Supporting Information

Di-*tert*-butyl peroxide (DTBP) promoted dehydrogenative coupling: An expedient and metal-free synthesis of oxindoles via intramolecular C(sp^2)-H and C(sp^3)-H bond activation

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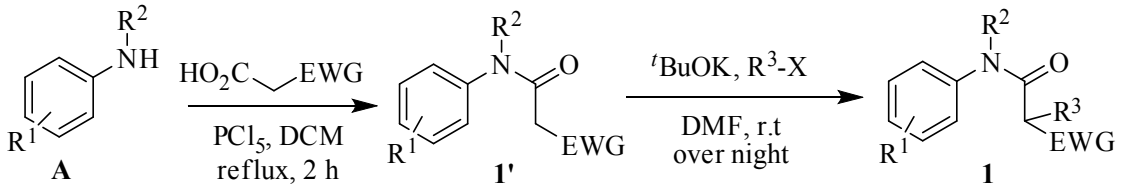
(1) General methods:

Aniline, alkyl halides and peroxides were purchased from commercial sources (Sigma-Aldrich, Alfa Aesar, Merck) and used without purification. All solvents were purchased from Spectrochem , India; dried and distilled immediately prior to use under nitrogen atmosphere. All other reagents were used as it from commercial sources. All reactions were performed in oven-dried glassware. Melting points were determined in open capillaries and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer in $CDCl_3$ with TMS as internal standard (chemical shift in δ). Chemical shifts of common trace PMR impurities ($CDCl_3$, ppm) in some samples: H_2O: ~1.58-1.68; $CHCl_3$: ~7.26. MS were recorded on a waters Q-TOF micro^{Tm} instrument. Silica gel [(60-120mesh), Rankem, India; (230-400 mesh) Spectrochem , India] was used for chromatographic separation. Silica gel G (CDH, India) was used for TLC. Petroleum ether refers to the fraction boiling between 60 °C and 80 °C.

(2) General procedure for the preparation of starting material (1) and products (2):

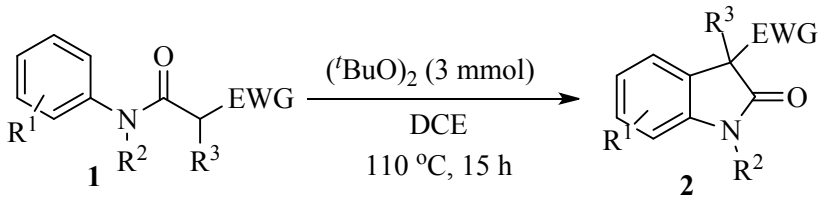
(a) Synthesis of compound 1:

1 equiv. of aniline (**A**) was taken in a 250 ml round bottom flask and dissolved it in 100 ml dry DCM; then 1.5 equiv. of PCl_5 was added to it. The mixture was refluxed for 30 minutes with stirring. After that the mixture was cooled to room temperature and 1.2 equiv. of acid (**B**) was added to it and refluxed for additional 1.5 h. The reaction mixture was cooled to room temperature and completion of the reaction was checked by TLC. Excess PCl_5 was neutralized by sodium bicarbonate and washed with water for three times. The organic layer was dried over Na_2SO_4, concentrated to afford compound **1'** (sufficiently pure) which was used for the next step without purification. 3 mmol of compound **1'** was dissolved in dry DMF and 3.6 mmol of ^{t}BuOK was added to it and stirred at room temperature for 15 minutes. After that 4.5 mmol of alkyl halide was added to it under stirring condition and stirred for overnight. Completion of reaction was checked by TLC, diluted with water and 50 ml of DCM, washed with H_2O (3 ×20 ml) and saturated NaCl (aq) (1 ×20 ml). The organic part was dried over Na_2SO_4, concentrated and purified by flash chromatography, using ethyl acetate/petroleum ether (2:8) as the eluent to afford product **1** in upto 84 % yield.



R^1 = H, Cl, NO_2; R^2 = Me, Et; EWG = CN, CO_2H; R^3 = Me, Et, ^{i}Pr, ^{n}Bu, Bn

(b) Synthesis of compound 2:



1 mmol of *β*-*N*-arylamido nitrile or ester, 3 mmol of di-*tert*-butyl peroxide (DTBP) and 4 ml DCE were taken in a sealed tube and the mixture was degassed with nitrogen for twice. Then the reaction mixture was heated at 110 °C for 15 h. The reaction mixture was cooled to room temperature and diluted with DCM. The mixture was then transferred to a 100 ml round bottom flask. The solvent was evaporated under reduced pressure and purified by flash coloumn chromatography, using ethyl acetate/petroleum ether (2:8) as the eluent to afford the product **2**.

(4) (a) Experimental characterization data of starting material (1):

All reactions were performed according to general procedure

2-Cyano-N-methyl-N-phenylpropanamide (1a):

White solid; MP: 60-62°C

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.45 (d, J = 7.2 Hz, 3H); 3.32 (s, 3H); 3.47 (q, J = 7.2 Hz, 1H); 7.29 (d, J = 7.6 Hz, 2H); 7.44 (t, J = 7.6 Hz, 1H); 7.51 (t, J = 8.0 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 15.9, 29.5, 38.2, 118.4, 127.3, 129.0, 130.4, 142.3, 165.6.

HRMS calcd. for $C_{11}H_{12}N_2ONa$ $[M + Na]^+$: 211.0847; found: 211.0842

2-Cyano-N-methyl-N-phenylbutanamide (1b):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.96 (t, J = 7.6 Hz, 3H); 1.87-1.94 (m, 2H); 3.29 (t, J = 7.2 Hz, 1H); 3.32 (s, 3H); 7.26 (d, J = 7.6 Hz, 2H); 7.44 (t, J = 7.2 Hz); 7.50 (t, J = 7.6Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 11.5, 24.2, 36.7, 38.1, 117.6, 127.3, 128.9, 130.4, 142.4, 165.0.

HRMS calcd. for $C_{12}H_{14}N_2ONa$ $[M + Na]^+$: 225.1004; found: 225.1007

2-Cyano-N-ethyl-N-phenylpropanamide (1c):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.51(t, J = 7.2 Hz, 3H); 1.44 (d, J = 7.2 Hz, 3H); 3.41 (q, J = 7.2 Hz, 1H); 3.69-3.76 (m, 1H); 3.80-3.87 (m, 1H); 7.27 (d, J = 7.2 Hz, 2H); 7.44-7.54 (m, 3H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 12.6, 15.8, 29.8, 45.1, 118.5, 128.3, 129.0, 130.3, 140.5, 165.0

HRMS calcd. for $C_{12}H_{14}N_2ONa$ $[M + Na]^+$: 225.1004; found: 225.1009

2-cyano-N-ethyl-N-phenylbutanamide (1d):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.95 (t, J = 7.6 Hz, 3H); 1.15 (t, J = 7.2 Hz, 3H); 1.84-1.93 (m, 2H); 3.28 (t, J = 7.6 Hz, 1H); 3.67-3.76 (m, 1H); 3.81-3.90 (m, 1H); 7.28 (d, J = 7.6 Hz, 2H); 7.46 (t, J = 7.2 Hz, 1H); 7.53 (t, J = 7.6 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 11.3, 12.6, 24.0, 36.9, 44.8, 117.6, 128.2, 128.8, 130.2, 140.6, 164.3.

HRMS calcd. for $C_{13}H_{16}N_2ONa$ $[M + Na]^+$: 239.1160; found: 239.1154

N-(4-chlorophenyl)-2-cyano-N-methylpropanamide (1e):

White soilid; MP: 50-52°C

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.46 (d, J = 6.8 Hz, 3H); 3.30 (s, 3H); 3.46 (q, J = 6.8 Hz, 1H); 7.27 (d, J = 8.4 Hz, 2H); 7.48 (d, J = 8.4 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 15.8, 29.5, 38.2, 118.3, 128.8, 130.6, 134.9, 140.7, 165.3.

HRMS calcd. for $C_{11}H_{11}ClN_2ONa$ $[M + Na]^+$: 245.0458; found: 245.0462

N-(4-chlorophenyl)-2-cyano-N-methylbutanamide (1f):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.98 (t, J = 7.2 Hz, 3H); 1.90 (quintet, J = 7.2 Hz, 2H); 3.26-3.36 (m, 4H, one NMe and one C-H); 7.27 (d, J = 8.4 Hz, 2H); 7.49 (d, J = 8.4 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 11.5, 24.1, 36.7, 38.1, 117.4, 128.8, 130.6, 134.7, 140.8, 164.8.

HRMS calcd. for $C_{12}H_{13}ClN_2ONa$ $[M + Na]^+$: 259.0614; found:259.0609

2-Cyano-N-methyl-N-(4-nitrophenyl)propanamide (1g):

Pale yellow solid; MP: 118-120°C

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.51 (d, J = 6.8 Hz, 3H); 3.40 (s, 3H); 3.51 (brs, 1H); 7.54 (d, J = 8.8 Hz); 8.37 (d, J = 8.8 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 15.6, 29.8, 38.3, 117.8, 125.7, 128.3, 147.3, 147.9, 164.9.

HRMS calcd. for $C_{11}H_{11}N_3O_3Na$ $[M + Na]^+$: 256.0698; found: 256.0695

2-Cyano-N-ethyl-3-methyl-N-phenylbutanamide (1h):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.90 (d, J = 6.4 Hz, 3H); 1.07 (d, J = 6.4 Hz, 3H); 2.27-2.36 (m, 1H); 3.15 (d, J = 8.4 Hz, 1H); 3.33 (s, 3H); 7.26 (d, J = 7.6 Hz, 2H); 7.45 (t, J = 8.0 Hz, 1H); 7.51 (t, J = 8.0 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 19.7, 20.6, 30.3, 38.0, 42.6, 117.0, 127.2, 128.9, 130.4, 142.5, 164.9.

HRMS calcd. for $C_{13}H_{16}N_2ONa$ $[M + Na]^+$: 239.1160; found: 239.1166

2-Cyano-N-ethyl-N,3-diphenylpropanamide (1i):

White solid; MP: 88-90°C

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.06 (t, J = 7.2 Hz, 3H); 3.06 (q, J = 6.4 Hz, 1H); 3.69 (q, J = 6.8 Hz, 2H); 7.06 (m, 2H); 7.25-7.29 (m, 4H); 7.38 (m, 4H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 12.7, 36.8, 37.3, 45.0, 117.4, 124.6, 128.2, 128.7, 129.0, 129.2, 130.2, 136.1, 140.5, 163.9.

