

Supporting Information

A new synthesis of flavones and pyranoflavone by *intramolecular* photochemical Wittig reaction in water

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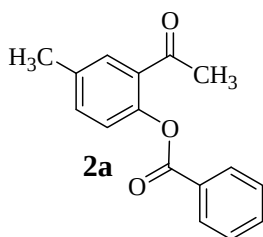
Contents:

General	S-2
Experimental Details and Characterization Data	S-2
References	S-14
^1H NMR, ^{13}C NMR, Mass Spectra & X-ray structure	S-15

General:

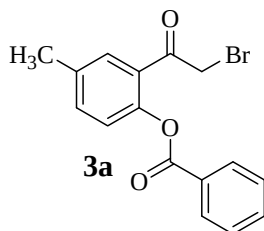
The melting points, recorded in sulfuric acid bath, or boiling points are uncorrected. Photochemical reactions were done using a 150 W tungsten lamp. Solvents were routinely purified; dried and chromatographic purifications were carried out with silica gel (60-120 mesh).

Experimental Details and Characterization Data



1-(2-Benzoyloxy-5-methylphenyl)-ethanone (2a):

Ortho-hydroxyacetophenone (3 g, 20 mmol) was taken in pyridine (20 mL) and to it benzoyl chloride (2.81 g, 20 mmol) was added drop wisely at 0°C with stirring and kept overnight. The reaction mixture was warmed on a water bath for 10 min and decomposed with ice cold hydrochloric acid (1:1), a precipitate was obtained, filtered and washed with brine (3 x 20 mL), and dried under *vacuo*. Crystallized from a mixture of acetone and petroleum ether (60-80°C), a colorless shining granular crystals was appear. Yield: 3.90 g (77%). mp. 60-61°C; ¹H NMR (300MHz, CDCl₃, 22°C): δ 8.21 (dd, *J* = 8.4 Hz, 1.2 Hz, 2H), 7.65 (m, 2H), 7.52 (m, 2H), 7.38 (dd, *J* = 8.2 Hz, 0.6 Hz, 1H), 7.11 (d, *J* = 8.2 Hz, 1H), 2.53 (s, 3H), 2.42 (s, 3H) ppm.

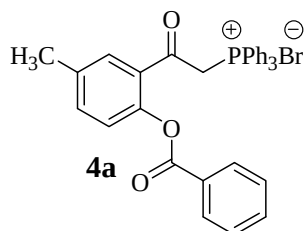


1-(2-Benzoyloxy-5-methylphenyl)-2-bromo ethanone (3a):

1-(2-Benzoyloxy-5-methylphenyl)-ethanone (2.54 g, 10 mmol) was taken in glacial acetic acid (20 mL) and to it bromine (1.60 g, 10 mmol) was added drop wisely at 0°C with stirring and kept overnight at 27°C. The reaction mixture was then added 100 g of crushed ice, a solid product comes out, filtered and washed with brine (3 x 20 mL) and dried under *vacuo*, and the residue on chromatography over silica gel afforded **3a** in ethyl acetate and petroleum ether (60-80°C) (15% v/v) mixture as eluent, crystallized further from a mixture of dichloromethane and petroleum ether (60-80°C) as colorless shining granular crystals. Yield: 2.30 g (69%). mp. 85-86°C; ¹H NMR (300MHz, CDCl₃, 22°C): δ 8.20 (m, 2H), 7.66 (m, 2H),

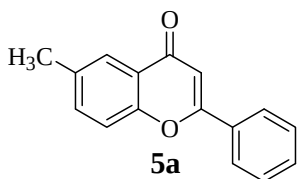
7.53 (t, $J = 7.6$ Hz, 2H), 7.42 (dd, $J = 8.2$ Hz, 1.8 Hz, 1H), 7.17 (d, $J = 8.3$ Hz, 1H), 4.39 (s, 2H), 2.43 (s, 3H) ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 190.9, 165.0, 147.3, 136.2, 134.7, 133.9, 130.8, 130.3, 129.1, 128.8, 128.1, 123.8, 34.2, 20.8 ppm.



2-Benzoyloxy-5-methyl-benzoyl methyl triphenyl phosphonium bromide (4a):

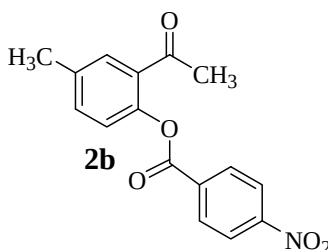
1-(2-Benzoyloxy-5-methylphenyl)-2-bromo ethanone (0.66 g, 2 mmol) was heated in dry condition with triphenyl phosphine (0.52 g, 2 mmol) for 20 min or reflux in toluene for 4h. The mass was crystallized from dichloromethane and ethyl acetate and **4a** was obtained as colorless granular crystals. Yield: 0.73 g (62%), mp 196-97°C; ^1H NMR (300MHz, CDCl_3 , 22°C) δ 9.00 (s, 1H), 7.92 (m, 2H), 7.85 (m, 6H), 7.64 (m, 4H), 7.54 (m, 6H), 7.45 (d, $J = 7.8$ Hz, 2H), 7.39 (d, $J = 10.9$ Hz, 1H), 7.01 (d, $J = 8.2$ Hz, 1H), 6.35 (d, $J = 12.1$ Hz, 2H), 2.54 (s, 3H) ppm.



6-Methyl-2-phenyl chromen-4-one (5a):

A suspension of 2-Benzoyloxy-5-methyl-benzoyl methyl triphenyl-phosphonium bromide (0.10 g, 0.17 mmol) in water (30 mL) and triethylamine (0.10 g, 1 mmol) was irradiated with a 150 W tungsten lamp (Philips India Ltd.) for 40 min. The reaction was monitored by TLC (after 10 minutes interval), and the product was isolated, after solvent extraction, by a column filtration using a mixture of ethyl acetate and petroleum ether (60-80°C) (20% v/v) over silica gel and crystallized from a mixture of acetone and petroleum ether (60-80°C) as colorless shining flakes. Yield: 0.035 g (88%), mp 120-21°C, (lit.¹ mp 122-23°C); IR (KBr) ν_{max} 1645, 1615, 1569, 1484, 1451, 1361 cm^{-1} ; ^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.04 (s, 1H), 7.96 (dd, $J = 6.5$ Hz, 2.1 Hz, 2H), 7.52 (m, 5H), 7.06 (s, 1H), 2.45 (s, 3H) ppm.
 ^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 178.6, 163.6, 154.6, 135.4, 135.2, 131.8, 131.7, 129.1, 126.4, 125.0, 123.4, 117.9, 107.2, 21.0 ppm.
 Mass (EI), m/z (%): 237.0994 ($\text{M}^+ + \text{H}$).

Similar procedures were followed for the preparation of esters (**2b-2h** & **7**), bromo-esters (**3b-3h**), phosphonium salts (**4b-4h** & **9**) and flavones (**5b-5h** & **10**).



1-[2-(4-Nitrobenzoyloxy)-5-methylphenyl]-ethanone (2b):

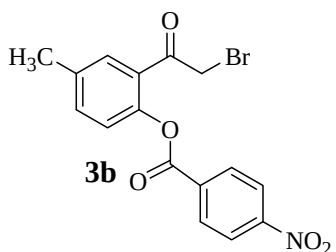
Straw yellow crystalline granules, mp. 110-11°C (acetone-petroleum ether) (60-80°C).

2-Hydroxy-5-methyl acetophenone: 3g, 20 mmol; 4-nitrobenzoic acid: 3.34 g, 20 mmol; SOCl₂: 3.57 g, 30 mmol;

Pyridine: 20 mL. Yield: 4.59g (77%).

¹H NMR (300MHz, CDCl₃, 22°C): δ 8.36 (br s, 4H), 7.69 (s, 1H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.13 (d, *J* = 8.1 Hz, 1H), 2.54 (s, 3H), 2.45 (s, 3H) ppm.

¹³C NMR-APT- (75 MHz, CDCl₃, 22°C): δ 197.3, 163.7, 150.9, 146.7, 136.5, 135.0, 134.3, 131.4, 131.1, 129.9, 123.7, 123.5, 29.1, 20.9 ppm.



1-[2-(4-Nitrobenzoyloxy)-5-methylphenyl]-2-bromo ethanone (3b):

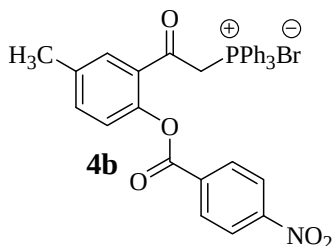
Straw yellow crystalline flakes, mp. 122-23°C (acetone-petroleum ether) (60-80°C).

1-[2-(4-Nitrobenzoyloxy)-5-methylphenyl]-ethanone: 1.50 g, 5 mmol; bromine: 0.80 g, 5 mmol; glacial acetic acid: 15 mL.

yield: 1.23 g (65%).

¹H NMR (300MHz, CDCl₃, 22°C): δ 8.35 (br s, 4H), 7.69 (s, 1H), 7.47 (d, *J* = 8.1 Hz, 1H), 7.19 (d, *J* = 8.1 Hz, 1H), 4.38 (s, 2H), 2.45 (s, 3H) ppm.

¹³C NMR-APT- (75 MHz, CDCl₃, 22°C): δ 190.4, 163.4, 150.9, 147.0, 136.6, 135.0, 134.6, 131.3, 131.28, 130.9, 123.8, 123.7, 33.0, 20.8 ppm.



2-(4-Nitrobenzoyloxy)-5-methyl-benzoyl methyl triphenyl-phosphonium bromide (4b):

Yellow crystalline granules, mp. > 250°C (dichloromethane-ethyl acetate).

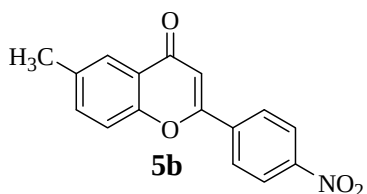
1-[2-(4-Nitrobenzoyloxy)-5-methylphenyl]-2-bromo ethanone: 0.38 g 1 mmol; triphenylphosphine: 0.26 g, 1 mmol. Yield:

0.39 g (61%).

¹H NMR (300MHz, CDCl₃, 22°C): δ 9.07 (s, 1H), 8.25 (m, 2H), 8.06 (d, *J* = 8.4 Hz, 2H), 7.85 (m, 5H), 7.69 (t, *J* = 6.6 Hz, 4H), 7.56 (br s, 6H), 7.43 (d, *J* = 8.1 Hz, 1H), 7.04 (d, *J* = 8.1 Hz, 1H), 6.38 (d, *J* = 9.6 Hz, 2H), 2.54 (s, 3H) ppm.

¹³C NMR-APT- (75 MHz, CDCl₃, 22°C): 191.73, 191.7, 163.4, 150.8, 146.0, 138.2, 135.9, 135.1, 135.0, 134.7, 134.59, 134.6, 134.2, 134.1, 132.2, 132.1, 131.4, 131.3, 130.1, 130.0,

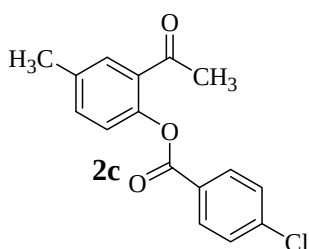
129.8, 129.7, 128.6, 128.5, 128.2, 128.1, 123.8, 123.7, 123.5, 123.4, 122.7, 119.3, 118.1, 41.9, 41.1, 20.8 ppm.



6-Methyl-2-(4-nitro phenyl)-chromen-4-one (5b): Yellow crystalline flakes, mp. 274-75°C (acetone-petroleum ether) (60-80°C) (lit.² mp 277°C).

2-(4-Nitrobenzoyloxy)-5-methyl-benzoyl methyl triphenyl-phosphonium bromide: 0.18 g, 0.27 mmol; Triethylamine: 0.1 g, 1 mmol; H₂O: 20 mL; Eluent: ethyl acetate-petroleum ether (60-80°C) (30% v/v); Yield: .006g (77%). IR (KBr) $\nu_{\max}$ 1640, 1617, 1523, 1484, 1343 cm⁻¹.

¹H NMR (300MHz, CDCl₃, 22°C): δ 8.38 (d, *J* = 8.7 Hz, 2H), 8.11 (d, *J* = 9.0 Hz, 2H), 8.03 (s, 1H), 7.54 (m, 2H), 6.89 (s, 1H), 2.49 (s, 3H), ppm.

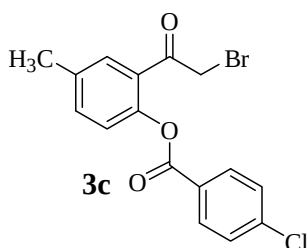


1-[2-(4-Chlorobenzoyloxy)-5-methylphenyl]-ethanone (2c): White crystalline granules, mp. 88-89°C (acetone-petroleum ether) (60-80°C).

2-Hydroxy-5-methyl acetophenone: 1.50 g, 10 mmol; 4-chlorobenzoic acid: 1.57 g, 10 mmol; SOCl₂: 2.38g, 20 mmol; Pyridine: 20 mL. Eluent: ethyl acetate-petroleum ether (60-80°C) (10% v/v); Yield: 2.20g (76%).

¹H NMR (300MHz, CDCl₃, 22°C): δ 8.14 (m, 2H), 7.65 (d, *J* = 1.5 Hz, 1H), 7.50 (m, 2H), 6.39 (dd, *J* = 8.1 Hz, 1.8 Hz, 1H), 7.11 (d, *J* = 8.4 Hz, 1H), 2.52 (s, 3H), 2.43 (s, 3H) ppm.

¹³C NMR (75 MHz, CDCl₃, 22°C): δ 197.5, 164.5, 147.0, 140.3, 136.1, 134.0, 131.6, 130.7, 129.0, 127.9, 123.6, 29.5, 20.8 ppm.

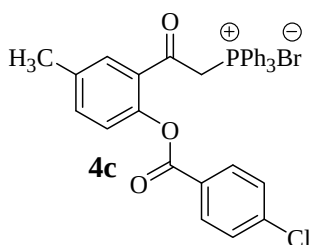


1-[2-(4-Chlorobenzoyloxy)-5-methylphenyl]-2-bromo ethanone (3c): White crystalline flakes, mp. 108-09°C (acetone-petroleum ether) (60-80°C).

1-[2-(4-Chlorobenzoyloxy)-5-methylphenyl]-ethanone: 0.58 g, 2 mmol; bromine: 0.32 g, 2 mmol; acetic acid: 15 mL. yield: 0.53 g (72%).

¹H NMR (300MHz, CDCl₃, 22°C): δ 8.14 (m, 2H), 7.67 (d, *J* = 1.8 Hz, 1H), 7.51 (m, 2H), 7.44 (dd, *J* = 8.3 Hz, 2.0 Hz, 1H), 7.16 (d, *J* = 8.1 Hz, 1H), 4.37 (s, 2H), 2.44 (s, 3H) pm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 190.8, 164.3, 147.3, 140.5, 136.4, 134.9, 131.7, 130.9, 129.1, 127.8, 127.6, 123.8, 33.8, 20.9 ppm.

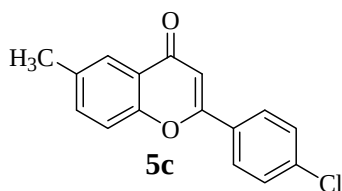


2-(4-Chlorobenzoyloxy)-5-methyl-benzoyl methyl triphenyl phosphonium bromide (4c): White crystalline granules, mp. 159-60°C (dichloromethane-ethyl acetate).

1-[2-(4-Chlorobenzoyloxy)-5-methylphenyl]-2-bromo ethanone: 0.37 g, 1 mmol; triphenylphosphine: 0.26 g 1 mmol. Yield:

0.40 g (63%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.99 (s, 1H), 7.82 (m, 8H), 7.64 (d, J = 6.9 Hz, 3H), 7.53 (br s, 6H), 7.40 (d, J = 8.4 Hz, 3H), 6.99 (d, J = 8.1 Hz, 1H), 6.34 (d, J = 11.4 Hz, 2H), 2.52 (s, 3H) ppm.

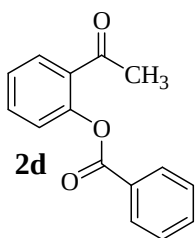


6-Methyl-2-(4-chlorophenyl)-chromen-4-one (5c): White crystalline needles, mp. 200-201°C (acetone-petroleum ether) (60-80°C) (lit.³ mp 198-99°C).

2-(4-Chlorobenzoyloxy)-5-methyl-benzoyl methyl triphenyl-phosphonium bromide: 0.16 g, 0.25 mmol; Triethylamine: 0.1 g, 1 mmol; H_2O : 20 mL; Eluent: ethyl acetate-petroleum ether (60-80°C) (15% v/v); Yield: .005g (73%); IR (KBr) ν_{max} 1643, 1621, 1493, 1484, 1364 cm^{-1} .

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.00 (s, 1H), 7.85 (dd, J = 7.7 Hz, 1.5 Hz, 2H), 7.48 (m, 4H), 6.77 (s, 1H), 2.46 (s, 3H) ppm.

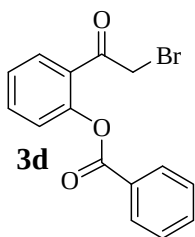
^{13}C NMR-APT- (75 MHz, CDCl_3 , 22°C): δ 178.4, 162.1, 154.5, 137.8, 135.4, 135.1, 130.4, 129.3, 127.5, 125.1, 123.6, 117.8, 107.6, 20.9 ppm.



1-[2-(Benzoyloxy)-phenyl]-ethanone (2d): White crystalline granules, mp. 87-88°C (dichloromethane-petroleum ether) (60-80°C).

Ortho hydroxy acetophenone: 4.08 g, 30 mmol; Benzoyl chloride: 4.20 g, 30 mmol; dry pyridine: 30mL; Yield: 6.10g (85%).

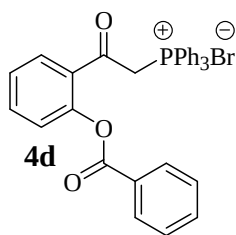
^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.22 (d, J = 8.1 Hz, 2H), 7.87 (d, J = 7.8 Hz, 1H), 7.65 (m, 1H), 7.55 (q, J = 8.5 Hz, 3H), 7.37 (t, J = 7.5 Hz, 1H), 7.24 (d, J = 8.1 Hz, 1H), 2.56 (s, 3H) ppm.



1-[2-(Benzoyloxy)-phenyl]-2-bromo ethanone (3d): White crystalline granules, mp. 74-75°C (dichloromethane-petroleum ether) (60-80°C).

1-[2-(Benzoyloxy)-phenyl]-ethanone: 2.40 g, 10 mmol; bromine: 1.60 g, 10mmol; acetic acid: 25 mL. yield: 2.20 g (69%).

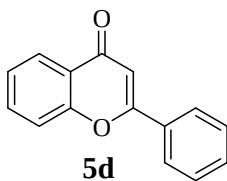
¹H NMR (300MHz, CDCl₃, 22°C): δ 8.23 (d, *J* = 7.8 Hz, 2H), 7.90 (d, *J* = 7.8 Hz, 1H), 7.67 (m, 2H), 7.55 (t, *J* = 7.7 Hz, 2H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.32 (d, *J* = 8.1 Hz, 1H), 4.42 (s, 2H) ppm.



2-(Benzoyloxy)-benzoyl methyl triphenyl phosphonium bromide (4d): White crystalline granules, mp. 204-06°C (dichloromethane-ethyl acetate).

1-[2-(Benzoyloxy)-phenyl]-2-bromo ethanone: 3.20 g, 10 mmol; triphenyl phosphine: 2.62 g, 10 mmol. Yield: 4.30 g (74%).

¹H NMR (300MHz, CDCl₃, 22°C): δ 8.98 (d, *J* = 7.8 Hz, 1H), 7.85 (d, *J* = 8.1 Hz, 2H), 7.80 (d, *J* = 8.1 Hz, 3H), 7.75 (d, *J* = 8.1 Hz, 3H), 7.59 (m, 4H), 7.48 (m, 7H), 7.40 (t, *J* = 7.7, 3H) 7.08 (d, *J* = 7.8 Hz, 1H), 6.28 (d, *J* = 12.0 Hz, 2H) ppm.

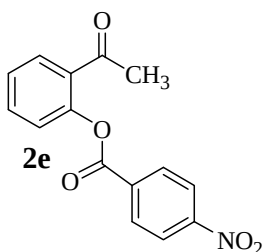


2-phenyl chromen-4-one (5d): White crystalline flakes, mp.97-98°C (acetone-petroleum ether) (60-80°C) (lit.⁴ mp 96°C).

2-(Benzoyloxy)-benzoyl methyl triphenyl phosphonium bromide: 0.58 g, 1 mmol; Triethylamine: 0.4 g, 4 mmol; H₂O: 25 mL; Eluent: ethyl acetate-petroleum ether (60-80°C) (20% v/v); Yield: 0.19 g (86%). IR (KBr) ν_{max} 1646, 1605, 1569, 1466, 1450, 1376 cm⁻¹.

¹H NMR (300MHz, CDCl₃, 22°C): δ 8.23 (d, *J* = 7.8 Hz, 1H), 7.92 (dd, *J* = 6.2 Hz, 2.7 Hz, 2H), 7.70 (t, *J* = 7.2 Hz, 1H), 7.54 (m, 4H), 7.42 (t, *J* = 7.5 Hz, 1H), 6.85 (s, 1H) ppm.

¹³C NMR (75 MHz, CDCl₃, 22°C): δ 178.5, 163.6, 156.3, 133.9, 131.7, 131.7, 129.1, 126.4, 125.7, 125.3, 123.9, 118.1, 107.5 ppm.

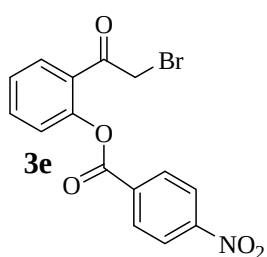


1-[2-(4-Nitrobenzoyloxy)-phenyl]-ethanone (2e): Straw yellow crystalline granules, mp. 95-96°C (acetone-petroleum ether) (60-80°C).

Ortho hydroxy acetophenone: 2.72 g, 20 mmol; 4-nitrobenzoic acid: 3.34 g, 20 mmol; SOCl₂: 3.57g, 30 mmol; Pyridine: 20 mL. Yield: 3.85g (68%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.35 (m, 4H), 7.90 (dd, $J = 7.8$ Hz, 1.5 Hz, 1H), 7.62 (dt, $J = 7.8$ Hz, 1.5 Hz, 1H), 7.42 (dt, $J = 7.7$ Hz, 1.1 Hz, 1H), 7.25 (dd, $J = 8.1$ Hz, 0.9 Hz, 1H), 2.55 (s, 3H) ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 197.2, 163.5, 150.8, 148.8, 134.9, 133.7, 131.4, 130.7, 130.2, 126.6, 123.8, 123.7, 29.0 ppm.

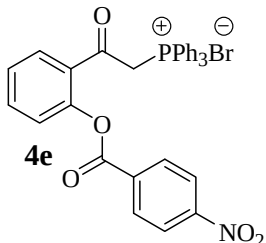


1-[2-(4-Nitrobenzoyloxy)-phenyl]-2-bromo ethanone (3e): Straw yellow crystalline flakes, mp. 94-95°C (acetone-petroleum ether) (60-80°C).

1-[2-(4-Nitrobenzoyloxy)-phenyl]-ethanone: 1.43 g 5 mmol; bromine: 0.80 g 5 mmol; glacial acetic acid: 15 mL. yield: 1.00 g (55%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.37 (br s, 4H), 7.92 (d, $J = 7.5$ Hz, 1H), 7.68 (t, $J = 7.4$ Hz, 1H), 7.45 (t, $J = 7.4$ Hz, 1H), 7.32 (d, $J = 8.1$ Hz, 1H), 4.39 (s, 2H) ppm.

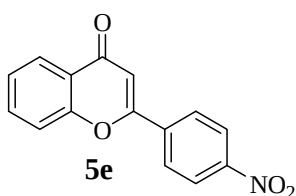
^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 190.3, 163.3, 151.0, 149.4, 134.5, 131.4, 130.6, 127.4, 126.7, 124.2, 123.8, 32.9 ppm.



2-(4-Nitrobenzoyloxy)-phenyl-benzoyl methyl triphenyl phosphonium bromide (4e): yellow crystalline solid, mp. 154°C (dec) (dichloromethane-ethyl acetate).

1-[2-(4-Nitrobenzoyloxy)-phenyl]-2-bromo ethanone: 0.55 g 1.5 mmol; triphenylphosphine: 0.39 g 1.5 mmol; toluene: 20ml; Yield: 0.63 g (67%).

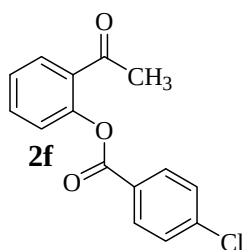
^1H NMR (300MHz, CDCl_3 , 22°C): δ 9.12 (s, 1H), 8.27 (d, $J = 8.4$ Hz, 2H), 8.06 (d, $J = 8.5$ Hz, 3H), 7.85 (m, 4H), 7.63 (m, 12H), 7.16 (d, $J = 7.6$ Hz, 1H), 6.46 (d, $J = 11.1$ Hz, 2H) ppm.



2-(4-Nitro phenyl)-chromen-4-one (5e): White crystalline solid, mp 239-40°C (acetone-petroleum ether) (60-80°C) (lit.⁴ mp 242-44°C).

2-(4-Nitrobenzoyloxy)-phenyl-benzoyl methyl triphenyl phosphonium bromide: 0.31 g, 0.5 mmol; Triethylamine: 0.2 g, 2 mmol; H_2O : 25 mL; Eluent: ethyl acetate-petroleum ether (60-80°C) (30% v/v); Yield: 0.09 g (69%). IR (KBr) ν_{max} 1660, 1610, 1520, 1468, 1417, 1346 cm^{-1} .

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.40 (d, J = 8.7 Hz, 2H), 8.25 (d, J = 7.8 Hz, 1H), 8.12 (d, J = 8.7 Hz, 2H), 7.76 (t, J = 7.7 Hz, 1H), 7.61 (d, J = 8.1 Hz, 1H), 7.48 (t, J = 7.5 Hz, 1H), 6.92 (s, 1H) ppm.

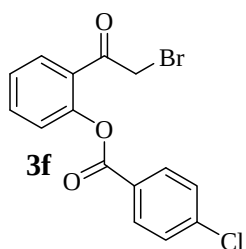


1-[2-(4-Chlorobenzoyloxy)-phenyl]-ethanone (2f): White crystalline granules, mp. 90-91°C (acetone-petroleum ether) (60-80°C).

Ortho hydroxy acetophenone: 4.08 g, 30 mmol; 4-chlorobenzoic acid: 4.7 g, 30 mmol; SOCl_2 : 5.4 g, 45 mmol; Pyridine: 30 mL. Yield: 6.4 g (78%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.14 (d, J = 8.1 Hz, 2H), 7.86 (d, J = 7.8 Hz, 1H), 7.58 (t, J = 7.7 Hz, 1H), 7.49 (d, J = 8.1 Hz, 2H), 7.37 (t, J = 7.5 Hz, 1H), 7.24 (d, J = 7.8 Hz, 1H), 2.53 (s, 3H) ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 197.3, 164.3, 149.1, 140.2, 133.4, 131.6, 130.9, 130.3, 129.0, 127.8, 126.2, 123.8, 29.4 ppm.

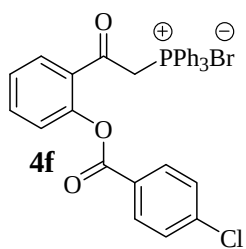


1-[2-(4-Chlorobenzoyloxy)-phenyl]-2-bromo ethanone (3f): White crystalline flakes, mp. 68-70°C (acetone-petroleum ether) (60-80°C).

1-[2-(4-Chlorobenzoyloxy)-phenyl]-ethanone: 2.75 g, 10 mmol; bromine: 1.60 g, 10 mmol; glacial acetic acid: 20 mL. yield: 1.90g (54%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.15 (d, J = 8.4 Hz, 2H), 7.89 (d, J = 7.8 Hz, 1H), 7.65 (t, J = 7.2 Hz, 1H), 7.52 (d, J = 8.4 Hz, 2H), 7.42 (t, J = 7.5 Hz, 1H), 7.29 (d, J = 8.1 Hz, 1H), 4.38 (s, 2H) ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 190.5, 164.0, 149.4, 140.5, 134.2, 131.6, 130.5, 129.1, 128.1, 127.4, 126.3, 124.1, 33.5 ppm.

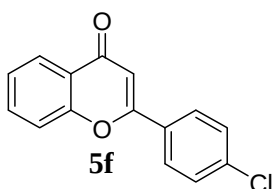


2-(4-Chlorobenzoyloxy)-phenyl)-benzoyl methyl triphenyl phosphonium bromide (4f): White crystalline granules, mp. 163-65°C (dichloromethane-ethyl acetate).

1-[2-(4-Chlorobenzoyloxy)-phenyl]-2-bromo ethanone: 0.35 g, 1 mmol; triphenylphosphine: 0.26 g, 1 mmol. Yield: 0.39 g (64%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.97 (d, J = 7.5, 1H), 7.78 (m, 8H), 7.61 (m, 3H), 7.47 (m, 8H), 7.37 (d, J = 8.4, 2H), 7.08 (d, J = 7.8, 1H), 6.29 (d, J = 12.3, 2H) ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 191.4, 191.3, 164.0, 148.2, 140.0, 134.8, 134.5, 134.4, 133.9, 133.8, 132.7, 131.5, 129.9, 129.8, 129.1, 129.0, 128.6, 127.3, 127.0, 123.1, 129.0, 117.8, 41.0, 40.2.

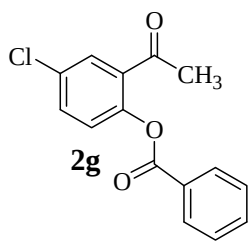


2-(4-Chlorophenyl)-chromen-4-one (5f): White crystalline needles, mp. 186-87°C (acetone- petroleum ether) (60-80°C) (lit.⁴ mp 186-88°C).

2-(4-Chlorobenzoyloxy)-phenyl)-benzoyl methyl triphenyl phosphonium bromide: 0.11 g, 0.18 mmol; Triethylamine: 0.1 g, 1 mmol; H_2O : 20 mL; Eluent: ethyl acetate-petroleum ether (60-80°C) (15% v/v); Yield: 0.04g (87%). IR (KBr) ν_{max} 1662, 1643, 1606, 1467, 1408, 1375 cm^{-1} .

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.23 (d, J = 7.8 Hz, 1H), 7.87 (d, J = 8.4 Hz, 2H), 7.72 (t, J = 7.4 Hz, 1H), 7.57 (d, J = 8.4 Hz, 1H), 7.51 (d, J = 8.4 Hz, 2H), 7.44 (t, J = 7.5 Hz, 1H), 6.83 (s, 1H) ppm.

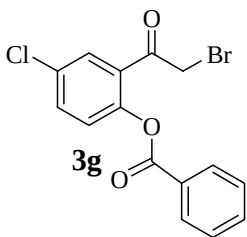
^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 178.3, 162.4, 156.2, 138.0, 134.0, 130.2, 129.4, 127.6, 125.8, 125.5, 123.8, 118.0, 107.6 ppm.



1-(2-Benzoyloxy-5-chlorophenyl)-ethanone (2g): Colorless crystalline granules, mp. 70-71°C (acetone-petroleum ether) (60-80°C).

2-Hydroxy-5-chloro acetophenone: 3.41 g, 20 mmol; benzoyl chloride: 2.81 g, 20 mmol; Pyridine: 30 mL. Yield: 4.91 g (89%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.19 (m, 2H), 7.81 (d, J = 2.6 Hz, 1H), 7.65 (m, 1H), 7.53 (m, 3H), 7.19 (d, J = 8.6 Hz, 1H), 2.52 (s, 3H) ppm.

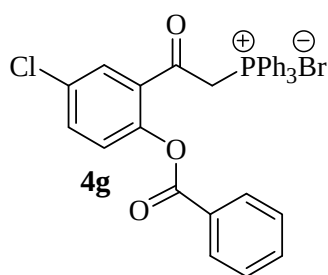


1-(2-Benzoyloxy-5-chlorophenyl)-2-bromo ethanone (3g): Colorless crystalline flakes, mp. 82-83°C (acetone-petroleum ether) (60-80°C).

1-(2-Benzoyloxy-5-chlorophenyl)-ethanone: 1.10 g, 4 mmol; bromine: 0.64 g, 4 mmol; glacial acetic acid: 20 mL. yield: 0.95g (67%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.19 (d, J = 7.5 Hz, 2H), 7.85 (d, J = 2.4 Hz, 1H), 7.69 (t, J = 7.4 Hz, 1H), 7.57 (m, 3H), 7.26 (d, J = 8.7 Hz, 1H), 4.36 (s, 2H) ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 189.8, 164.5, 147.9, 134.3, 133.8, 131.9, 130.3, 129.7, 128.9, 128.6, 125.5, 33.7 ppm.

