Supporting Information

Silver-Catalyzed C(sp^3)–H Chlorination

Jun Ozawa and Motomu Kanai

Graduate School of Pharmaceutical Sciences, The University of Tokyo
Kanai Life Science Catalysis Project, ERATO, Japan Science Technology Agency,

7-3-1, Hongo, Bunkyo-ku, Tokyo 113-0033, Japan.

Phone: +81-3-5841-4830
Fax: +81-3-5684-5206
Email: kanai@mol.f.u-tokyo.ac.jp (Kanai)

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1. General method

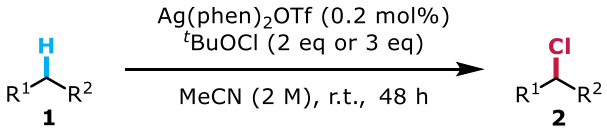
AgOTf, 1,10-phenanthroline, and ^{t}BuOCl were purchased from Wako Pure Chemical Industries, Ltd., Aldrich, and Tokyo Chemical Industry Co., Ltd. (TCI), respectively, which were used without any purification. NMR spectra were recorded on JEOL ECX-500 (500.16 MHz for ^{1}H NMR and 125.77 MHz for ^{13}C NMR), and JEOL ECS-400 (391.78 MHz for ^{1}H NMR, 98.52 MHz for ^{13}C NMR) spectrometers. For ^{1}H and ^{13}C NMR, chemical shifts were reported in the scale relative to the solvent used as an internal reference (δ = 7.26 and 77.00 ppm ($CDCl_3$), 2.04 and 29.80 ppm (acetone-d_6), 2.50 and 39.52 ppm (DMSO-d_6); ^{1}H and ^{13}C NMR respectively). Infrared (IR) spectra were recorded on a JASCO FT/IR 410 Fourier transform infrared spectrophotometer. ESI-MS spectra were measured on a Waters ZQ4000 spectrometer (for LRMS), and a JEOL JMS-T100LC AccuTOF spectrometer (for HRMS). Optical rotations were measured on a JASCO P-1010 polarimeter. HPLC was conducted by JASCO HPLC systems (pump: PU-2080; detector: UV-2075, measured at 254 nm). Column chromatography was performed with silica gel Merck 60 (230–400 mesh ASTM). NMR yields were calculated by ^{1}H NMR of crude products using $CHCl_2CHCl_2$ (δ = 5.9 ppm in $CDCl_3$) as an internal standard.

2. Preparation of Ag(phen)$_2$OTf[1]

To a stirred mixture of AgOTf (256.9 mg, 1 mmol, 1 eq) in MeOH (5 mL) was added 1,10-phenanthroline anhydrate (360.4 mg, 2 mmol, 2 eq) in MeOH (10 mL). The colorless solution turned into yellow suspension on adding a 1,10-phenanthroline solution. After stirring for three hours the solid was collected on a Kiriyama filter and washed with MeOH, then dried under vacuum to give Ag(phen)$_2$OTf (542.0 mg, 88%) as yellow solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.17 (4H, dd, J = 4.5 Hz, 1.8 Hz), 8.80 (4H, dd, J = 8.1 Hz, 1.8 Hz), 8.24 (4H, s), 8.02 (4H, dd, J = 8.1 Hz, 4.5 Hz); identical to the reported data.

3. Typical C(sp^3)–H chlorination procedure



A) 2 mmol scale reaction

To a stirred mixture of substrate **1** (2 mmol, 1 eq) and Ag(phen)$_2$OTf (2.5 mg, 0.004 mmol, 0.2 mol%) in MeCN (1 mL, 2 M to **1**) was added ^{t}BuOCl (678.6 μL, 6 mmol, 3 eq)*, and then a cap was put on the flask. The color of the reaction mixture turned to brown by adding ^{t}BuOCl. After stirring for 48 hours the reaction mixture was passed through a silica gel pad in a Pasteur pipet (about 4 cm, the eluent was Et_2O or EtOAc) to remove the silver catalyst and the volatiles were removed by rotavap. The residue was purified by silica gel column chromatography to obtain the chlorinated compound **2**.

*In chlorination of a benzylic C(sp^3)–H bond, we used 1) 2 eq of ^{t}BuOCl to minimize dichlorination and 2) dry MeCN, and ran the reaction under argon atmosphere to avoid S_N2 substitution of Cl by H_2O; otherwise

[1] C. Santini, C. Pettinari, G. Gioia Lobbia, D. Leonesi, G. Valle, S. Calogero *Polyhedron* **1998**, *17*, 3201–3210.

significant amounts of acetophenones were produced.

B) 5 mmol scale reaction

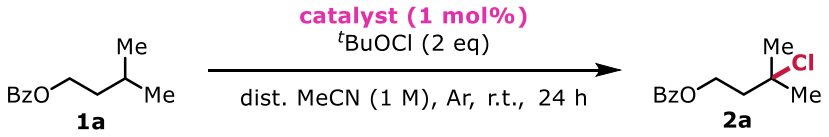
The workup procedure was different from that of 2 mmol scale reaction. To a stirred reaction mixture cooled by ice bath was added $NaHSO_3$ aq and Et_2O. The organic layer was separated, dried over Na_2SO_4, filtrated, and evaporated to give a crude mixture. The following procedure was the same as above.

4. Optimization of the reaction conditions

4.1. Chlorination of *tert*-C(sp^3)–H bonds

The chlorination conditions were screened to maximize the yield using 0.2 mmol of **1a**. First, we screened catalysts (Table S1). We found that AgOTf with a bidentate ligand catalyzed the chlorination in good yield (entry 5–7). Free Ag salts didn't work well (entry 1–4), and Cu(I) and Fe(II) salts, which are catalysts for Fenton-type reactions, failed efficient chlorination, either (entry 8–11). The ligand itself was unable to be the catalyst (entry 13–15), and almost no reaction proceeded without any catalyst (entry 12). The results suggested that $Ag(phen)_2OTf$ was the best among the catalysts tested.

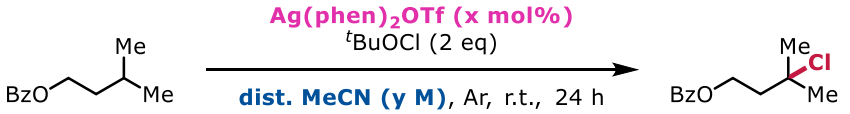
Table S1 The condition screening — catalysts.



entry	catalyst	NMR yield	NMR rsm
1	AgOTf	12%	87%
2	$AgNO_3$	7%	92%
3	AgOAc	8%	91%
4	AgTFA	16%	83%
5	**$Ag(1,10\text{-phen})_2OTf$**	**76%**	**23%**
6	Ag(2,2'-bpy)OTf	60%	39%
7	$Ag(2,2'\text{-bpy})_2OTf$	69%	30%
8	CuCl (5 mol%)	24%	75%
9	CuCl + 1,10-phen (5+10 mol%)	51%	44%
10	CuCl + 2,2'-bpy (5+10 mol%)	24%	74%
11	$Fe(OTf)_2$ (5 mol%)	< 10% (TLC)	–
12	(none)	6%	93%
13	1,10-phen (10 mol%)	5%	94%
14	2,2'-bpy (10 mol%)	13%	86%
15	4,4'-bpy (10 mol%)	1%	99%

Next, we screened the amount of the solvent and the catalyst (Table S2). The yield did not change a lot as the concentration was changed (entry 1–4), and 2 M should be the most proper concentration. The amount of the catalyst was able to decrease as low as 0.2 mol% (entry 6). Even though higher NMR yield was obtained in entry 7, the difference in the yields is not large considering 2.5 times as much as the catalyst was used.

Table S2 The condition screening — the amount of MeCN and Ag(phen)$_2$OTf.

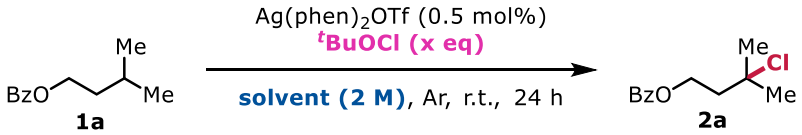


entry	Ag cat	MeCN	NMR yield	NMR rsm
1	1 mol%	0.5 M	66%	33%
2	1 mol%	1 M	76%	23%
3	**1 mol%**	**2 M**	**77%**	**22%**
4	1 mol%	4 M	71%	28%
5	0.1 mol%	2 M	56%	43%
6	**0.2 mol%**	**2 M**	**71%**	**28%**
7	0.5 mol%*	2 M	74%	25%
8	1 mol%*	2 M	58%	41%
9	2 mol%*	2 M	59%	40%

* Ag(phen)$_2$OTf did not dissolve completely in MeCN.

Then we screened solvents and oxidants (Table S3). Ag(phen)$_2$OTf did not dissolve well in the solvents except for MeCN and DMF, which may be one reason for the yield to be decreased by replacing MeCN with other solvents (entry 1–8). Water contamination did not affect the yield (entry 1 vs 9), so MeCN without any purification (as purchased) was chosen as the best solvent. DMSO furiously reacted with ^{t}BuOCl so that no chlorination occurred on **1a**. Other oxidants like *N*-chlorosuccinimide or aqueous NaOCl didn't promote efficient chlorination.

Table S3 The condition screening — solvents and oxidants.



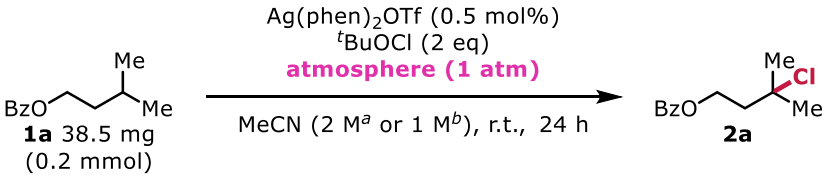
entry	^{t}BuOCl	solvent	NMR yield	NMR rsm
1	**2 eq**	**dist. MeCN**	**78%**	**21%**
2	2 eq	dry EtOAc	50%	44%
3	2 eq	dry CH_2Cl_2	37%	43%
4	2 eq	dry $CHCl_3$	27%	58%
5	2 eq	dry ^{t}BuOH	49%	49%
6	2 eq	dry DMF	0% (TLC)	–
7	2 eq	dry DMSO	N.R. (TLC)	–
8	2 eq	PhCl	65%	25%
9	**2 eq**	**MeCN/water (1/1)**	**79%**	**12%**
10	2 eq	MeCN[a]	76%	23%
11	NCS 2 eq	MeCN[a]	N.R. (TLC)	–
12	NaOCl 2 eq	MeCN[a]	3%	96%
13	3 eq	MeCN[a]	84%	11%
14	4 eq	MeCN[a]	87%	11%
15[b]	2 eq	MeCN[a]	80%	19%
16[b]	**3 eq**	**MeCN[a]**	**92%**	**7%**

[a] neither distilled nor anhydrous [b] 48 h

Finally, we tried the chlorination under air or O_2 atmosphere (Table S4). Oxygen had

adverse effects on the chlorination process, but aerobic conditions were acceptable if a small reaction vessel was used.

Table S4 Reaction atmosphere: Ar, O_2 or air.



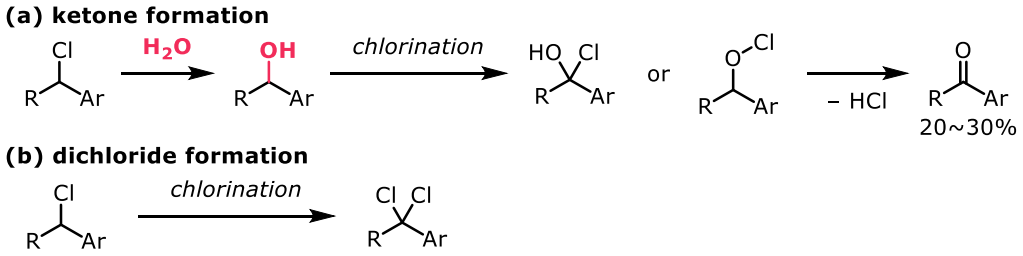
entry	atmosphere	NMR yield[a]	NMR rsm[a]	NMR yield[b]	NMR rsm[b]
1	Ar	79%	18%	79%	20%
2	O_2	64%	35%	53%	46%
3	air	76%	23%	59%	40%

[a] capped tube, ϕ16 mm × 100 mm (12.5 mL) [b] test tube with balloon

4.2. Chlorination of benzylic C(sp^3)–H bonds

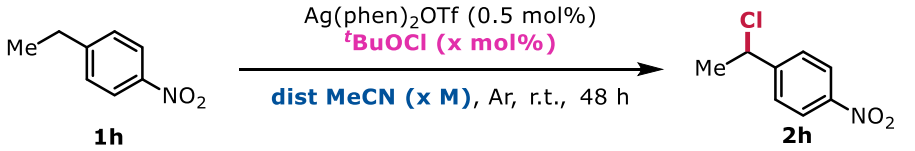
When the optimized conditions above were applied to benzylic C(sp^3)–H chlorination, two problems arose: ketone formation and dichlorination (Scheme S1). To improve the situation, we further optimized the reaction conditions using **1h**.

Scheme S1 Undesirable side reactions.

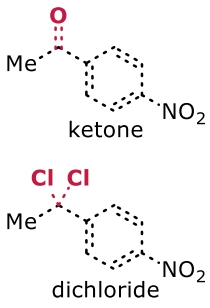


Use of dry MeCN would suppress the ketone formation, and a proper amount of ^{t}BuOCl would decrease the dichlorination. Table S5 shows that anhydrous conditions could suppress the ketone formation (entry 1–4). Use of smaller equivalents of the oxidant did not result in higher yield of monochloride, even though the dichlorination did decrease (entry 5–7).

Table S5 The condition screening — benzylic C(sp^3)–H chloriation.



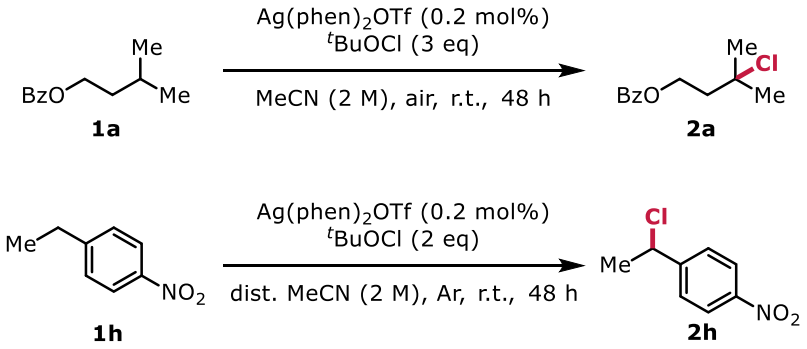
entry	^{t}BuOCl	MeCN	NMR yield	NMR rsm	ketone	dichloride
1	3 eq	0.2 M	63%	27%	3%	7%
2	3 eq	0.5 M	65%	21%	2%	11%
3	3 eq	1 M	67%	15%	2%	15%
4	3 eq	2 M	67%	10%	2%	20%
5	1.2 eq	2 M	53%	41%	1%	5%
6	1.5 eq	2 M	53%	33%	10%	3%
7	**2 eq**	**2 M**	**66%**	**19%**	**4%**	**10%**



After all we determined the optimal reaction conditions as below (Scheme S2). As there

was no problem for secondary C(sp^3)–H chlorination under *tert*-C(sp^3)–H chlorination conditions, no optimization was conducted further.

Scheme S2 The optimal reaction conditions.

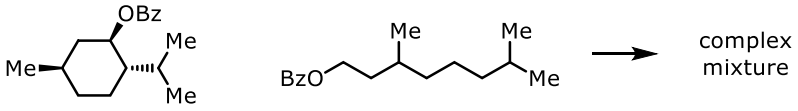


5. Limitation in the substrate scope

Listed below are compounds that didn't give satisfactory yields in the C(sp^3)–H chlorination.

1. A lot of C(sp^3)–H bonds

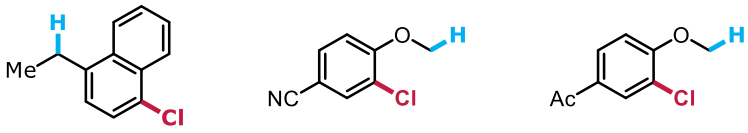
The optimized chlorination conditions were so reactive that regioselective chlorination of C(sp^3)–H bonds was not successful.



2. Nucleophilic attack to ^{t}BuOCl

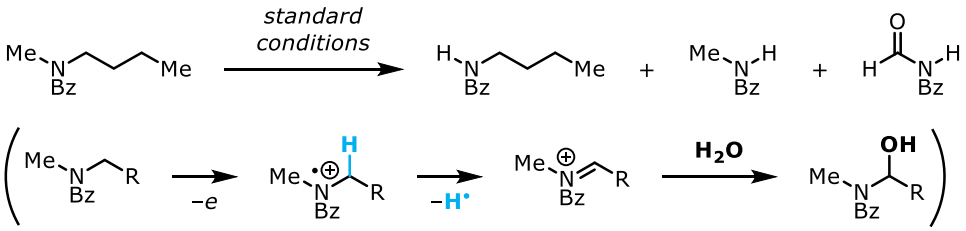
Anisole or naphthalene derivatives reacted directly with ^{t}BuOCl irrespective of the Ag catalyst.

undesired aryl chlorides:



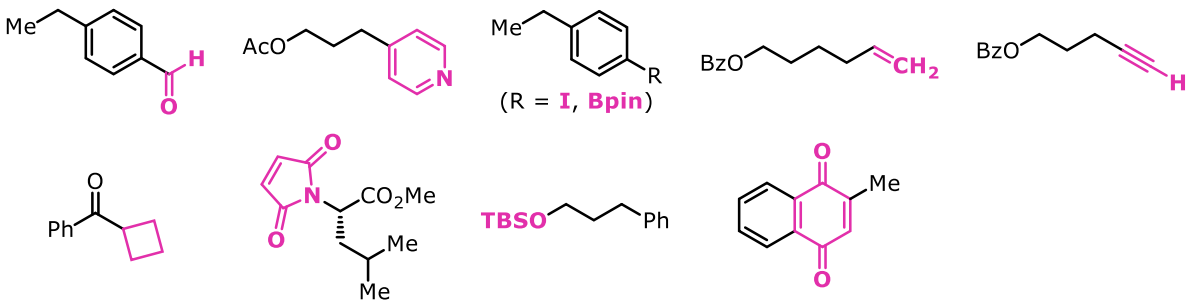
3. Direct oxidation of amide nitrogen atom by high-valent Ag

Amide nitrogen atoms seemed to be oxidized by a high-valent Ag that had formed from Ag catalyst and ^{t}BuOCl. The resulting iminium cations should be attacked by water, giving C–N-bond-cleaved products.



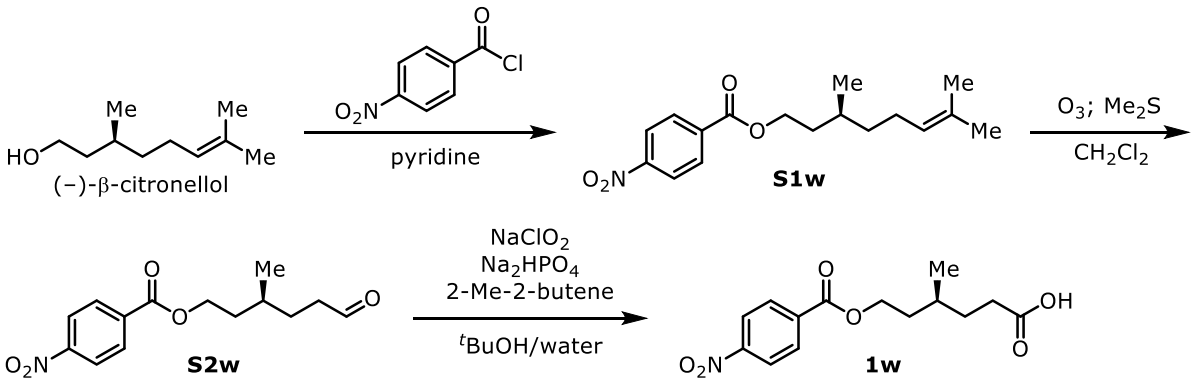
4. Labile functional groups

Compounds listed below gave rather complicated reaction mixtures under the optimized chlorination conditions.



6. Chlorination of a chiral compound

6.1. Synthesis of 1w from (−)-β-citronellol, and conversion of 2w to 2x



(−)-β-citronellol to **S1w**

To a stirred mixture of (−)-β-citronellol (781 mg, 5 mmol, 1 eq) in pyridine (5 mL) was added 4-nitrobenzoyl chloride (1.39 g. 1.5 eq) at room temperature. HCl in water was added after 8 h, and the mixture was extracted with Et_2O. The combined organic layer was washed with HCl aq, dried over Na_2SO_4, and filtrated, and volatiles were removed by rotavap. The residue was purified by silica gel column chromatography (hexane/EtOAc = 20/1) so that **S1w** was obtained as yellow liquid (1.37 g, 90%).

S1w to **S2w**

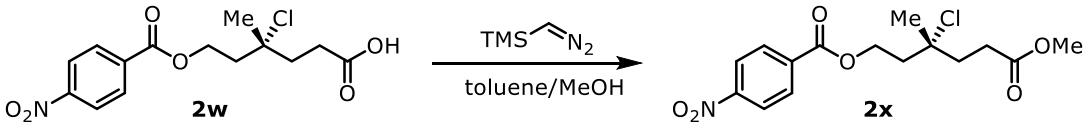
A mixture of **S1w** (663 mg, 2.17 mmol, 1 eq) in CH_2Cl_2 (5 mL, 0.4 M) cooled by acetone/dry ice bath was treated with a stream of O_3/O_2. After 5 min, the solution color turned blue, and then Ar was flushed through the mixture until the color disappeared. Me_2S (635 µL, 8.68 mmol, 4 eq) was added, and the cold bath was removed. After 15 h, the reaction mixture was washed with HCl aq, and the organic layer was dried over Na_2SO_4. After filtration, volatiles were removed by rotavap. The residue was purified by silica gel column chromatography (hexane/EtOAc = 4/1 to 2/1 to 1/2) so that **S2w** was obtained as yellow liquid (561 mg, 92%), along with **1w** (27.7 mg).

S2w to **1w**

To a stirred mixture of **S2w** (539 mg, 1.93 mmol, 1 eq), 2-methyl-2-butene (2.05 mL, 19.3 mmol, 10 eq), and Na_2HPO_4 (1.37 g, 9.65 mmol, 5 eq) in ^{t}BuOH/water (4 mL/4 mL, 0.25 M) was added $NaClO_2$ (655 mg, 5.79 mmol, 3 eq) at room temperature. After 1h, NaCl aq was added and the mixture was extracted with EtOAc. The organic layer was washed with HCl aq and dried over Na_2SO_4. After filtration, the volatiles were removed by rotavap. The

residue was purified by silica gel column chromatography (hexane/EtOAc = 3/1 to 1/2) so that **1w** was obtained as yellow liquid, which gradually solidified into yellow solid (495 mg, 87%).

[note] Minerals on silica gel sometimes eluted with the carboxylic acid **1w**. An Et_2O solution of **1w** was washed with HCl aq to remove the minerals in such a case.



2w to **2x**

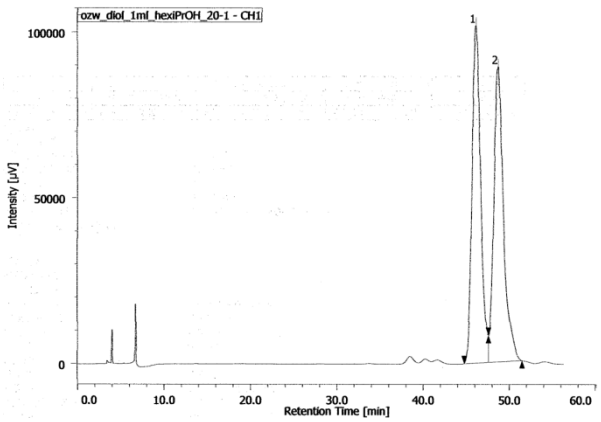
To a solution of **2w** (38.4 mg, 0.11 mmol, 1 eq) in toluene/MeOH (330 µL/110 µL, 0.25 M) was added 0.6 M hexane solution of trimethylsilyldiazomethane (200 µL, 1.05 eq) at room temperature under air. After 10 min, AcOH (a few drops) was added to quench trimethylsilyldiazomethane. The volatiles were removed by rotavap, and the residue was purified by PTLC (hexane/EtOAc = 4/1) so that **2x** was obtained as light yellow liquid (28.1 mg, 74%).

6.2. Chiral HPLC analysis of 2x

We analyzed **2x** using a chiral HPLC and the results are shown below. The charts indicated that the chirality disappeared during the chlorination.

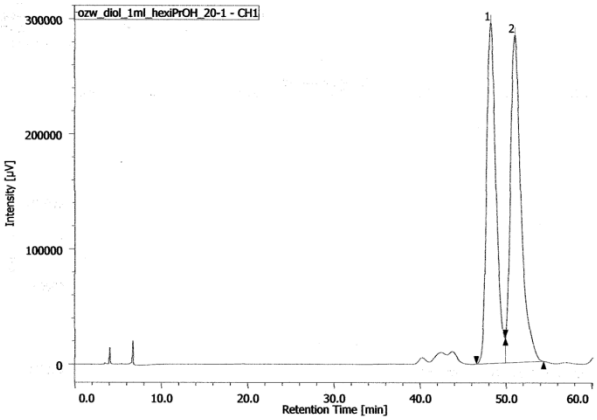
HPLC conditions	
column:	CHIRALPAK® IC ϕ 4.6 mm × 250 mm
eluent:	hexane/2-propanol = 20/1
flow rate:	1.0 mL/min

SM: (−)-**1w**



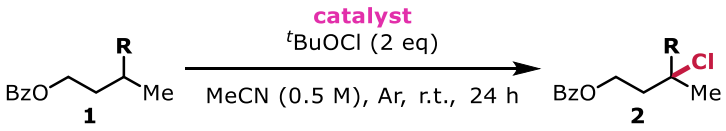
#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%
1	Unknown	1	46.108	7054078	101820	50.099
2	Unknown	1	48.667	7026092	89041	49.901

SM: (±)-**1w**



#	ピーク名	CH	tR [min]	面積 [µV·sec]	高さ [µV]	面積%
1	Unknown	1	48.175	22451983	295785	48.026
2	Unknown	1	50.975	24297273	284276	51.974

7. Procedure for free *tert*-BuO radical reactions



A) *N*-hydroxyphthalimide (NHPI) conditions

To a stirred mixture of the substrate **1** (0.1 mmol, 1 eq) and NHPI (4.1 mg, 0.025 mmol, 25 mol%) in distilled MeCN (200 µL, 0.5 M to **1**) was added ^{t}BuOCl (22.6 µL, 0.2 mmol, 2 eq) under Ar atmosphere. The color of the reaction mixture turned into yellow first, and into dark yellow on adding ^{t}BuOCl. After stirring for 24 h, Na_2SO_3 and water were added to the reaction mixture. The mixture was extracted with Et_2O and the volatiles were removed by rotavap. The NMR yields were calculated using 1,1,2,2-tetrachloroethane as an internal standard.

B) photoirradiation conditions

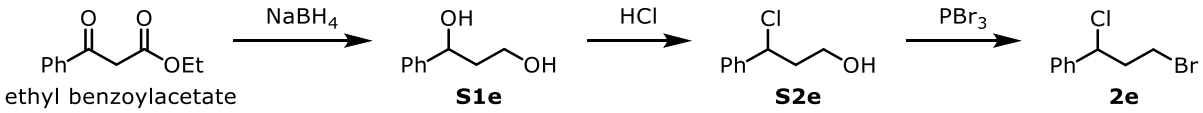
To a stirred solution of the substrate **1** (0.2 mmol, 1 eq) in distilled MeCN (400 µL, 0.5 M to **1**) was added ^{t}BuOCl (45.2 µL, 0.4 mmol, 2 eq) under Ar atmosphere. The color of the reaction mixture didn't change on adding ^{t}BuOCl. Then the reaction was irrradiated with a white LED lump*. After stirring for 24 h, Na_2SO_3 and water were added to the reaction mixture. The mixture was extracted with Et_2O and the volatiles were removed by rotavap. The NMR yields were calculated using 1,1,2,2-tetrachloroethane as an internal standard.

* Irradiations were performed with VBL-SL150-WW as the photon source, white LED lamp for plant science research purchased from Valore Ltd., Kyoto, Japan.

8. Synthesis of authentic samples of 2e and 2j

Authentic samples for **2e** and **2j** were synthesized to identify the reaction products of **1e** and **1j**, because **2e** and **2j** were not detected in HRMS (ESI, DART, nor EI).

8.1. Synthesis of authentic 2e



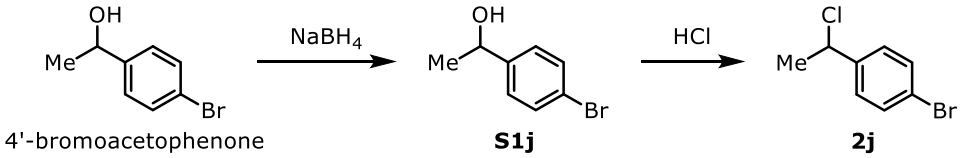
A solution of ethyl benzoylacetate (192.2 mg, 1 mmol, 1 eq) in MeOH (2 mL, 0.5 M) was treated with $NaBH_4$ (113.5 mg, 3 mmol, 3 eq) at room temperature. After 1 h, water was added to the reaction mixture. The mixture was extracted with EtOAc, and the volatiles were removed by rotavap. The residue was purified by PTLC (hexane/EtOAc = 1/2) to give **S1e** (79.9 mg, 53%).

S1e (188.2 mg, 1.2 mmol, 1 eq) in toluene (2.4 mL, 0.5 M) was treated with HCl aq (12 N, 250 µL, 3 mmol, 2.5 eq) at room temperature. After 15 h, water was added, the reaction mixture was extracted with Et_2O, and the volatiles were removed by rotavap. The residue was purified by silica gel column chromatography (hexane/EtOAc = 3/1) so that **S2e** was obtained as light dark orange liquid (132.3 mg, 64%).

A flask was charged with **S2e** (132.3 mg, 0.77 mmol, 1 eq) and dry CH_2Cl_2 (1.6 mL, 0.5 M)

under argon atmosphere. PBr_3 (36.7 µL, 0.39 mmol, 0.5 eq) in dry CH_2Cl_2 (400 µL) was added to the flask at room temperature. After 3 h, water and Et_2O were added. The organic layer was washed with water and NaCl aq, dried over Na_2SO_4, filtrated, and the volatiles were removed by rotavap. The residue was purified by silica gel column chromatography (hexane only) so that **2e** was obtained as light yellow liquid (58.4 mg, 32%).

8.2. Synthesis of authentic 2j



4'-bromoacetophenone **S1j** **2j**

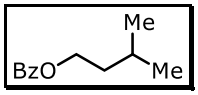
A solution of 4'-bromoacetophenone (99.5 mg, 0.5 mmol, 1 eq) in MeOH (500 µL, 1 M) was treated with $NaBH_4$ (28.4 mg, 0.75 mmol, 1.5 eq) at room temperature. After 4 h, water was added to the reaction mixture. The mixture was extracted with EtOAc, and the volatiles were removed by rotavap to give **S1j** (108 mg, containing EtOAc). **S1j** was used in the next step without further purification.

S1j was treated with HCl aq (6 N, 1 mL, around 0.5 M to **S1j**) at room temperature. After 40 h, the reaction mixture was extracted with Et_2O, and the volatiles were removed by rotavap. The residue was purified by PTLC (hexane only) so that **2j** was obtained as light yellow liquid (64.5 mg, 59%).

9. Analytical data

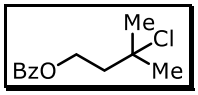
^{1}H and ^{13}C NMR data are described for **1**, which are known materials except for **1w**; full spectroscopic data for **S1w**, **S2w**, **1w** and **2** no matter whether they are new or known. HRMS for **2e** and **2j** are not described because no appropriate signals were detected. The amount of the chlorinated products is also described.

isopentyl benzoate (1a)



colorless liquid, R_f = 0.50 (hexane/EtOAc = 10/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.04 (2H, d, *J* = 7.6 Hz), 7.55 (1H, t, *J* = 7.6 Hz), 7.43 (2H, t, *J* = 7.6 Hz), 4.36 (2H, t, *J* = 6.9 Hz), 1.75–1.85 (1H, m), 1.64–1.71 (2H, m), 0.98 (6H, d, *J* = 6.9 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.6, 132.8, 130.5, 129.5, 128.3, 63.6, 37.4, 25.2, 22.5

3-chloro-3-methylbutyl benzoate (2a)



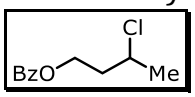
pale yellow liquid, R_f = 0.40 (hexane/EtOAc = 10/1)
Yield: 370 mg, 82%; 791 mg (1 g scale), 70%; 853 mg (1 g scale, AgOTf and 1,10-phen as the catalyst), 75%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.02–8.05 (2H, m), 7.53–7.58 (1H, m), 7.41–7.46 (2H, m), 4.56 (2H, t, *J* = 6.8 Hz), 2.25 (2H, t, *J* = 6.8 Hz), 1.68 (6H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.4, 133.0, 130.1, 129.5, 128.3, 68.4, 62.0, 44.0, 32.9

IR (neat, cm^{-1}) ν: 2923, 1720, 1452, 1275, 1114, 712
HRMS (ESI): *m/z* calcd for $C_{12}H_{15}ClO_2Na$ $[M+Na]^+$ 249.0653, Found 249.0644

butyl benzoate (1b)

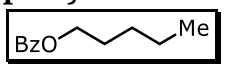
colorless liquid, R_f = 0.46 (hexane/EtOAc = 10/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05 (2H, dd, *J* = 8.0 H, 1.1 Hz), 7.52–7.57 (1H, m), 7.41–7.46 (2H, m), 4.33 (2H, t, *J* = 6.6 Hz), 1.70–1.79 (2H, m), 1.43–1.53 (2H, m), 0.98 (3H, t, *J* = 7.4 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.6, 132.7, 130.5, 129.5, 128.2, 64.8, 30.7, 19.2, 13.7

3-chlorobutyl benzoate (2b)



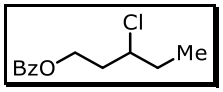
pale yellow liquid, R_f = 0.34 (hexane/EtOAc = 10/1)
Yield: 152 mg, 35%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.04 (2H, dd, *J* = 8.0 Hz, 1.1 Hz), 7.54–7.58 (1H, m), 7.44 (2H, t, *J* = 7.7 Hz), 4.43–4.55 (2H, m), 4.20–4.28 (1H, m), 2.18–2.26 (1H, m), 2.07–2.15 (1H, m), 1.60 (3H, d, *J* = 6.9 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.3, 133.0, 130.0, 129.5, 128.3, 62.0, 54.8, 39.1, 25.4
IR (neat, cm^{-1}) ν: 2974, 1720, 1452, 1274, 1114, 712
HRMS (ESI): *m/z* calcd for $C_{11}H_{13}ClO_2Na$ $[M+Na]^+$ 235.0496, Found 235.0491

pentyl benzoate (1c)



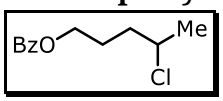
colorless liquid, R_f = 0.58 (hexane/EtOAc = 10/1)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.05 (2H, d, *J* = 7.4 Hz), 7.55 (1H, t, *J* = 7.4 Hz), 7.44 (2H, t, *J* = 7.4 Hz), 4.32 (2H, t, *J* = 6.7 Hz), 1.71–1.84 (2H, m), 1.32–1.49 (4H, m), 0.93 (3H, t, *J* = 7.0 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.7, 132.8, 130.5, 129.5, 128.3, 65.1, 28.4, 28.1, 22.4, 14.0

3-chloropentyl benzoate (2c)



pale yellow liquid, R_f = 0.50 (hexane/EtOAc = 10/1)
Yield: 288 mg (**2c** + **2c'**), 64%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.01–8.05 (2H, m), 7.54–7.59 (1H, m), 7.42–7.47 (2H, m), 4.44–4.58 (2H, m), 4.01–4.09 (1H, m), 2.20–2.28 (1H, m), 2.05–2.15 (1H, m), 1.73–1.92 (2H, m), 1.07 (3H, t, *J* = 7.4 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.4, 133.0, 130.1, 129.5, 128.4, 62.1, 61.6, 37.0, 31.6, 10.8
IR (neat, cm^{-1}) ν: 2970, 1720, 1452, 1274, 1114, 712
HRMS (ESI): *m/z* calcd for $C_{12}H_{15}ClO_2Na$ $[M+Na]^+$ 249.0653, Found 249.0661

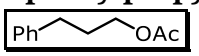
4-chloropentyl benzoate (2c')



pale yellow liquid, R_f = 0.46 (hexane/EtOAc = 10/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.02–8.06 (2H, m), 7.54–7.58 (1H, m), 7.43–7.48 (2H, m), 4.32–4.37 (m, 2H), 4.06–4.13 (1H, m), 1.99–2.05 (1H, m), 1.82–1.94 (3H, m), 1.55 (3H, d, *J* = 6.3 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.5, 132.9, 130.2, 129.5, 128.3, 64.3, 58.1, 36.8, 26.0, 25.4

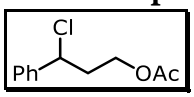
IR (neat, cm^{-1}) ν: 2964, 1719, 1451, 1274, 1112, 711
HRMS (ESI): *m/z* calcd for $C_{12}H_{15}ClO_2Na$ $[M+Na]^+$ 249.0653, Found 249.0643

3-phenylpropyl acetate (1d)



pale yellow liquid, R_f = 0.52 (hexane/EtOAc = 5/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.27–7.33 (2H, m), 7.17–7.23 (3H, m), 4.10 (2H, t, *J* = 6.6 Hz), 2.70 (2H, t, *J* = 7.7 Hz), 2.06 (3H, s), 1.94–2.01 (2H, m)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 171.1, 141.1, 128.4, 128.3, 125.9, 63.8, 32.1, 30.1, 20.9

3-chloro-3-phenylpropyl acetate (2d)

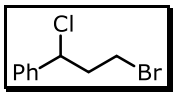


pale yellow liquid, R_f = 0.46 (hexane/EtOAc = 5/1)
Yield: 319 mg, 75%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.29–7.42 (5H, m), 4.98–5.03 (1H, m), 4.21–4.28 (1H, m), 4.12–4.18 (1H, m), 2.31–2.48 (2H, m), 2.05 (3H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 170.8, 140.9, 128.7, 128.5, 126.9, 61.5, 59.8, 38.6, 20.8
IR (neat, cm^{-1}) ν: 2961, 1741, 1365, 1237, 1038, 761, 698
HRMS (ESI): *m/z* calcd for $C_{11}H_{13}ClO_2Na$ $[M+Na]^+$ 235.0496, Found 235.0504

(3-bromopropyl)benzene (1e)

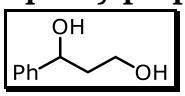
colorless liquid, R_f = 0.54 (hexane/EtOAc = 1/0)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.29–7.35 (2H, m), 7.20–7.25 (3H, m), 3.41 (2H, t, *J* = 6.6 Hz), 2.80 (2H, t, *J* = 7.4 Hz), 2.15–2.22 (2H, m)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 140.5, 128.52, 128.47, 126.1, 34.1, 33.9, 33.1

(3-bromo-1-chloropropyl)benzene (2e)



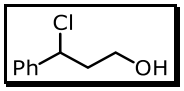
light yellow liquid, R_f = 0.44 (hexane/EtOAc = 1/0)
Yield: 234 mg, 50%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.31–7.44 (5H, m), 5.11–5.15 (1H, m), 3.56–3.62 (1H, m), 3.39–3.45 (1H, m), 2.59–2.68 (1H, m), 2.44–2.52 (1H, m)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 140.4, 128.8, 128.6, 127.0, 61.1, 42.3, 30.1
IR (neat, cm^{-1}) ν: 3031, 2967, 1492, 1454, 1258, 757, 697
HRMS (ESI, DART, EI): not detected

1-phenylpropane-1,3-diol (S1e)



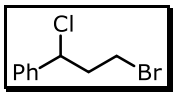
light yellow liquid, R_f = 0.42 (hexane/EtOAc = 1/2)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.21–7.36 (5H, m), 4.86 (1H, dd, *J* = 8.5 Hz, 4.0 Hz), 3.12–4.32 (2H, m), 3.68–3.80 (2H, m), 1.79–1.98 (2H, m)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.2, 128.3, 127.3, 125.6, 73.5, 60.7, 40.3

3-chloro-3-phenylpropan-1-ol (S2e)



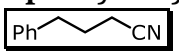
dark orange liquid, R_f = 0.77 (hexane/EtOAc = 1/2)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.28–7.43 (5H, m), 5.13 (1H, dd, *J* = 9.4 Hz, 5.4 Hz), 3.82–3.90 (1H, m), 3.68–3.76 (1H, m), 2.20–2.39 (2H, m), 2.14 (1H, bs)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.4, 128.7, 128.3, 126.9, 60.3, 59.6, 42.1

(3-bromo-1-chloropropyl)benzene (2e, authentic sample)



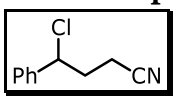
light yellow liquid, R_f = 0.44 (hexane/EtOAc = 1/0)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.31–7.44 (5H, m), 5.13 (1H, dd, *J* = 9.0 Hz, 5.4 Hz), 3.54–3.64 (1H, m), 3.38–3.46 (1H, m), 2.58–2.70 (1H, m), 2.42–2.54 (1H, m)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 140.4, 128.8, 128.6, 127.0, 61.1, 42.3, 30.1
IR (neat, cm^{-1}) ν: 3032, 2968, 1493, 1454, 1258, 758, 697

4-phenylbutyronitrile (1f)



colorless liquid, R_f = 0.48 (hexane/EtOAc = 5/1)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.29–7.36 (2H, m), 7.17–7.28 (3H, m), 2.79 (2H, t, *J* = 7.4 Hz), 2.32 (2H, t, *J* = 7.2 Hz), 1.94–2.03 (2H, m)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 139.6, 128.5, 128.3, 126.4, 119.4, 34.2, 26.8, 16.2

4-chloro-4-phenylbutyronitrile (2f)

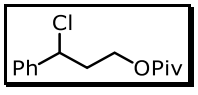


yellow liquid; , R_f = 0.40 (hexane/EtOAc = 5/1)
Yield: 255 mg, 71%
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32–7.43 (5H, m), 4.96–5.01 (1H, m), 2.28–2.64 (4H, m)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 139.6, 128.9, 126.7, 118.4, 61.1, 35.3, 15.2
IR (neat, cm^{-1}) ν: 2934, 2247, 1492, 1455, 1247, 761
HRMS (ESI): *m/z* calcd for $C_{10}H_{10}ClNNa$ $[M+Na]^+$ 202.0394, Found 202.0397

3-phenylpropyl pivalate (1g)

colorless liquid, R_f = 0.54 (hexane/EtOAc = 10/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.30 (2H, t, *J* = 7.4 Hz), 7.20 (3H, t, *J* = 8.3 Hz), 4.09 (2H, t, *J* = 6.6 Hz), 2.71 (2H, t, *J* = 7.7 Hz), 1.94–2.01 (2H, m), 1.23 (9H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 178.5, 141.2, 128.39, 128.36, 125.9, 63.5, 38.7, 32.1, 30.3, 27.1

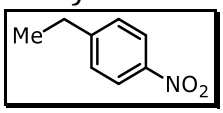
3-chloro-3-phenylpropyl pivalate (2g)



light yellow liquid, R_f = 0.48 (hexane/EtOAc = 10/1)
Yield: 406 mg, 80%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.32–7.41 (5H, m), 4.99 (1H, dd, *J* = 8.6 Hz, 5.7 Hz), 4.20–4.26 (1H, m), 4.08–4.14 (1H, m), 2.32–2.48 (2H, m), 1.21 (9H, s)

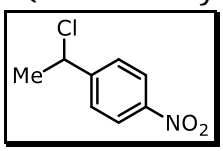
^{13}C NMR (100 MHz, $CDCl_3$) δ: 178.2, 140.9, 128.8, 128.5, 126.8, 61.4, 59.9, 38.8, 38.7, 27.1
IR (neat, cm^{-1}) ν: 2972, 1730, 1479, 1283, 1154, 1035, 759, 698
HRMS (ESI): *m/z* calcd for $C_{14}H_{19}ClO_2Na$ $[M+Na]^+$ 277.0966, Found 277.0956

1-ethyl-4-nitrobenzene (1h)



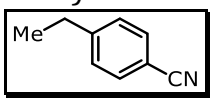
yellow liquid, R_f = 0.65 (hexane/EtOAc = 5/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.08–8.13 (2H, m), 7.32 (2H, d, *J* = 8.6 Hz), 2.74 (2H, q, *J* = 7.7 Hz), 1.26 (3H, t, *J* = 7.7 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.0, 146.1, 128.5, 123.5, 28.7, 14.9

1-(1-chloroethyl)-4-nitrobenzene (2h)



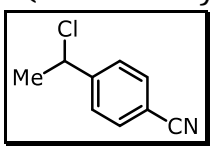
yellow liquid, R_f = 0.52 (hexane/EtOAc = 5/1)
Yield: 231 mg, 62%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.21 (2H, d, *J* = 8.9 Hz), 7.59 (2H, d, *J* = 8.9 Hz), 5.13 (1H, q, *J* = 6.9 Hz), 1.86 (3H, d, *J* = 6.9 Hz);
^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.6, 147.5, 127.5, 123.9, 56.8, 26.3
IR (neat, cm^{-1}) ν: 2982, 1606, 1523, 1349, 856, 781, 698
HRMS (ESI): *m/z* calcd for $C_8H_8ClNO_2Na$ $[M+Na]^+$ 208.0136, Found 208.0146

4-ethylbenzonitrile (1i)



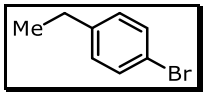
yellow liquid, R_f = 0.60 (hexane/EtOAc = 5/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.55 (2H, d, *J* = 8.3 Hz), 7.28 (2H, d, *J* = 8.3 Hz), 2.69 (2H, q, *J* = 7.4 Hz), 1.24 (3H, t, *J* = 7.4 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.7, 132.0, 128.6, 119.1, 109.3, 28.9, 14.9

4-(1-chloroethyl)benzonitrile (2i)



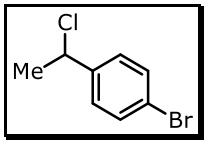
light yellow liquid, R_f = 0.50 (hexane/EtOAc = 5/1)
Yield: 200 mg, 60%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.64 (2H, d, *J* = 8.3 Hz), 7.52 (2H, d, *J* = 8.3 Hz), 5.07 (1H, q, *J* = 6.9 H), 1.83 (3H, d, *J* = 6.9 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 147.7, 132.4, 127.3, 118.4, 111.9, 57.2, 26.2
IR (neat, cm^{-1}) ν: 2981, 2360, 2229, 1922, 1608, 1505, 1415, 1232, 1047, 838, 668
HRMS (ESI): *m/z* calcd for C_9H_8ClNNa $[M+Na]^+$ 188.0238, Found 188.0230

1-bromo-4-ethylbenzene (1j)



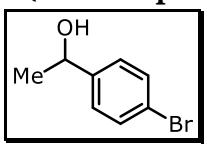
colorless liquid, R_f = 0.66 (hexane/EtOAc = 1/0)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.41 (2H, d, *J* = 8.0 Hz), 7.08 (2H, d, *J* = 8.0 Hz), 2.61 (2H, q, *J* = 7.5 Hz), 1.23 (3H, t, *J* = 7.5 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 143.1, 131.3, 129.6, 119.2, 28.3, 15.4

1-bromo-4-(1-chloroethyl)benzene (2j)



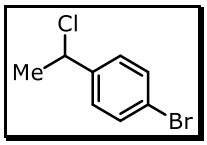
light yellow liquid, R_f = 0.52 (hexane/EtOAc = 1/0)
Yield: 200 mg, 46%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.47–7.50 (2H, m), 7.29–7.31 (2H, m), 5.04 (1H, q, *J* = 6.9 Hz), 1.82 (3H, d, *J* = 6.9 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 141.8, 131.7, 128.2, 122.1, 57.8, 26.4
IR (neat, cm^{-1}) ν: 2978, 2926, 1592, 1489, 1406, 1226, 1074, 1011, 825, 786, 717
HRMS (ESI, DART, EI): not detected

1-(4-bromophenyl)ethan-1-ol (S1j)



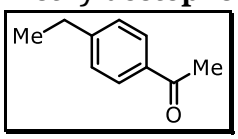
Light yellow liquid, R_f = 0.42 (hexane/EtOAc = 3/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.46 (2H, d, *J* = 7.2 Hz), 7.23 (2H, d, *J* = 7.2 Hz), 4.84 (1H, q, *J* = 6.7 Hz), 2.09 (bs, 1H), 1.45 (3H, d, *J* = 6.7 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 144.7, 131.5, 127.1, 121.1, 69.7, 25.2

1-bromo-4-(1-chloroethyl)benzene (2j, authentic sample)



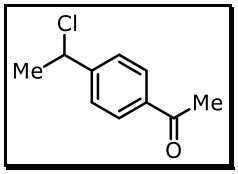
light yellow liquid, R_f = 0.52 (hexane/EtOAc = 1/0)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.49 (2H, d, *J* = 8.6 Hz), 7.30 (2H, d, *J* = 8.6 Hz), 5.04 (1H, q, *J* = 6.9 Hz), 1.83 (3H, d, *J* = 6.9 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 141.8, 131.7, 128.2, 122.1, 57.8, 26.4
IR (neat, cm^{-1}) ν: 2976, 2926, 1592, 1489, 1406, 1226, 1074, 1011, 825, 785, 717

4'-ethylacetophenone (1k)



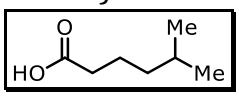
colorless liquid, R_f = 0.54 (hexane/EtOAc = 5/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.88 (2H, d, *J* = 8.3 Hz), 7.27 (2H, d, *J* = 8.3 Hz), 2.70 (2H, q, *J* = 7.5 Hz), 2.57 (3H, s), 1.25 (3H, t, *J* = 7.5 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 197.8, 150.0, 134.8, 128.5, 128.0, 28.9, 26.5, 15.1

1-(4-(1-chloroethyl)phenyl)ethan-1-one (2k)



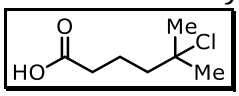
light yellow liquid, R_f = 0.44 (hexane/EtOAc = 5/1)
Yield: 274 mg, 75%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.94 (2H, d, *J* = 8.3 Hz), 7.50 (2H, d, *J* = 8.3 Hz), 5.10 (1H, q, *J* = 6.9 Hz), 2.59 (3H, s), 1.84 (3H, d, *J* = 6.9 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 197.4, 147.7, 136.8, 128.7, 126.7, 57.7, 26.6, 26.3
IR (neat, cm^{-1}) ν: 2979, 1685, 1608, 1415, 1359, 1267, 1047, 838, 683
HRMS (ESI): *m/z* calcd for $C_{10}H_{11}ClONa$ $[M+Na]^+$ 205.0391, Found 205.0397

5-methylhexanoic acid (1l)



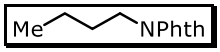
colorless liquid, R_f = 0.52 (hexane/EtOAc = 2/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 10.82 (1H, bs), 2.33 (2H, t, *J* = 7.4 Hz), 1.60–1.67 (2H, m), 1.51–1.60 (1H, m), 1.18–1.24 (2H, m), 0.88 (6H, d, *J* = 6.3 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 180.6, 38.2, 34.3, 27.7, 22.5, 22.4

5-chloro-5-methylhexanoic acid (2l)



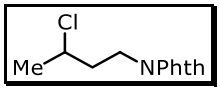
yellow liquid, R_f = 0.36 (hexane/EtOAc = 2/1)
Yield: 281 mg, 85%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.45 (1H, bs), 2.38 (2H, t, *J* = 7.2 Hz), 1.73–1.87 (4H, m), 1.56 (6H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 179.5, 70.3, 45.0, 33.8, 32.3, 20.3
IR (neat, cm^{-1}) ν: 2972, 2667, 1714, 1281, 1115, 938, 826
HRMS (ESI): *m/z* calcd for $C_7H_{13}ClO_2Na$ $[M+Na]^+$ 187.0496, Found 187.0503

2-butylisoindoline-1,3-dione (1m)



white solid, R_f = 0.60 (hexane/EtOAc = 3/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.80 (2H, dd, *J* = 5.1 Hz, 3.1 Hz), 7.67 (2H, dd, *J* = 5.1 Hz, 3.1 Hz), 3.65 (2H, t, *J* = 7.4 Hz), 1.58–1.66 (2H, m), 1.29–1.38 (2H, m), 0.91 (3H, t, *J* = 7.2 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 168.3, 133.7, 132.1, 123.0, 37.7, 30.5, 20.0, 13.6

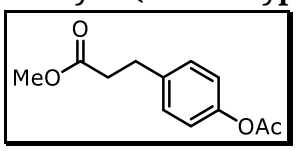
2-(3-chlorobutyl)isoindoline-1,3-dione (2m)



white solid, R_f = 0.48 (hexane/EtOAc = 3/1)
Yield: 295 mg, 62%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.79–7.85 (2H, m), 7.67–7.73 (2H, m), 3.98–4.07 (1H, m), 3.76–3.90 (2H, m), 1.99–2.13 (2H, m), 1.54 (3H, d, *J* = 6.3 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 168.2, 133.9, 132.0, 123.2, 55.5, 38.6, 35.6, 25.2
IR (neat, cm^{-1}) ν: 2975, 1773, 1712, 1397, 720

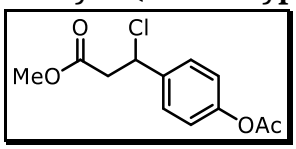
HRMS (ESI): *m/z* calcd for $C_{12}H_{12}ClNO_2Na$ $[M+Na]^+$ 260.0449, Found 260.0445

methyl 3-(4-acetoxyphenyl)propanoate (1n)



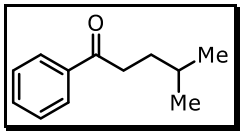
colorless liquid, R_f = 0.52 for hexane/EtOAc = 2/1; 0.48 for hexane/EtOAc = 10/1 × 4 times
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.20 (2H, d, *J* = 8.5 Hz), 6.99 (2H, d, *J* = 8.5 Hz), 3.66 (3H, s), 2.93 (2H, t, *J* = 7.8 Hz), 2.62 (2H, t, *J* = 7.8 Hz), 2.28 (3H, s)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.1, 169.6, 149.0, 138.0, 129.2, 121.5, 51.6, 35.5, 30.2, 21.0

methyl 3-(4-acetoxyphenyl)-3-chloropropanoate (2n)



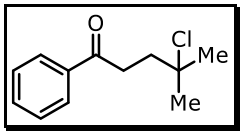
white solid, R_f = 0.52 for hexane/EtOAc = 2/1; 0.40 for hexane/EtOAc = 10/1 × 4 times
Yield: 30 mg, 58% (0.2 mmol scale)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.42 (2H, dt, *J* = 9.2 Hz, 2.5 Hz), 7.08 (2H, dt, *J* = 9.2 Hz, 2.5 Hz), 5.34 (1H, dd, *J* = 9.3 Hz, 5.7 Hz), 3.70 (3H, s), 3.16 (1H, dd, *J* = 16.2 Hz, 9.3 Hz), 3.01 (1H, dd, *J* = 16.2 Hz, 5.7 Hz), 2.29 (3H, s)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 169.9, 169.2, 150.6, 137.7, 128.1, 121.9, 57.3, 52.1, 44.6, 21.1
IR (neat, cm^{-1}) ν: 2954, 1741, 1508, 1437, 1370, 1201, 1016, 910
HRMS (ESI): *m/z* calcd for $C_{12}H_{13}ClO_4Na$ $[M+Na]^+$ 279.0395, Found 279.0394

4-methyl-1-phenylpentan-1-one (1o)[2]



colorless liquid, R_f = 0.62 (hexane/EtOAc = 10/1)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94–7.98 (2H, m), 7.52–7.57 (1H, m), 7.42–7.48 (2H, m), 2.96 (2H, t, *J* = 7.4 Hz), 1.59–1.70 (3H, m), 0.95 (6H, d, *J* = 6.3 Hz)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 200.7, 137.0, 132.8, 128.5, 128.0, 36.6, 33.2, 27.8, 22.4

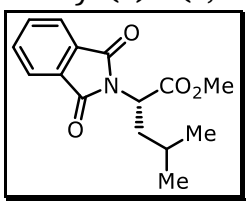
4-chloro-4-methyl-1-phenylpentan-1-one (2o)



white solid, R_f = 0.44 (hexane/EtOAc = 10/1)
Yield: 214 mg, 51%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.00 (2H, d, *J* = 7.4 Hz), 7.57 (1H, t, *J* = 7.4 Hz), 7.48 (2H, t, *J* = 7.4 Hz), 3.24 (2H, t, *J* = 7.7 Hz), 2.20 (2H, t, *J* = 7.7 Hz), 1.64 (6H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 199.4, 136.8, 133.1, 128.6, 128.1, 70.4, 39.6, 34.7, 32.6
IR (neat, cm^{-1}) ν: 2972, 1686, 1448, 1371, 1215, 1110, 742, 689
HRMS (ESI): *m/z* calcd for $C_{12}H_{15}ClONa$ $[M+Na]^+$ 233.0704, Found 233.0706

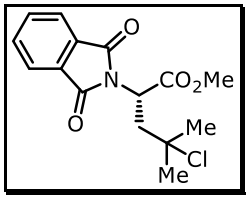
[2] Liang, Y.-F.; Zhou, X.-F.; Tang, S.-Y.; Huang, Y.-B.; Feng, Y.-S.; Xu, H.-J. *RSC Adv.* **2013**, *3*, 7739–7742.

methyl (S)-2-(1,3-dioxoisoindolin-2-yl)-4-methylpentanoate (1p)



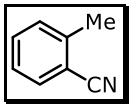
light yellow liquid, R_f = 0.37 (hexane/EtOAc = 5/1), $[\alpha]_D^{19}$ = −27.2 (c = 1.08, MeOH)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85 (2H, dd, *J* = 5.5 Hz, 3.0 Hz), 7.73 (2H, dd, *J* = 5.5 Hz, 3.0 Hz), 4.93 (1H, dd, *J* = 11.4 Hz, 4.3 Hz), 3.71 (3H, s), 2.27–2.37 (1H, m), 1.89–1.99 (1H, m), 1.40–1.53 (1H, m), 0.92 (6H, dd, *J* = 10.5 Hz, 6.5 Hz)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.2, 167.7, 134.1, 131.7, 123.5, 52.7, 50.5, 37.2, 25.0, 23.1, 20.9

methyl (S)-4-chloro-2-(1,3-dioxoisoindolin-2-yl)-4-methylpentanoate (2p)



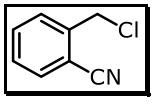
white solid, R_f = 0.34 (hexane/EtOAc = 3/1), $[\alpha]_D^{19}$ = −24.7 (c = 0.930, MeOH)
Yield: 585 mg, 94%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.83–7.90 (2H, m), 7.71–7.76 (2H, m), 5.20 (1H, dd, *J* = 9.6 Hz, 2.9 Hz), 3.70–3.74 (3H, m), 2.69–2.85 (2H, m), 1.64 (3H, s), 1.55 (3H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 169.6, 167.6, 134.2, 131.6, 123.6, 68.1, 53.1, 49.2, 42.8, 33.2, 31.9
IR (neat, cm^{-1}) ν: 2977, 1746, 1715, 1389, 1271, 1138, 720
HRMS (ESI): *m/z* calcd for $C_{15}H_{16}ClNO_4Na$ $[M+Na]^+$ 332.0660, Found 332.0664

2-methylbenzonitrile (1q)



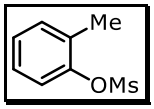
colorless liquid, R_f = 0.65 (hexane/EtOAc = 5/1)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58 (1H, dd, *J* = 7.6 Hz, 0.9 Hz), 7.47 (1H, td, *J* = 7.7 Hz, 1.2 Hz), 7.31 (1H, dd, *J* = 7.9 Hz, 0.6 Hz), 7.24–7.28 (1H, m), 2.54 (3H, s)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.9, 132.6, 132.4, 130.2, 126.2, 118.1, 112.7, 20.4