HRMS calcd. for $C_{18}H_{18}N_2ONa$ $[M + Na]^+$: 301.1317; found: 301.1320

2-Cyano-N-ethyl-N-phenylhexanamide (1j):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.84 (t, J = 7.2 Hz, 3H); 1.17-1.34 (m, 4H); 1.86 (q, J = 7.6 Hz, 2H); 3.32 (s, 3H); 3.36 (t, J = 7.6 Hz, 1H); 7.27 (d, J = 7.2 Hz, 2H); 7.44-7.46 (m, 1H); 7.51 (t, J = 7.6 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 13.6, 21.8, 28.9, 30.2, 35.1, 38.1, 117.7, 127.2, 128.9, 130.4, 142.4, 165.2.

HRMS calcd. for $C_{14}H_{18}N_2ONa$ $[M + Na]^+$: 253.1317; found: 253.1312

2-Cyano-N-ethyl-N-phenylpent-4-enamide (1k):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 2.53-2.67 (m, 2H); 3.32 (s, 3H); 3.41 (t, J = 7.6 Hz, 1H); 5.10-5.15 (m, 2H); 5.61-5.71 (m, 1H); 7.27 (d, J = 7.6 Hz, 2H); 7.44 (t, J = 7.2 Hz, 1H); 7.50 (t, J = 7.6 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 34.3, 35.0, 38.1, 117.2, 119.3, 127.3, 129.0, 130.4, 132.2, 142.2, 164.3.

HRMS calcd. for $C_{13}H_{14}N_2ONa$ $[M + Na]^+$: 237.1004; found: 237.1007

Ethyl 2-(ethyl(phenyl)carbamoyl)butanoate (1l):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.85 (t, J = 6.4 Hz, 3H); 1.16 (t, J = 6.8 Hz, 3H); 1.25 (t, J = 6.8 Hz, 3H); 1.82-1.87 (m, 1H); 1.91-1.96 (m, 1H); 3.18 (dd, J = 8.8Hz and 2.4 Hz, 2H); 3.69-3.78 (m, 1H); 3.82-3.91 (m, 1H); 4.12 (q, J = 7.2 Hz, 2H); 7.25 (d, J = 7.2 Hz, 2H); 7.39 (t, J = 7.2 Hz, 1H); 7.46 (t, J = 7.6 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 11.8, 12.8, 13.9, 22.5, 44.3, 50.7, 60.8, 128.2, 128.7, 129.6, 141.7, 168.3, 17.0.

HRMS calcd. for $C_{15}H_{21}NO_3Na$ $[M + Na]^+$: 286.1419; found: 286.1416

Ethyl 3-(ethyl(phenyl)amino)-2-methyl-3-oxopropanoate (1m):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.15 (t, J = 7.2 Hz, 3H); 1.24 (t, J = 6.8 Hz, 3H); 1.30 (d, J = 7.2 Hz, 3H); 3.34 (q, J = 7.2 Hz, 1H); 3.68-3.77 (m, 1H); 3.80-3.89 (m, 1H); 4.11 (q, J = 7.2 Hz, 2H); 7.25 (d, J = 7.2 Hz, 2H); 7.40 (t, J = 7.6 Hz, 1H); 7.46 (t, J = 7.6 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 12.7, 13.9, 43.7, 44.2, 60.9, 128.2, 128.4, 129.7, 141.7, 169.4, 170.7.

HRMS calcd. for $C_{14}H_{19}NO_3Na$ $[M + Na]^+$: 272.1263; found: 272.1259

Ethyl 2-benzyl-3-(methyl(phenyl)amino)-3-oxopropanoate (1n):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.25 (t, J = 7.2 Hz, 3H); 3.07 (dd, J = 13.2 Hz and 4.8 Hz, 1H); 3.17 (s, 3H); 3.22 (dd, J = 13.2 Hz and 6.4 Hz, 1H); 3.55 (q, J = 4.8 Hz, 1H); 4.11-4.18 (m, 2H); 6.64 (brs, 1H); 7.02-7.04 (m, 2H); 7.23-7.36 (m, 7H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 14.1, 35.2, 37.4, 51.0, 61.3, 126.6, 127.6, 128.0, 128.4, 129.2, 129.5, 138.4, 143.1, 168.5, 169.4.

HRMS calcd. for $C_{19}H_{21}NO_3Na$ $[M + Na]^+$: 334.1419; found: 334.1411

Ethyl 2-(methyl(phenyl)carbamoyl)butanoate (1o):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.84 (t, J = 7.6 Hz, 3H); 1.24 (t, J = 7.2 Hz, 3H); 1.81-1.97 (m, 2H); 3.25-3.29 (m, 1H); 3.32 (s, 3H); 4.08-4.15 (m, 2H); 7.26 (d, J = 7.6 Hz, 2H); 7.38 (t, J = 7.6 Hz, 1H); 7.46 (t, J = 7.6 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 11.8, 14.0, 22.7, 37.5, 50.4, 60.9, 127.6, 128.1, 129.8, 143.4, 169.0, 169.9.

HRMS calcd. for $C_{14}H_{19}NO_3Na$ $[M + Na]^+$: 272.1263; found: 272.1267

Ethyl 2-methyl-3-(methyl(phenyl)amino)-3-oxopropanoate (1p):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.23 (t, J = 7.2 Hz, 3H); 1.30 (d, J = 7.2 Hz, 3H); 3.31 (s, 3H); 3.42 (q, J = 6.8 Hz, 1H); 4.07-4.14 (m, 2H); 7.27 (d, J = 7.2 Hz, 2H); 7.38 (t, J = 7.2 Hz, 1H); 7.46 (t, J = 7.6 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 14.0, 14.0, 37.5, 43.4, 61.1, 127.4, 128.2, 129.9, 143.4, 170.1, 170.7.

HRMS calcd. for $C_{13}H_{17}NO_3Na$ $[M + Na]^+$: 258.1106; found: 258.1101

2-Cyano-N-methyl-N-phenylhept-6-enamide (1q):

Gummy liquid

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.34-1.50 (m, 2H); 1.85-1.91 (m, 2H); 1.94-1.99 (m, 2H); 3.32 (s, 3H); 3.36 (t, J = 7.6 Hz, 1H); 4.90-4.92 (m, 1H); 4.95 (s, 1H); 5.63-5.73 (m, 1H); 7.25 (d, J = 7.2 Hz, 2H); 7.42-7.46 (m, 1H); 7.50 (t, J = 8.0 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 26.0, 29.8, 32.7, 34.9, 38.1, 115.5, 117.6, 127.2, 129.0, 130.4, 137.3, 142.4, 165.0.

HRMS calcd. for $C_{15}H_{19}N_2O$ $[M + H]^+$: 243.1497; found: 243.1495.

Diethyl 1-(methyl(phenyl)amino)-1-oxopropan-2-ylphosphonate (1r):

Gummy liquid

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.25 (t, J = 7.6 Hz, 3H); 1.28-1.38 (m, 6H); 3.05 (dq, J = 13.2 and 7.2 Hz, 1H); 3.30 (d, J = 1.2 Hz, 3H); 4.01-4.11 (m, 2H); 4.13-4.26 (m, 2H); 7.31 (d, J = 7.2 Hz, 2H); 7.34-7.38 (m, 1H); 7.44 (t, J = 7.2 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 13.1 (d, J = 7.0 Hz), 16.2 (d = 6.0 Hz), 16.4 (d, J = 6.0 Hz), 36.0, 37.4 (d, J = 260.0 Hz), 62.0 (d, J = 7 Hz), 62.9 (d, J = 6.0 Hz), 127.4, 128.1, 129.9, 143.7, 169.5 (d, J = 4.0 Hz).

HRMS calcd. for $C_{14}H_{23}NO_4P$ $[M + H]^+$: 300.1365; found: 300.1363.

2-Cyano-N-(4-methoxyphenyl)propanamide (1s):

White solid; MP: 137-139 ^oC

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.66 (d, J = 7.6 Hz, 3H); 3.56 (q, J = 7.6 Hz, 3H); 3.80 (s, 3H); 6.87 (d, J = 8.8 Hz, 2H); 7.39 (d, J = 8.8 Hz, 2H); 7.84 (brs, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 15.7, 32.9, 55.5, 114.3, 118.9, 122.5, 129.5, 157.3, 163.0.

HRMS calcd. for $C_{11}H_{13}N_2O_2$ $[M + H]^+$: 205.0977; found: 205.0971.

2-Cyano-N-methyl-N-phenylacetamide[1, 2] (1a'):

White solid; MP: 82-84°C

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 3.25 (s, 2H); 3.32 (s, 3H); 7.25 (d, J = 7.6 Hz, 2H); 7.43 (t, J = 7.2 Hz, 1H); 7.49 (t, J = 8.0 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 25.5, 37.9, 114.2, 127.1, 129.1, 130.5, 142.4, 161.8.

HRMS calcd. for $C_{10}H_{10}N_2ONa$ $[M + Na]^+$: 197.0691; found:197.0687

(b) Experimental characterization data of products (2):

All reactions were performed according to general procedure

1,3-Dimethyl-2-oxoindoline-3-carbonitrile[1] (2a):

Colourless solid; MP: 77-79°C

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.82 (s, 3H); 3.27 (s, 3H); 6.91 (d, J = 8.0 Hz, 1H); 7.18 (t, J = 7.6 Hz, 1H); 7.39-7.44 (m, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 23.4, 27.0, 42.1, 109.2, 117.7, 123.8, 123.9, 126.8, 130.3, 142.6, 171.0.

HRMS calcd. for $C_{11}H_{10}N_2ONa$ $[M + Na]^+$: 209.0691; found: 209.0684

3-Ethyl-1-methyl-2-oxoindoline-3-carbonitrile (2b):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.99 (t, J = 7.2 Hz, 3H); 2.16-2.26 (m, 2H); 3.26 (s, 3H); 6.90 (d, J = 8.4 Hz, 1H); 7.17 (t, J = 7.6 Hz, 1H); 7.39-7.43 (m, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 8.4, 26.9, 30.8, 47.1, 109.0, 117.1, 123.7, 124.1, 125.2, 130.3, 143.1, 170.5.

HRMS calcd. for $C_{12}H_{12}N_2ONa$ $[M + Na]^+$: 223.0847; found: 223.0841

1-Ethyl-3-methyl-2-oxoindoline-3-carbonitrile (2c):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.30 (t, J = 7.2 Hz, 3H); 1.81 (s, 3H); 3.80 (quintet, J = 7.2 Hz, 2H); 6.92 (d, J = 7.6 Hz, 1H); 7.14-7.18 (m, 1H); 7.38-7.44 (m, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 12.0, 23.0, 35.1, 41.6, 108.8, 117.2, 123.2, 123.4, 127.2, 129.8, 141.2, 170.1.