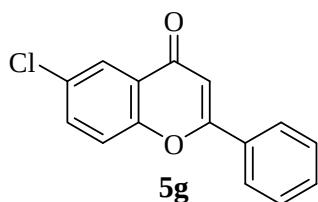


2-Benzoyloxy-5-chloro-benzoyl methyl triphenyl phosphonium bromide (4g): White crystalline granules, mp.

181-83°C (dichloromethane-ethyl acetate).

1-(2-Benzoyloxy-5-chlorophenyl)-2-bromo ethanone: 0.71 g, 2 mmol; triphenyl phosphine: 0.52 g 2 mmol. Yield: 0.89 g (72%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 9.04 (s, 1H), 7.91 (d, J = 7.9 Hz, 2H), 7.80 (m, 6H), 7.62 (d, J = 5.3 Hz, 4H), 7.48 (m, 9H), 7.07 (d, J = 8.6 Hz, 1H), 6.41 (d, J = 12.1 Hz, 2H) ppm. IR (KBr) ν_{max} 1662, 1643, 1606, 1467, 1408, 1375 cm^{-1} .



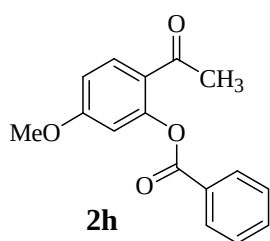
6-Chloro-2-phenyl chromen-4-one (5g): White crystalline needles, mp. 185-186°C (acetone- petroleum ether) (60-80°C) (lit.¹ mp 184-85°C).

2-Benzoyloxy-5-chloro-benzoyl methyl triphenyl phosphonium bromide: 0.31 g, 0.5 mmol; Triethyl amine: 0.5 g, 5 mmol; H_2O : 20 mL; Eluent: ethyl acetate-petroleum ether (60-80°C) (20% v/v); Yield: .012 g (91%).

IR (KBr) ν_{max} 1648, 1602, 1566, 1458, 1438, 1354 cm^{-1} .

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.20 (d, J = 2.5 Hz, 1H), 7.92 (m, 2H), 7.65 (dd, J = 9.0 Hz, 2.6 Hz, 2H), 7.53 (m, 4H), 6.83 (s, 1H) ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 177.2, 163.7, 154.6, 134.0, 131.8, 131.4, 131.2, 129.1, 126.3, 125.2, 124.9, 119.8, 107.5 ppm.

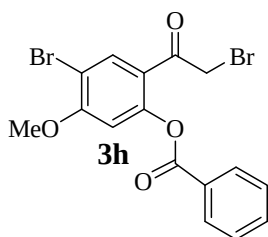


1-(2-Benzoyloxy-5-methoxyphenyl)-ethanone (2h): Colorless liquid.

1-(2-Hydroxy-4-methoxy phenyl)-ethanone: 1.66 g, 10 mmol; Benzoyl chloride: 1.4 g, 10 mmol; Pyridine: 20 mL; Eluent: ethyl acetate and petroleum ether (60-80°C) (20% v/v), Yield: 2.2 g (81%).

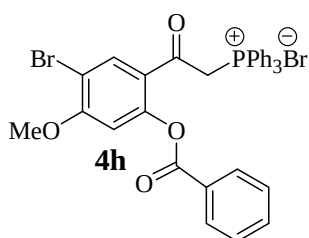
^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.22 (d, J = 7.9 Hz, 2H), 7.89 (d, J = 8.8 Hz, 1H), 7.65 (t, J = 6.9 Hz, 1H), 7.55 (t, J = 7.5 Hz, 2H), 6.87 (d, J = 8.8 Hz, 1H), 6.73 (s, 1H), 3.86 (s, 3H), 2.49 (s, 3H) ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 195.7, 165.1, 163.8, 151.7, 133.8, 132.4, 130.3, 129.4, 128.7, 123.6, 112.0, 109.3, 55.7, 29.5 ppm.

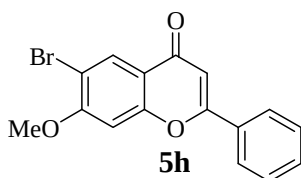


1-[2-Benzoyloxy-4-methoxy-5-bromo-phenyl]-2-bromo-ethanone (3h): Colorless sticky liquid.

1-(2-Benzoyloxy-5-methoxyphenyl)-ethanone: 2.70 g, 10 mmol;
Bromine: 1.60 g, 10 mmol; glacial acetic acid: 20 mL; yield: 2.98 g (70%).



2-Benzoyloxy-4-methoxy-5-bromo-benzoyl methyl triphenyl phosphonium bromide (4h): White crystalline granules, mp. 197-98°C (dec) (dichloromethane-ethyl acetate). 1-[2-Benzoyloxy-4-methoxy-5-bromo-phenyl]-2-bromo-ethanone: 0.60 g, 2 mmol; triphenylphosphine : 0.52 g 2 mmol. Yield: 0.59 g (56%).



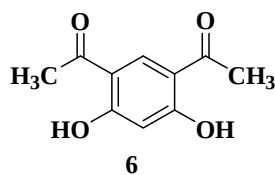
6-Bromo-7-methoxy-2-phenyl-chromene-4-one (5h): White crystalline needles, mp. 191-92°C (dec) (acetone-petroleum ether, 60-80°C).

2-Benzoyloxy-4-methoxy-5-bromo-benzoyl methyl triphenyl phosphonium bromide : 0.35 g 0.5 mmol; Triethylamine: .5 g, 5 mmol; H₂O: 20 mL; Eluent: ethyl acetate-petroleum ether (60-80°C) (20% v/v); Yield: .01g (63%). IR (KBr) $\nu_{\max}$ 1637, 1597, 1452, 1430, 1358 cm⁻¹.

¹H NMR (500MHz, CDCl₃, 22°C): δ 8.41 (s, 1H), 7.90 (m, 2H), 7.53 (m, 3H), 7.00 (s, 1H), 6.78 (s, 1H), 4.03 (s, 3H) ppm.

¹³C NMR (75 MHz, CDCl₃, 22°C): δ 176.6, 163.3, 160.1, 156.9, 131.6, 130.1, 129.1, 126.2, 118.6, 110.1, 107.6, 100.1, 56.9 ppm.

The crystal structure has been deposited at the Cambridge Crystallographic Data Centre and allocated the deposition number **CCDC 838053**.



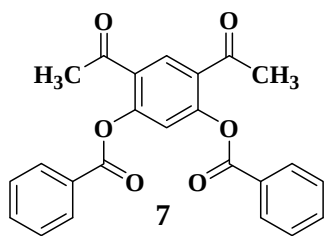
1-(2,4-Dihydroxy-5-acetylphenyl)-ethanone (6):

colourless crystalline needles, mp 178-79°C (petroleum ether, 60-80°C).

A mixture of freshly prepared fused zinc chloride (10.1 g, 100 mmol) and resorcinol (5.5g, 50 mmol) in dry acetic anhydride (15.3 g, 150 mmol) are taken in a round-bottom flask and heated gently in an oil bath to 142°C for 15 min with the help of a magnetic stirrer. The reaction mixture was allowed to cool at room temperature and a syrupy mass was obtained. After then 150 gm crushed ice and conc HCl (80mL) was added to it and

stirred for 30 min, an orange red color crystalline material separated out. The crude product crystallized from petroleum ether (60-80°C) to give the expected product as colorless crystalline needles. Yield: .5.9 g (61%); ^1H NMR (300MHz, CDCl_3 , 22°C): δ 12.91 (s, 2H), 8.18 (s, 1H), 6.38 (s, 1H), 2.61 (s, 6H) ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 202.4, 168.8, 136.2, 113.6, 104.9, 26.0 ppm.

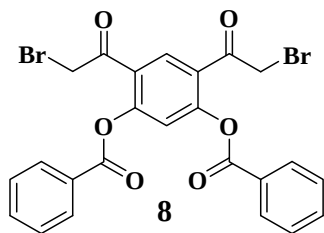


1-(2,4-Dibenzoyloxy-5-acetylphenyl)-ethanone (7): colourless crystalline granules, mp 118-19°C (acetone-petroleum ether, 60-80°C).

1-(2,4-Dihydroxy-5-acetylphenyl)-ethanone: 1.94 g, 10 mmol;
Benzoyl chloride: 2.81 g, 20 mmol; dry pyridine: 30 mL; Yield: 2.90 g (72%).

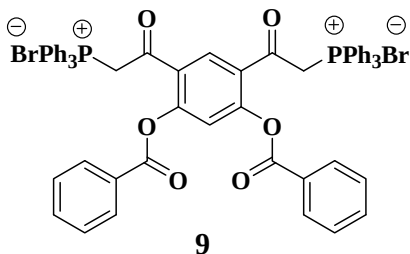
^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.41 (s, 1H), 8.20 (dd, J = 8.0 Hz, 1.5 Hz, 4H), 7.67 (m, 2H), 7.54 (t, J = 7.7 Hz, 4H), 7.25 (s, 1H), 2.60 (s, 6H), ppm.

^{13}C NMR (75 MHz, CDCl_3 , 22°C): δ 195.6, 164.3, 152.7, 134.2, 132.6, 130.4, 129.0, 128.8, 128.5, 119.8, 29.8 ppm.



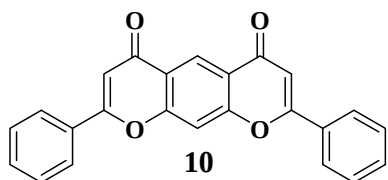
1-[2,4-Dibenzoyloxy-5-(2-bromoacetyl)-phenyl]-2-bromo-ethanone (8): 1-(2,4-Dibenzoyloxy-5-acetylphenyl)-ethanone (1.05g, 2.50 mmol) and acetic acid (25 mL) was taken in a 100 mL round-bottomed flask and bromine (0.8 g, 5 mmol) added to it, and the whole reaction mixture was heated gently until the red color of the reaction mixture turned to colorless or straw yellow. It was then cooled at room temperature and 150 g crushed ice added to it. A sticky semi solid mass was so obtained after solvent extraction. Yield: .0.95 g (65%).

^1H NMR (300MHz, CDCl_3 , 22°C): δ 8.42 (d, J = 10.2 Hz, 1H), 8.16 (d, J = 7.6 Hz, 4H), 7.65 (t, J = 7.0 Hz, 2H) 7.51 (t, J = 7.5 Hz, 4H) 7.38 (s, 1H) 4.43 (s, 4H) ppm.



2, 4-Dibenzoyloxy-5-(2-oxoethyltriphenylphosphonium)-benzoyl methyl triphenyl phosphonium dibromide (9): colourless crystalline granules, mp > 270°C (dichloromethane-ethyl acetate).

1-[2,4-Dibenzoyloxy-5-(2-bromoacetyl)-phenyl]-2-bromoethanone: 0.56 g, 1 mmol; triphenylphosphine: 0.52 g, 2 mmol. Yield: 0.77 g (71%).



2, 8-Diphenyl-pyrano [3, 2-g] chromene-4, 6-dione (10):

colourless crystalline needles, mp 268-69°C (acetone-petroleum ether, 60-80°C).

2, 4-Dibenzoyloxy-5-(2-oxoethyltriphenylphosphonium)-benzoyl methyl triphenyl phosphonium dibromide: 0.22 g, 0.20 mmol; Triethylamine: 1.01 g, 10 mmol; H₂O: 40 mL; Eluent: ethyl acetate-petroleum ether (60-80°C) (30% v/v); Yield: 0.053g (72%). IR (KBr) $\nu_{\max}$ 1646, 1609, 1450, 1467, 1366, cm⁻¹.

¹H NMR (300MHz, CDCl₃, 22°C): δ 9.14 (s, 1H), 7.95 (m, 4H), 7.73 (s, 1H), 7.57 (d, *J* = 6.1 Hz, 6H), 6.85 (s, 2H) ppm.

¹³C NMR (75 MHz, CDCl₃, 22°C): δ 177.2, 163.8, 158.6, 132.0, 131.2, 129.2, 126.3, 126.1, 121.7, 107.5, 106.4, ppm.

Mass (EI), *m/z* (%): 389.13 (M⁺ + Na).

References:

1. Bennardi, D.; Romanelli, G.; Autino, J.; Pizzio, L.; Vazquez, P.; Caceres, C.; Blanco, M. *Reac Kinet Mech Cat* **2010**, *100*, 165-174.
2. Sarada, S. R.; Pathan, M. Y.; Paike, V. V.; Pachmase, P. R.; Jadhav, W. N.; Pawar, R. P. *Arkivoc.* **2006**, (XVI), 43-48.
3. Sing, O. V.; Muthukrishnan, M.; Raj, G. *Synth. Commun.* **2005**, *35*, 2723-2728.
4. Kumar, P.; Bodas, M. S. *Org. Lett.* **2000**, *2*, 3821-3823.



Fig. 1: ^1H NMR spectrum of 1-(2-Benzoyloxy-5-methylphenyl)-ethanone (2a)

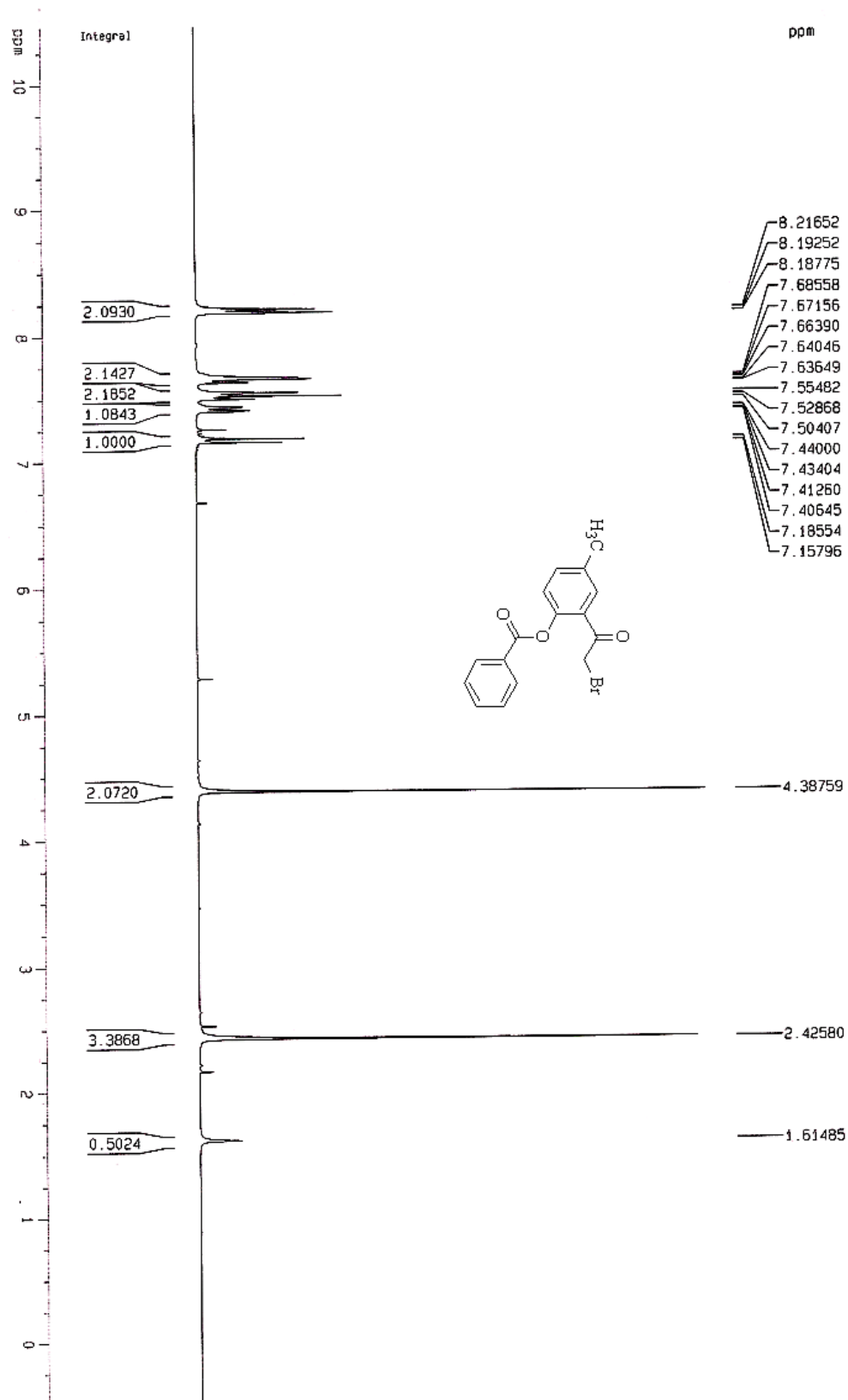


Fig. 2: ^1H NMR spectrum of 1-(2-Benzoyloxy-5-methylphenyl)-2-bromo ethanone (3a)

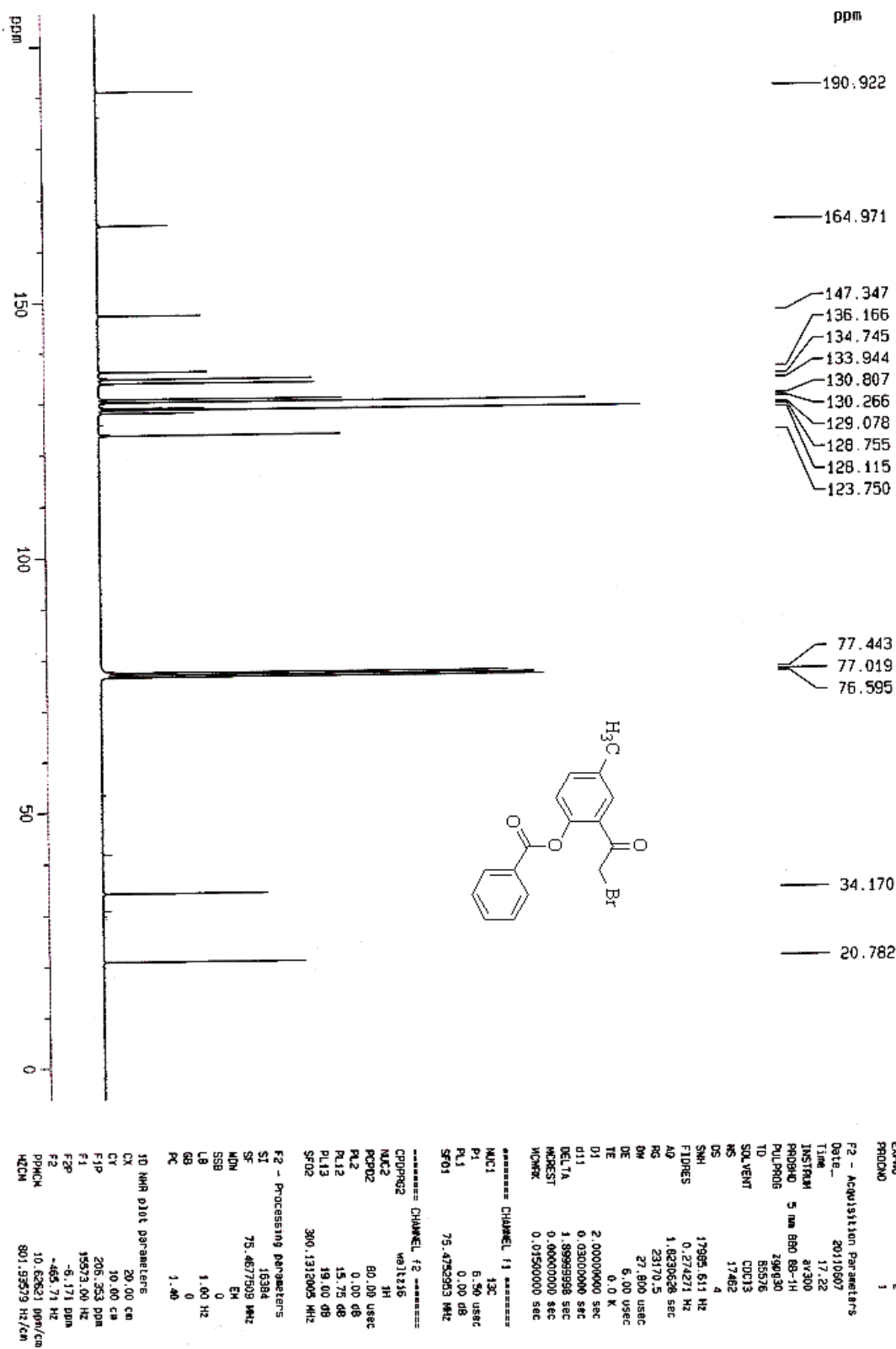


Fig. 3: ^{13}C NMR spectrum of 1-(2-Benzoyloxy-5-methylphenyl)-2-bromo ethanone (3a)

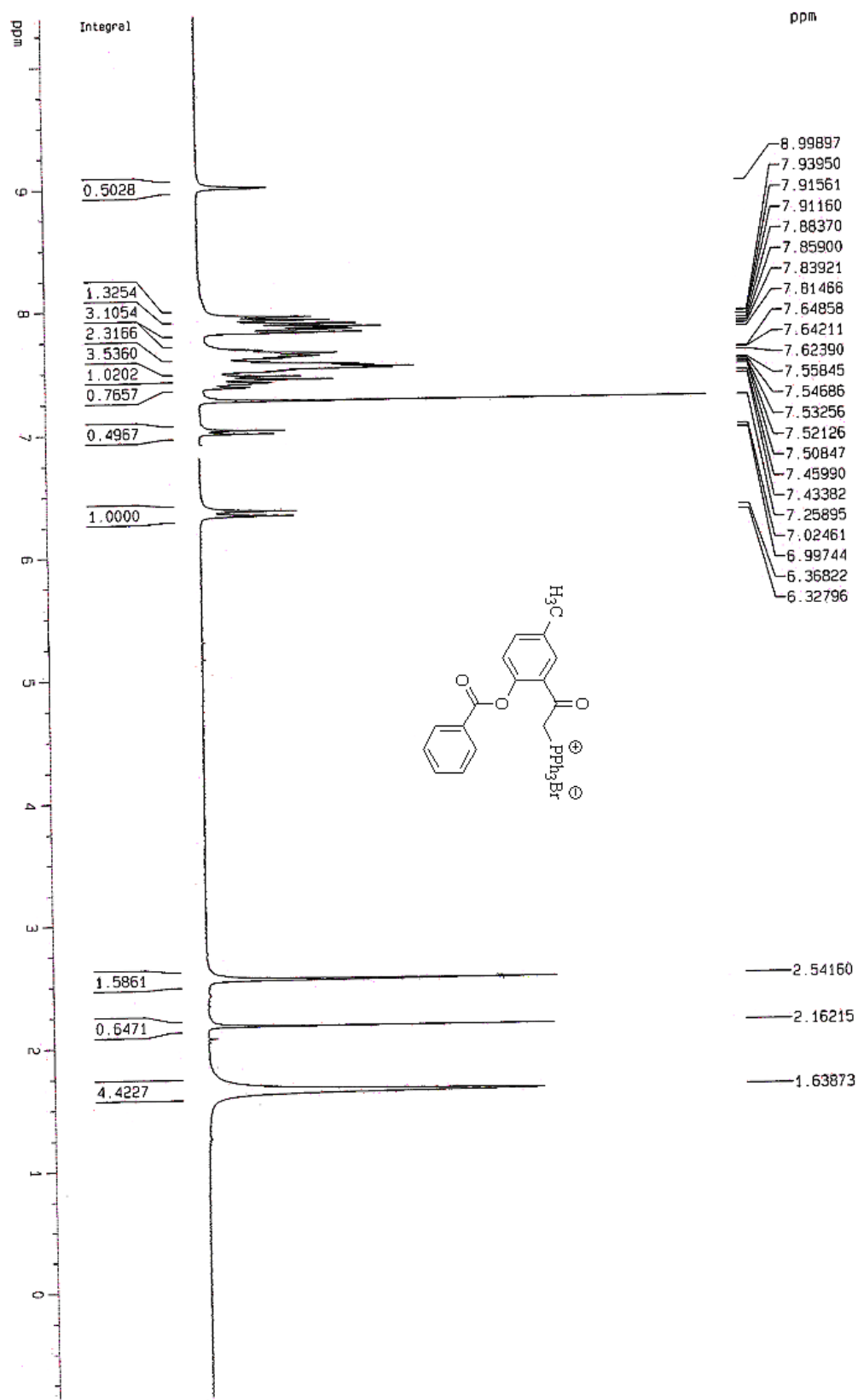


Fig. 4: ^1H NMR spectrum of 2-Benzoyloxy-5-methyl-benzoyl methyl triphenyl phosphonium bromide (4a)

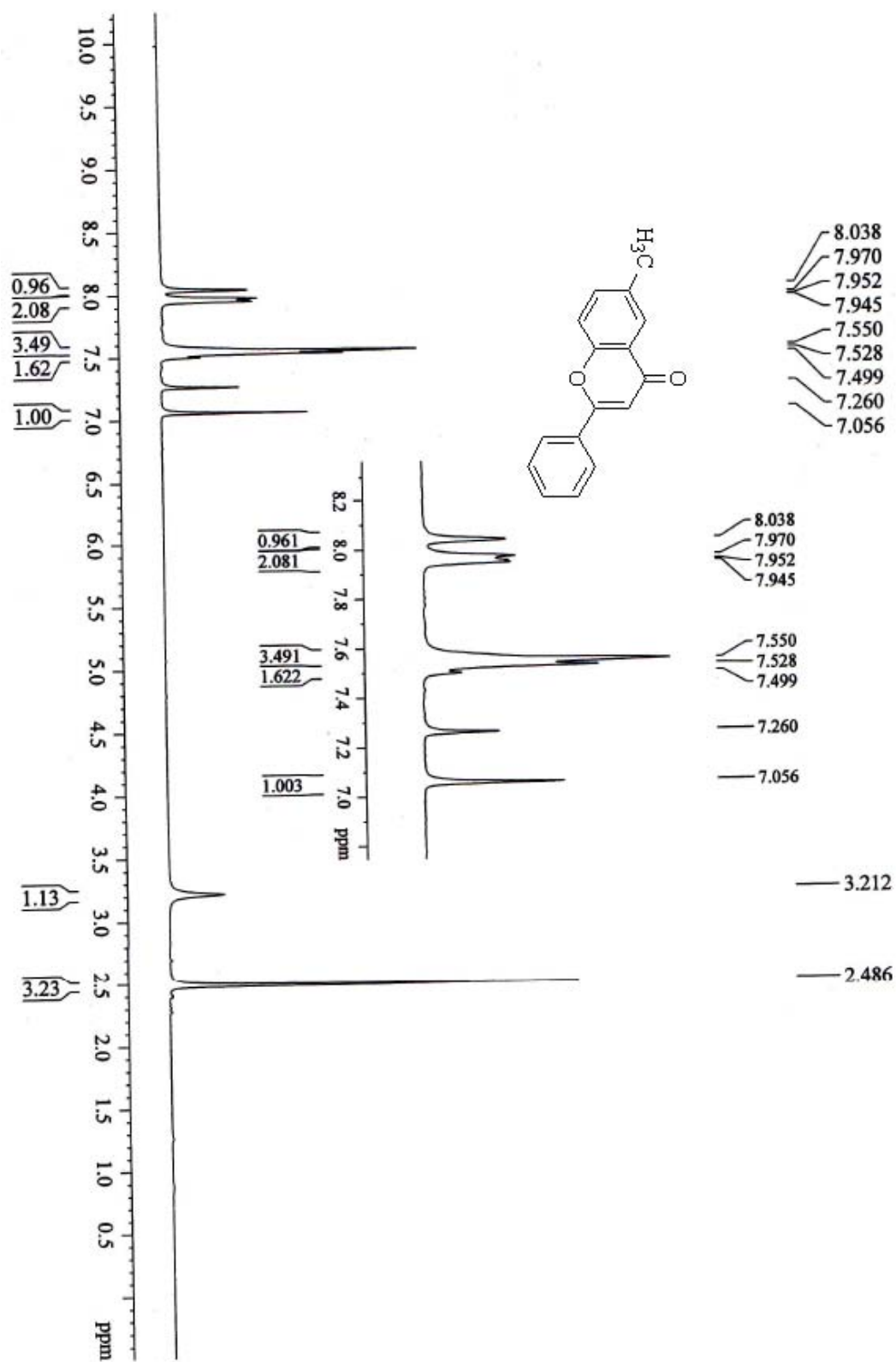


Fig. 5: ^1H NMR spectrum of 6-Methyl-2-phenyl chromen-4-one (5a)

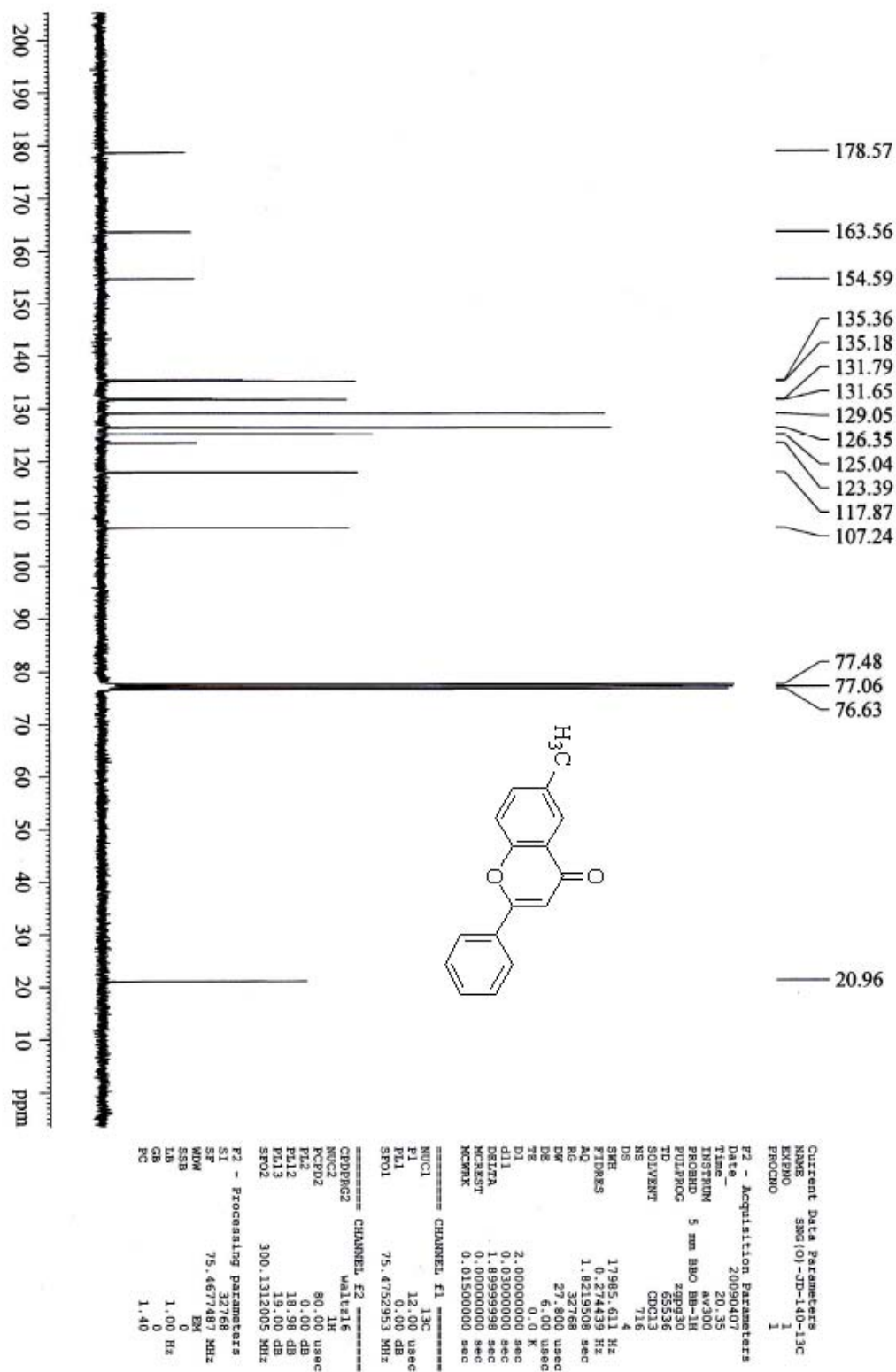


Fig. 6: ¹³C NMR spectrum of 6-Methyl-2-phenyl chromen-4-one (5a)