2-(chloromethyl)benzonitrile (2q)



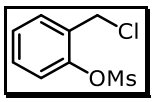
light yellow solid, R_f = 0.50 (hexane/EtOAc = 5/1)
Yield: 128 mg, 42%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.67 (1H, d, *J* = 7.6 Hz), 7.56–7.65 (2H, m), 7.44 (1H, td, *J* = 7.4 Hz, 1.6 Hz), 4.75 (2H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 140.6, 133.2, 133.0, 130.0, 128.9, 116.7, 112.2, 43.1
IR (neat, cm^{-1}) ν: 3021, 2225, 1597, 1489, 1452, 1271, 1217, 832, 754, 673
HRMS (ESI): *m/z* calcd for C_8H_6ClNNa $[M+Na]^+$ 174.0081, Found 174.0074

***o*-tolyl methanesulfonate (1r)**



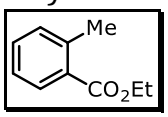
colorless liquid, R_f = 0.50 (hexane/EtOAc = 3/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.19–7.30 (4H, m), 3.18 (3H, s), 2.37 (3H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 147.8, 131.8, 131.2, 127.2, 122.0, 38.0, 16.5

2-(chloromethyl)phenyl methanesulfonate (2r)



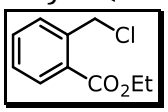
colorless liquid, R_f = 0.48 (hexane/EtOAc = 3/1)
Yield: 14 mg, 32% (0.2 mmol scale)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.52 (1H, dd, *J* = 7.6 Hz, 1.7 Hz), 7.37–7.44 (2H, m), 7.33 (1H, td, *J* = 7.3 Hz, 16 Hz), 4.69 (2H, s), 3.27 (3H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 146.9, 131.5, 130.4, 130.2, 127.6, 122.3, 40.5, 38.3
IR (neat, cm^{-1}) ν: 3030, 2939, 1585, 1490, 1363, 1153, 1090, 970, 876, 805, 670
HRMS (ESI): *m/z* calcd for $C_8H_9ClO_3SNa$ $[M+Na]^+$ 242.9853, Found 242.9861

ethyl 2-methylbenzoate (1s)



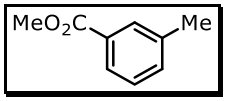
colorless liquid, R_f = 0.56 (hexane/EtOAc = 10/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.91 (1H, dd, *J* = 8.0 Hz, 1.3 Hz), 7.39 (1H, td, *J* = 7.5 Hz, 1.3 Hz), 7.22–7.27 (2H, m), 4.36 (2H, q, *J* = 7.2 Hz), 2.60 (3H, s), 1.39 (3H, t, *J* = 7.2 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 167.7, 139.9, 131.8, 131.6, 130.4, 129.9, 125.6, 60.6, 21.7, 14.3

ethyl 2-(chloromethyl)benzoate (2s)



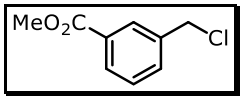
light yellow liquid, R_f = 0.42 (hexane/EtOAc = 10/1)
Yield: 177 mg, 45%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.98 (1H, d, *J* = 7.4 Hz), 7.49–7.55 (2H, m), 7.37–7.41 (1H, m), 5.04 (2H, s), 4.40 (2H, q, *J* = 7.2 Hz), 1.41 (3H, t, *J* = 7.2 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.7, 138.6, 132.3, 131.0, 130.8, 129.4, 128.3, 61.2, 44.4, 14.2
IR (neat, cm^{-1}) ν: 2981, 1716, 1602, 1448, 1366, 1269, 1131, 1080, 714, 679
HRMS (ESI): *m/z* calcd for $C_{10}H_{11}ClO_2Na$ $[M+Na]^+$ 221.0340, Found 221.0331

methyl 3-methylbenzoate (1t)



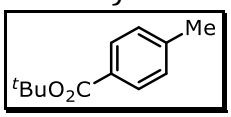
colorless liquid, R_f = 0.52 (hexane/EtOAc = 10/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.82–7.87 (2H, m), 7.30–7.38 (2H, m), 3.91 (3H, s), 2.40 (3H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 167.3, 138.1, 133.6, 130.1, 130.0, 128.2, 126.7, 52.0, 21.2

methyl 3-(chloromethyl)benzoate (2t)



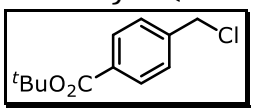
light yellow liquid, R_f = 0.40 (hexane/EtOAc = 10/1)
Yield: 14 mg, 38% (0.2 mmol scale)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.07 (1H, bs), 8.00 (1H, d, *J* = 7.4 Hz), 7.59 (1H, d, *J* = 7.4 Hz), 7.45 (1H, t, *J* = 7.7 Hz), 4.62 (2H, s), 3.93 (3H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.6, 137.8, 133.0, 130.7, 129.6, 129.5, 128.9, 52.2, 45.5
IR (neat, cm^{-1}) ν: 2952, 1724, 1591, 1434, 1291, 1205, 1108, 990, 759, 708
HRMS (ESI): *m/z* calcd for $C_9H_9ClO_2Na$ $[M+Na]^+$ 207.0183, Found 207.0174

***tert*-butyl 4-methylbenzoate (1u)**



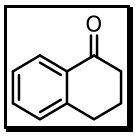
colorless liquid, R_f = 0.48 (hexane/EtOAc = 10/1)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.88 (2H, d, *J* = 8.3 Hz), 7.21 (2H, d, *J* = 8.3 Hz), 2.39 (3H, s), 1.59 (9H, s)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.9, 142.9, 129.4, 129.2, 128.8, 80.6, 28.2, 21.5

***tert*-butyl 4-(chloromethyl)benzoate (2u)**



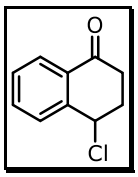
white solid, R_f = 0.42 (hexane/EtOAc = 10/1)
Yield: 17 mg, 36% (0.2 mmol scale)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.98 (2H, d, *J* = 8.0 Hz), 7.43 (2H, d, *J* = 8.0 Hz), 4.60 (2H, s), 1.59 (9H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 165.2, 141.7, 132.0, 129.8, 128.3, 81.2, 45.4, 28.1
IR (neat, cm^{-1}) ν: 2979, 1714, 1612, 1414, 1368, 1294, 1167, 1119, 1019, 849, 713
HRMS (ESI): *m/z* calcd for $C_{12}H_{15}ClO_2Na$ $[M+Na]^+$ 249.0653, Found 249.0652

1-tetralone (1v)



yellow liquid, R_f = 0.38 (hexane/EtOAc = 5/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.02 (1H, d, *J* = 8.0 Hz), 7.45 (1H, td, *J* = 7.4 Hz, 1.1 Hz), 7.29 (1H, t, *J* = 7.4 Hz), 7.24 (1H, d, *J* = 8.0 Hz), 2.95 (2H, t, *J* = 6.3 Hz), 2.64 (2H, t, *J* = 6.3 Hz), 2.09–2.16 (2H, m)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 198.3, 144.4, 133.3, 132.5, 128.7, 127.0, 126.5, 39.1, 29.6, 23.2

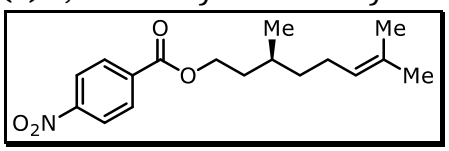
4-chloro-1-tetralone (2v)



yellow liquid, R_f = 0.34 (hexane/EtOAc = 5/1)
Yield: 219 mg, 61%

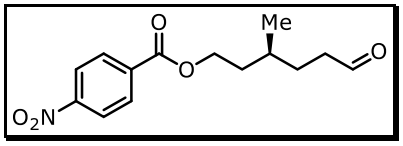
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05 (1H, d, *J* = 8.6 Hz), 7.58 (1H, t, *J* = 8.3 Hz), 7.42–7.51 (2H, m), 5.38 (1H, t, *J* = 4.0 Hz), 3.11–3.20 (1H, m), 2.49–2.74 (3H, m)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 196.3, 142.2, 134.1, 131.0, 129.3, 129.0, 127.3, 56.6, 33.9, 31.8
IR (neat, cm^{-1}) ν: 2959, 1687, 1599, 1455, 1285, 1234, 921, 881, 777, 720
HRMS (ESI): *m/z* calcd for $C_{10}H_9ClONa$ $[M+Na]^+$ 203.0234, Found 203.0234

(*S*)-3,7-dimethyloct-6-en-1-yl 4-nitrobenzoate (S1w)



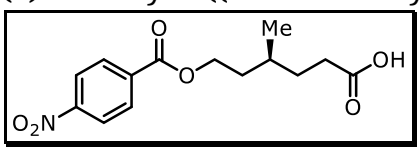
yellow liquid, R_f = 0.60 (hexane/EtOAc = 5/1), $[\alpha]_D^{22}$ = −1.9 (c = 1.45, MeOH)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.27 (2H, d, *J* = 8.9 Hz), 8.19 (2H, d, *J* = 8.9 Hz), 5.07 (1H, t, *J* = 7.2 Hz), 4.34–4.46 (2H, m), 1.92–2.07 (2H, m), 1.78–1.87 (1H, m), 1.55–1.71 (2H, m), 1.65 (3H, s), 1.59 (3H, s), 1.34–1.44 (1H, m), 1.19–1.30 (1H, m), 0.97 (3H, d, *J* = 6.3 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 164.6, 150.4, 135.8, 131.4, 130.6, 124.4, 123.4, 64.4, 36.8, 35.3, 29.5, 25.6, 25.3, 19.4, 17.6
IR (neat, cm^{-1}) ν: 2925, 1726, 1608, 1529, 1349, 1276, 1102, 719
HRMS (ESI): *m/z* calcd for $C_{17}H_{23}NO_4Na$ $[M+Na]^+$ 328.1519, Found 328.1523

(*S*)-3-methyl-6-oxohexyl 4-nitrobenzoate (S2w)



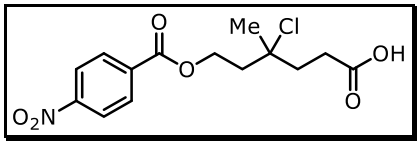
yellow liquid, R_f = 0.48 (hexane/EtOAc = 2/1), $[\alpha]_D^{21}$ = −3.9 (c = 2.17, MeOH)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.77 (1H, s), 8.26 (2H, d, *J* = 8.0 Hz), 8.17 (2H, d, *J* = 8.0 Hz), 4.36–4.46 (2H, m), 2.42–2.55 (2H, m), 1.59–1.87 (4H, m), 1.47–1.56 (1H, m), 0.97 (3H, d, *J* = 6.3 Hz)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 202.2, 164.6, 150.4, 135.6, 130.6, 123.5, 64.0, 41.4, 35.2, 29.5, 28.6, 19.1
IR (neat, cm^{-1}) ν: 2960, 2723, 1724, 1607, 1528, 1350, 1278, 1104, 875, 720
HRMS (ESI): *m/z* calcd for $C_{14}H_{17}NO_5Na$ $[M+Na]^+$ 302.0999, Found 302.0995

(*S*)-4-methyl-6-((4-nitrobenzoyl)oxy)hexanoic acid (1w)



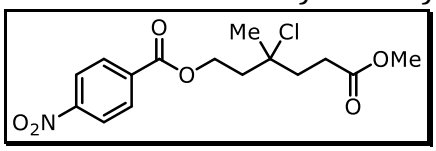
yellow solid, R_f = 0.40 (hexane/EtOAc = 1/1), $[\alpha]_D^{21}$ = 0 (c = 1.69, MeOH)
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.28 (2H, d, *J* = 8.5 Hz), 8.19 (2H, d, *J* = 8.5 Hz), 4.35–4.48 (2H, m), 2.31–2.50 (2H, m), 1.47–1.90 (5H, m), 0.99 (3H, d, *J* = 6.3 Hz)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.0, 164.7, 150.4, 135.7, 130.6, 123.5, 64.0, 35.1, 31.5, 31.3, 29.3, 19.0
IR (neat, cm^{-1}) ν: 3112, 2961, 1723, 1608, 1528, 1350, 1277, 1103, 876, 719
HRMS (ESI): *m/z* calcd for $C_{14}H_{17}NO_6Na$ $[M+Na]^+$ 318.0948, Found 318.0938

DL-4-chloro-4-methyl-6-((4-nitrobenzoyl)oxy)hexanoic acid (2w)



white solid, R_f = 0.32 (hexane/EtOAc = 1/1)
Yield: 38.4 mg (0.3 mmol scale), 37%
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.28 (2H, d, *J* = 8.6 Hz), 8.20 (2H, d, *J* = 8.6 Hz), 4.63 (2H, t, *J* = 6.3 Hz), 2.59–2.73 (2H, m), 2.10–2.36 (4H, m), 1.64 (3H, s)
^{13}C NMR (126 MHz, $CDCl_3$) δ: 178.9, 164.5, 150.5, 135.3, 130.7, 123.5, 70.3, 62.5, 42.3, 38.7, 29.7, 29.6
IR (neat, cm^{-1}) ν: 3426, 1724, 1608, 1528, 1413, 1350, 1276, 1104, 874, 719
HRMS (ESI): *m/z* calcd for $C_{14}H_{16}ClNO_6Na$ $[M+Na]^+$ 352.0558, Found 352.0566

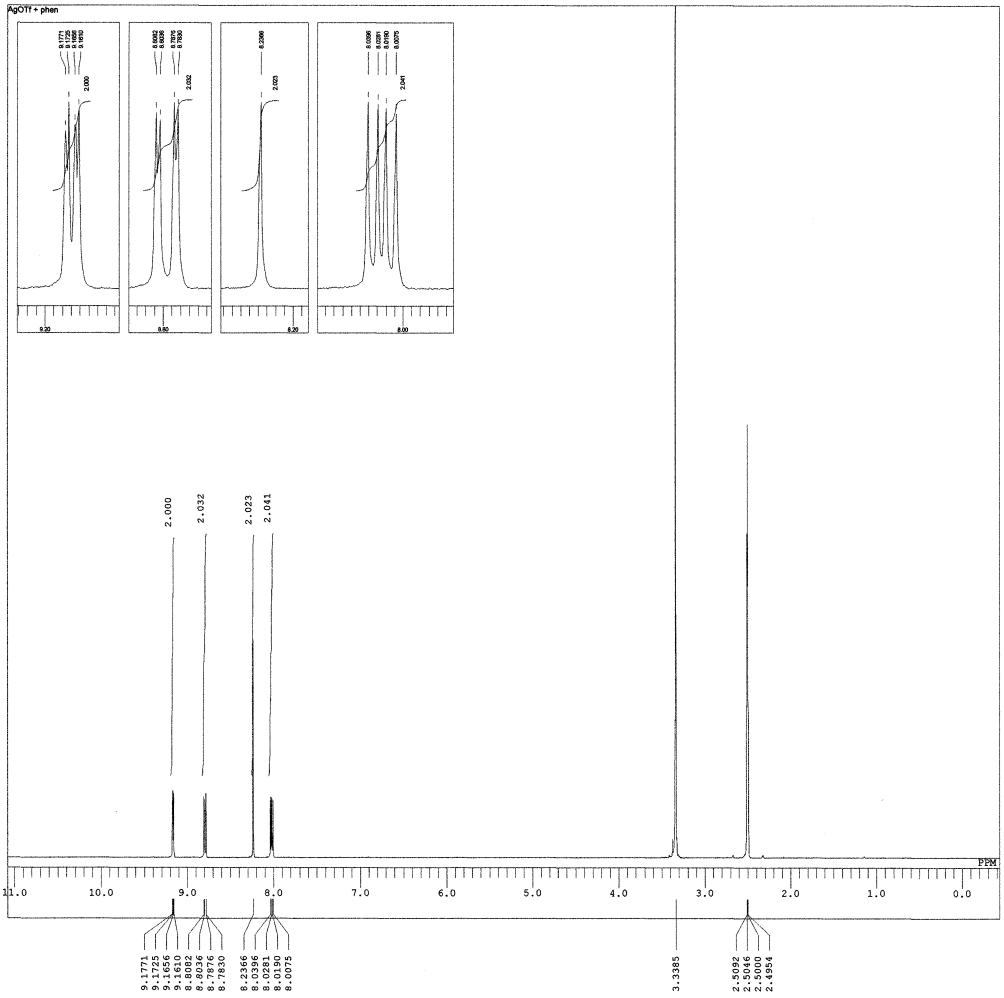
DL-3-chloro-6-methoxy-3-methyl-6-oxohexyl 4-nitrobenzoate (2x)



light yellow liquid, R_f = 0.50 (hexane/EtOAc = 2/1)
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.28 (2H, d, *J* = 8.3 Hz), 8.19 (2H, d, *J* = 8.3 Hz), 4.60 (2H, t, *J* = 6.6 Hz), 3.68 (3H, s), 2.52–2.63 (2H, m), 2.09–2.34 (4H, m), 1.61 (3H, s)
^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.2, 164.4, 150.5, 135.3, 130.6, 123.5, 70.4, 62.4, 51.8, 42.2, 39.0, 29.58, 29.55
IR (neat, cm^{-1}) ν: 2953, 1730, 1607, 1530, 1436, 1271, 1102, 874, 718
HRMS (ESI): *m/z* calcd for $C_{15}H_{18}ClNO_6Na$ $[M+Na]^+$ 366.0715, Found 366.0706

10. NMR spectra

A ^{1}H NMR spectrum for $Ag(phen)_2OTf$, and ^{1}H and ^{13}C NMR spectra for the compounds in **9. Analytical data** are appended below.

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CMNT   AgOTf + phen
DATIM  24-08-2015 15:11:50
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         16384
FREQU       7352.94 Hz
SCANS             8
ACQTM          2.2282 sec
PD             5.0000 sec
PW1              4.99 usec
IRNUC  1H
CTEMP            21.7 c
SLVNT  DMSO
EXREF            2.50 ppm
BF               0.12 Hz
RGAIN              44
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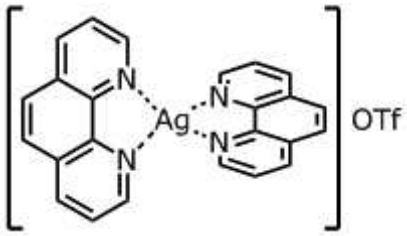

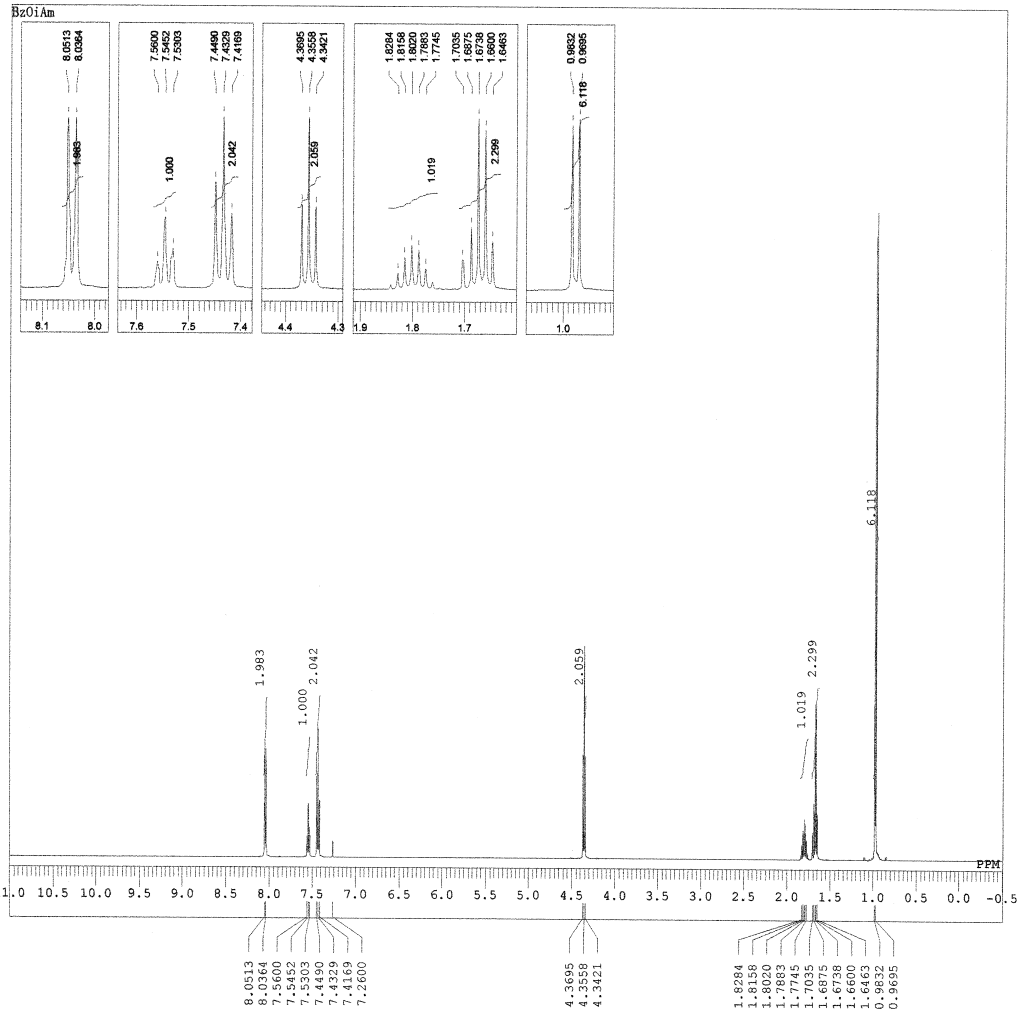


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EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           21.2 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
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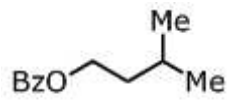
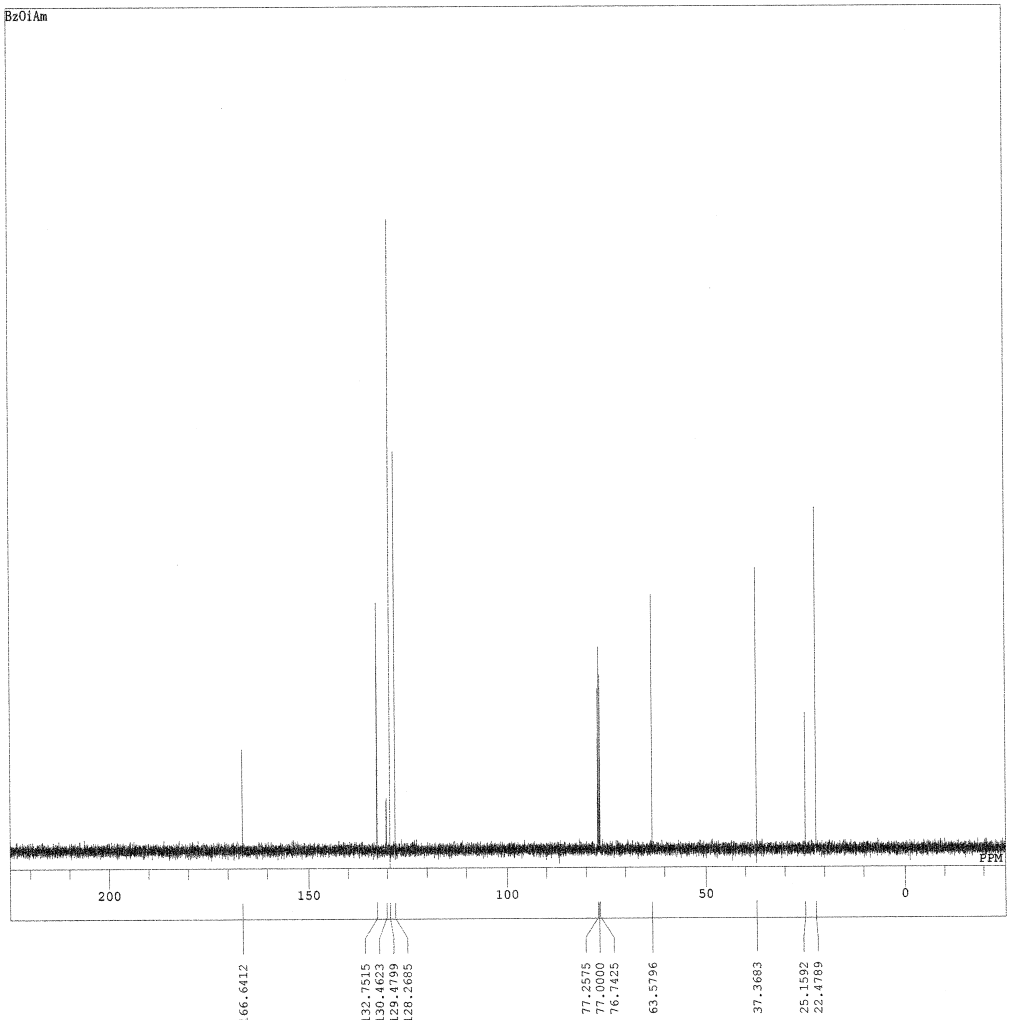


1a

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EXMOD   carbon.jxp
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OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU       39308.18 Hz
SCANS             20
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           21.4 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
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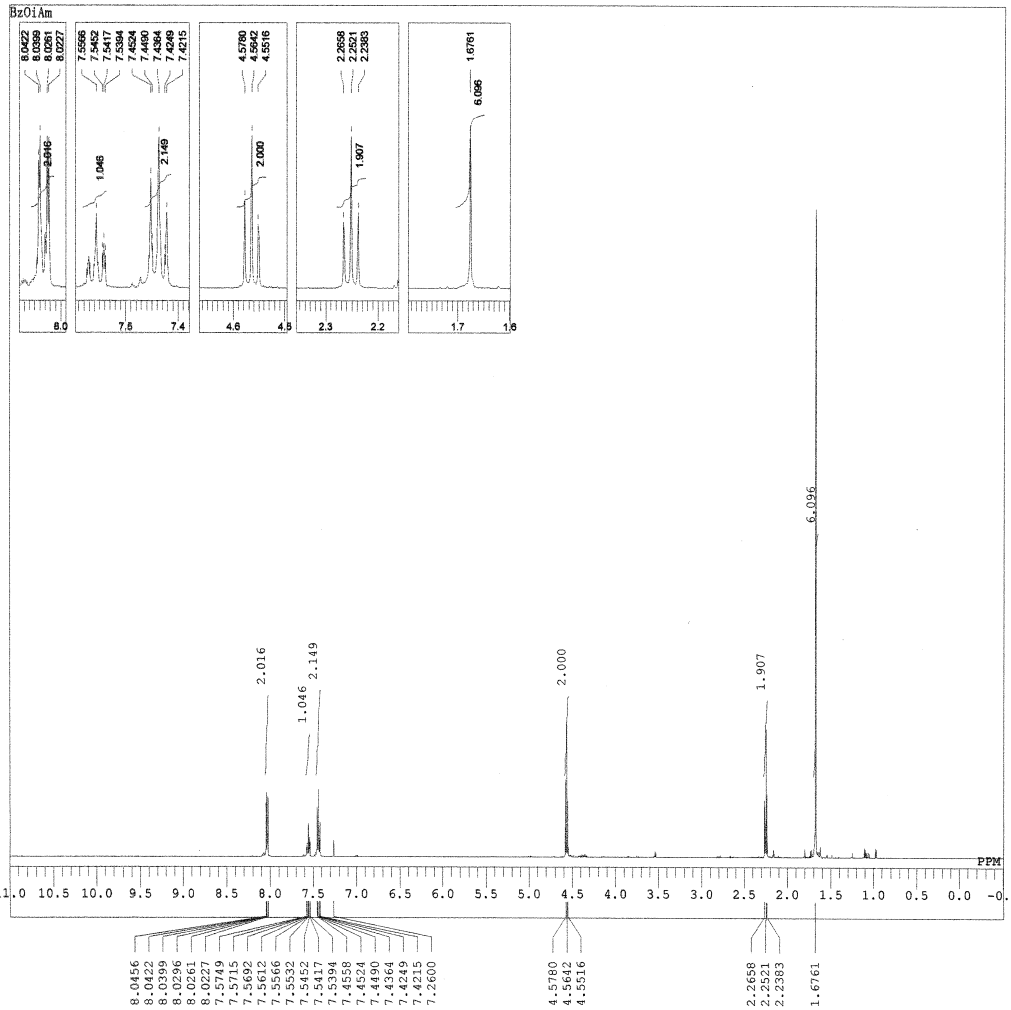


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OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS              4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           21.2 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
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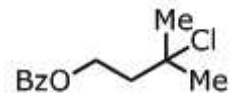
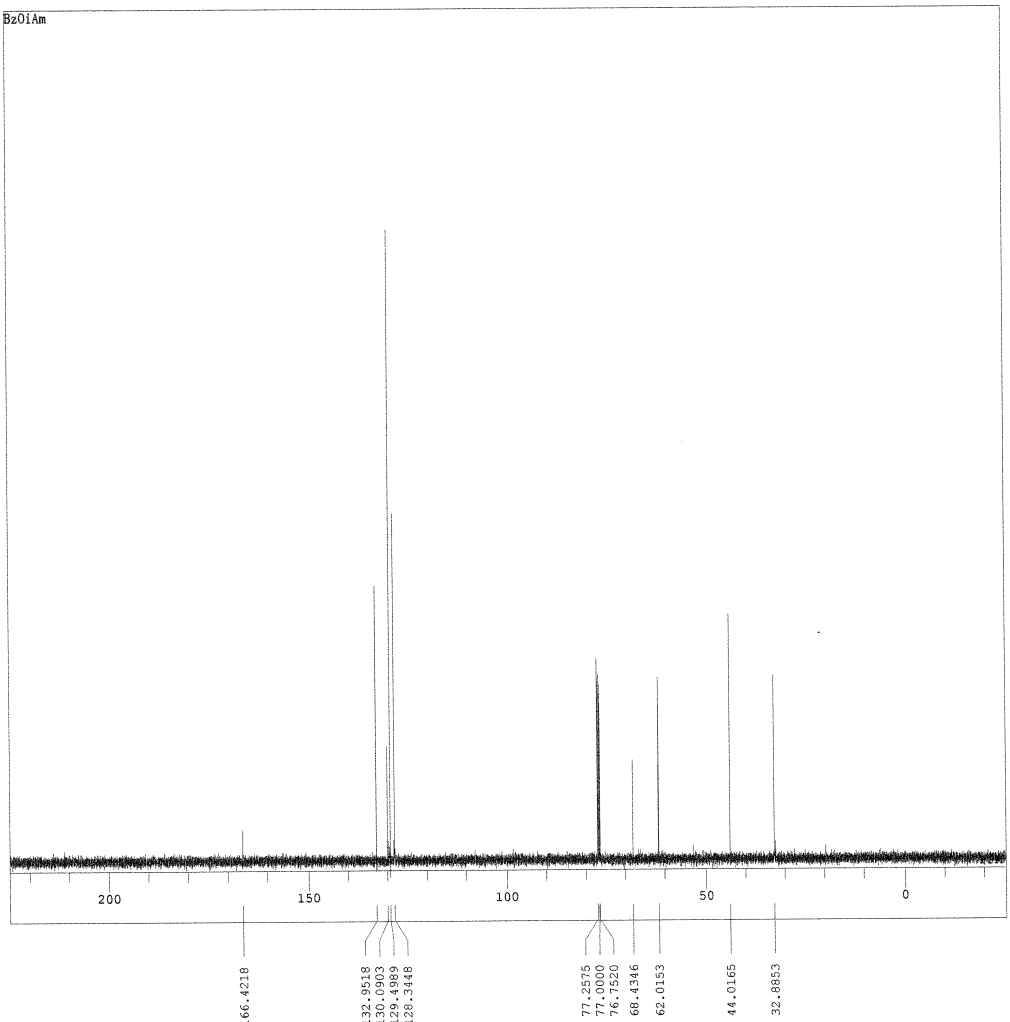


2a

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EXMOD   carbon.jxp
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OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU        39308.18 Hz
SCANS             32
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           21.4 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
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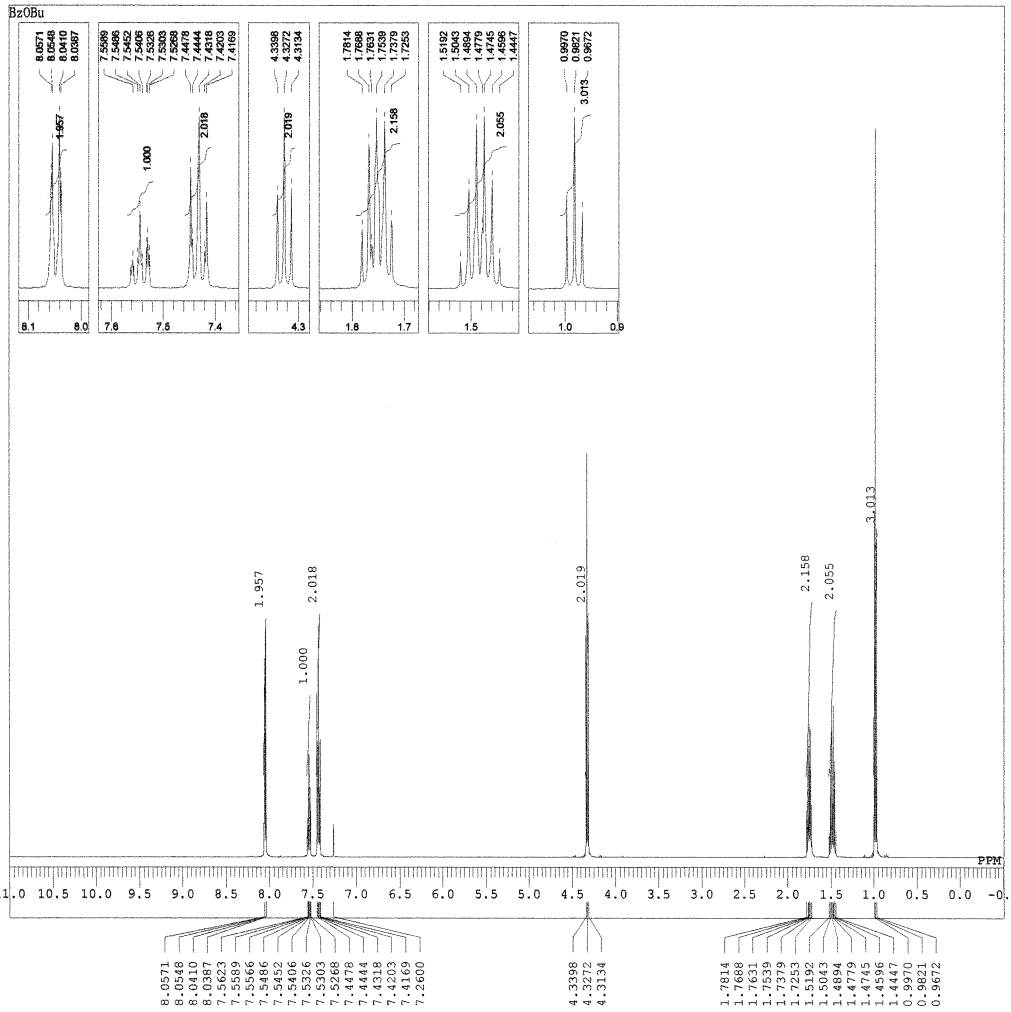


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EXMOD   proton.jxp
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OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS                4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           22.8 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             28
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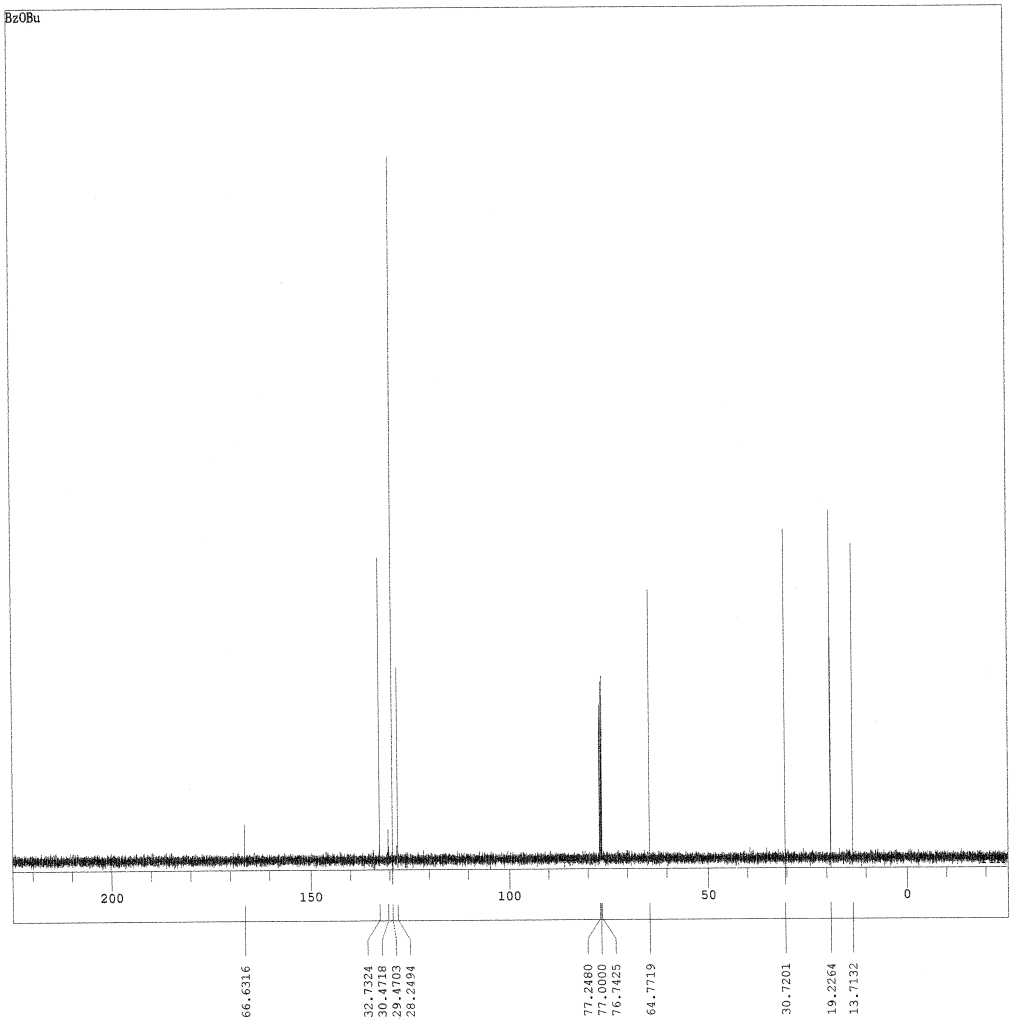
1b

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EXMOD   carbon.jxp
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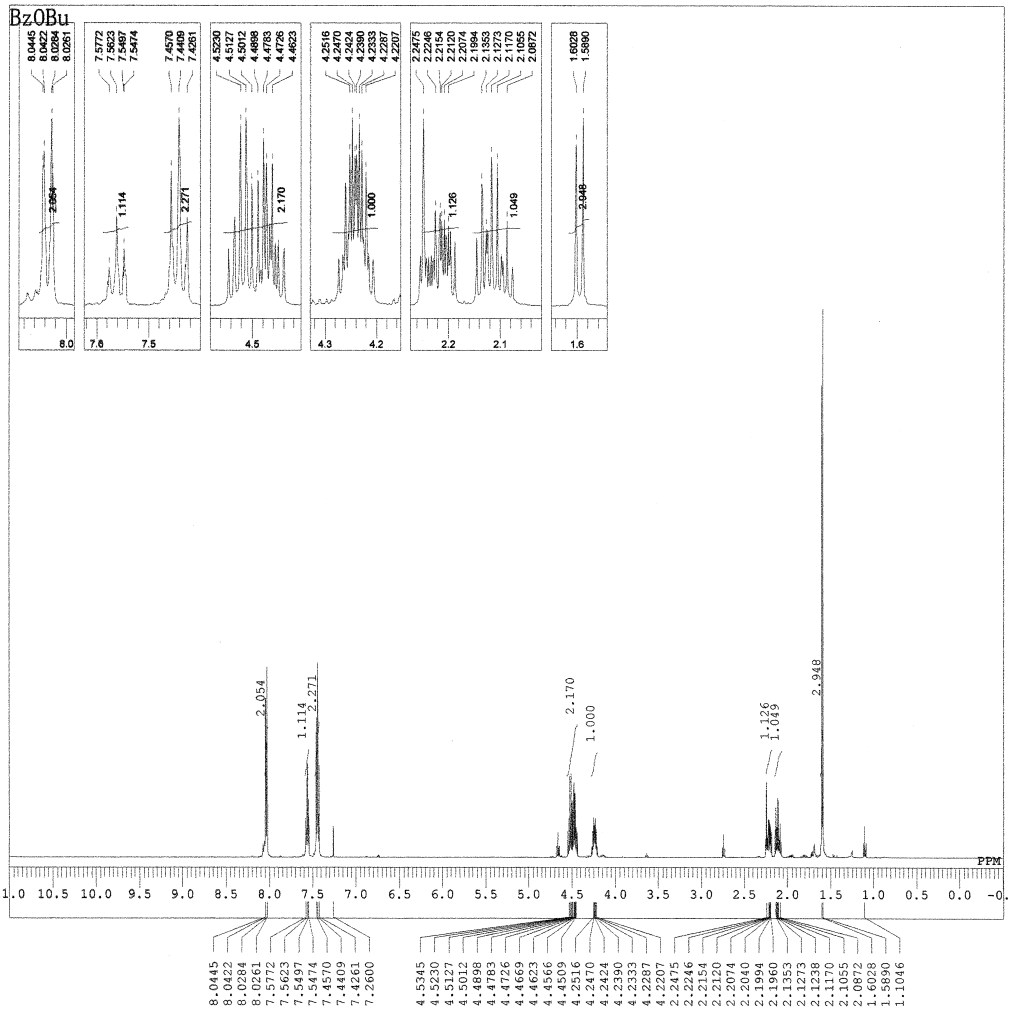
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU       39308.18 Hz
SCANS              28
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           22.5 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```

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OBNUC   1H
EXMOD   proton.jxp
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OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           21.8 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
```
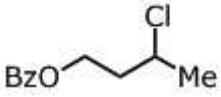
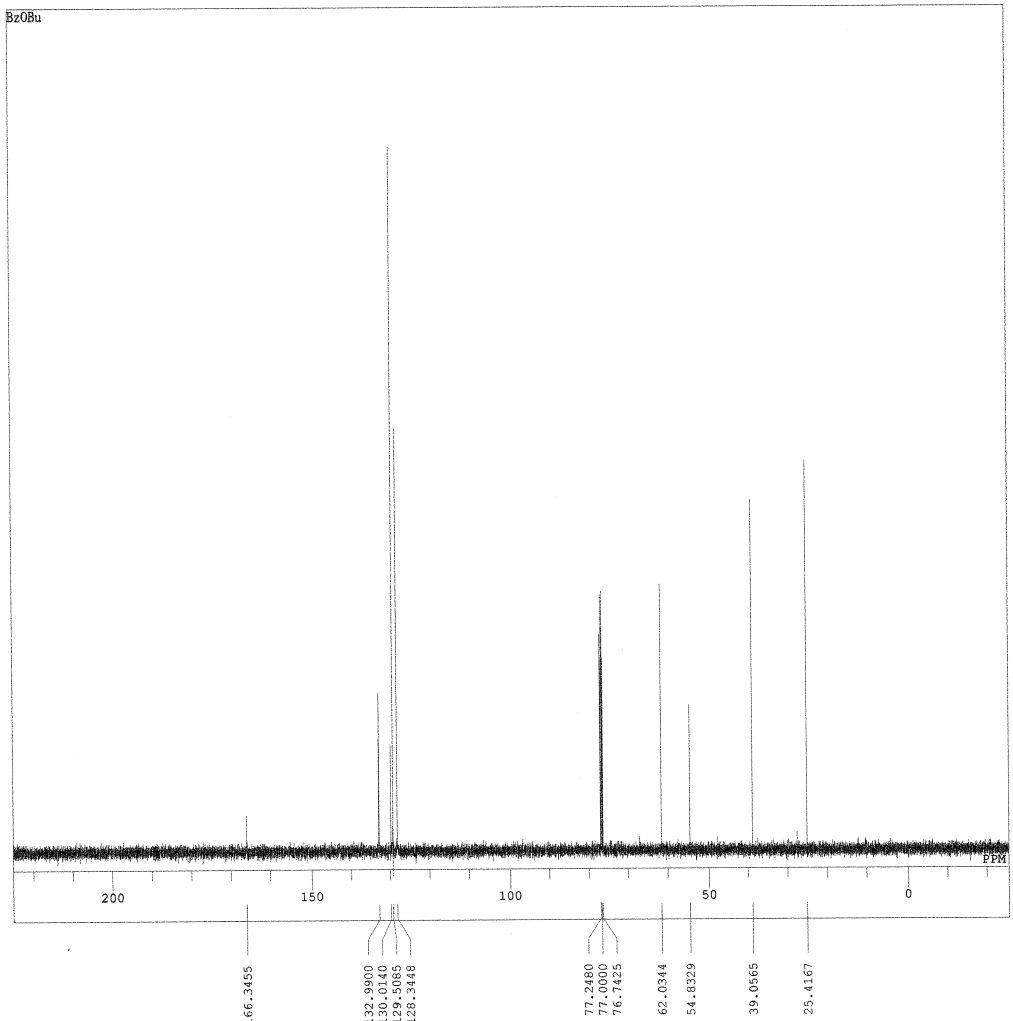
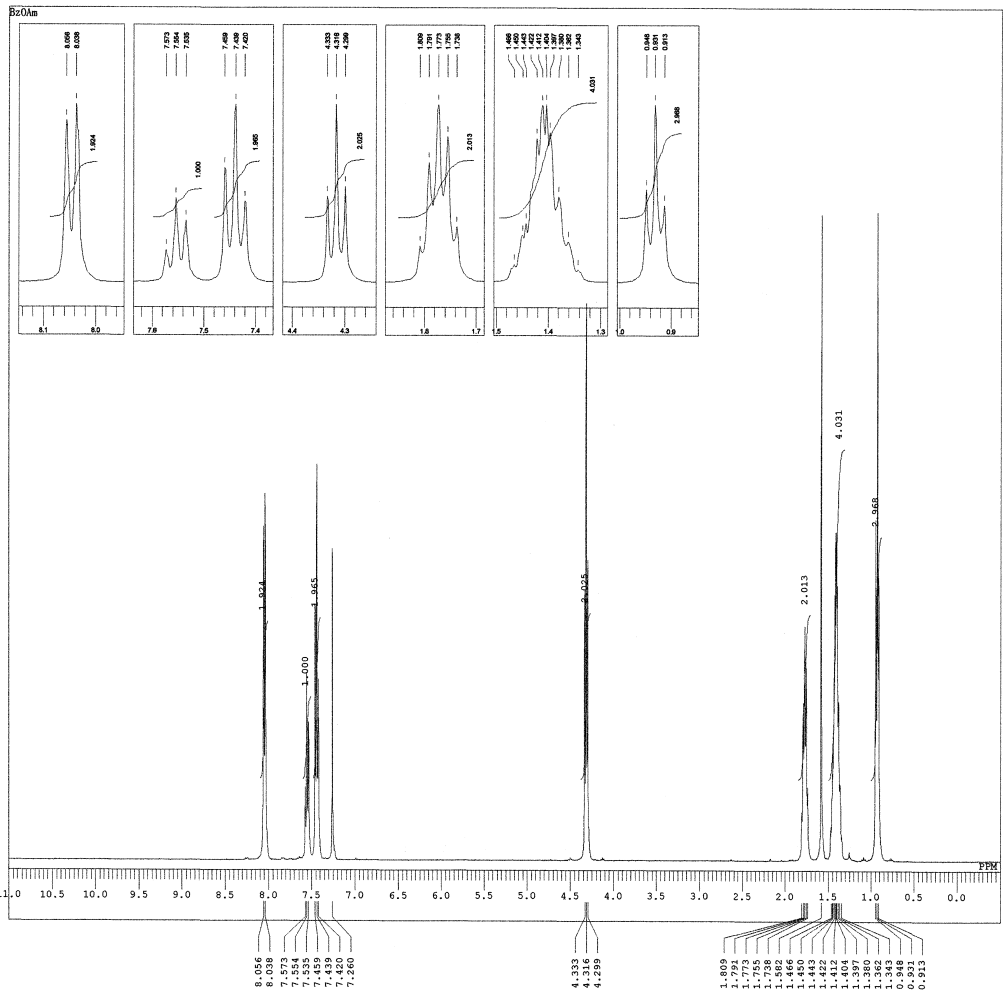



2b

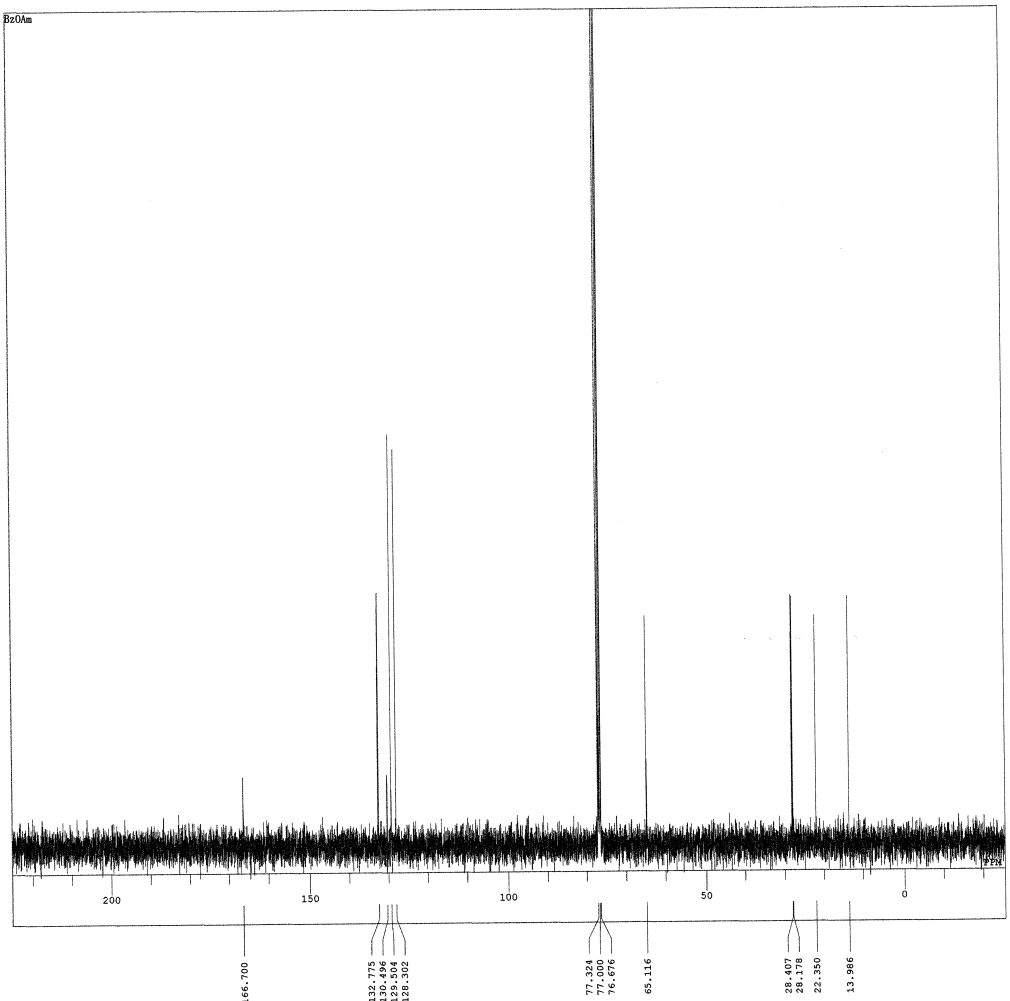



```
DFILE   ozawa08-141_13C.jdf
COMNT   BzOBu
DATIM   2015-09-29 12:05:09
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU        39308.18 Hz
SCANS             40
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           21.9 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```

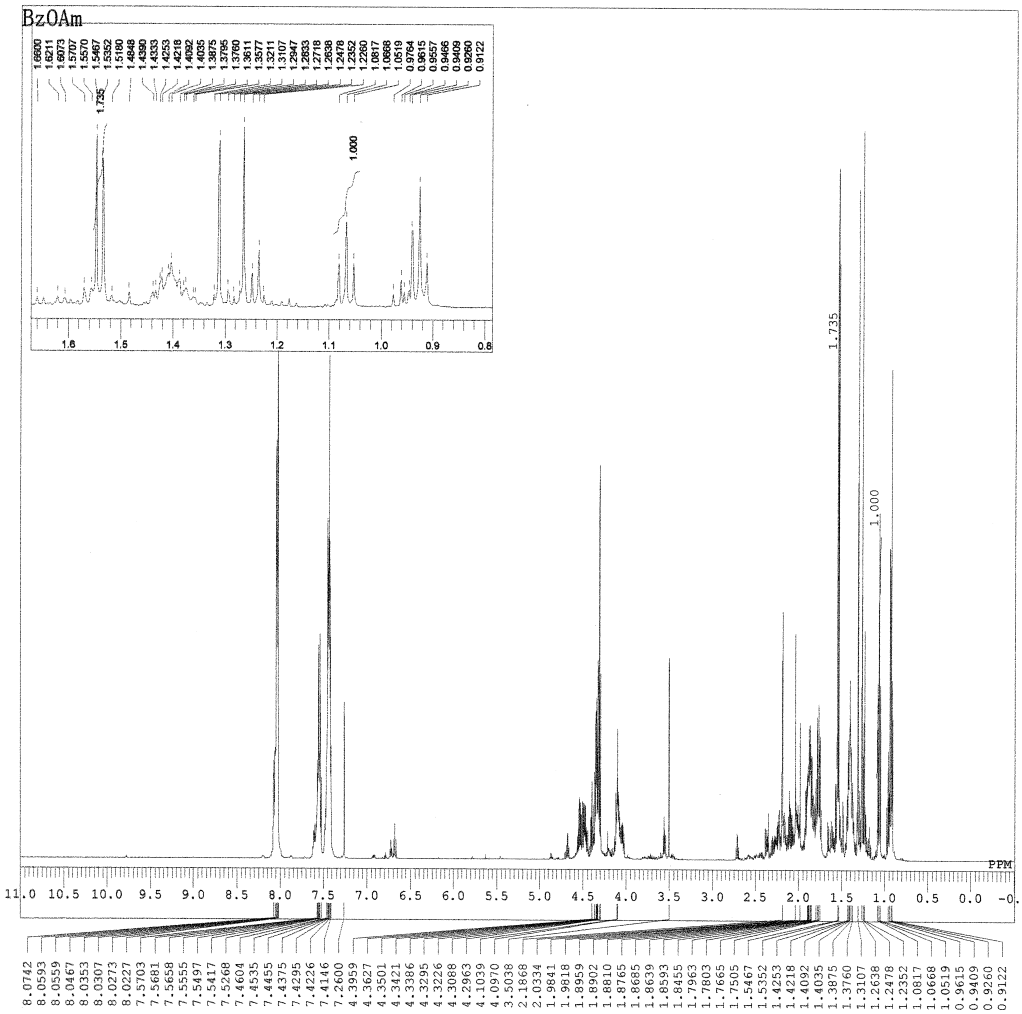



```
DFILE  ozawa08-138_1H.als
COMNT  BzOAm
DATIM  25-09-2015 15:54:13
OBNUC  1H
EXMOD  proton.jxp
OBFRQ          391.78 MHz
OBSET            8.51 KHz
OBFIN            3.34 Hz
POINT           13107
FREQU         5882.35 Hz
SCANS               4
ACQTM          2.2282 sec
PD             5.0000 sec
PW1              4.99 usec
IRNUC  1H
CTEMP           20.4 c
SLVNT  CDCL3
EXREF           7.26 ppm
BF              1.20 Hz
RGAIN             40
```

1c

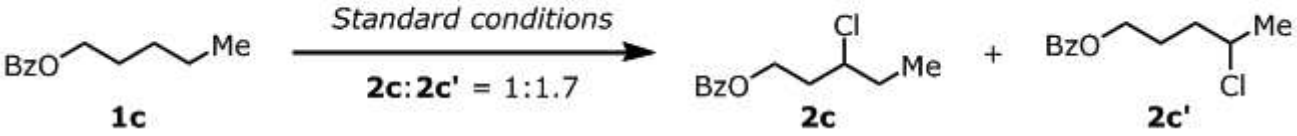



```
DFILE  ozawa08-138_13C.jdf
COMNT  BzOAm
DATIM  25-09-2015 16:08:53
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ           98.52 MHz
OBSET            4.64 KHz
OBFIN            9.74 Hz
POINT           32767
FREQU        30788.18 Hz
SCANS             128
ACQTM          1.0643 sec
PD             2.0000 sec
PW1              3.16 usec
IRNUC  1H
CTEMP           20.5 c
SLVNT  CDCL3
EXREF           77.00 ppm
BF              1.20 Hz
RGAIN             60
```

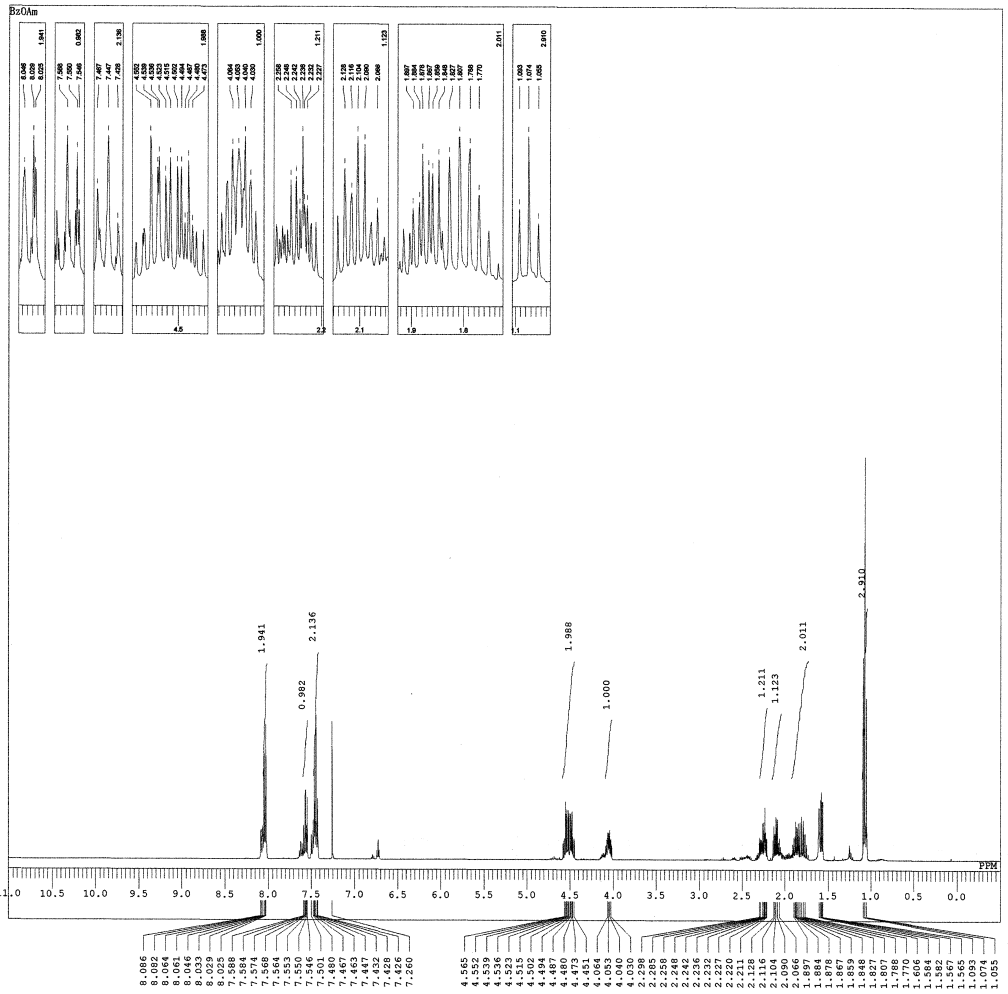



```
DFILE   ozawa08-147_C.jdf
COMNT   BzOAm
DATIM   2015-10-03 10:00:22
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             8
ACQTM         1.7459 sec
PD            5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           33.3 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             28
```

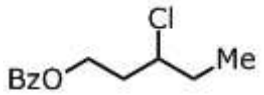
^{1}H NMR of crude mixture.