HRMS calcd. for $C_{12}H_{12}N_2ONa$ $[M + Na]^+$: 223.0847; found: 223.0854

1,3-Diethyl-2-oxoindoline-3-carbonitrile (2d):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.90 (t, J = 7.6 Hz, 3H); 1.29 (t, J = 7.2 Hz, 3H); 2.17-2.28 (m, 2H); 3.71-3.79 (m, 1H); 3.80-3.88 (m, 1H); 6.93 (d, J = 8.0 Hz, 1H); 7.16 (t, J = 7.2 Hz, 1H); 7.40 (t, J = 7.2 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 8.2, 12.5, 30.9, 35.5, 47.0, 109.2, 117.2, 123.5, 124.3, 125.3, 130.2, 142.3, 170.0.

HRMS calcd. for $C_{13}H_{15}N_2O$ $[M + H]^+$: 215.1184; found: 215.1180

5-Chloro-1,3-dimethyl-2-oxoindoline-3-carbonitrile (2e):

White solid; MP: 124-126°C

H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.82 (s, 3H); 3.25 (s, 3H); 6.85 (d, J = 8.0 Hz, 1H); 7.39 (dd, J = 8.0 Hz and 2.0 Hz, 1H); 7.42 (d, J = 2.0 Hz, 1H);

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 23.3, 27.2, 42.1, 110.3, 117.1, 124.3, 128.1, 129.2, 130.4, 141.1, 170.5.

HRMS calcd. for $C_{11}H_{10}ClN_2O$ $[M + H]^+$: 221.0482; found: 221.0484

5-Chloro-3-ethyl-1-methyl-2-oxoindoline-3-carbonitrile (2f):

White solid; MP: 82-84°C

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.99 (t, J = 7.6 Hz, 3H); 2.13-2.28 (m, 2H), 3.25 (s, 3H); 6.85 (d, J = 8.8 Hz, 1H); 7.39-7.40 (m, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 8.3, 27.1, 30.8, 47.1, 110.1, 116.5, 124.6, 126.6, 129.1, 130.3, 141.7, 169.9.

HRMS calcd. for $C_{12}H_{12}ClN_2O$ [M + H]$^+$: 235.0638; found: 235.0642

1,3-Dimethyl-5-nitro-2-oxoindoline-3-carbonitrile (2g):

White solid; MP: 162-164°C

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.89 (s, 3H); 3.36 (s, 3H); 7.05 (d, J = 8.8 Hz, 1H); 8.35 (d, J = 2.0 Hz, 1H); 8.39 (dd, J = 8.8 Hz and 2.0 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 23.2, 27.5, 41.8, 109.1, 116.2, 119.9, 127.3, 127.4, 144.2, 148.0, 170.9.

HRMS calcd. for $C_{11}H_{10}N_3O_3$ [M + H]$^+$: 232.0722; found: 232.0727

1-Ethyl-3-isopropyl-2-oxoindoline-3-carbonitrile (2h):

White solid; MP: 82-84 ^{o}C

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.90 (d, J = 6.8 Hz, 3H); 1.18 (d, J = 6.8 Hz, 3H); 2.58 (septet, J = 6.8 Hz, 1H); 3.24 (s, 3H); 6.92 (d, J = 8.0 Hz, 1H); 7.16 (t, J = 7.2 Hz, 1H); 7.39-7.44 (m, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 16.4, 17.4, 26.8, 36.2, 51.2, 109.0, 117.2, 123.5, 123.8, 124.6, 130.3, 143.5, 170.2.

HRMS calcd. for $C_{13}H_{15}N_2O$ [M + H]$^+$: 215.1184; found: 215.1189

3-Benzyl-1-ethyl-2-oxoindoline-3-carbonitrile (2i):

White solid, MP: 118-120°C;

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.94 (t, J = 7.2 Hz, 3H); 3.40 (d, J = 12.8 Hz, 1H); 3.43-3.52 (m, 1H); 3.54 (d, J = 13.2 Hz, 1H); 3.67-3.74 (m, 1H); 6.71 (d, J = 7.6 Hz, 1H); 6.93 (d, J = 7.2 Hz, 2H); 7.13 (t, J = 7.2 Hz, 3H); 7.17-7.19 (m, 1H); 7.22 (dd, J = 8.0 Hz and 1.2 Hz, 1H); 7.33 (dt, J = 7.6 Hz and 0.8 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 12.0, 35.3, 42.8, 47.7, 109.0, 117.1, 123.2, 124.5, 124.9, 127.9, 128.1, 130.3, 132.2, 142.2, 169.4.

HRMS calcd. for $C_{18}H_{17}N_2O$ [M + H]$^+$: 277.1341; found: 277.1338.

3-Butyl-1-ethyl-2-oxoindoline-3-carbonitrile (2j):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.87 (t, J = 7.2 Hz, 3H); 1.28-1.36 (m, 4H); 2.09-2.18 (m, 2H); 3.25 (s, 3H); 6.91 (d, J = 8.0 Hz, 1H); 7.17 (t, J = 8.0 Hz, 1H); 7.39-7.43 (m, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 13.7, 22.2, 25.8, 26.9, 36.9, 46.5, 109.1, 117.2, 123.7, 124.1, 125.4, 130.3, 143.0, 170.5.

HRMS calcd. for $C_{14}H_{16}N_2ONa$ $[M+H]^+$: 251.1160; found: 251.1167

3-Allyl-1-ethyl-2-oxoindoline-3-carbonitrile (2k):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 2.73 (dd, J = 13.6 Hz and 8.0 Hz, 1H); 3.00 (dd, J = 13.6 Hz and 6.4 Hz, 1H); 3.25 (s, 3H); 5.15-5.21 (m, 2H); 5.63-5.69 (m, 1H); 6.90 (d, J = 8.0 Hz, 1H); 7.16 (t, J = 7.6 Hz, 1H); 7.39-7.43 (m, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 13.6, 22.1, 26.0, 28.4, 28.6, 29.8, 31.2, 50.0, 63.4, 86.3, 112.9, 122.2, 126.5, 128.9, 138.1, 143.0, 155.2.

HRMS calcd. for $C_{13}H_{13}N_2O$ $[M + H]^+$: 213.1028; found: 213.1023

Ethyl 1,3-diethyl-2-oxoindoline-3-carboxylate (2l):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.63 (t, J = 7.6 Hz, 3H); 1.25 (J = 6.8 Hz, 3H); 1.27 (t, J = 7.6 Hz, 3H); 2.23-2.34 (m, 2H); 3.77-3.85 (m, 2H); 4.06-4.17 (m, 2H); 6.88 (d, J = 7.6 hz, 1H); 7.06 (dt, J = 7.6 Hz and 0.8 Hz, 1H); 7.24-7.26 (m, 1H); 7.32 (dt, J = 7.6 Hz and 1.2 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 7.9, 12.5, 13.8, 27.1, 34.8, 60.0, 61.7, 108.3, 122.5, 123.4, 128.1, 128.8, 143.4, 169.6, 173.9.

HRMS calcd. for $C_{15}H_{19}NO_3Na$ $[M + H]^+$: 284.1263; found: 284.1271

Ethyl 1-ethyl-3-methyl-2-oxoindoline-3-carboxylate (2m):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.14 (t, J = 7.2 Hz, 3H); 1.28 (t, J = 7.2 Hz, 3H); 1.65 (s, 3H); 3.73-3.88 (m, 2H); 4.04-4.17 (m, 2H); 6.90 (d, J = 8.0 Hz, 1H); 7.05 (dt, J = 7.6 Hz and 0.8 Hz, 1H); 7.26 (d, J = 6.8 Hz, 1H); 7.32 (dt, J = 7.6 Hz and 0.8 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 14.4, 54.3, 61.1, 63.7, 85.9, 111.6, 122.9, 125.2, 128.0, 128.8, 129.2, 131.6, 134.4, 140.9, 143.8, 160.2, 164.9.

HRMS calcd. for $C_{14}H_{17}NO_3Na$ $[M + H]^+$: 270.1106; found: 270.1113

Ethyl 3-benzyl-1-methyl-2-oxoindoline-3-carboxylate (2n):

White solid, mp: 124-126 ^oC

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.19 (t, J = 7.2 Hz, 3H); 2.94 (s, 3H); 3.54 (s, 2H); 4.15-4.22 (m, 2H); 6.56 (d, J = 7.6 Hz, 1H); 6.84 (d, J = 6.8 Hz, 2H); 6.98-7.08 (m, 4H); 7.21 (t, J = 7.6 Hz, 1H); 7.33 (d, J = 7.6 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 14.0, 26.1, 40.0, 60.9, 62.1, 108.1, 122.5, 123.8, 126.7, 127.4, 127.5, 129.0, 129.9, 134.4, 144.0, 169.3, 173.5.

HRMS calcd. for $C_{19}H_{19}NO_3Na$ $[M + Na]^+$: 332.1263; found: 332.1248

Ethyl 3-ethyl-1-methyl-2-oxoindoline-3-carboxylate (2o):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 0.65 (t, J = 7.6 Hz, 3H); 1.16 (t, J = 7.2 Hz, 3H); 2.22-2.35 (m, 2H); 3.25 (s, 3H); 4.08-4.17 (m, 2H); 6.87 (d, J = 8.0 Hz, 1H); 7.08 (t, J = 7.2 Hz, 1H); 7.25 (d, J = 7.2 Hz, 1H); 7.33 (dt, J = 8.0 Hz and 0.8 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 8.0, 13.9, 26.4, 27.4, 60.0, 61.8, 108.2, 122.8, 123.3, 127.9, 128.9, 144.3, 169.5, 174.2.

HRMS calcd. for $C_{14}H_{17}NO_3Na$ $[M + Na]^+$: 270.1106; found: 270.1113

Ethyl 1,3-dimethyl-2-oxoindoline-3-carboxylate[3, 4] (2p):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.15 (t, J = 7.2 Hz, 3H); 1.66 (t, J = 8.4 Hz, 3H); 3.26 (s, 3H); 4.07-4.17 (m, 2H); 6.88 (d, J = 7.6 Hz, 1H); 7.07 (dt, J = 7.6 Hz and 0.8 Hz, 1H); 7.26 (d, J = 7.2 Hz, 1H); 7.33 (dt, J = 8.0 Hz and 1.2 Hz. 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 13.9, 20.1, 26.5, 55.0, 61.9, 108.4, 122.9, 129.0, 130.2, 143.6, 169.7, 175.2.

HRMS calcd. for $C_{13}H_{15}NO_3Na$ $[M + Na]^+$: 256.0950; found: 256.0954

1-Methyl-2-oxo-3-(pent-4-enyl)indoline-3-carbonitrile (2q):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.44-1.52 (m, 2H); 2.05 (q, J = 7.2 Hz, 2H); 2.10-2.20 (m, 2H); 3.25 (s, 3H); 4.95-5.00 (m, 2H); 5.69 (dd, J = 17.2 Hz and 10.4 Hz); 6.90 (d, J = 8.0 Hz, 1H); 7.17 (t, J = 8.0 Hz, 1H); 7.41 (t, J = 6.8 Hz, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 23.0, 26.9, 33.0, 36.5, 46.4, 109.1, 115.8, 117.1, 123.7, 124.1, 125.3, 130.3, 137.1, 143.0, 170.4.

