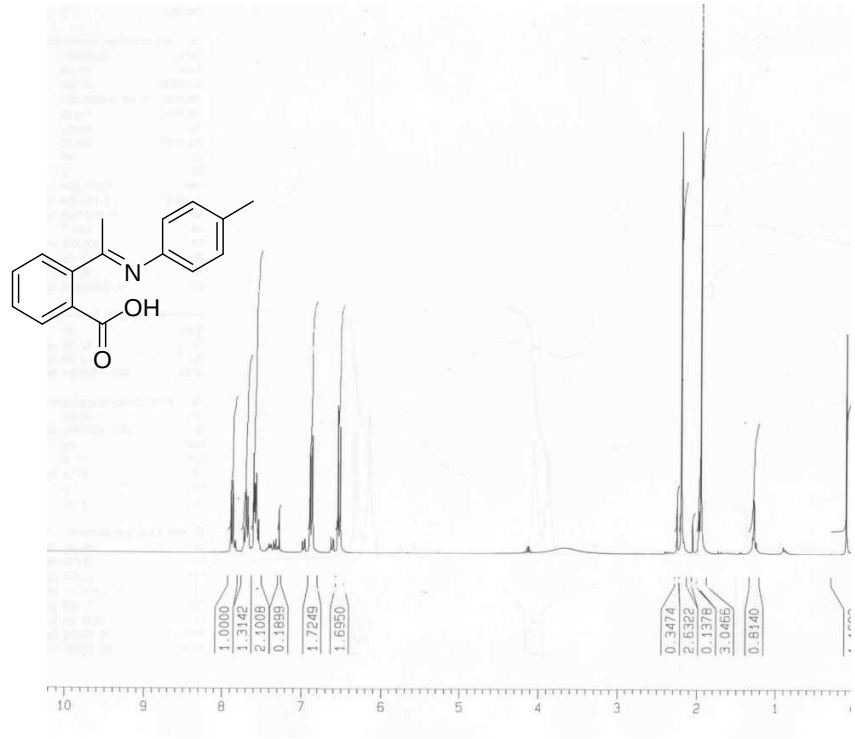


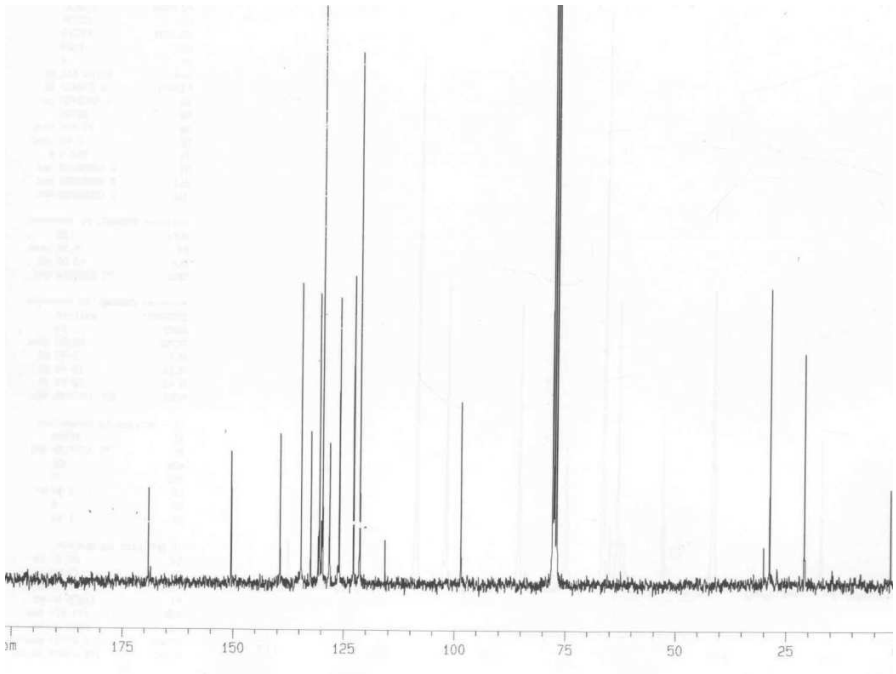
2-(1’-Methyl-N-phenylimino)benzoic acid 7a (Scheme 1)





2-(1’-Methyl-N-tolylimino)benzoic acid 7c (Scheme 1)


N
OH
O
1.0000
1.3142
2.1008
0.1899
1.7249
1.6950
0.3474
2.6322
0.1378
3.0465
0.8140
10
9
8
7
6
5
4
3
2
1


175
150
125
100
75
50
25