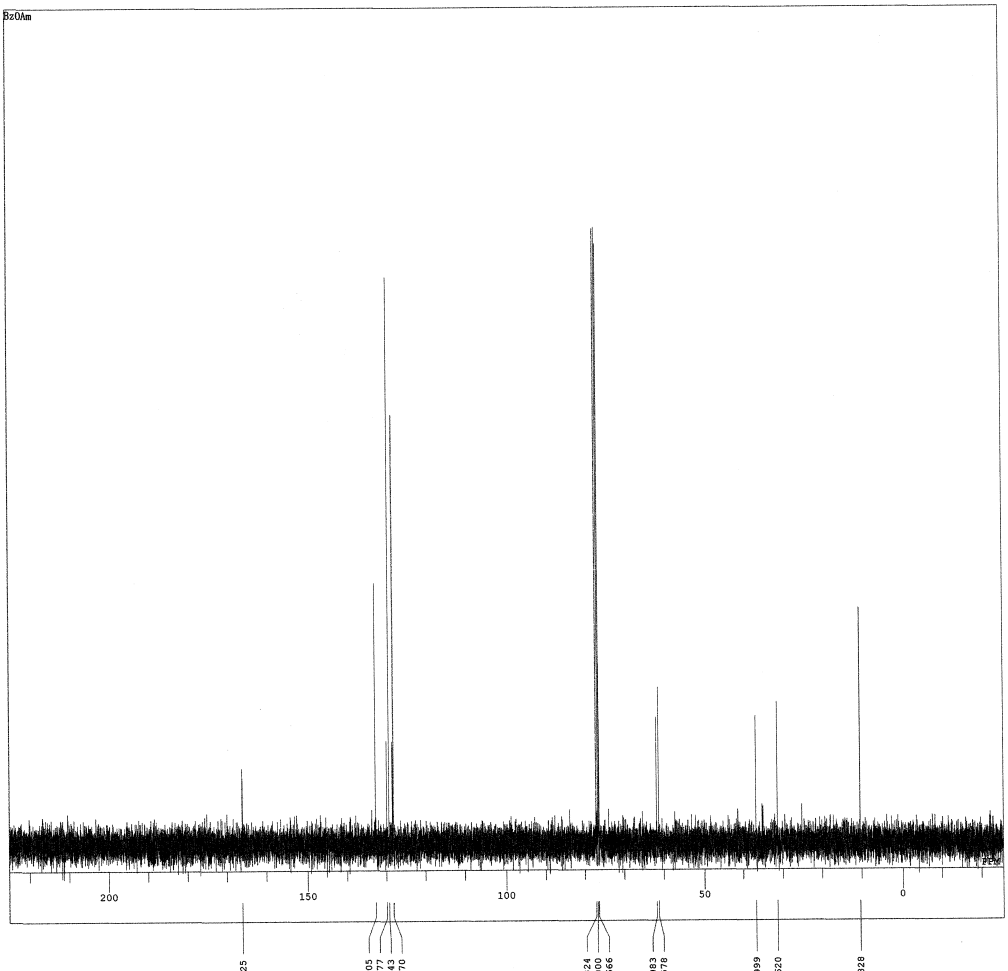
1c **2c** **2c'**



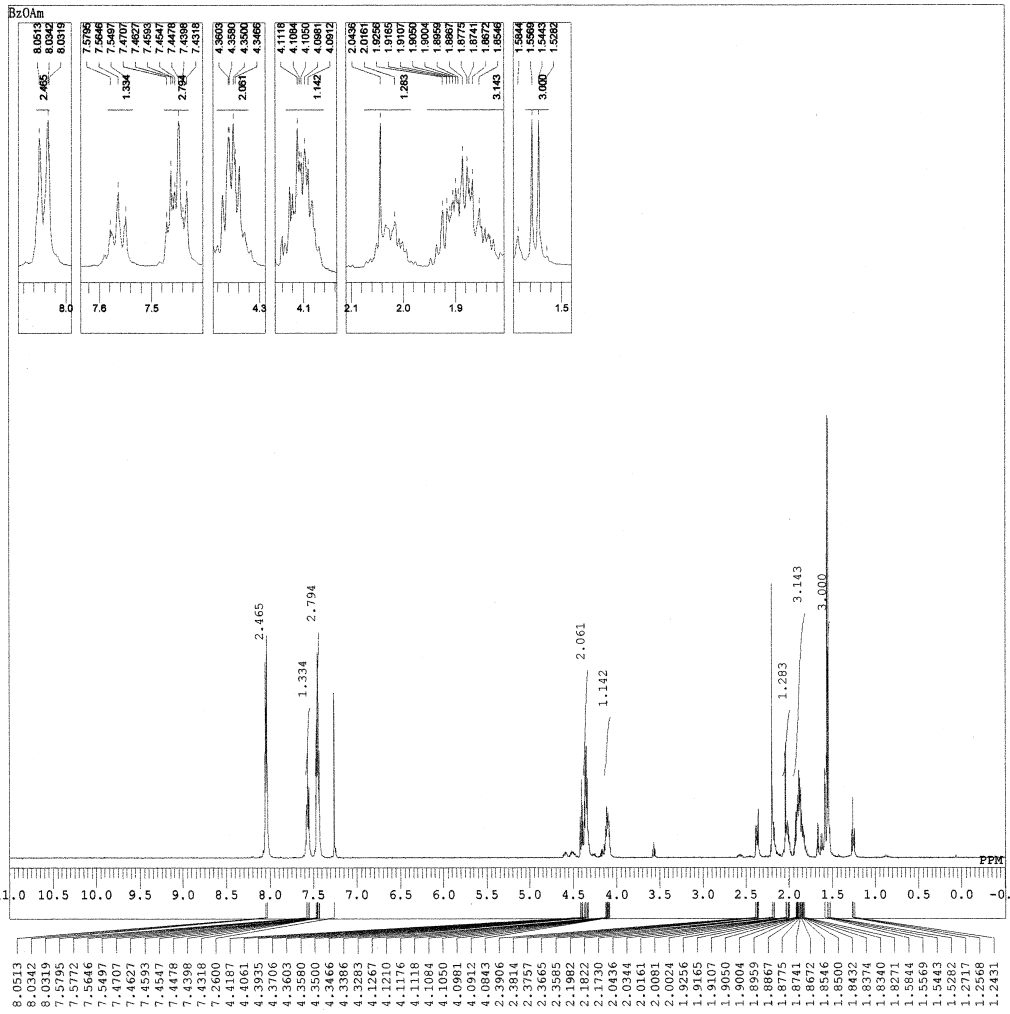

```
DFILE  ozawa08-142a_1H.jdf
COMNT  BzOAm
DATIM  01-10-2015 16:54:01
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         16384
FREQU       7352.94 Hz
SCANS             4
ACQTM         2.2282 sec
PD            5.0000 sec
PW1             4.99 usec
IRNUC  1H
CTEMP           20.4 c
SLVNT  CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             34
```



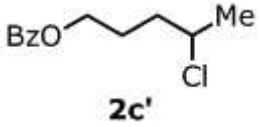
2c



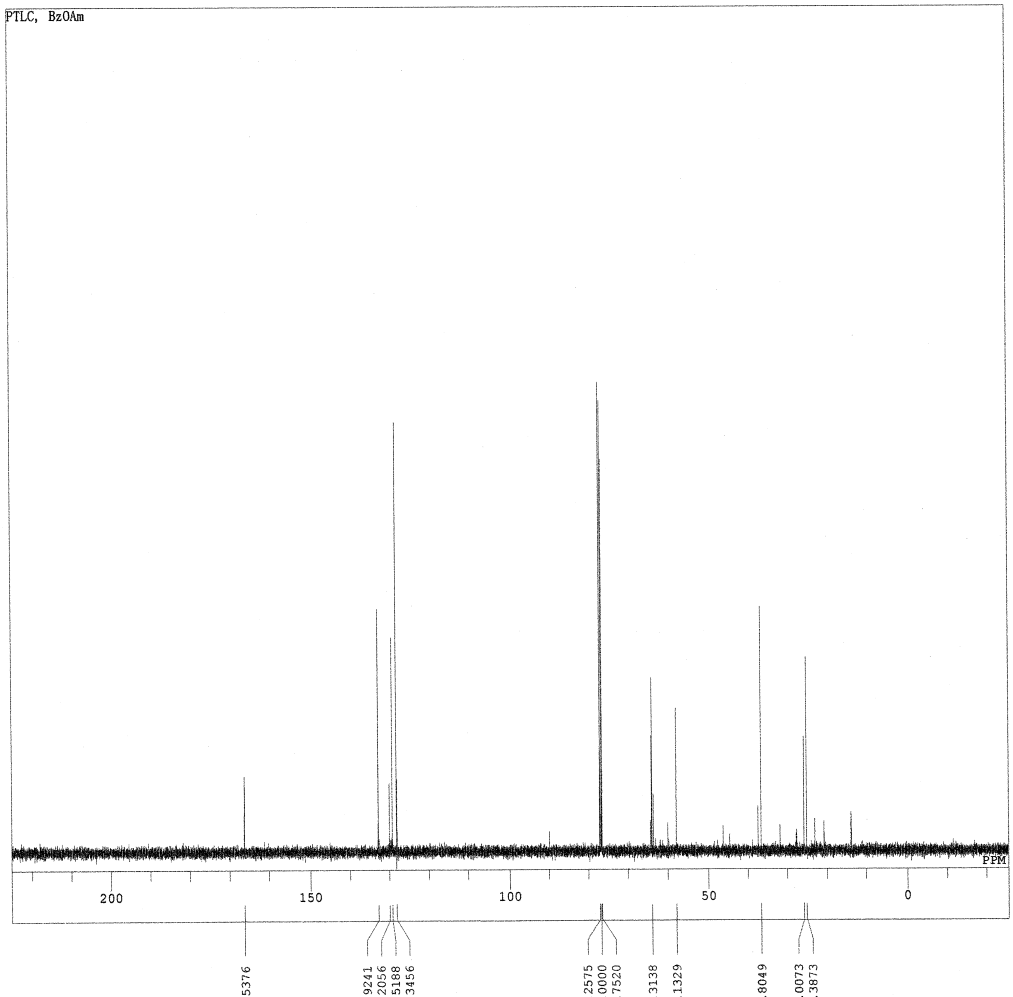

```
DFILE  ozawa08-142a_13C.als
COMNT  BzOAm
DATIM  01-10-2015 16:55:04
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         26214
FREQU      24630.54 Hz
SCANS            64
ACQTM         1.0643 sec
PD            2.0000 sec
PW1             3.16 usec
IRNUC  1H
CTEMP           20.6 c
SLVNT  CDCL3
EXREF          77.00 ppm
BF              0.12 Hz
RGAIN             60
```



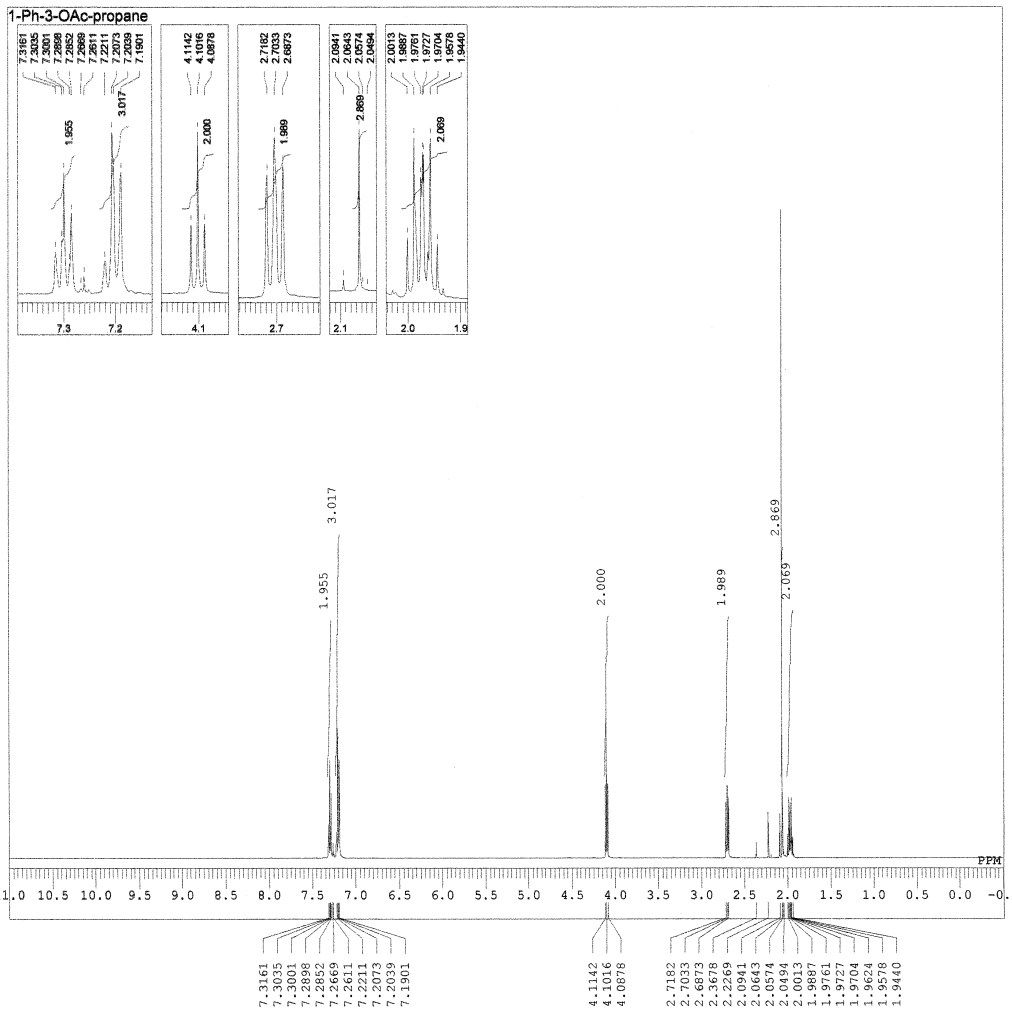

```
DFILE   ozawa08-142b_upper.als
COMNT   BzOAm
DATIM   2016-01-12 09:47:02
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          13107
FREQU        7507.51 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           30.3 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
```



2c'

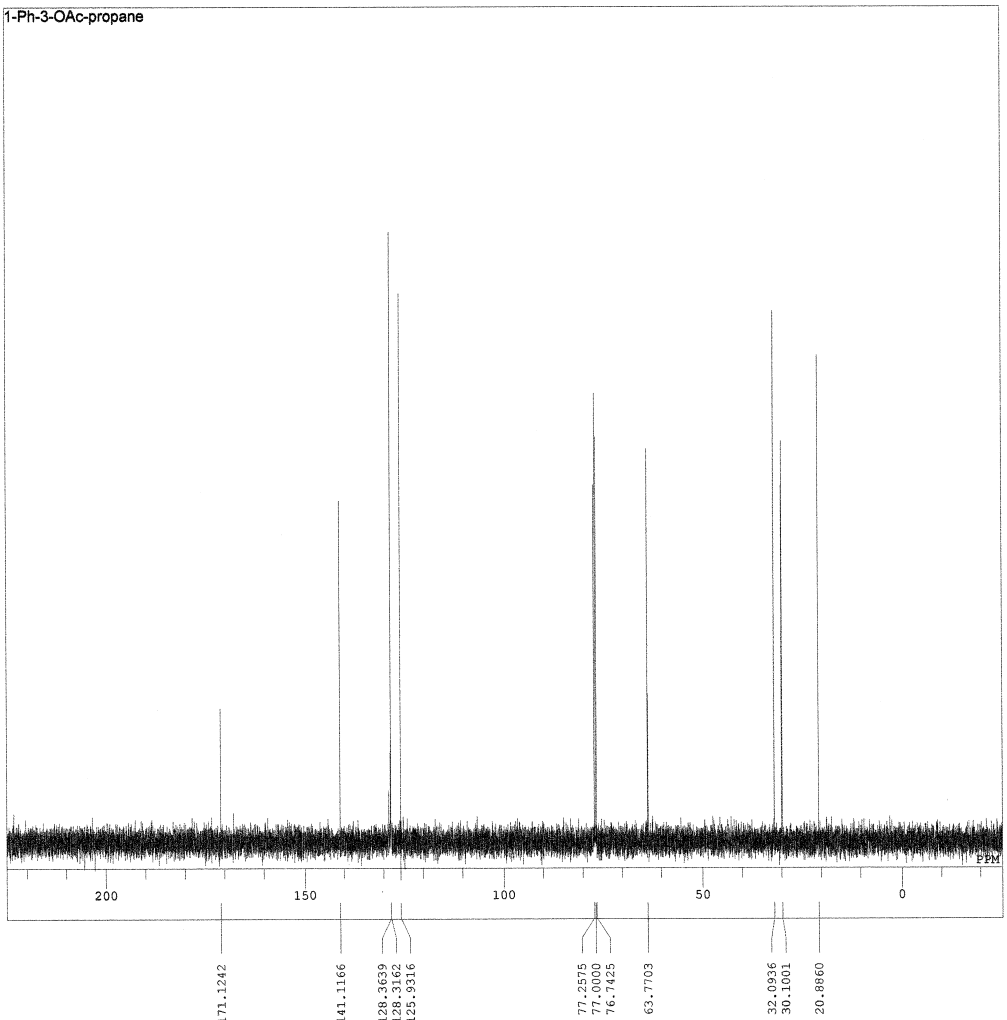



```
DFILE   ozawa08-142b_13C.als
COMNT   PTLC, BzOAm
DATIM   2016-01-07 14:40:55
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          26214
FREQU        31446.54 Hz
SCANS            160
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           27.1 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```

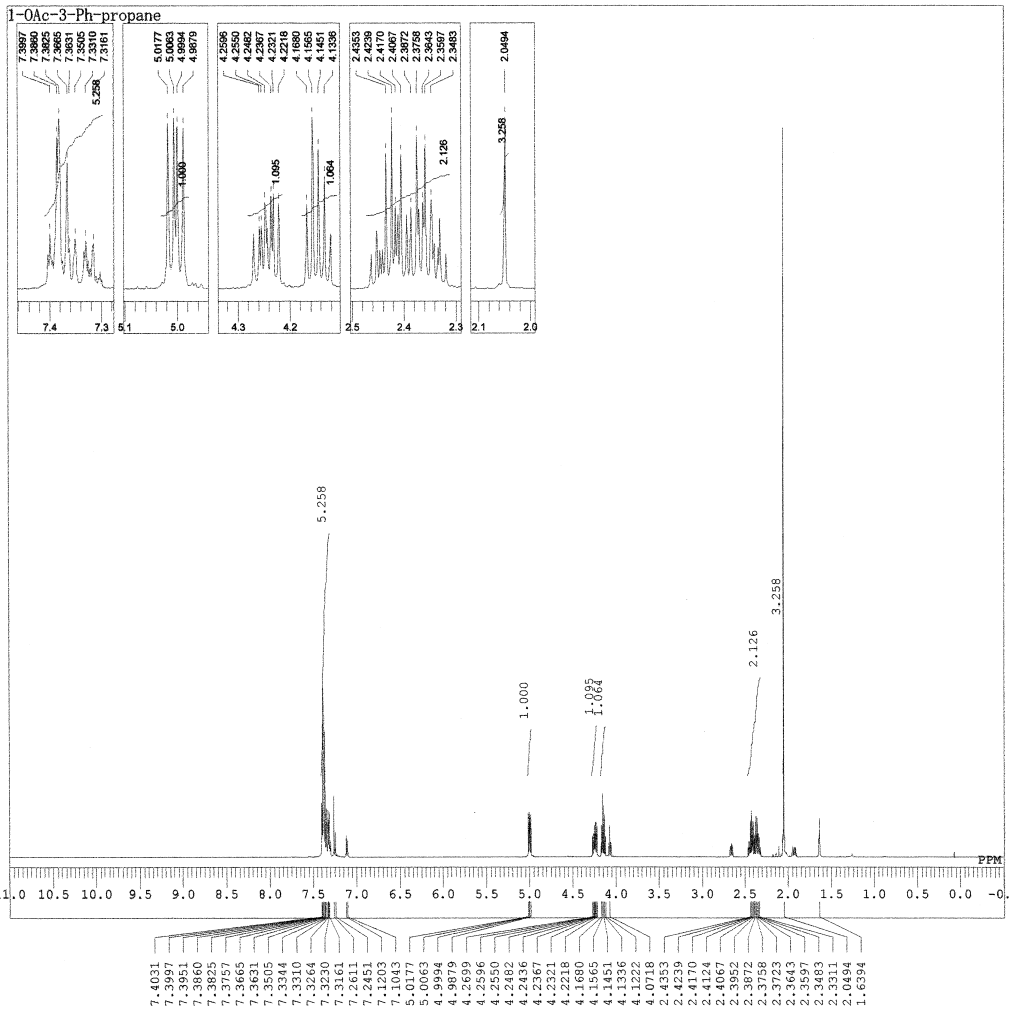


DFILE ozawa07-149_1H.jdf
COMNT 1-Ph-3-OAc-propane
DATIM 2015-08-26 10:11:46
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 30

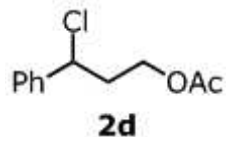
1d



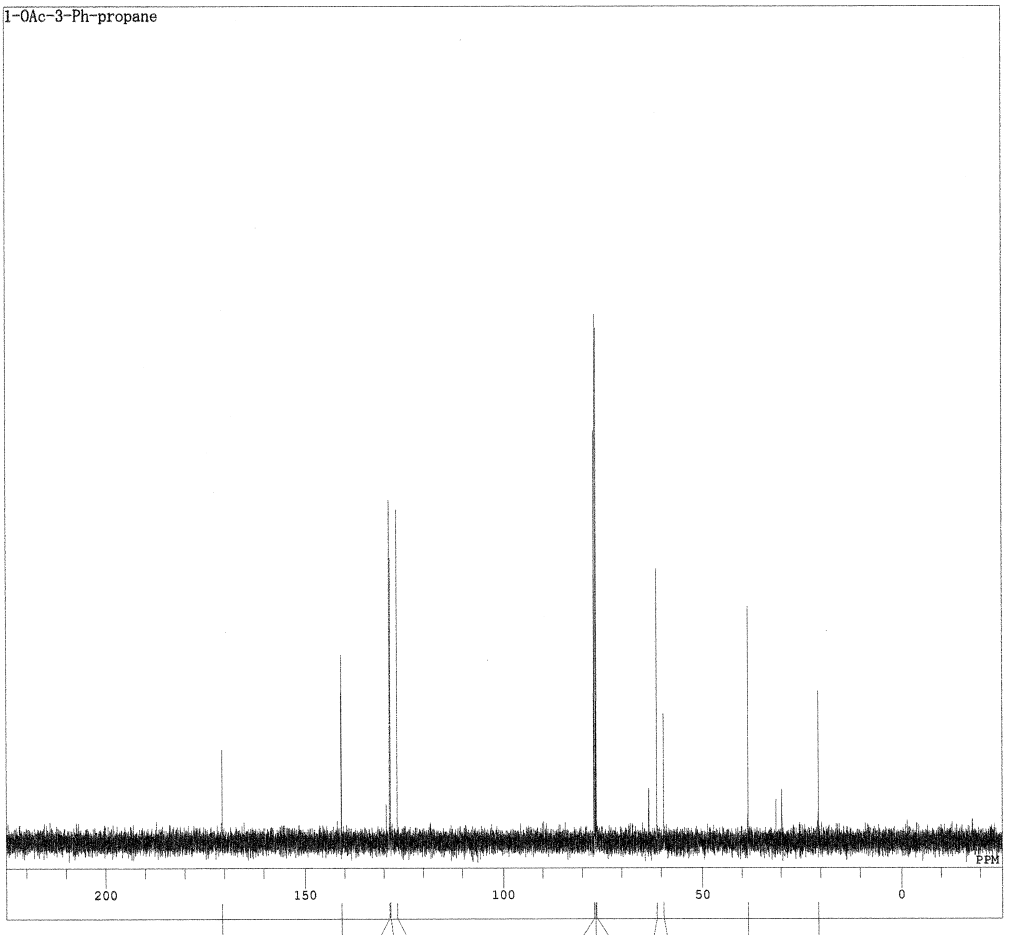
DFILE ozawa07-149_13C.jdf
COMNT 1-Ph-3-OAc-propane
DATIM 2015-08-26 10:12:50
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32767
FREQU 39308.18 Hz
SCANS 16
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



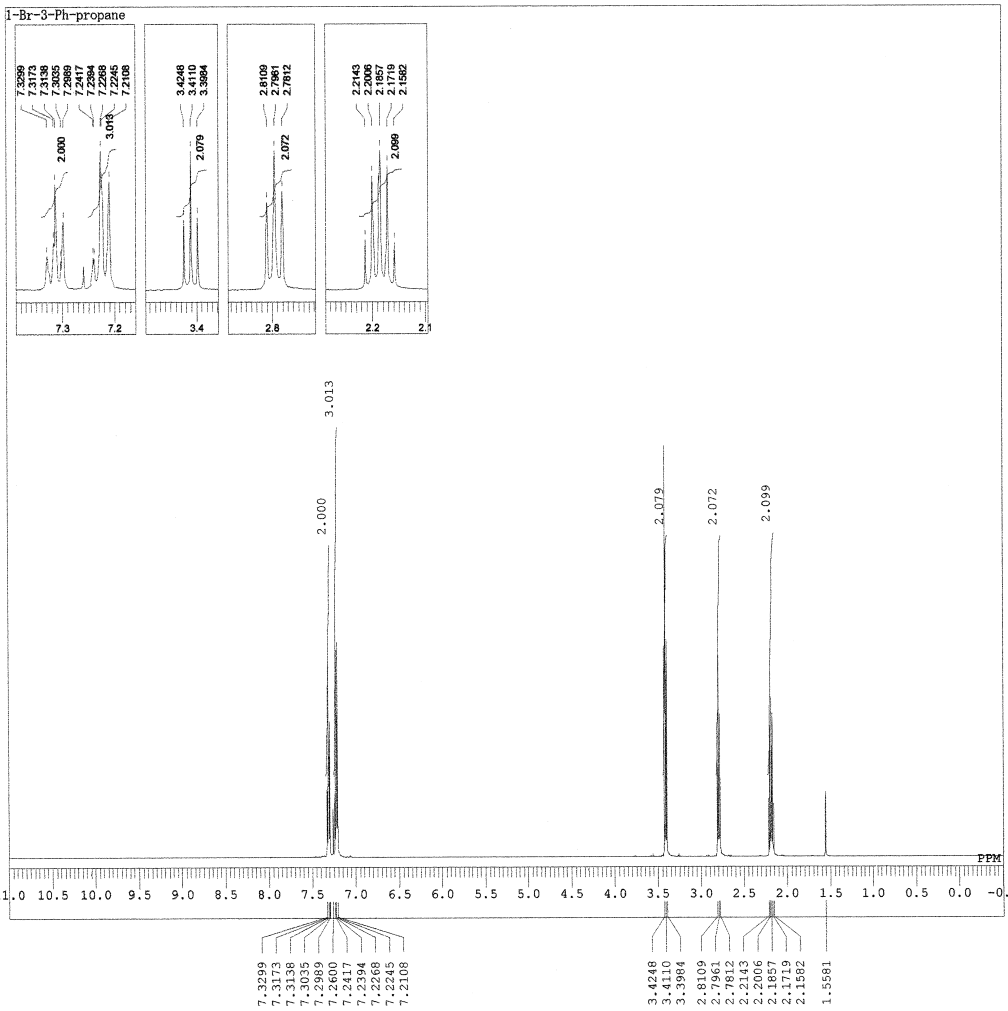

```
DFILE   ozawa08-133_1H.jdf
COMNT   1-OAc-3-Ph-propane
DATIM   2015-09-26 13:00:35
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS              4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           22.9 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
```



2d

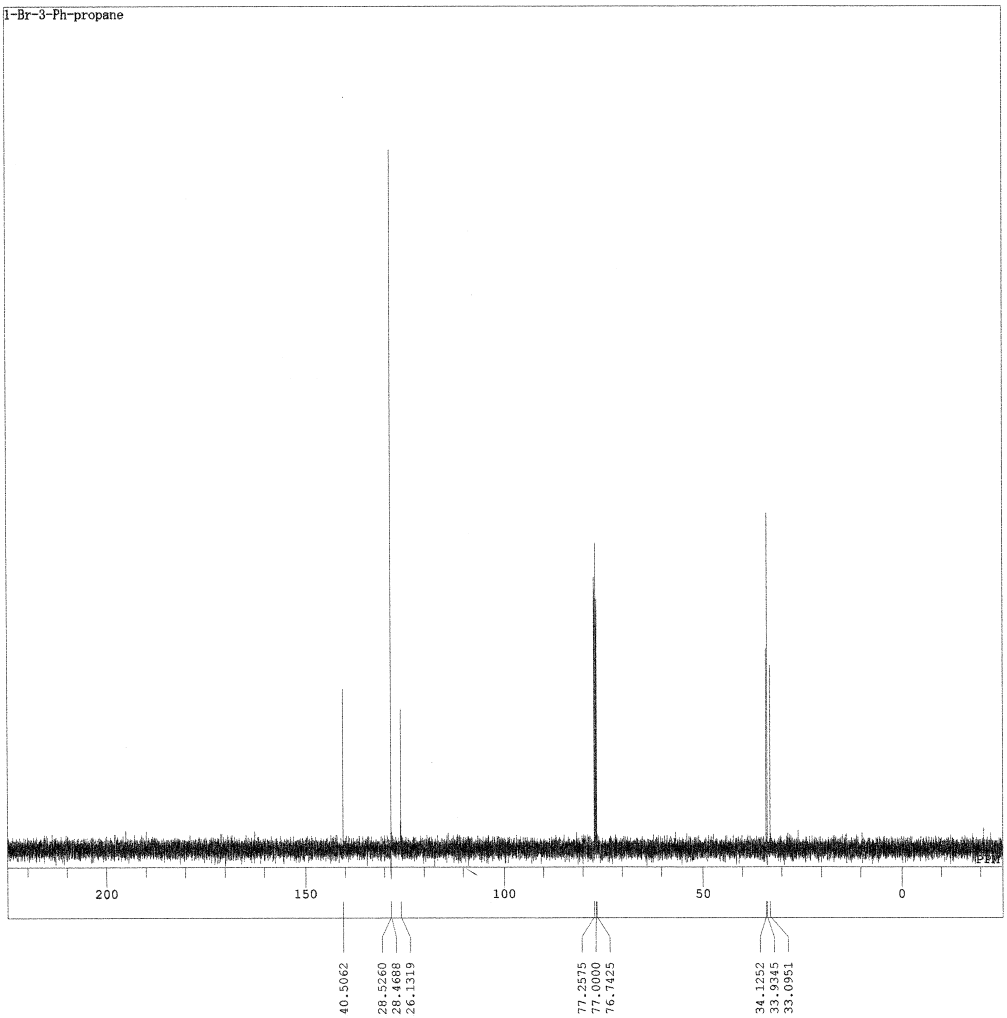



```
DFILE   ozawa06-133_13C.jdf
COMNT   1-OAc-3-Ph-propane
DATIM   2015-09-26 13:01:37
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU       39308.18 Hz
SCANS             48
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           22.7 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```

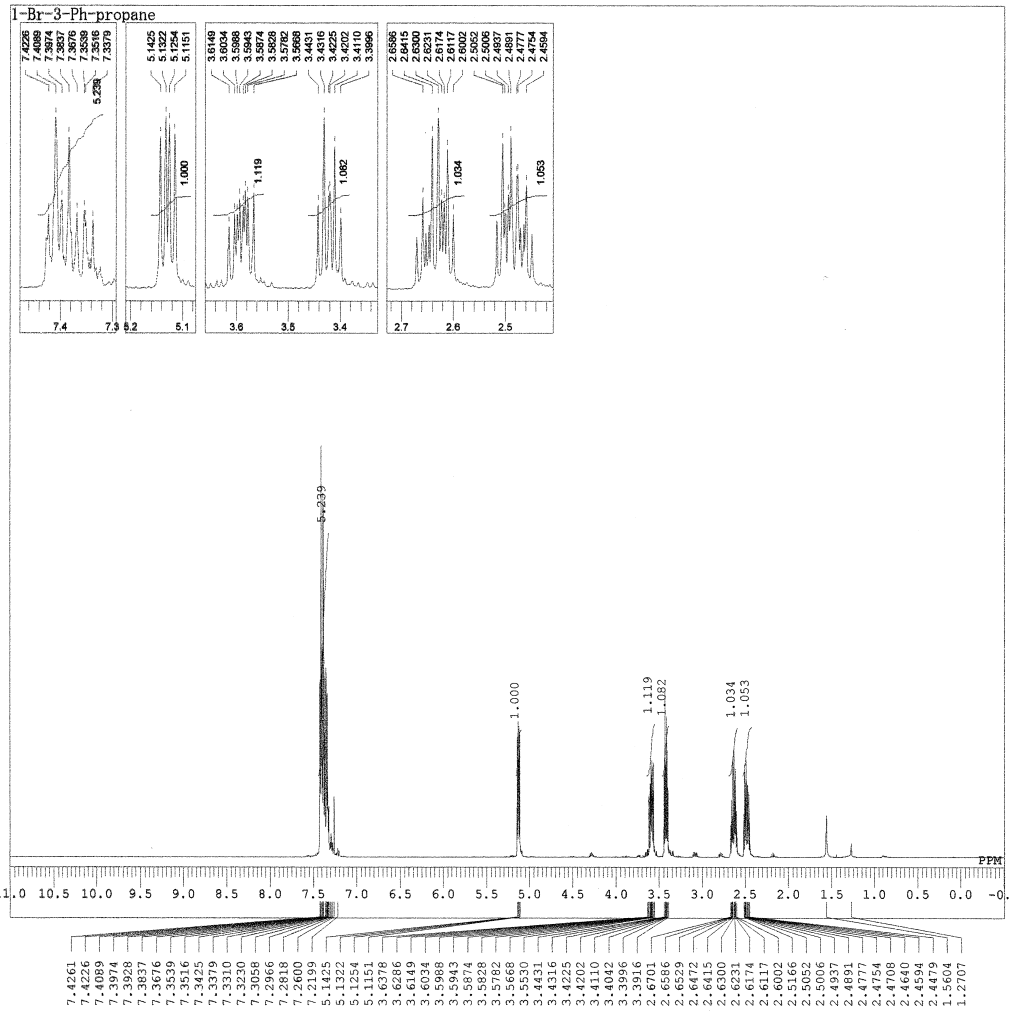


DFILE _1-Br-3-Ph-propane_1H.jdf
COMNT 1-Br-3-Ph-propane
DATIM 2015-08-28 12:41:51
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 30

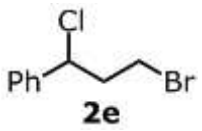
1e



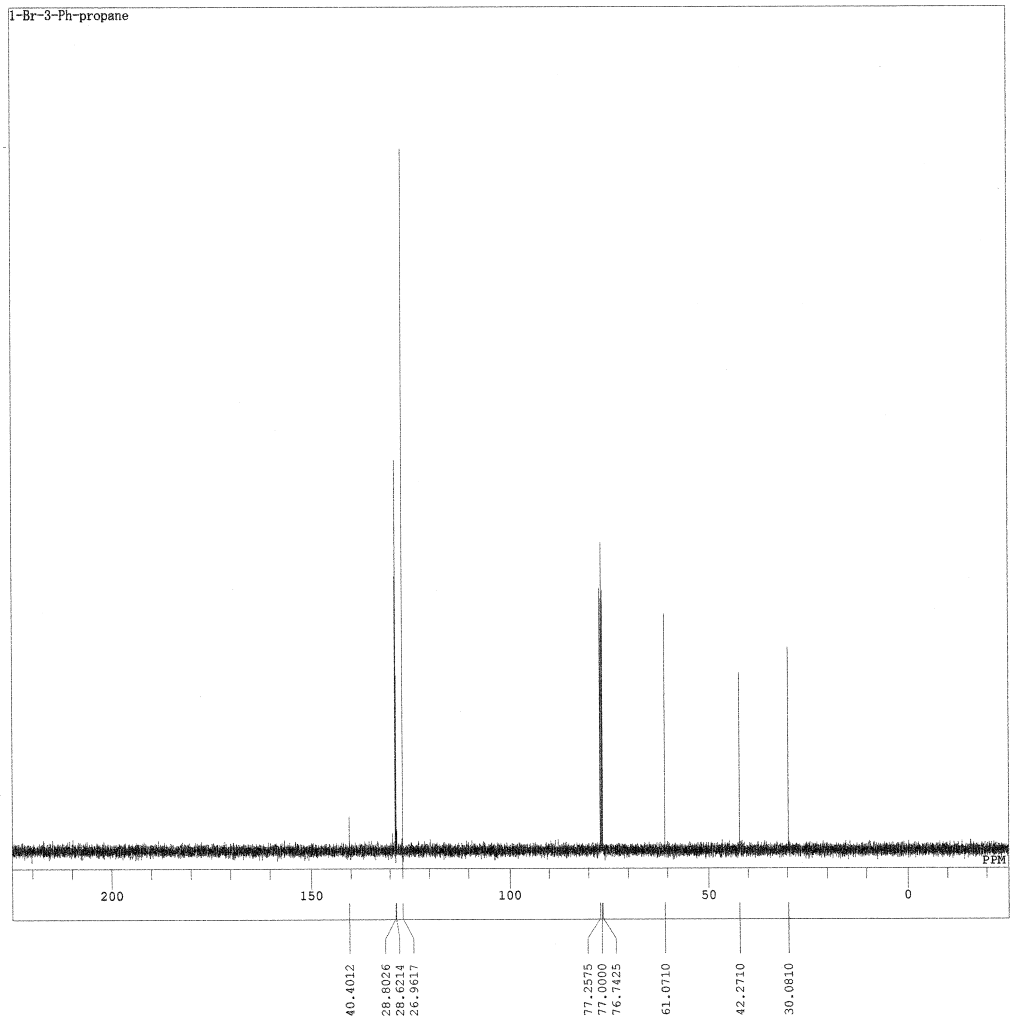
DFILE _1-Br-3-Ph-propane_13C.jdf
COMNT 1-Br-3-Ph-propane
DATIM 2015-08-28 12:42:53
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32767
FREQU 39308.18 Hz
SCANS 20
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



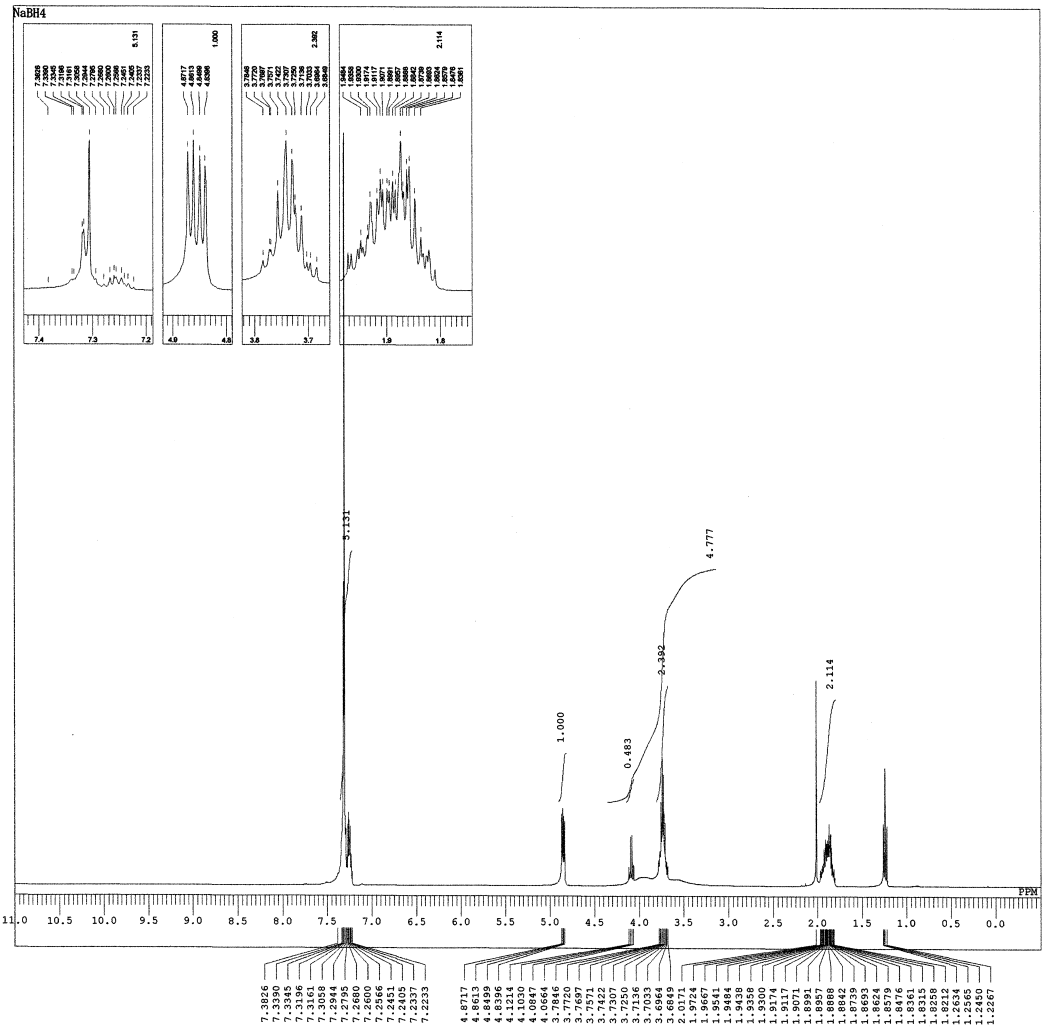

```
DFILE   ozawa08-128_1H.jdf
COMNT   1-Br-3-Ph-propane
DATIM   2015-09-24 10:04:19
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1            5.55 usec
IRNUC   1H
CTEMP          23.2 c
SLVNT   CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            30
```



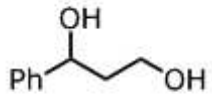
2e



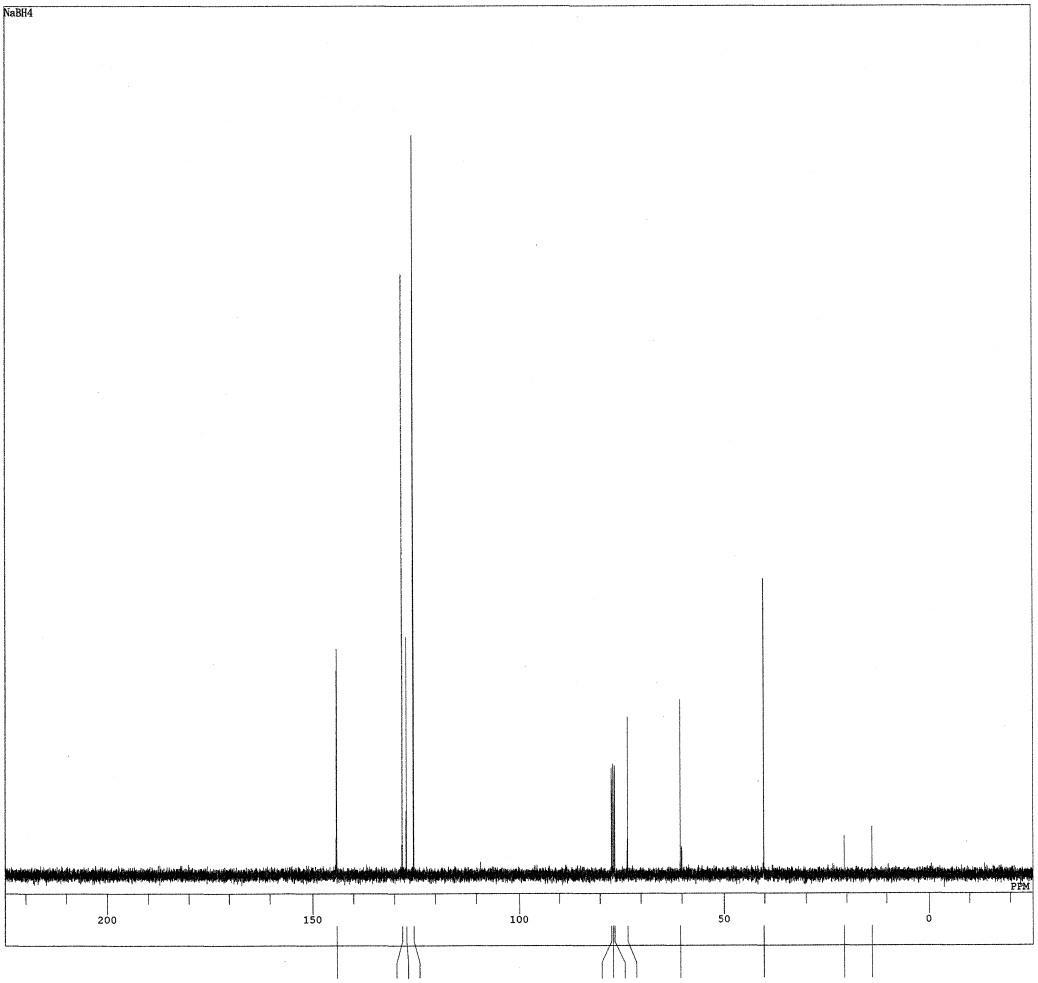

```
DFILE   ozawa08-128_13C.jdf
COMNT   1-Br-3-Ph-propane
DATIM   2015-09-24 10:05:23
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU        39308.18 Hz
SCANS            60
ACQTM          0.8336 sec
PD             2.0000 sec
PW1            3.40 usec
IRNUC   1H
CTEMP          23.3 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF             0.12 Hz
RGAIN            60
```



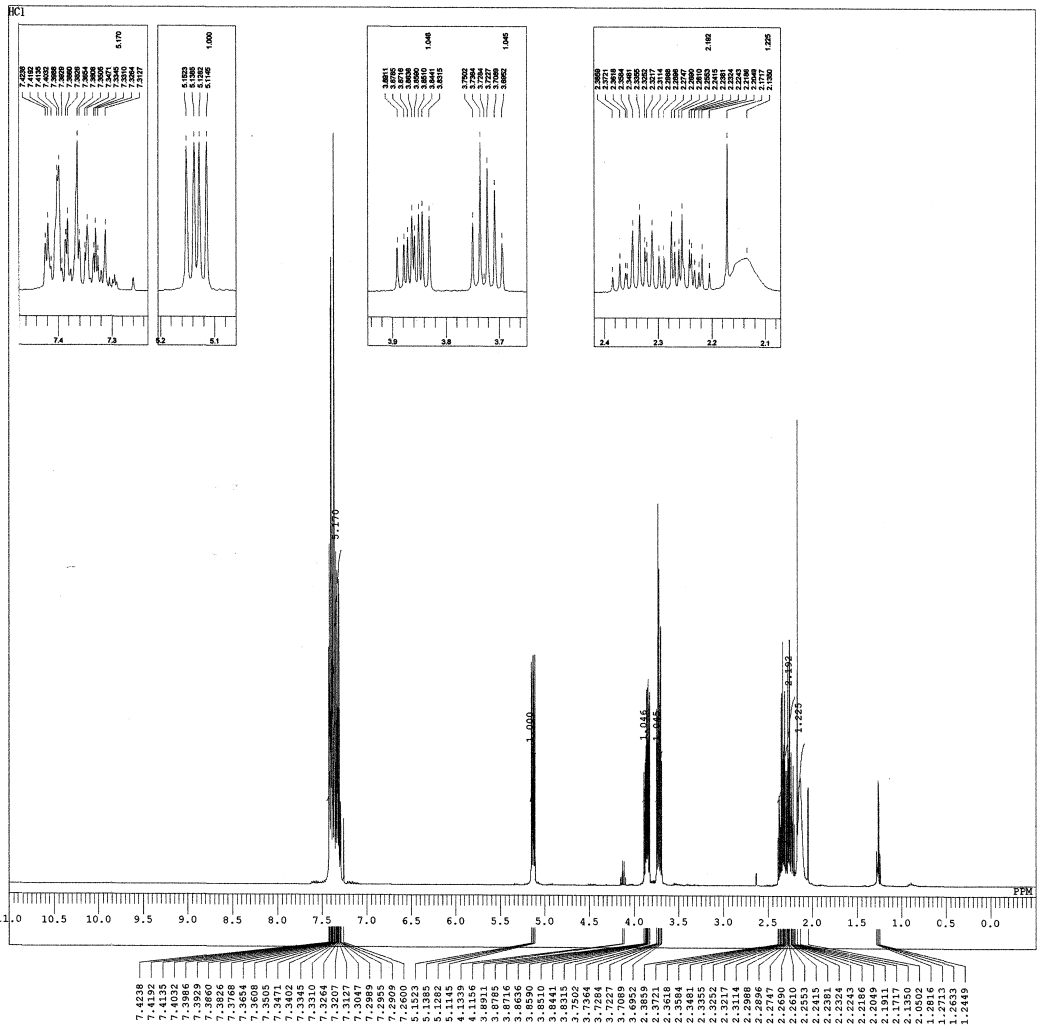

```
DFILE  ozawa09-126_1H.jdf
COMNT  NaBH4
DATIM  15-08-2016 14:48:01
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         16384
FREQU       7352.94 Hz
SCANS             4
ACQTM          2.2282 sec
PD             5.0000 sec
PW1            6.45 usec
IRNUC  1H
CTEMP        460.0 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            22
```



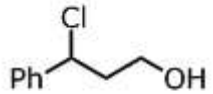
S1e



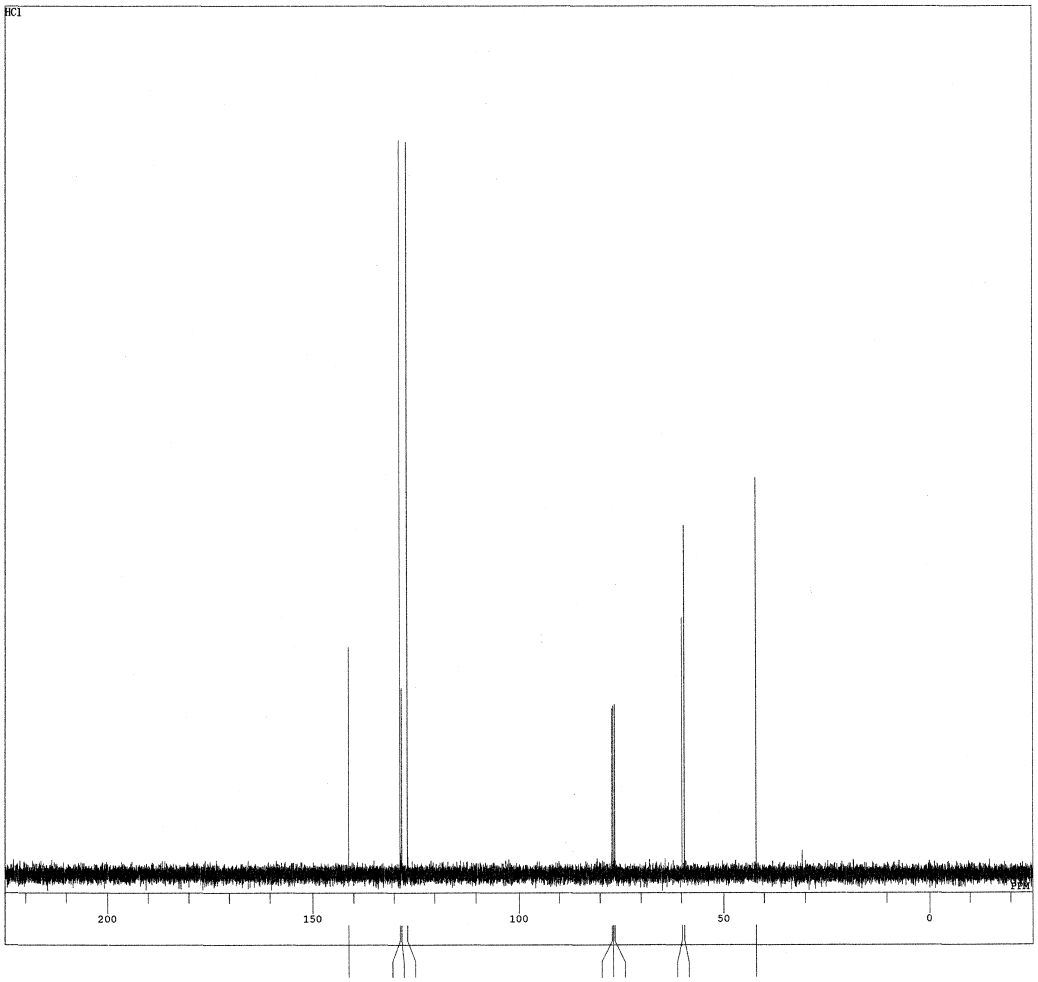

```
DFILE  ozawa09-126_13C.jdf
COMNT  NaBH4
DATIM  15-08-2016 14:49:27
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         32767
FREQU      30788.18 Hz
SCANS            32
ACQTM          1.0643 sec
PD             2.0000 sec
PW1            3.73 usec
IRNUC  1H
CTEMP        460.0 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.12 Hz
RGAIN            60
```



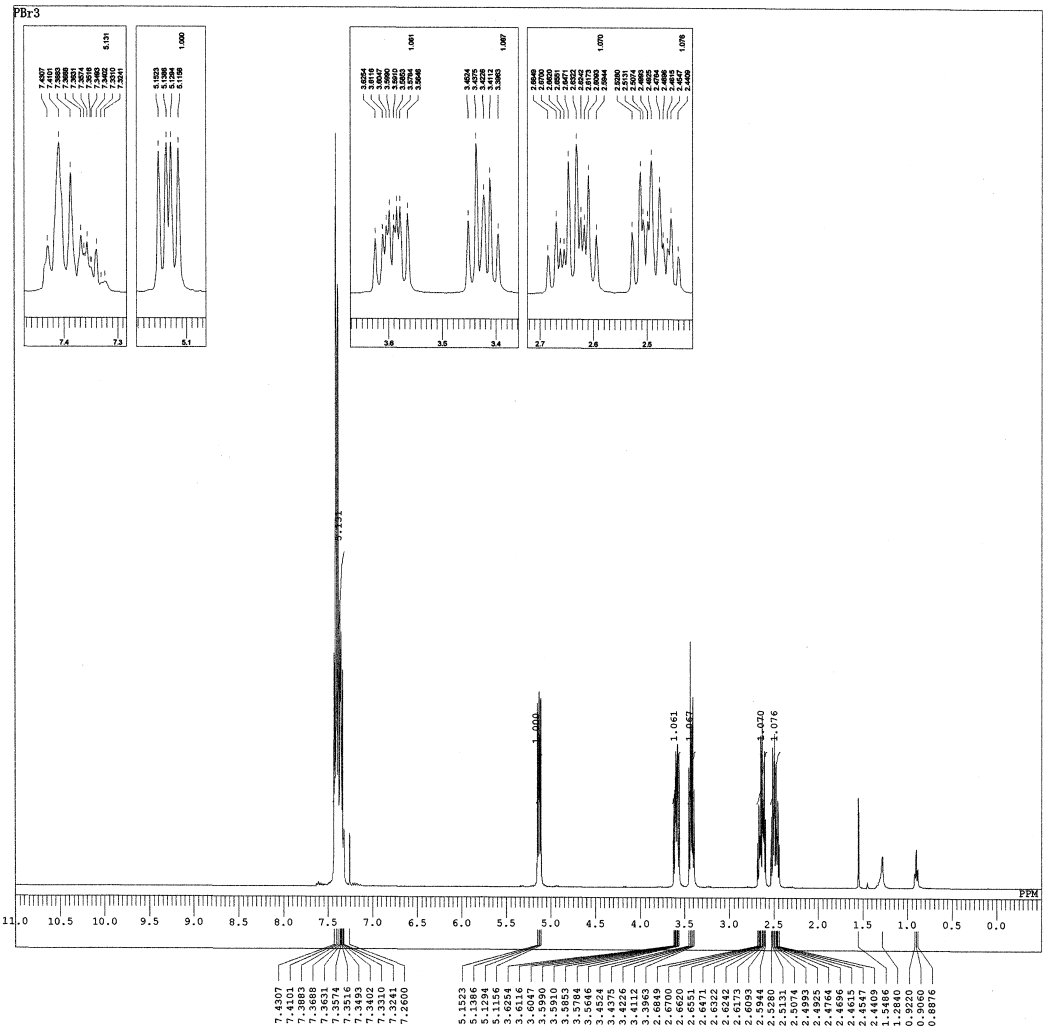

```
DFILE  ozawa09-130_1H.als
COMNT  HCl
DATIM  18-08-2016 15:28:00
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         13107
FREQU       5882.35 Hz
SCANS             4
ACQTM         2.2282 sec
PD            5.0000 sec
PW1             6.45 usec
IRNUC  1H
CTEMP         460.0 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            24
```



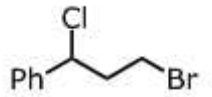
S2e



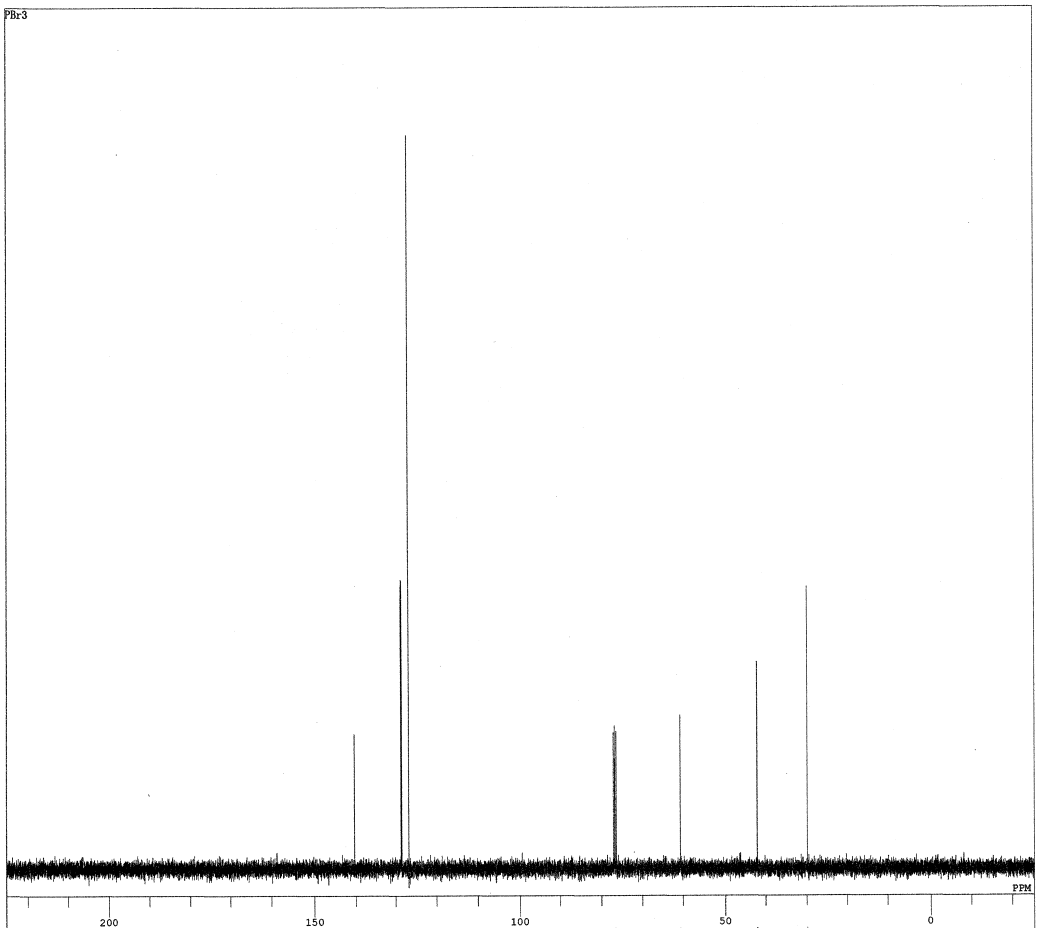

```
DFILE  ozawa09-130_13C.jdf
COMNT  HCl
DATIM  18-08-2016 15:29:20
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         32767
FREQU      30788.18 Hz
SCANS            20
ACQTM         1.0643 sec
PD            2.0000 sec
PW1             3.73 usec
IRNUC  1H
CTEMP         460.0 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.12 Hz
RGAIN            60
```