HRMS calcd. for $C_{15}H_{17}N_2O$ $[M + H]^+$: 241.1341; found: 241.1346

Diethyl 1,3-dimethyl-2-oxoindolin-3-ylphosphonate (2r):

Colourless gummy

^{1}H-NMR (400 MHz, $CDCl_3$, ppm): δ 1.08 (t, J = 7.2 Hz, 3H); 1.34 (t, J = 7.2 Hz, 3H); 1.72 (d, J = 17.2 Hz, 3H); 3.24 (s, 3H); 3.83-3.90 (m, 1H); 3.94-4.00 (m, 1H); 4.16-4.23 (m, 2H); 6.85 (d, J = 8.0 Hz, 1H); 7.10 (t, J = 7.6 Hz, 1H); 7.30-7.34 (m, 1H); 7.48 (d, J = 7.6 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$, ppm): δ 16.1 (d, J = 6.0 Hz), 16.4 (d, J = 6.0 Hz), 18.5 (d, J = 6.0 Hz), 26.6, 49.3 (d, J = 145.0 Hz), 63.2 (d, J = 7.0 Hz); 64.0 (d, J = 6.0 Hz), 108.0, 122.7 (d, J = 3.0 Hz), 125.2 (d, J = 4.0 Hz), 127.8 (d, J = 6.0 Hz), 128.8 (d, J = 4.0 Hz), 143.8, 174.8.

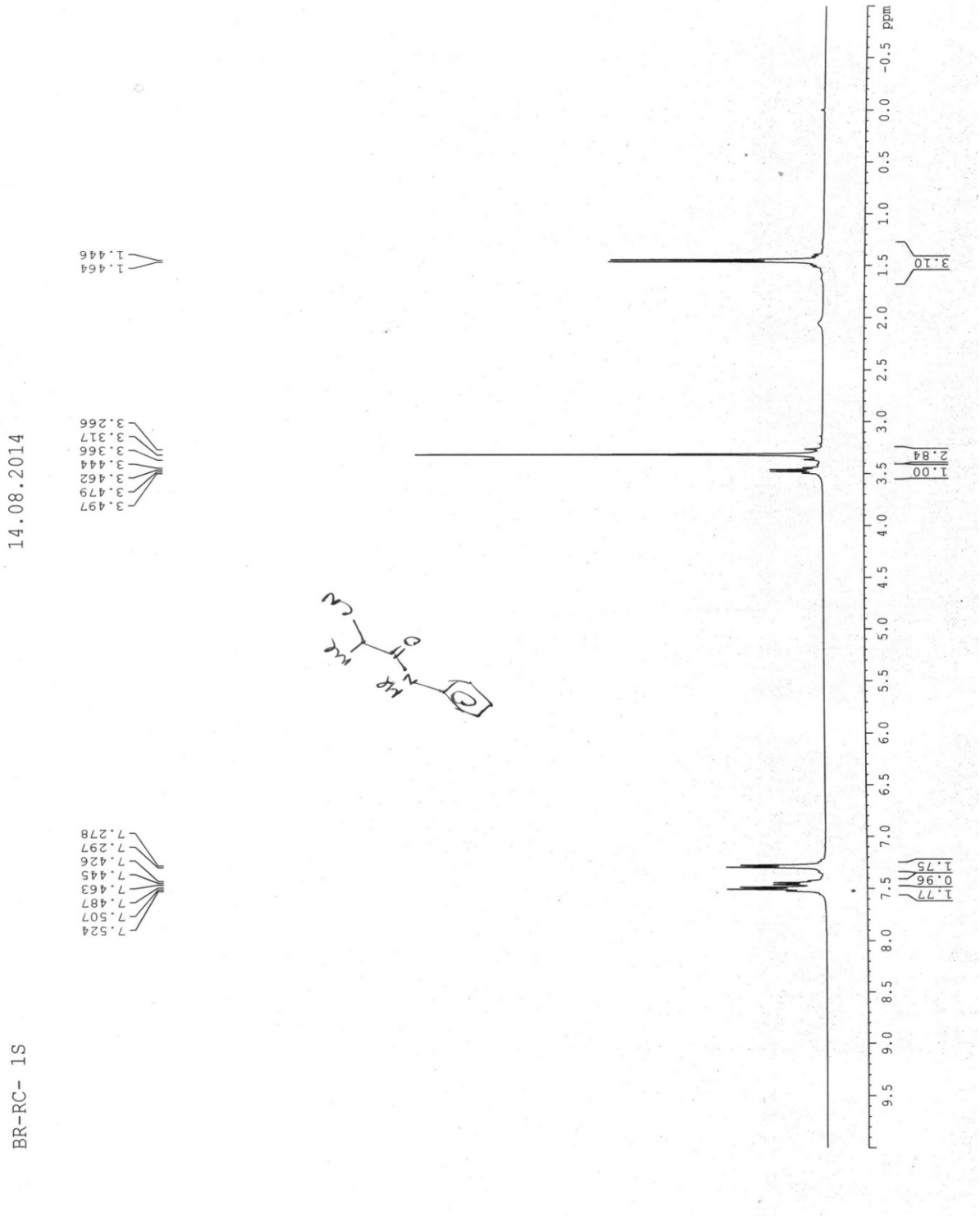
HRMS calcd. for $C_{14}H_{21}NO_4P$ $[M + H]^+$: 298.1208; found: 298.1214.

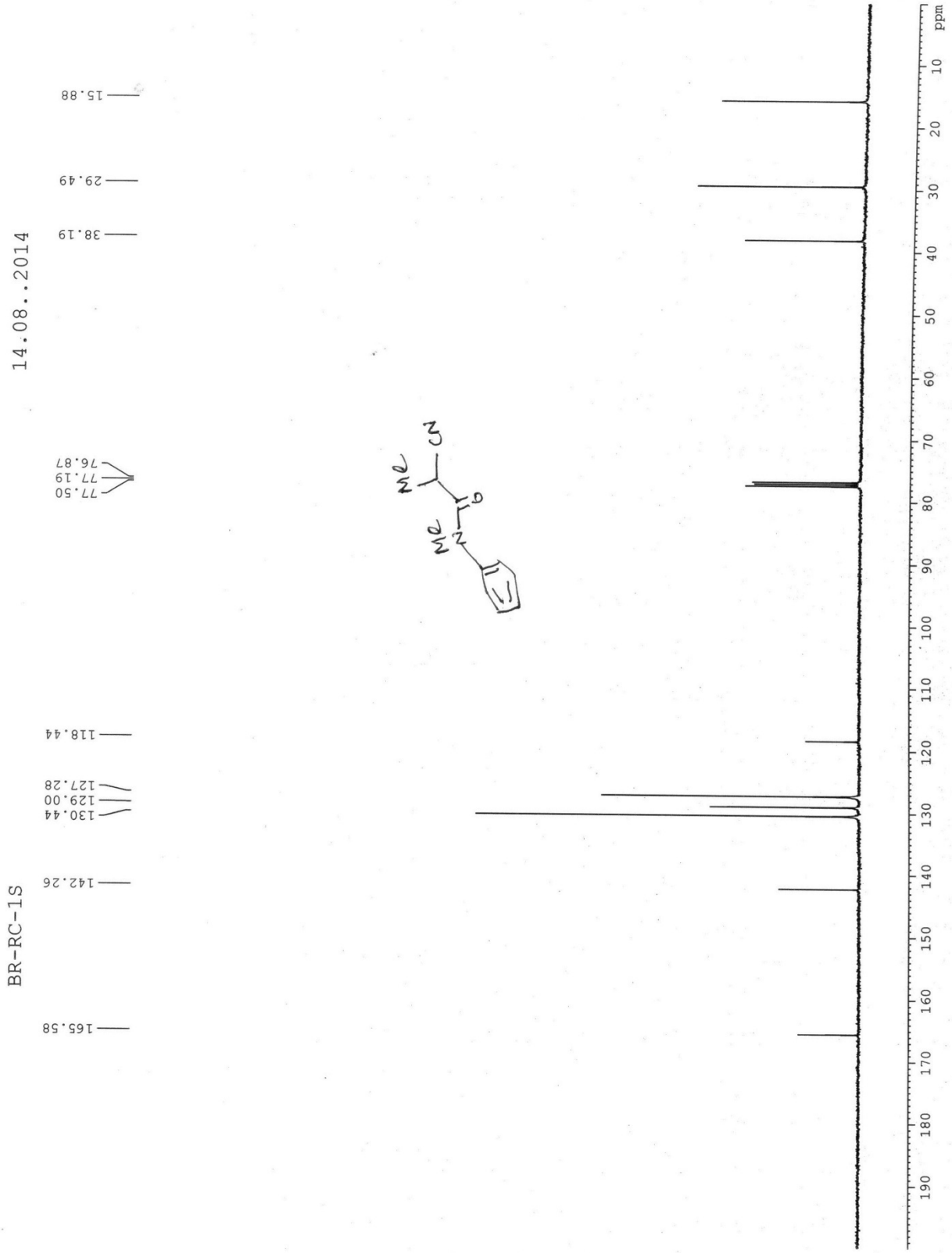
Reference:

1. S. Ghosh, S. De, B. N. Kakde, S. Bhunia, A. Adhikary and A. Bisai, *Org. Lett.,* 2012, ***14***, 5864-5867.
2. Y. Kobayashi and T. Harayama *Org. Lett.*, 2009, ***11***, 1603-1606.
3. S. Bhunia , S. Ghosh , D. Dey , and A. Bisai *Org. Lett.*, 2013, ***15***, 2426-2429.
4. X. Ju, Y. Liang, P. Jia, W. Lia and W. Yu *Org. Biomol. Chem.,* 2012, ***10***, 498-501.