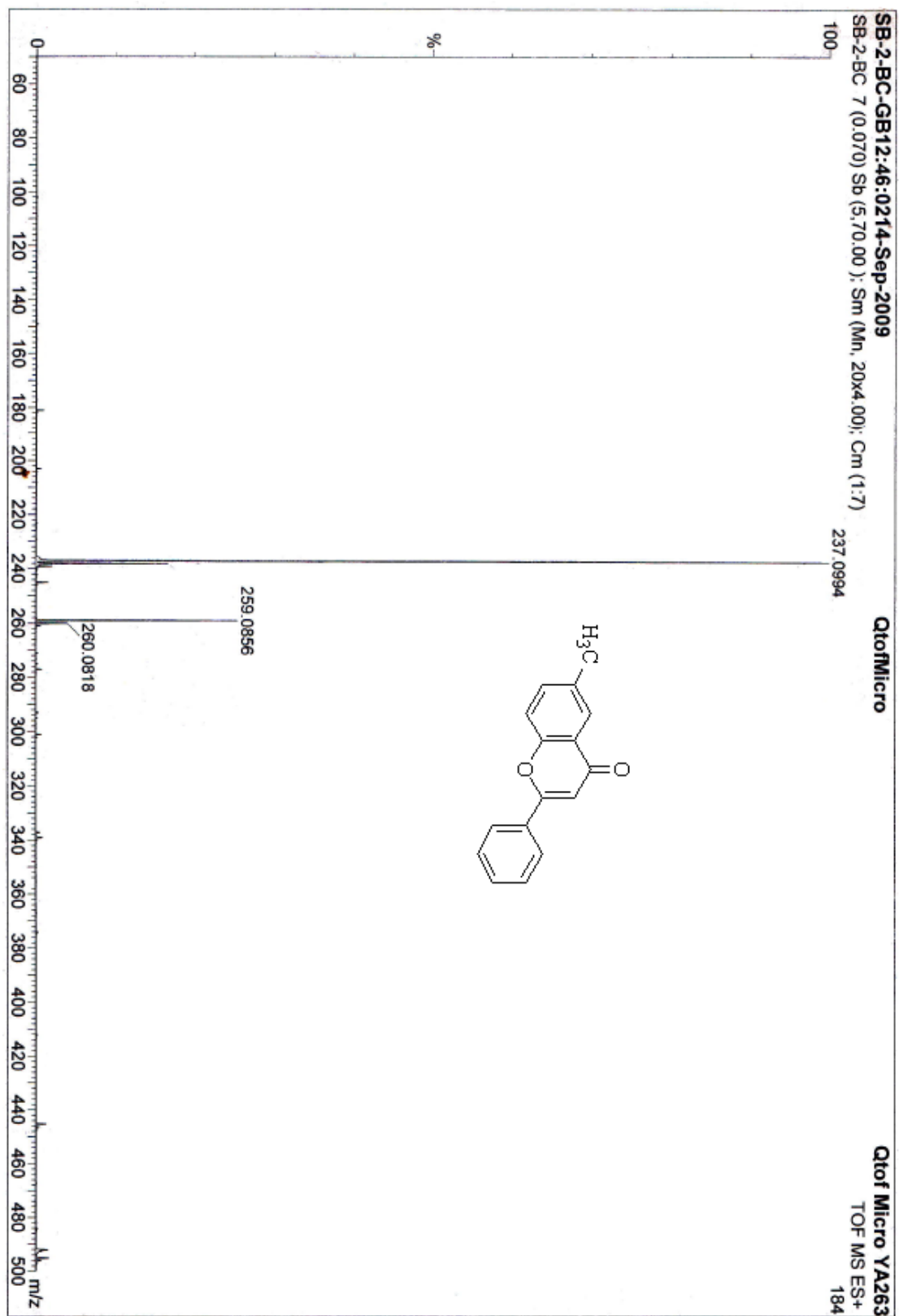


Fig 7: High Resolution Mass spectrum of 6-Methyl-2-phenyl chromen-4-one (5a):

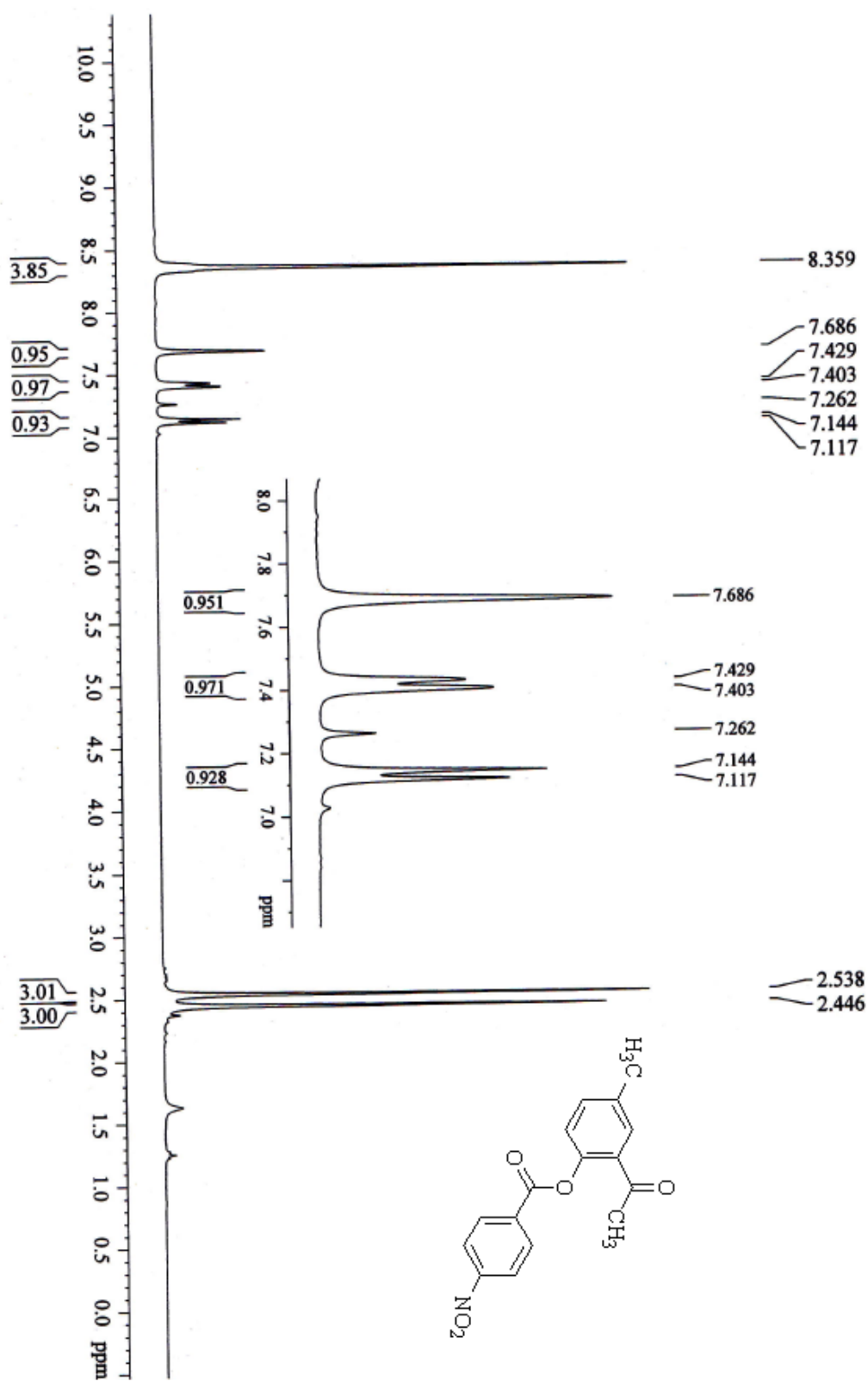


Fig 8: ^1H NMR spectrum of 1-[2-(4-Nitrobenzoyloxy)-5-methylphenyl]-ethanone (2b)

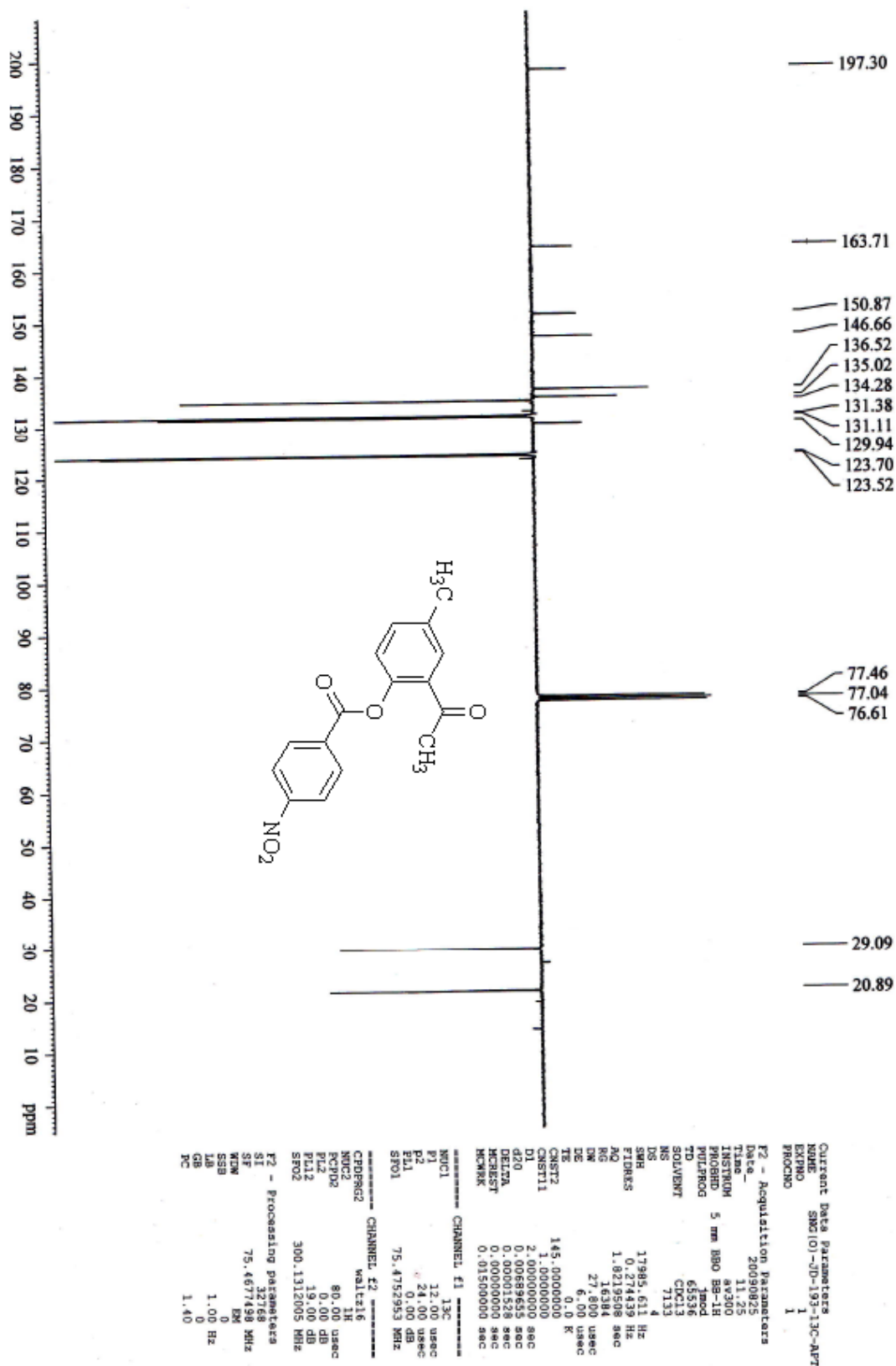


Fig. 9: ¹³C NMR spectrum of 1-[2-(4-Nitrobenzoyloxy)-5-methylphenyl]-ethanone (2b)

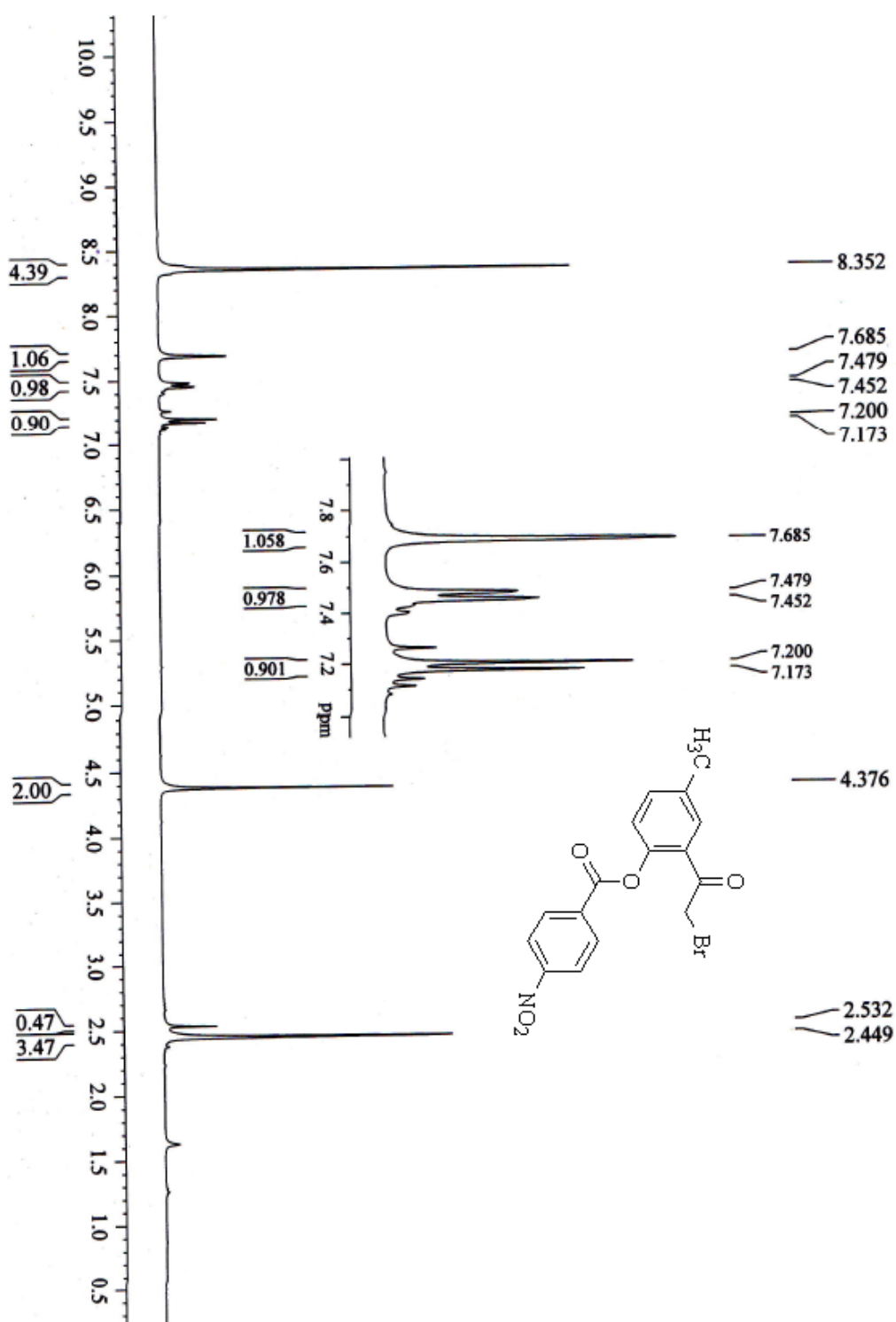


Fig. 10: ¹H NMR spectrum of 1-[2-(4-Nitrobenzoyloxy)-5-methylphenyl]-2-bromoethanone (3b)

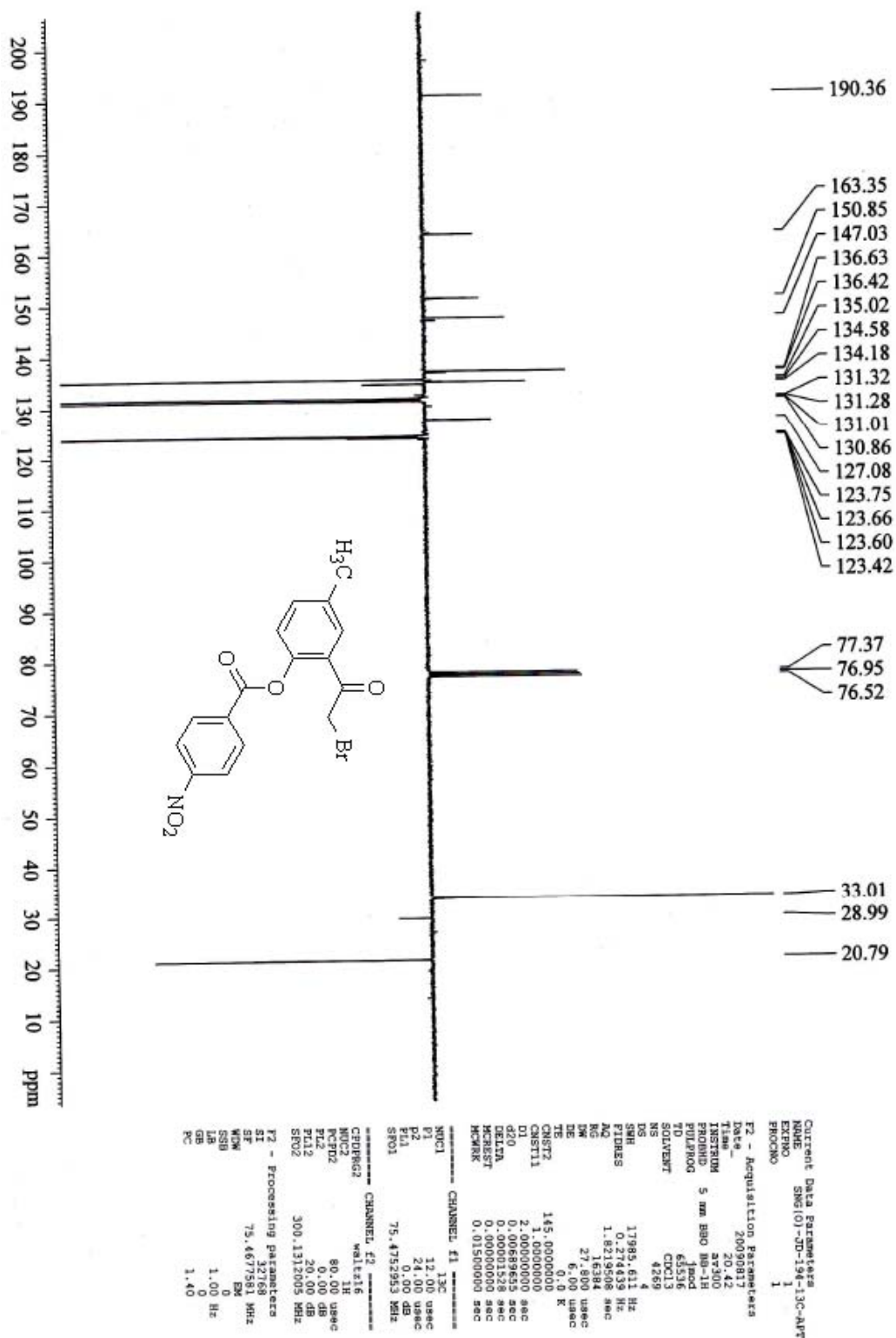


Fig. 11: ¹³C NMR spectrum of 1-[2-(4-Nitrobenzoyloxy)-5-methylphenyl]-2-bromoethanone (3b)

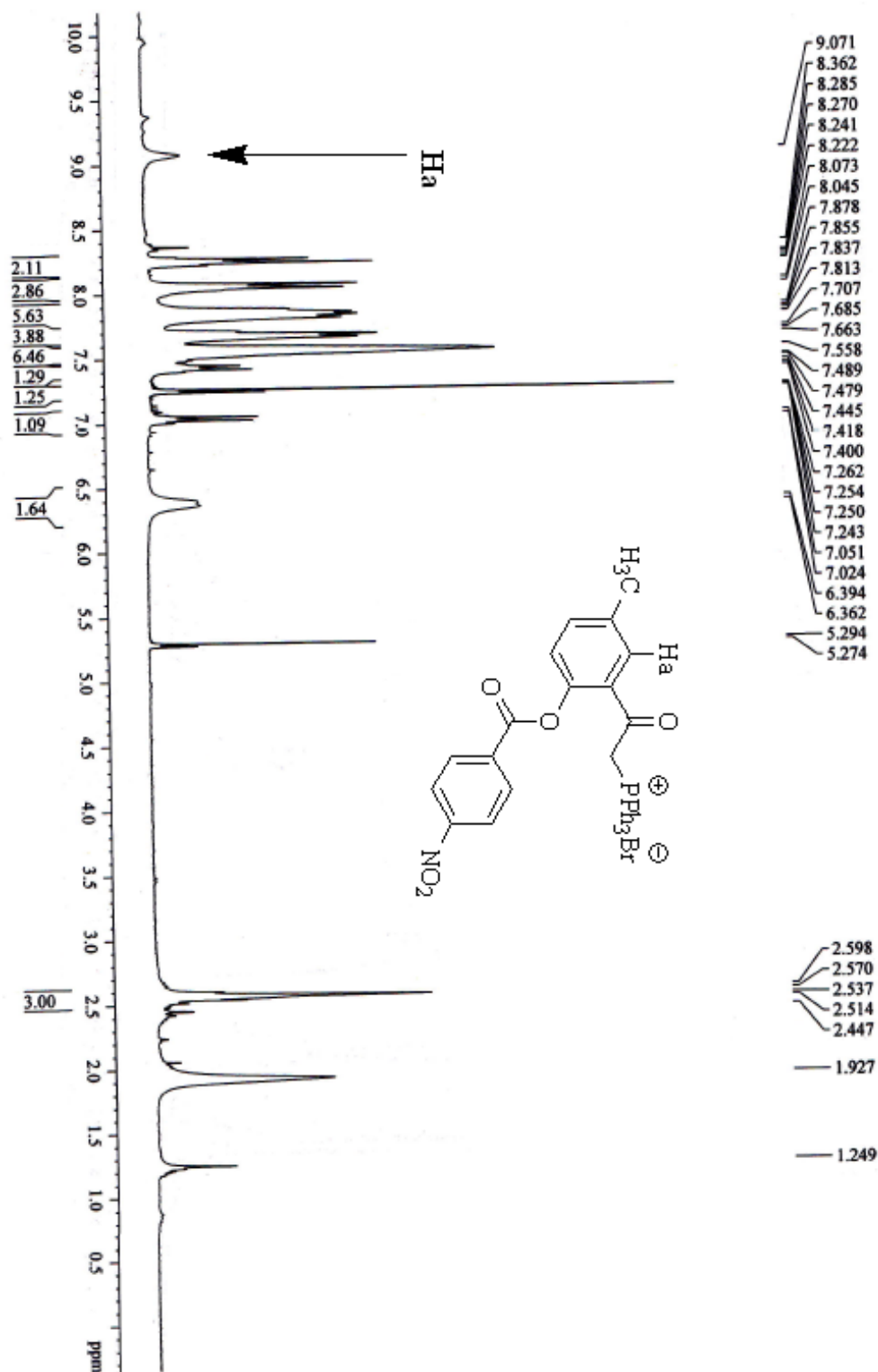


Fig. 12: ¹H NMR spectrum of 2-(4-Nitrobenzoyloxy)-5-methyl-benzoyl methyl triphenyl phosphonium bromide (4b)

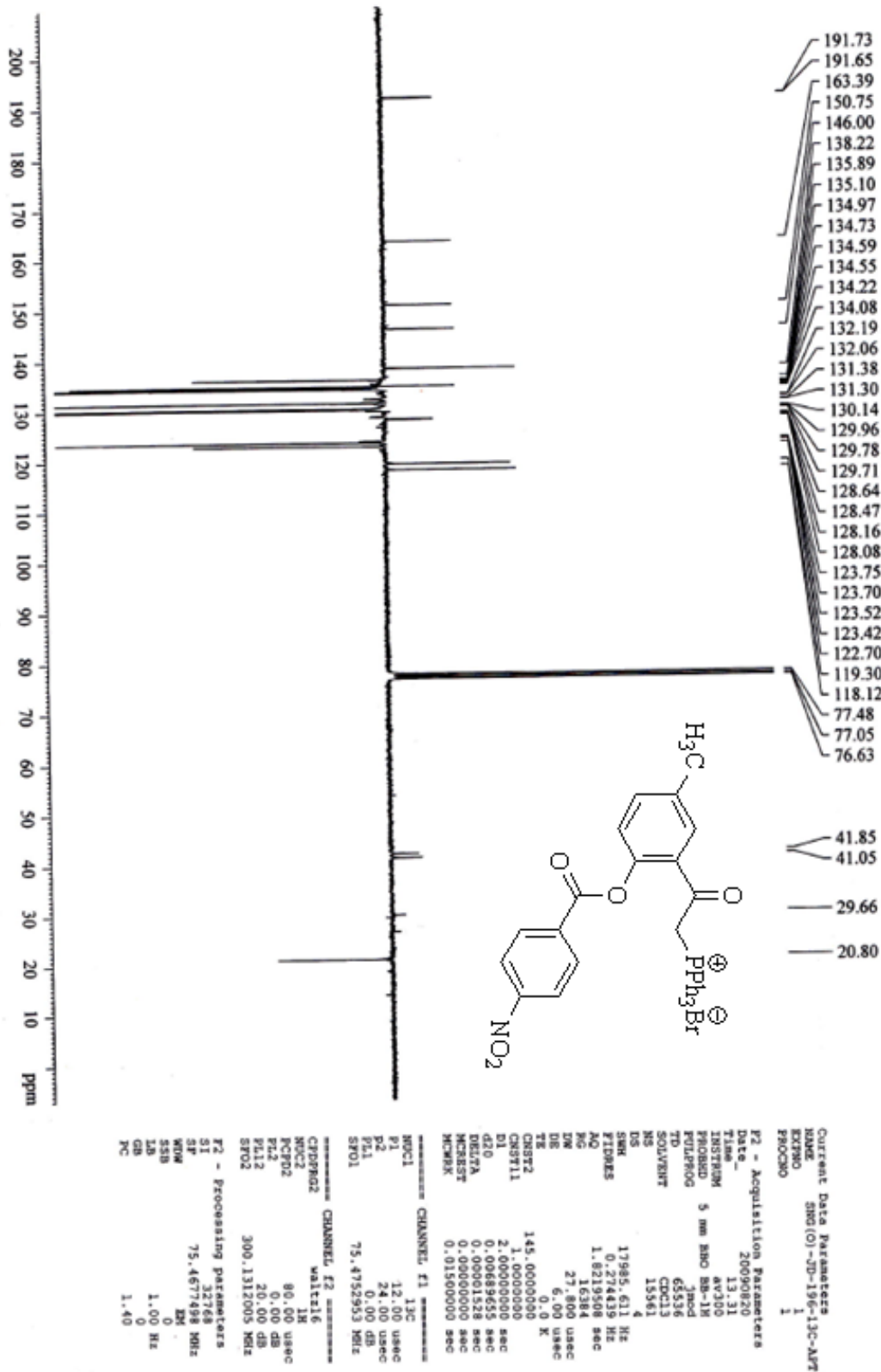


Fig. 13: ^{13}C NMR spectrum of 2-(4-Nitrobenzoyloxy)-5-methyl-benzoyl methyl triphenyl phosphonium bromide (4b)

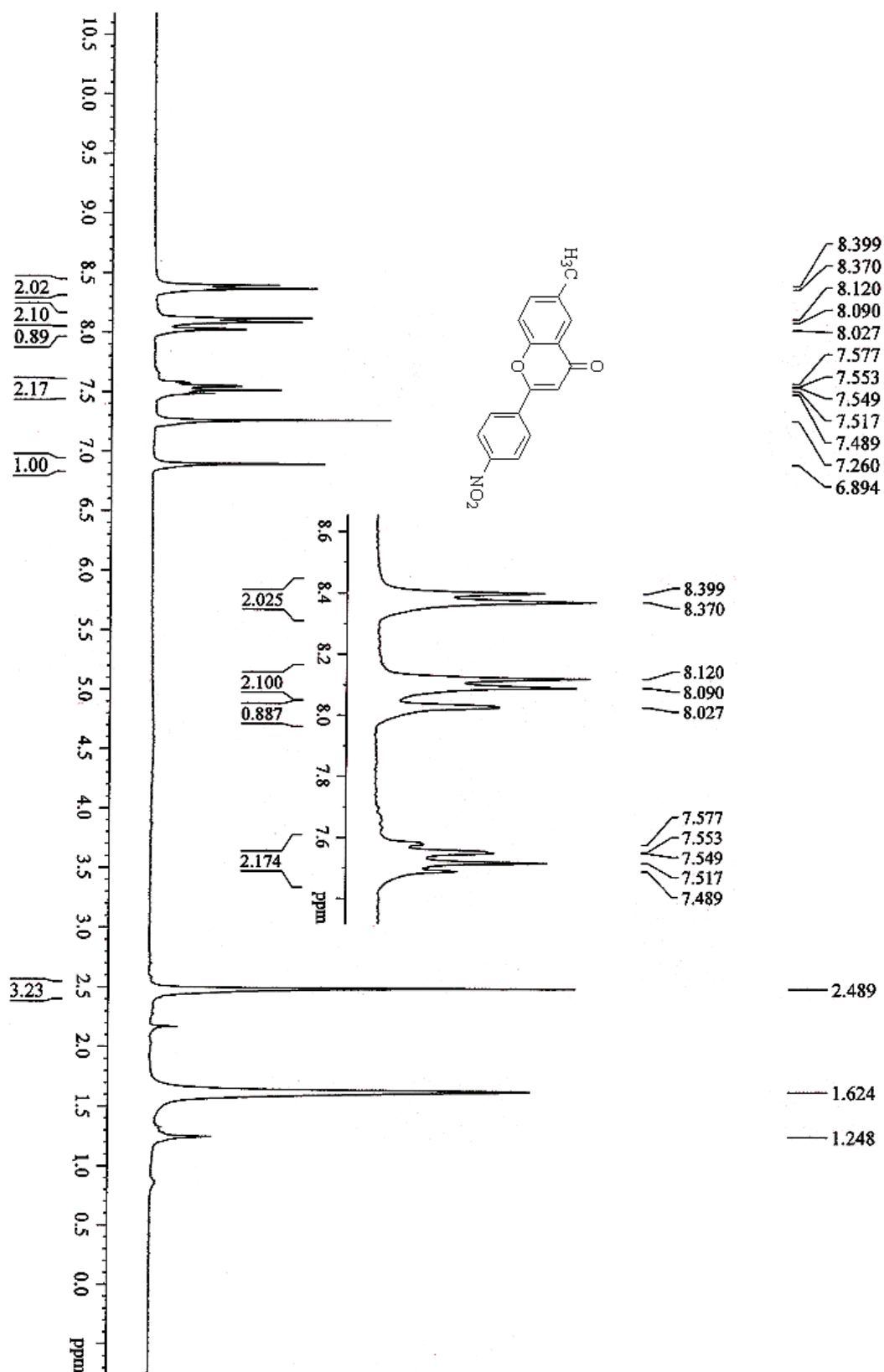


Fig. 14: ¹H NMR spectrum of 6-Methyl-2-(4-nitro phenyl)-chromen-4-one (5b)