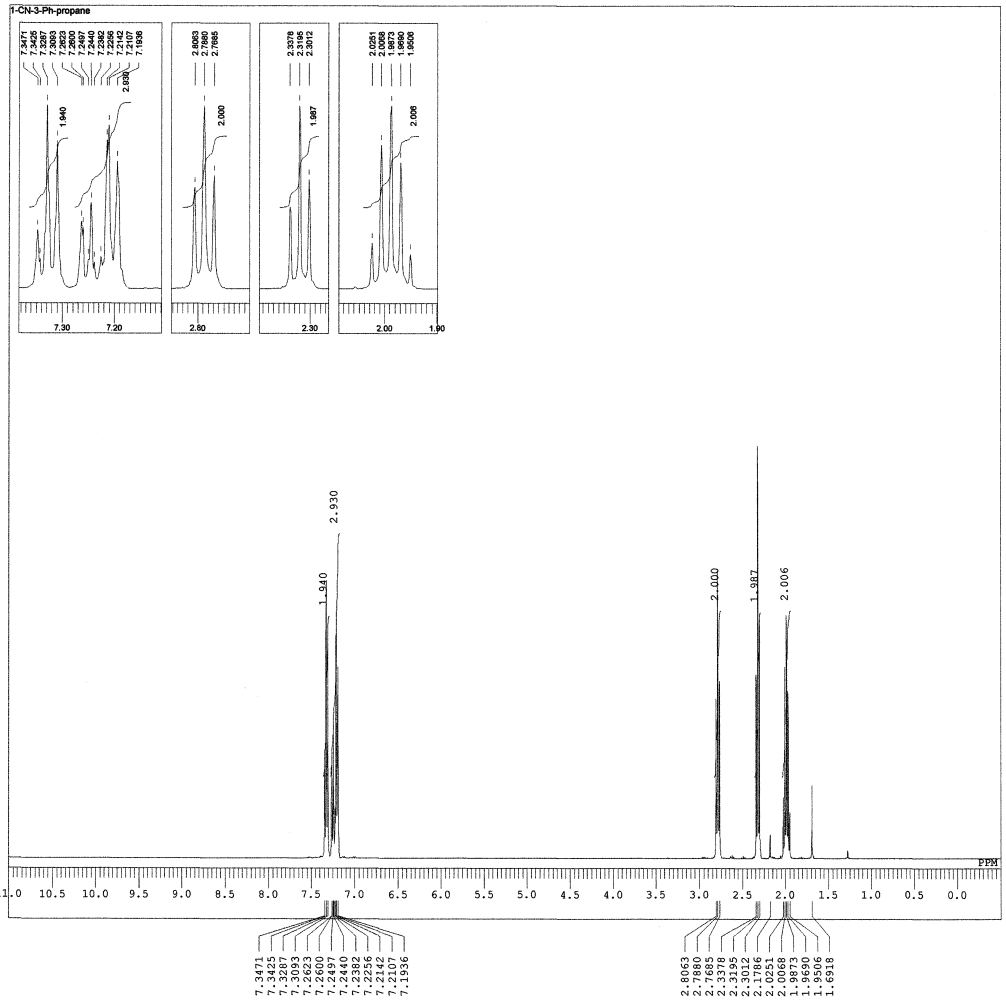

```
DFILE  ozawa09-128_1H.jdf
COMNT  PBr3
DATIM  25-08-2016 16:05:32
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         16384
FREQU       7352.94 Hz
SCANS             4
ACQTM          2.2282 sec
PD             5.0000 sec
PW1            6.45 usec
IRNUC  1H
CTEMP        460.0 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.20 Hz
RGAIN            26
```



2e (authentic)

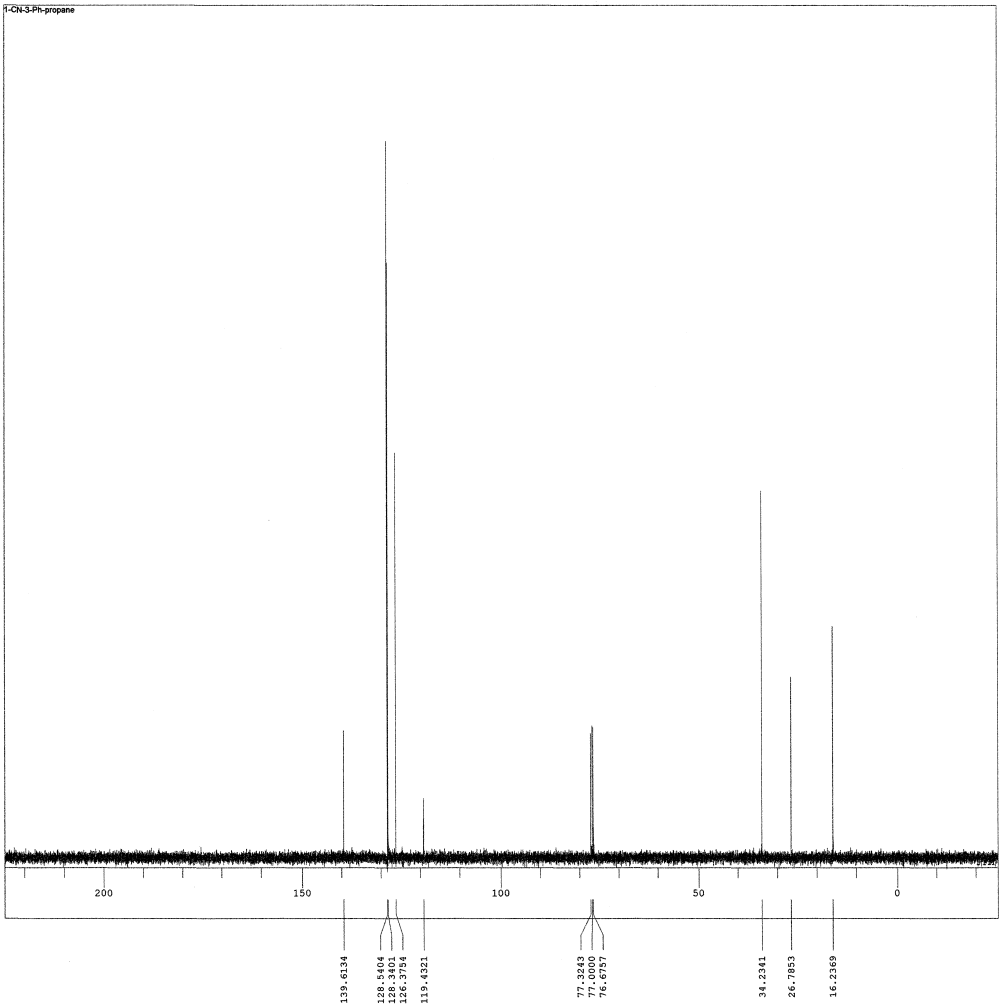



```
DFILE  ozawa09-128_13C.jdf
COMNT  PBr3
DATIM  25-08-2016 16:07:52
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         32767
FREQU      30788.18 Hz
SCANS            20
ACQTM          1.0643 sec
PD             2.0000 sec
PW1            3.73 usec
IRNUC  1H
CTEMP        460.0 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.20 Hz
RGAIN            60
```

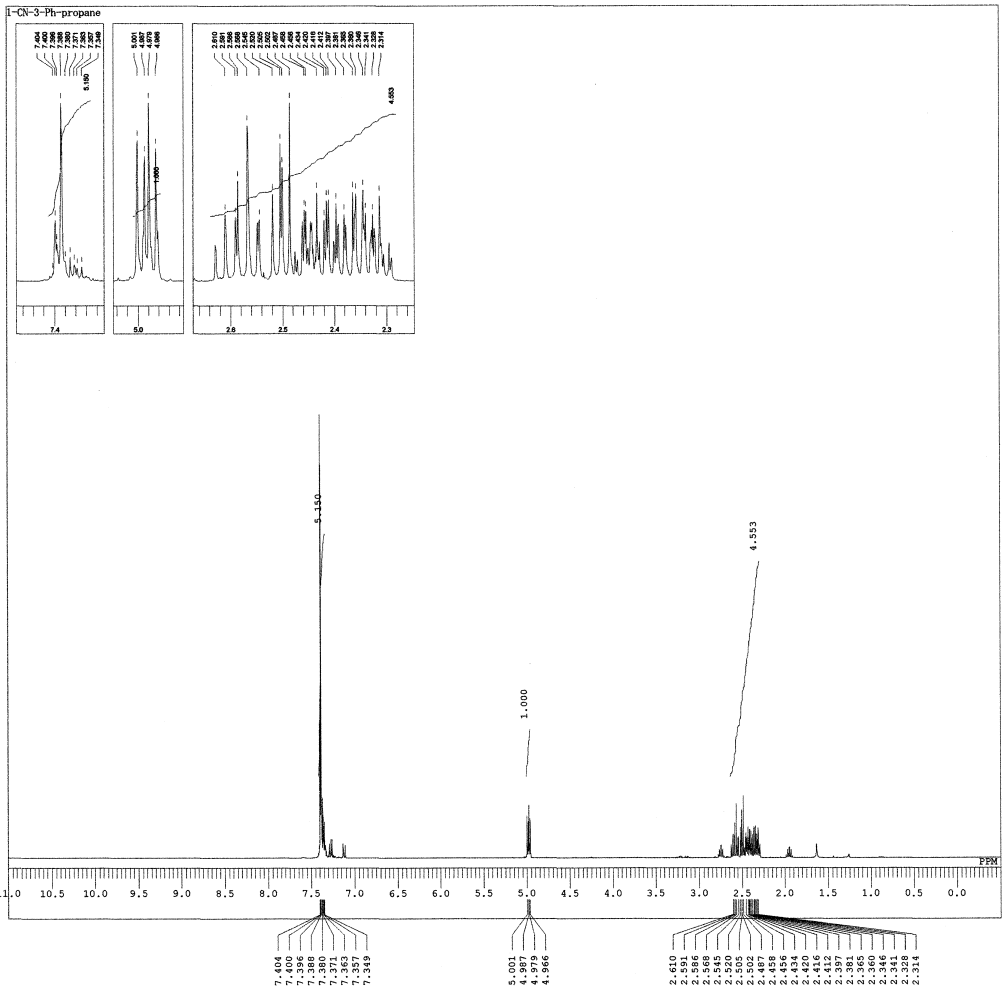


DFILE ozawa08-082_1H.jdf
COMNT 1-CN-3-Ph-propane
DATIM 26-08-2015 15:08:17
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 16384
FREQU 7352.94 Hz
SCANS 4
ACQTM 2.2282 sec
PD 5.0000 sec
PW1 4.99 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 24

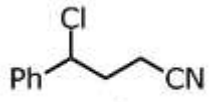
1f



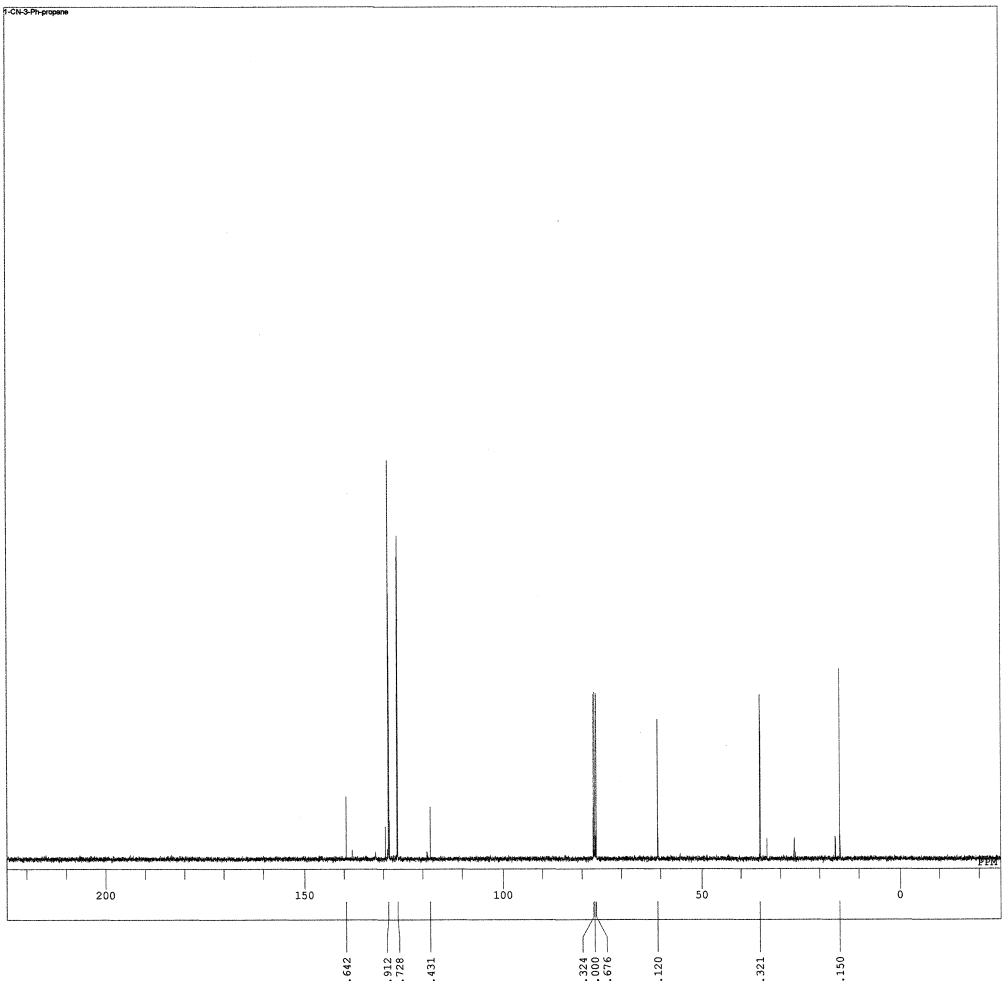
DFILE ozawa08-082_13C.jdf
COMNT 1-CN-3-Ph-propane
DATIM 26-08-2015 15:09:20
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 32767
FREQU 30788.18 Hz
SCANS 16
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.16 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



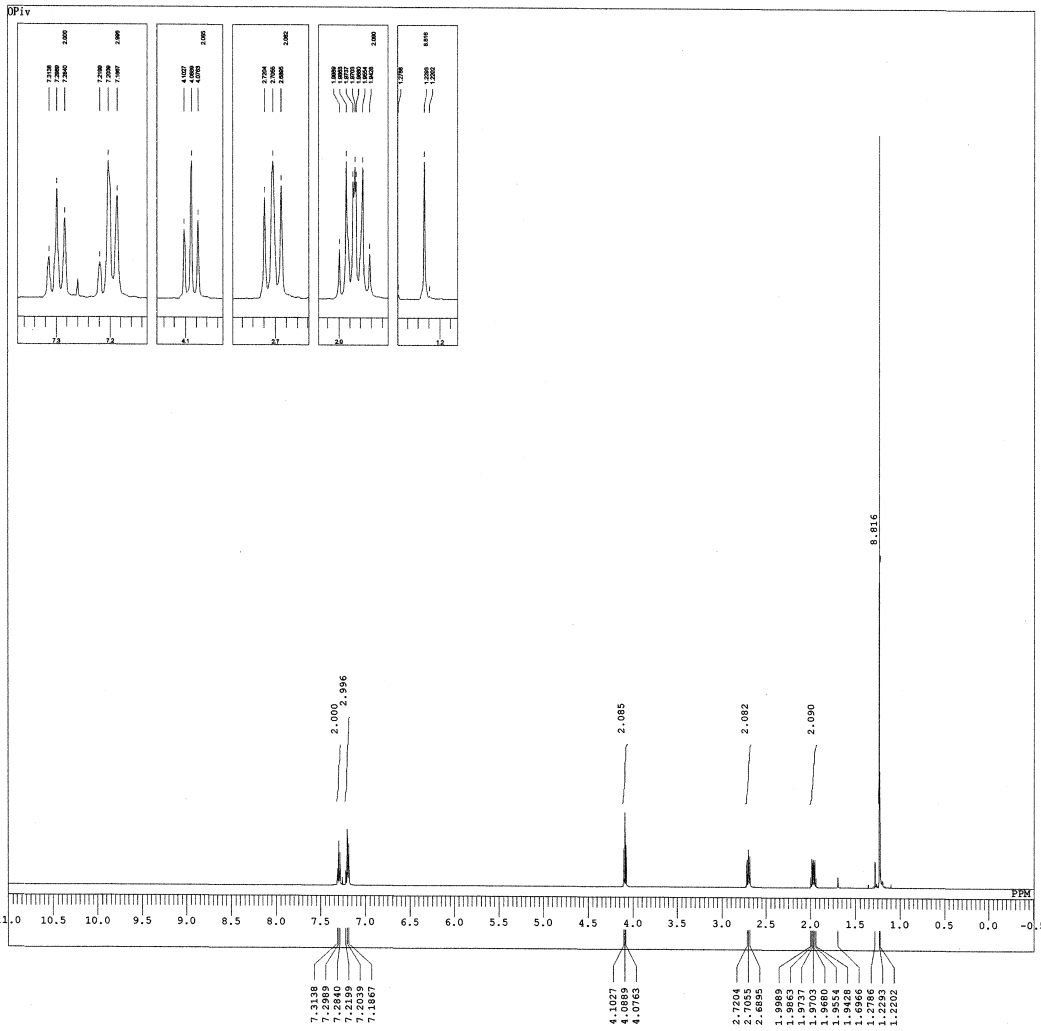
DFILE ozawa08-135_1H.jdf
COMNT 1-CN-3-Ph-propane
DATIM 26-09-2015 14:59:48
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 16384
FREQU 7352.94 Hz
SCANS 4
ACQTM 2.2282 sec
PD 5.0000 sec
PW1 4.99 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 28



2f

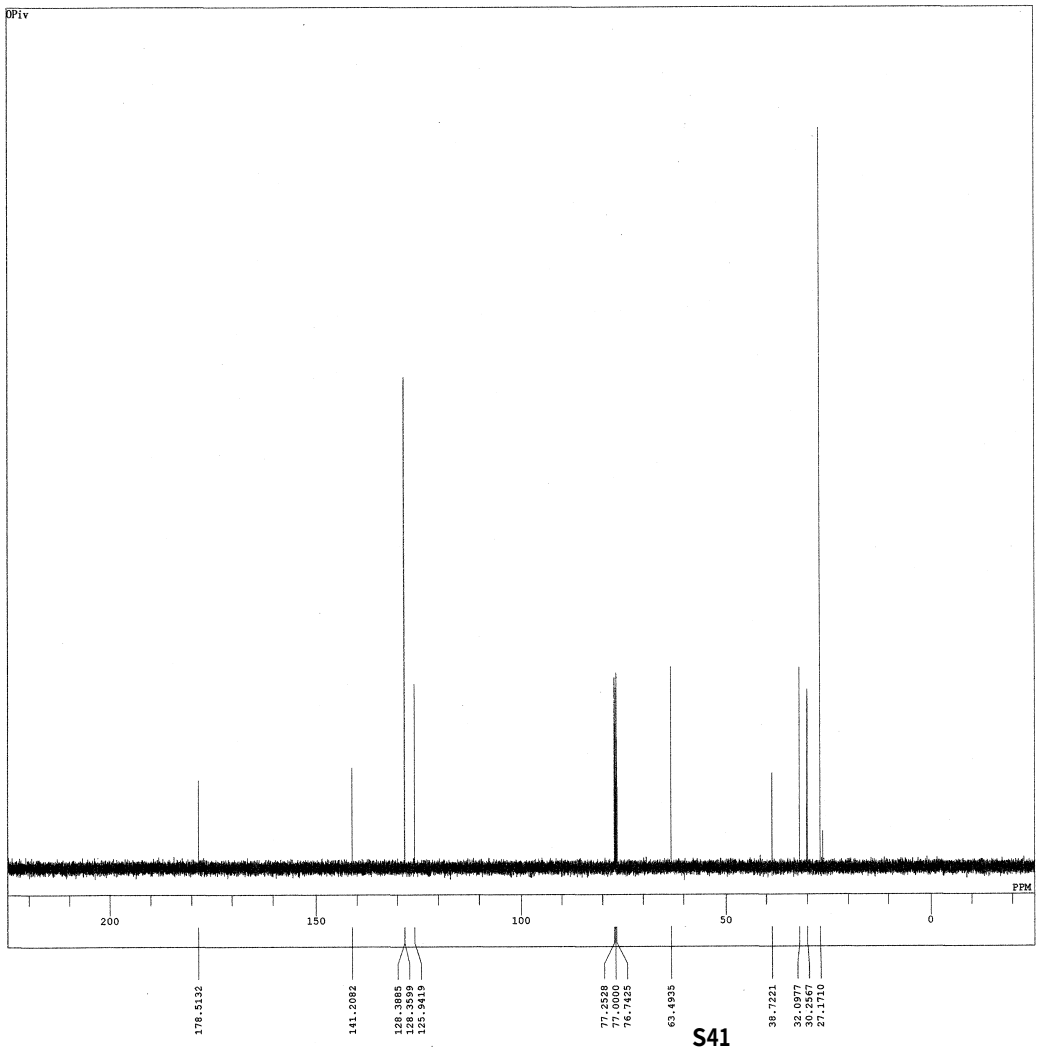


DFILE ozawa08-135_13C.jdf
COMNT 1-CN-3-Ph-propane
DATIM 26-09-2015 15:00:50
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 32767
FREQU 30788.18 Hz
SCANS 100
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.16 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

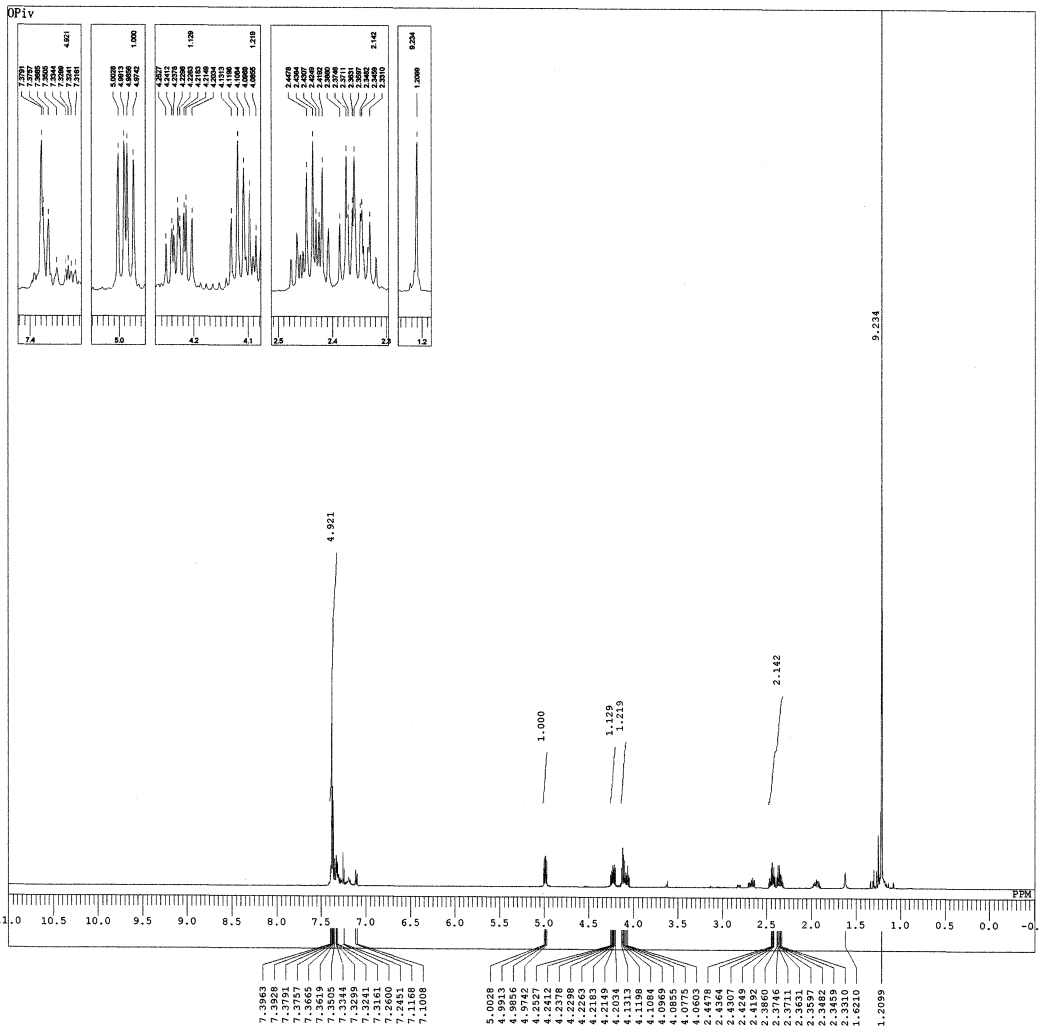


DFILE ozawa09-197_1H.als
COMNT OPiv
DATIM 2016-09-27 10:16:50
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 24

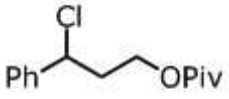
1g



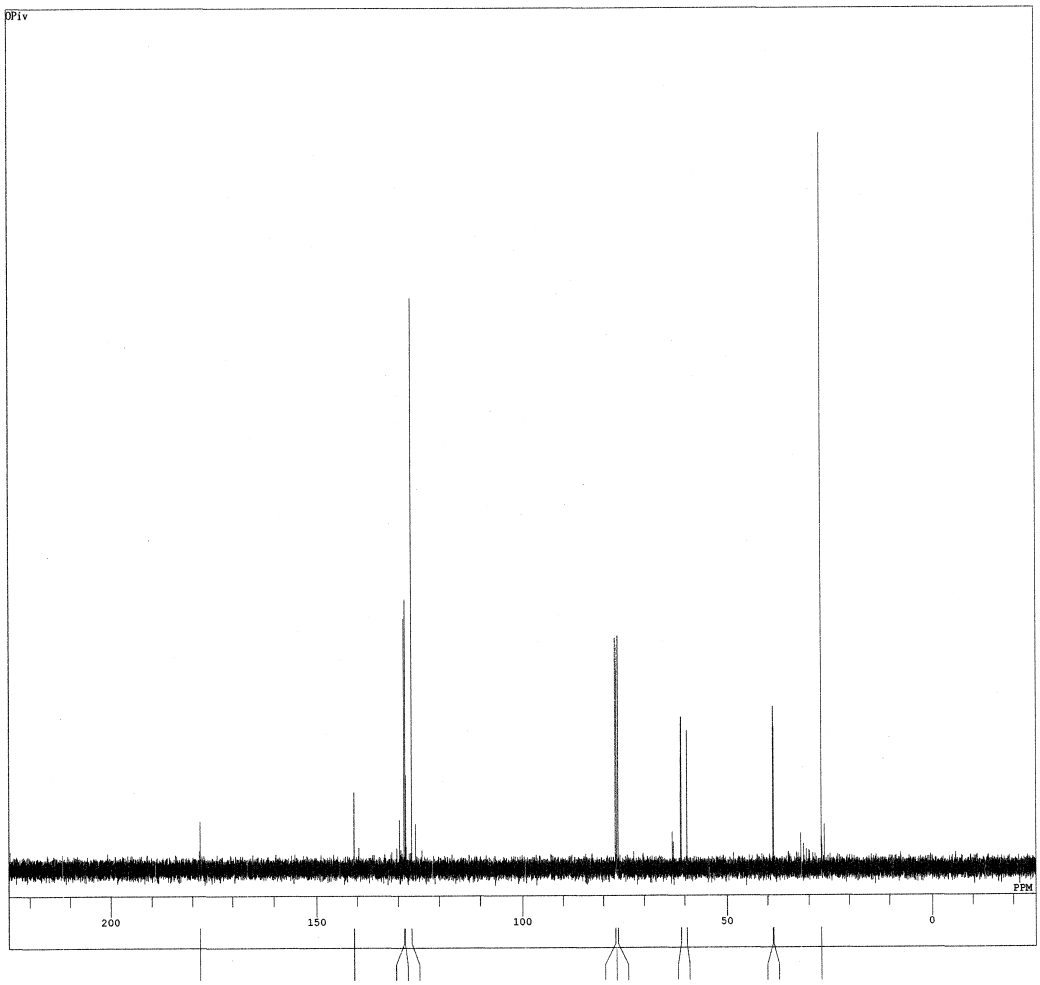
DFILE ozawa09-197_13C.als
COMNT OPiv
DATIM 2016-09-27 10:17:50
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.54 Hz
SCANS 16
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



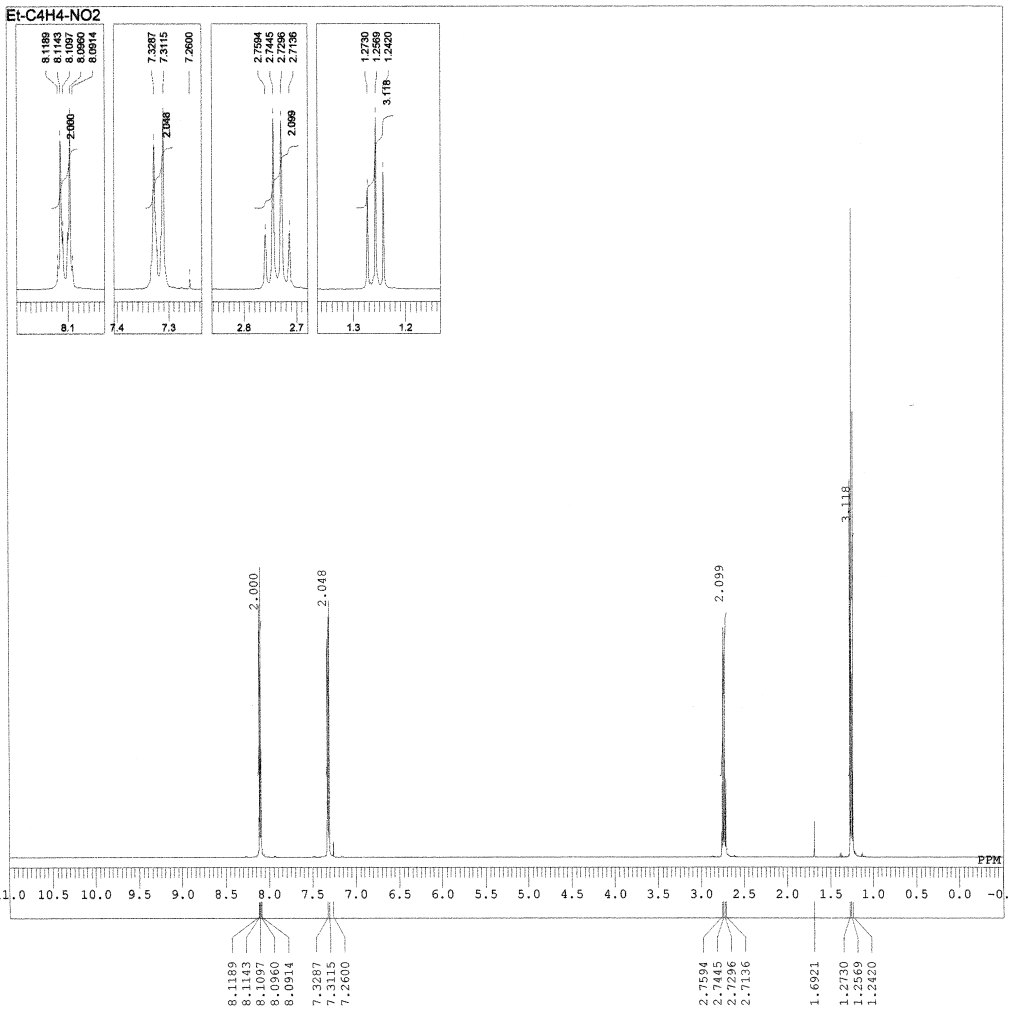

```
DFILE  ozawa09-200_1H.als
COMNT  OPiv
DATIM  2016-09-30 17:21:19
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.51 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1            5.55 usec
IRNUC  1H
CTEMP         21.2 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            28
```



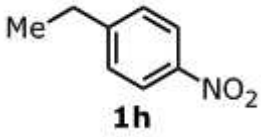
2g



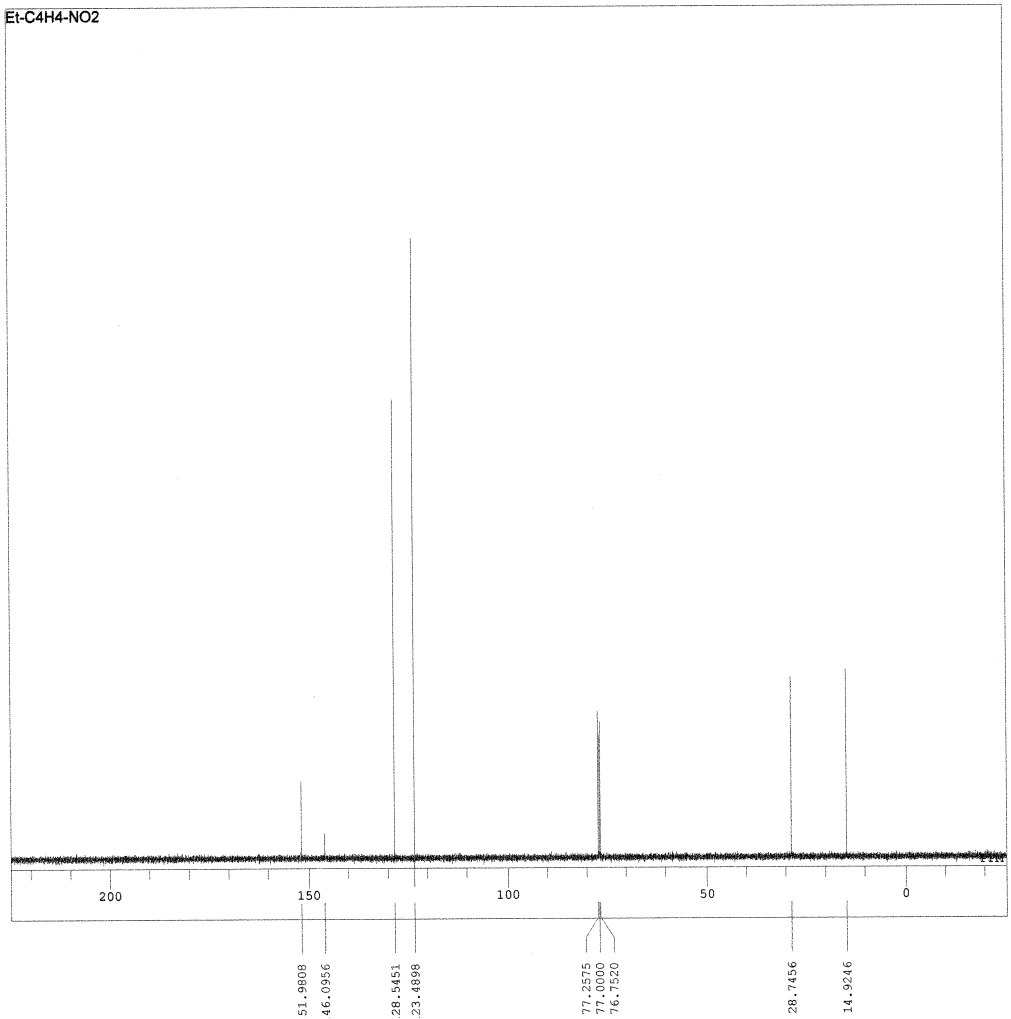

```
DFILE  ozawa09-200_13C.als
COMNT  OPiv
DATIM  30-09-2016 09:35:38
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         52428
FREQU      24630.54 Hz
SCANS            32
ACQTM          2.1286 sec
PD             2.0000 sec
PW1            3.73 usec
IRNUC  1H
CTEMP        460.0 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.12 Hz
RGAIN            60
```



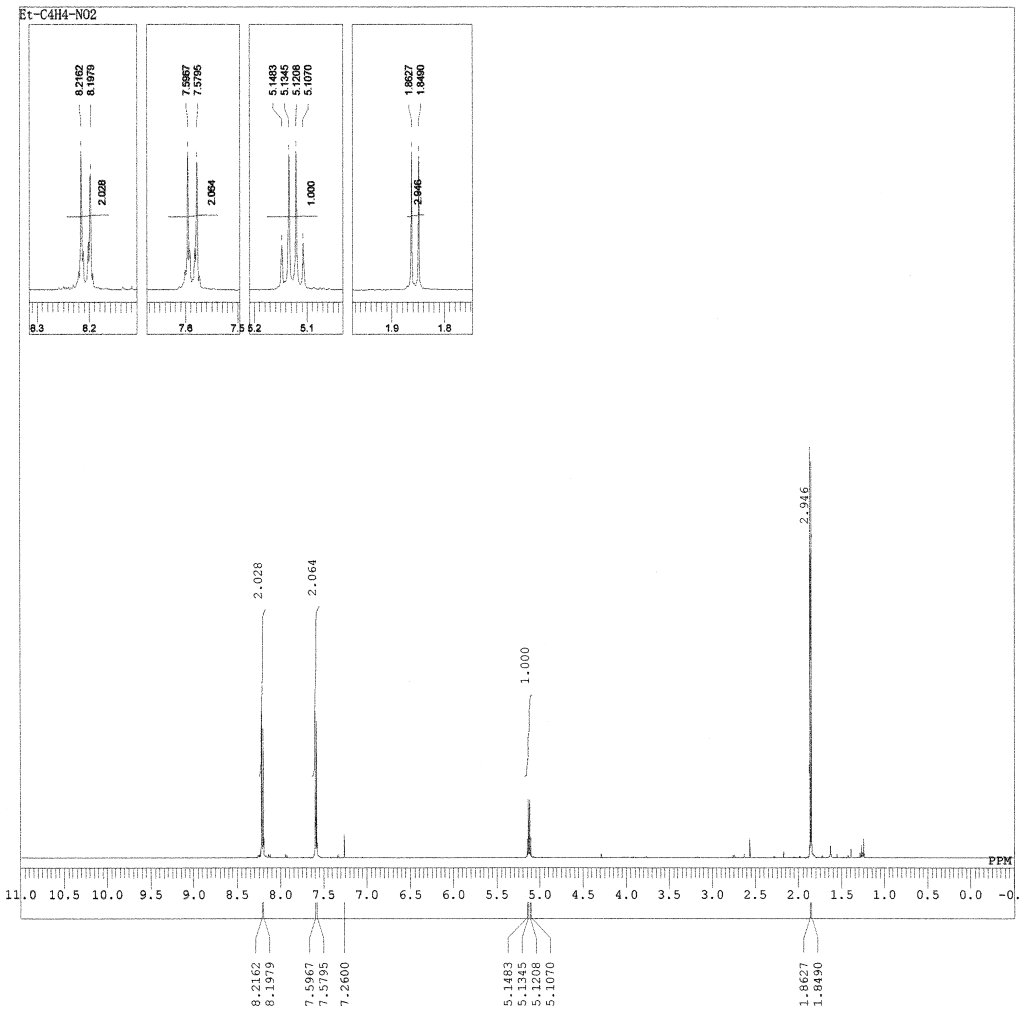

```
DFILE   _Et-C4H4-NO2_1H.jdf
COMNT   Et-C4H4-NO2
DATIM   2015-08-26 10:20:26
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1               5.55 usec
IRNUC   1H
CTEMP            21.5 c
SLVNT   CDCL3
EXREF            7.26 ppm
BF               0.12 Hz
RGAIN              28
```



1h

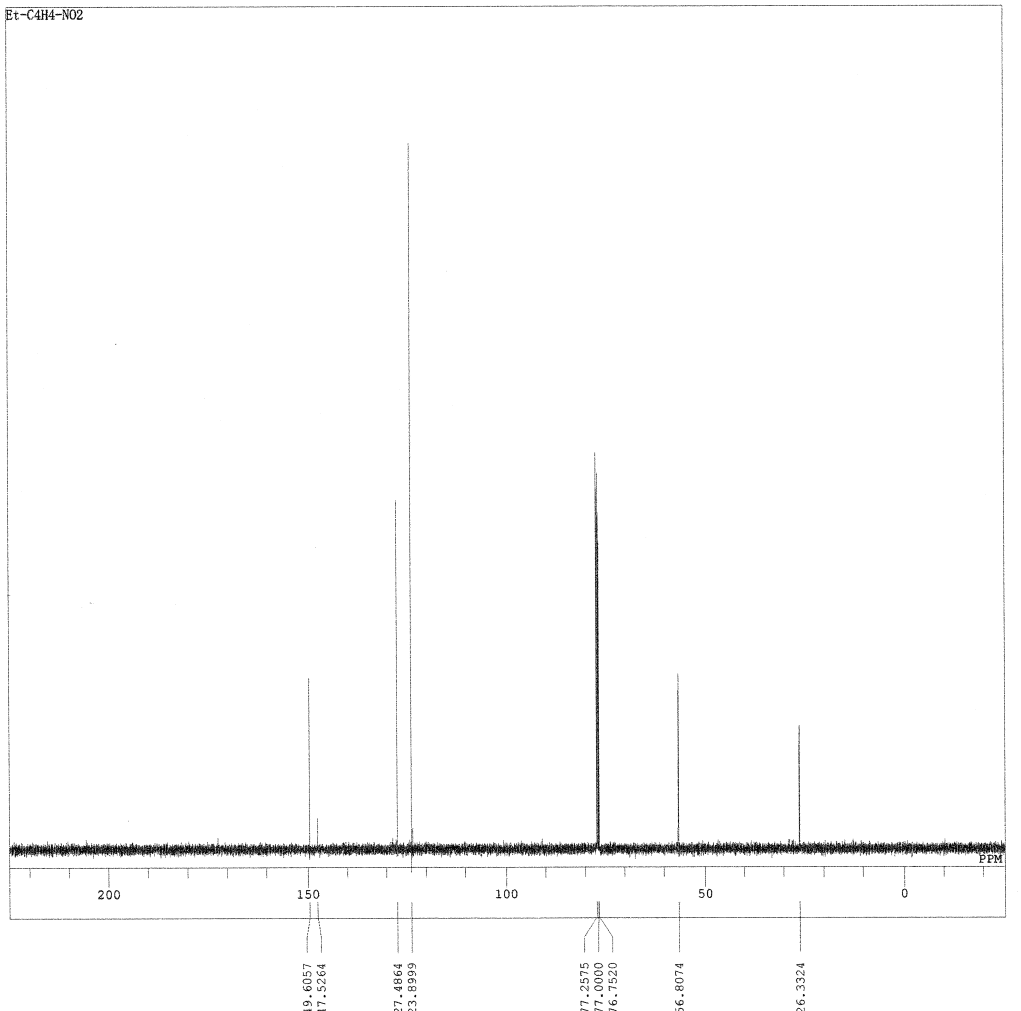



```
DFILE   _Et-C4H4-NO2_13C.jdf
COMNT   Et-C4H4-NO2
DATIM   2015-08-26 10:22:07
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU       39308.18 Hz
SCANS             12
ACQTM          0.8336 sec
PD            10.0000 sec
PW1               3.40 usec
IRNUC   1H
CTEMP            21.7 c
SLVNT   CDCL3
EXREF            77.00 ppm
BF               0.12 Hz
RGAIN              60
```

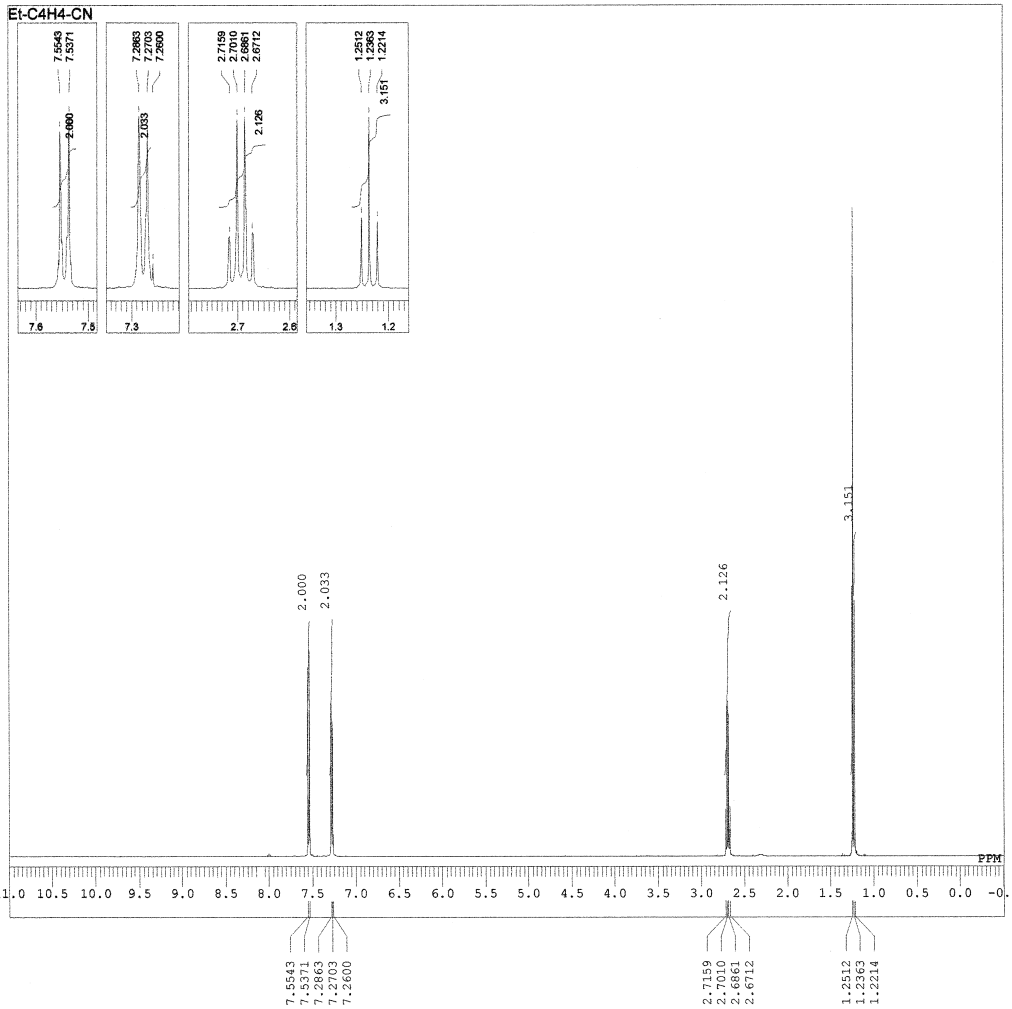



```
DFILE   ozawa08-086_1H.jdf
COMNT   Et-C4H4-NO2
DATIM   2015-08-28 12:26:07
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           21.5 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
```

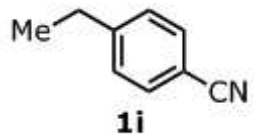
2h



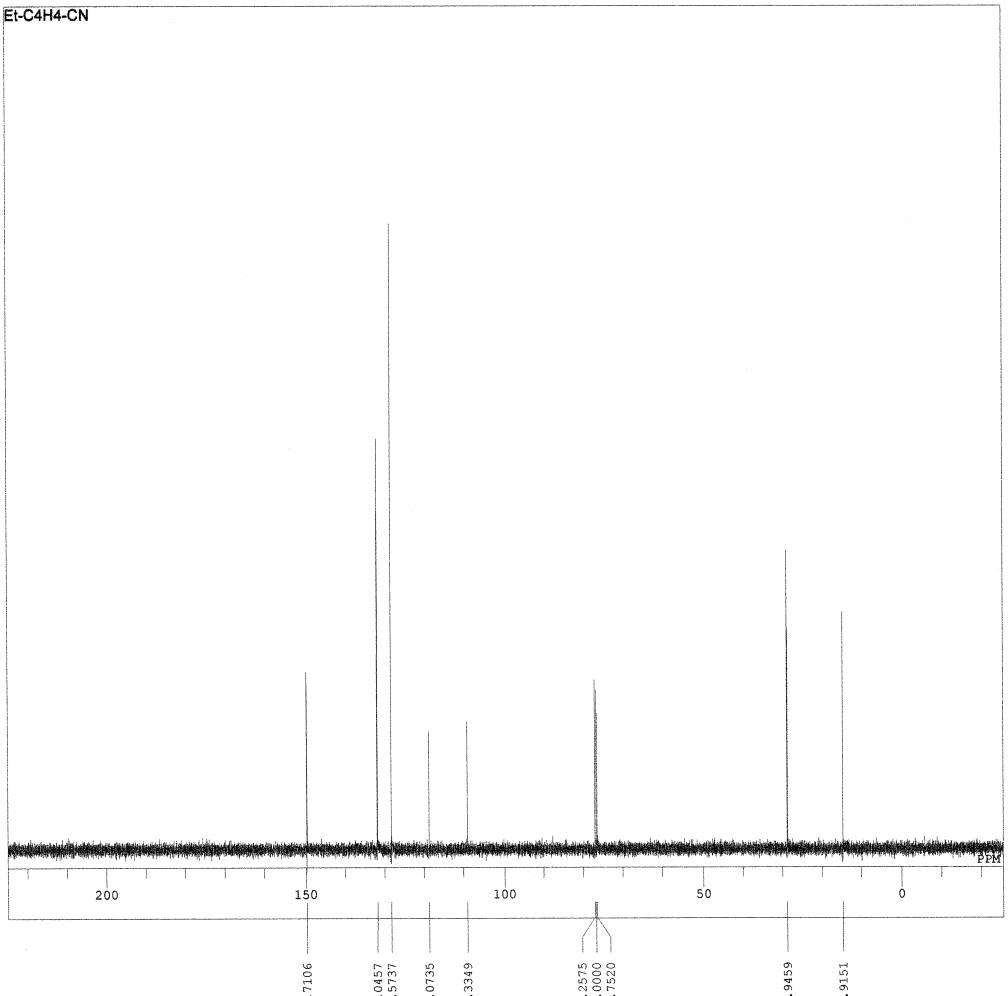

```
DFILE   ozawa08-086_13C.jdf
COMNT   Et-C4H4-NO2
DATIM   2015-08-28 12:28:57
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU        39308.18 Hz
SCANS             32
ACQTM          0.8336 sec
PD            10.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           21.8 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```



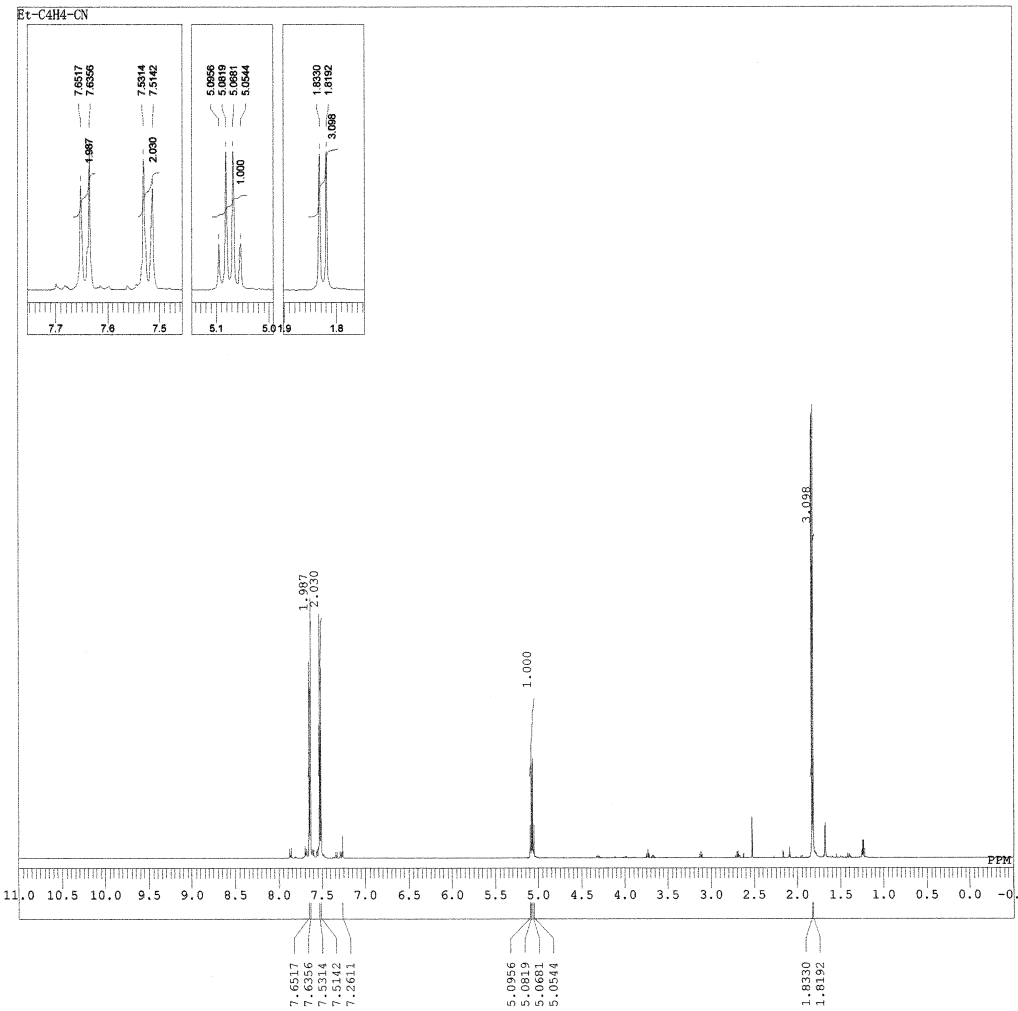

```
DFILE   _Et-C4H4-CN_1H.jdf
COMNT   Et-C4H4-CN
DATIM   2015-08-26 10:16:11
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS              4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           21.5 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
```



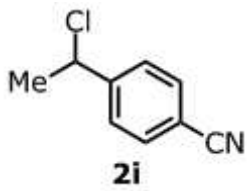
1i



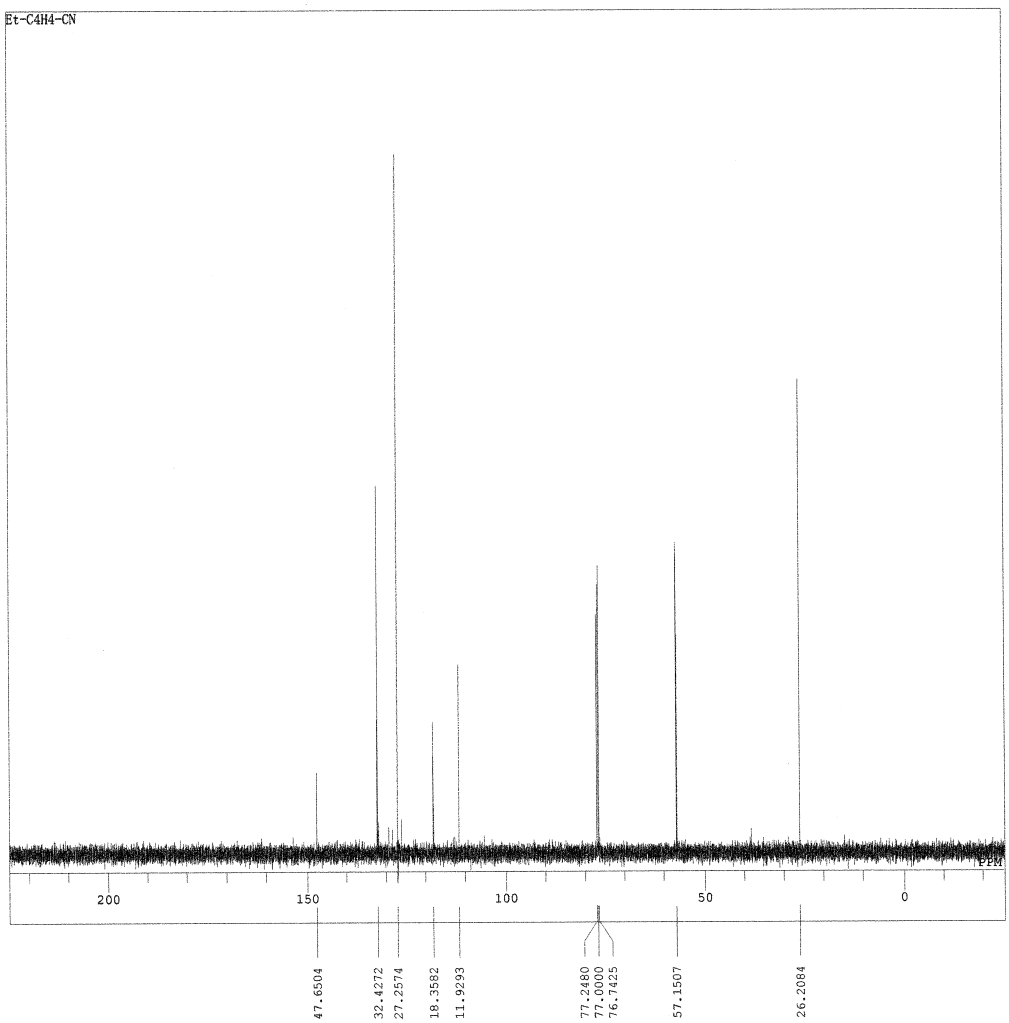

```
DFILE   _Et-C4H4-CN_13C.jdf
COMNT   Et-C4H4-CN
DATIM   2015-08-26 10:17:13
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU       39308.18 Hz
SCANS             12
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           21.6 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```



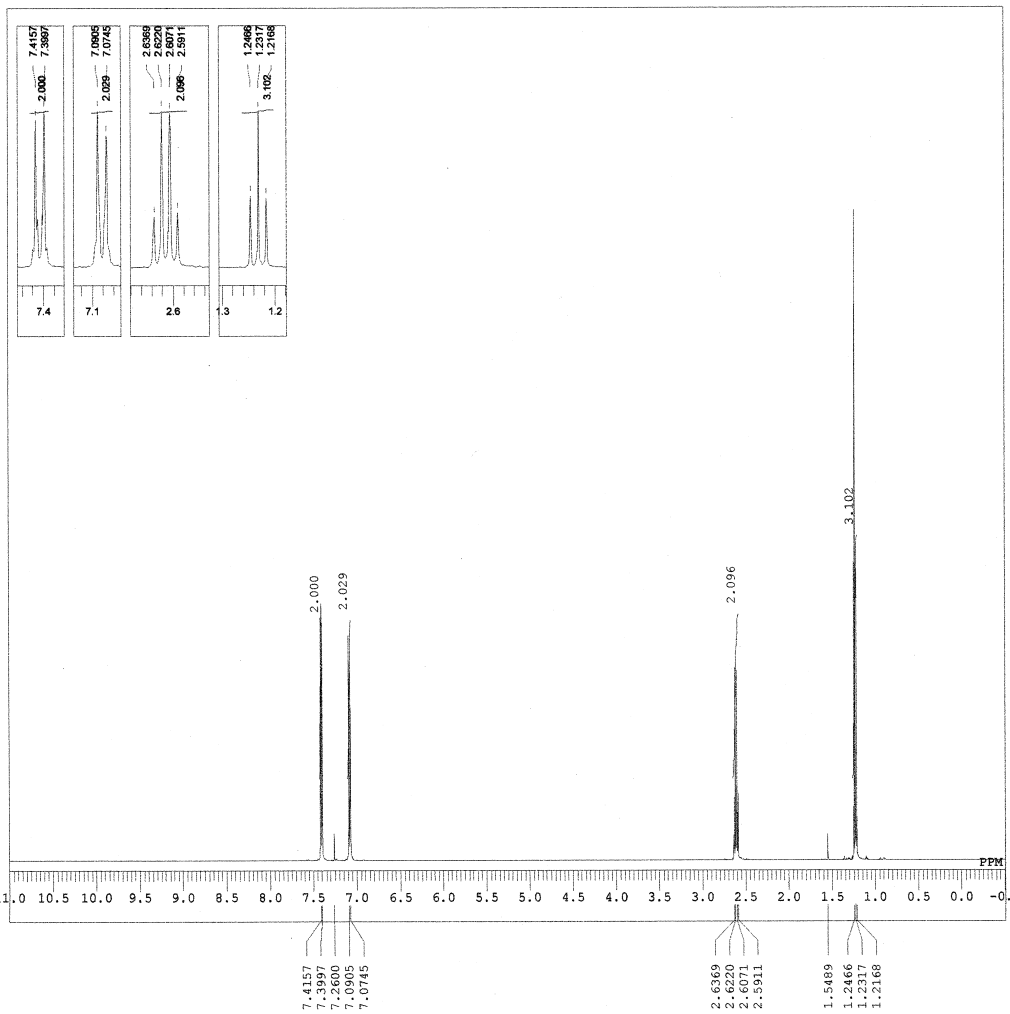

```
DFILE   ozawa08-087_1H.jdf
COMNT   Et-C4H4-CN
DATIM   2015-08-28 12:37:27
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS                4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1               5.55 usec
IRNUC   1H
CTEMP            21.6 c
SLVNT   CDCL3
EXREF            7.26 ppm
BF               0.12 Hz
RGAIN              30
```