Copies of NMR (^{1}H & ^{13}C) spectra of starting material:

2-Cyano-N-methyl-N-phenylpropanamide (1a):

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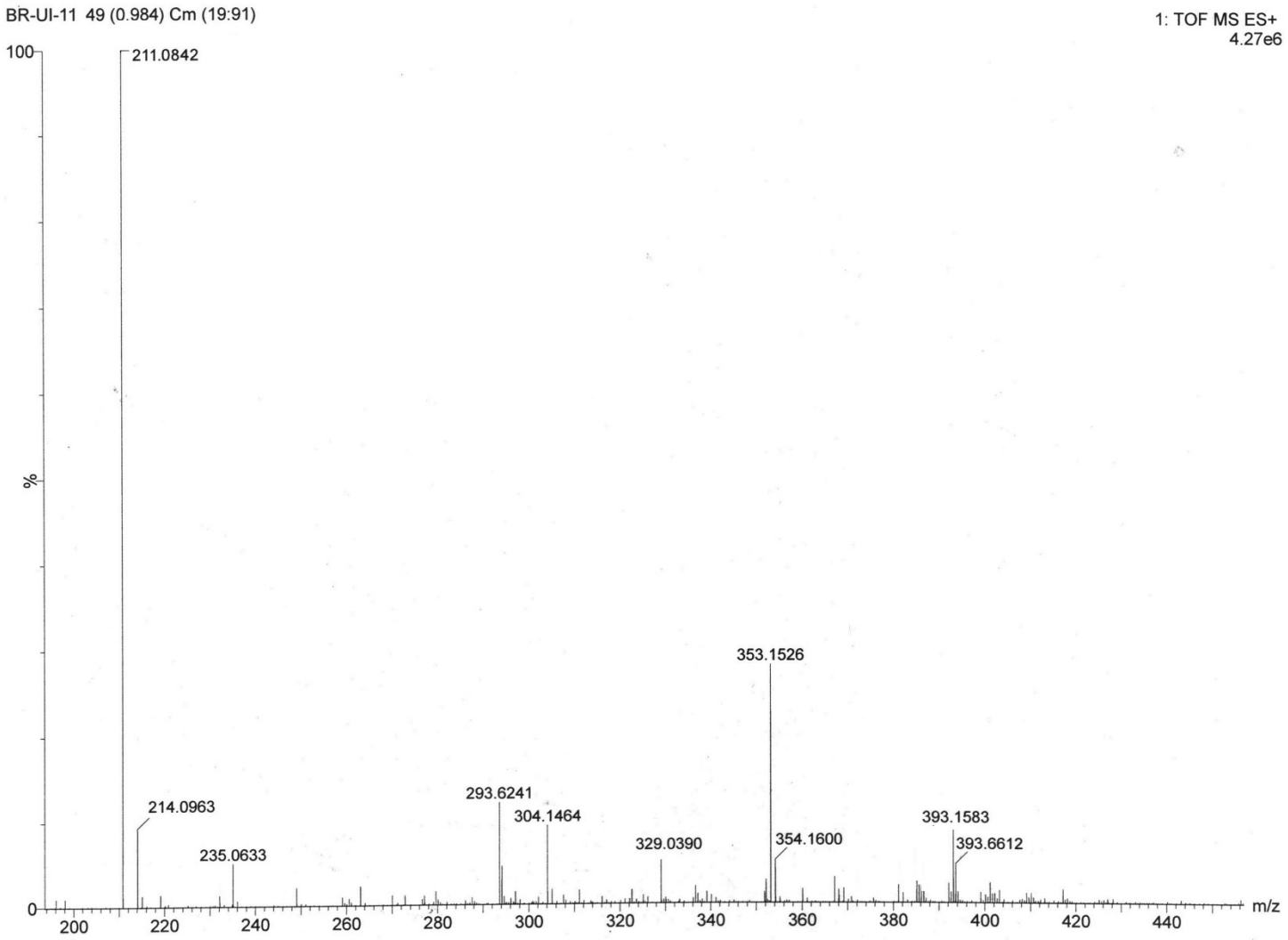
29.49
15.88
ppm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%
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293.6241
304.1464
329.0390
353.1526
354.1600
393.1583
393.6612
200
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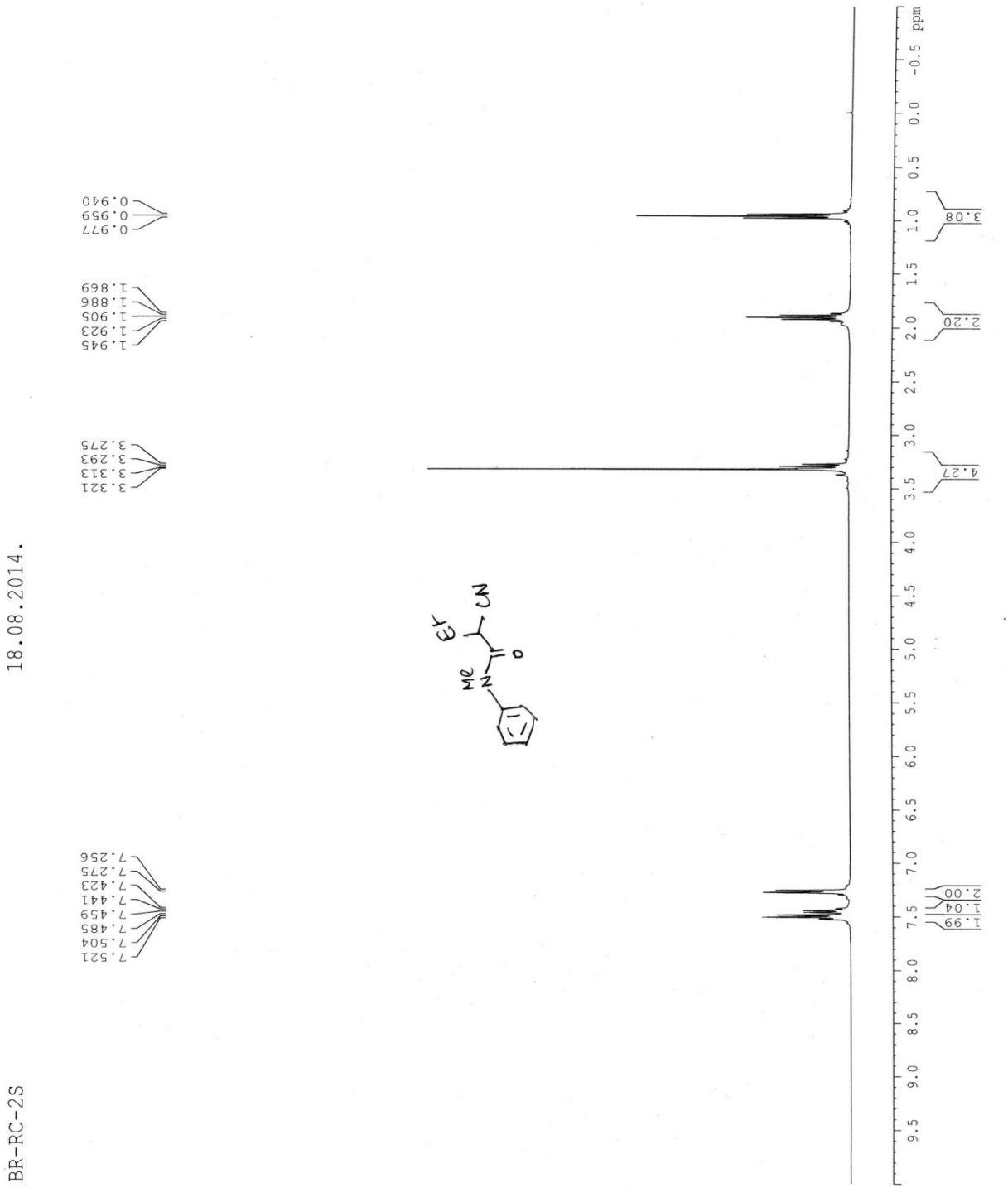
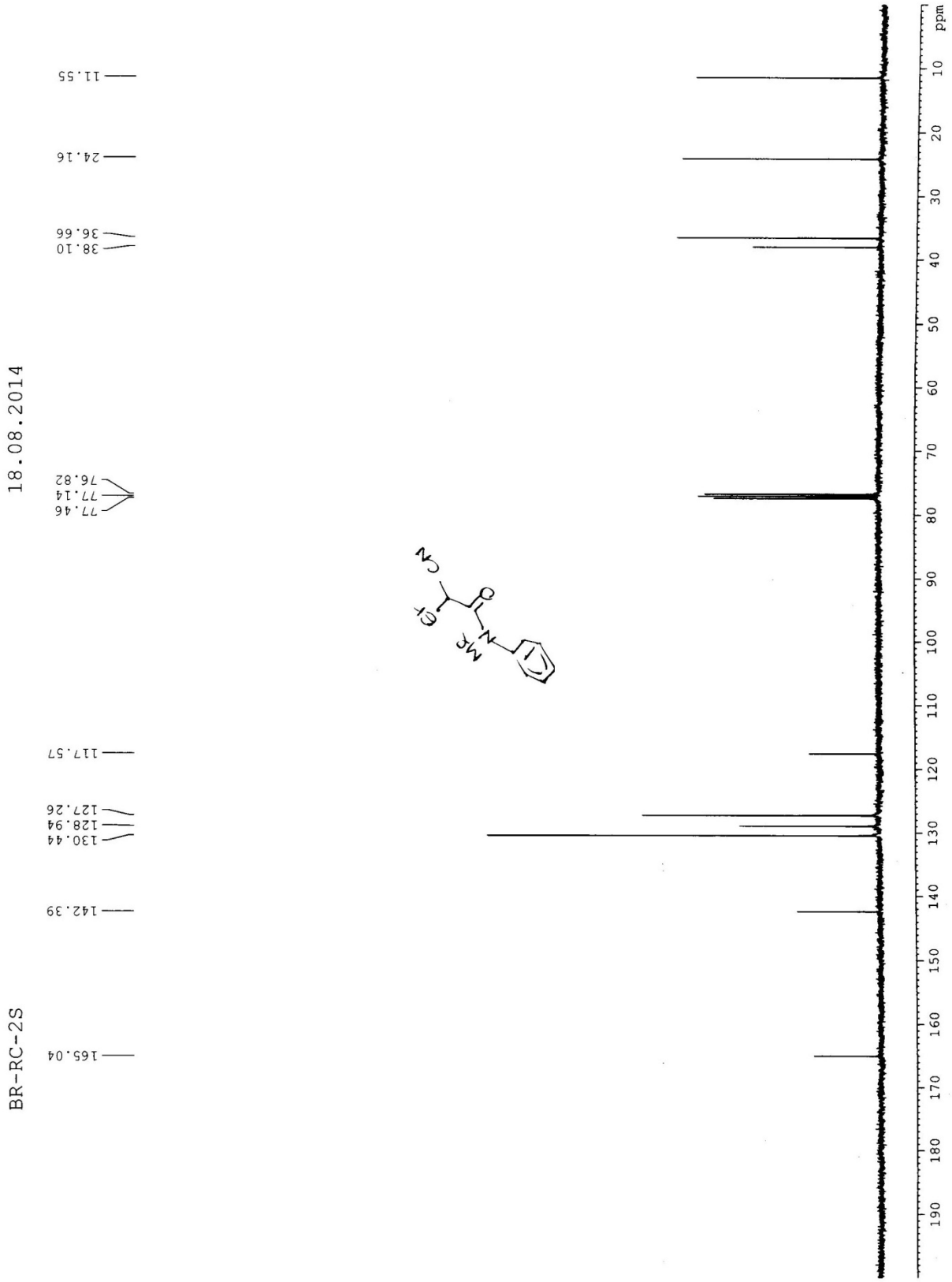
300
320
340
360
380
400
420
440
m/z