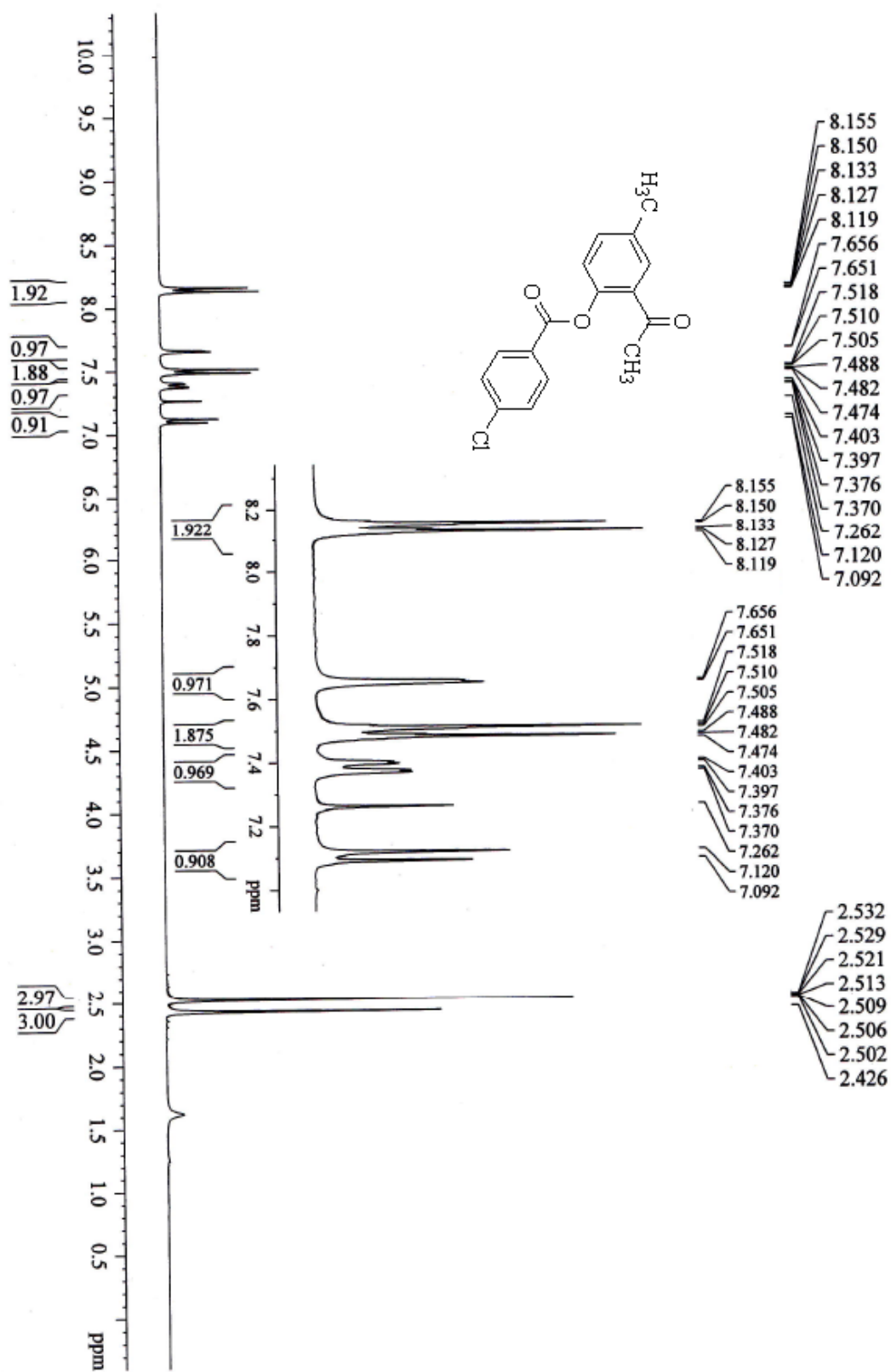


Fig. 15: ¹H NMR spectrum of 1-[2-(4-Chlorobenzoyloxy)-5-methylphenyl]-ethanone (2c)

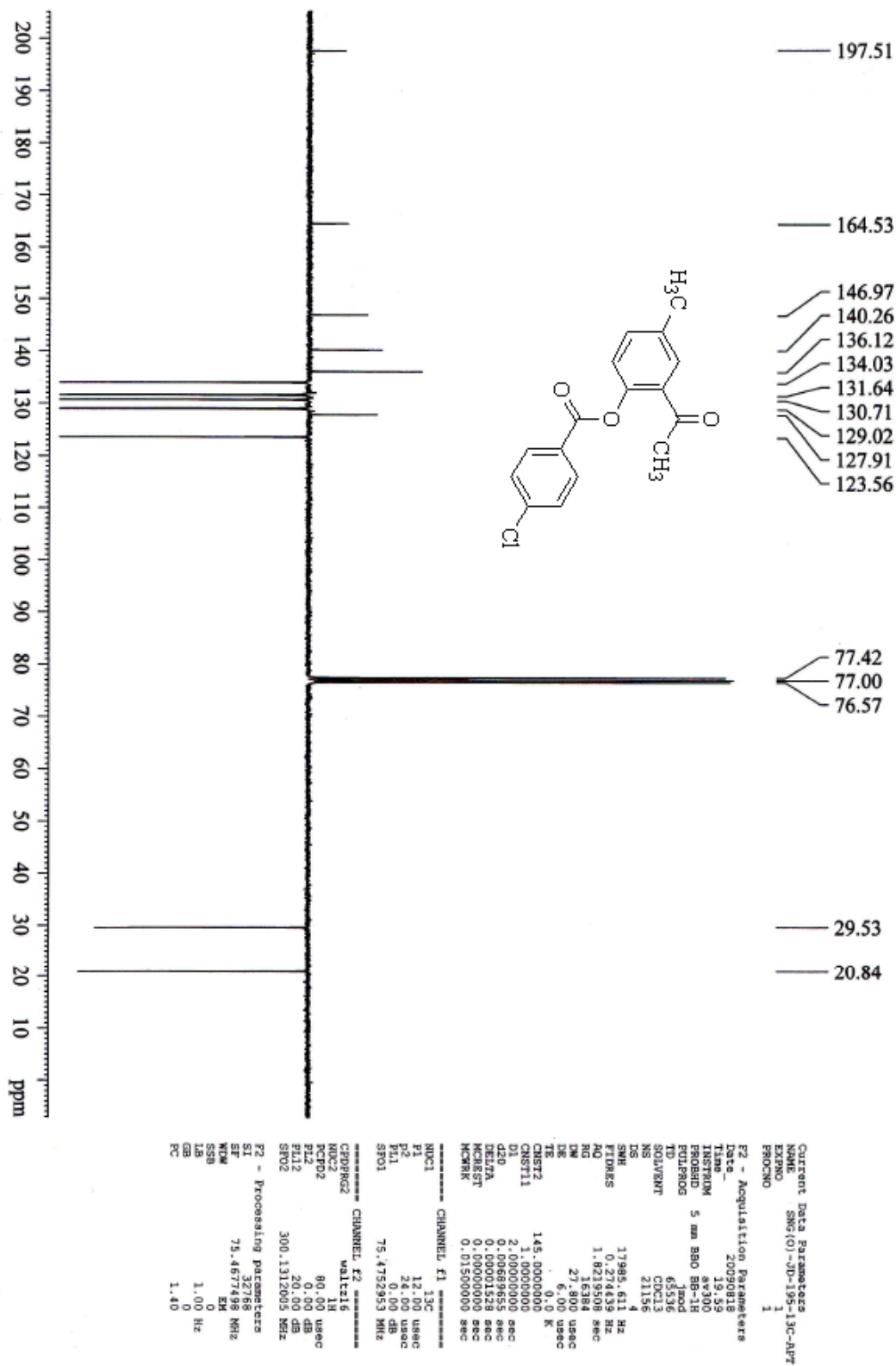


Fig. 16: ^{13}C NMR spectrum of 1-[2-(4-Chlorobenzoyloxy)-5-methylphenyl]-ethanone (2c)

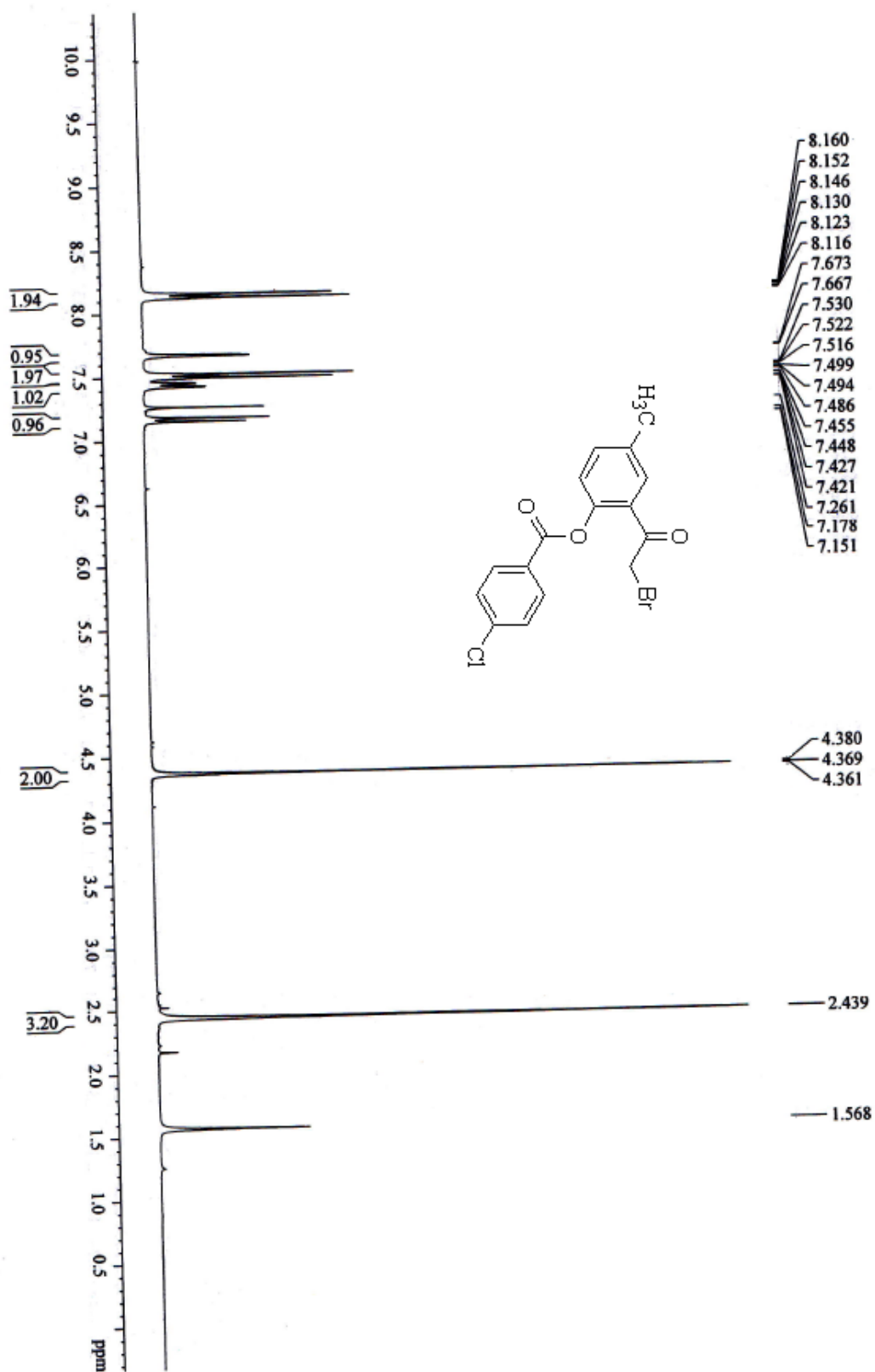


Fig. 17: ¹H NMR spectrum of 1-[2-(4-Chlorobenzoyloxy)-5-methylphenyl]-2-bromoethanone (3c)

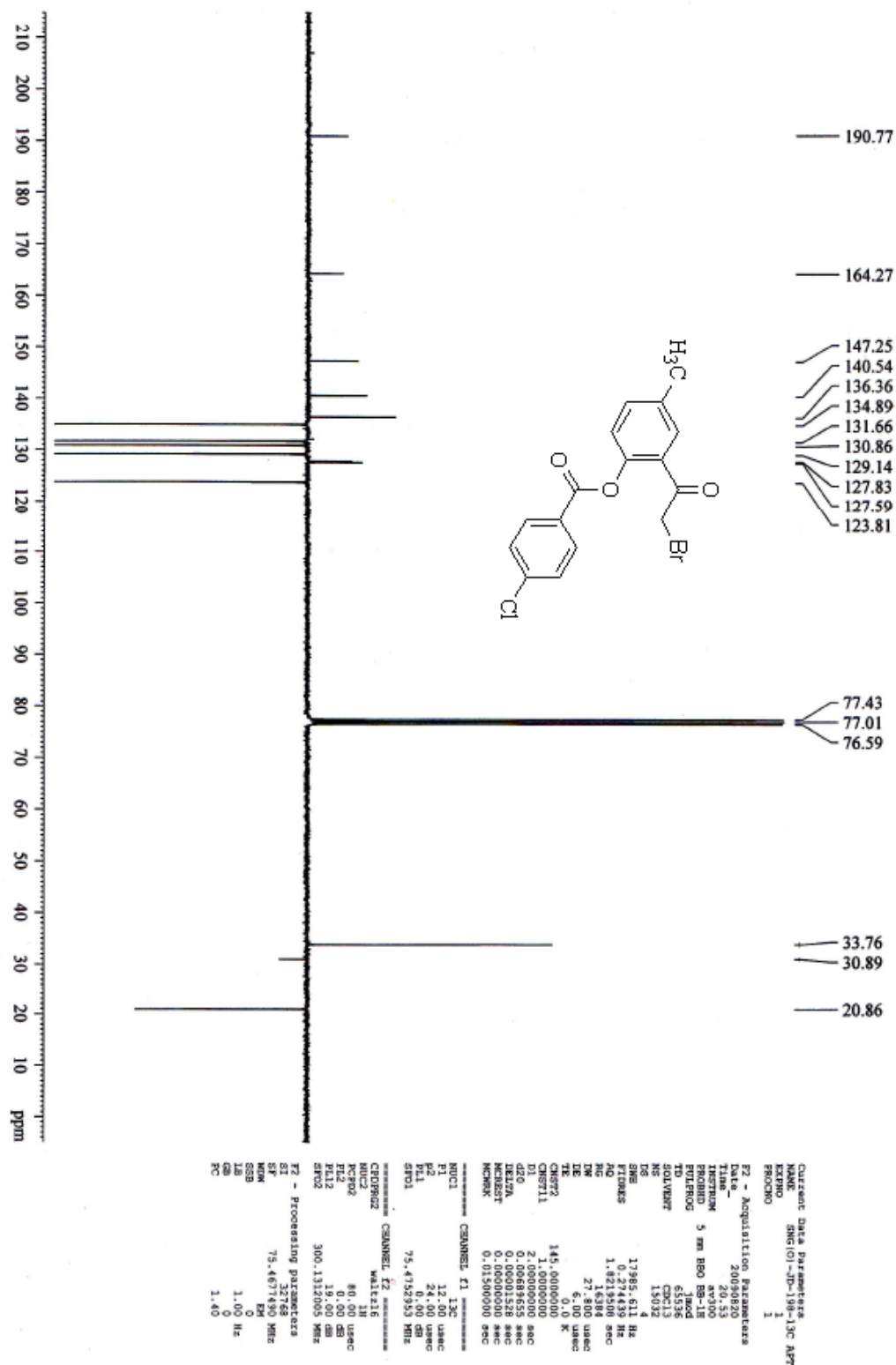


Fig. 19: ¹³C NMR spectrum of 1-[2-(4-Chlorobenzoyloxy)-5-methylphenyl]-2-bromoethanone (3c)

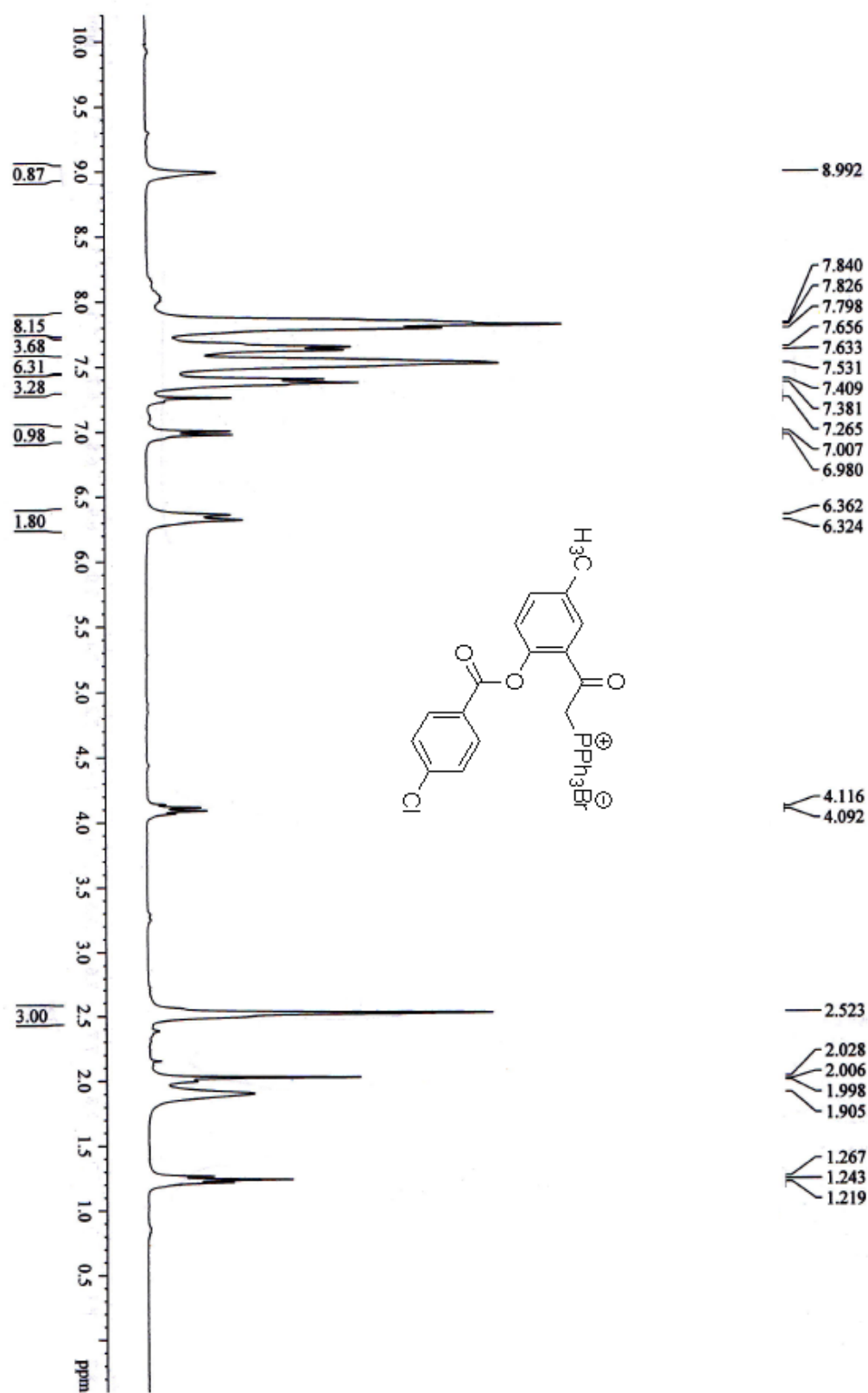


Fig. 20: ¹H NMR spectrum of 2-(4-Chlorobenzoyloxy)-5-methyl-benzoyl methyl triphenyl phosphonium bromide (4c)

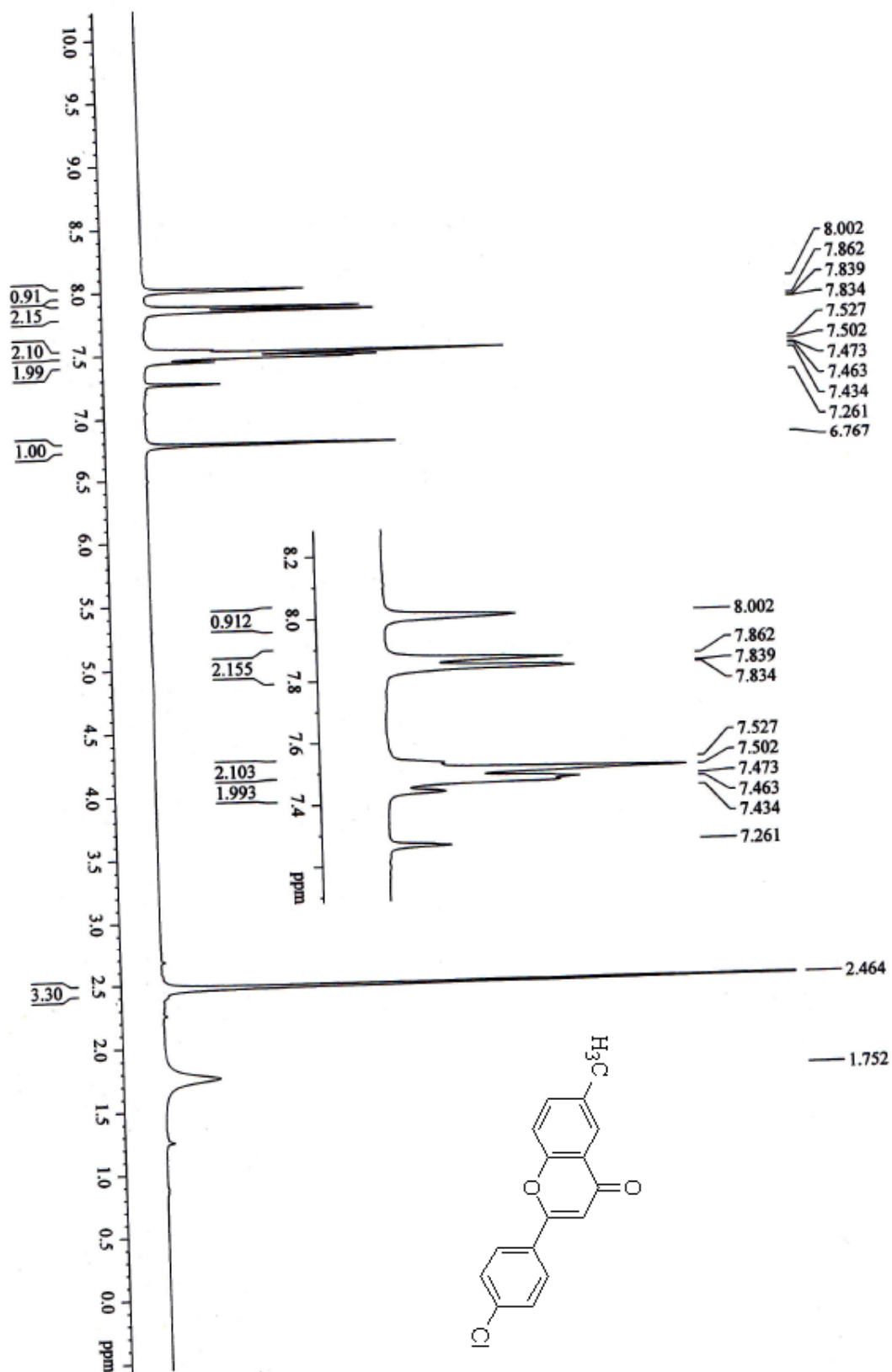


Fig. 21: ¹H NMR spectrum of 6-Methyl-2-(4-chlorophenyl)-chromen-4-one (5c)



Fig. 22: ¹³C NMR spectrum of 6-Methyl-2-(4-chlorophenyl)-chromen-4-one (5c)

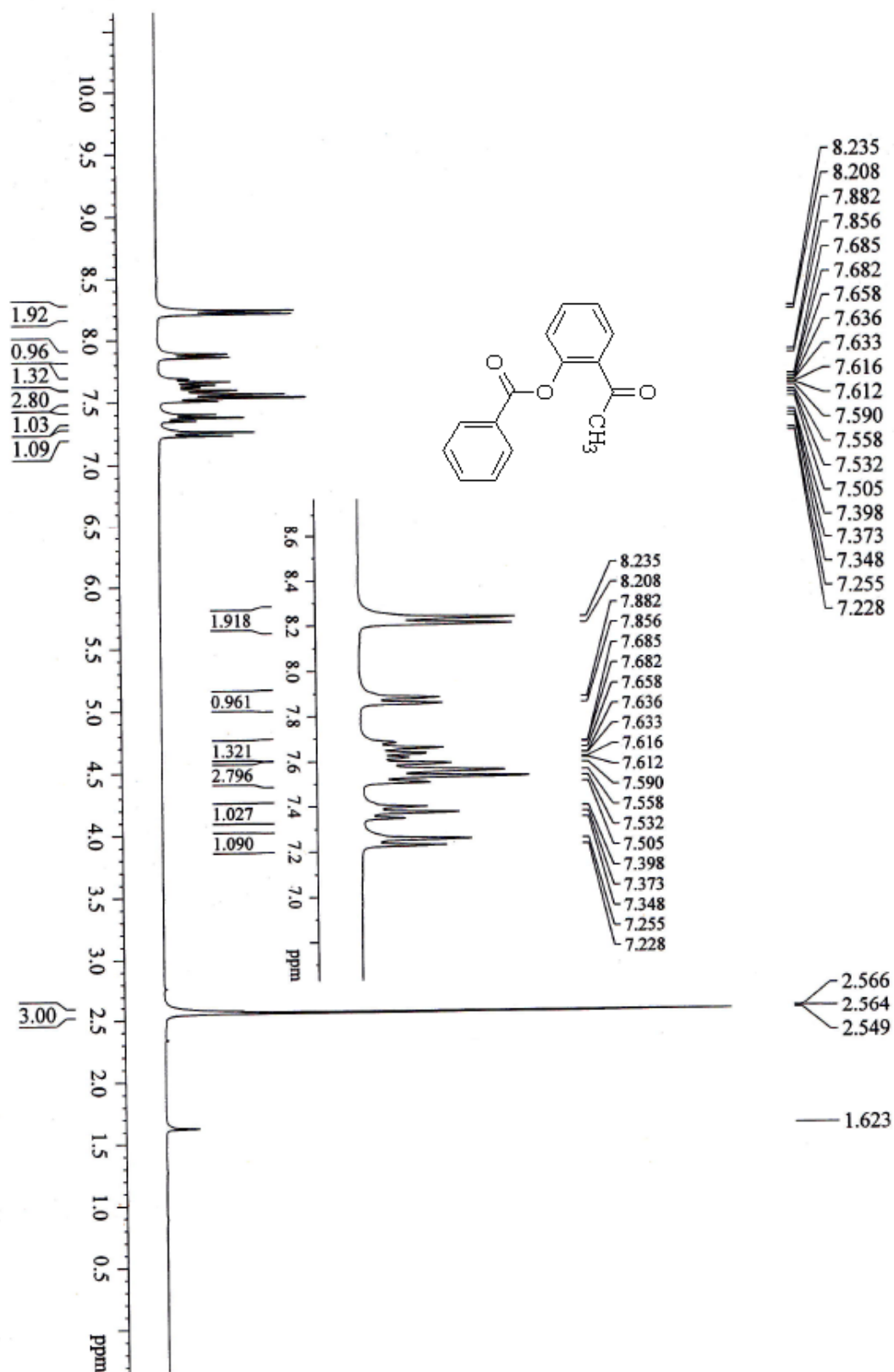


Fig. 23: ¹H NMR spectrum of 1-[2-(Benzoyloxy)-phenyl]-ethanone (2d)

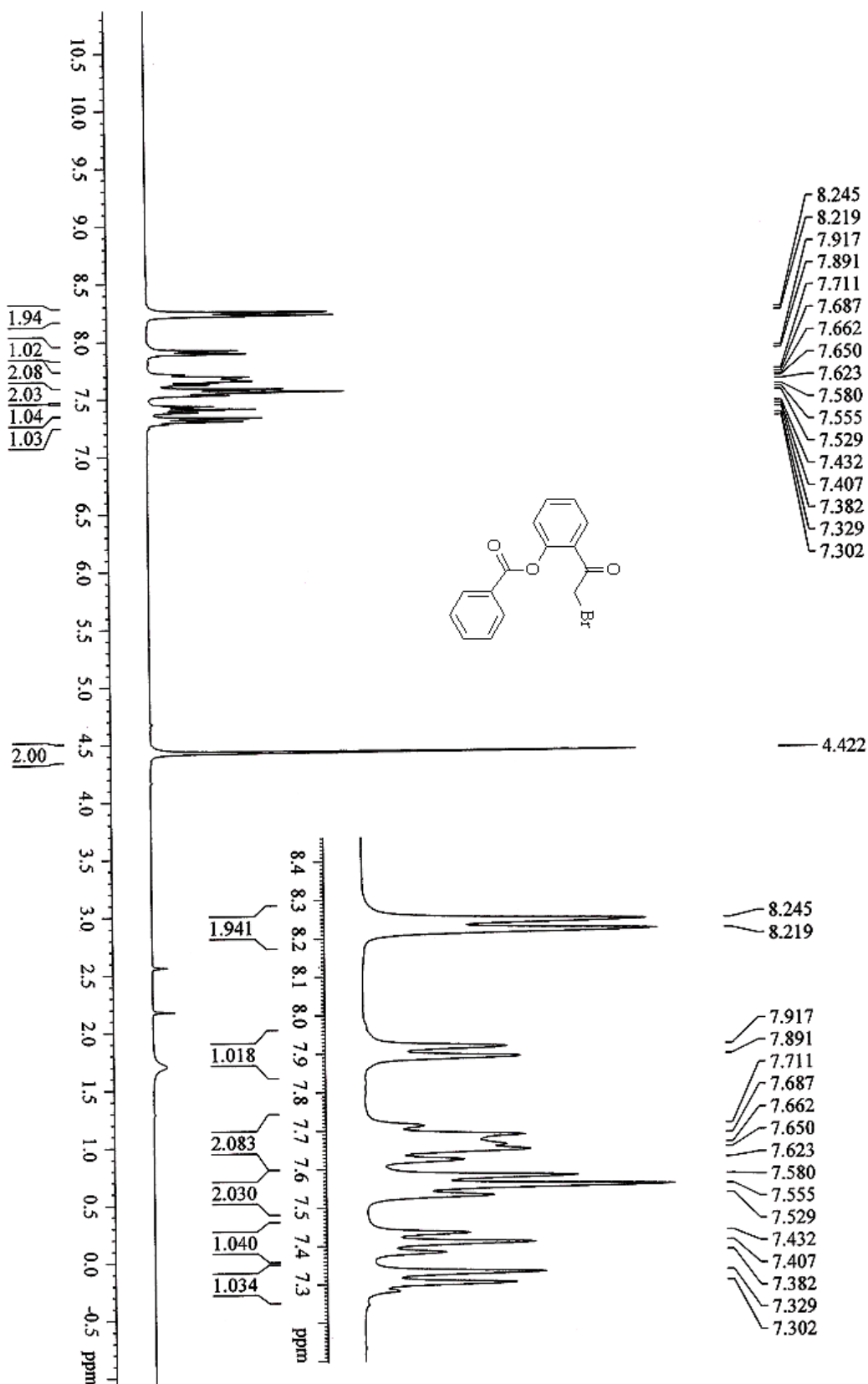


Fig. 24: ¹H NMR spectrum of 1-[2-(Benzoyloxy)-phenyl]-2-bromo ethanone (3d)

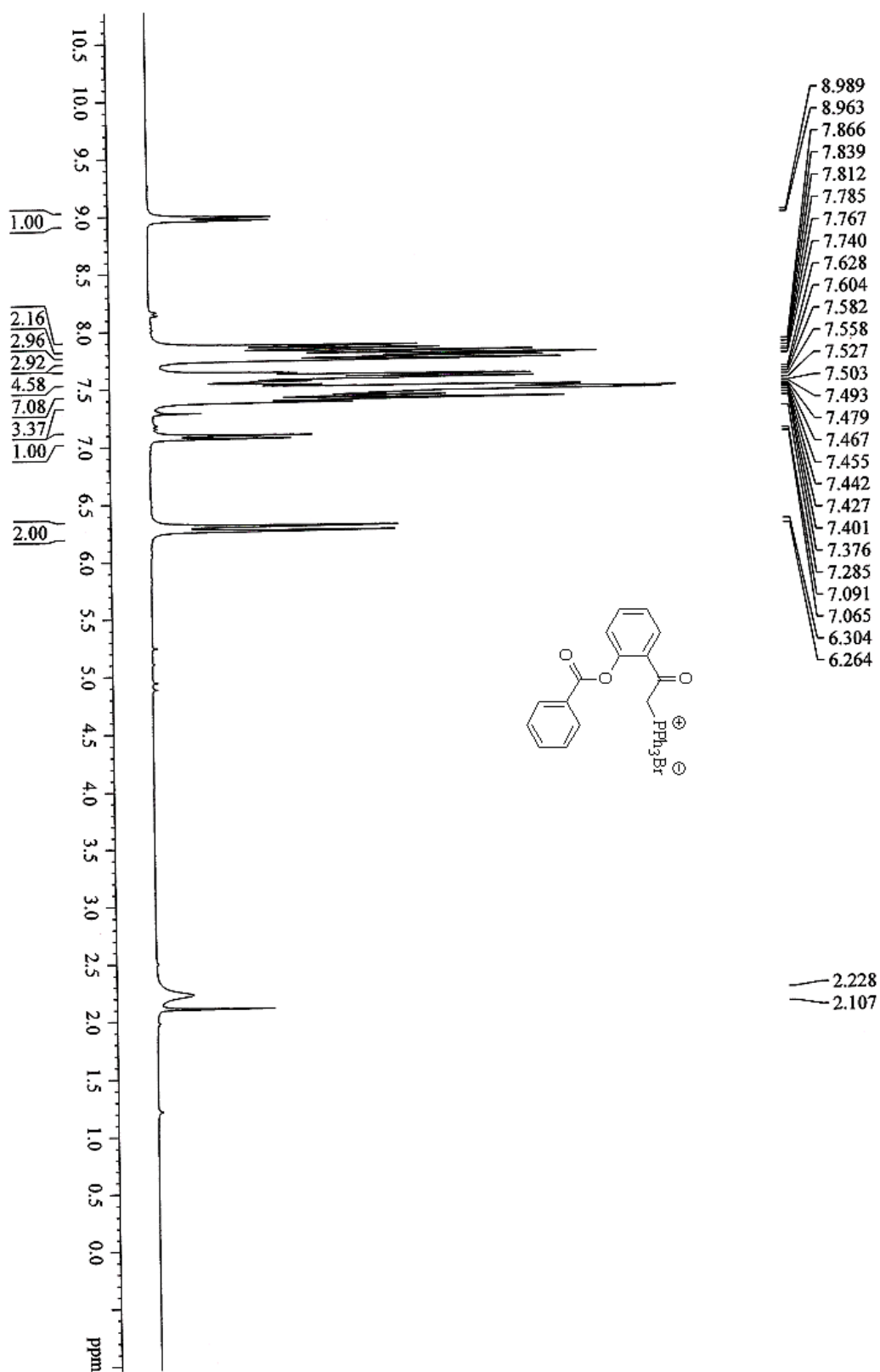


Fig. 25: ^1H NMR spectrum of 2-(Benzoyloxy)-benzoyl methyl triphenyl phosphonium bromide (4d)