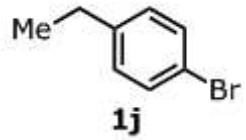
2i



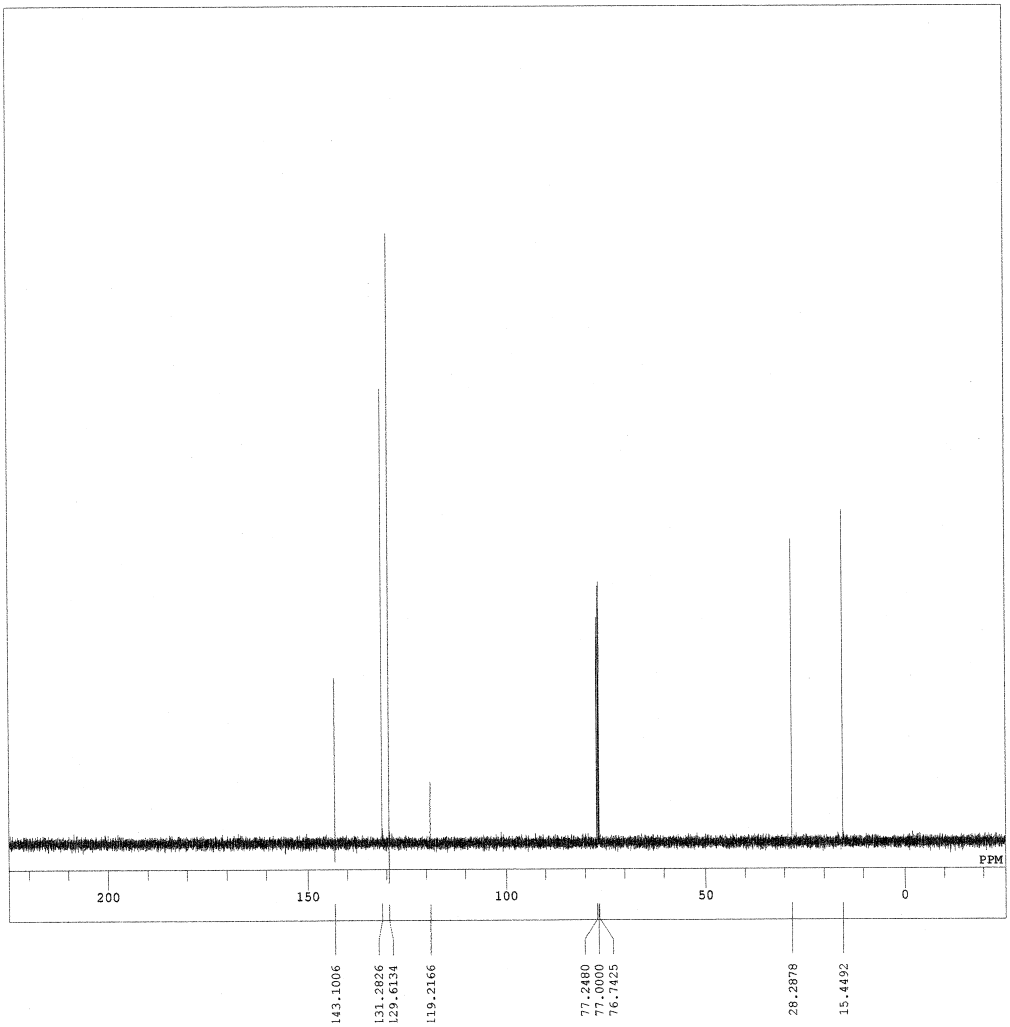

```
DFILE   ozawa08-087_13C.jdf
COMNT   Et-C4H4-CN
DATIM   2015-08-28 12:38:29
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU       39308.18 Hz
SCANS             20
ACQTM          0.8336 sec
PD             2.0000 sec
PW1               3.40 usec
IRNUC   1H
CTEMP            21.7 c
SLVNT   CDCL3
EXREF            77.00 ppm
BF               0.12 Hz
RGAIN              60
```



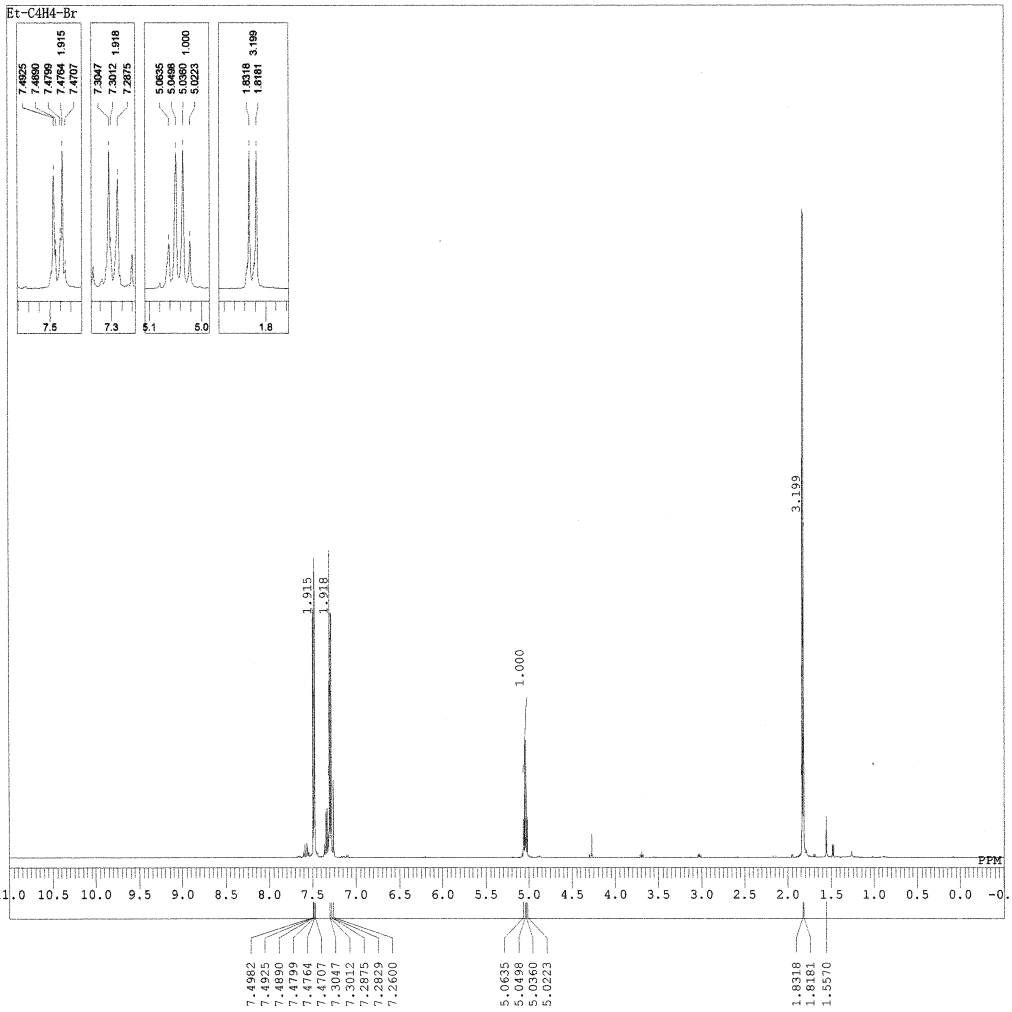

```
DFILE   _Et-C4H4-Br_1H.jdf
COMNT
DATIM   2016-01-12 10:40:32
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS              4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1            5.55 usec
IRNUC   1H
CTEMP           32.4 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             28
```



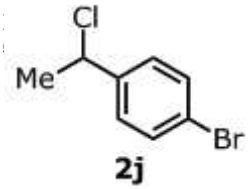
1j



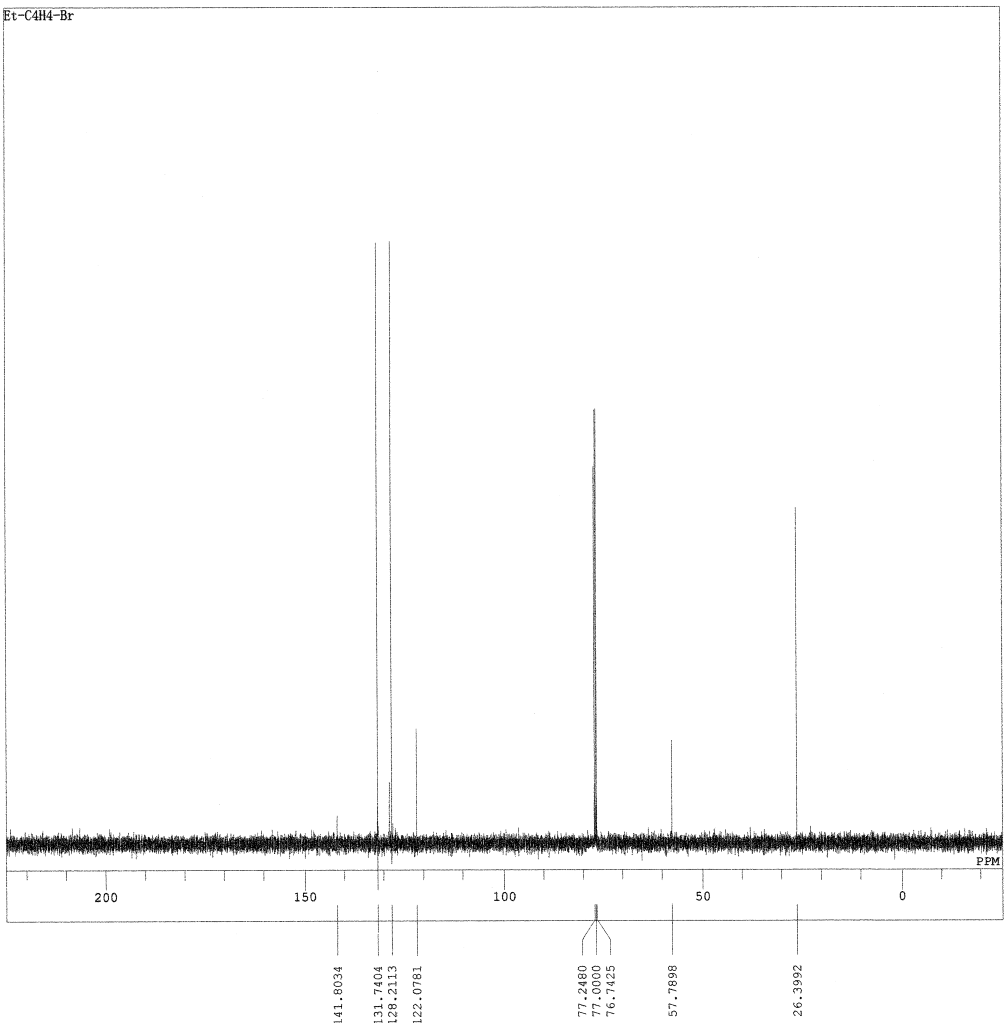

```
DFILE   _Et-C4H4-Br_13C.jdf
COMNT
DATIM   2016-01-12 10:41:34
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU        39308.18 Hz
SCANS             44
ACQTM          0.8336 sec
PD             2.0000 sec
PW1             3.40 usec
IRNUC   1H
CTEMP           33.1 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```



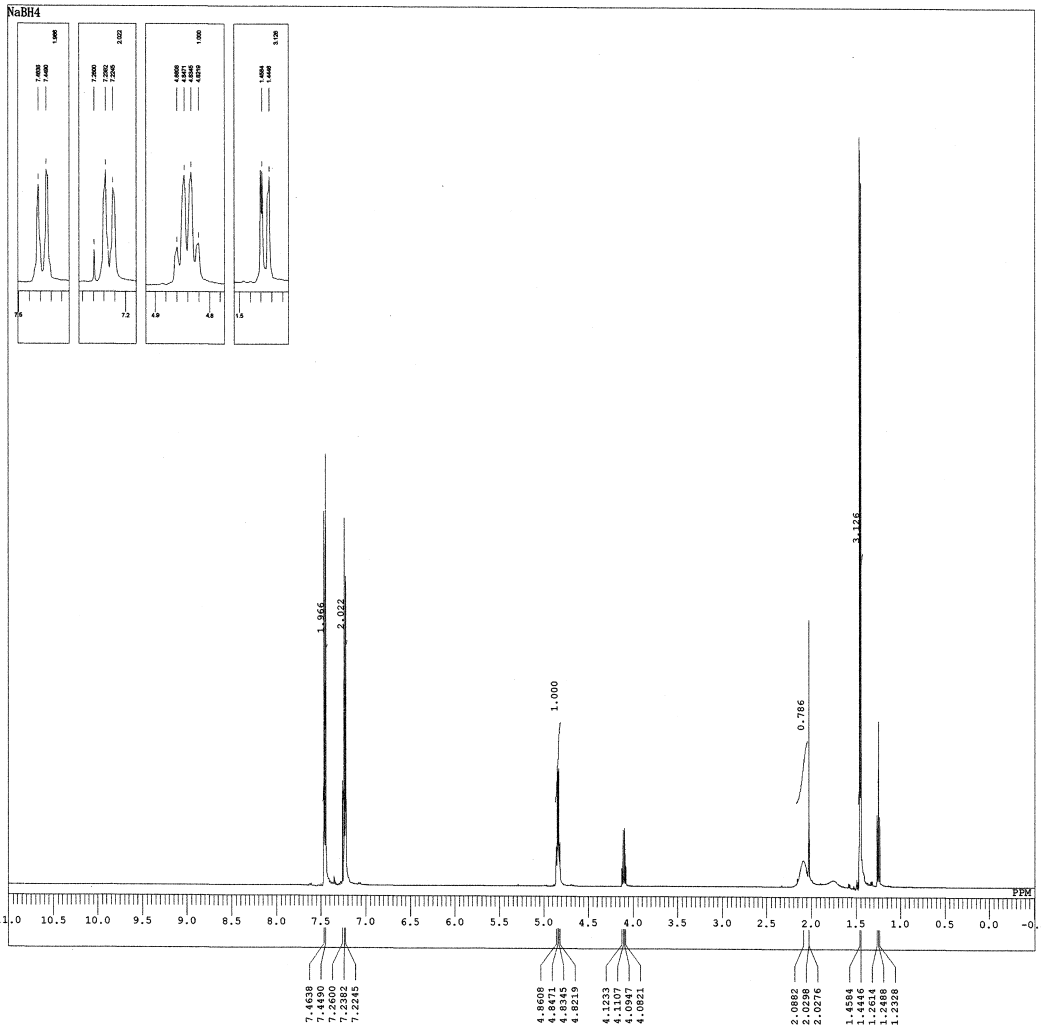

```
DFILE   ozawa08-154_1H.jdf
COMNT   Et-C4H4-Br
DATIM   2015-12-28 14:09:47
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           33.2 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
```



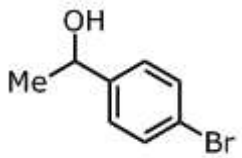
2j



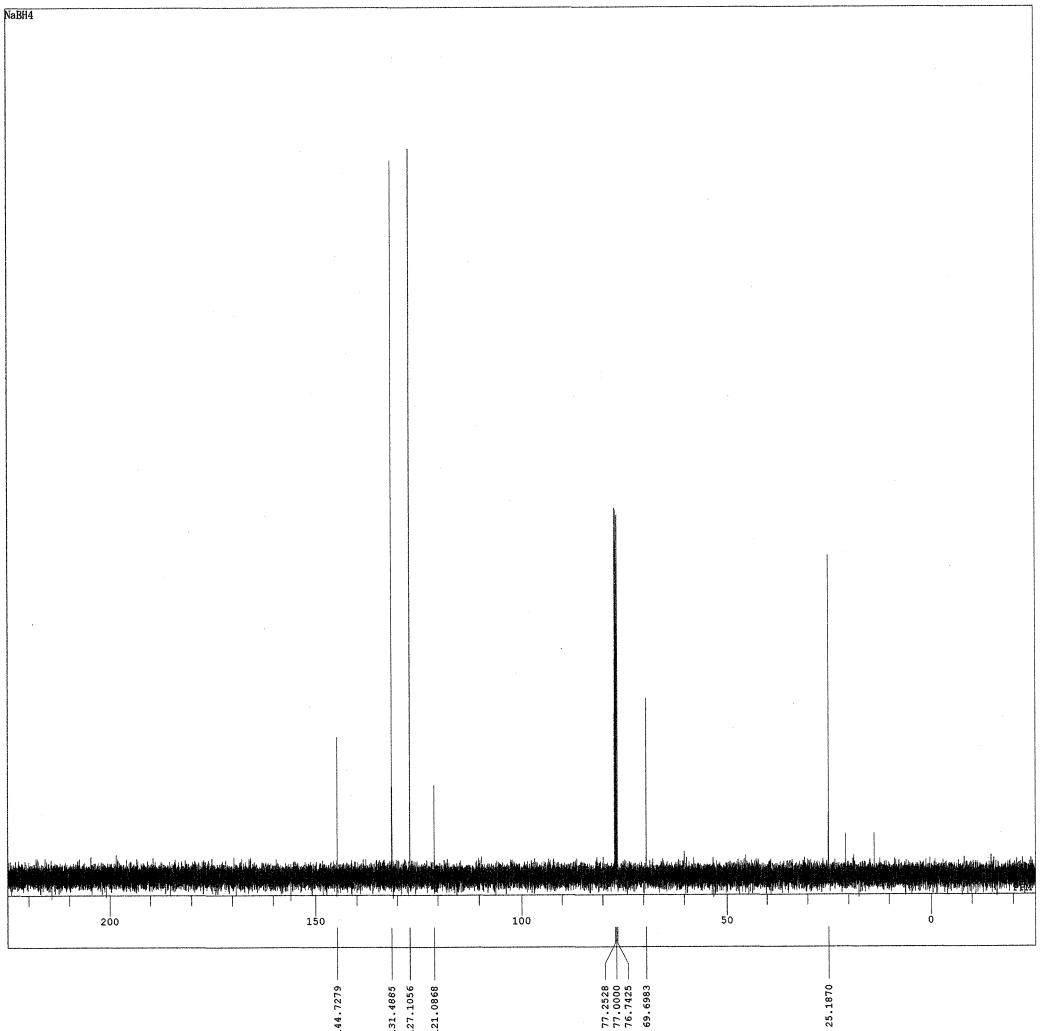

```
DFILE   ozawa08-154_13C.jdf
COMNT   Et-C4H4-Br
DATIM   2015-12-28 14:11:14
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.07 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU       39308.18 Hz
SCANS             60
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           30.2 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```



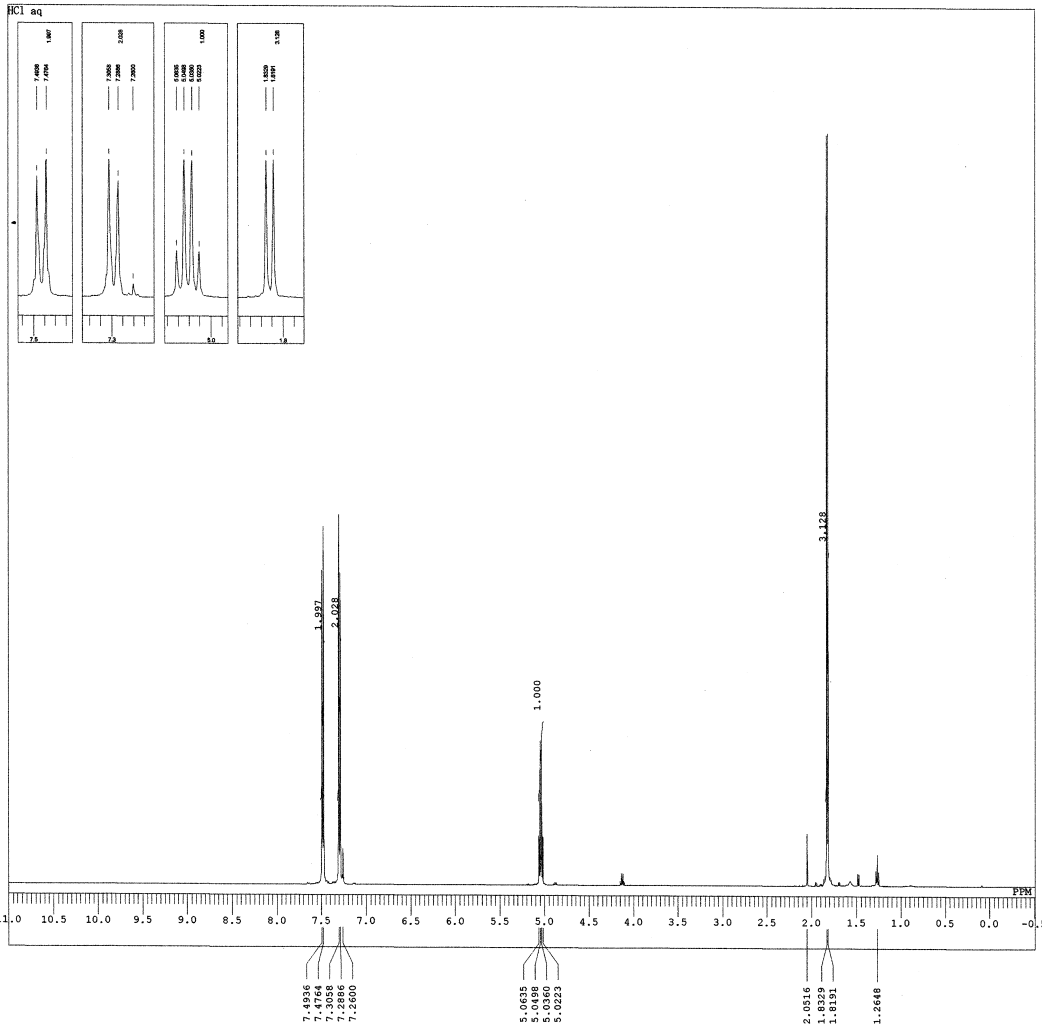
DFILE ozawa09-124_1H.als
COMNT NaBH4
DATIM 2016-08-10 15:18:53
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 30



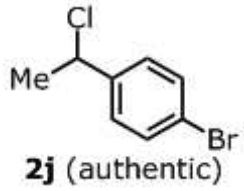
S1j



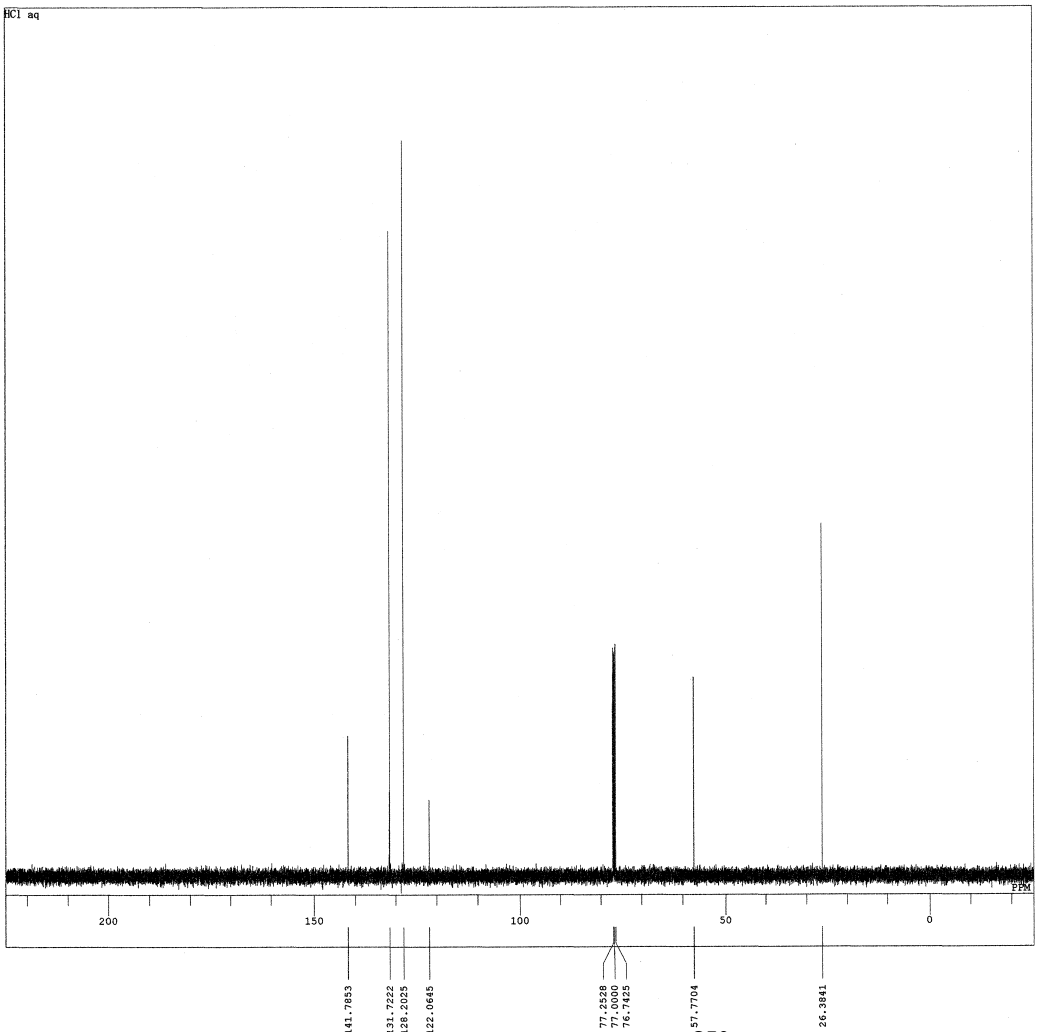
DFILE ozawa09-124_13C.als
COMNT NaBH4
DATIM 2016-08-10 15:19:53
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.54 Hz
SCANS 20
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



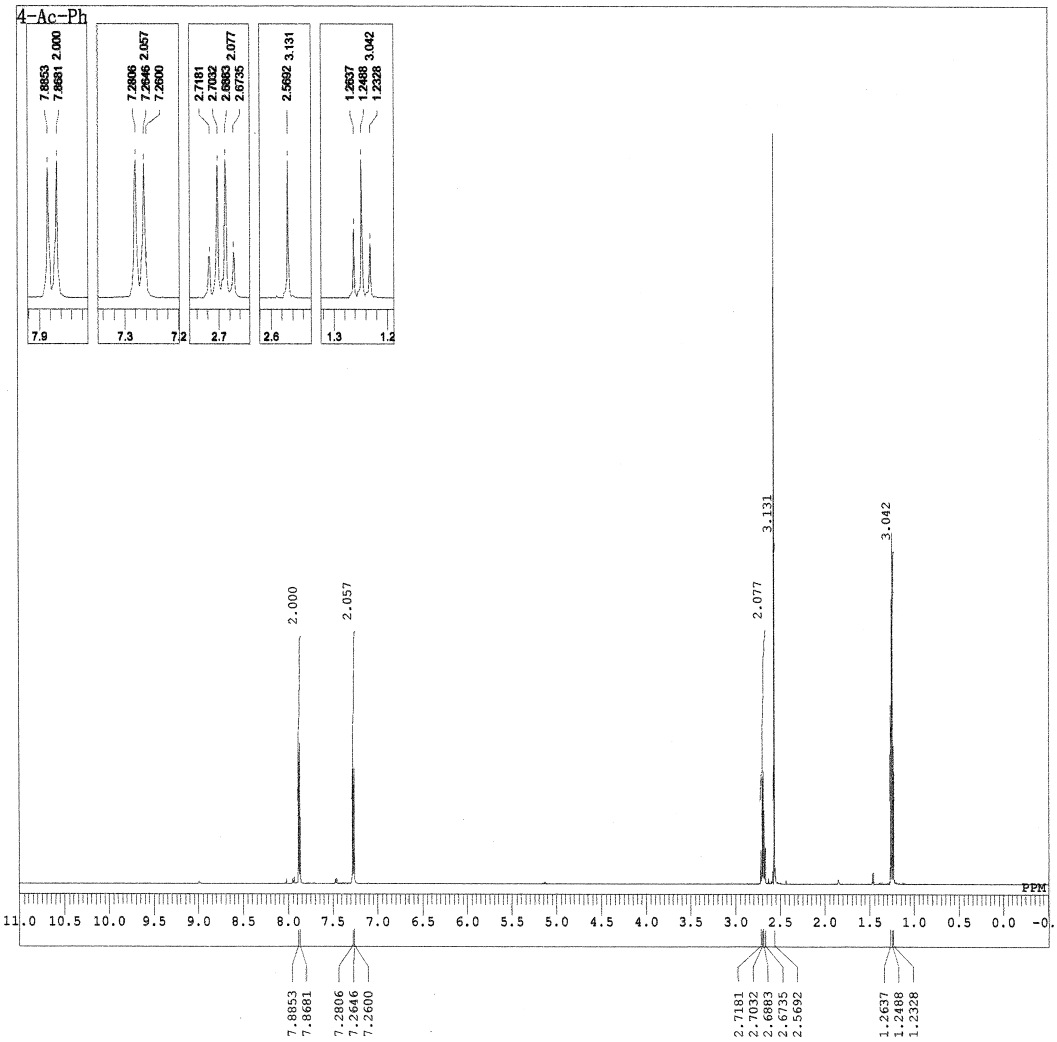

```
DFILE  ozawa09-125_1H.als
COMNT  HCl aq
DATIM  2016-08-12 14:51:49
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.51 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1            5.55 usec
IRNUC  1H
CTEMP         21.3 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            30
```



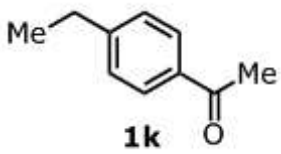
2j (authentic)



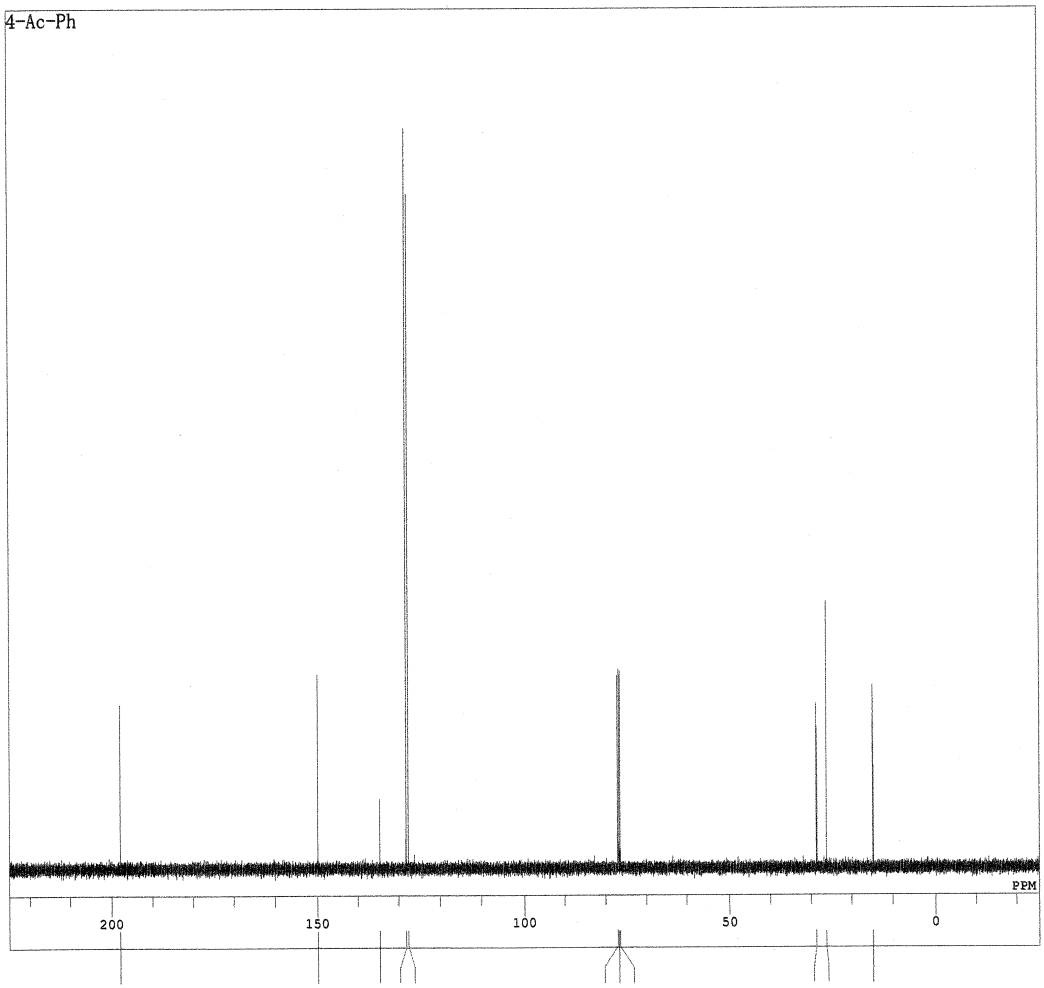

```
DFILE  ozawa09-125_13C.als
COMNT  HCl aq
DATIM  2016-08-12 14:52:48
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         52428
FREQU       31446.54 Hz
SCANS            20
ACQTM          1.6672 sec
PD             2.0000 sec
PW1            3.40 usec
IRNUC  1H
CTEMP         21.5 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.12 Hz
RGAIN            60
```



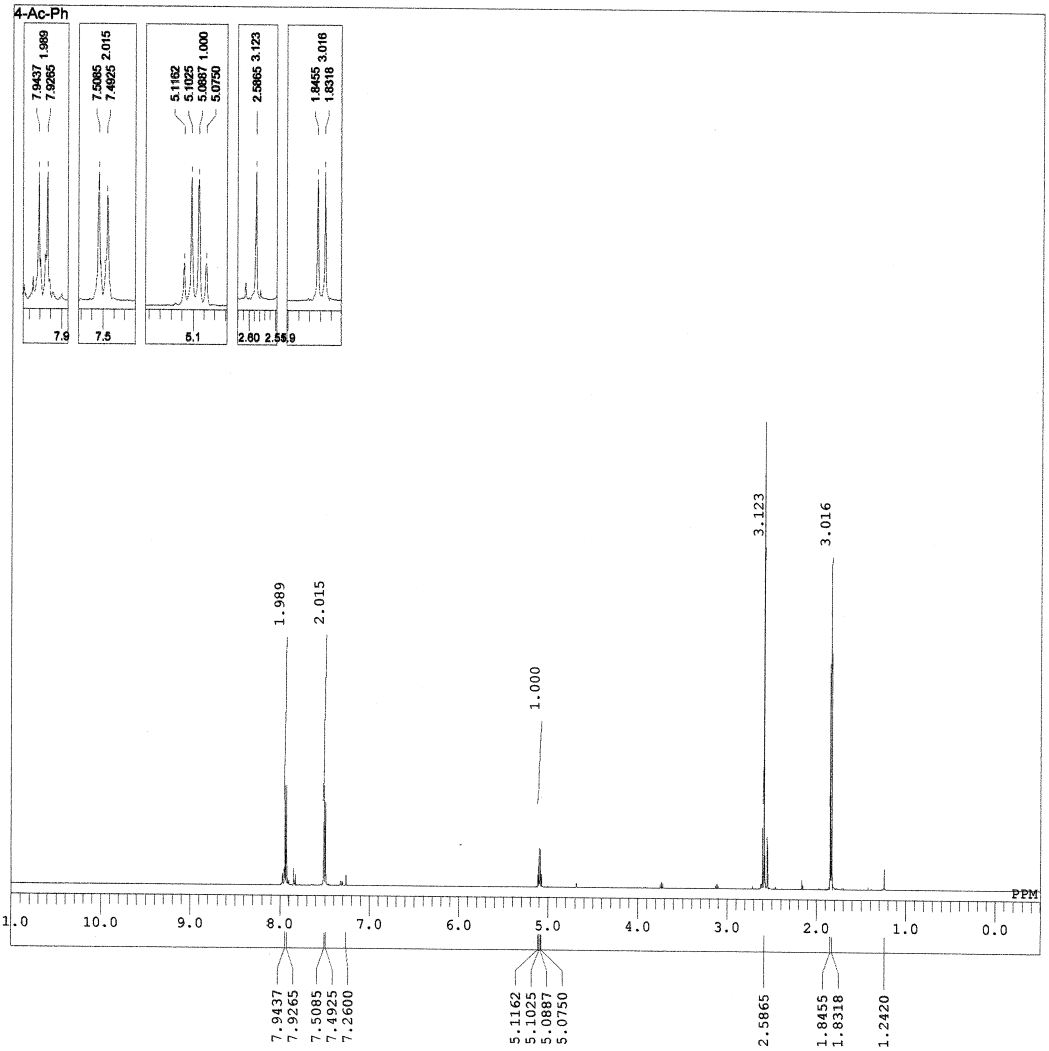

```
DFILE   ozawa10-046_SM_1H.als
COMNT   4-Ac-Ph
DATIM   2016-11-28 16:02:25
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          13107
FREQU        7507.51 Hz
SCANS                4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           21.3 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             28
```



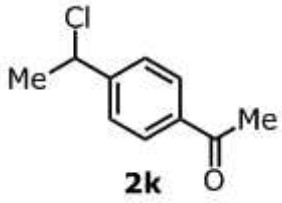
1k

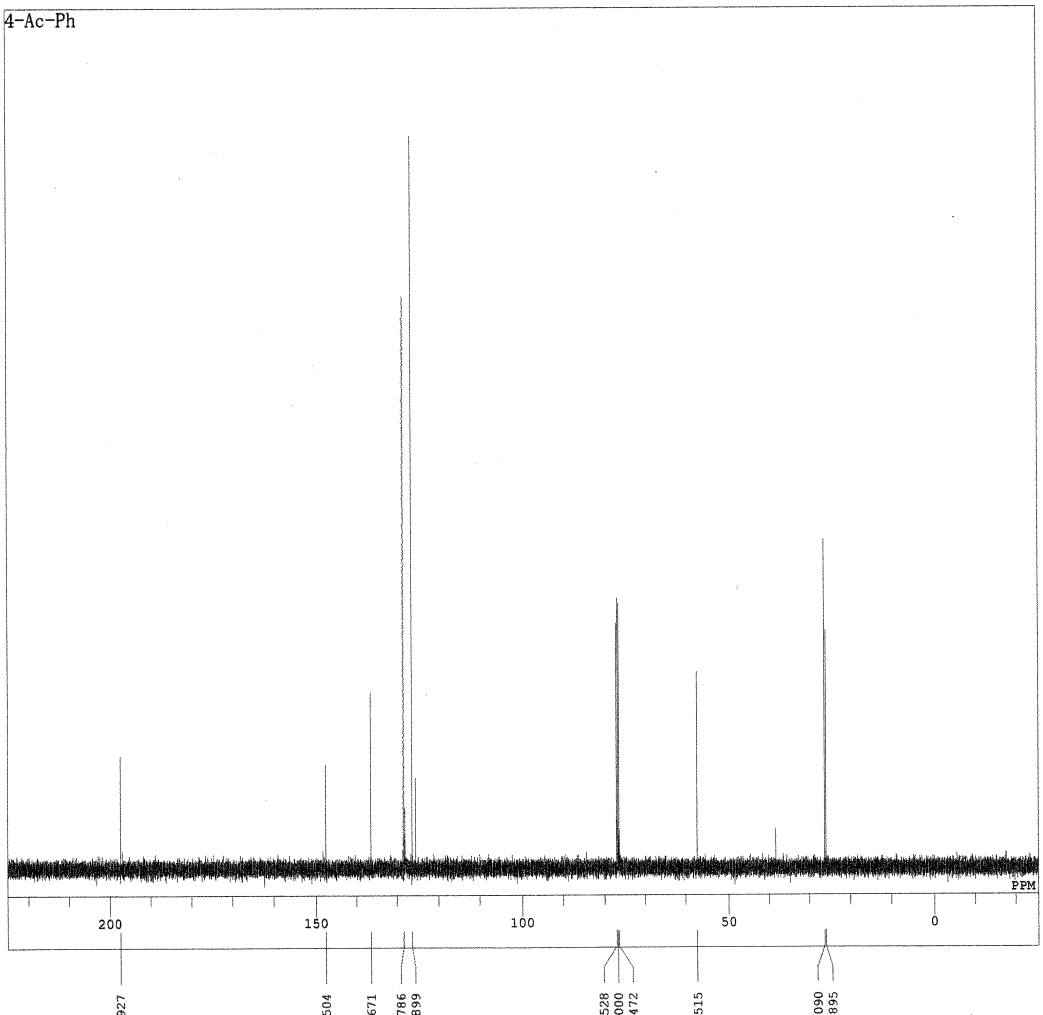



```
DFILE   ozawa10-046_SM_13C.jdf
COMNT   4-Ac-Ph
DATIM   2016-11-28 16:04:20
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          65535
FREQU       39308.18 Hz
SCANS              20
ACQTM          1.6672 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           21.3 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             58
```

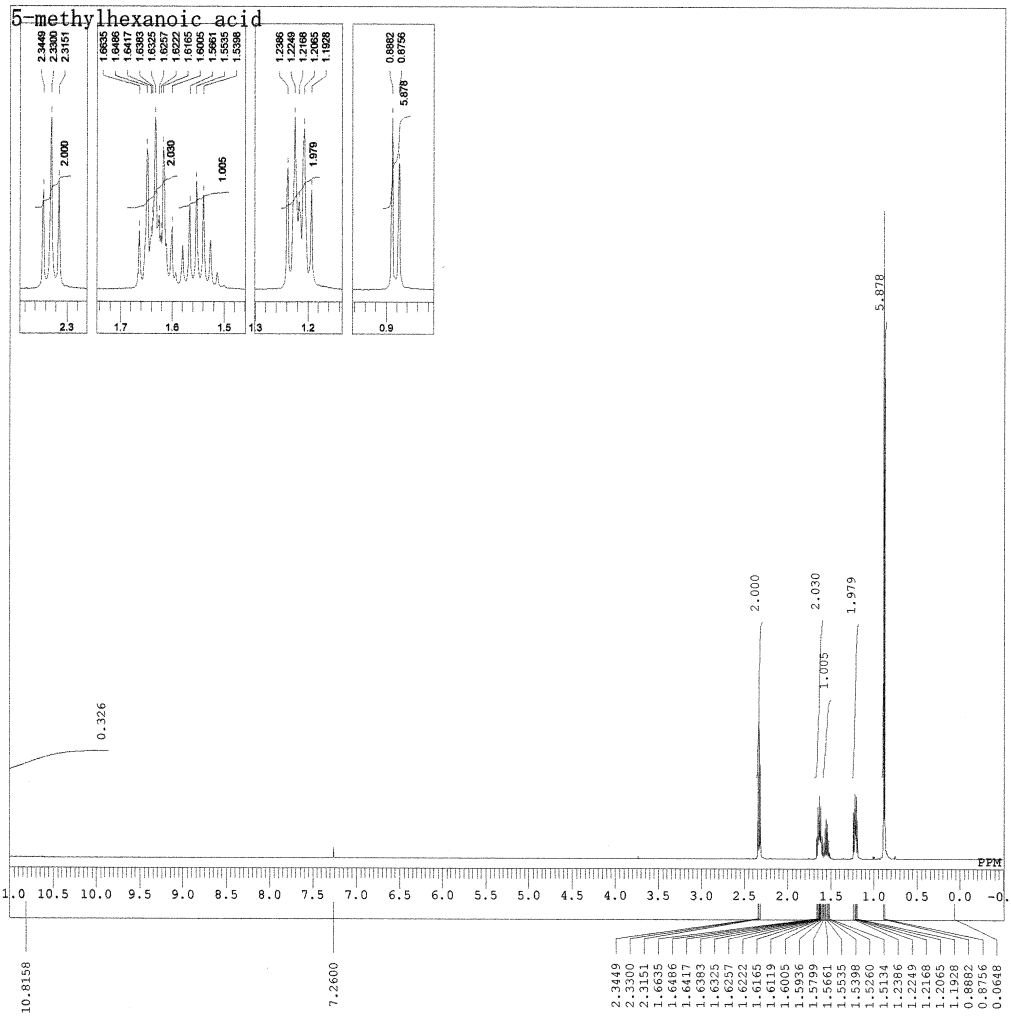



```
DFILE   ozawa10-046_1H.jdf
COMNT   4-Ac-Ph
DATIM   2016-11-30 13:21:09
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           21.0 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             28
```

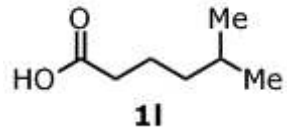




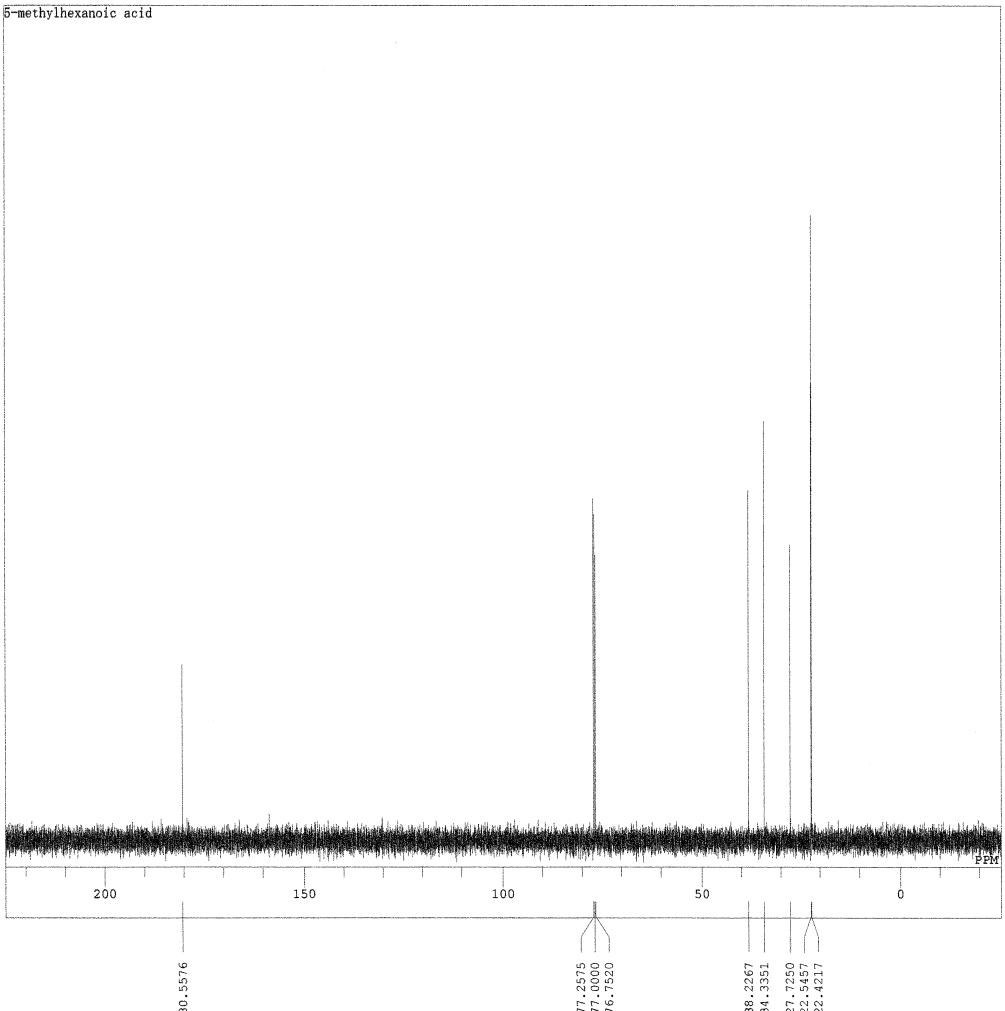

```
DFILE   ozawa10-046_13C.jdf
COMNT   4-Ac-Ph
DATIM   2016-11-30 13:22:13
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          65535
FREQU       39308.18 Hz
SCANS            16
ACQTM          1.6672 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           21.1 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             58
```



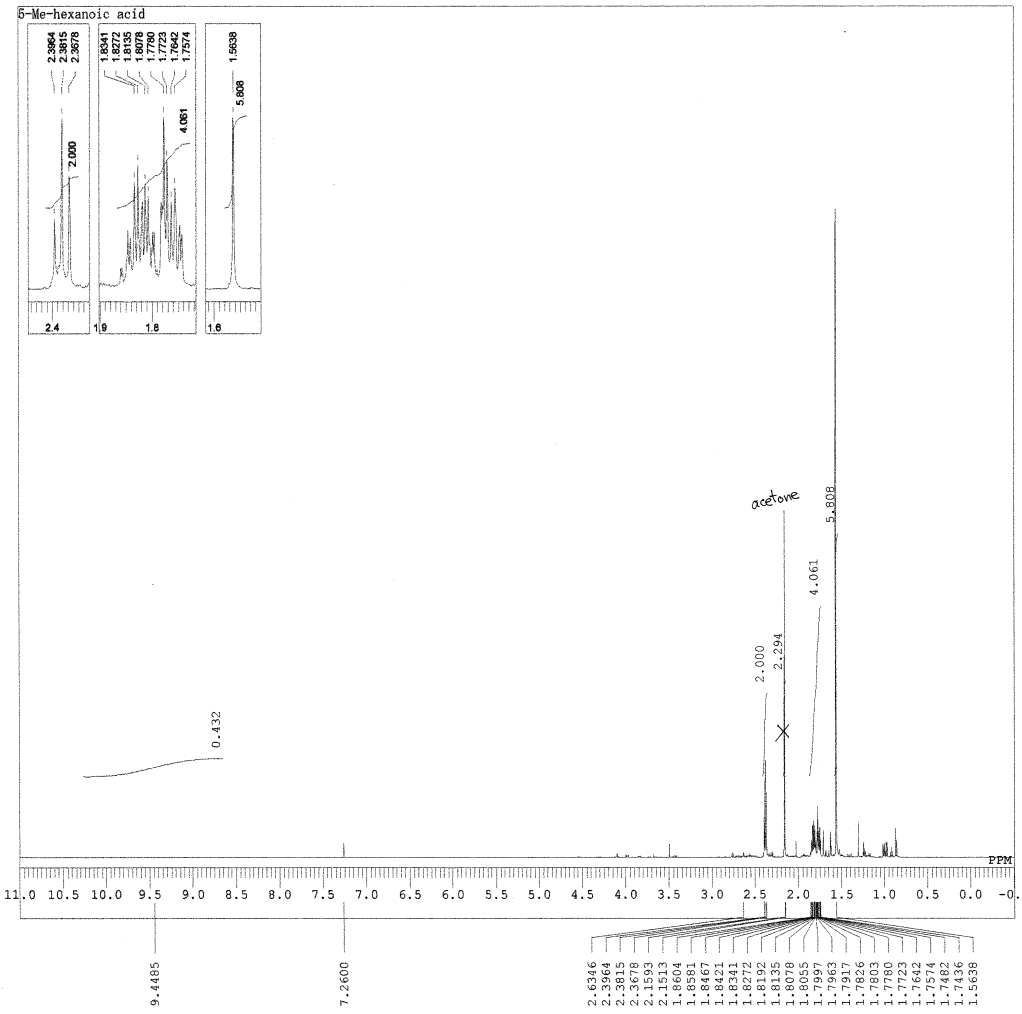

```
DFILE   _5-methylhexanoic acid_1H.jdf
COMNT   5-methylhexanoic acid
DATIM   2015-08-25 12:34:05
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1            5.55 usec
IRNUC   1H
CTEMP          21.3 c
SLVNT   CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            30
```



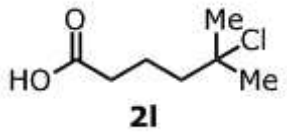
1l



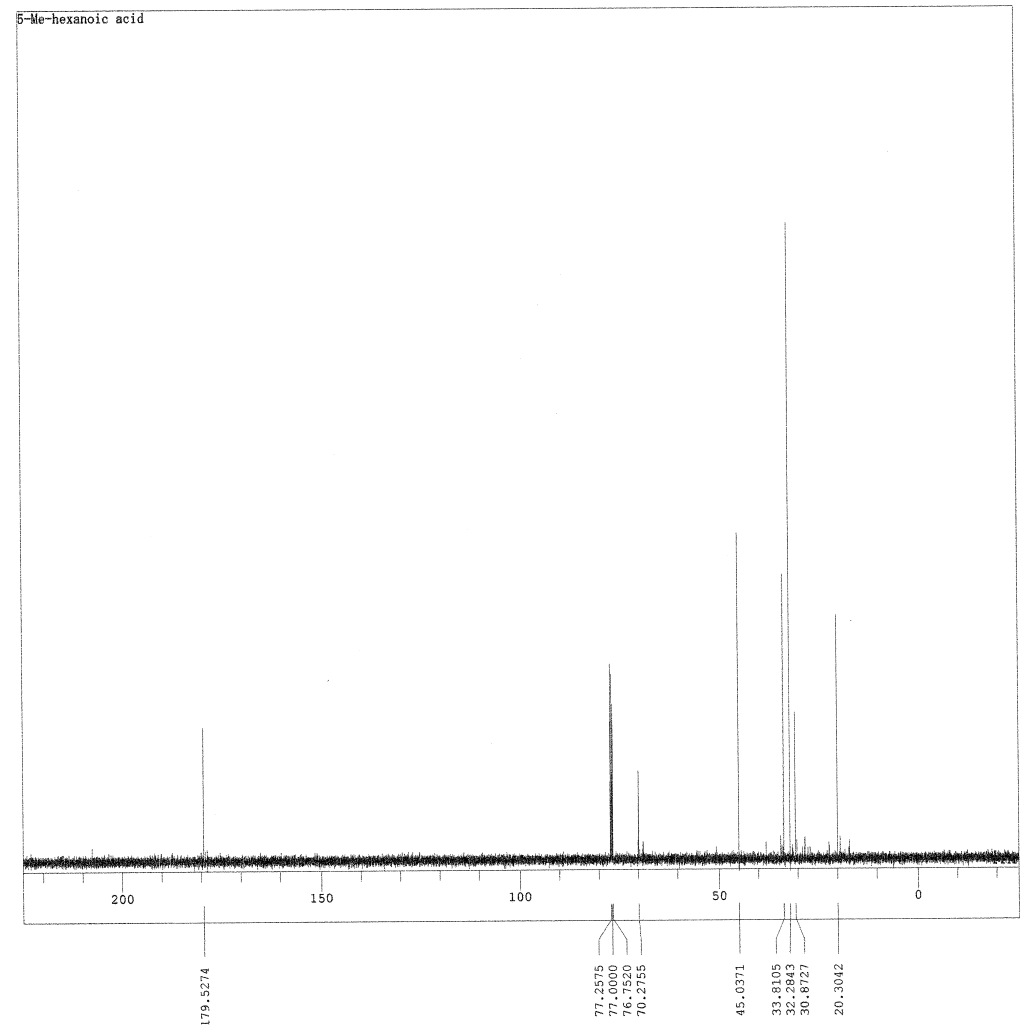

```
DFILE   _5-methylhexanoic acid_13C.jdf
COMNT   5-methylhexanoic acid
DATIM   2015-08-25 12:35:07
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU       39308.18 Hz
SCANS            12
ACQTM          0.8336 sec
PD             2.0000 sec
PW1            3.40 usec
IRNUC   1H
CTEMP          21.4 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF             0.12 Hz
RGAIN            60
```



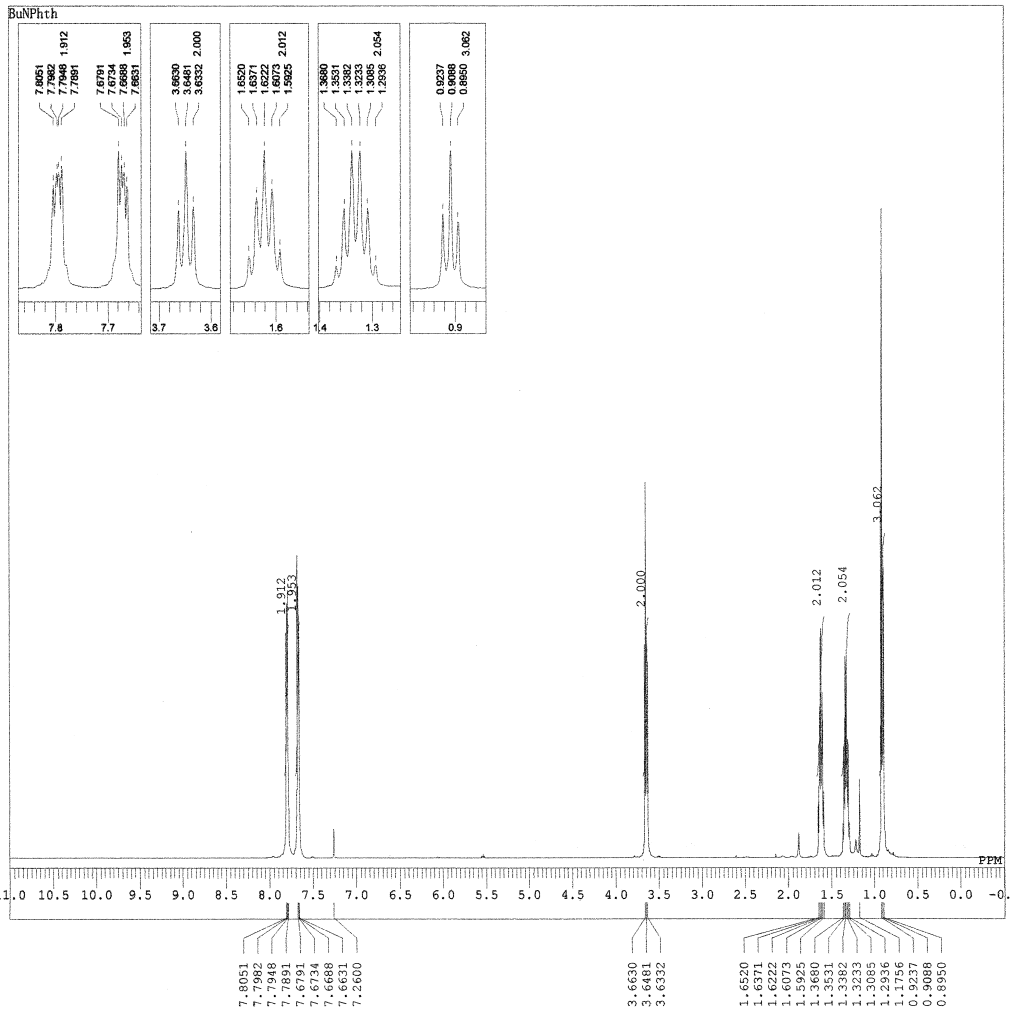

```
DFILE   ozawa08-076_1H.jdf
COMNT   5-Me-hexanoic acid
DATIM   2015-08-25 12:43:19
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS                4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           21.2 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             28
```