2-Cyano-N-methyl-N-phenylbutanamide (1b):

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11.55
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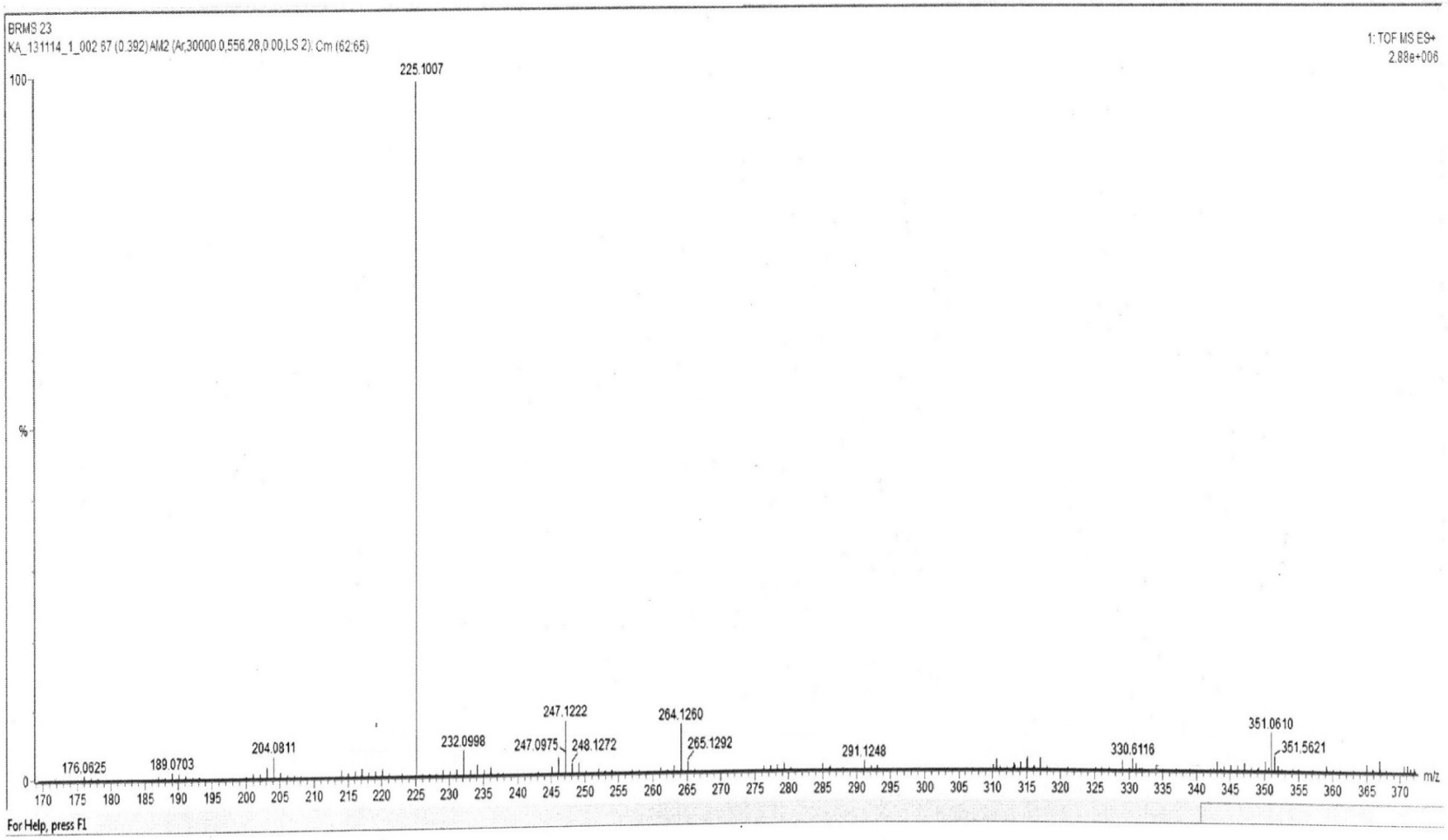
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%
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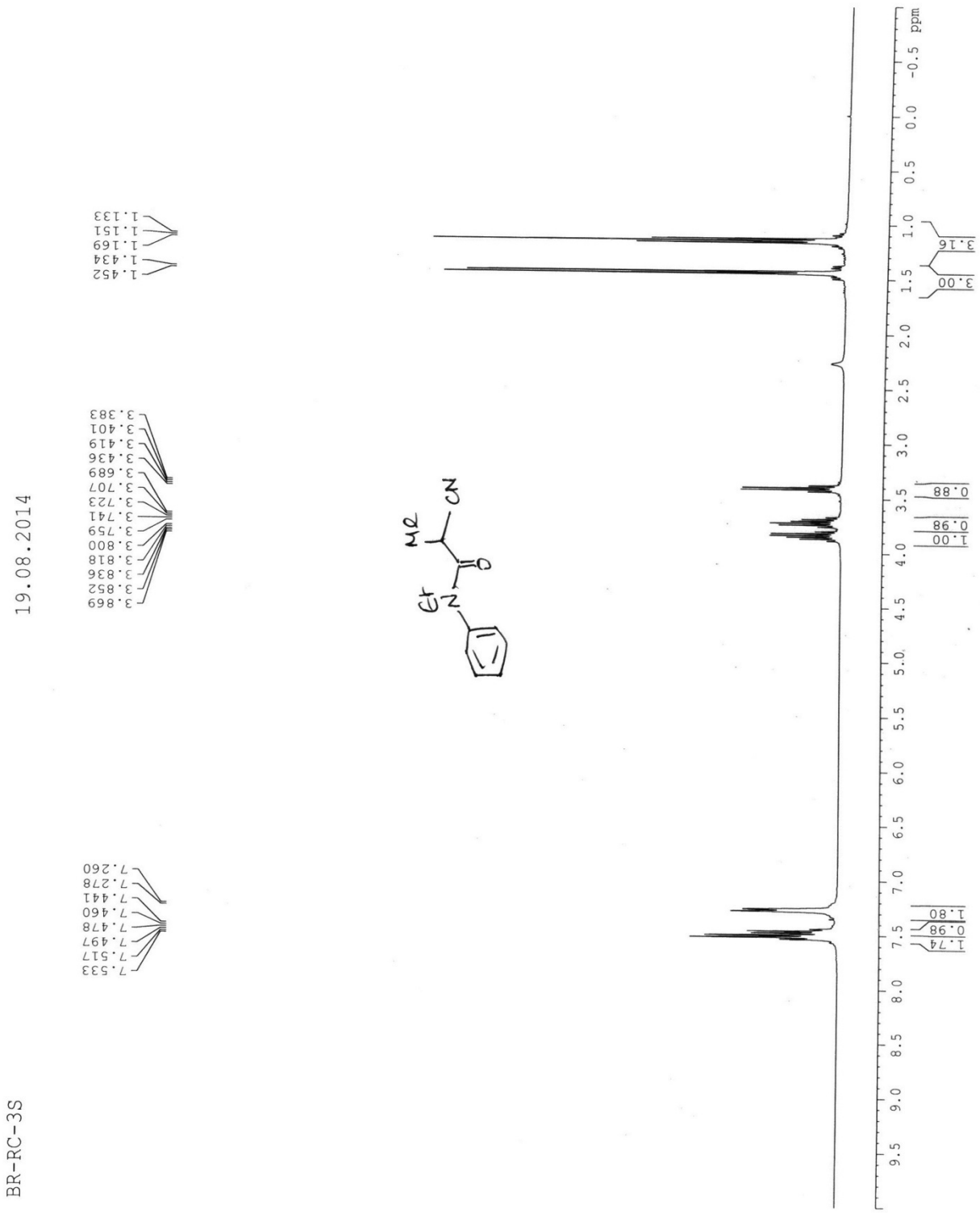
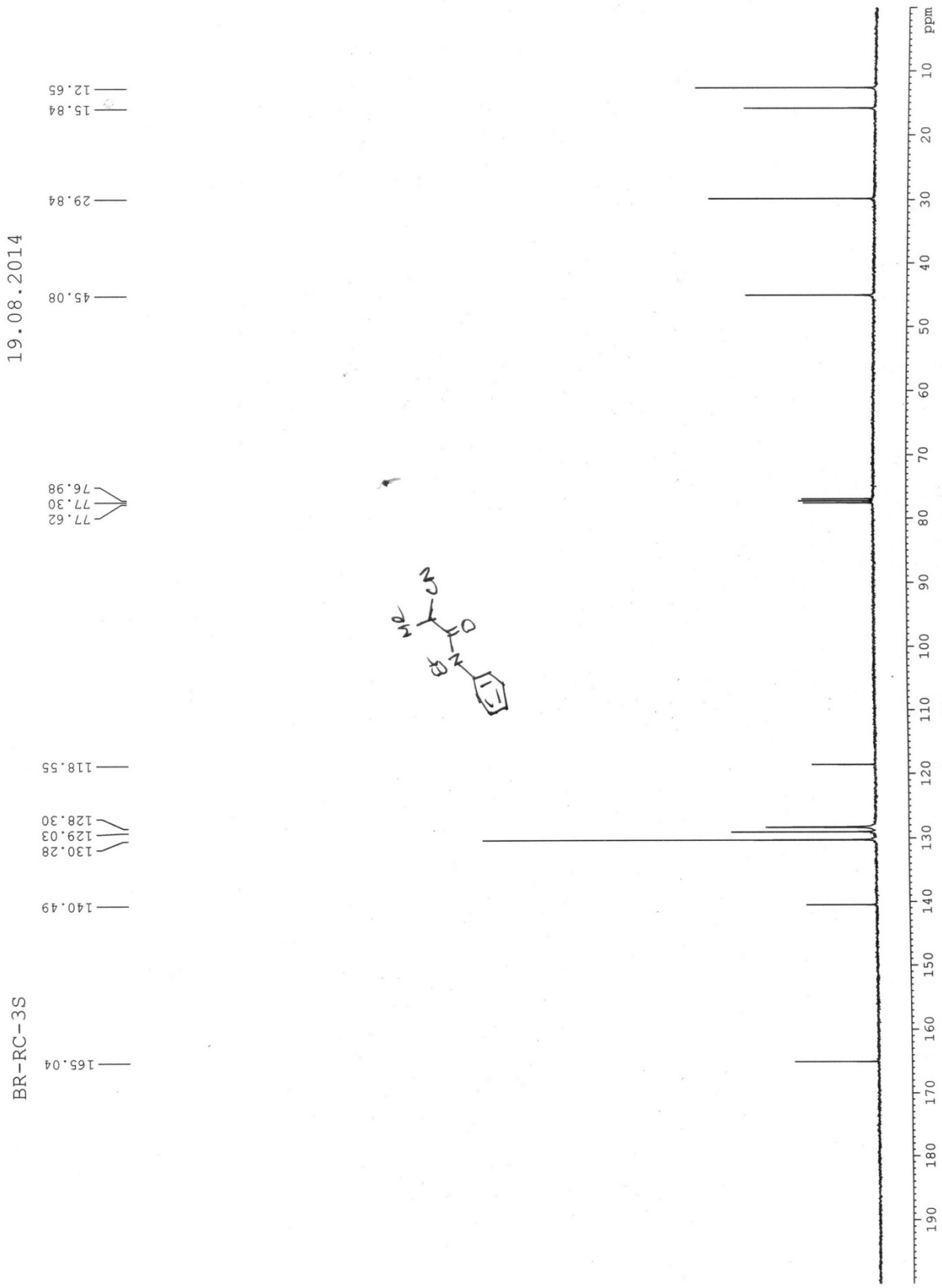
264.1260
351.0610
204.0811
232.0998
247.0975
248.1272
265.1292
291.1248
330.6116
351.5621
176.0625
189.0703
170 175 180 185 190 195 200 205 210 215 220 225 230 235 240 245 250 255 260 265 270 275 280 285 290 295 300 305 310 315 320 325 330 335 340 345 350 355 360 365 370
m/z
For Help, press F1