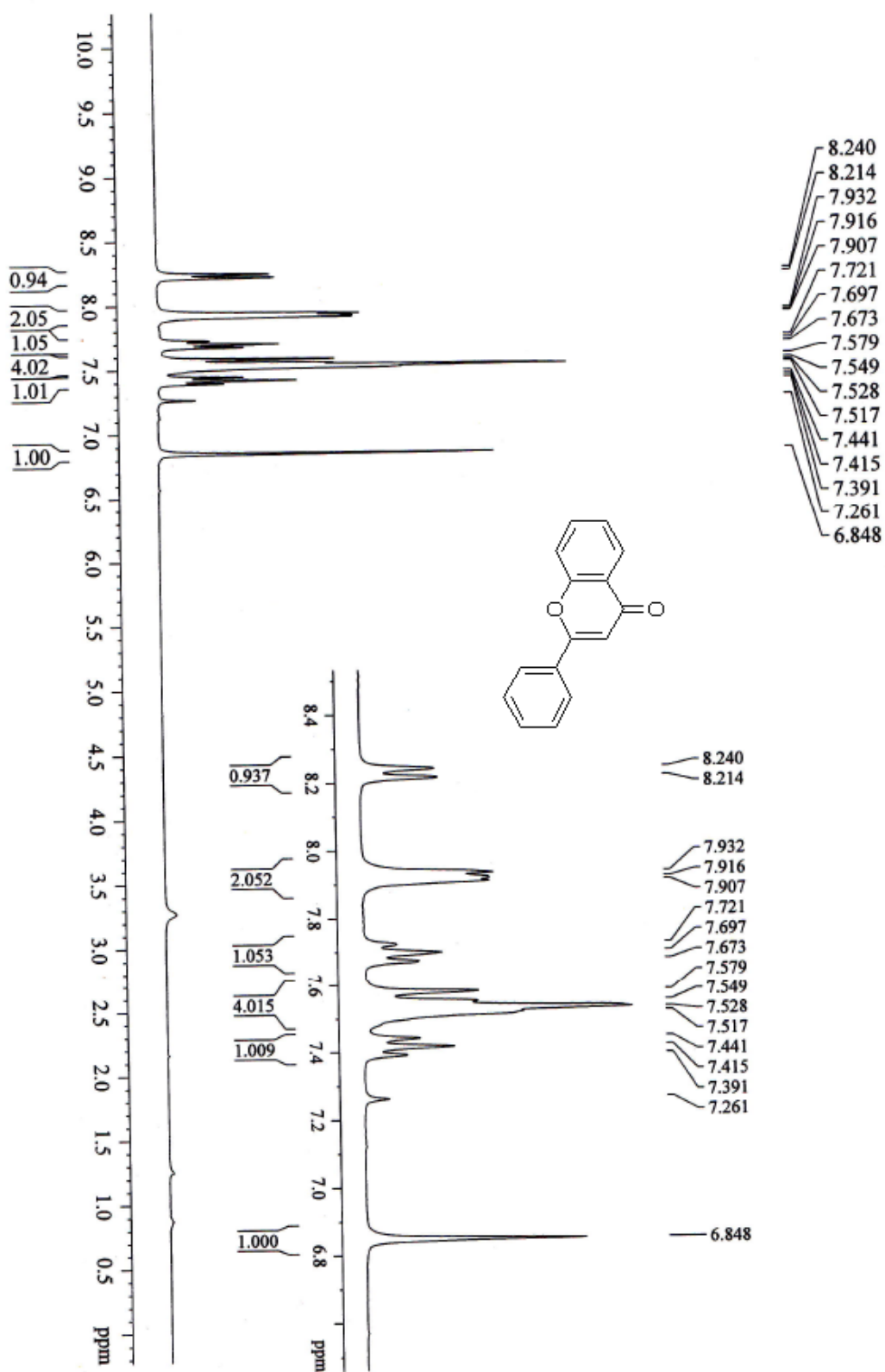


Fig. 26: ¹H NMR spectrum of 2-phenyl chromen-4-one (5d):

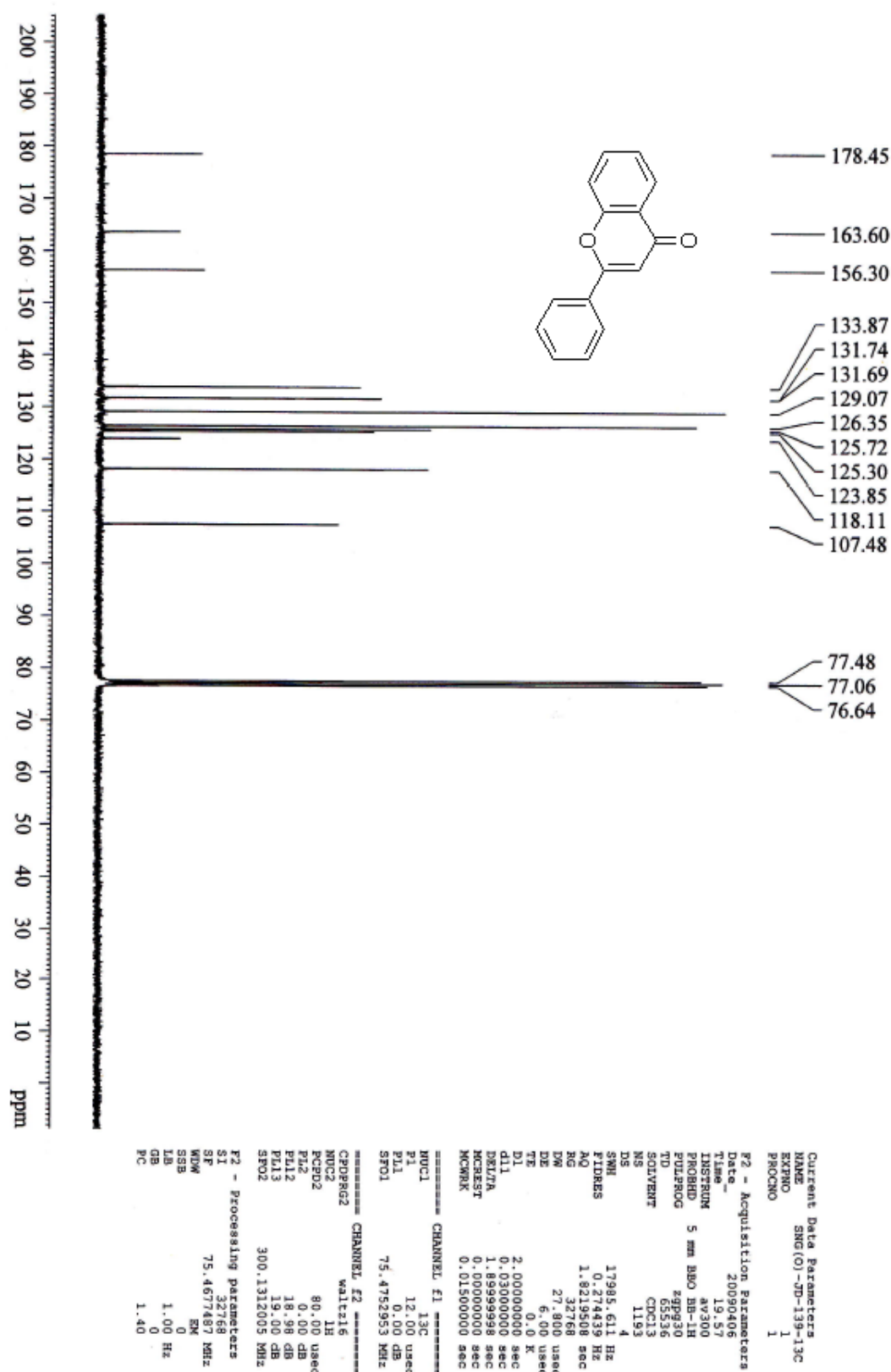


Fig. 27: ^{13}C NMR spectrum of 2-phenyl chromen-4-one (5d)

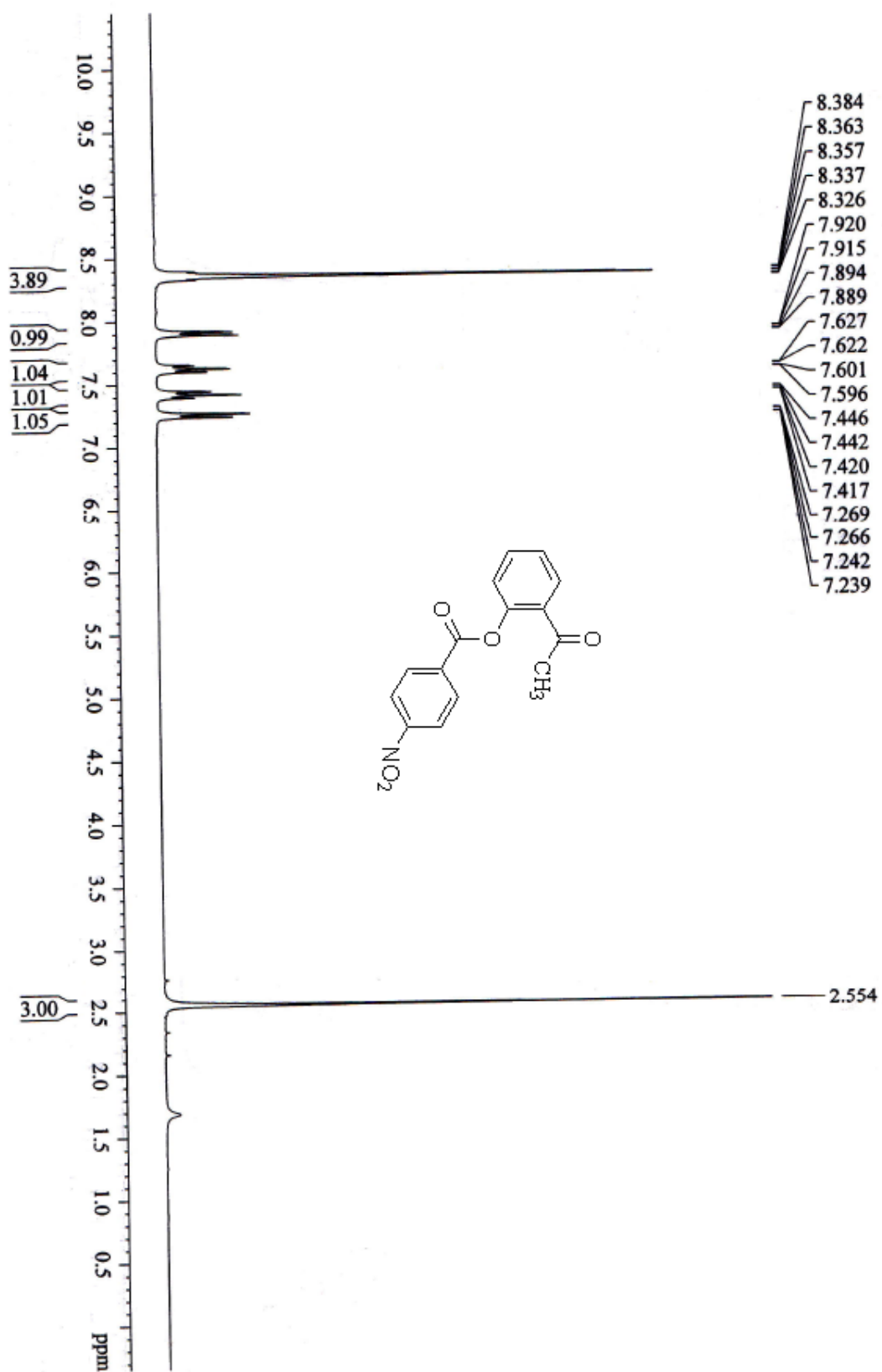
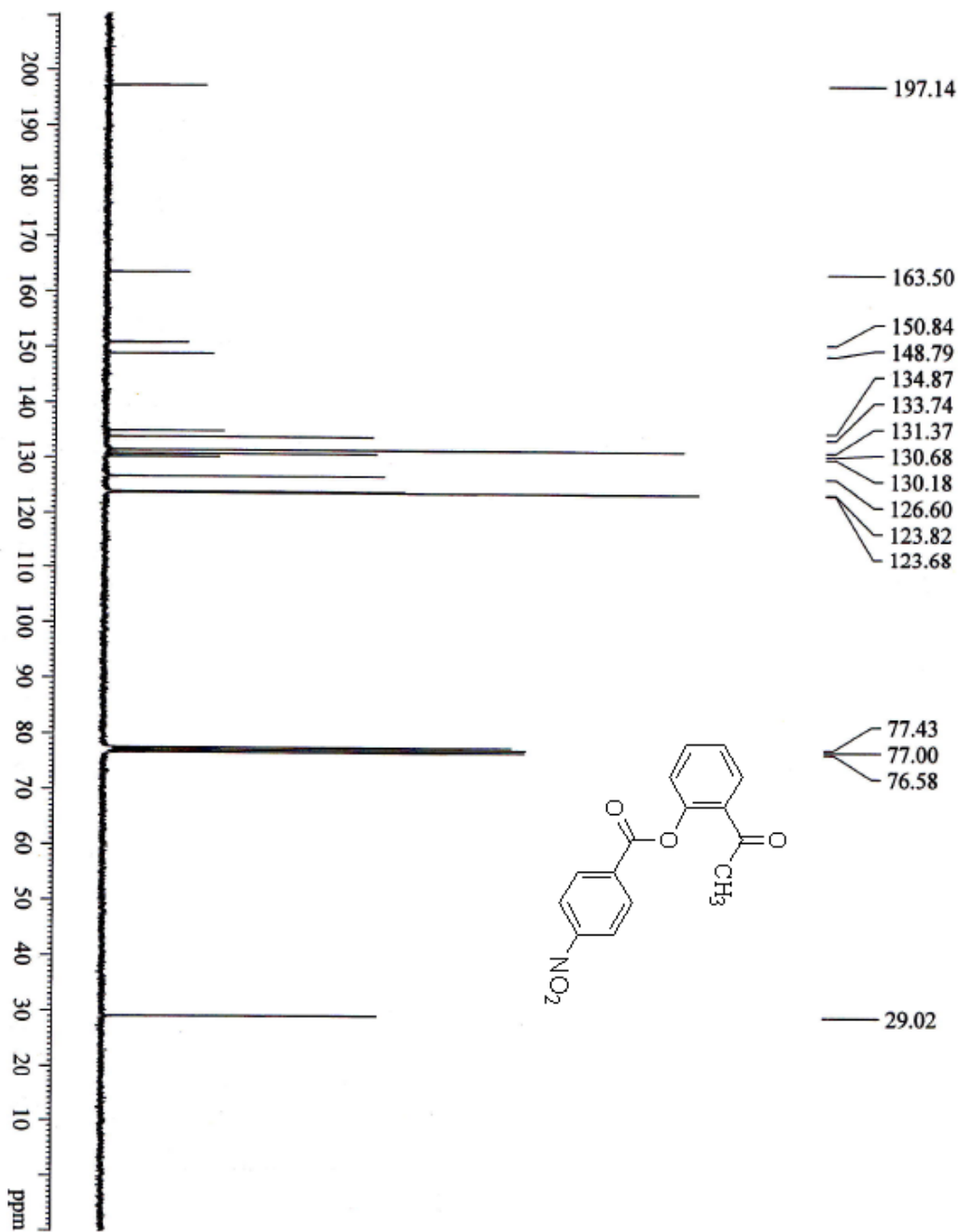


Fig. 28: ¹H NMR spectrum of 1-[2-(4-Nitrobenzoyloxy)-phenyl]-ethanone (2e)



Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 19:48
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 874
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
BW 27.800 usec
DE 6.00 usec
TE 300.2 K
NUC1 13C
P1 12.00 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

CPDPRG2 waltz16
CHANEL F2
NUC2 1H
PCPD2 80.00 usec
PL2 19.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4671517 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Fig. 29: ¹³C NMR spectrum of 1-[2-(4-Nitrobenzoyloxy)-phenyl]-ethanone (2e)

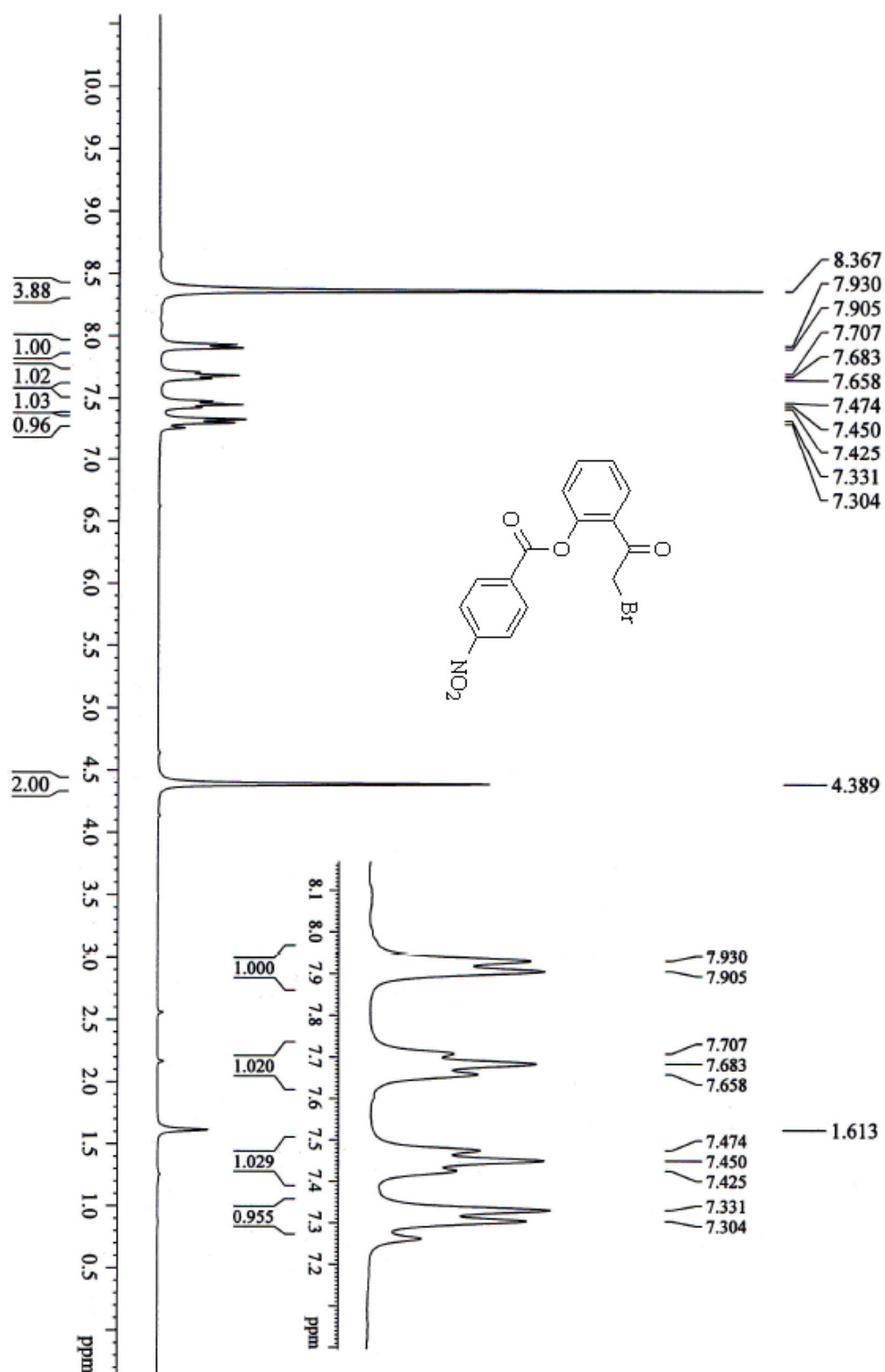


Fig. 30: ¹H NMR spectrum of 1-[2-(4-Nitrobenzoyloxy)-phenyl]-2-bromo ethanone (3e)

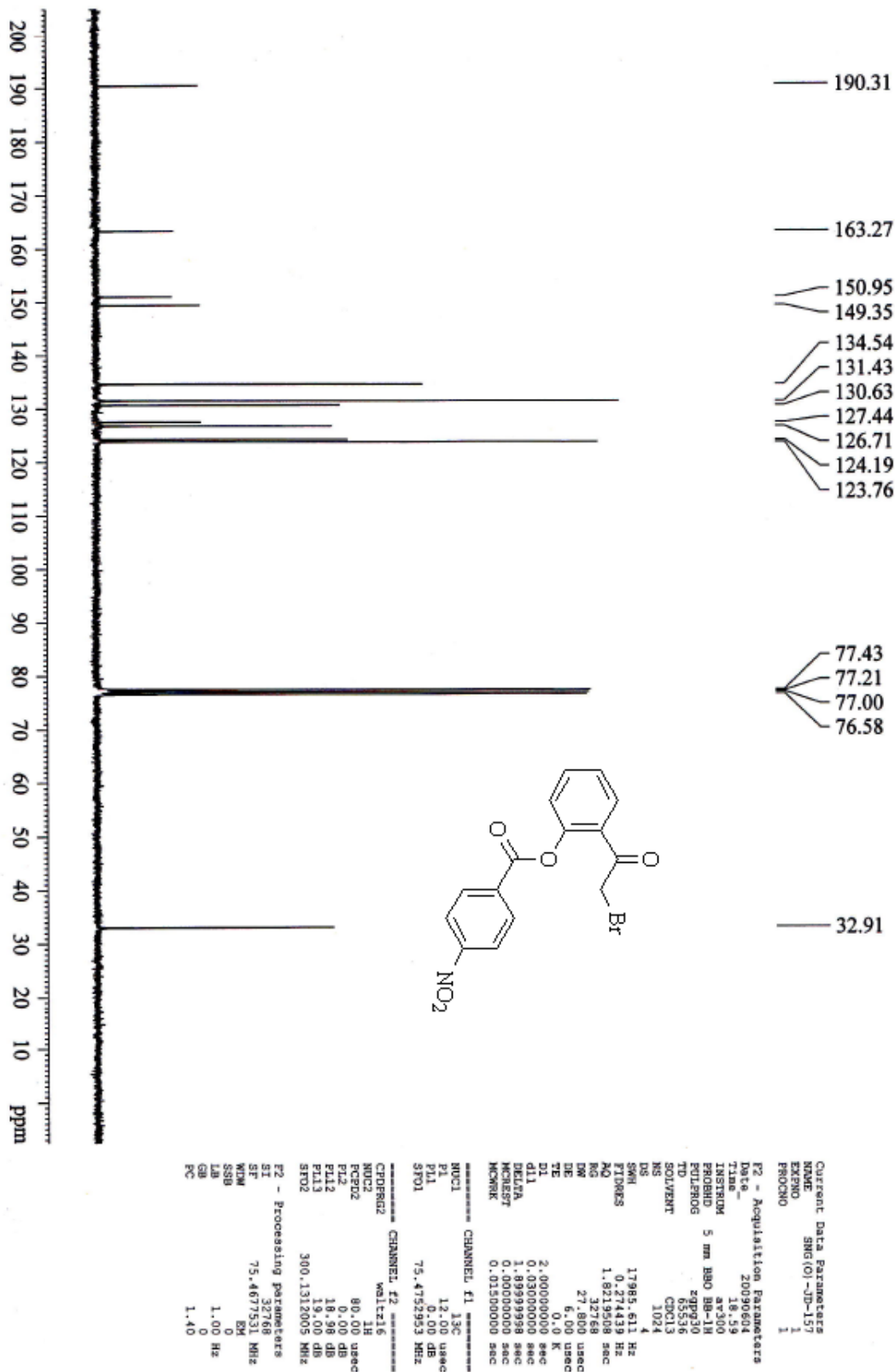


Fig. 31: ¹³C NMR spectrum of 1-[2-(4-Nitrobenzoyloxy)-phenyl]-2-bromo ethanone (3e)

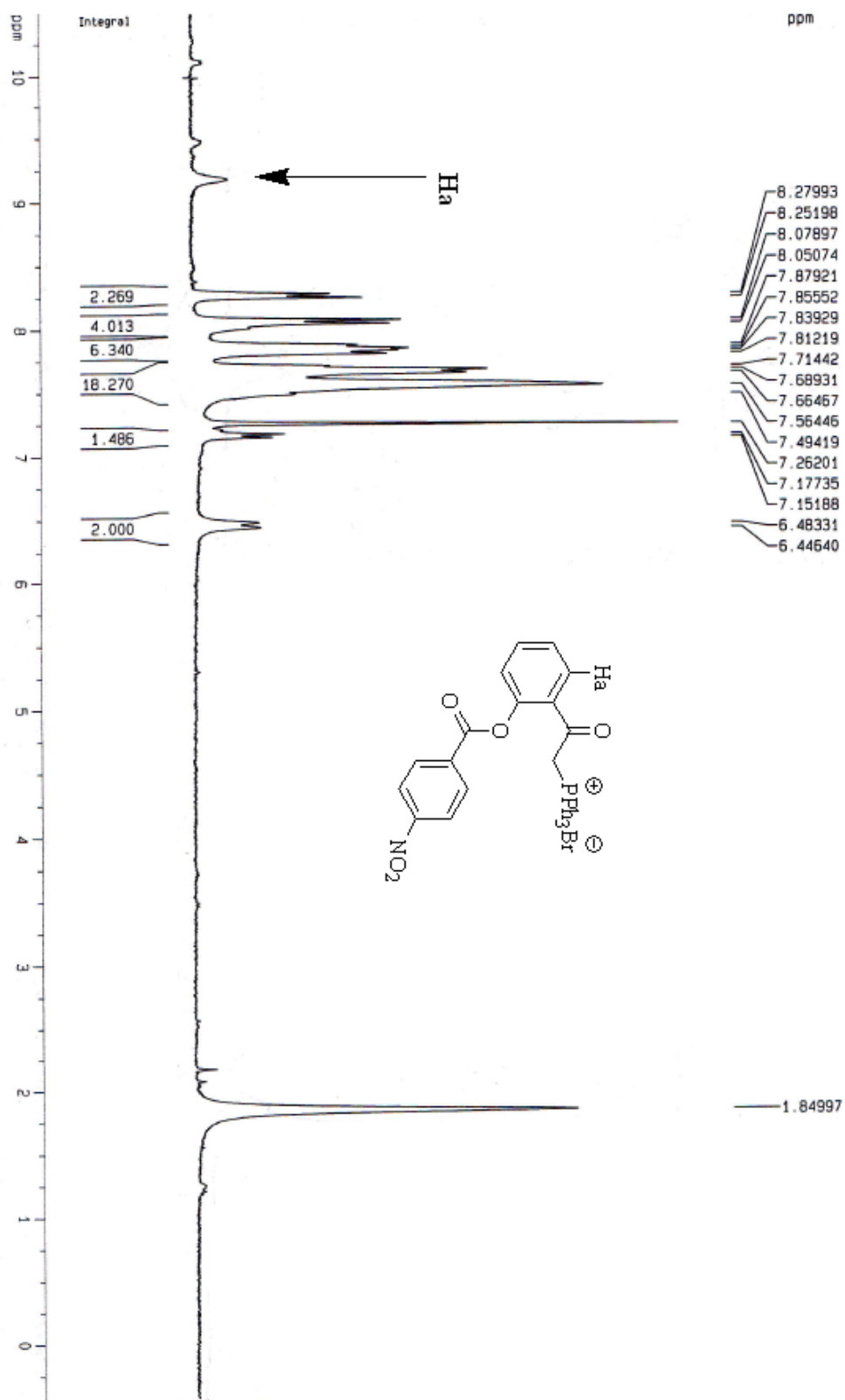


Fig. 32: ^1H NMR spectrum of 2-(4-Nitrobenzoyloxy)-phenyl)-benzoyl methyl triphenyl phosphonium bromide (4e)

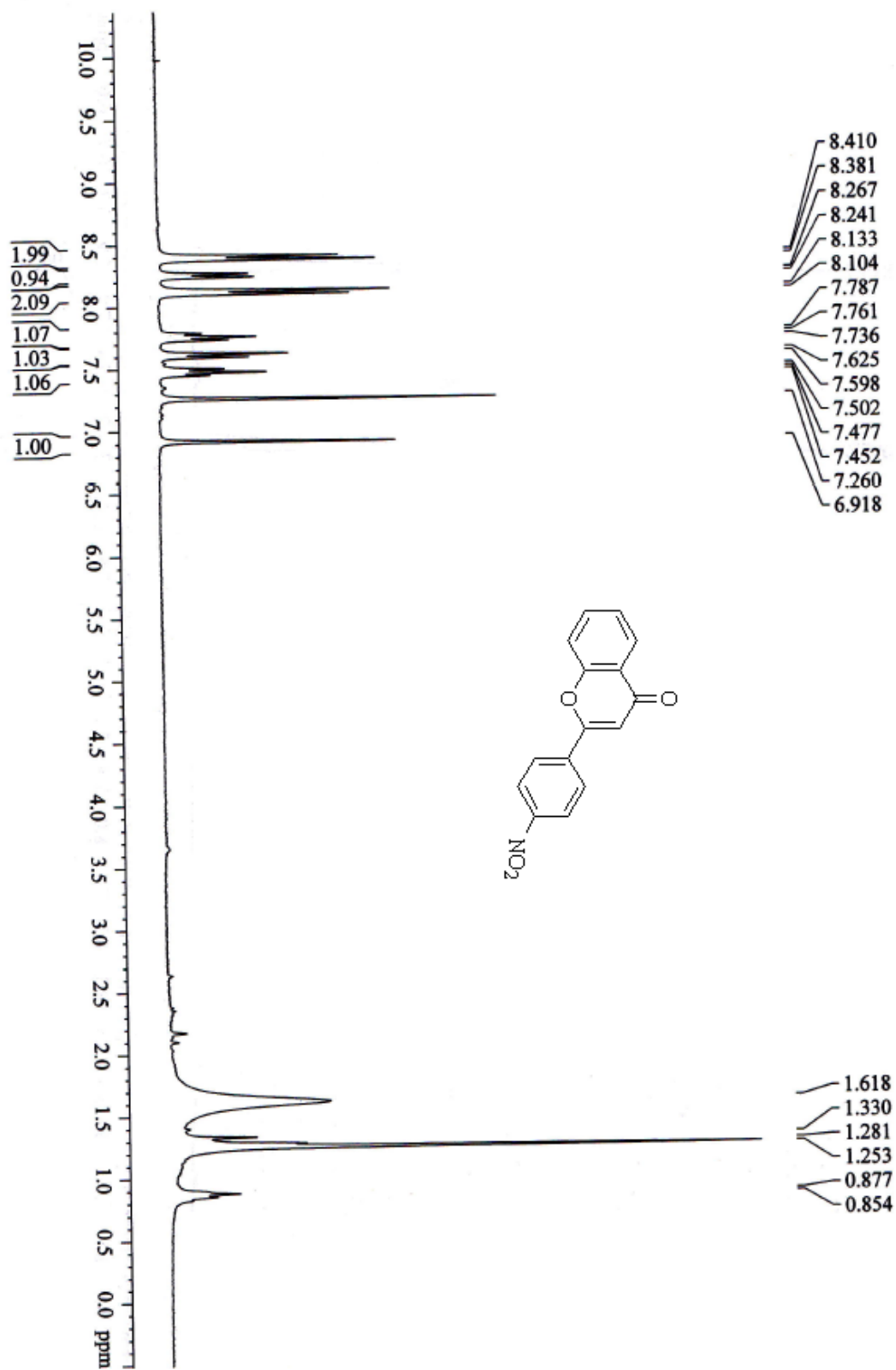


Fig. 33: ¹H NMR spectrum of 2-(4-Nitro phenyl)-chromen-4-one (5e)