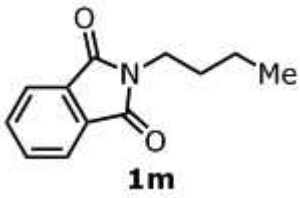
2l



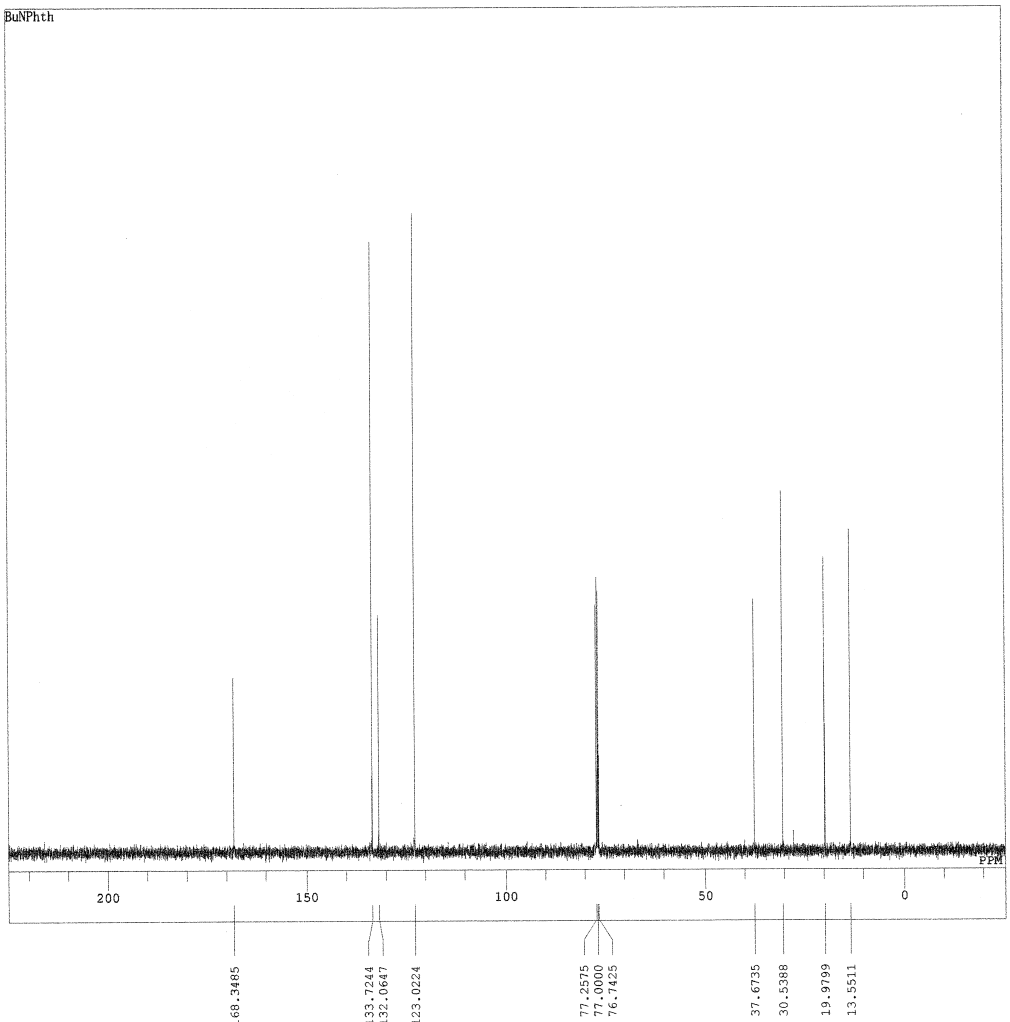

```
DFILE   ozawa08-076_13C.jdf
COMNT   5-Me-hexanoic acid
DATIM   2015-08-25 12:44:22
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU        39308.18 Hz
SCANS              32
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           21.5 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```



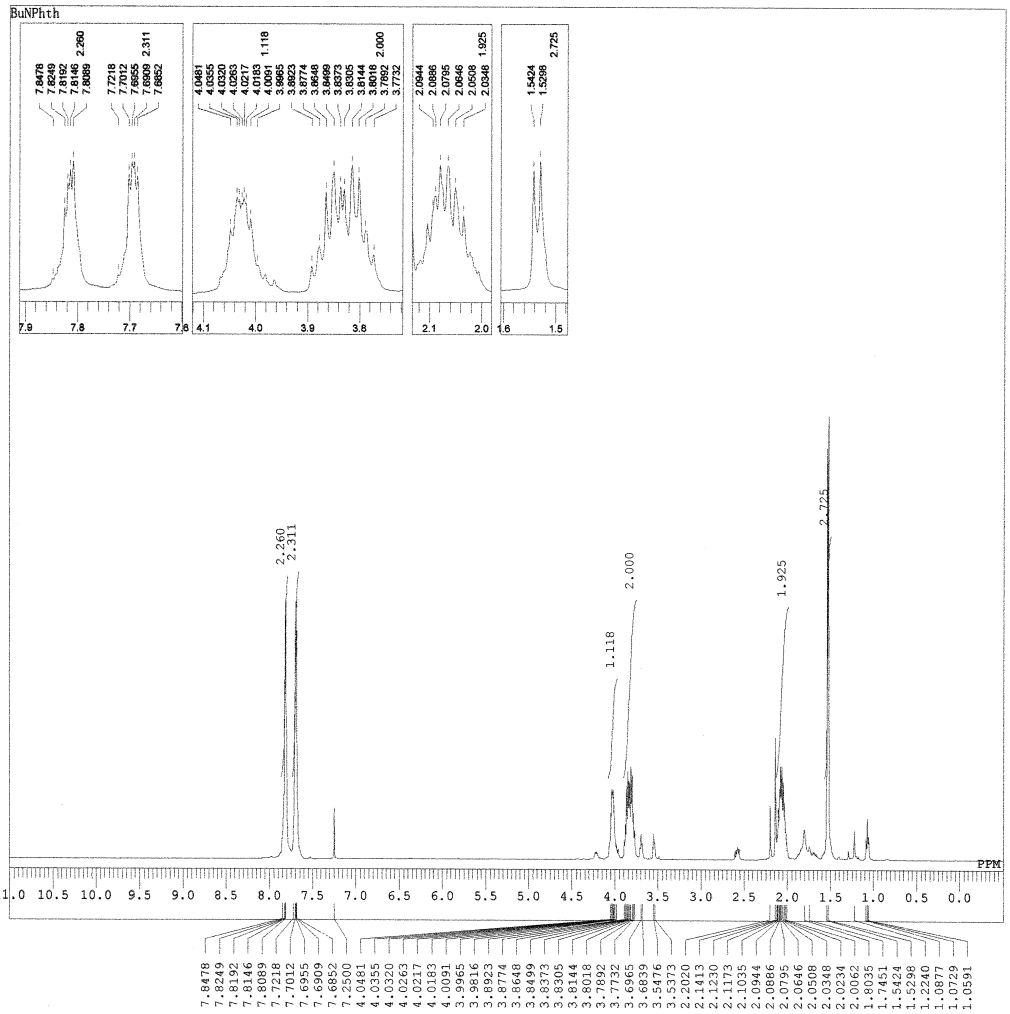

```
DFILE   ozawa09-065_rsm_1H.jdf
COMNT   BuNPhth
DATIM   2015-12-24 17:06:28
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1               5.55 usec
IRNUC   1H
CTEMP            33.8 c
SLVNT   CDCL3
EXREF             7.26 ppm
BF                1.20 Hz
RGAIN               24
```



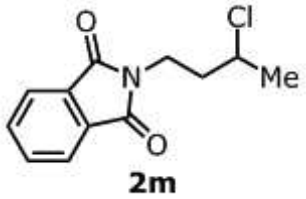
1m



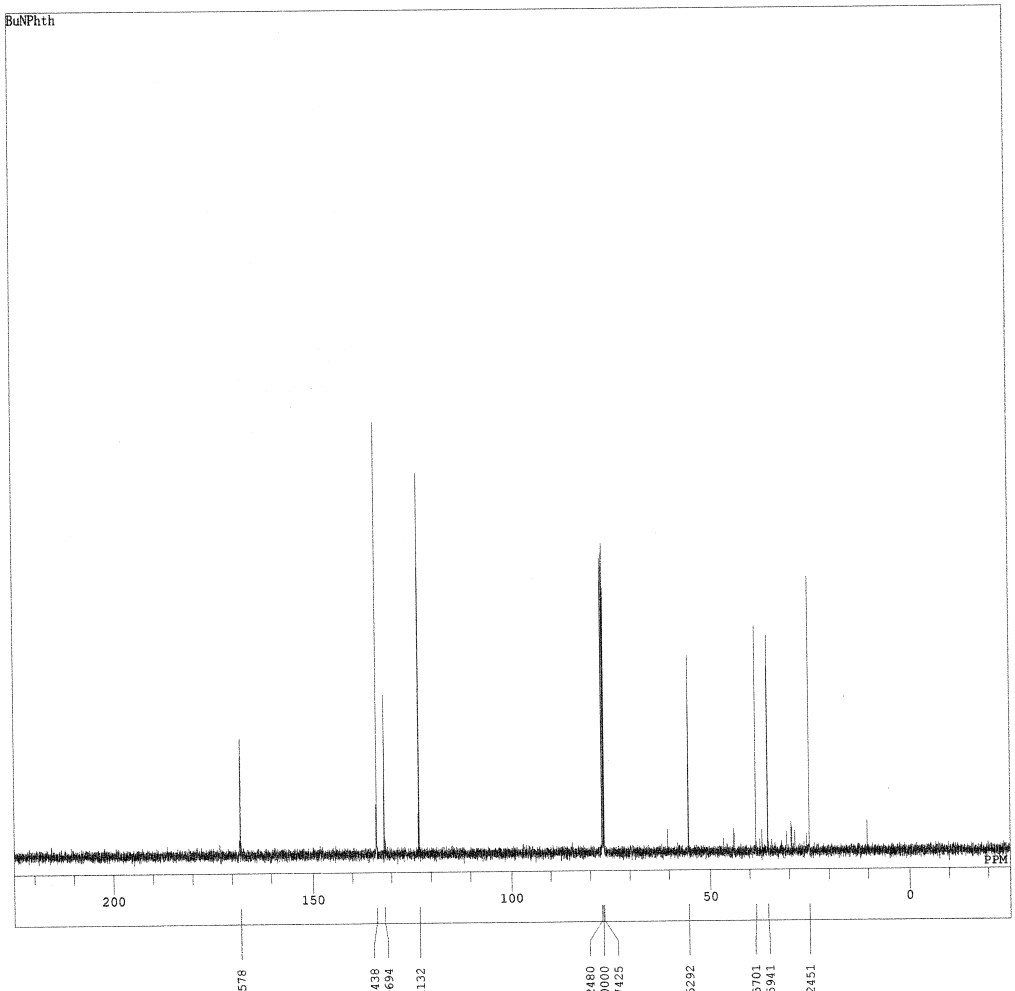

```
DFILE   ozawa09-065_rsm_13C.jdf
COMNT   BuNPhth
DATIM   2015-12-24 17:07:35
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU        39308.18 Hz
SCANS             17
ACQTM          0.8336 sec
PD             2.0000 sec
PW1               3.40 usec
IRNUC   1H
CTEMP            30.0 c
SLVNT   CDCL3
EXREF             77.00 ppm
BF                1.20 Hz
RGAIN               60
```



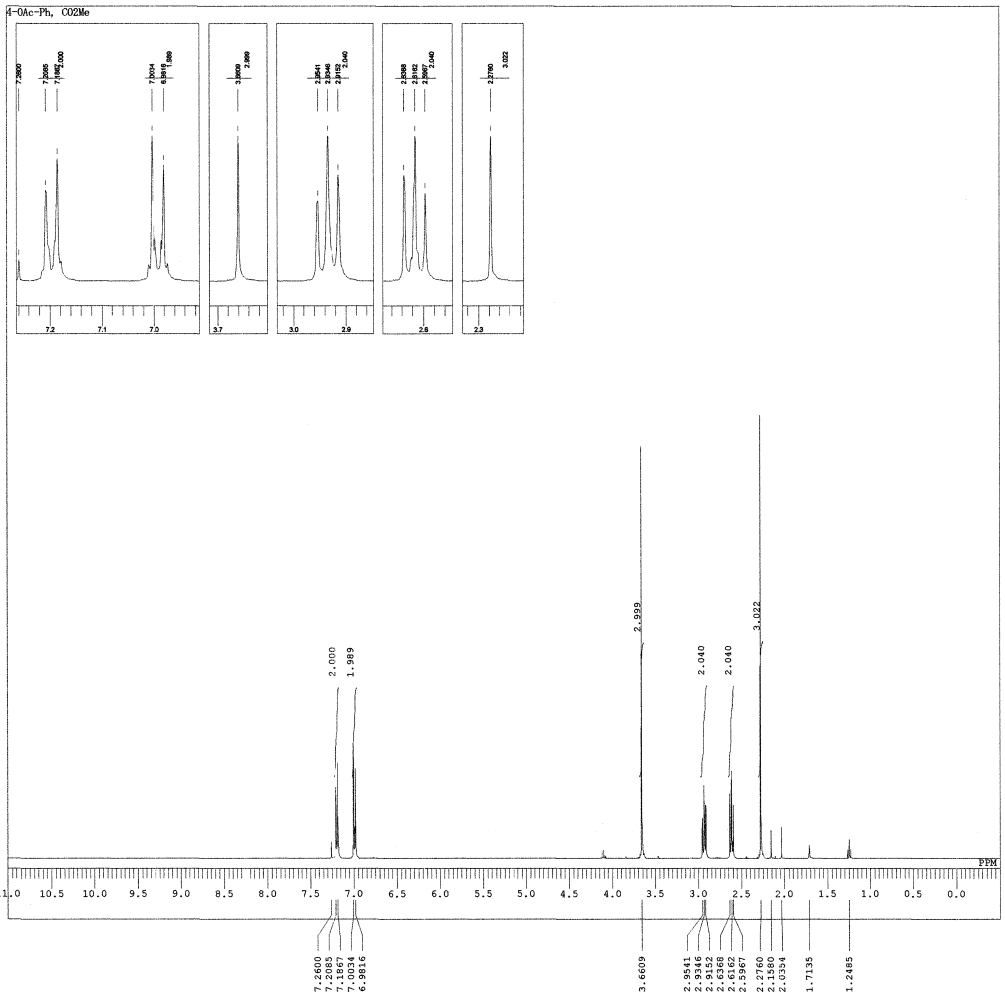

```
DFILE   ozawa09-065_1H.jdf
COMNT   BuNPhth
DATIM   2015-12-24 16:59:35
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           31.9 c
SLVNT   CDCL3
EXREF           7.25 ppm
BF              1.20 Hz
RGAIN             26
```



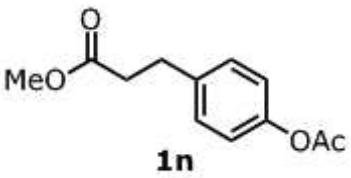
2m



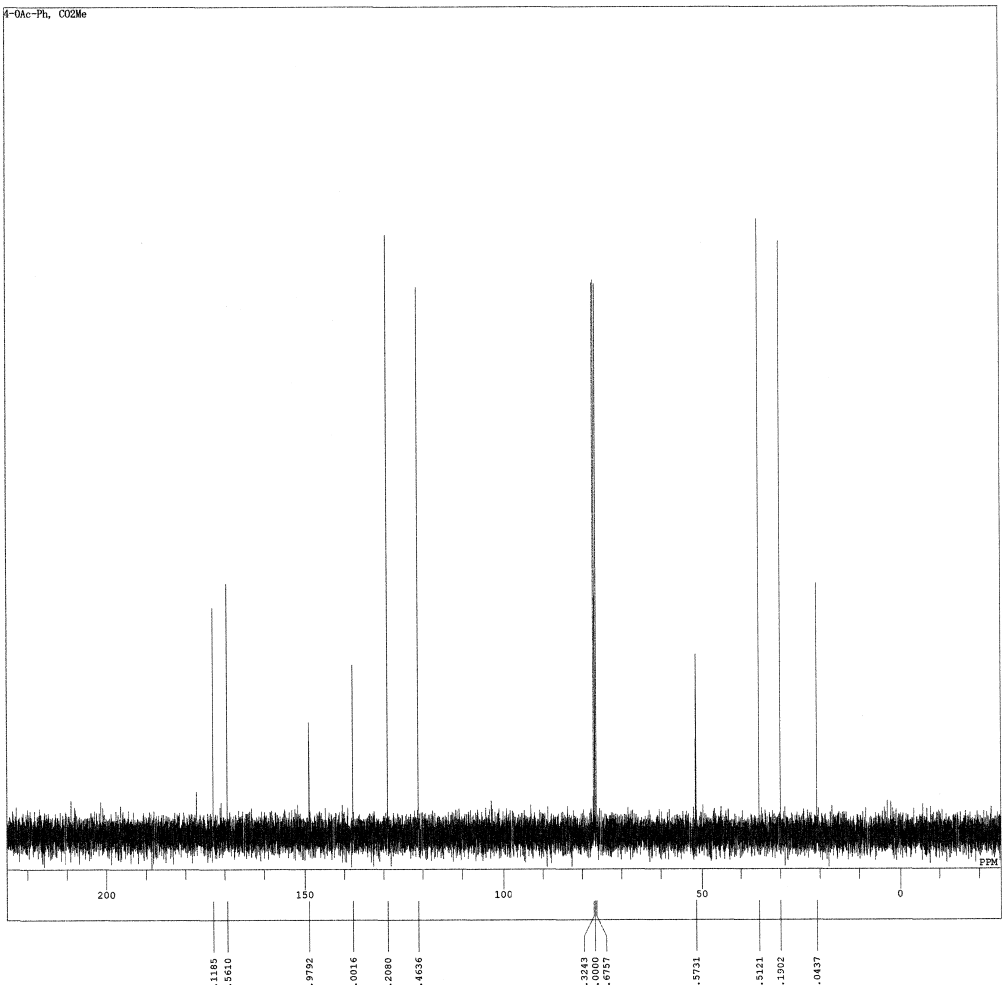

```
DFILE   ozawa09-065_13C.jdf
COMNT   BuNPhth
DATIM   2015-12-24 17:00:37
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU       39308.18 Hz
SCANS             60
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           33.3 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              1.20 Hz
RGAIN             60
```



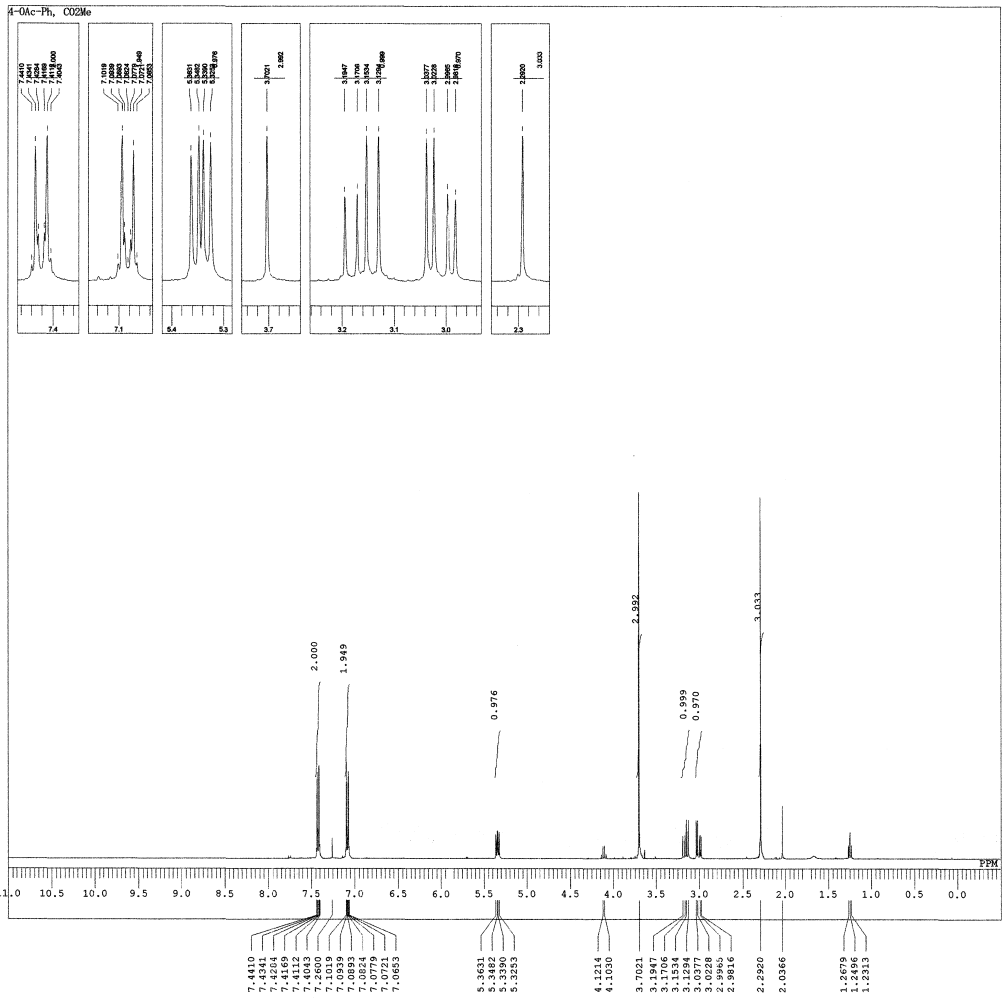

```
DFILE  ozawa09-016_1H.jdf
COMNT  4-OAc-Ph, CO2Me
DATIM  18-11-2015 17:55:28
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         16384
FREQU       7352.94 Hz
SCANS             4
ACQTM          2.2282 sec
PD             5.0000 sec
PW1              4.99 usec
IRNUC  1H
CTEMP           20.1 c
SLVNT  CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             26
```



1n

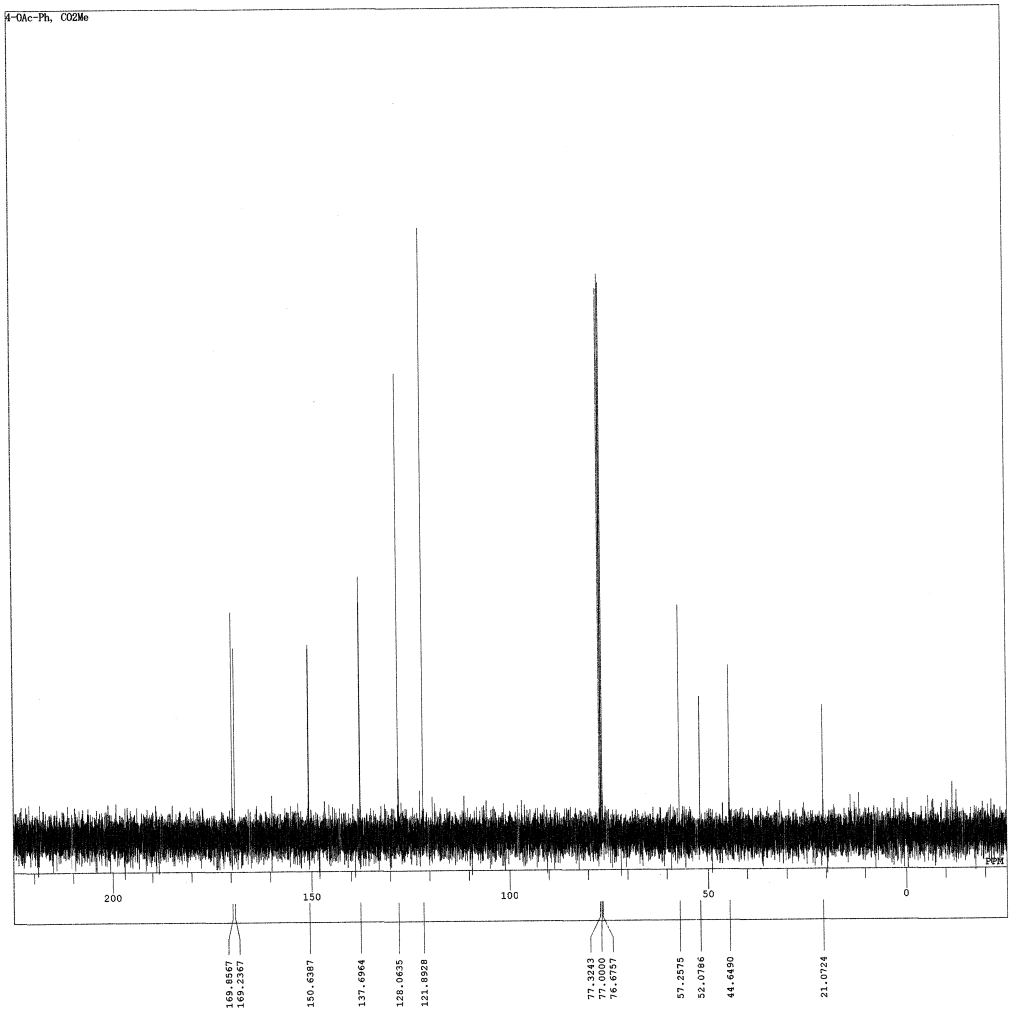



```
DFILE  ozawa09-016_13C.jdf
COMNT  4-OAc-Ph, CO2Me
DATIM  18-11-2015 17:56:31
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         32767
FREQU      30788.18 Hz
SCANS            44
ACQTM          1.0643 sec
PD             2.0000 sec
PW1              3.16 usec
IRNUC  1H
CTEMP           20.3 c
SLVNT  CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```

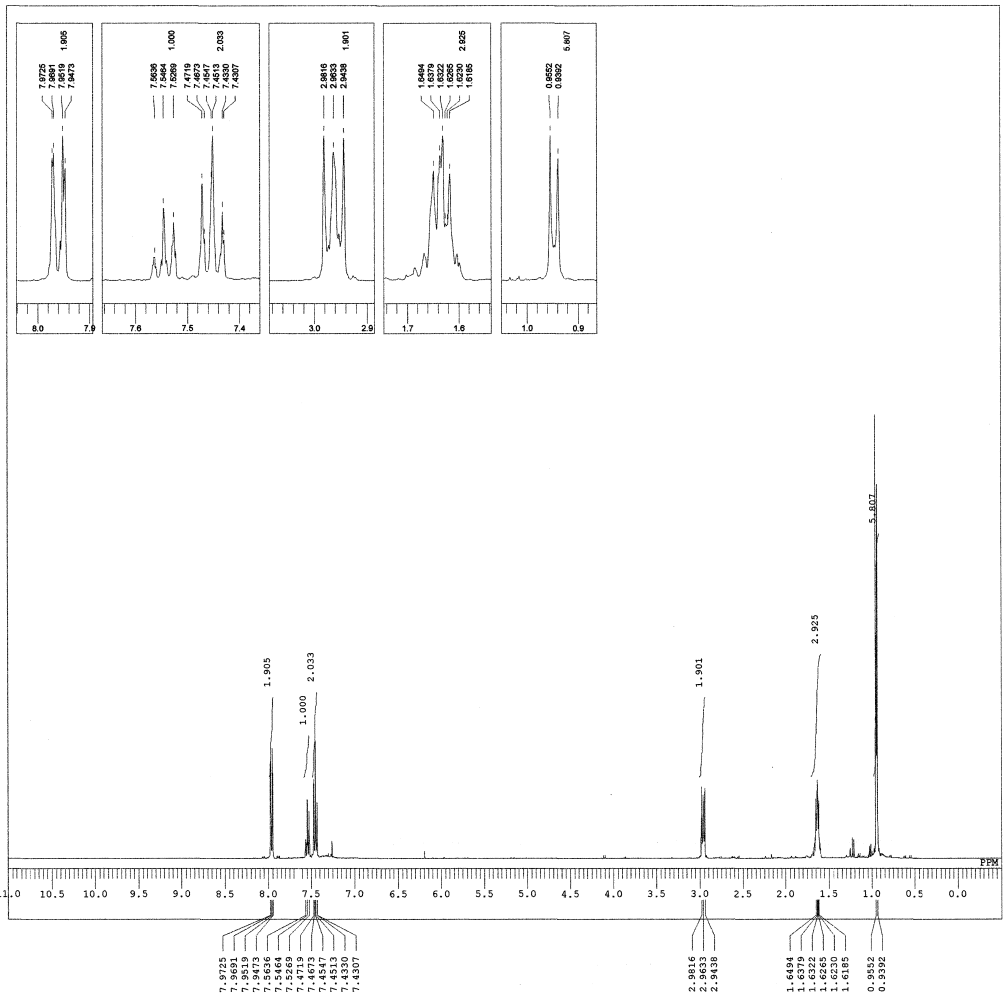



```
DFILE  ozawa09-019_1H.jdf
COMNT  4-OAc-Ph, CO2Me
DATIM  18-11-2015 18:01:12
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         16384
FREQU       7352.94 Hz
SCANS             4
ACQTM         2.2282 sec
PD            5.0000 sec
PW1             4.99 usec
IRNUC  1H
CTEMP          20.1 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            30
```

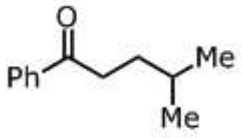
2n



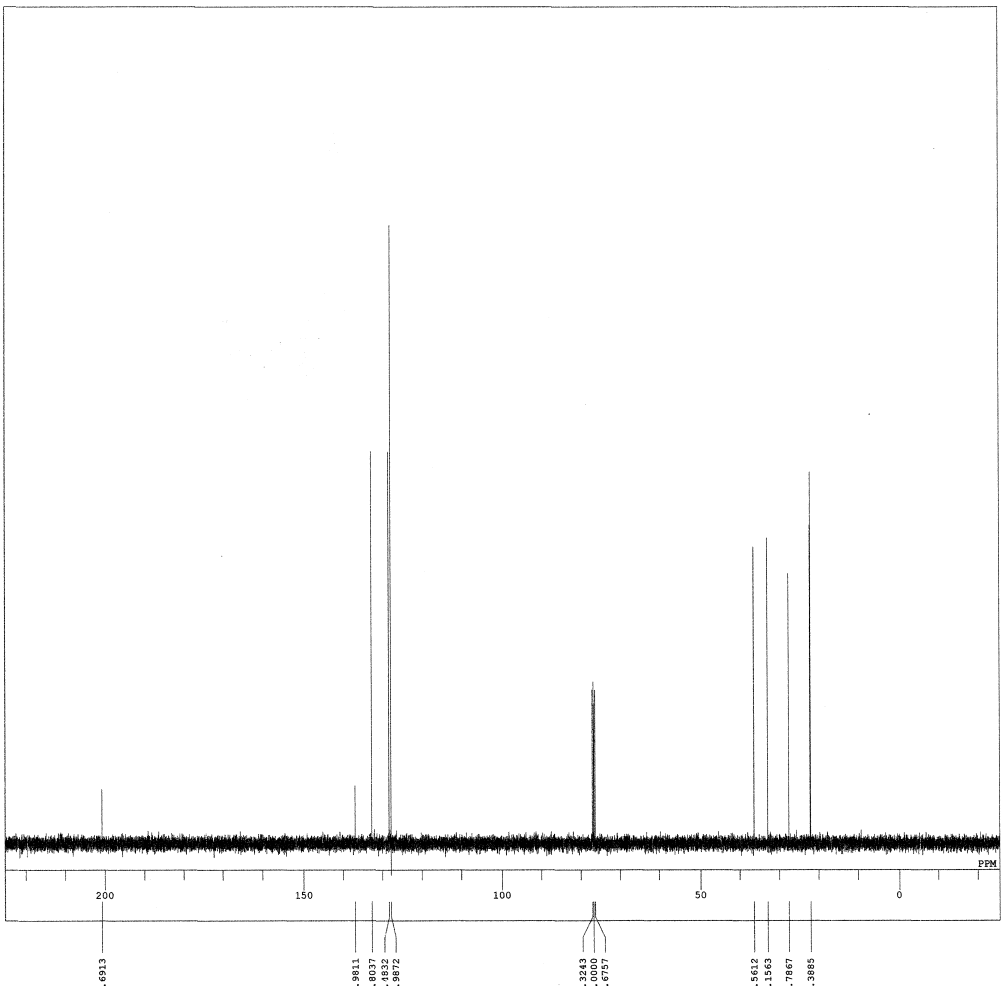

```
DFILE  ozawa09-019_13C.jdf
COMNT  4-OAc-Ph, CO2Me
DATIM  18-11-2015 18:02:14
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         32767
FREQU      30788.18 Hz
SCANS            32
ACQTM         1.0643 sec
PD            2.0000 sec
PW1             3.16 usec
IRNUC  1H
CTEMP          20.2 c
SLVNT  CDCL3
EXREF          77.00 ppm
BF             0.12 Hz
RGAIN            60
```



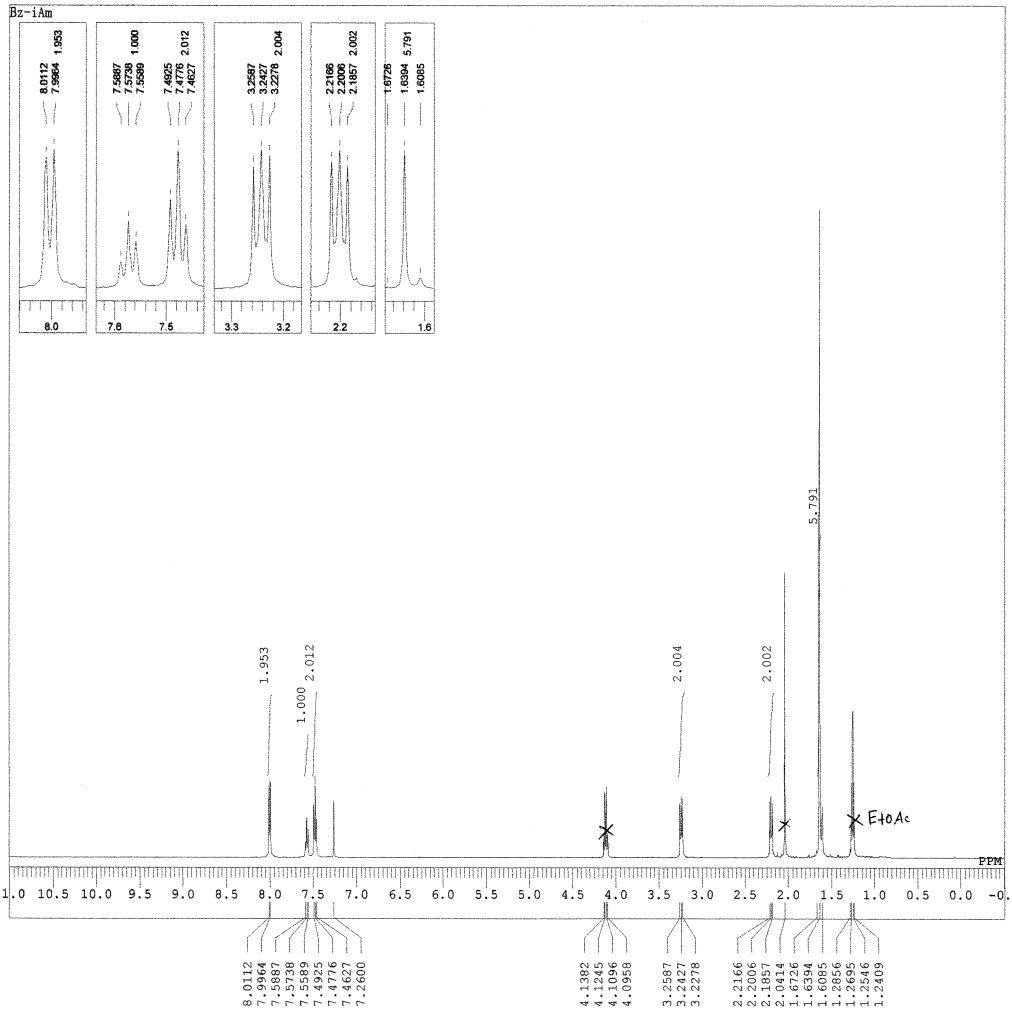
```
EFILE  ozawa09-059_1H.jdf
COMNT
DATIM  19-12-2015 17:03:04
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         16384
FREQU       7352.94 Hz
SCANS             4
ACQTM        2.2282 sec
PD           5.0000 sec
PW1             4.99 usec
IRNUC  1H
CTEMP           19.8 c
SLVNT  CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             22
```



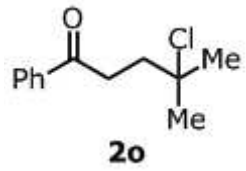
1o



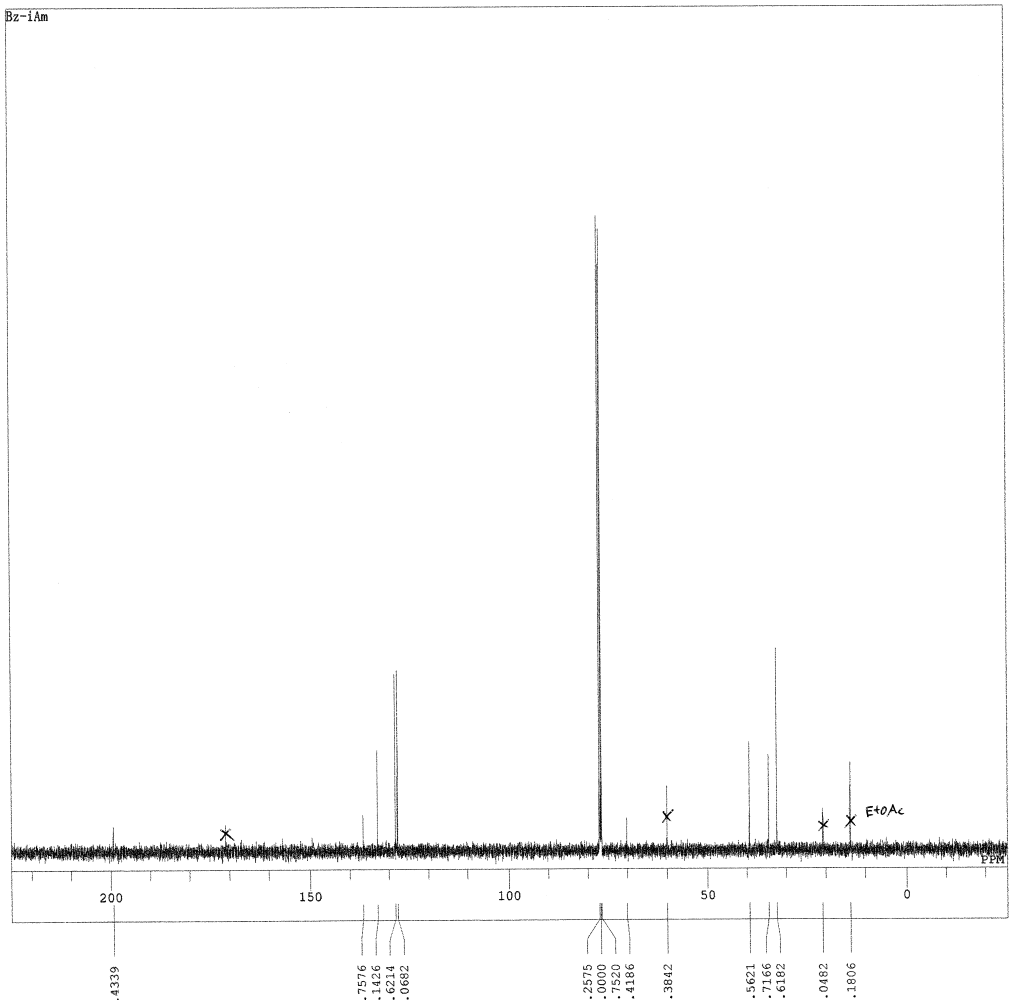
```
DFILE  ozawa09-059_13C.jdf
COMNT
DATIM  19-12-2015 17:05:22
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ          98.52 MHz
OBSET           4.64 KHz
OBFIN           8.74 Hz
POINT          32767
FREQU       30788.18 Hz
SCANS             20
ACQTM         1.0643 sec
PD            2.0000 sec
PW1              3.16 usec
IRNUC  1H
CTEMP           19.9 c
SLVNT  CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```



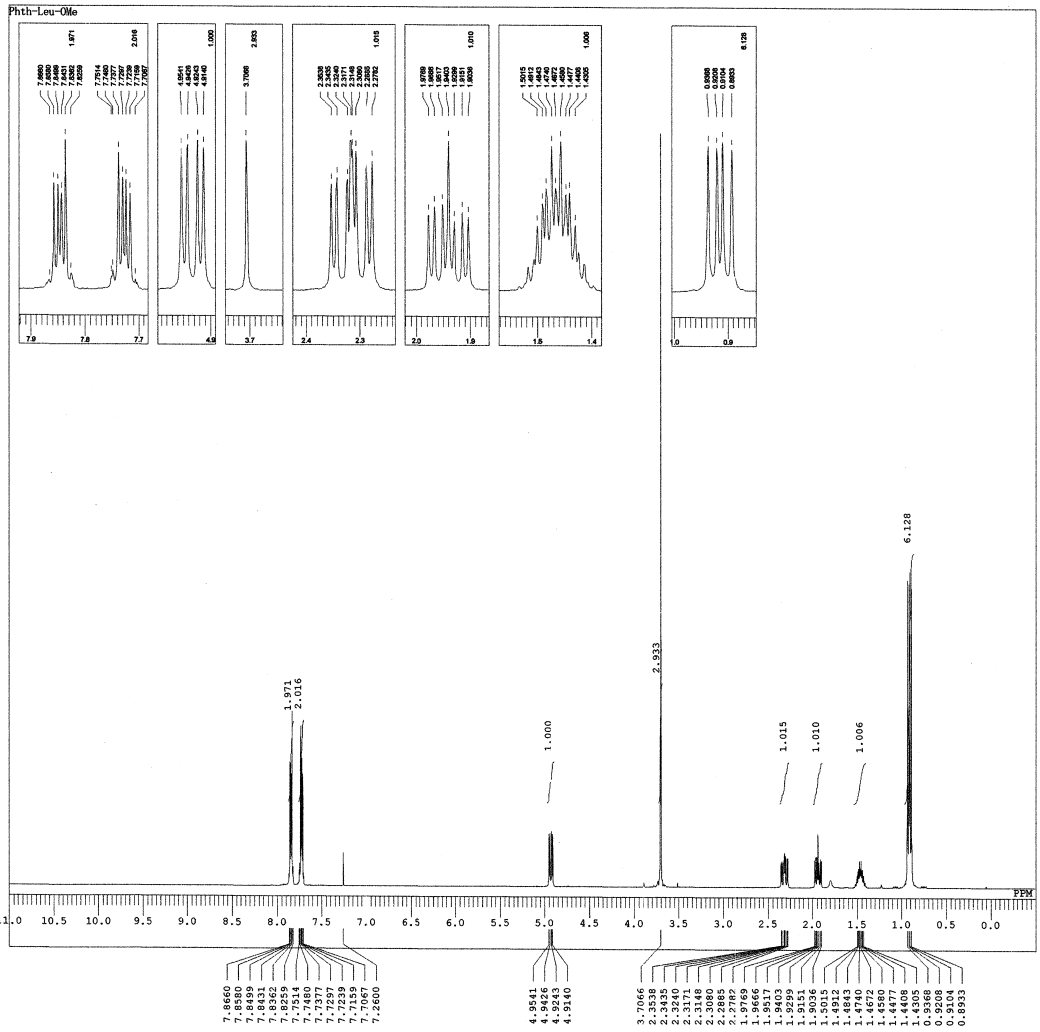

```
DFILE   ozawa09-066_1H.jdf
COMNT   Bz-iAm
DATIM   2015-12-24 17:13:48
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           32.9 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              1.20 Hz
RGAIN             30
```



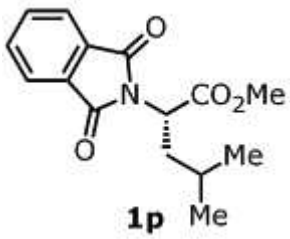
2o



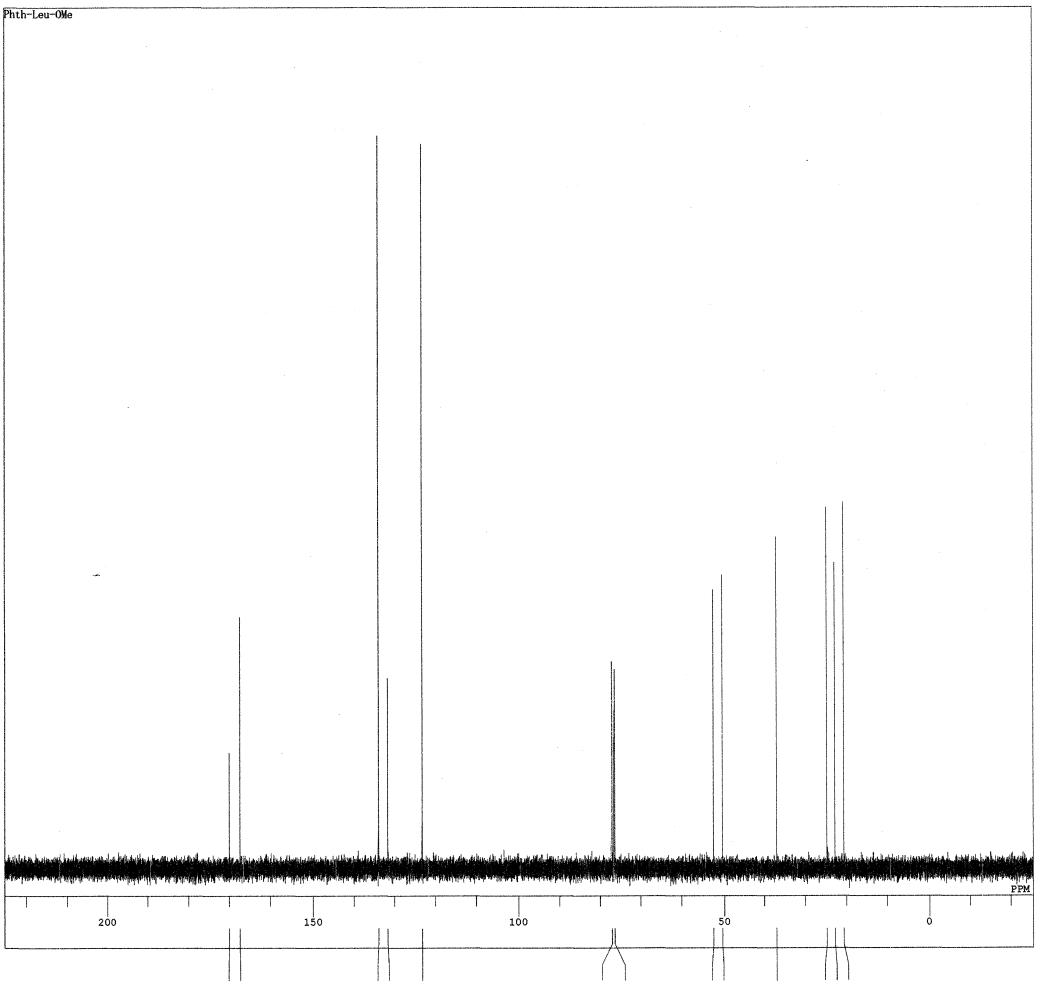

```
DFILE   ozawa09-066_13C.jdf
COMNT   Bz-iAm
DATIM   2015-12-24 17:15:27
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32767
FREQU        39308.18 Hz
SCANS            112
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC   1H
CTEMP           30.3 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              1.20 Hz
RGAIN             60
```



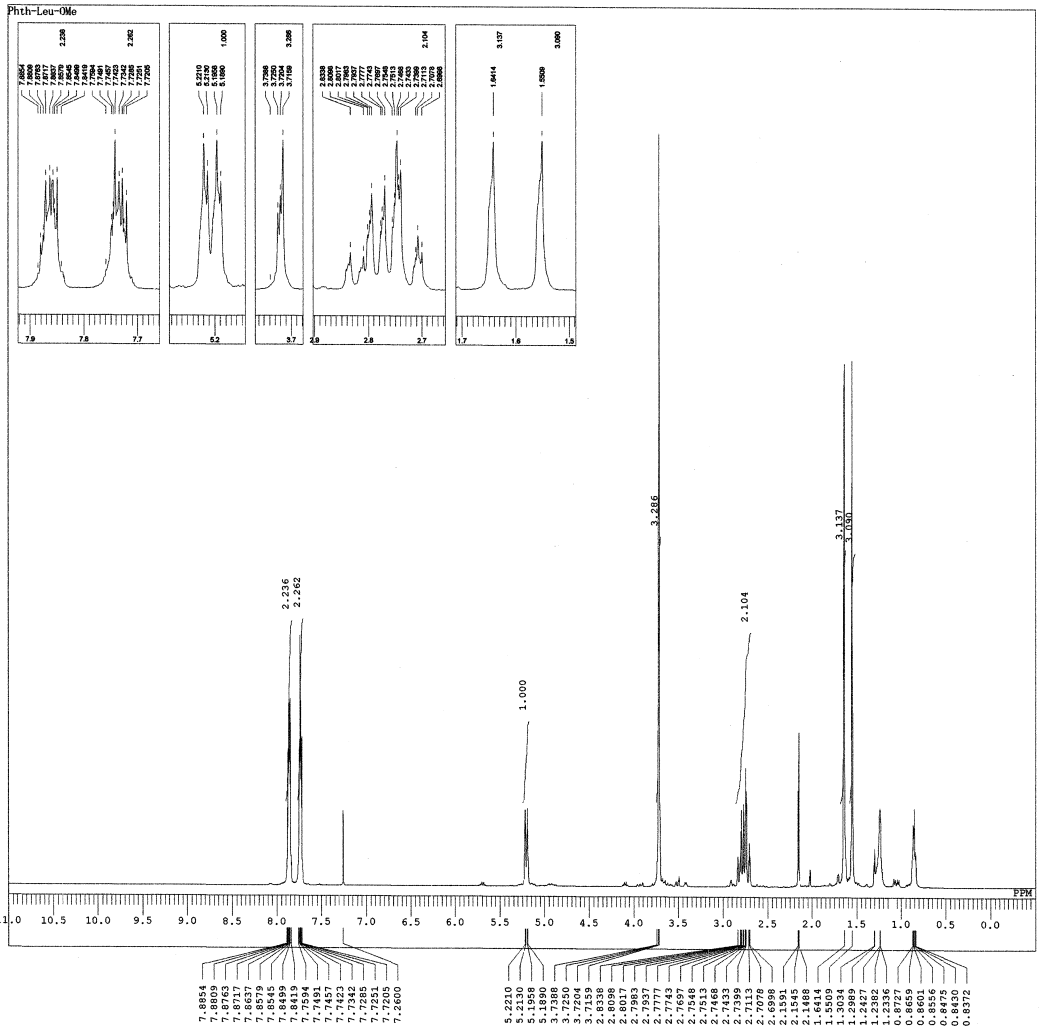

```
DFILE  ozawa09-157_1H.als
COMNT  Phth-Leu-OMe
DATIM  09-09-2016 16:46:10
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         13107
FREQU       5882.35 Hz
SCANS             4
ACQTM          2.2282 sec
PD             5.0000 sec
PW1              6.45 usec
IRNUC  1H
CTEMP          460.0 c
SLVNT  CDCL3
EXREF            7.26 ppm
BF               0.12 Hz
RGAIN              24
```



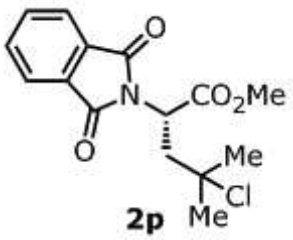
1p



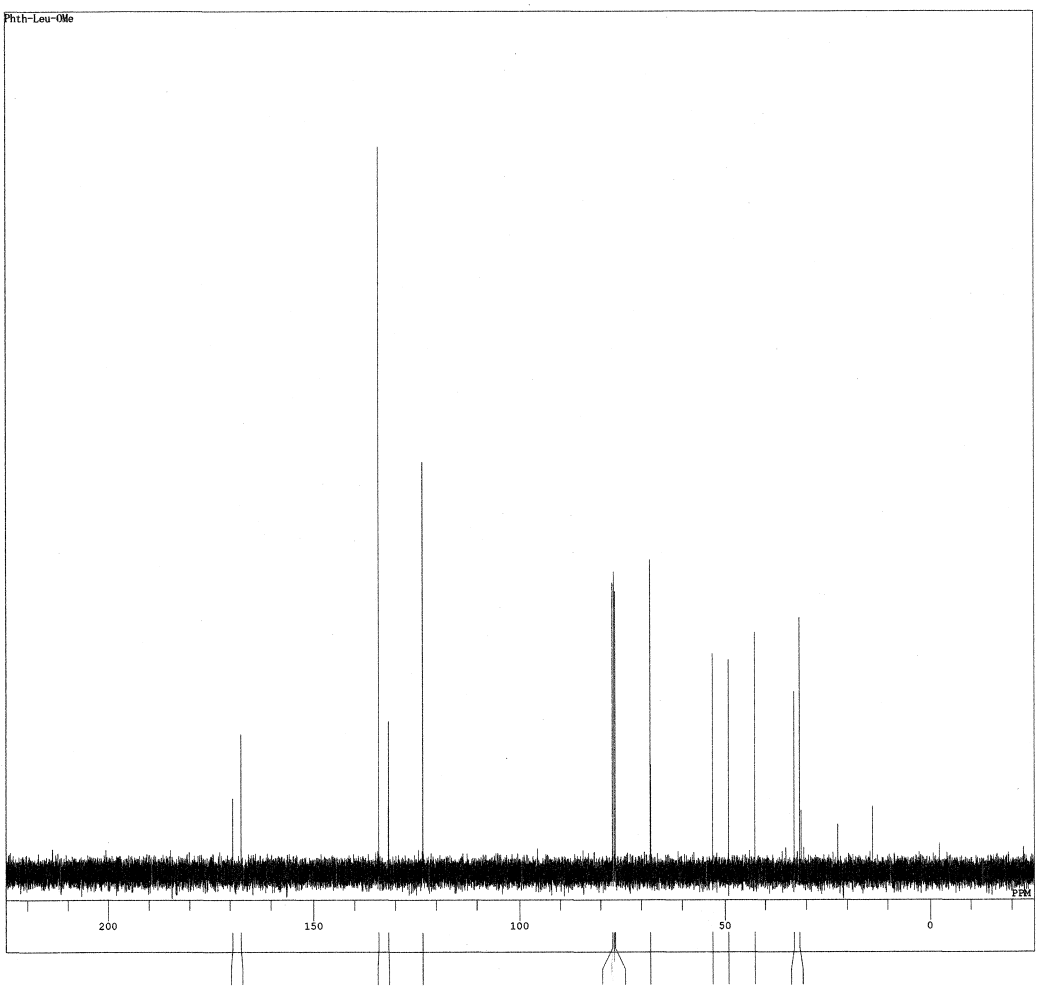

```
DFILE  ozawa09-157_13C.jdf
COMNT  Phth-Leu-OMe
DATIM  09-09-2016 16:47:38
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         65535
FREQU      30788.18 Hz
SCANS            24
ACQTM          2.1286 sec
PD             2.0000 sec
PW1              3.73 usec
IRNUC  1H
CTEMP          460.0 c
SLVNT  CDCL3
EXREF           77.00 ppm
BF               0.12 Hz
RGAIN              60
```



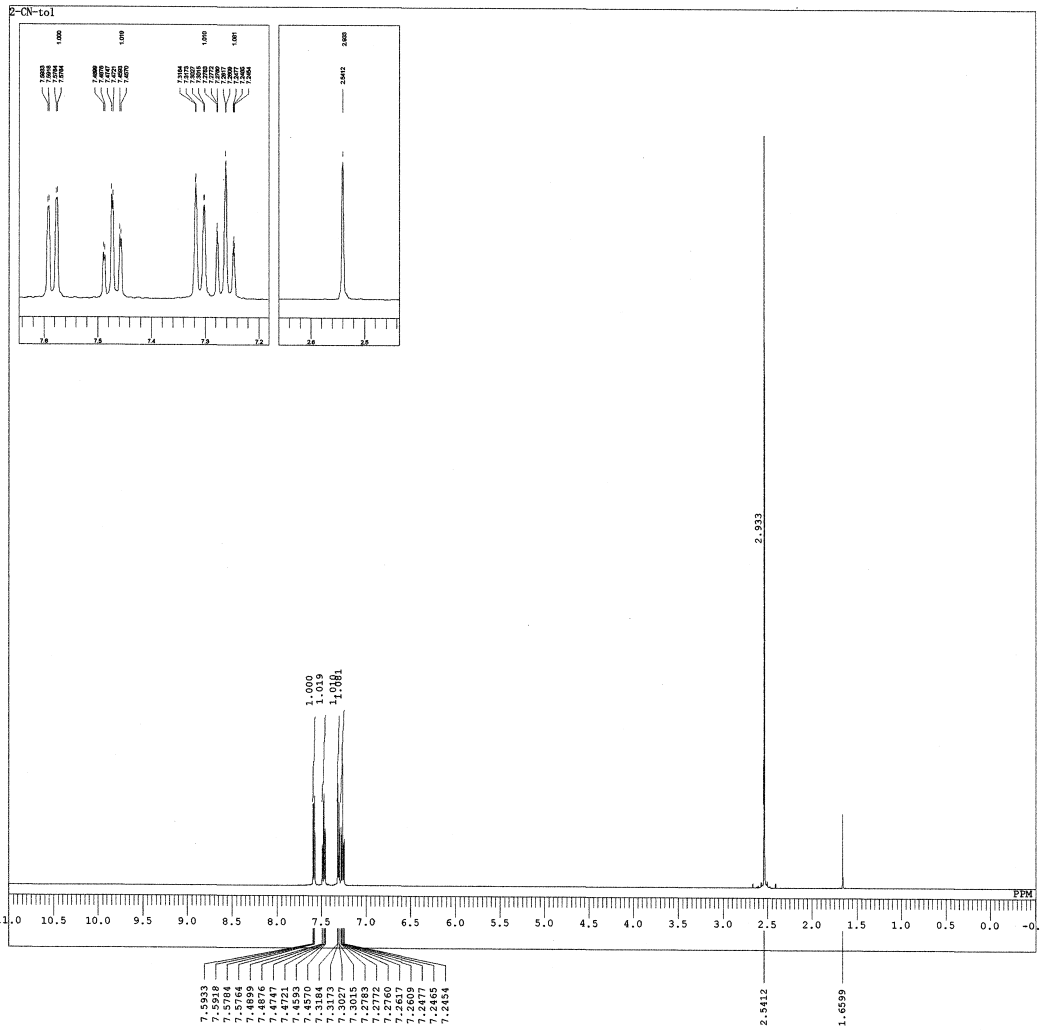

```
DFILE  ozawa09-158_1H.jdf
COMNT  Phth-Leu-OMe
DATIM  09-09-2016 16:52:35
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         16384
FREQU       7352.94 Hz
SCANS             4
ACQTM          2.2282 sec
PD             5.0000 sec
PW1              6.45 usec
IRNUC  1H
CTEMP          460.0 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            26
```



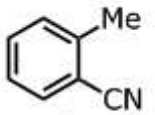
2p



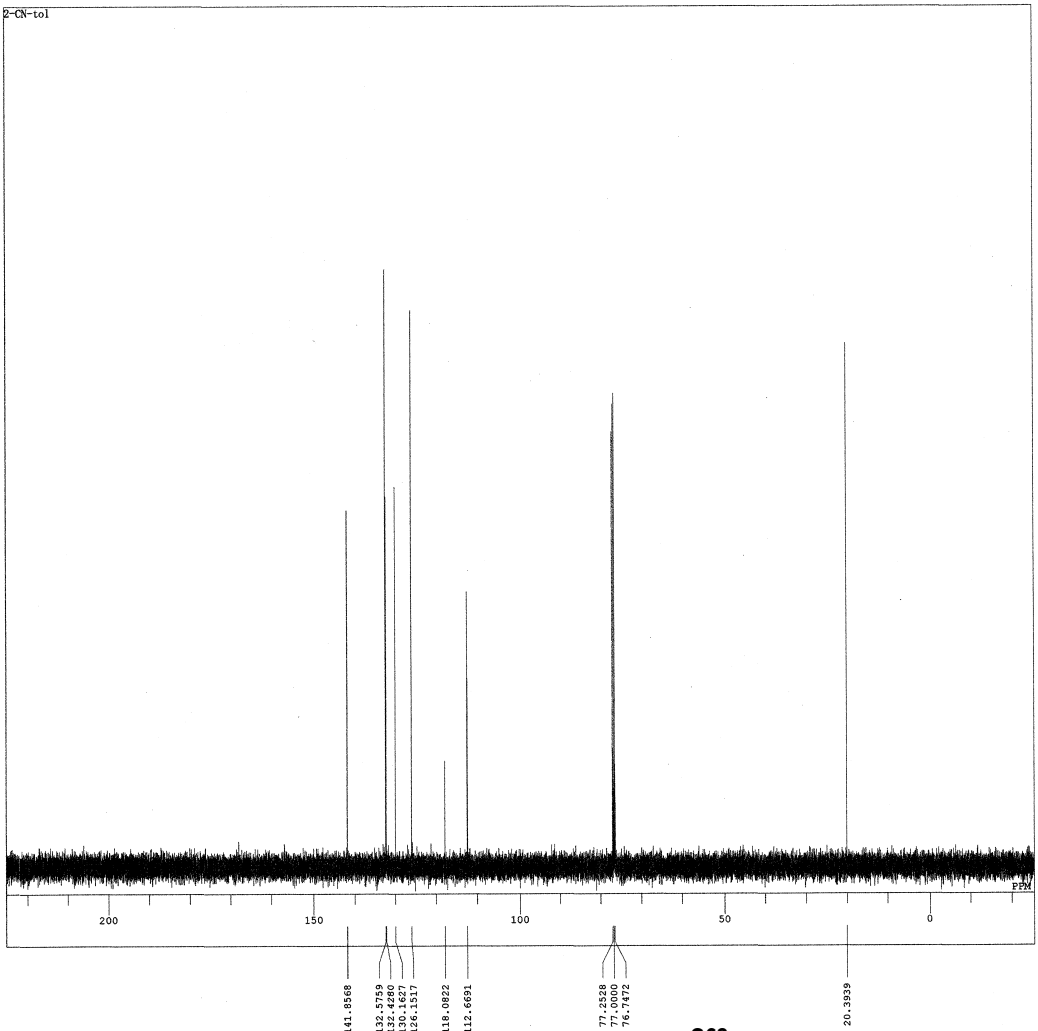