2-Cyano-N-ethyl-N-phenylpropanamide (1c):

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45.08
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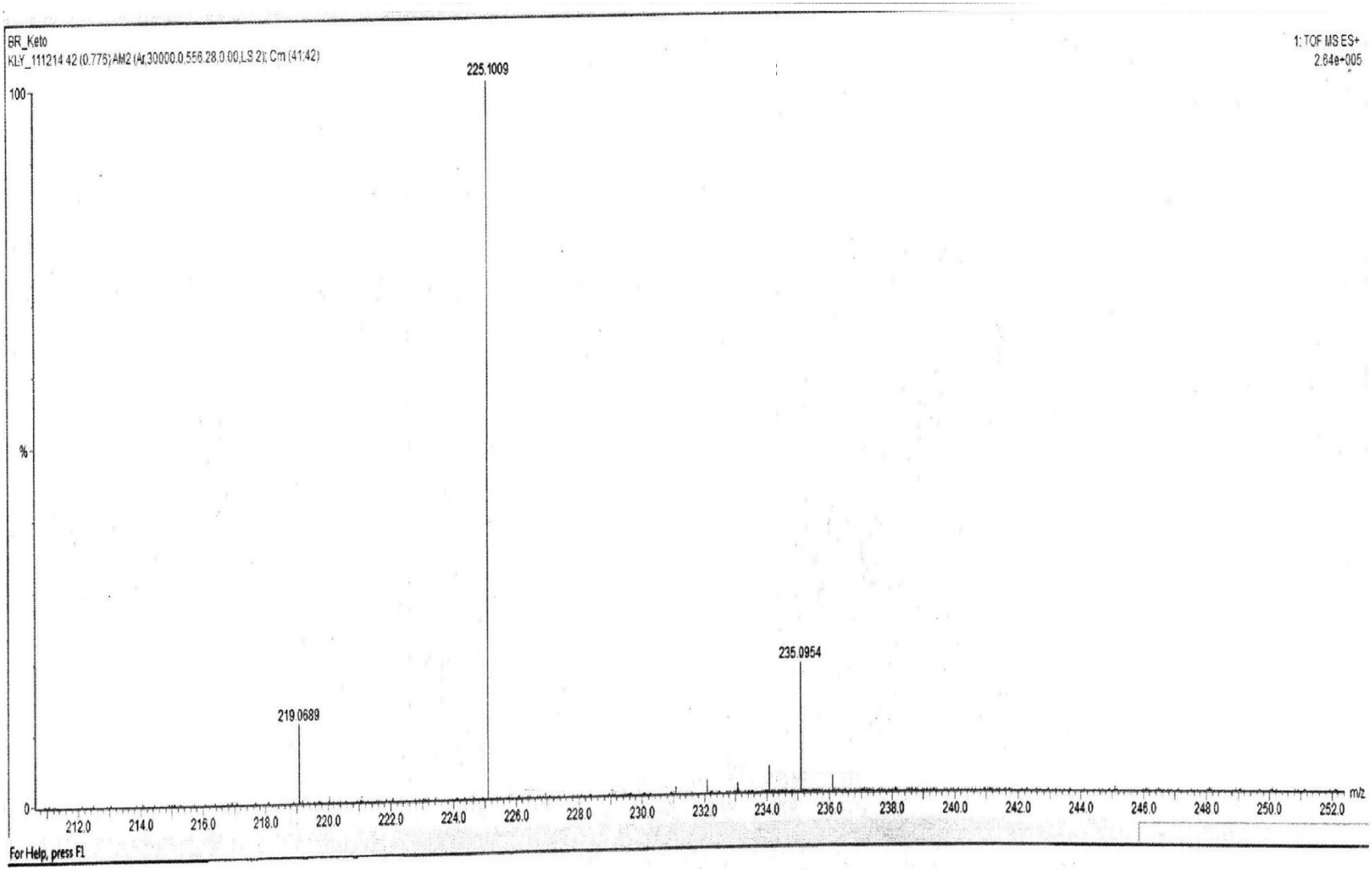
15.84
12.65
ppm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219.0689
100
%
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214.0
216.0
218.0
220.0
222.0
224.0
226.0
228.0
230.0
232.0
234.0
236.0
238.0
240.0
242.0
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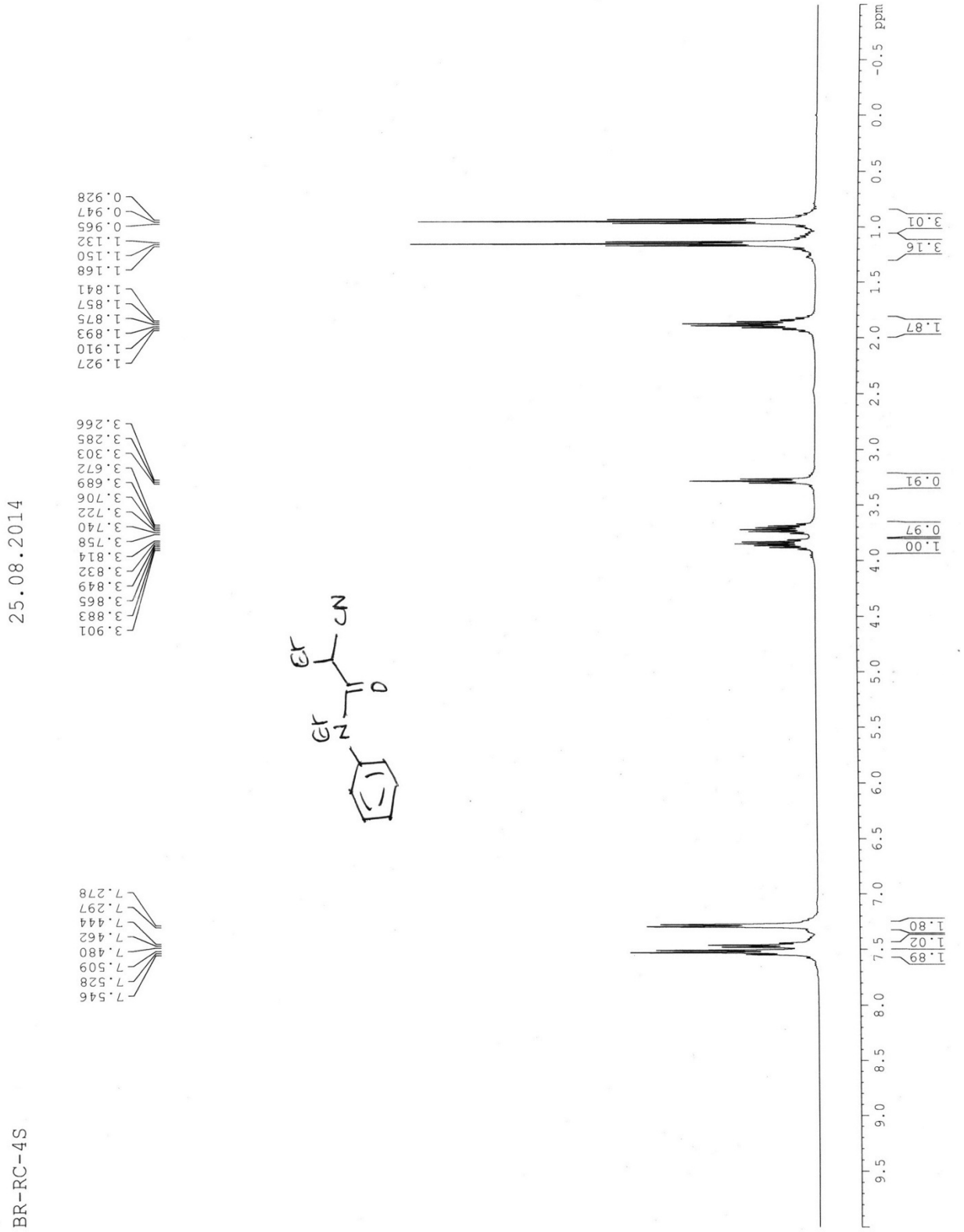
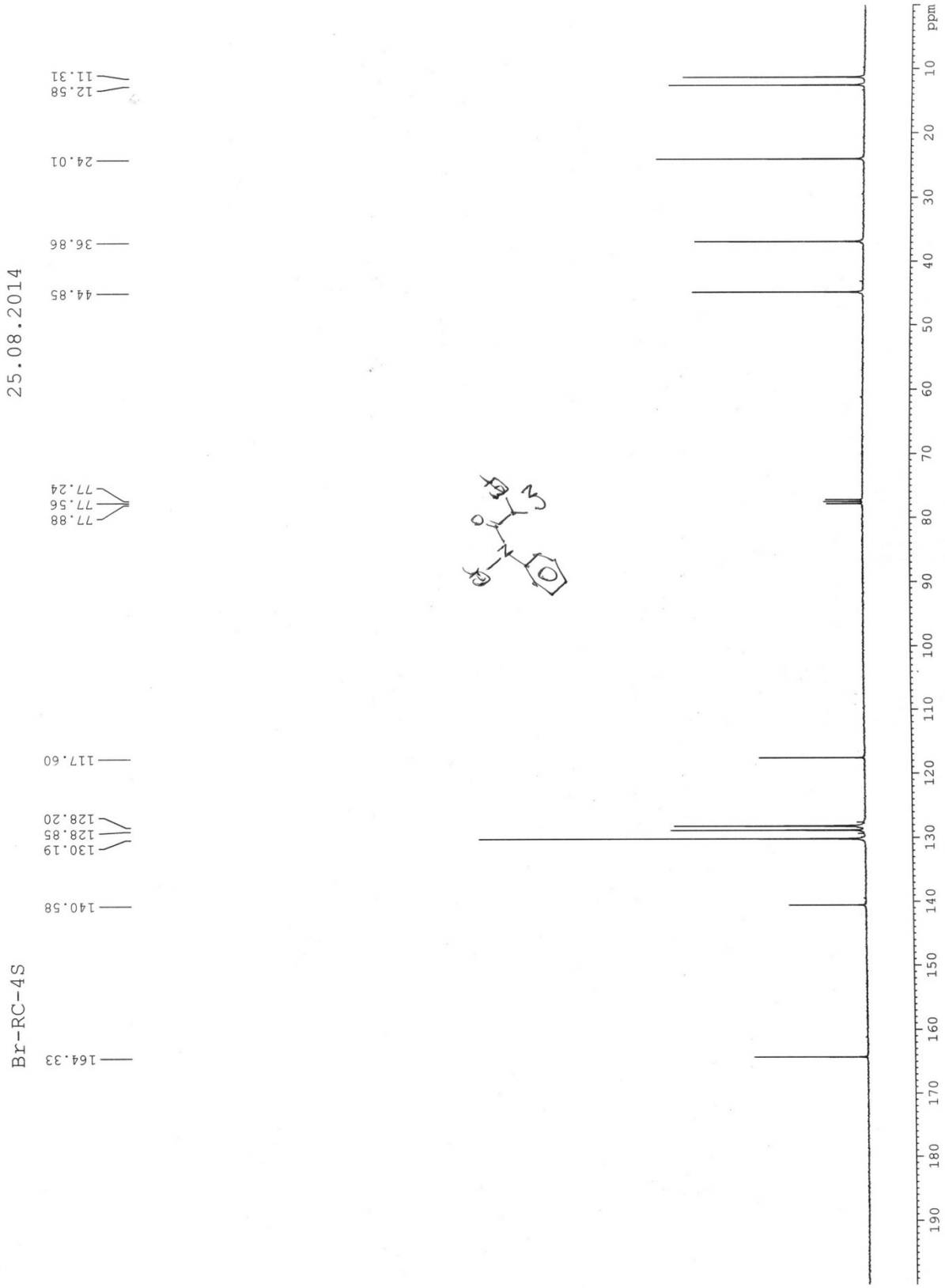
248.0
250.0
252.0
m/z
For Help, press F1