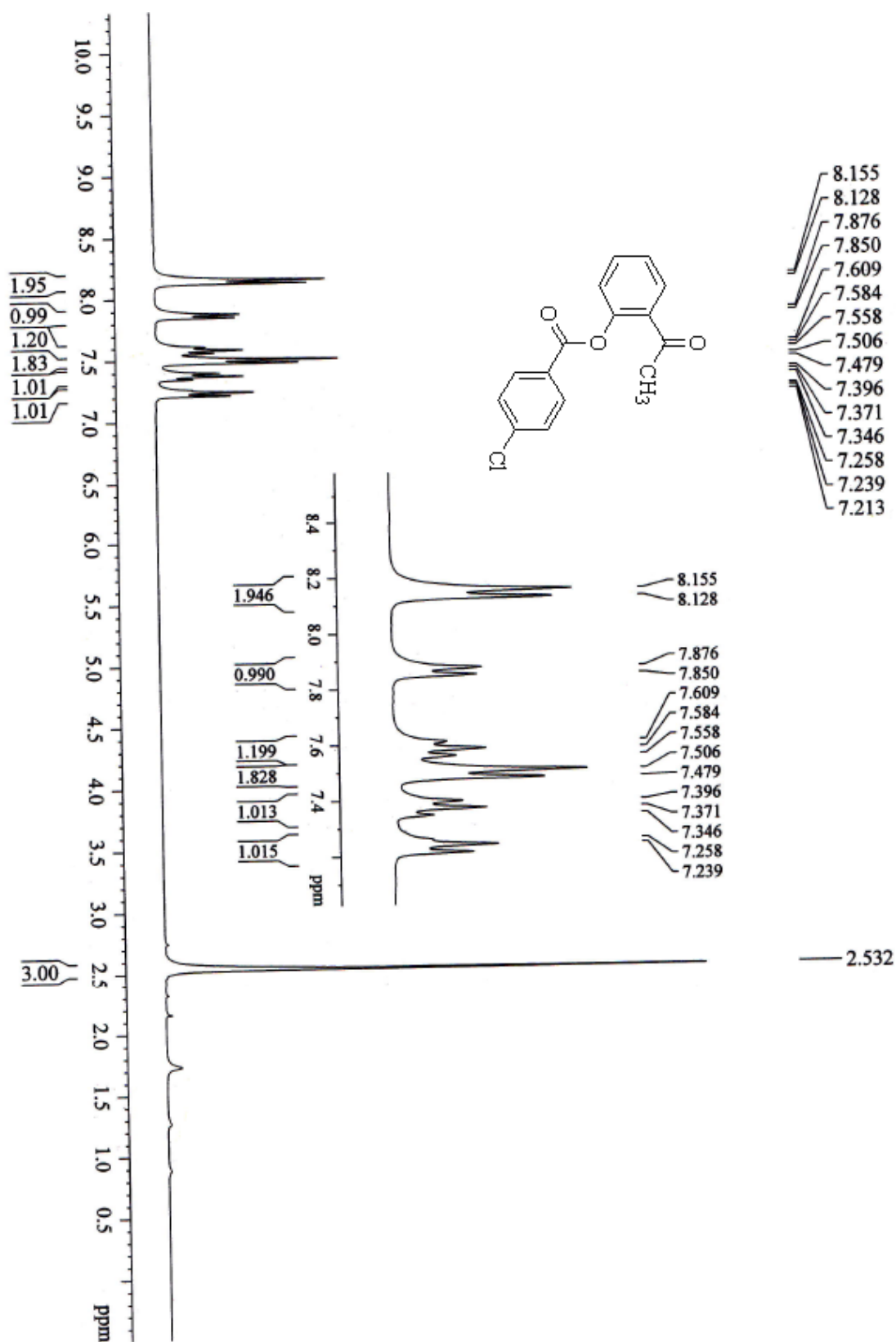
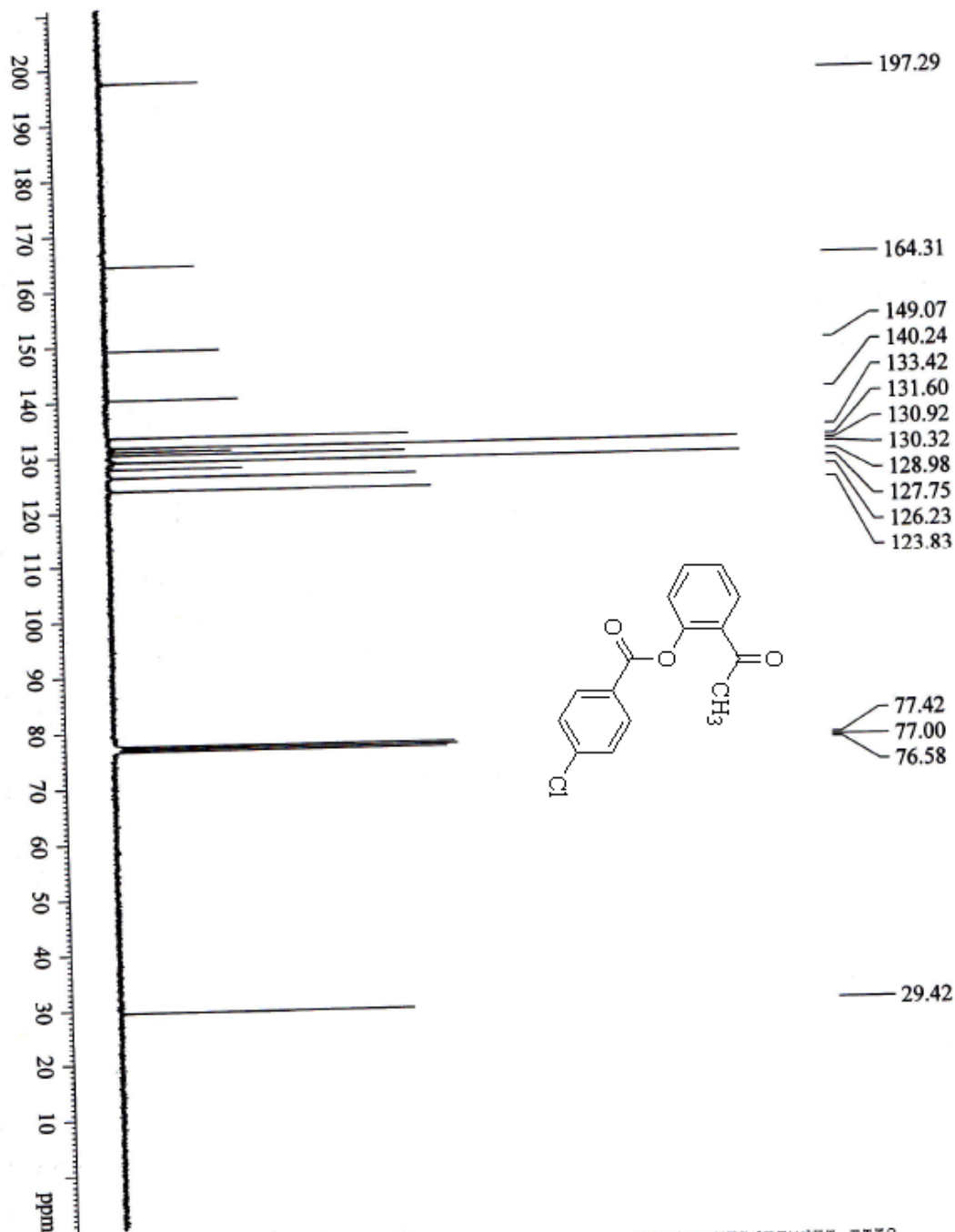


Fig. 34: ¹H NMR spectrum of 1-[2-(4-Chlorobenzoyloxy)-phenyl]-ethanone (2f)



Current Data Parameters
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PROCNO 1
F2 - Acquisition Parameters
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Time 15:18
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DM 27.800 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
WDELT 0.00000000 sec
WDELT 0.03000000 sec
MCWMT 0.03000000 sec
MCWMT 0.03000000 sec
CHANNEL F1
NUC1 13C
P1 12.00 usec
PL 0.00 dB
SFO1 75.4732953 MHz
===== CHANNEL F2 =====
CPDPRG2 mltc16
NUC2 1H
PCPD2 0.00 usec
PL2 0.00 dB
PL12 18.50 dB
PL13 19.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.467758 MHz
RG 0
WDW 0
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Fig. 35: ¹³C NMR spectrum of 1-[2-(4-Chlorobenzoyloxy)-phenyl]-ethanone (2f)

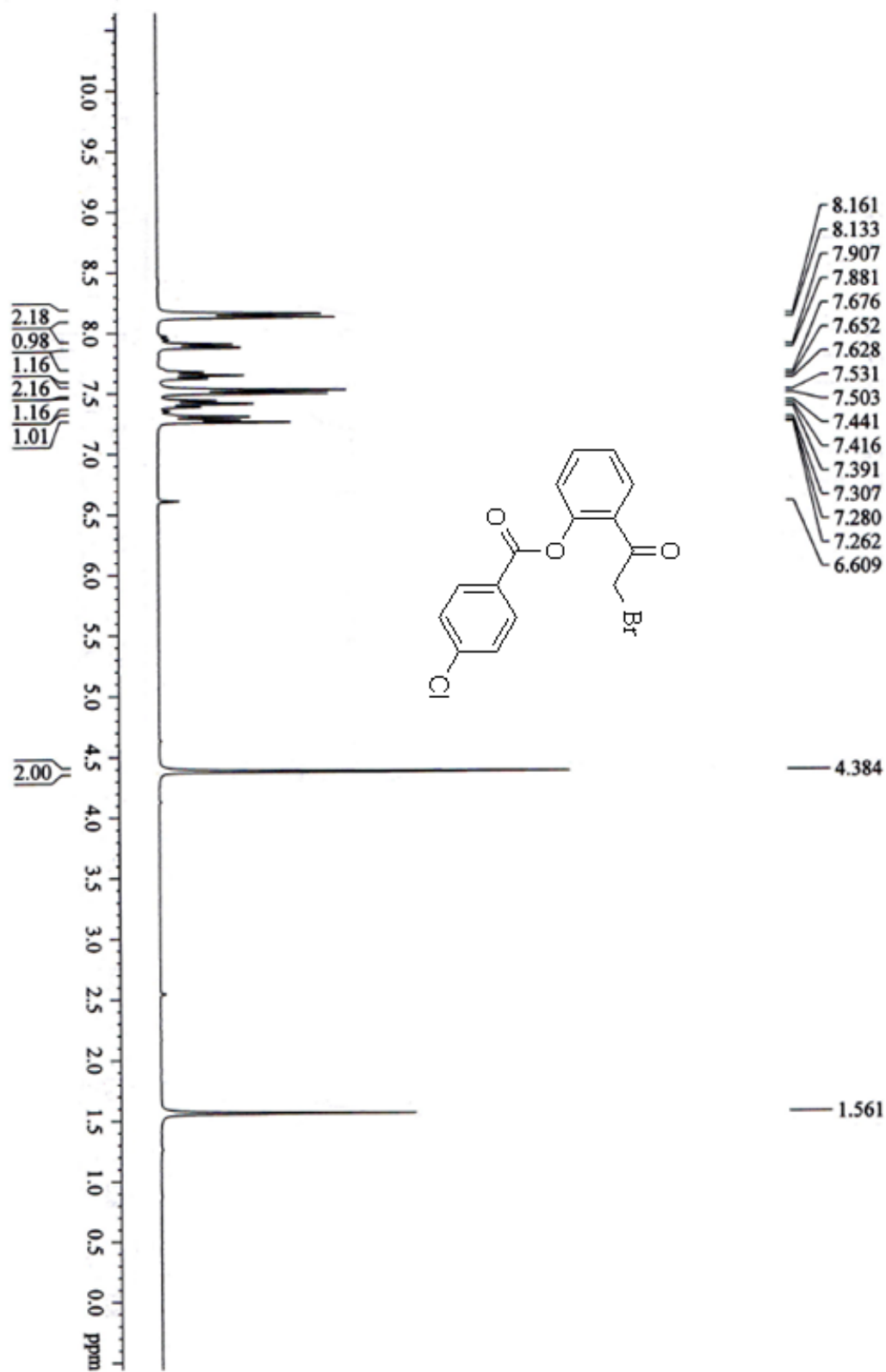


Fig. 36: ¹H NMR spectrum of 1-[2-(4-Chlorobenzoyloxy)-phenyl]-2-bromo ethanone (3f)

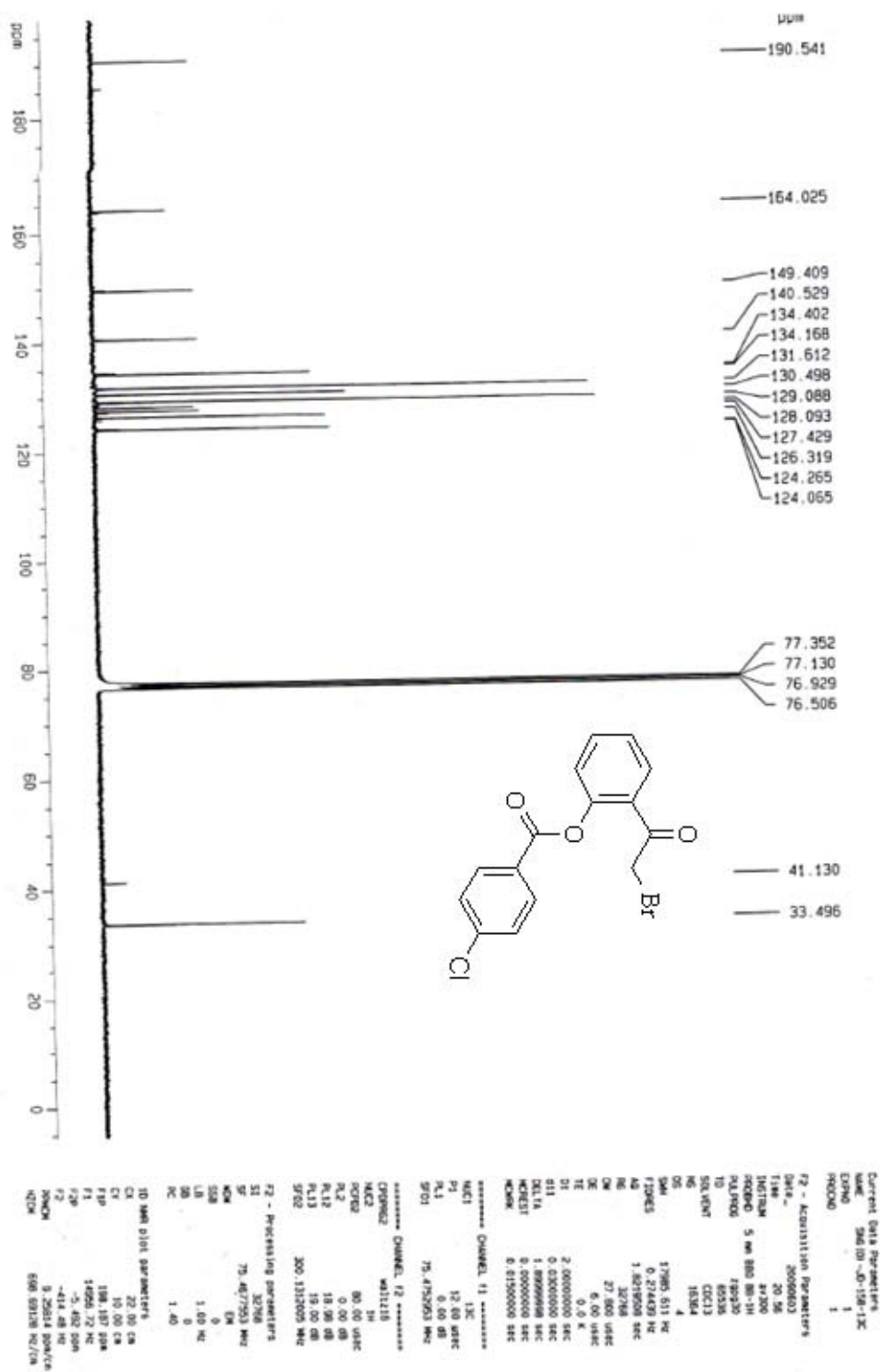


Fig. 37: ^{13}C NMR spectrum of 1-[2-(4-Chlorobenzoyloxy)-phenyl]-2-bromo ethanone (3f)

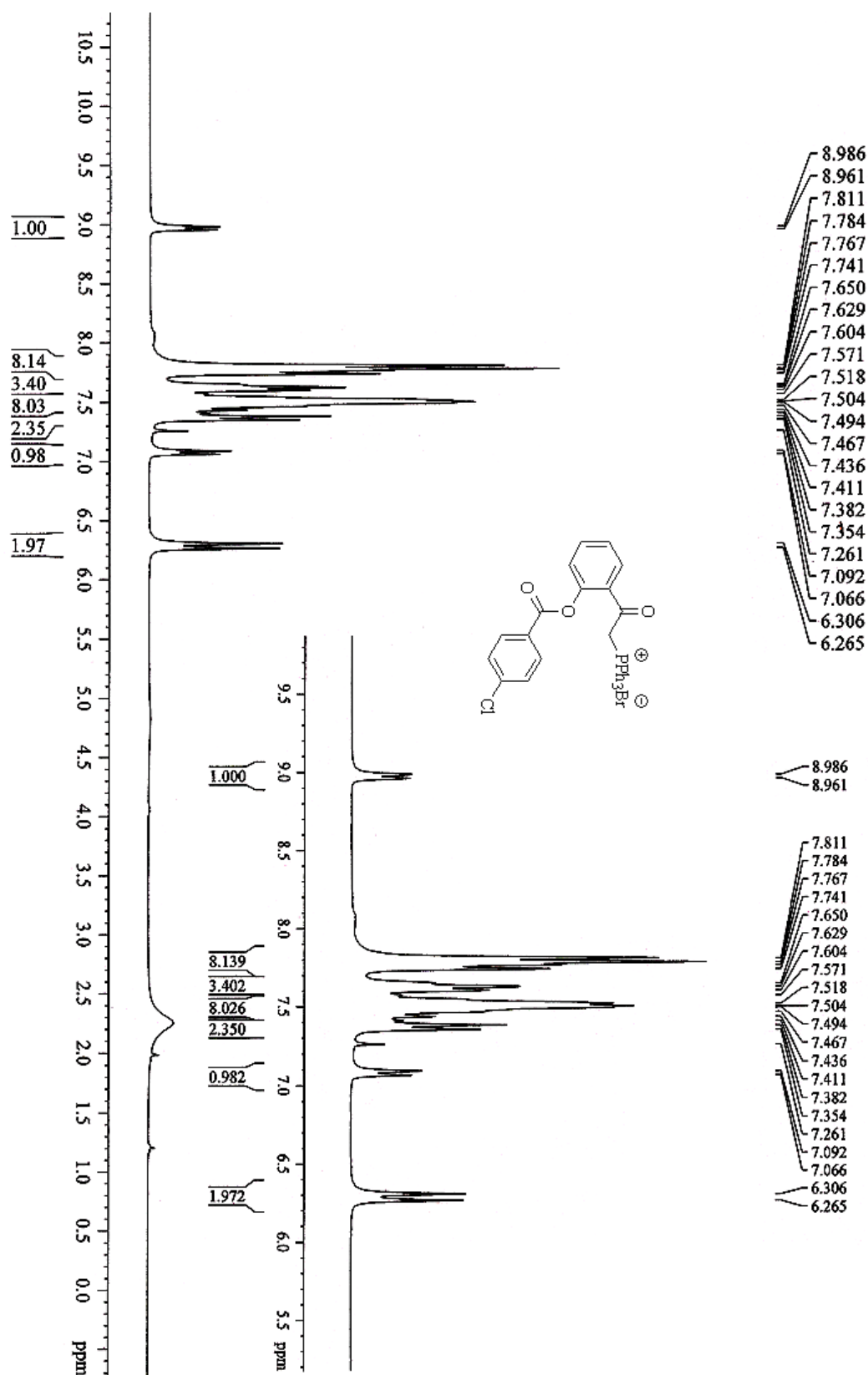


Fig. 38: ¹H NMR spectrum of 2-(4-Chlorobenzoyloxy)-phenyl)-benzoyl methyl triphenyl phosphonium bromide (4f)

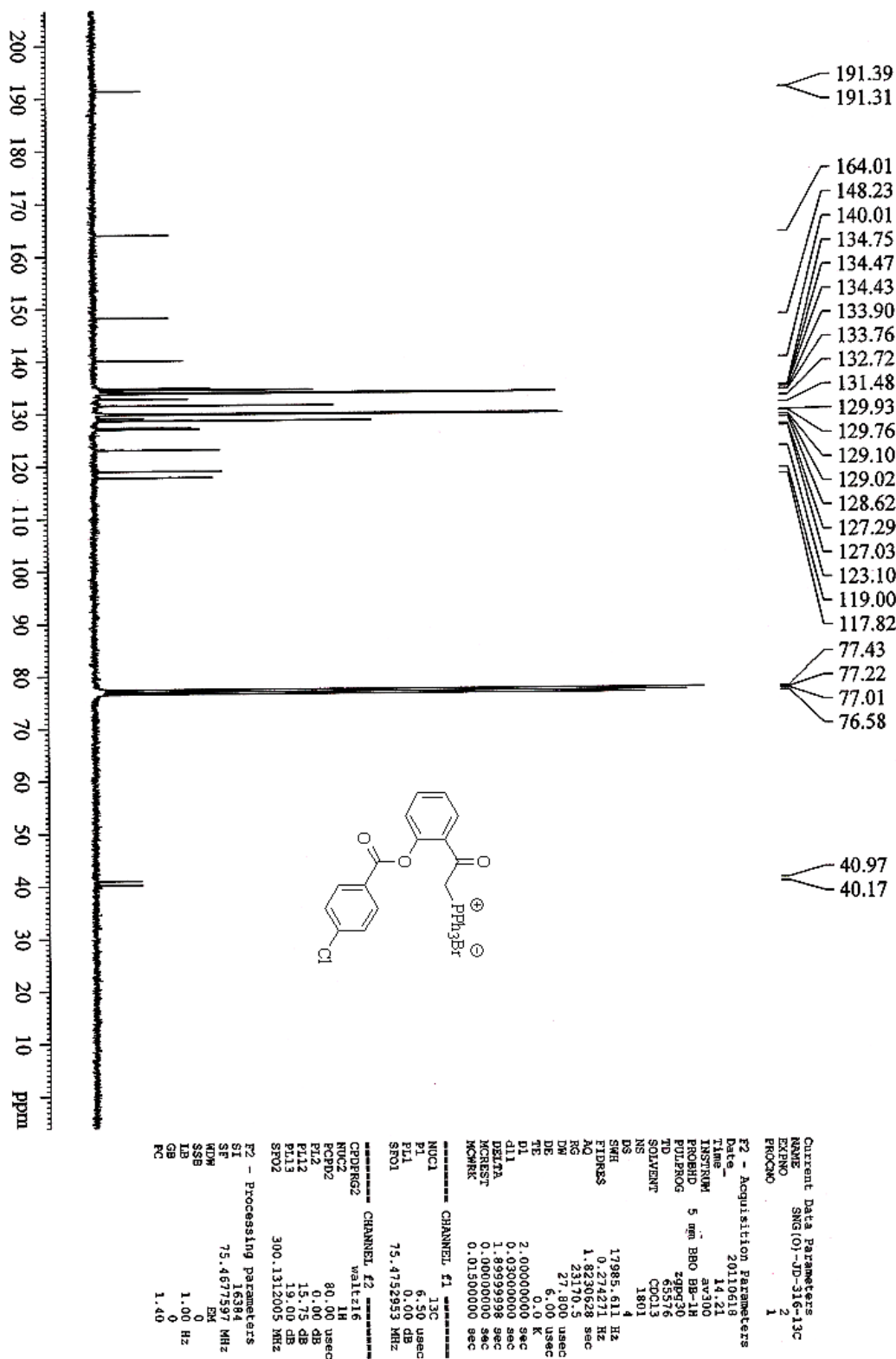


Fig. 39: ¹³C NMR spectrum of 2-(4-Chlorobenzoyloxy)-phenyl)-benzoyl methyl triphenyl phosphonium bromide (4f)

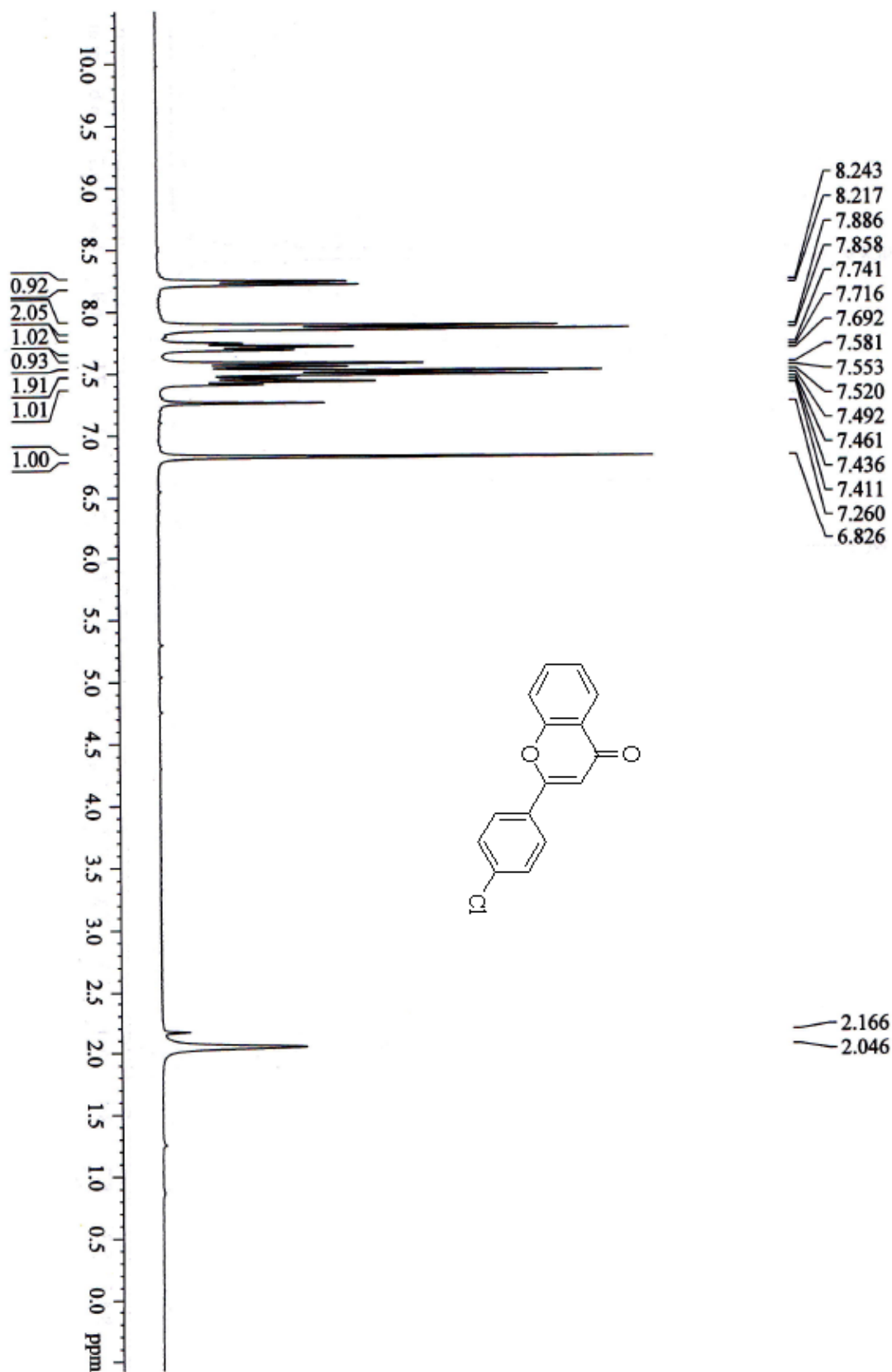


Fig. 40: ¹H NMR spectrum of 2-(4-Chlorophenyl)-chromen-4-one (5f)

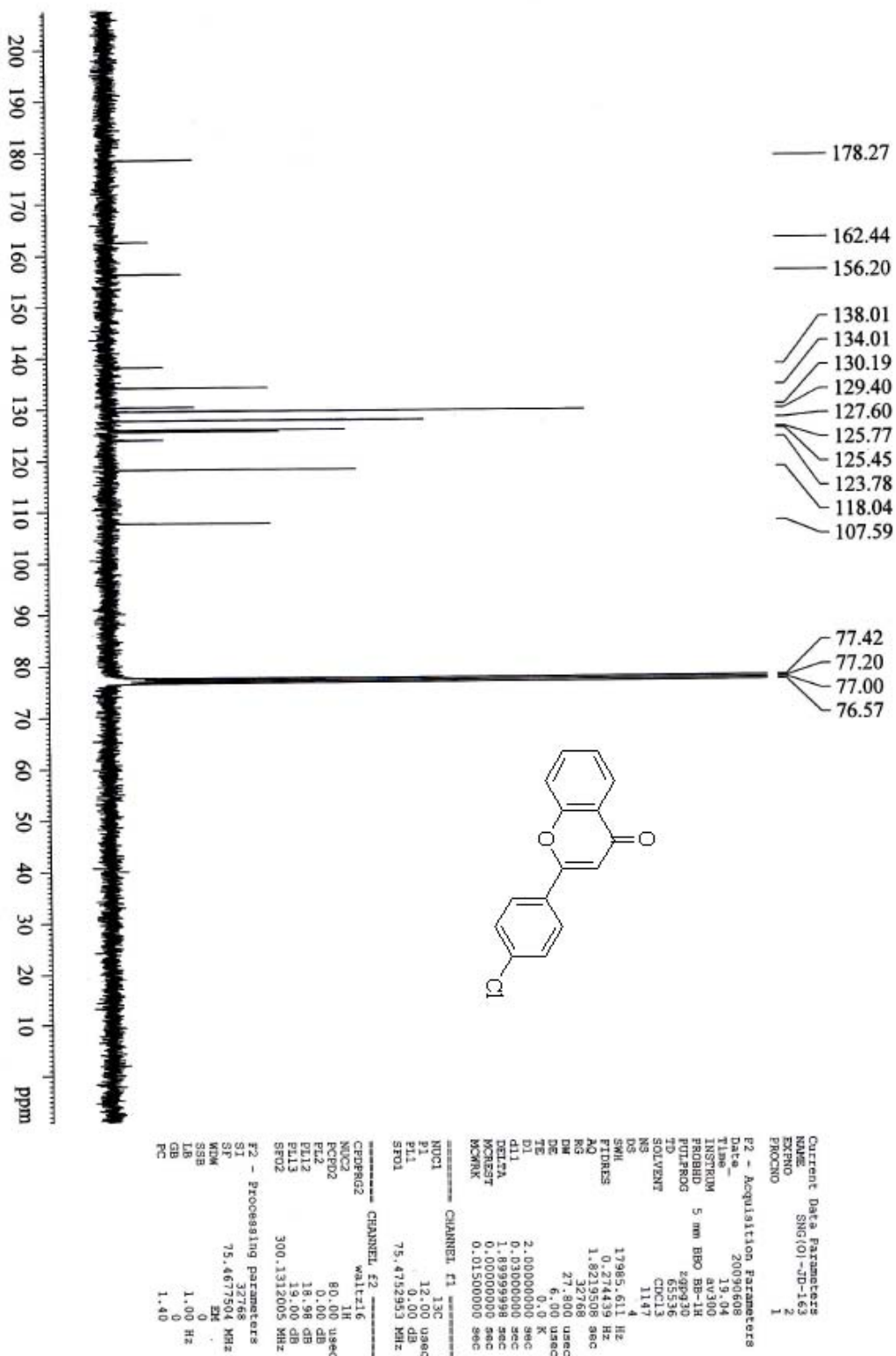


Fig. 41: ^{13}C NMR spectrum of 2-(4-Chlorophenyl)-chromen-4-one (5f)