```
DFILE  ozawa09-158_13C.jdf
COMNT  Phth-Leu-OMe
DATIM  09-09-2016 16:54:12
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         65535
FREQU      30788.18 Hz
SCANS            52
ACQTM          2.1286 sec
PD             2.0000 sec
PW1              3.73 usec
IRNUC  1H
CTEMP          460.0 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.12 Hz
RGAIN            60
```



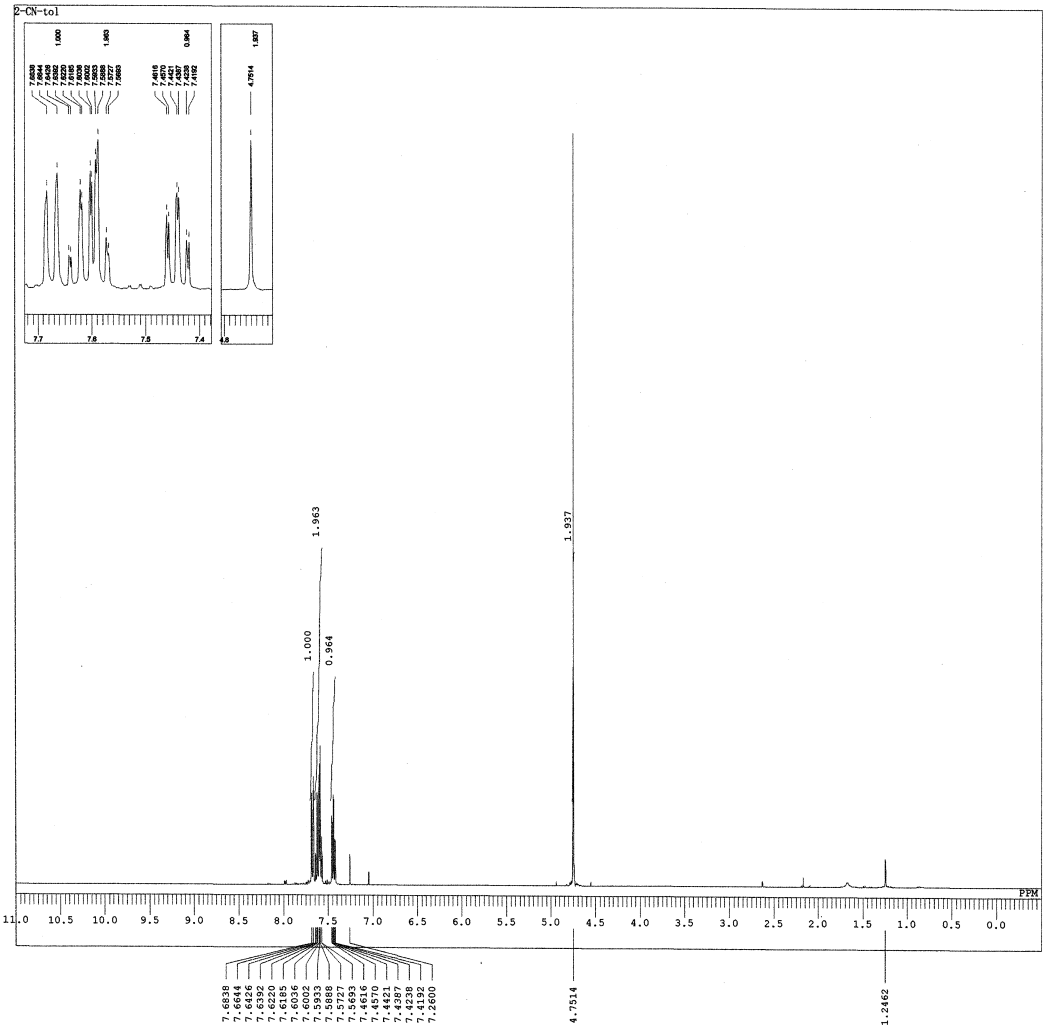
DFILE ozawa09-166_SM_1H.als
COMNT 2-CN-tol
DATIM 2016-09-27 14:47:35
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 52428
FREQU 7507.51 Hz
SCANS 4
ACQTM 6.9835 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 28



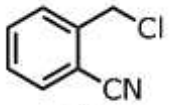
1q



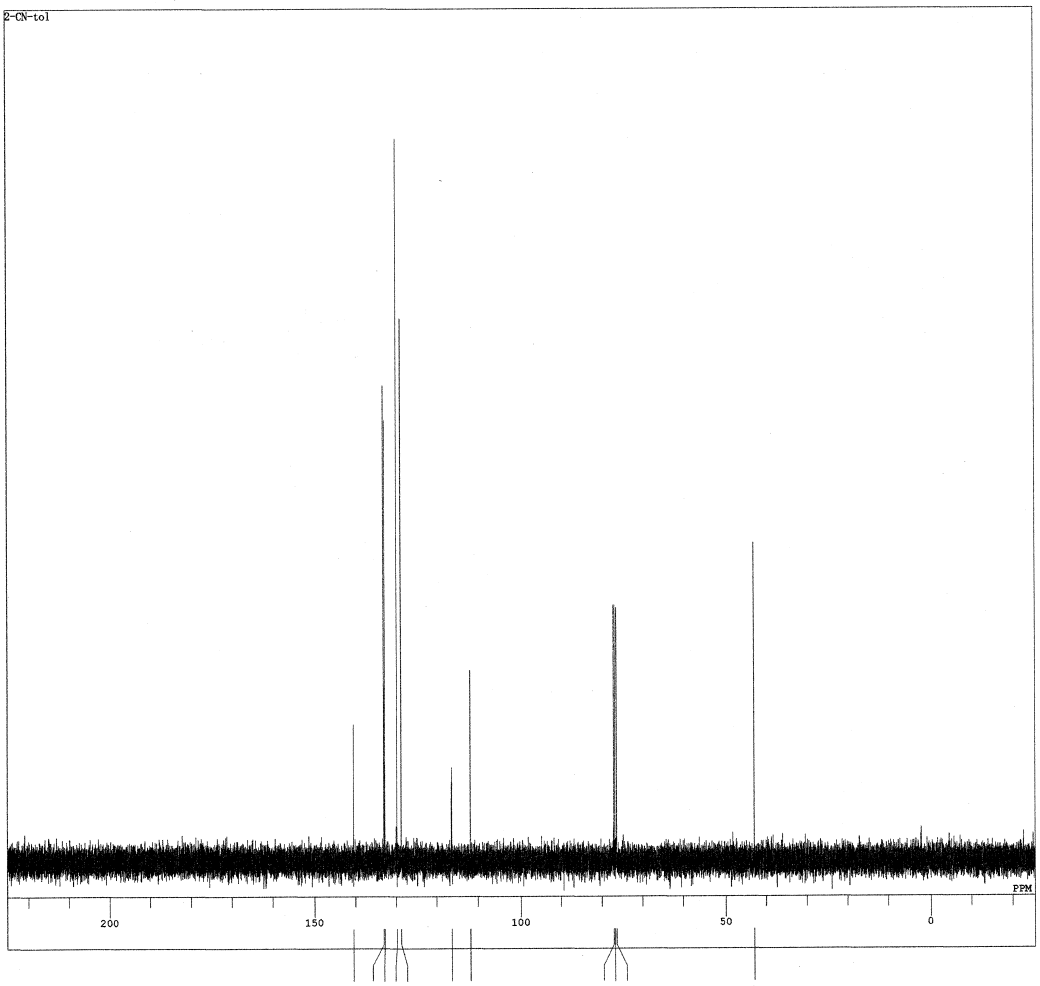
DFILE ozawa09-166_SM_13C.als
COMNT 2-CN-tol
DATIM 2016-09-27 14:49:03
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.54 Hz
SCANS 24
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



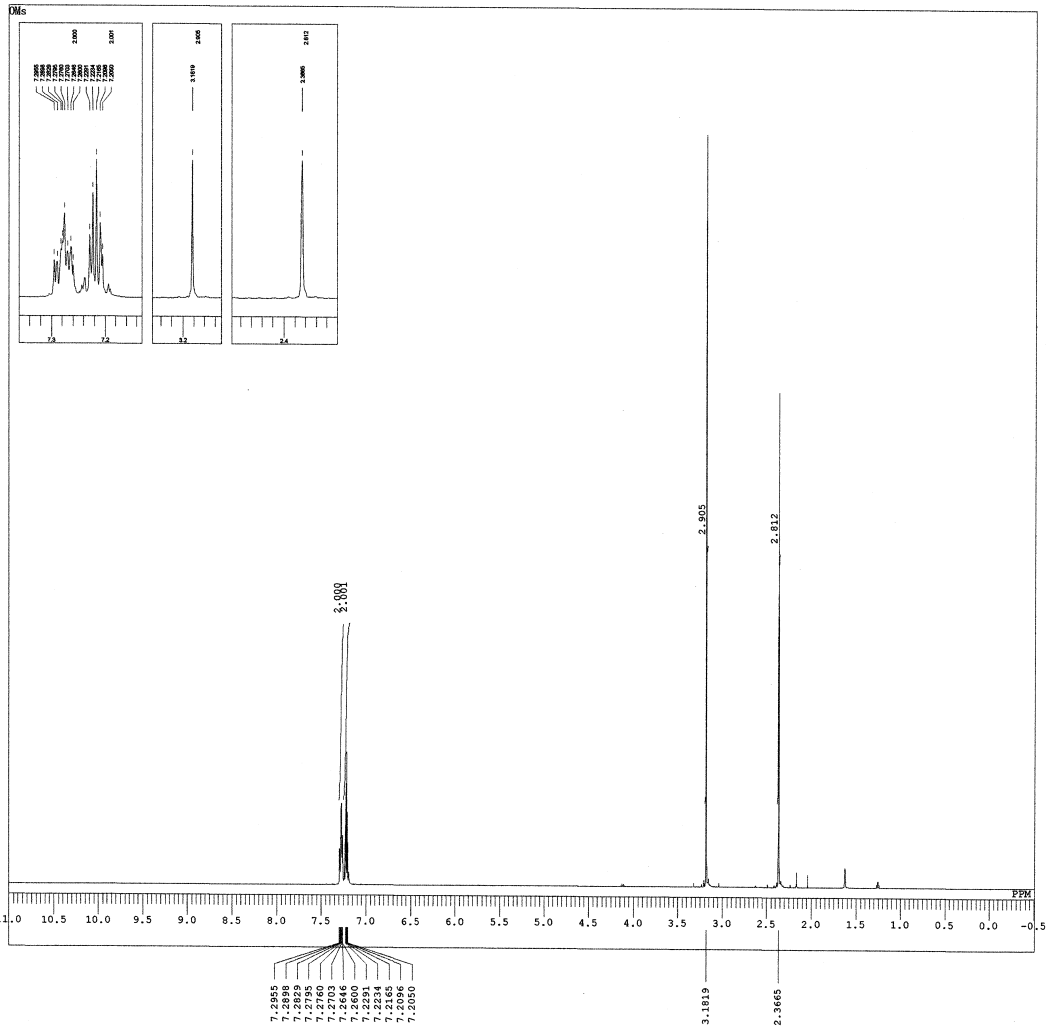
DFILE ozawa09-166_1H.jdf
COMNT 2-CN-tol
DATIM 14-09-2016 14:19:53
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 16384
FREQU 7352.94 Hz
SCANS 4
ACQTM 2.2282 sec
PD 5.0000 sec
PW1 6.45 usec
IRNUC 1H
CTEMP 460.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 28



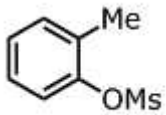
2q



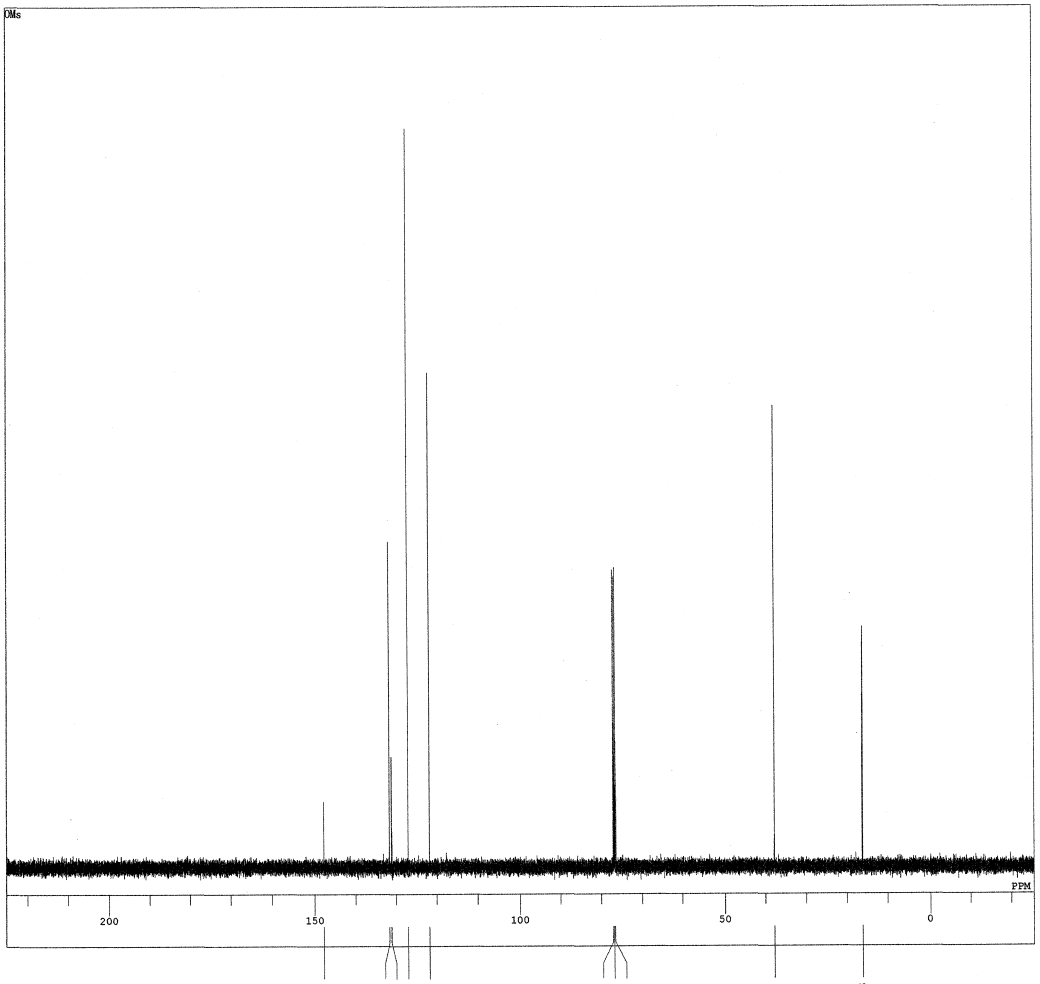
DFILE ozawa09-166_13C.jdf
COMNT 2-CN-tol
DATIM 14-09-2016 14:21:22
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 65535
FREQU 30788.18 Hz
SCANS 16
ACQTM 2.1286 sec
PD 2.0000 sec
PW1 3.73 usec
IRNUC 1H
CTEMP 460.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



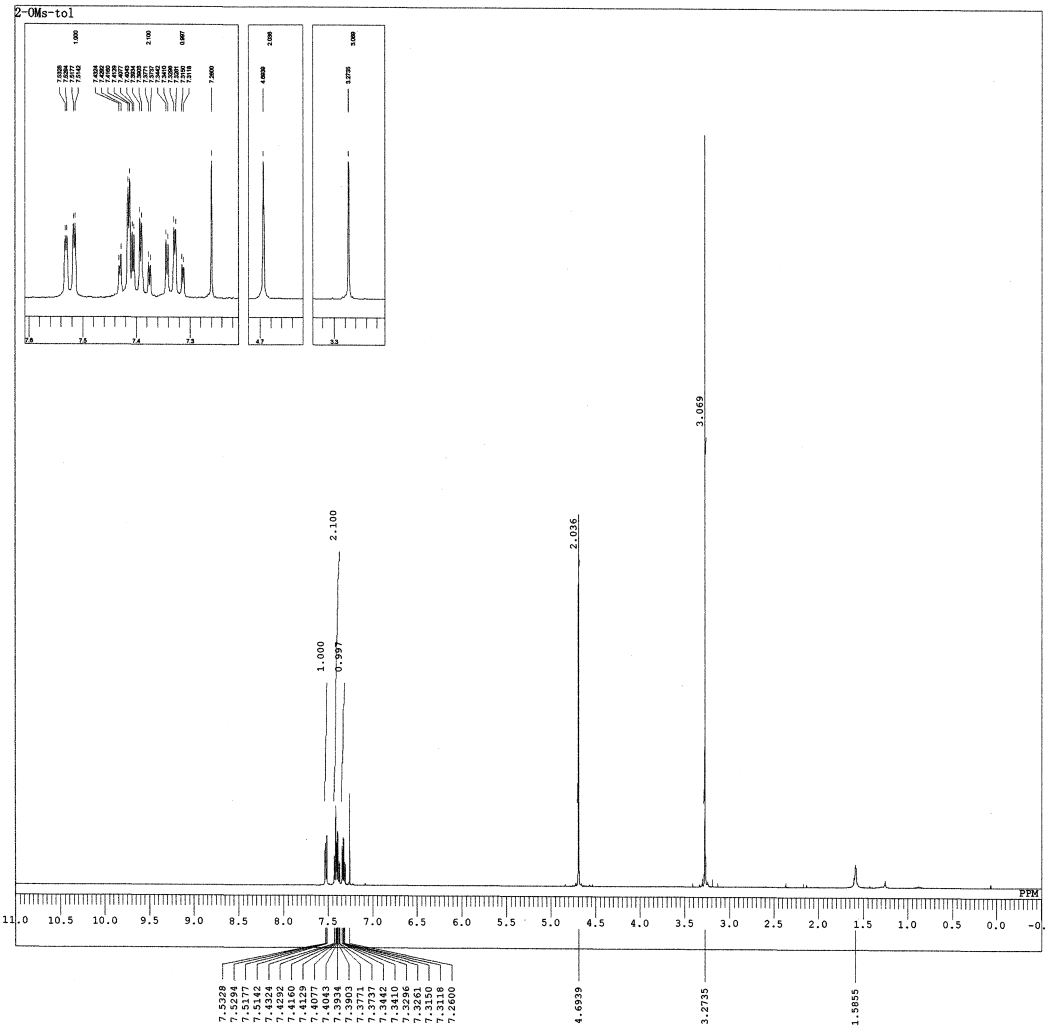
DFILE ozawa09-185_1H.als
COMNT OMs
DATIM 2016-09-27 10:30:60
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 28



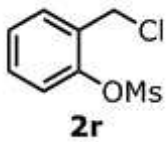
1r



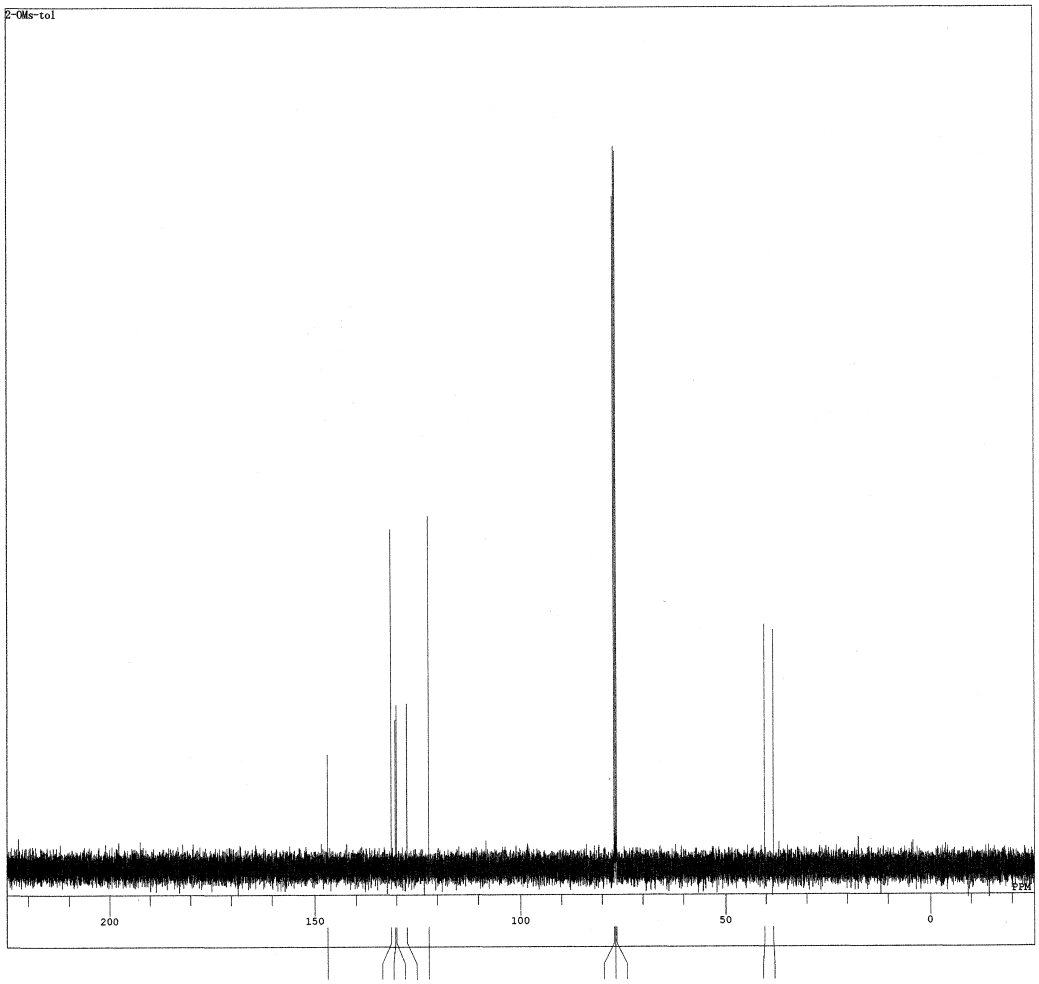
DFILE ozawa09-185_13C.als
COMNT OMs
DATIM 2016-09-27 10:31:59
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.54 Hz
SCANS 44
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



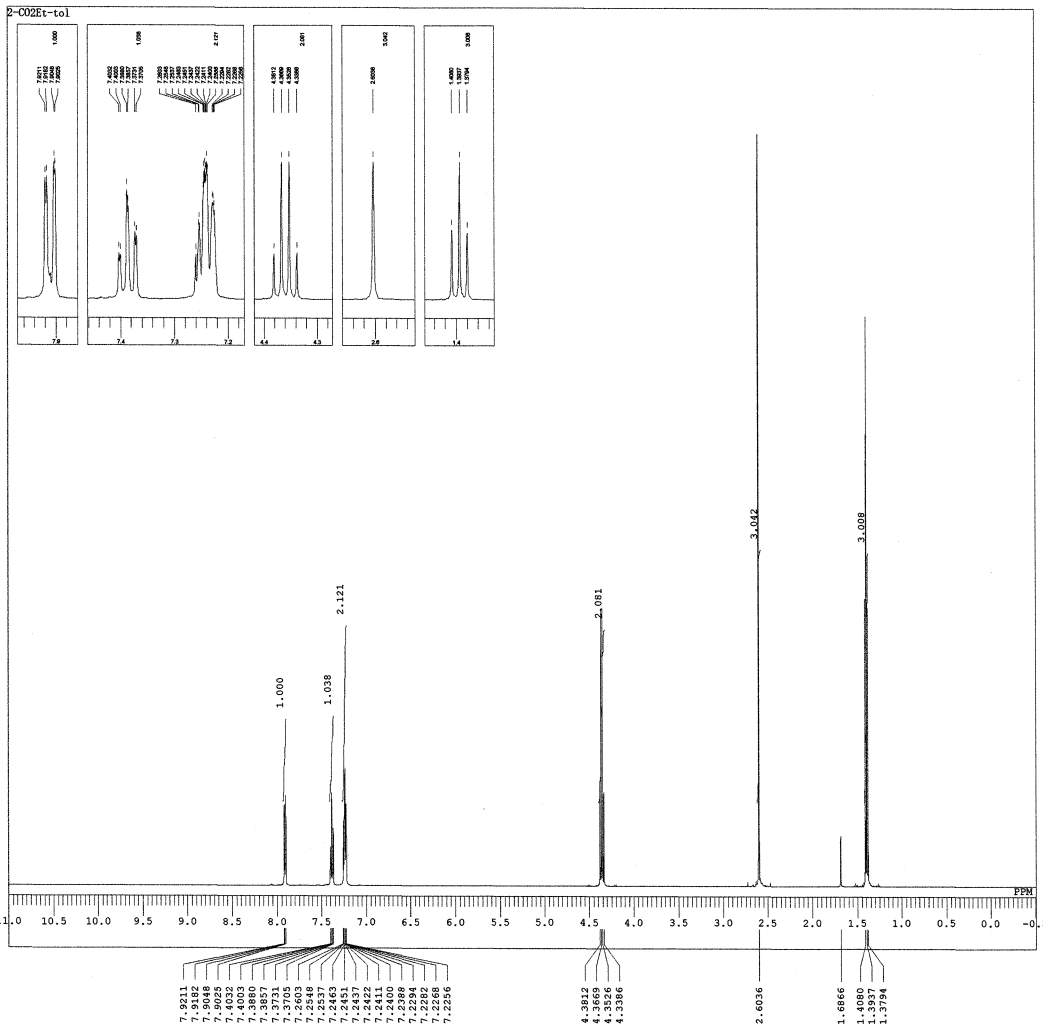

```
DFILE  ozawa09-192_1H.als
COMNT  2-OMs-tol
DATIM  2016-09-28 13:41:24
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         52428
FREQU       7507.51 Hz
SCANS             4
ACQTM          6.9835 sec
PD             5.0000 sec
PW1            5.55 usec
IRNUC  1H
CTEMP           21.5 c
SLVNT  CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
```



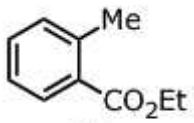
2r



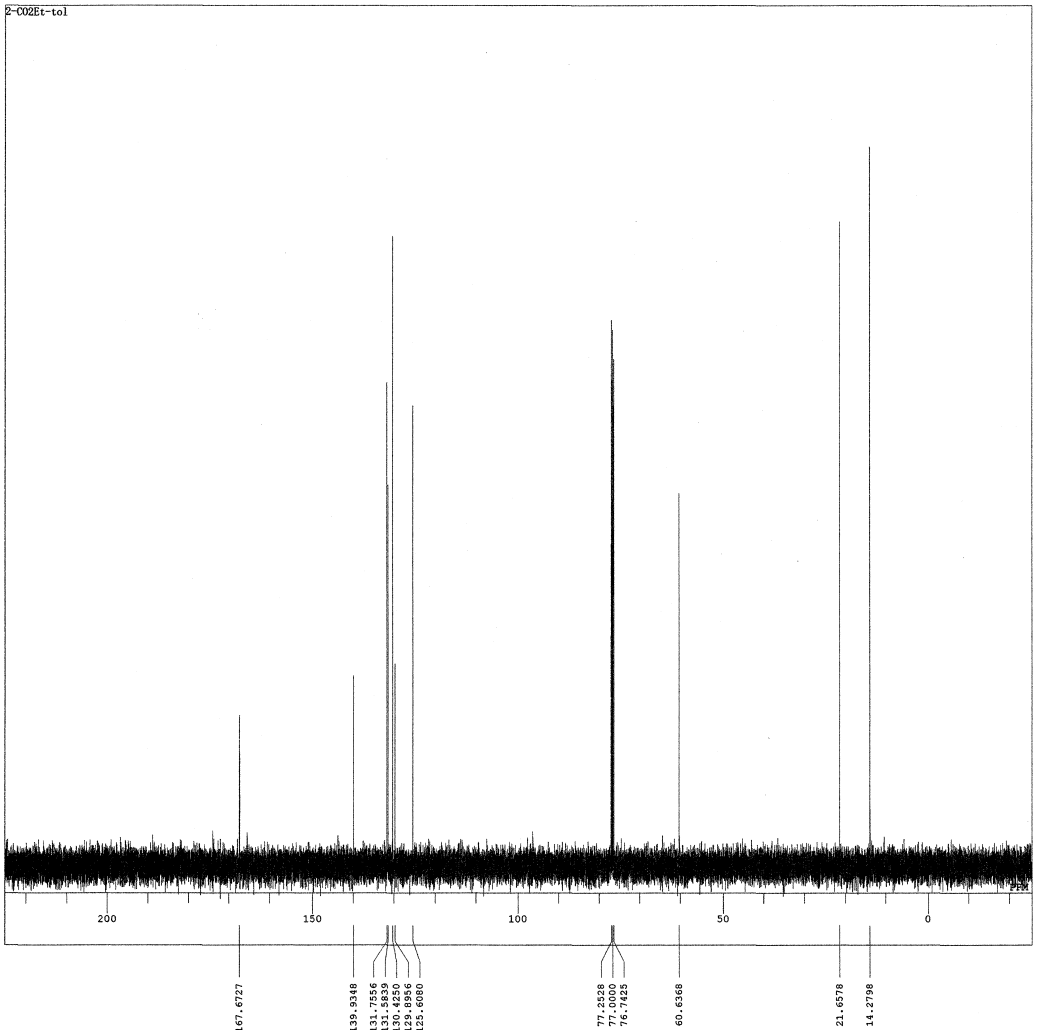

```
DFILE  ozawa09-192_13C.als
COMNT  2-OMs-tol
DATIM  2016-09-28 13:42:48
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         52428
FREQU       31446.54 Hz
SCANS            56
ACQTM          1.6672 sec
PD             2.0000 sec
PW1            3.40 usec
IRNUC  1H
CTEMP           21.6 c
SLVNT  CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```



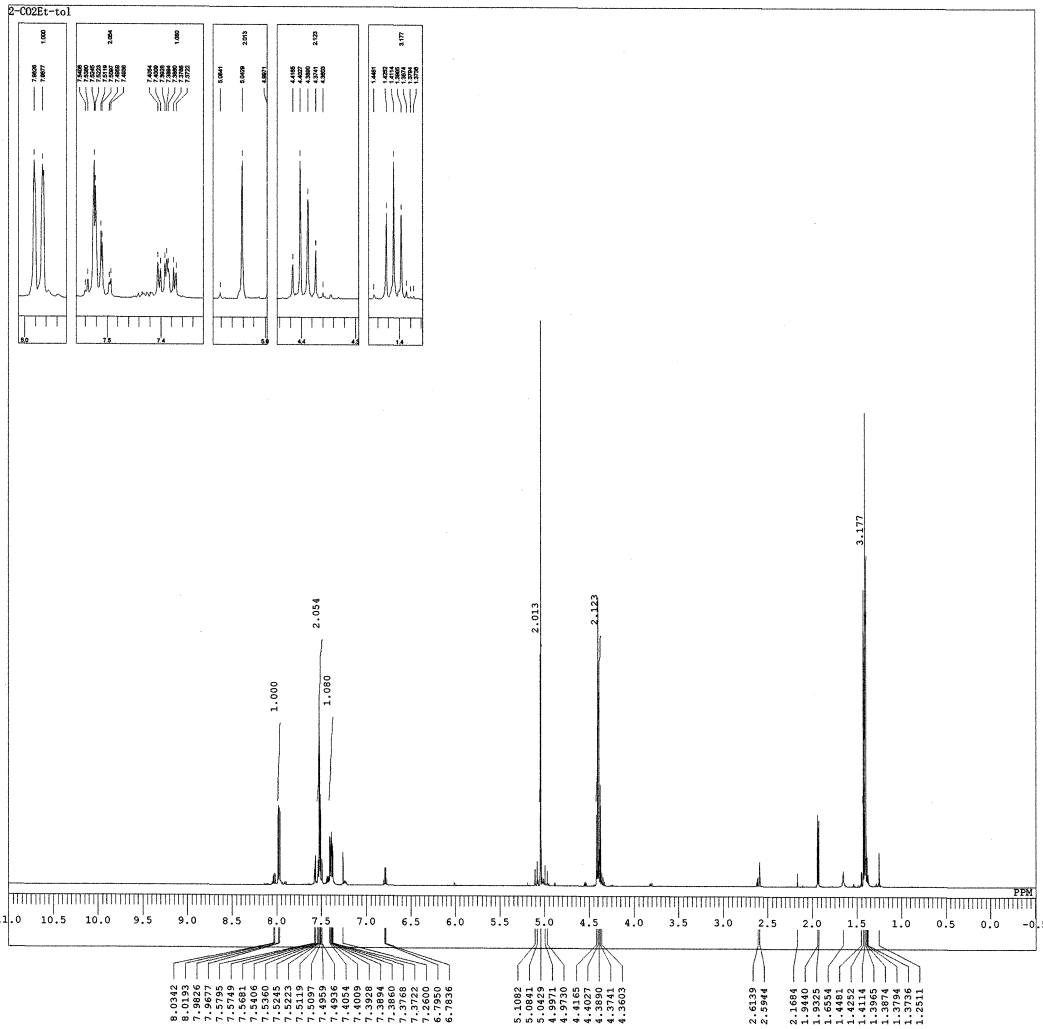
DFILE ozawa09-196_SM_1H.als
COMNT 2-CO2Et-tol
DATIM 2016-09-27 14:41:33
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 52428
FREQU 7507.51 Hz
SCANS 4
ACQTM 6.9835 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 26



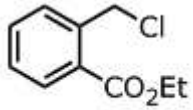
1s



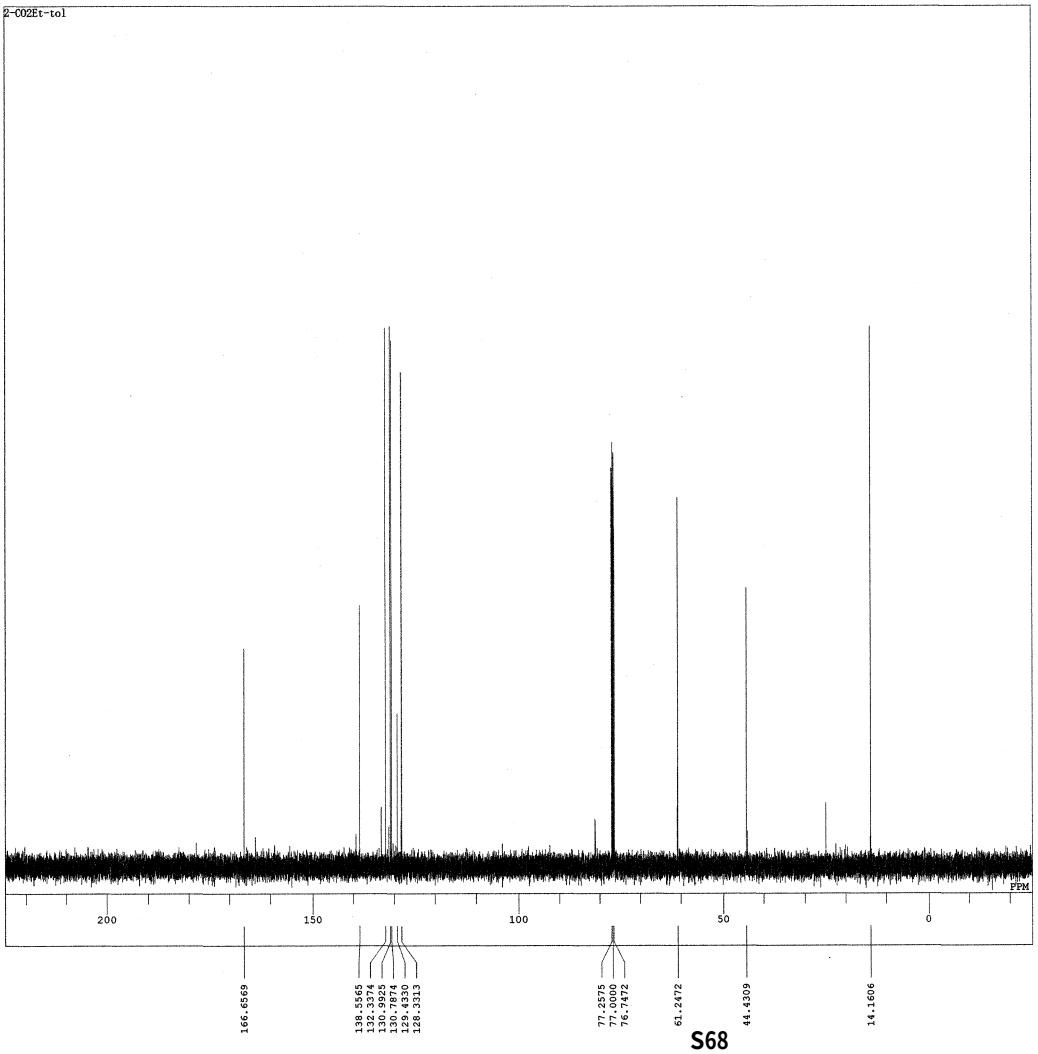
DFILE ozawa09-196_SM_13C.als
COMNT 2-CO2Et-tol
DATIM 2016-09-27 14:43:00
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.54 Hz
SCANS 16
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



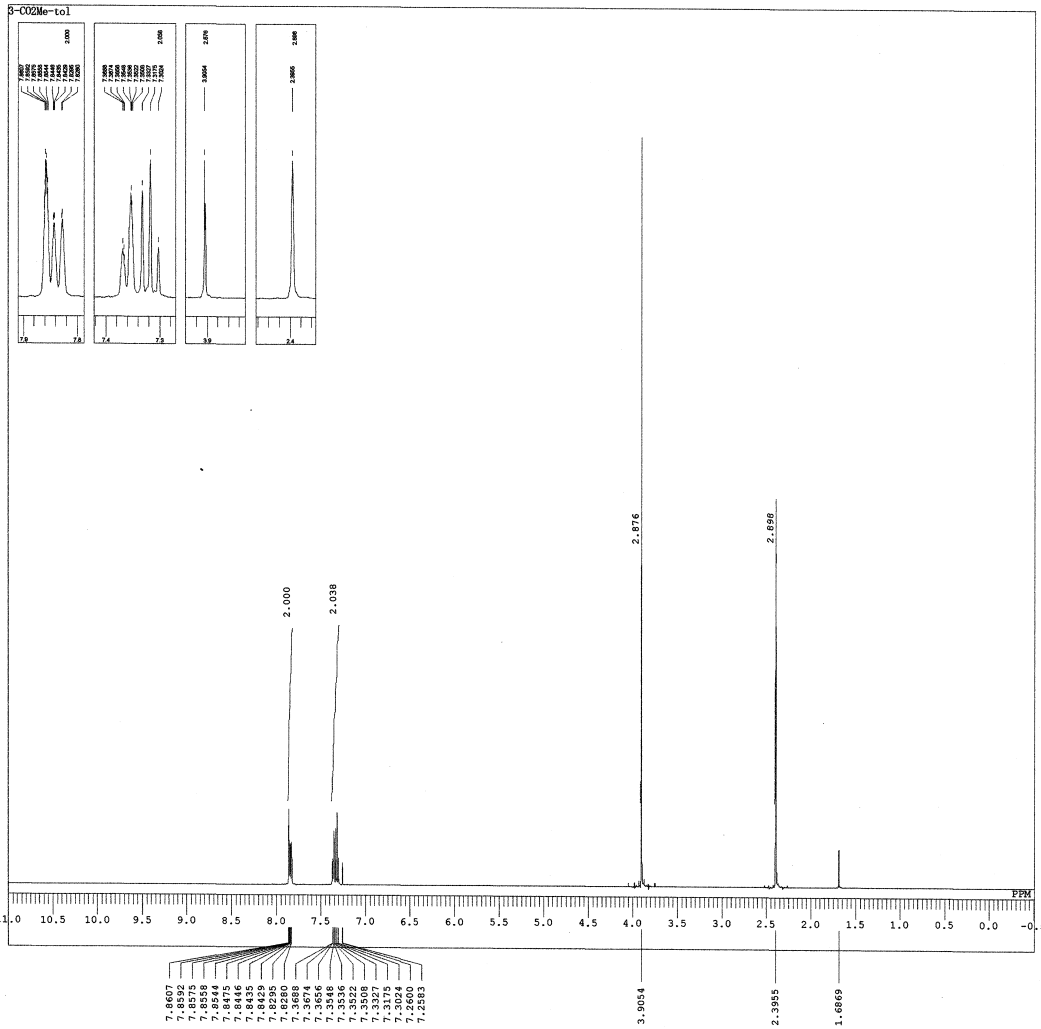
DFILE ozawa09-196_1H.als
COMNT 2-CO2Et-tol
DATIM 2016-09-27 10:25:17
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 26



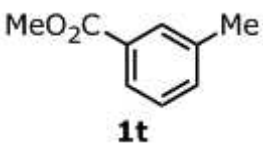
2s



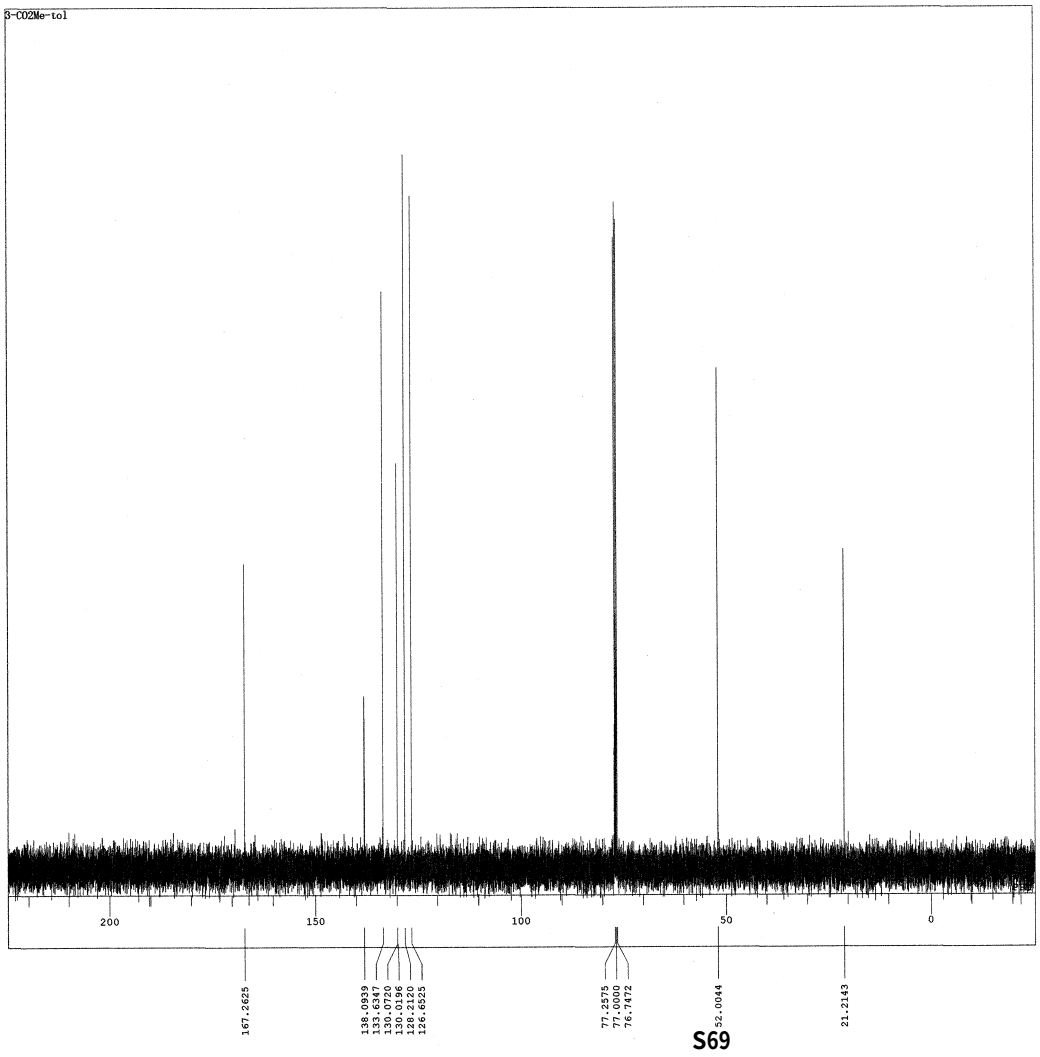
DFILE ozawa09-196_13C.als
COMNT 2-CO2Et-tol
DATIM 2016-09-27 10:26:18
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.54 Hz
SCANS 24
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



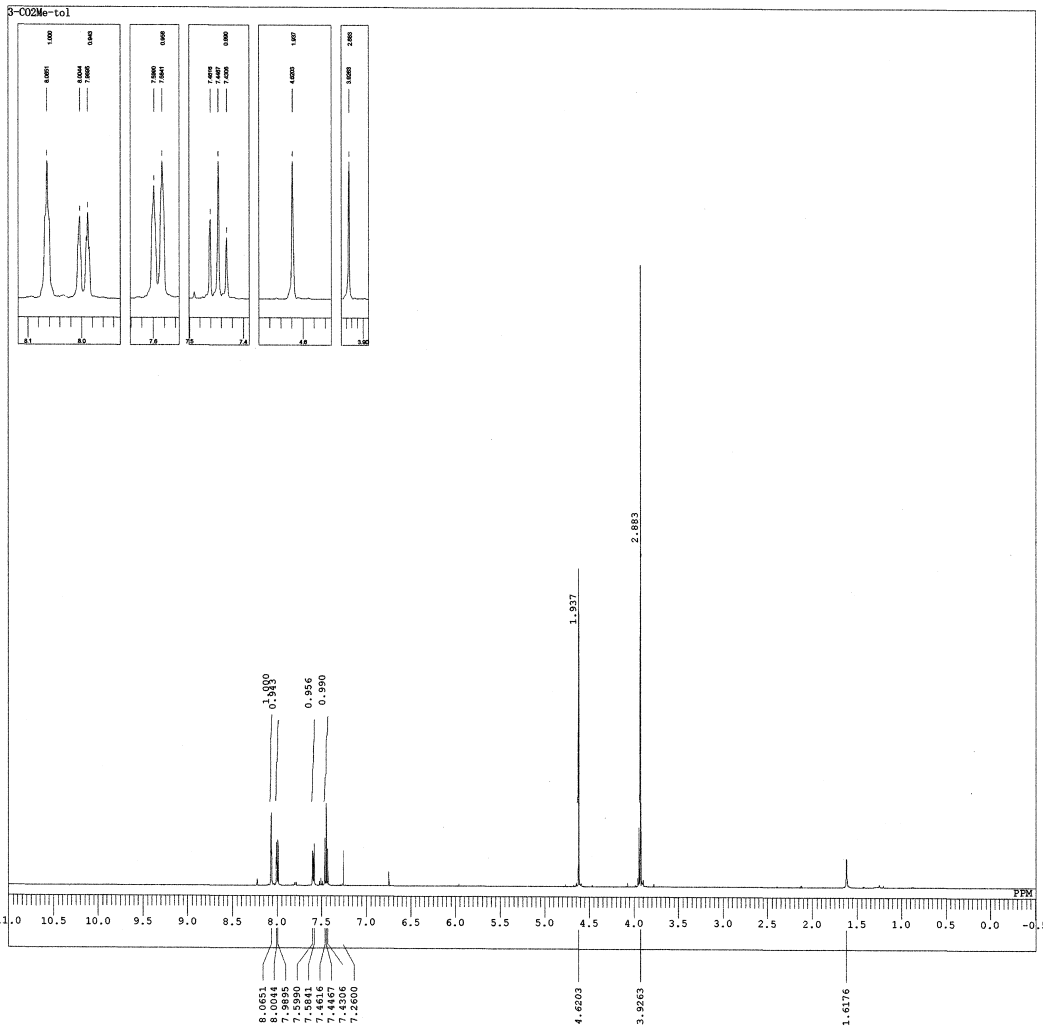

```
DFILE  ozawa09-195_SM_1H.als
COMNT  3-CO2Me-tol
DATIM  2016-09-27 14:35:41
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         52428
FREQU       7507.51 Hz
SCANS             4
ACQTM          6.9835 sec
PD             5.0000 sec
PW1            5.55 usec
IRNUC  1H
CTEMP         21.4 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            28
```



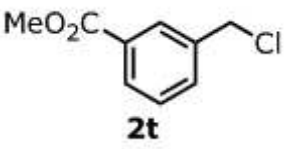
1t



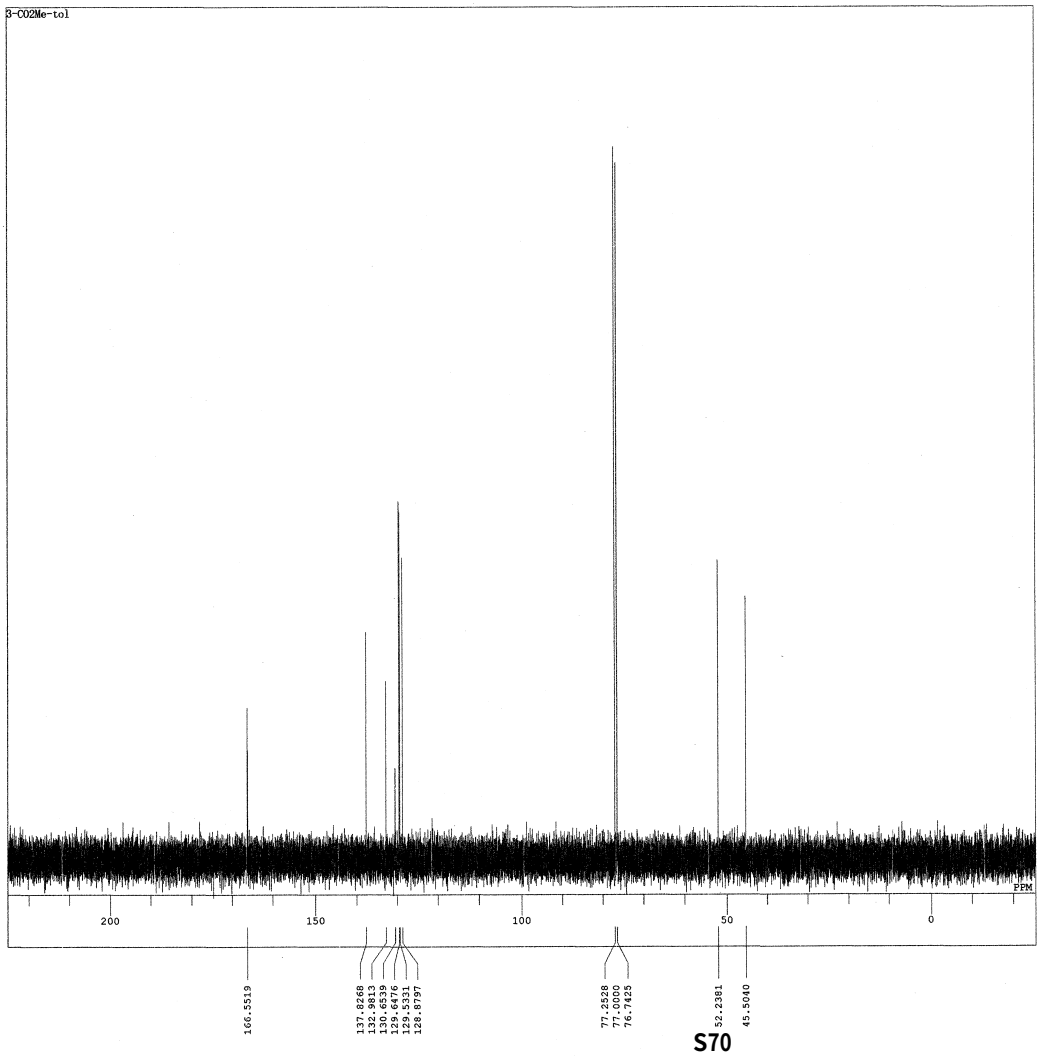

```
DFILE  ozawa09-195_SM_13C.als
COMNT  3-CO2Me-tol
DATIM  2016-09-27 14:37:08
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         52428
FREQU       31446.54 Hz
SCANS            16
ACQTM          1.6672 sec
PD             2.0000 sec
PW1            3.40 usec
IRNUC  1H
CTEMP         21.5 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.12 Hz
RGAIN            60
```



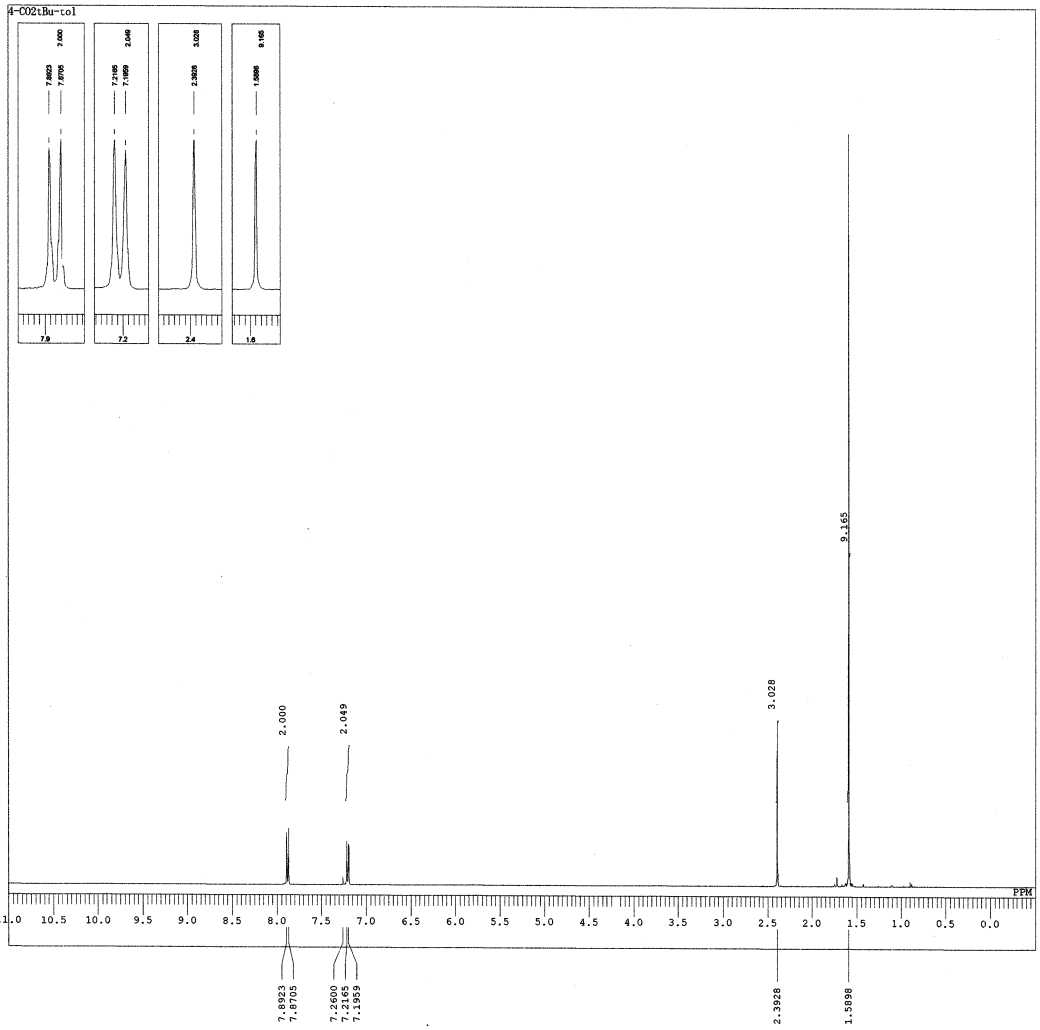

```
DFILE  ozawa10-003_1H.als
COMNT  3-CO2Me-tol
DATIM  2016-09-30 17:29:22
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.51 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC  1H
CTEMP           21.2 c
SLVNT  CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             30
```



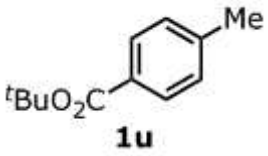
2t



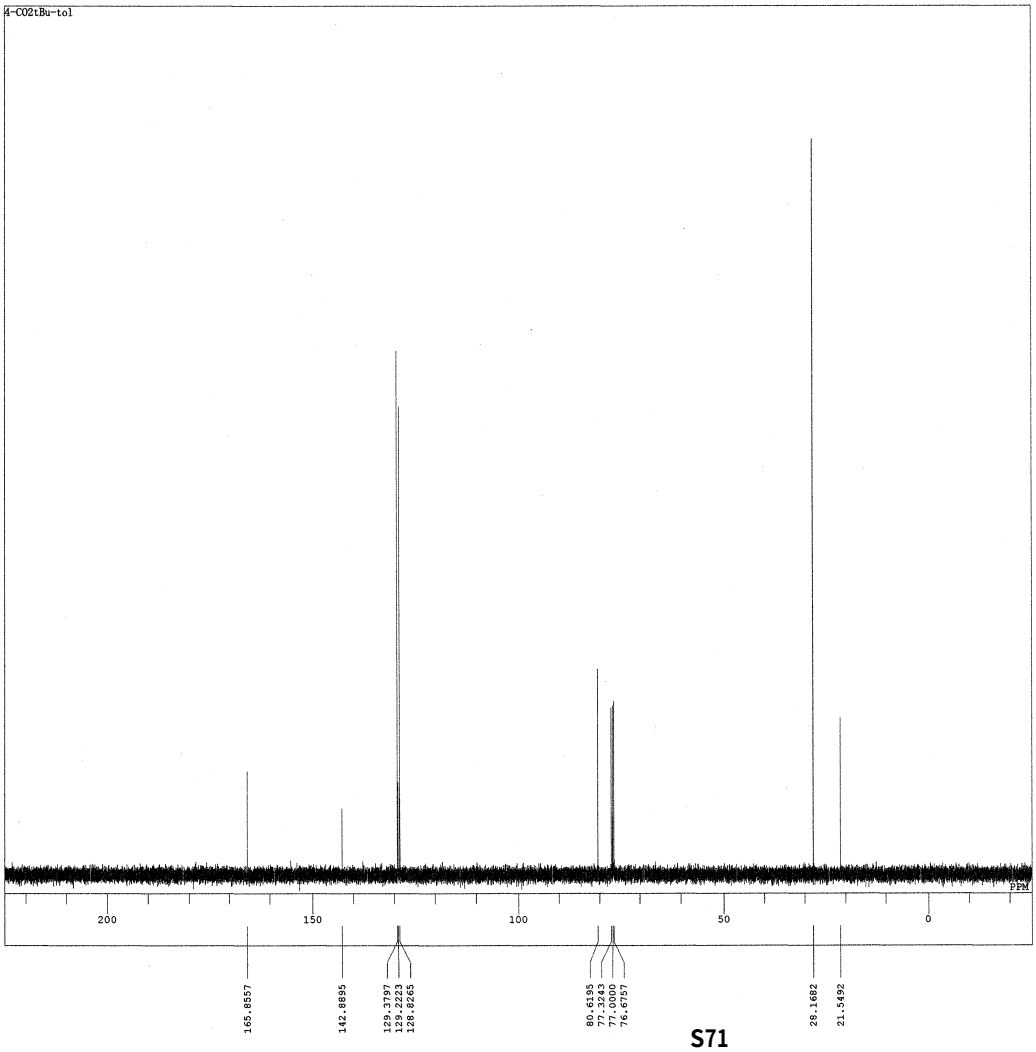

```
DFILE  ozawa10-003_13C.als
COMNT  3-CO2Me-tol
DATIM  2016-09-30 17:30:17
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         52428
FREQU      31446.54 Hz
SCANS            20
ACQTM          1.6672 sec
PD             2.0000 sec
PW1              3.40 usec
IRNUC  1H
CTEMP           21.3 c
SLVNT  CDCL3
EXREF           77.00 ppm
BF              0.12 Hz
RGAIN             60
```



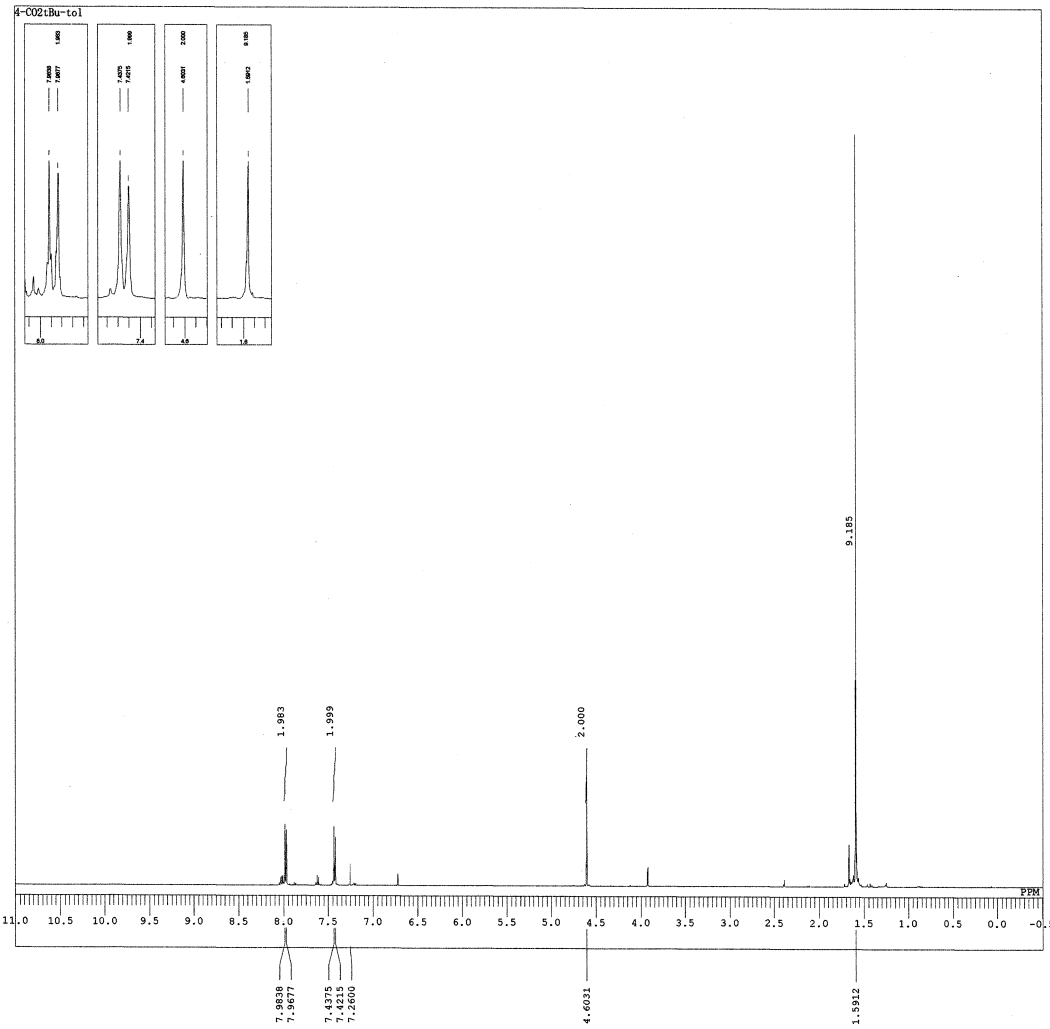

```
DFILE  ozawa10-002_1H.jdf
COMNT  4-CO2tBu-tol
DATIM  30-09-2016 10:21:24
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         16384
FREQU       7352.94 Hz
SCANS             4
ACQTM          2.2282 sec
PD             5.0000 sec
PW1            6.45 usec
IRNUC  1H
CTEMP        460.0 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            24
```



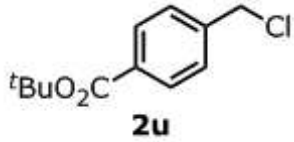
1u



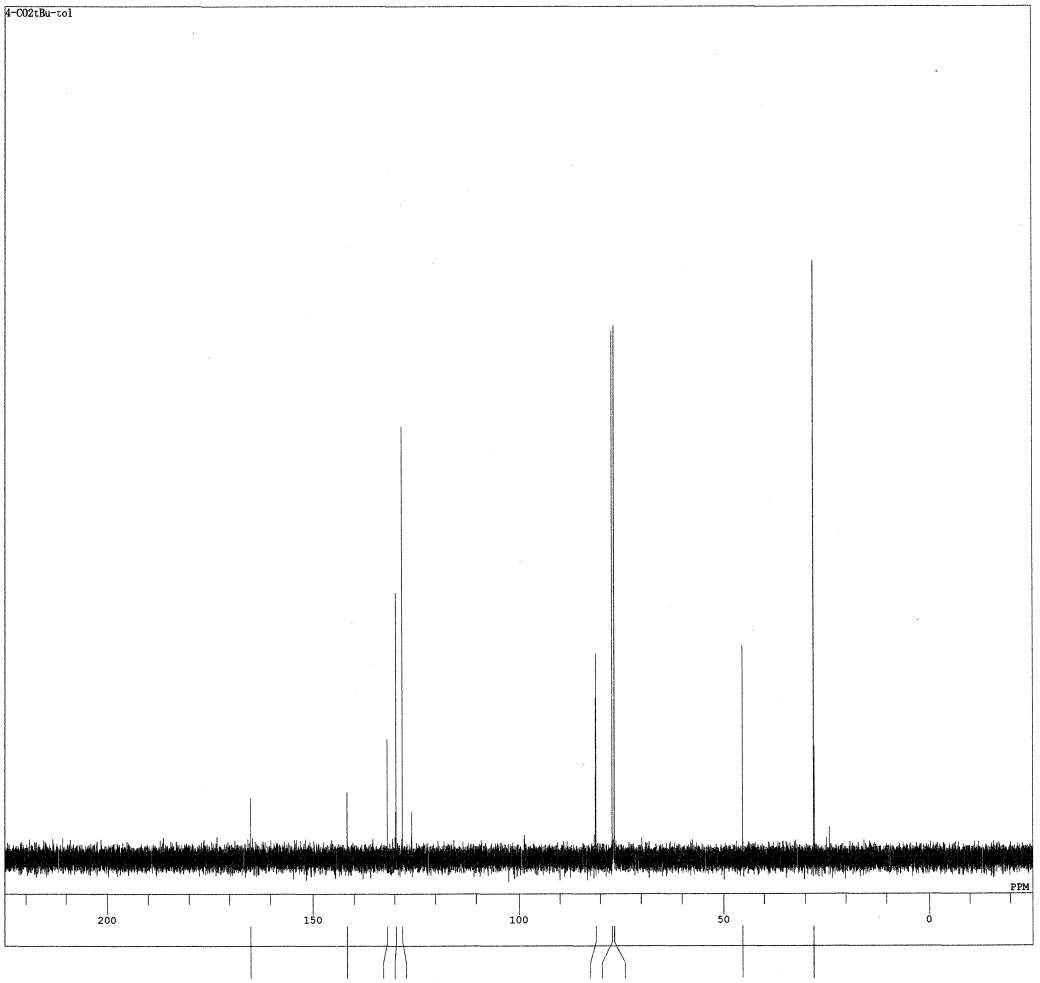

```
DFILE  ozawa10-002_13C-1-1.jdf
COMNT  4-CO2tBu-tol
DATIM  30-09-2016 10:22:24
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         65535
FREQU      30788.18 Hz
SCANS            12
ACQTM          2.1286 sec
PD             2.0000 sec
PW1            3.73 usec
IRNUC  1H
CTEMP        460.0 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.12 Hz
RGAIN            60
```



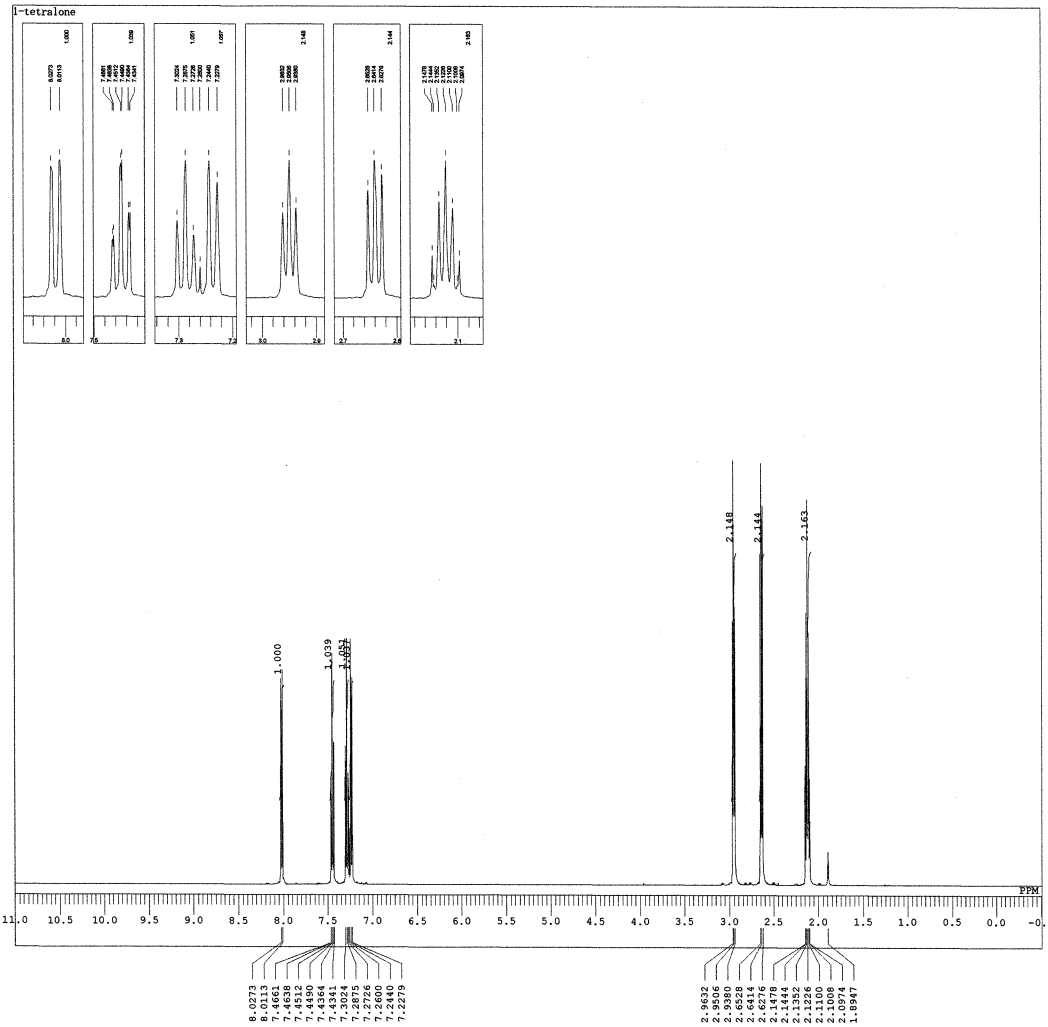

```
DFILE  ozawa10-009_1H.als
COMNT  4-CO2tBu-tol
DATIM  2016-10-05 17:05:17
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.51 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1            5.55 usec
IRNUC  1H
CTEMP         21.3 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.10 Hz
RGAIN            30
```



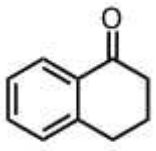
2u



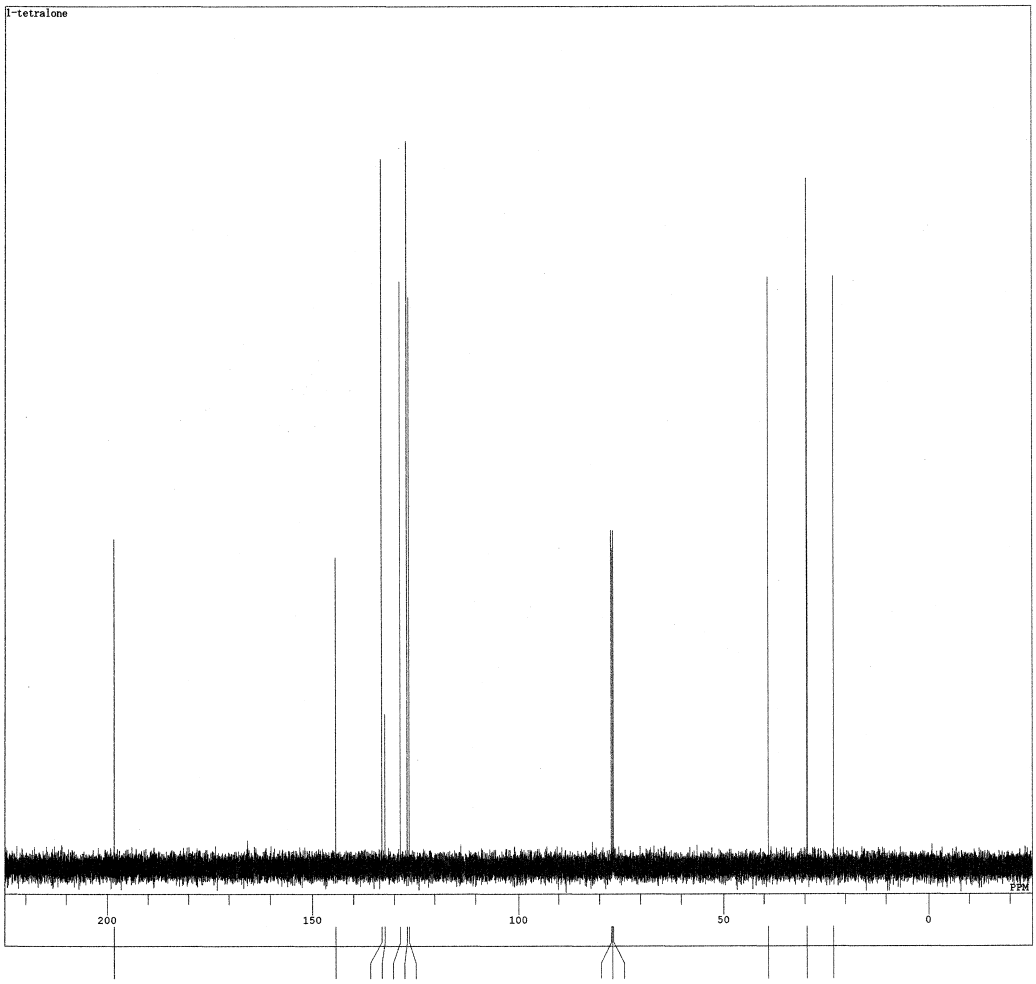

```
DFILE  ozawa10-009_13C.als
COMNT  4-CO2tBu-tol
DATIM  2016-10-05 17:06:14
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         52428
FREQU      31446.54 Hz
SCANS            48
ACQTM          1.6672 sec
PD             2.0000 sec
PW1            3.40 usec
IRNUC  1H
CTEMP         21.5 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.10 Hz
RGAIN            60
```



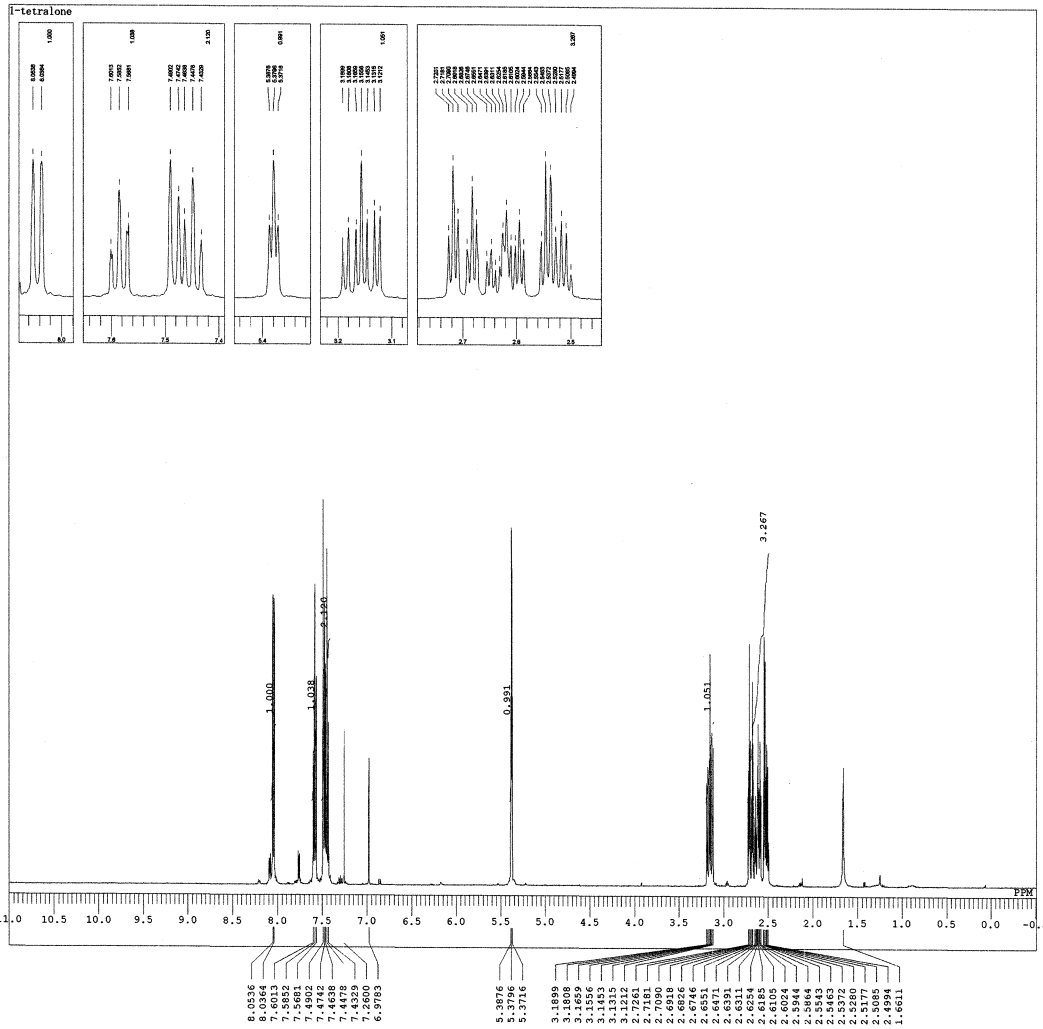
DFILE ozawa09-180_SM_1H.als
COMNT 1-tetralone
DATIM 2016-09-21 15:53:09
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 26



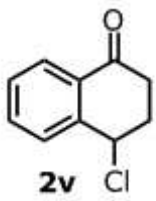
1v



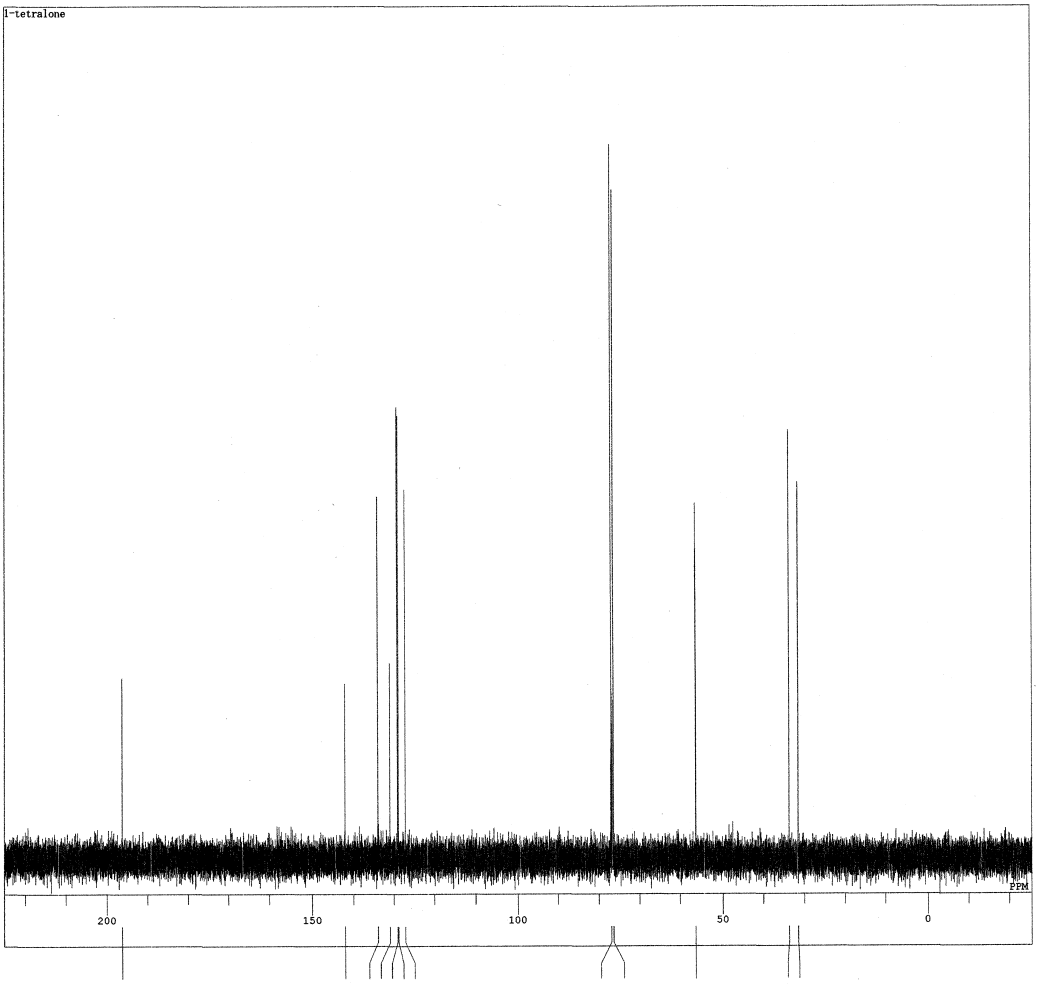
DFILE ozawa09-180_SM_13C.als
COMNT 1-tetralone
DATIM 2016-09-21 15:54:16
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.54 Hz
SCANS 12
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



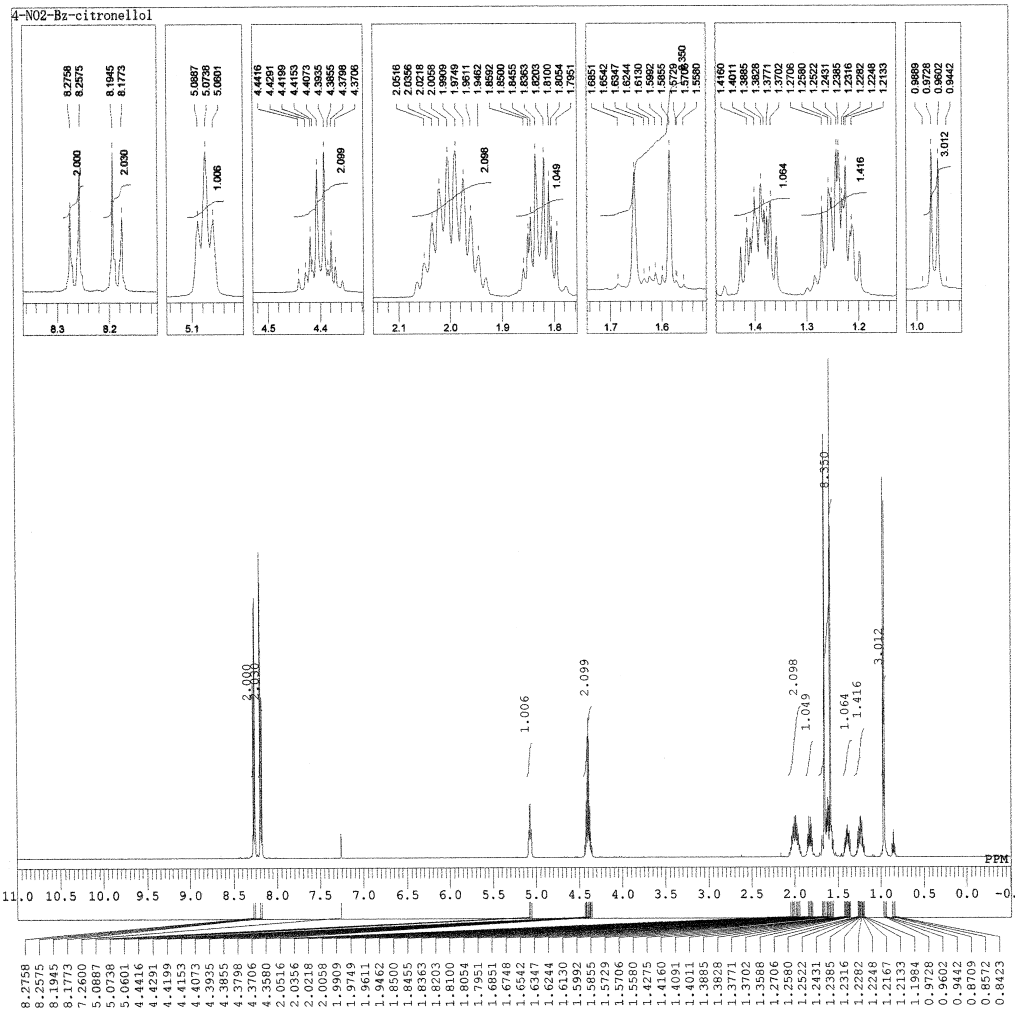
DFILE ozawa09-180_1H.als
COMNT 1-tetralone
DATIM 2016-09-30 17:24:21
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 30



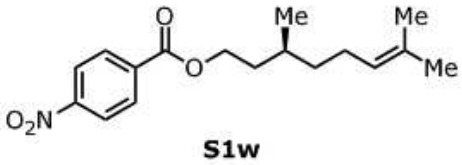
2v



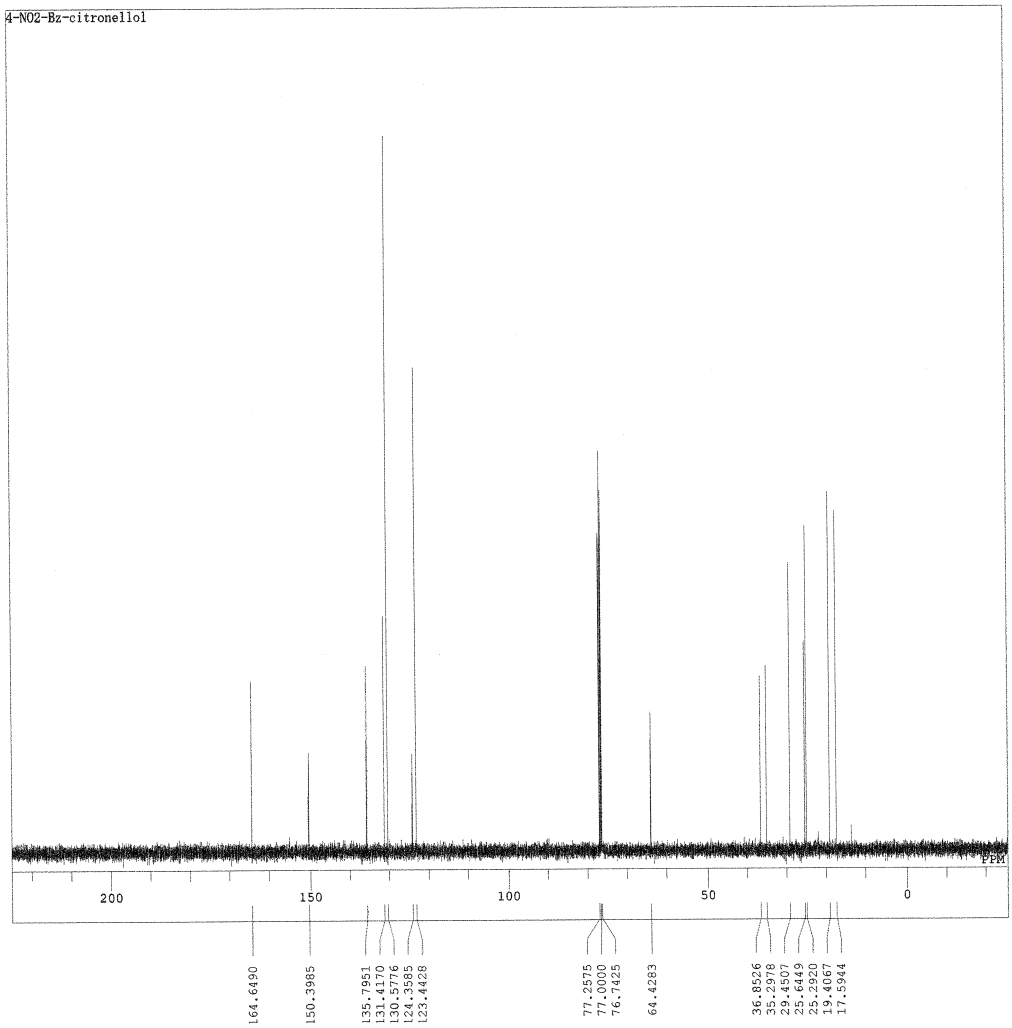
DFILE ozawa09-180_13C.als
COMNT 1-tetralone
DATIM 2016-09-30 17:25:18
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 52428
FREQU 31446.54 Hz
SCANS 28
ACQTM 1.6672 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



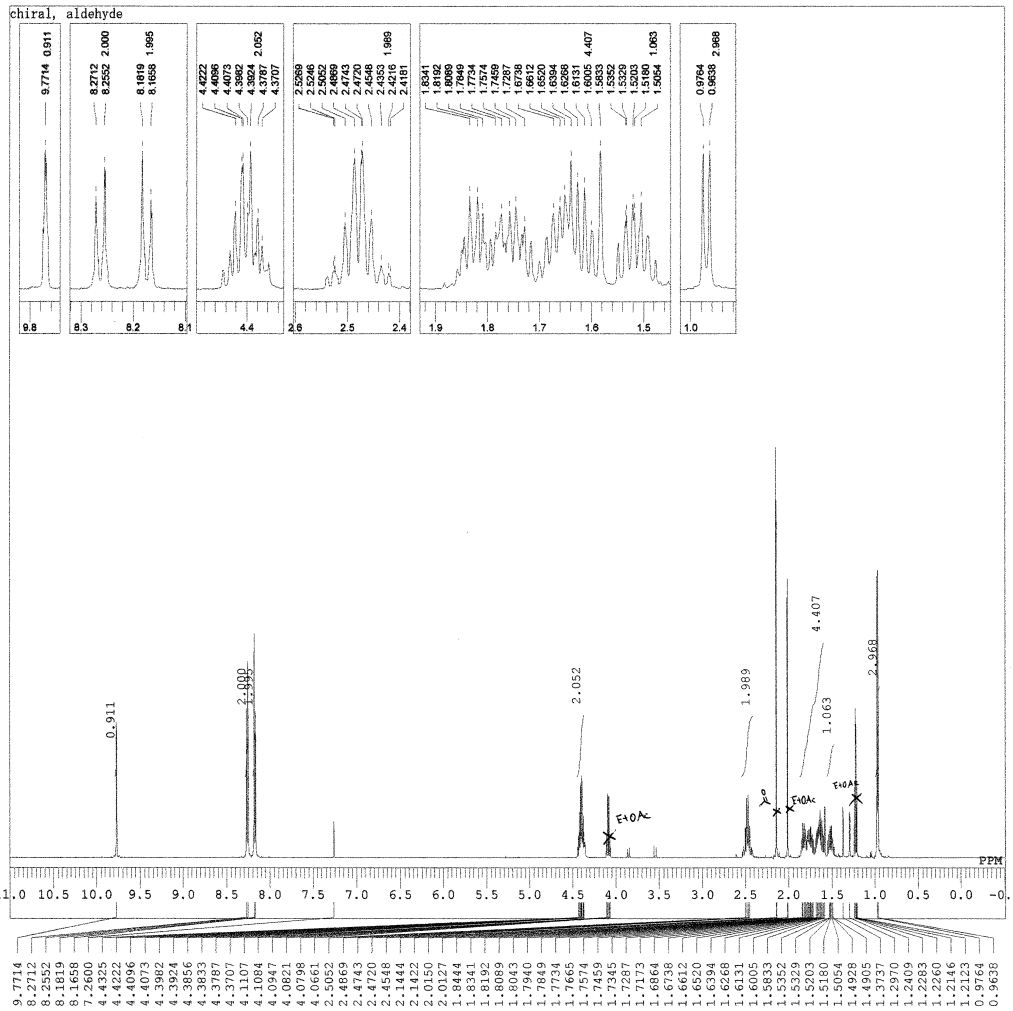

```
DFILE   ozawa08-174_1H.als
COMNT   4-NO2-Bz-citronellol
DATIM   2015-10-15 10:05:36
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          13107
FREQU        7507.51 Hz
SCANS             4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1            5.55 usec
IRNUC   1H
CTEMP          25.6 c
SLVNT   CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            22
```



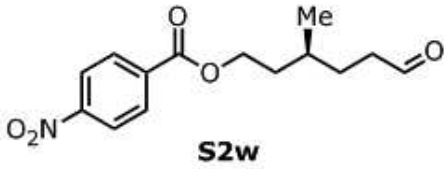
S1w



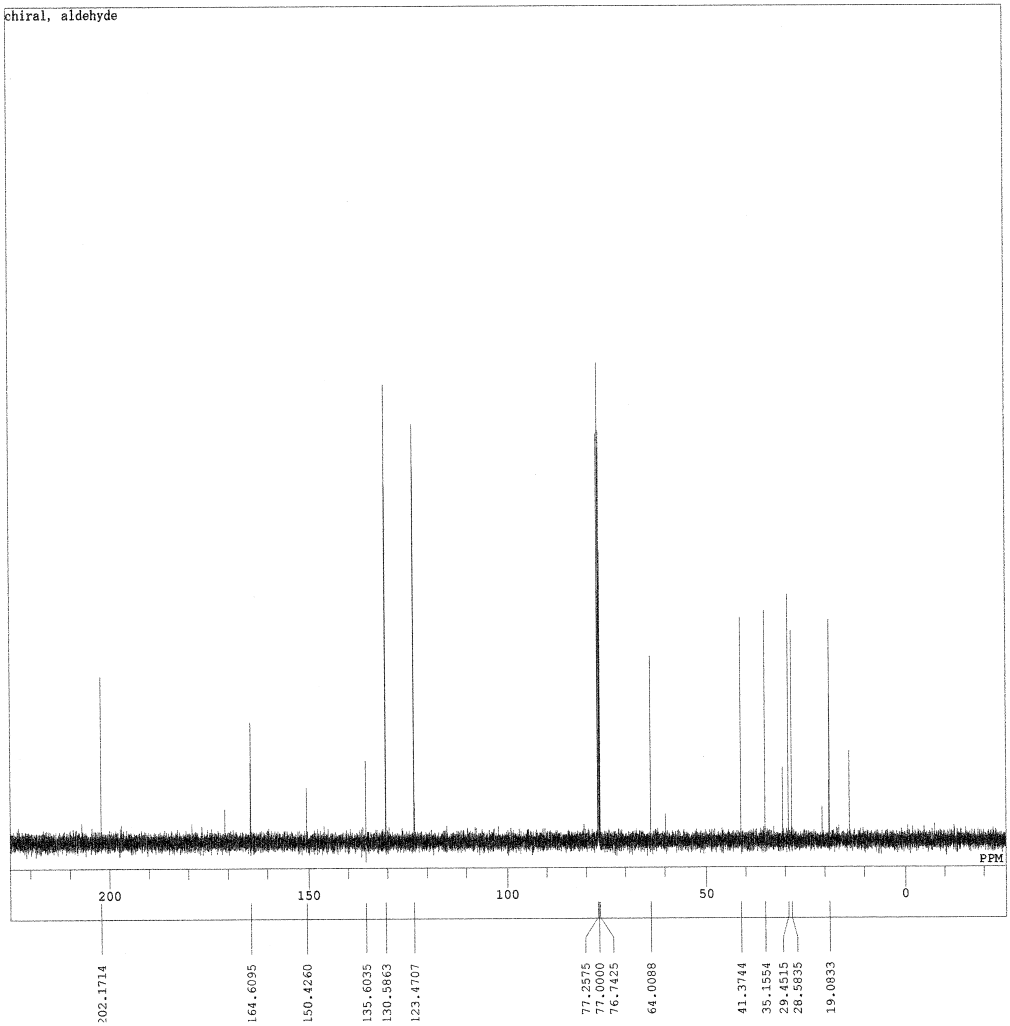

```
DFILE   ozawa08-174_13C.als
COMNT   4-NO2-Bz-citronellol
DATIM   2015-10-15 10:07:19
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          26214
FREQU       31446.54 Hz
SCANS            20
ACQTM          0.8336 sec
PD            10.0000 sec
PW1            3.40 usec
IRNUC   1H
CTEMP          24.3 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF             0.12 Hz
RGAIN            60
```



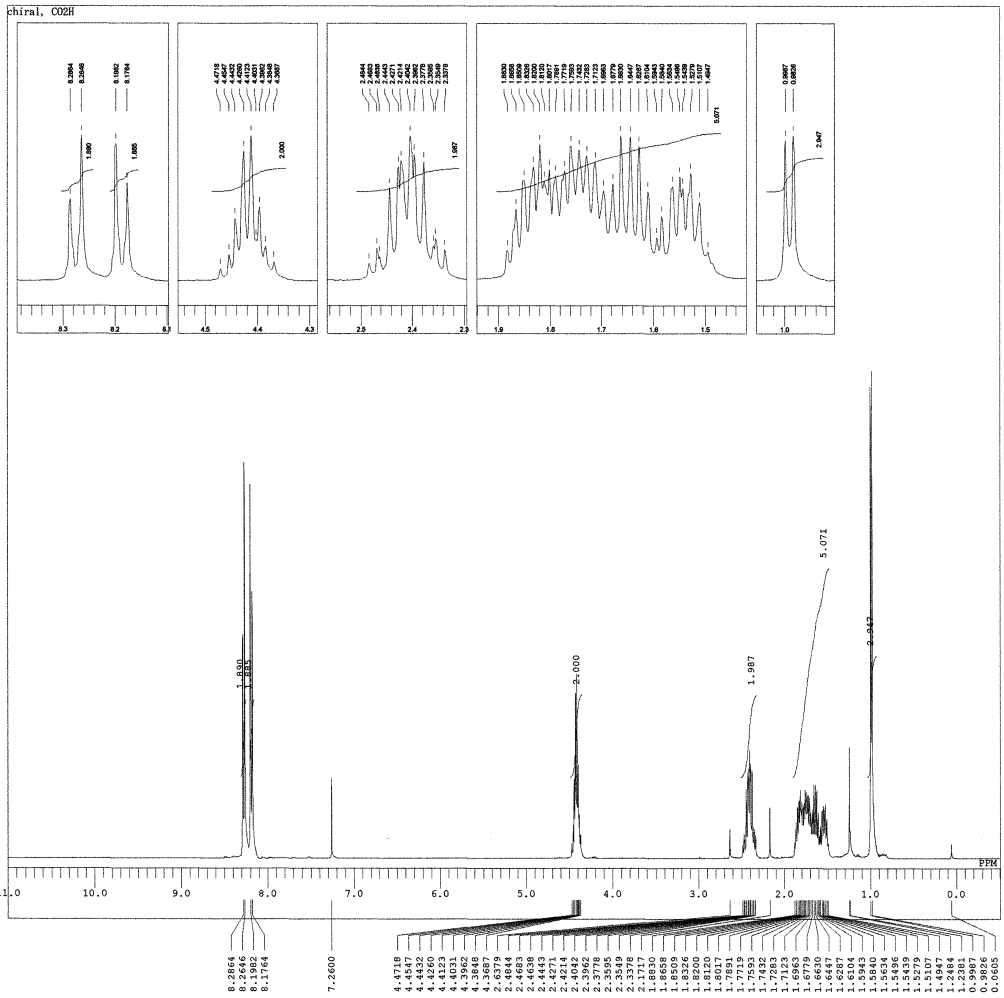
DFILE ozawa08-194_1H.jdf
COMNT chiral, aldehyde
DATIM 2015-10-30 12:45:36
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 28.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 24



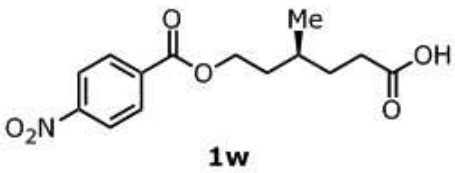
S2w



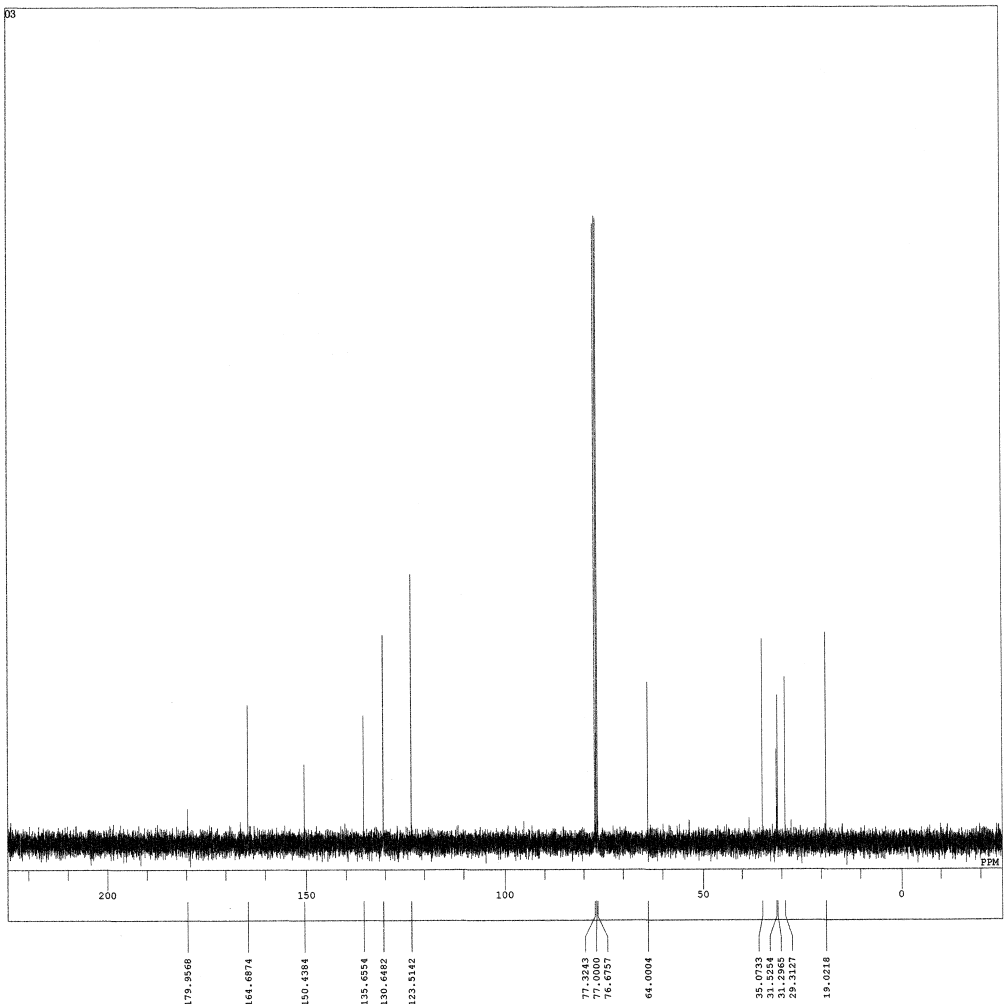
DFILE ozawa08-194_13C.jdf
COMNT chiral, aldehyde
DATIM 2015-10-30 12:47:19
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32767
FREQU 39308.18 Hz
SCANS 16
ACQTM 0.8336 sec
PD 10.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 28.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60



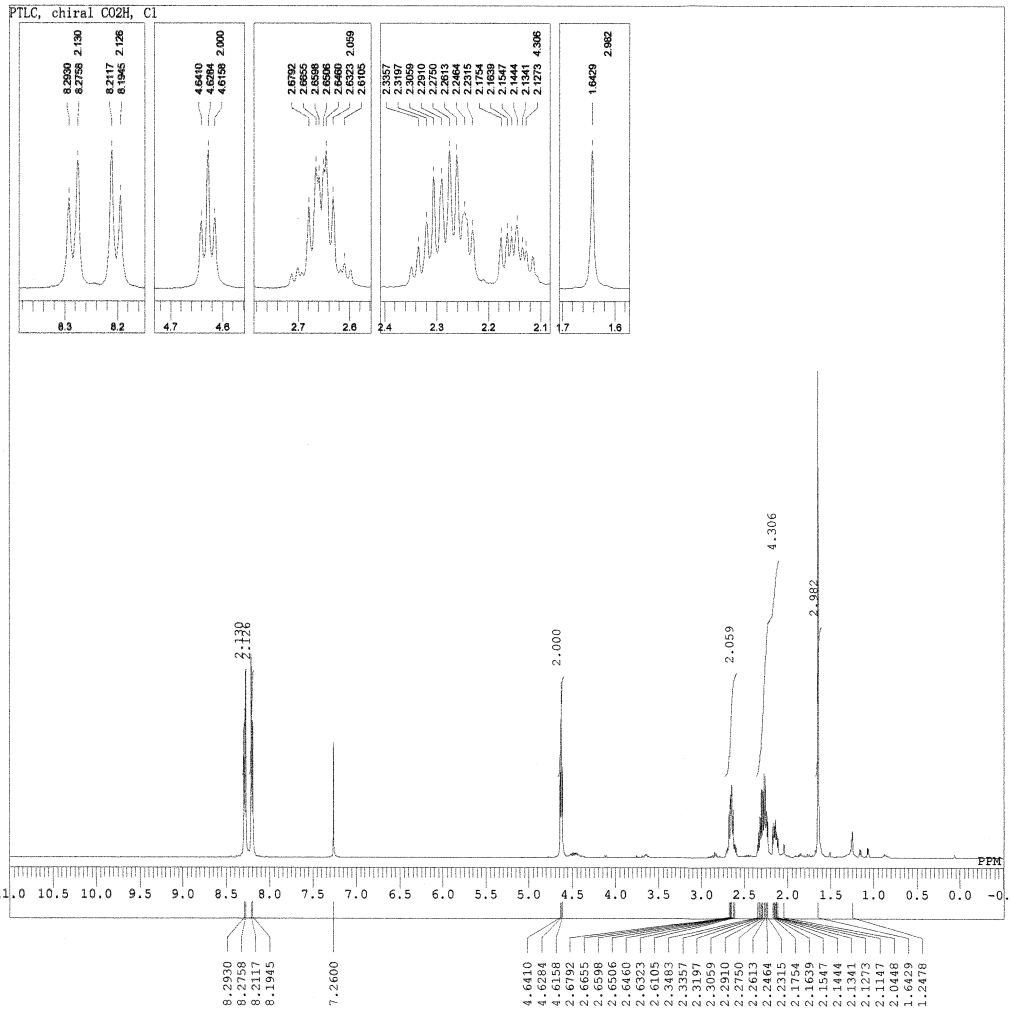

```
DFILE  ozawa08-194_MP.als
COMNT  chiral, CO2H
DATIM  31-10-2015 09:51:47
OBNUC  1H
EXMOD  proton.jxp
OBFRQ        391.78 MHz
OBSET          8.51 KHz
OBFIN          3.34 Hz
POINT         13107
FREQU       5882.35 Hz
SCANS             4
ACQTM          2.2282 sec
PD             5.0000 sec
PW1            4.99 usec
IRNUC  1H
CTEMP         23.0 c
SLVNT  CDCL3
EXREF          7.26 ppm
BF             0.12 Hz
RGAIN            30
```



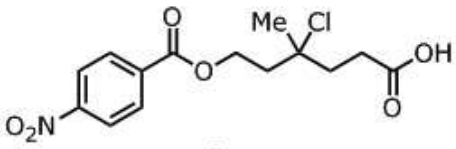
1w



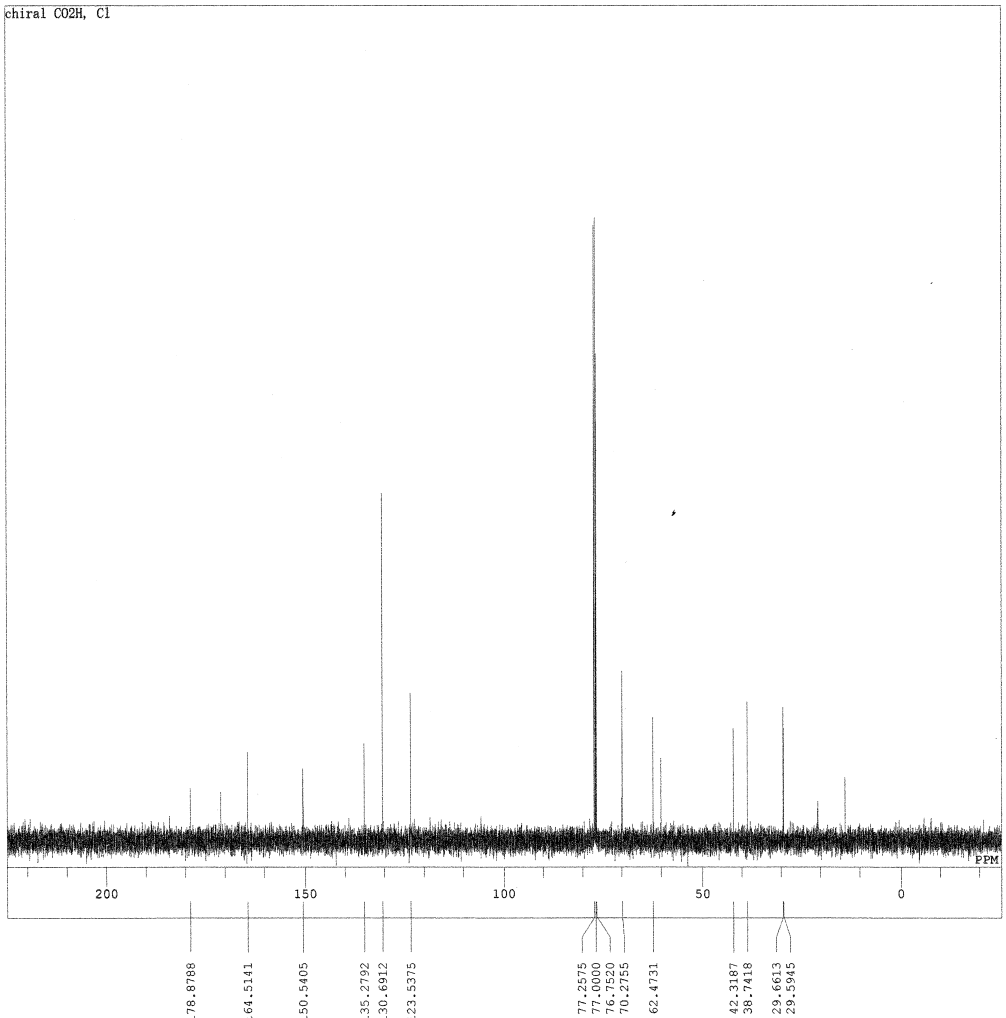

```
DFILE  ozawa09-194_MP_13C.jdf
COMNT  O3
DATIM  31-10-2015 10:20:05
OBNUC  13C
EXMOD  carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         32767
FREQU      30788.18 Hz
SCANS            28
ACQTM          1.0643 sec
PD            10.0000 sec
PW1            3.16 usec
IRNUC  1H
CTEMP         20.3 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             0.12 Hz
RGAIN            60
```



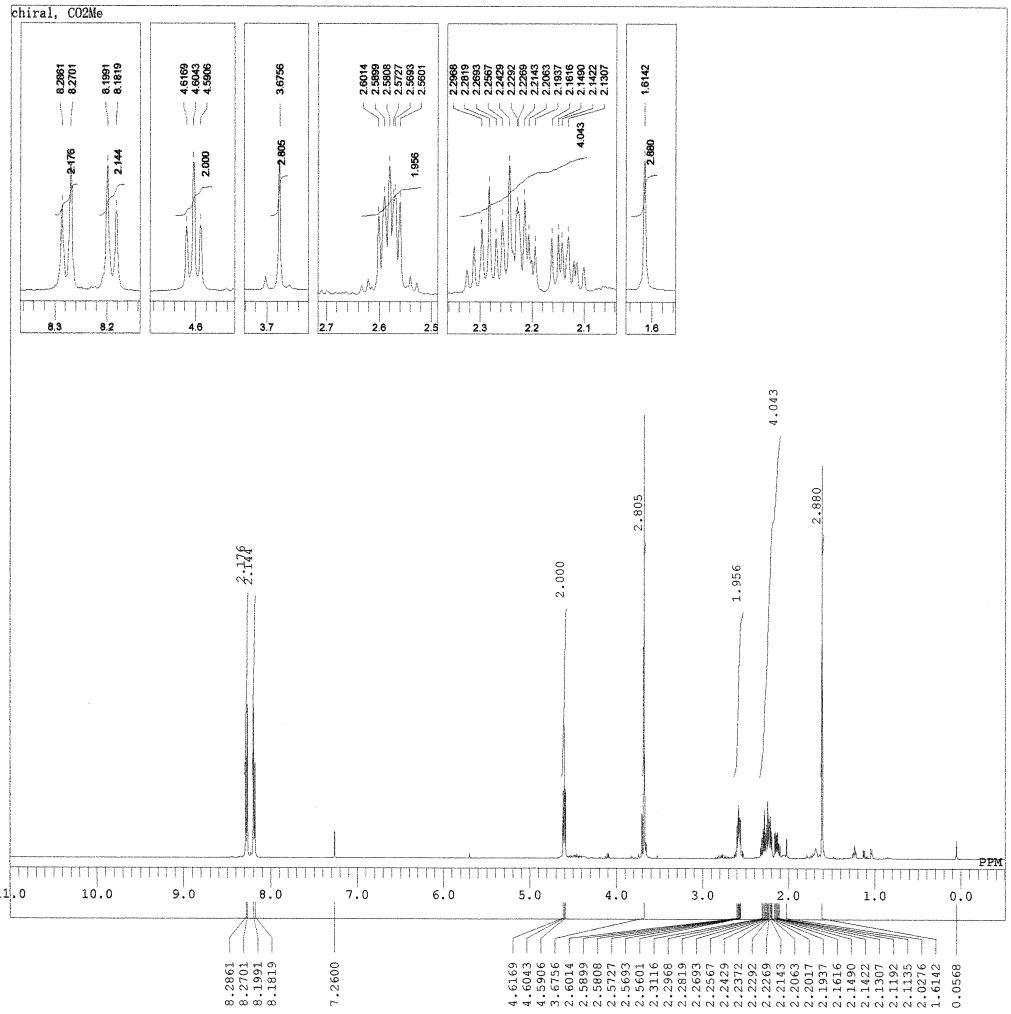
DFILE ozawa08-179_1H.jdf
COMNT PTLC, chiral CO2H, Cl
DATIM 2015-11-03 14:56:14
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 25.6 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 1.20 Hz
RGAIN 28



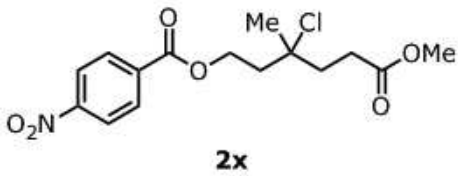
2w



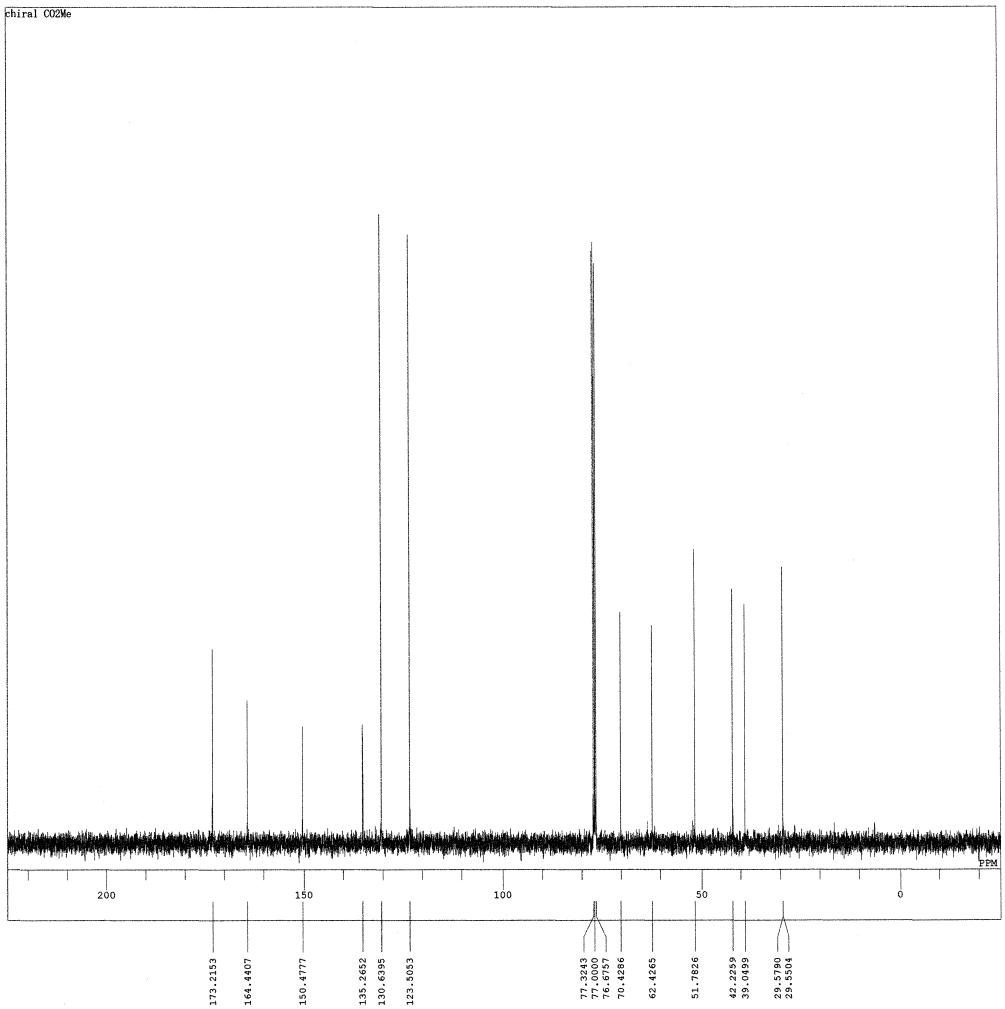
DFILE ozawa08-196_13C.jdf
COMNT chiral CO2H, Cl
DATIM 2015-11-03 15:46:15
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32767
FREQU 39308.18 Hz
SCANS 16
ACQTM 0.8336 sec
PD 10.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 28.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60




```
DFILE   ozawa08-183_1H.jdf
COMNT   chiral, CO2Me
DATIM   2015-10-27 16:41:26
OBNUC   1H
EXMOD   proton.jxp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        9384.38 Hz
SCANS              4
ACQTM          1.7459 sec
PD             5.0000 sec
PW1              5.55 usec
IRNUC   1H
CTEMP           31.2 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.12 Hz
RGAIN             26
```



2x




```
DFILE   ozawa08-183_13C.als
COMNT   chiral CO2Me
DATIM   24-10-2015 12:47:39
OBNUC   13C
EXMOD   carbon.jxp
OBFRQ         98.52 MHz
OBSET          4.64 KHz
OBFIN          8.74 Hz
POINT         26214
FREQU      24630.54 Hz
SCANS            16
ACQTM        1.0643 sec
PD          10.0000 sec
PW1            3.16 usec
IRNUC   1H
CTEMP          20.5 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            60
```