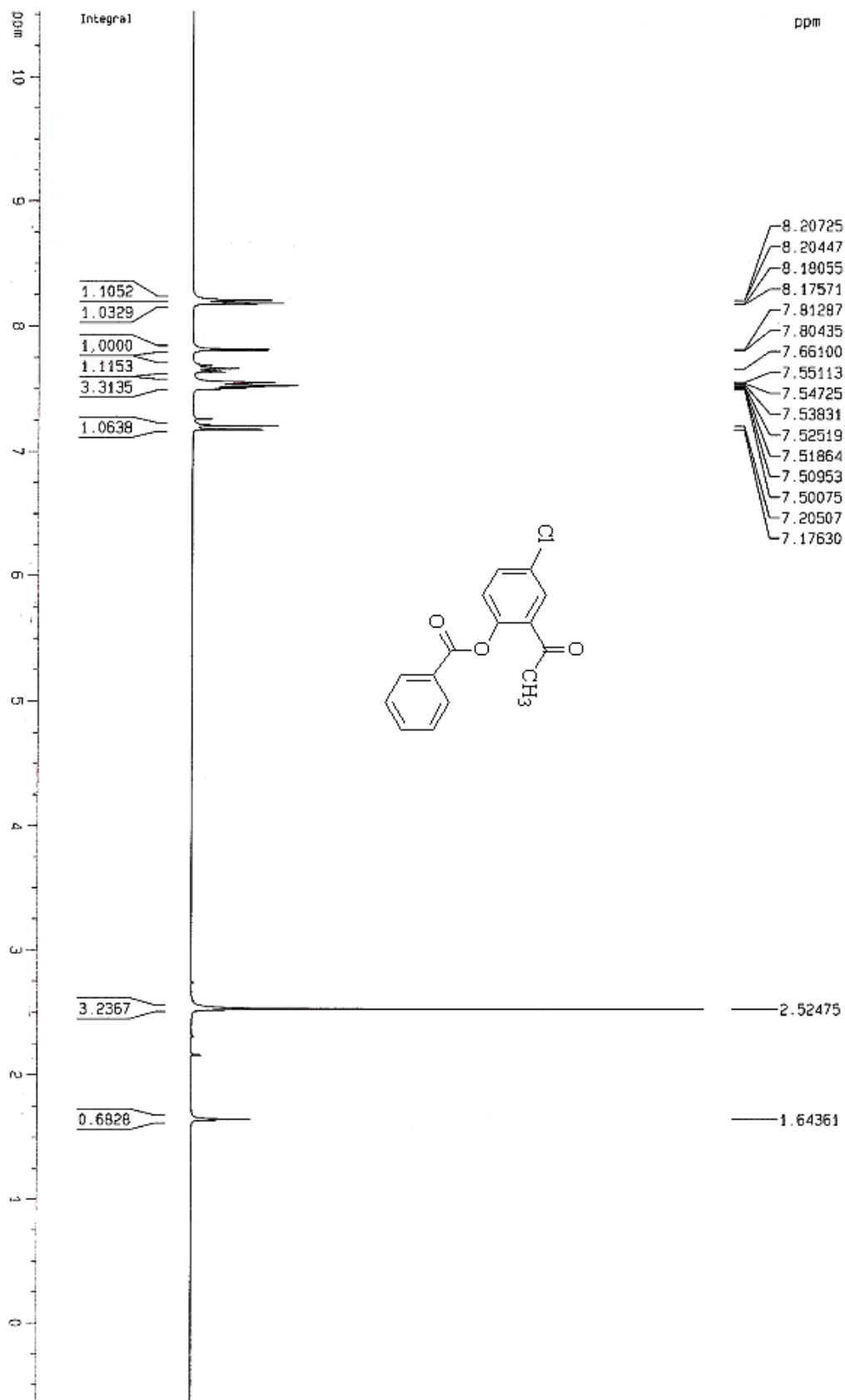


Fig. 42: ¹H NMR spectrum of 1-(2-Benzoyloxy-5-chlorophenyl)-ethanone (2g)

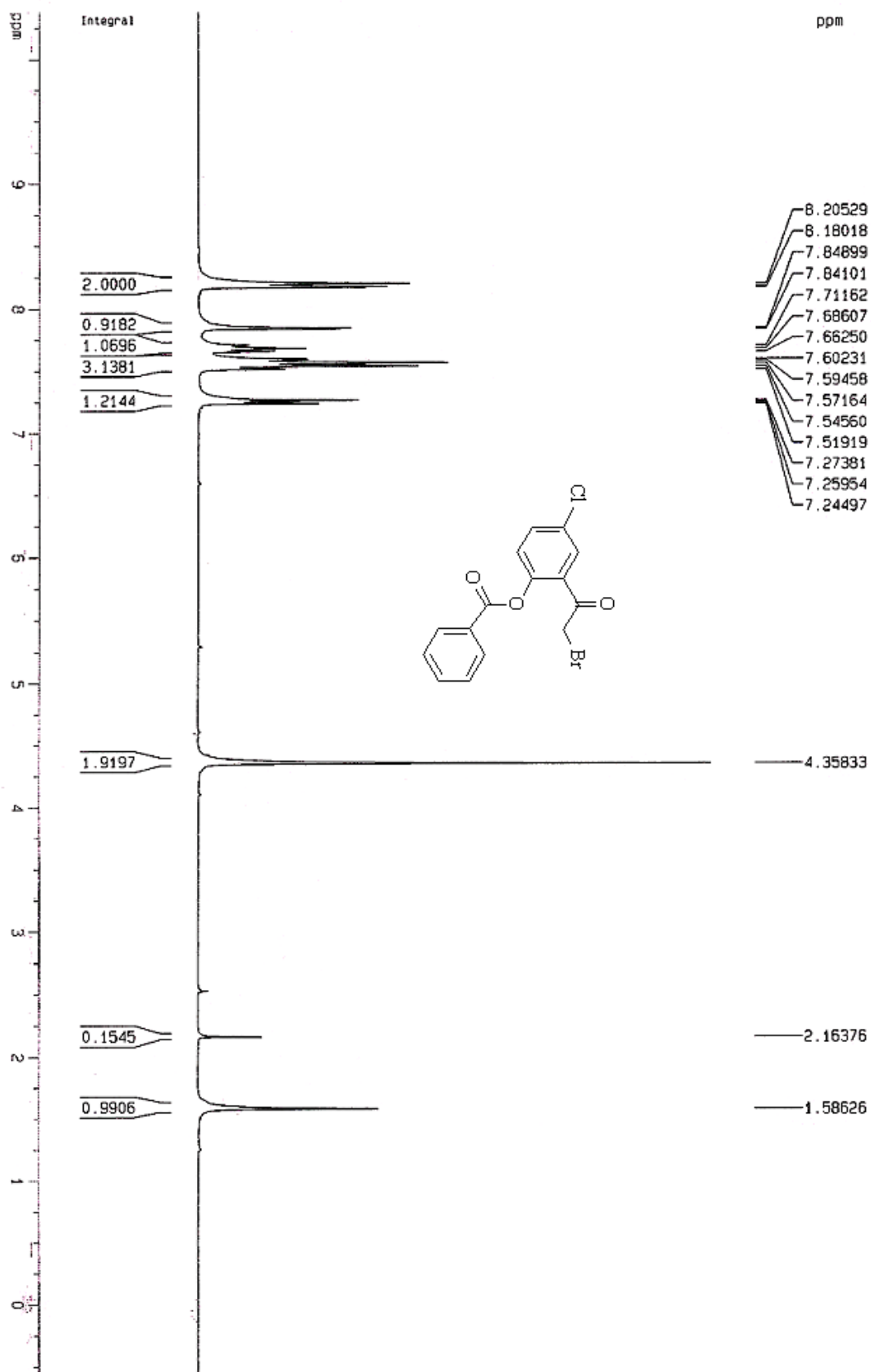


Fig. 43: ^1H NMR spectrum of 1-(2-Benzoyloxy-5-chlorophenyl)-2-bromo ethanone (3g)

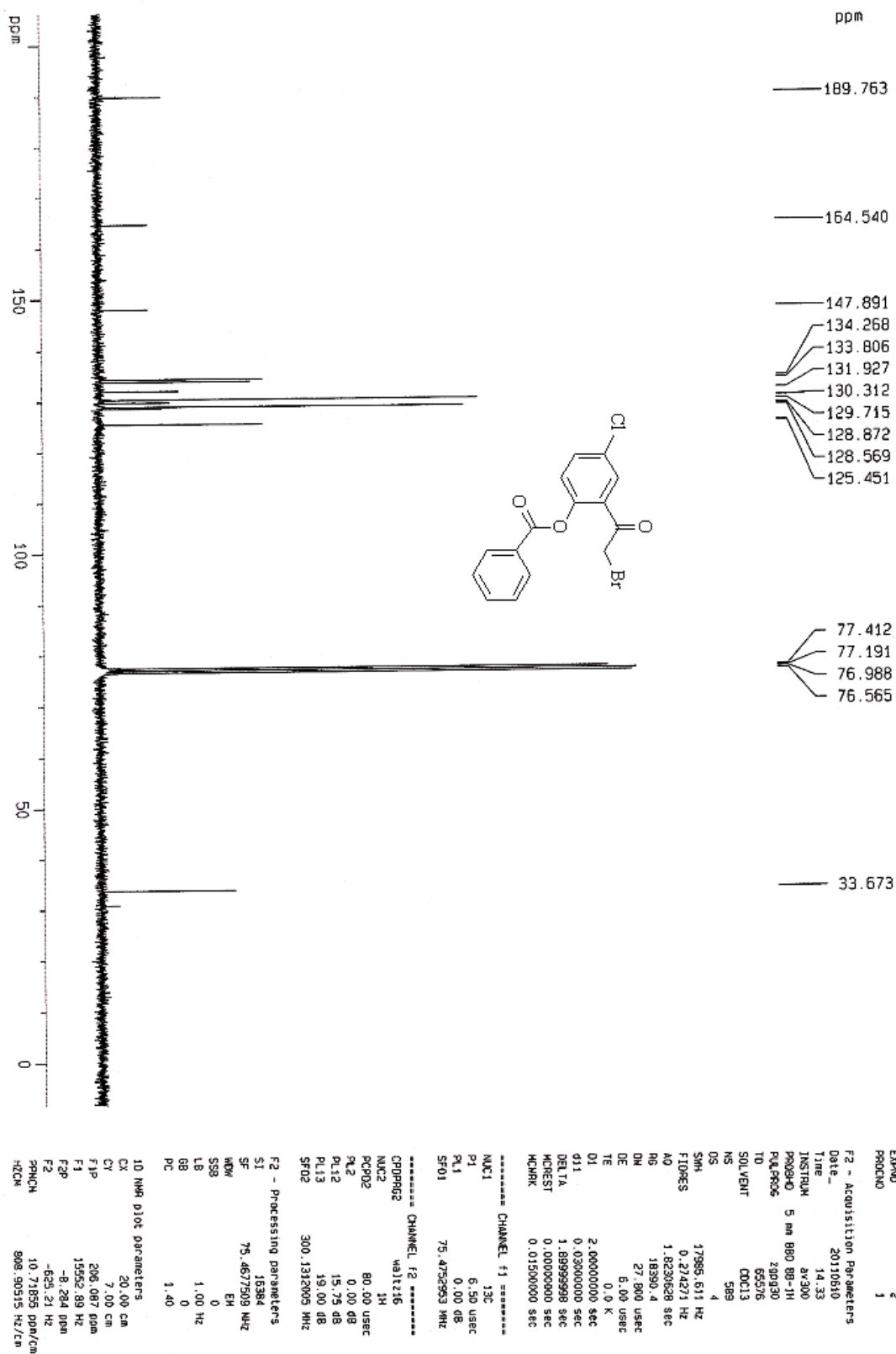


Fig. 44: ^{13}C NMR spectrum of 1-(2-Benzoyloxy-5-chlorophenyl)-2-bromo ethanone (3g)

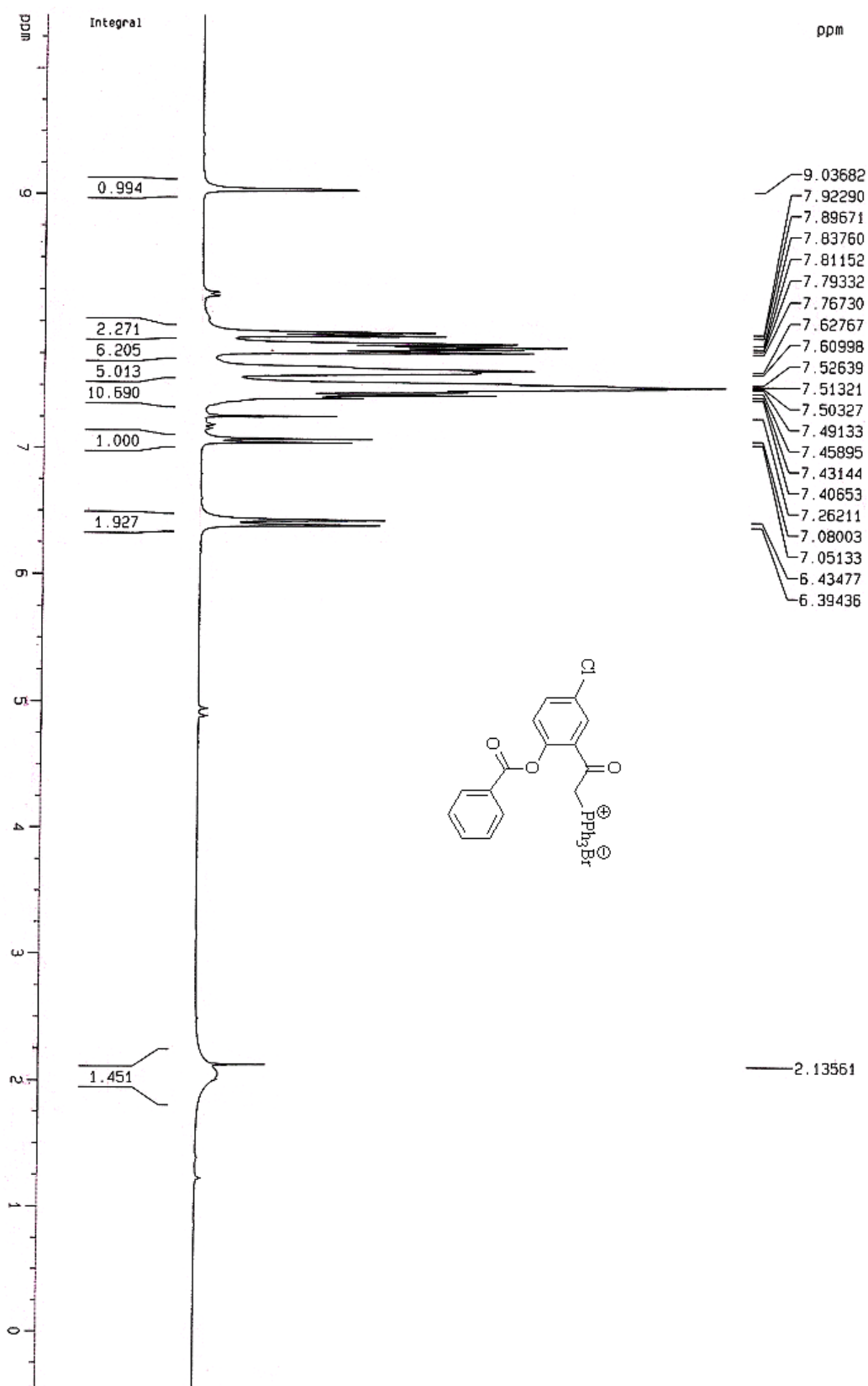


Fig. 45: ^1H NMR spectrum of 2-Benzoyloxy-5-chloro-benzoyl methyl triphenyl phosphonium bromide (4g)

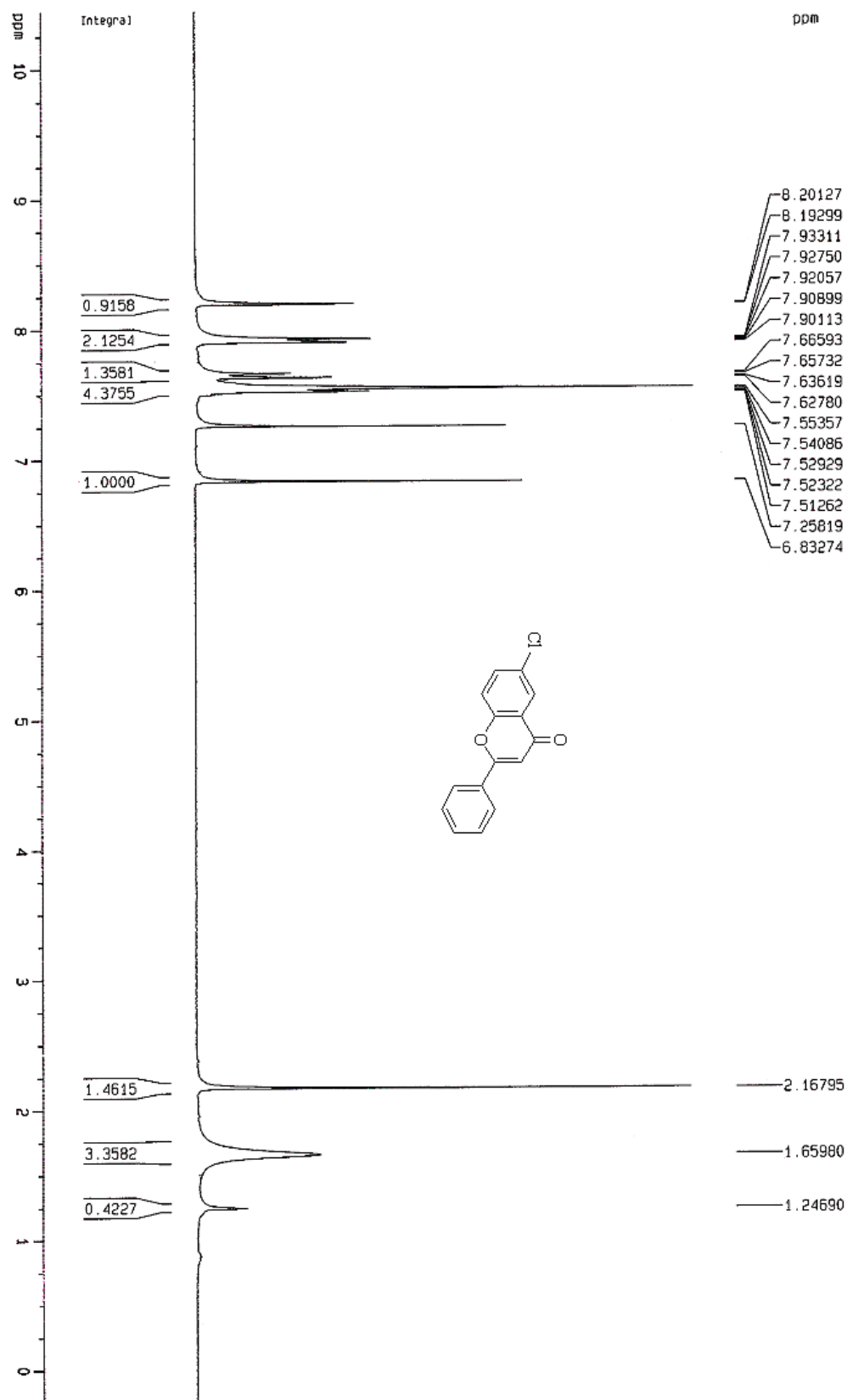


Fig. 46: ^1H NMR spectrum of 6-Chloro-2-phenyl chromen-4-one (5g)

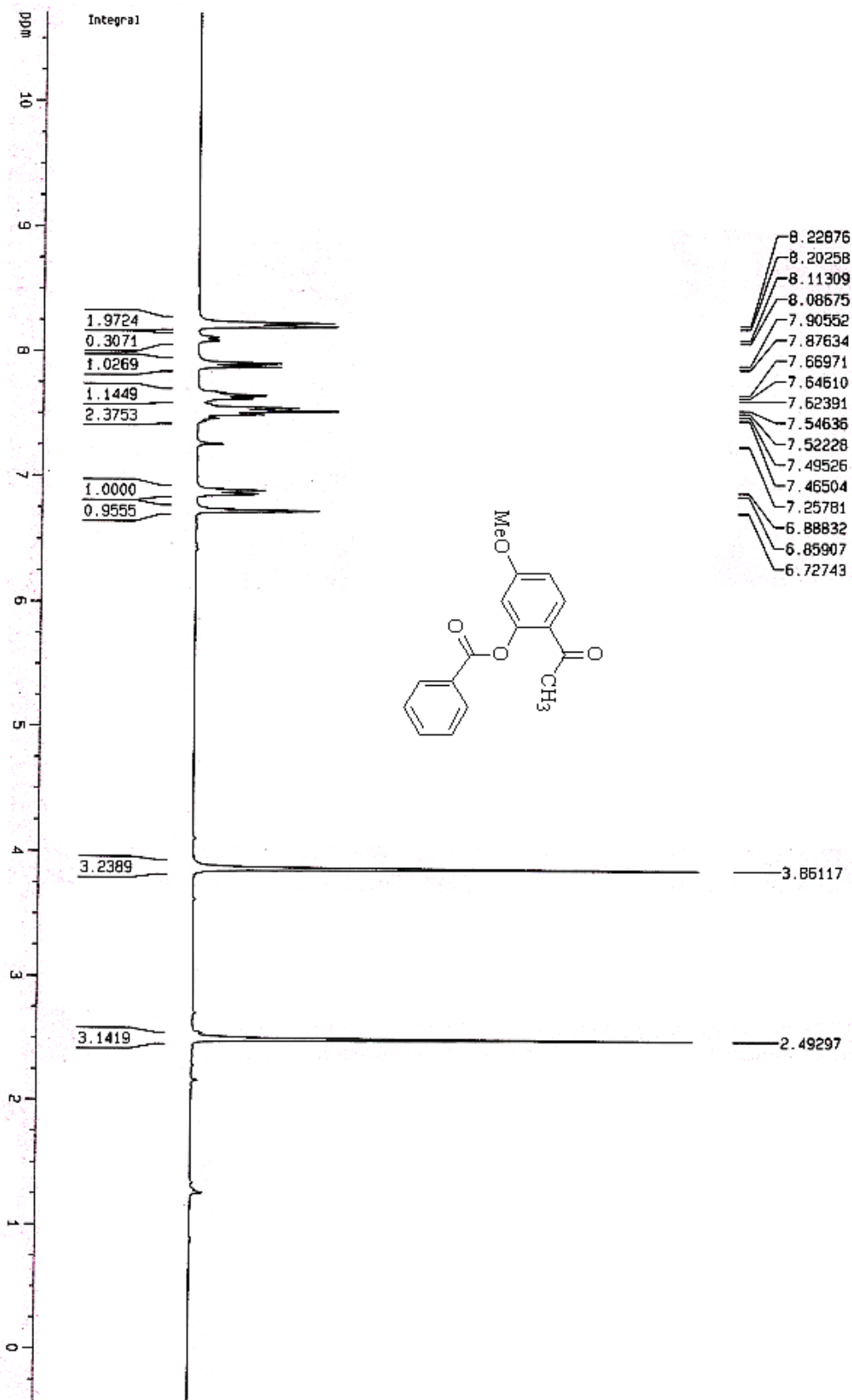


Fig. 48: ¹H NMR spectrum of 1-(2-Benzoyloxy-5-methoxyphenyl)-ethanone (2h)

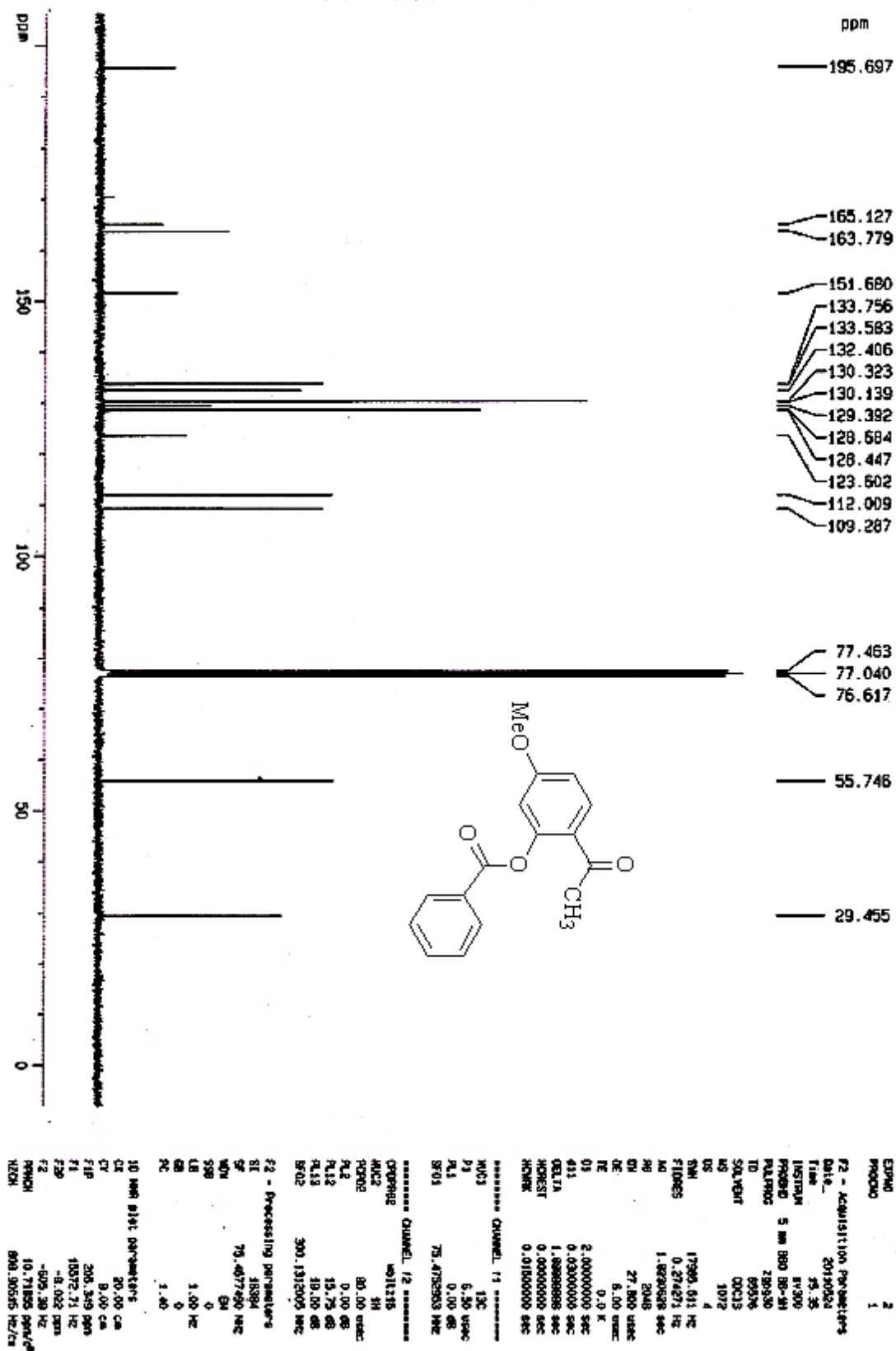


Fig. 49: ¹³C NMR spectrum of 1-(2-Benzoyloxy-5-methoxyphenyl)-ethanone (2h)

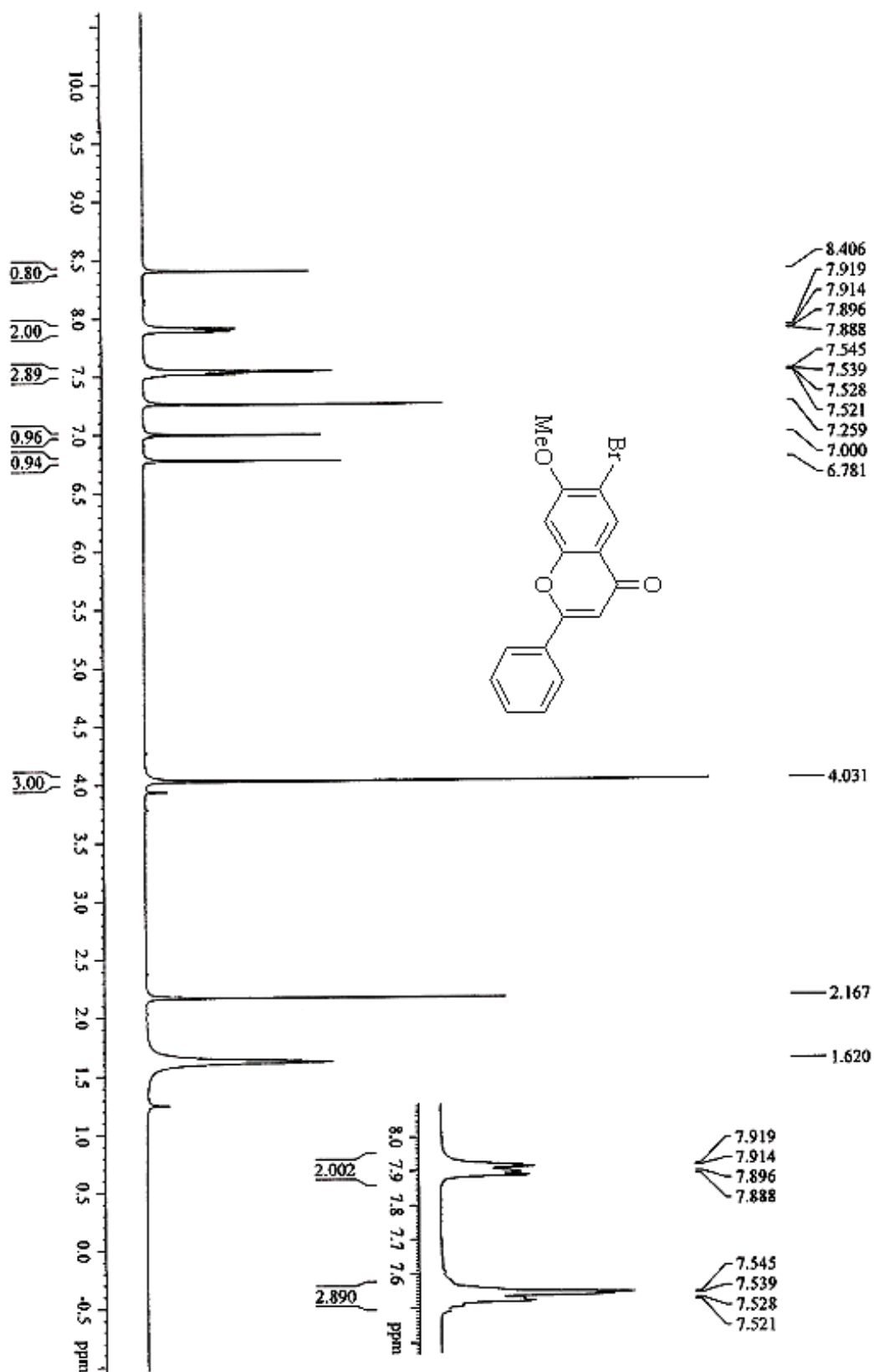


Fig. 50: ¹H NMR spectrum of 6-Bromo-7-methoxy-2-phenyl-chromene-4-one (5h)

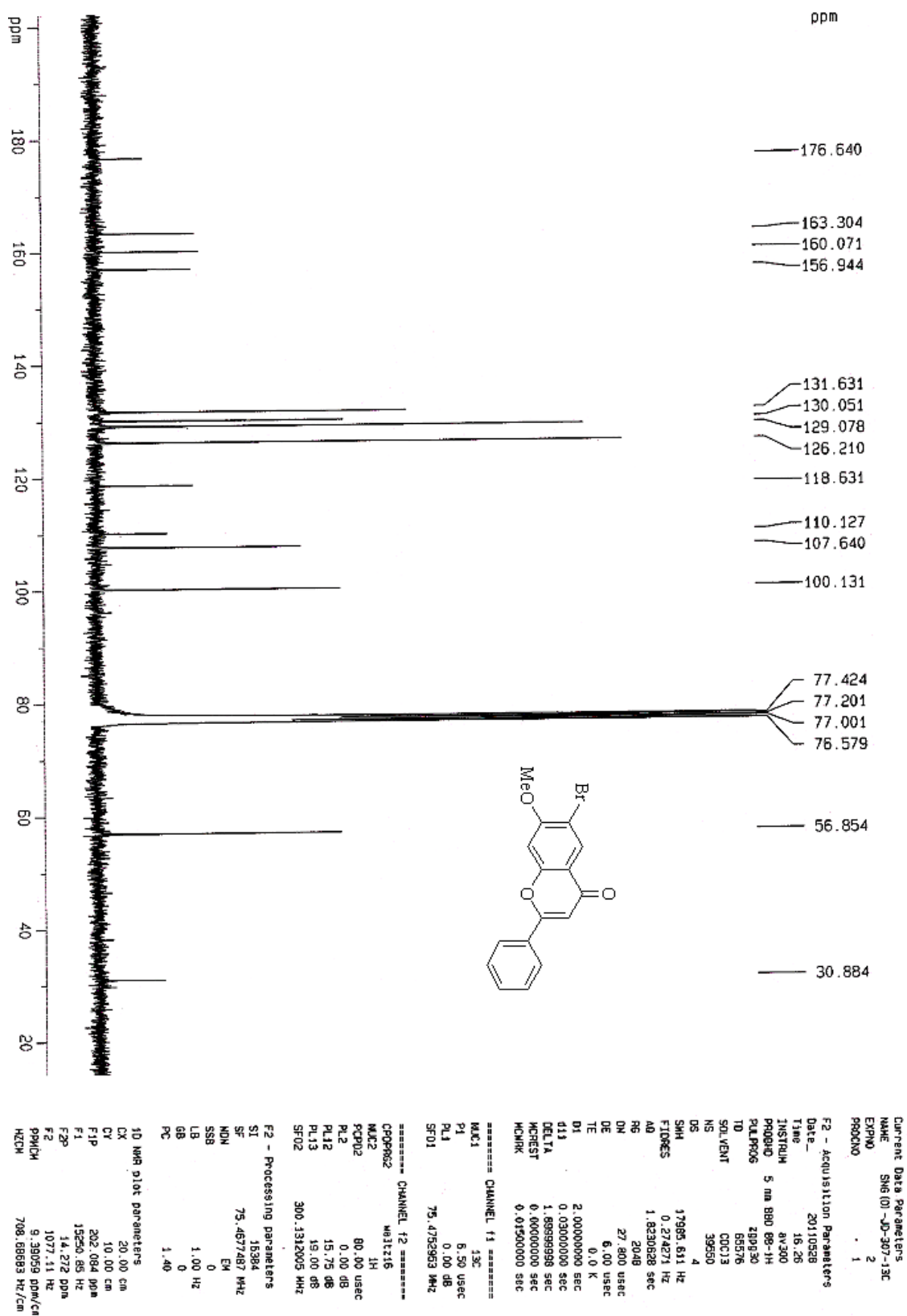


Fig. 51: ^{13}C NMR spectrum of 6-Bromo-7-methoxy-2-phenyl-chromene-4-one (5h)

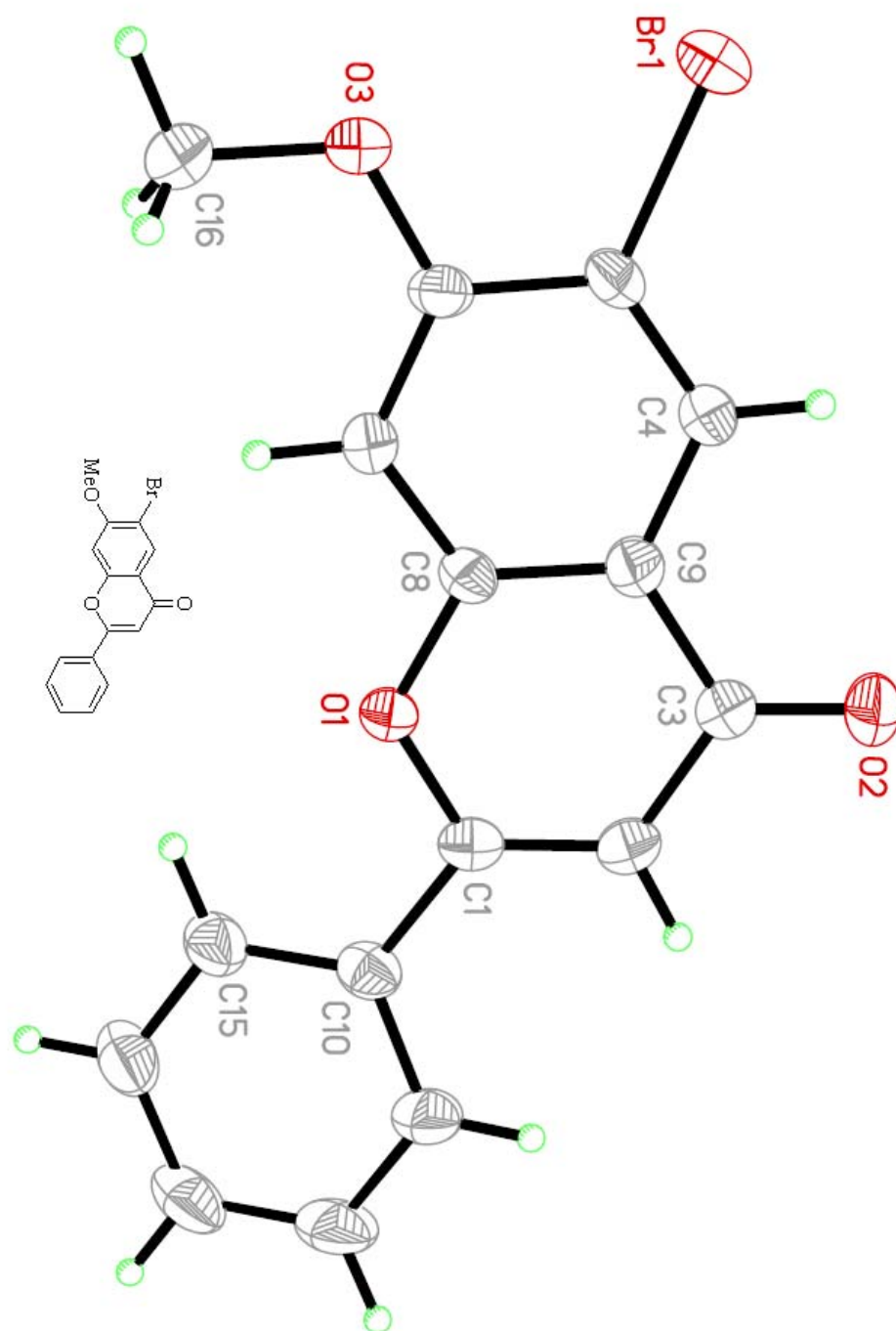


Fig. 52: ORTEP view of 6-Bromo-7-methoxy-2-phenyl-chromene-4-one is represented by their 35% thermal probability ellipsoid (5h) and this crystal structure has been deposited at the Cambridge Crystallographic Data Centre (deposition number CCDC 838053).

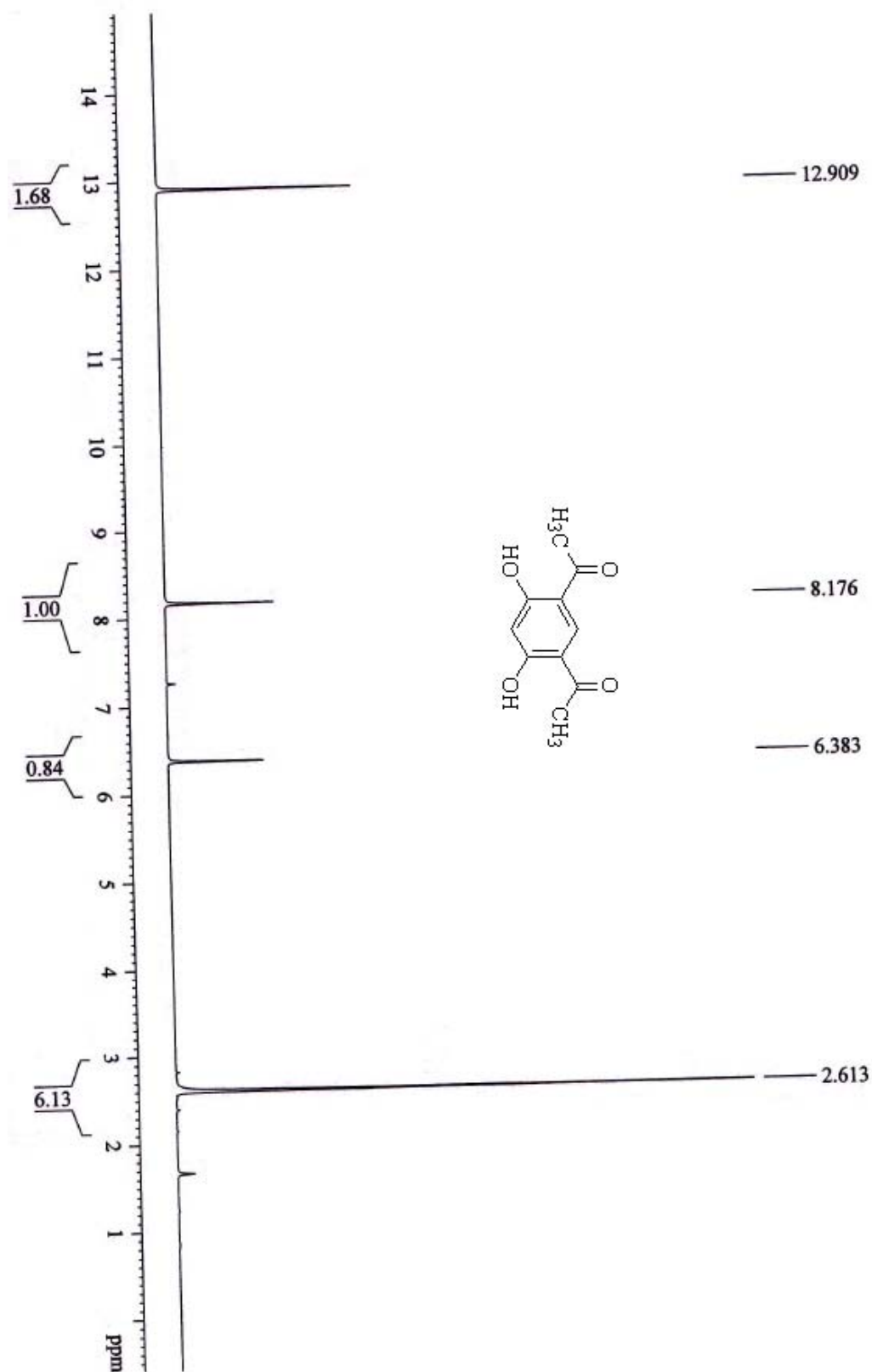


Fig. 53: ^1H NMR spectrum of 1-(2,4-Dihydroxy-5-acetylphenyl)-ethanone (6)



Fig. 54: ^{13}C NMR spectrum of 1-(2,4-Dihydroxy-5-acetylphenyl)-ethanone (6)

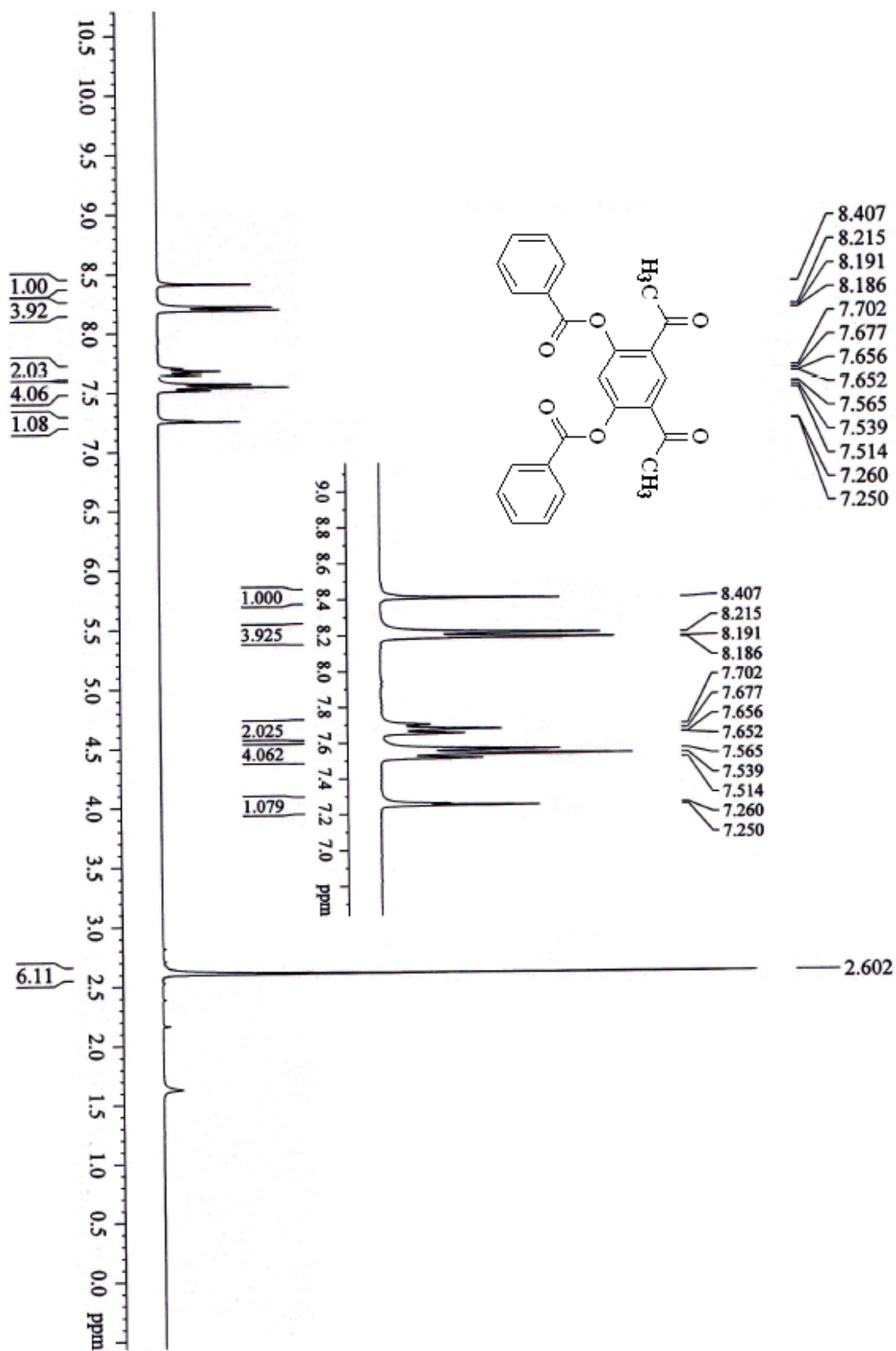


Fig. 55: ¹H NMR spectrum of 1-(2,4-Dibenzoyloxy-5-acetylphenyl)-ethanone (7)



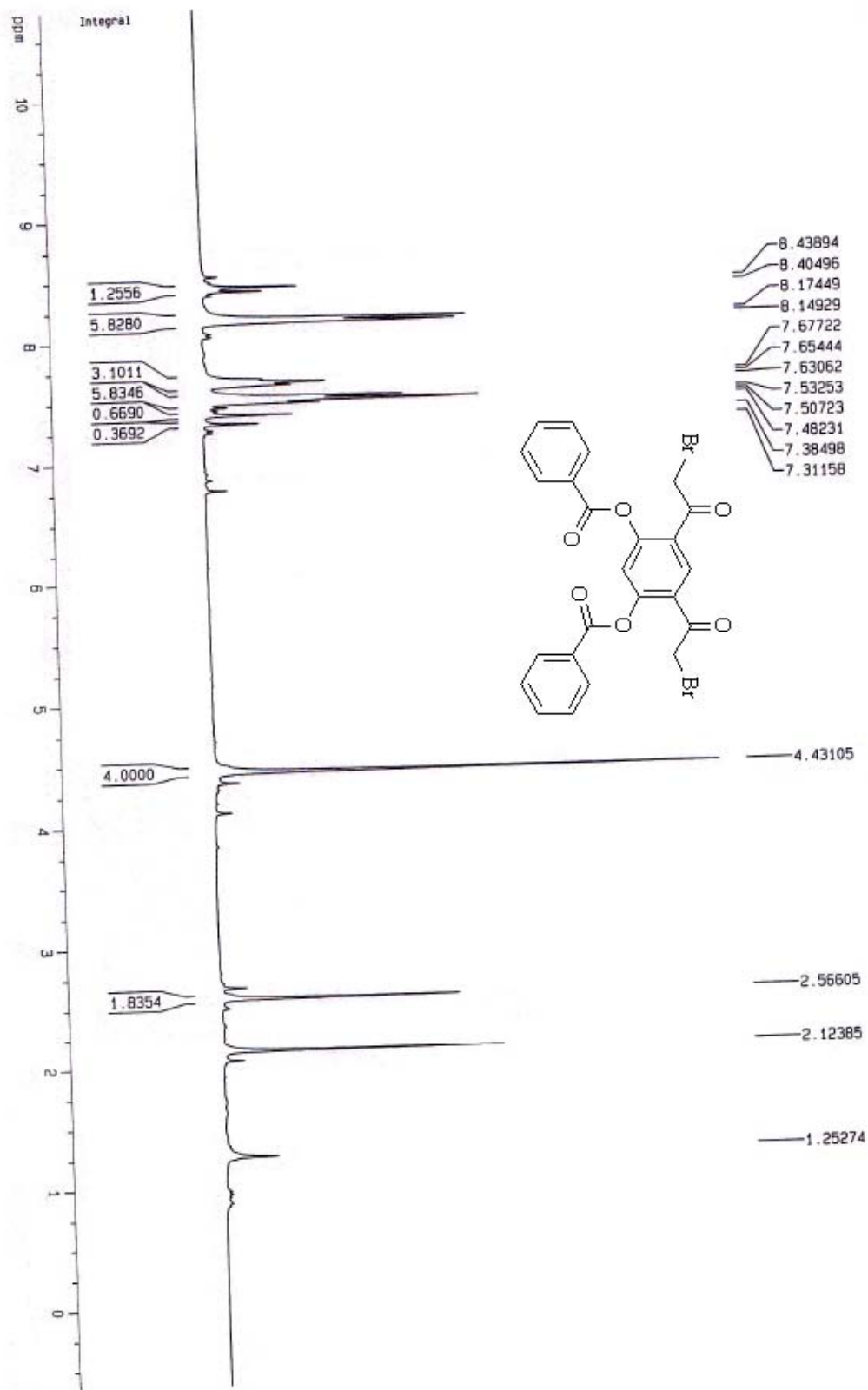


Fig. 57: ^1H NMR spectrum of 1-[2,4-Dibenzoyloxy-5-(2-bromoacetyl)-phenyl]-2-bromoethanone (8)

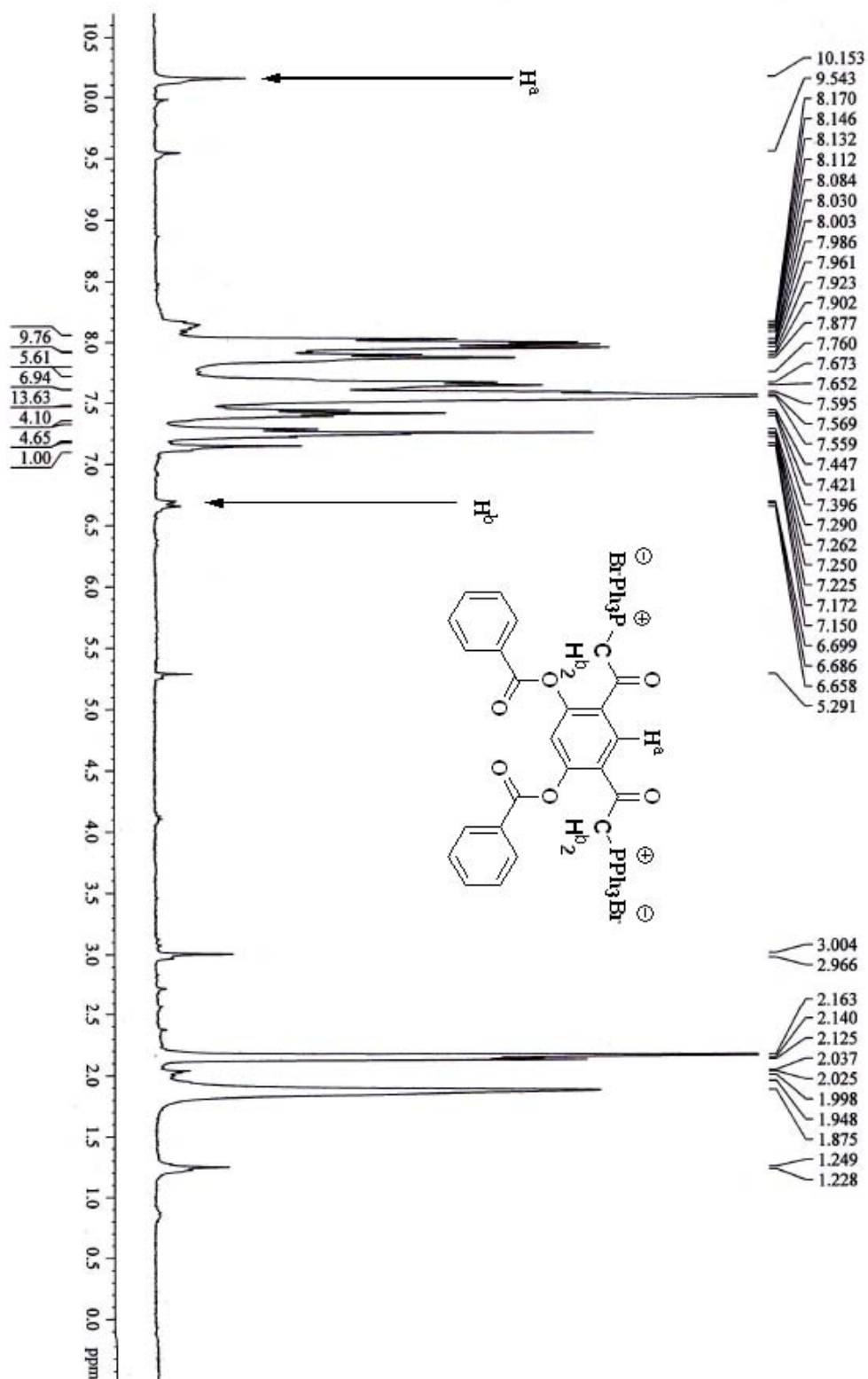


Fig. 58: ¹H NMR spectrum of 2, 4-Dibenzoyloxy-5-(2-oxoethyltriphenylphosphonium)-benzoyl methyl triphenyl phosphonium dibromide (9)

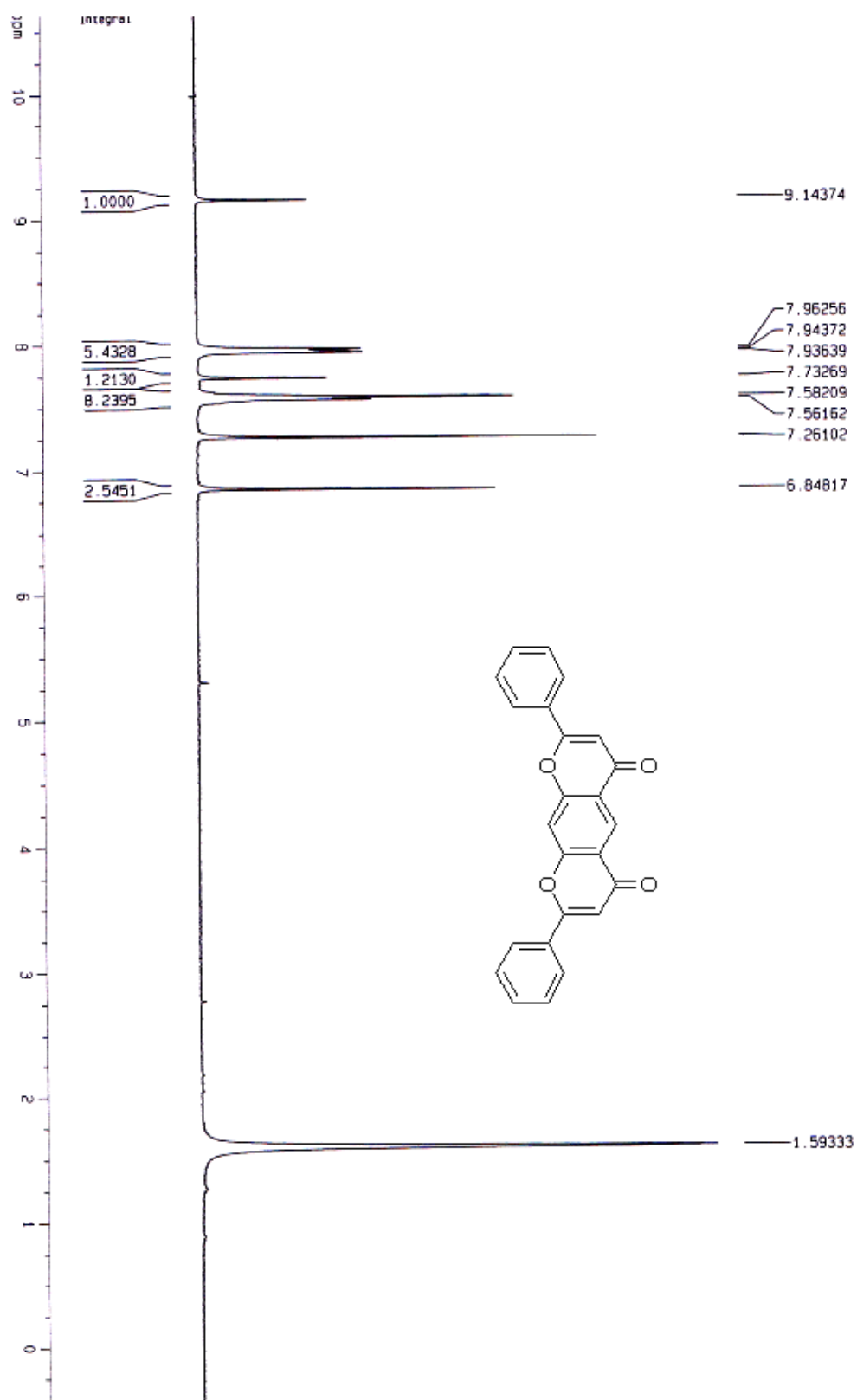


Fig. 59: ^1H NMR spectrum of 2, 8-Diphenyl-pyrano [3, 2-g] chromene-4, 6-dione (10)

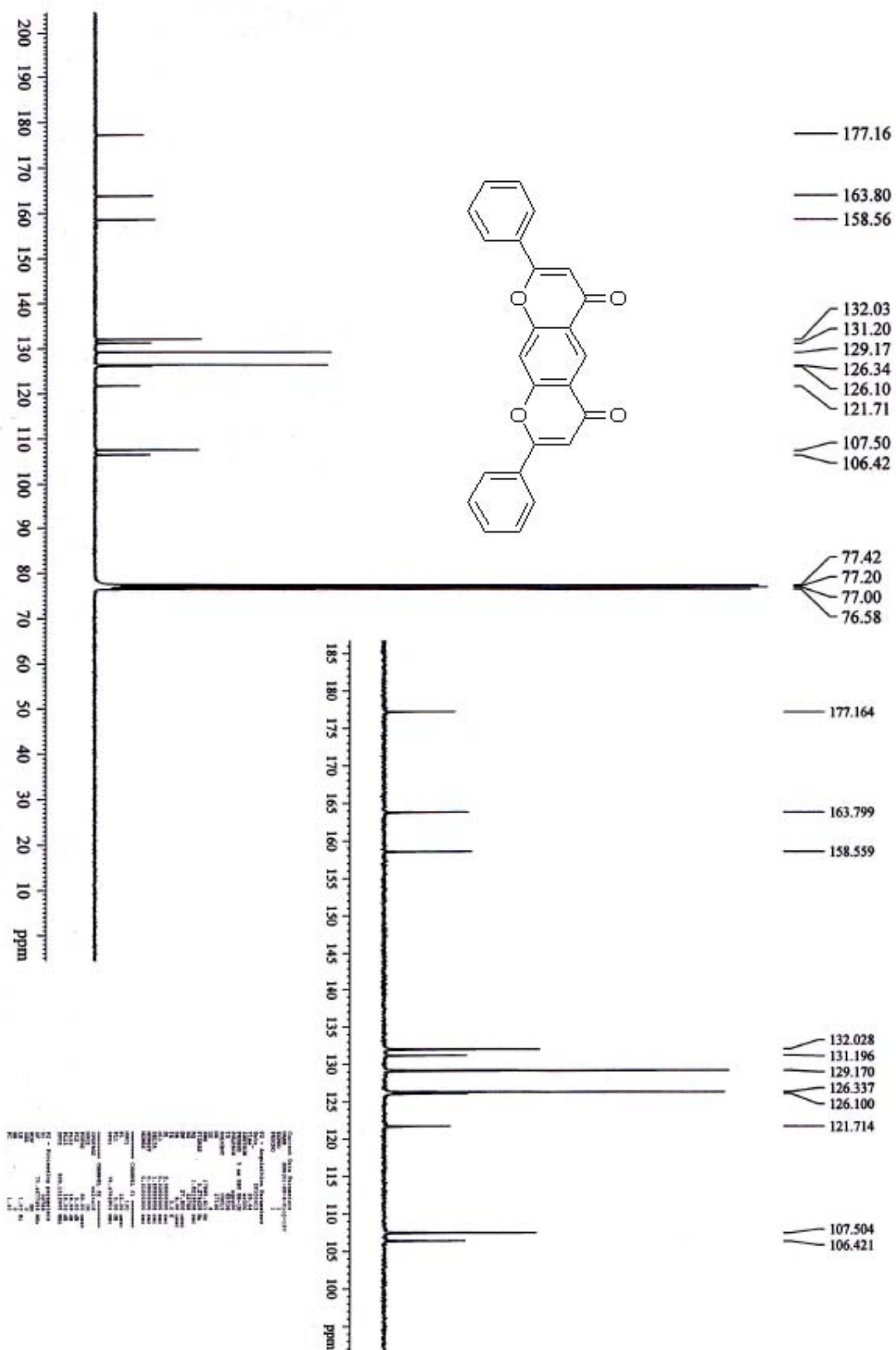


Fig. 60: ¹³C NMR spectrum of 2, 8-Diphenyl-pyrano [3, 2-g] chromene-4, 6-dione (10)

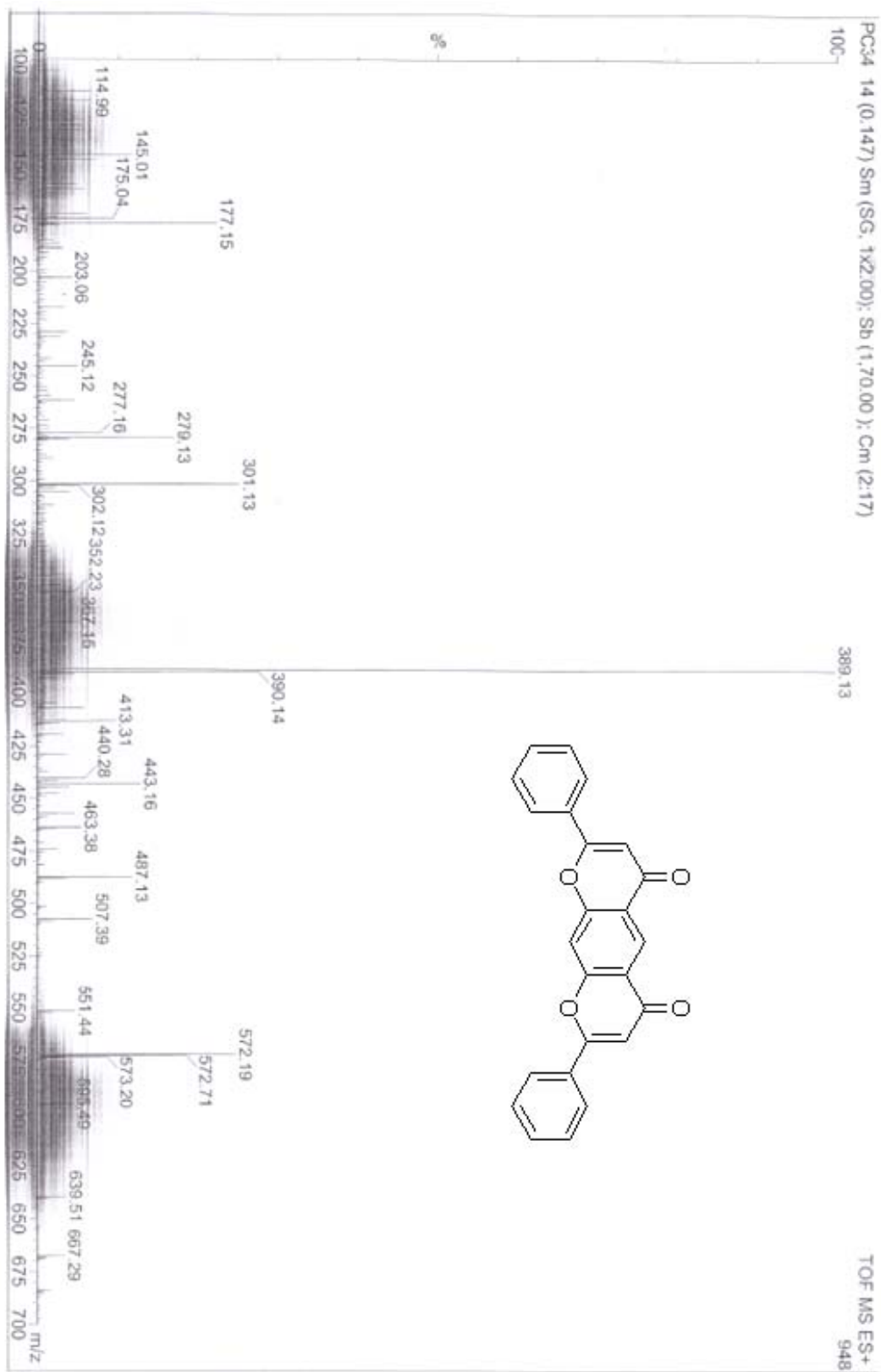


Fig. 61: High Resolution Mass spectrum of 2, 8-Diphenyl-pyrano [3, 2-g] chromene-4, 6-dione (10)