2-Cyano-N-ethyl-N-phenylbutanamide (1d):

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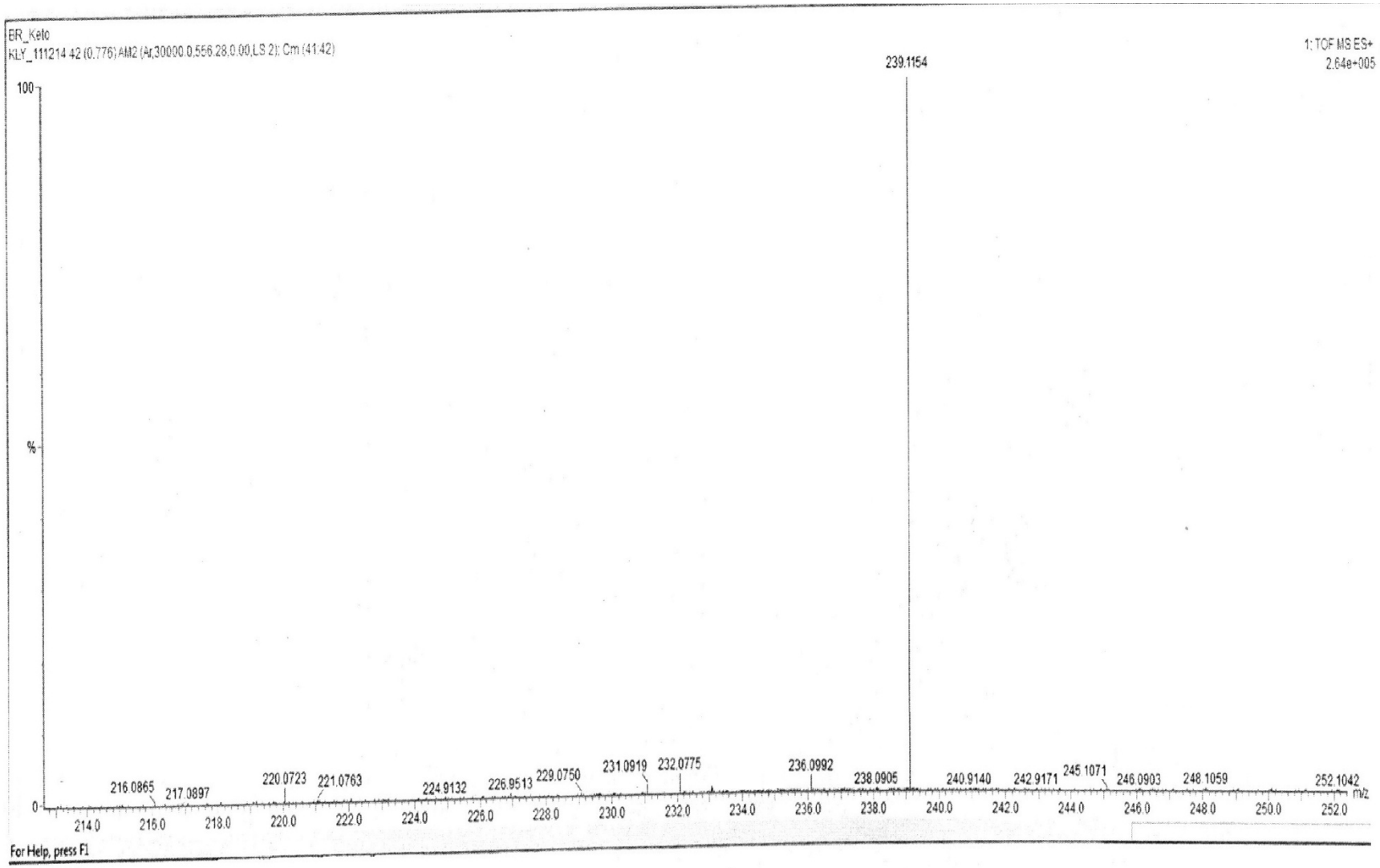
36.86
24.01
12.58
11.31
ppm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100
%
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229.0750
231.0919
232.0775
236.0992
238.0905
240.9140
242.9171
245.1071
246.0903
248.1059
252.1042
214.0
216.0
218.0
220.0
222.0
224.0
226.0
228.0
230.0
232.0
234.0
236.0
238.0
240.0
242.0
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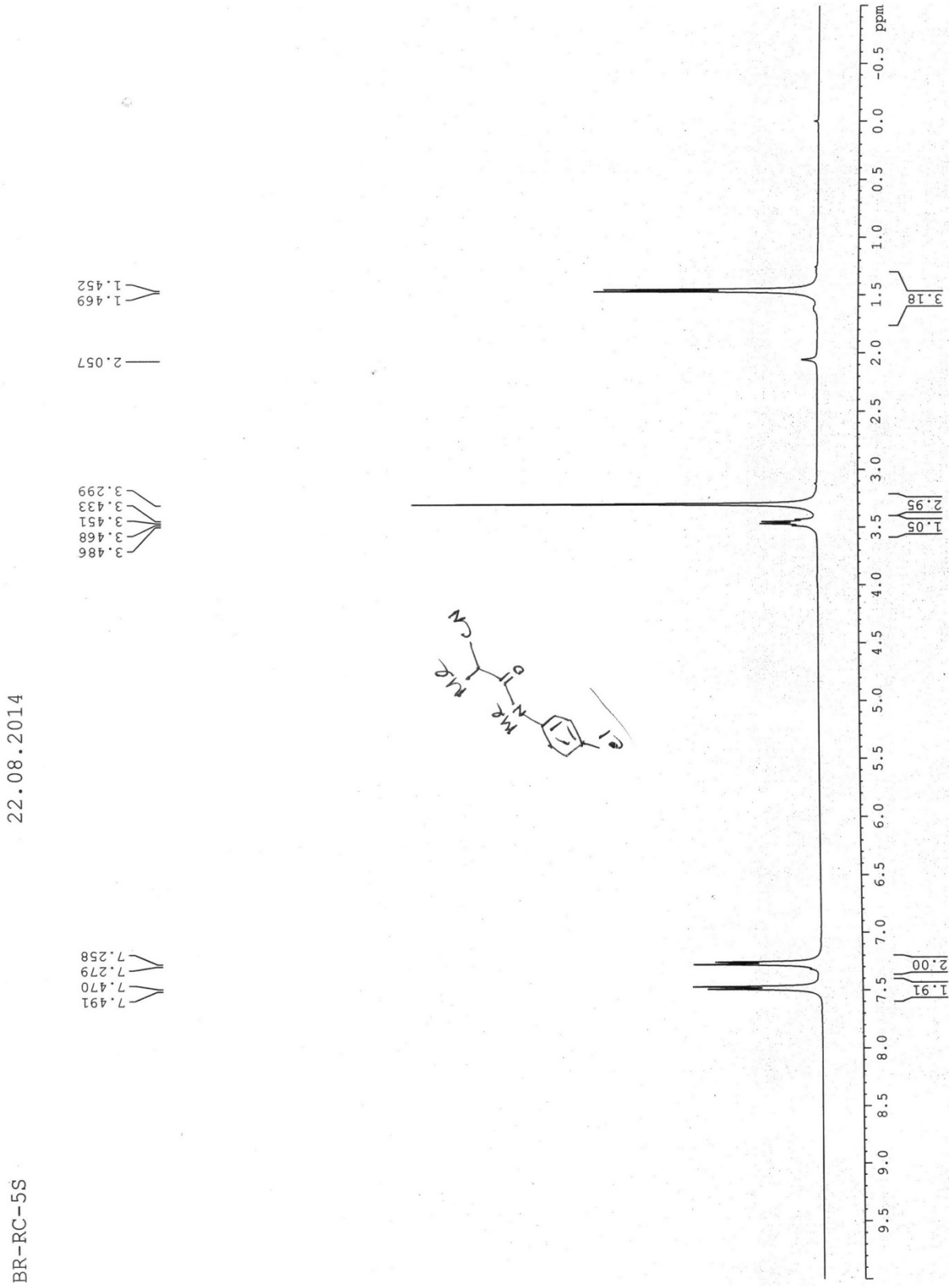
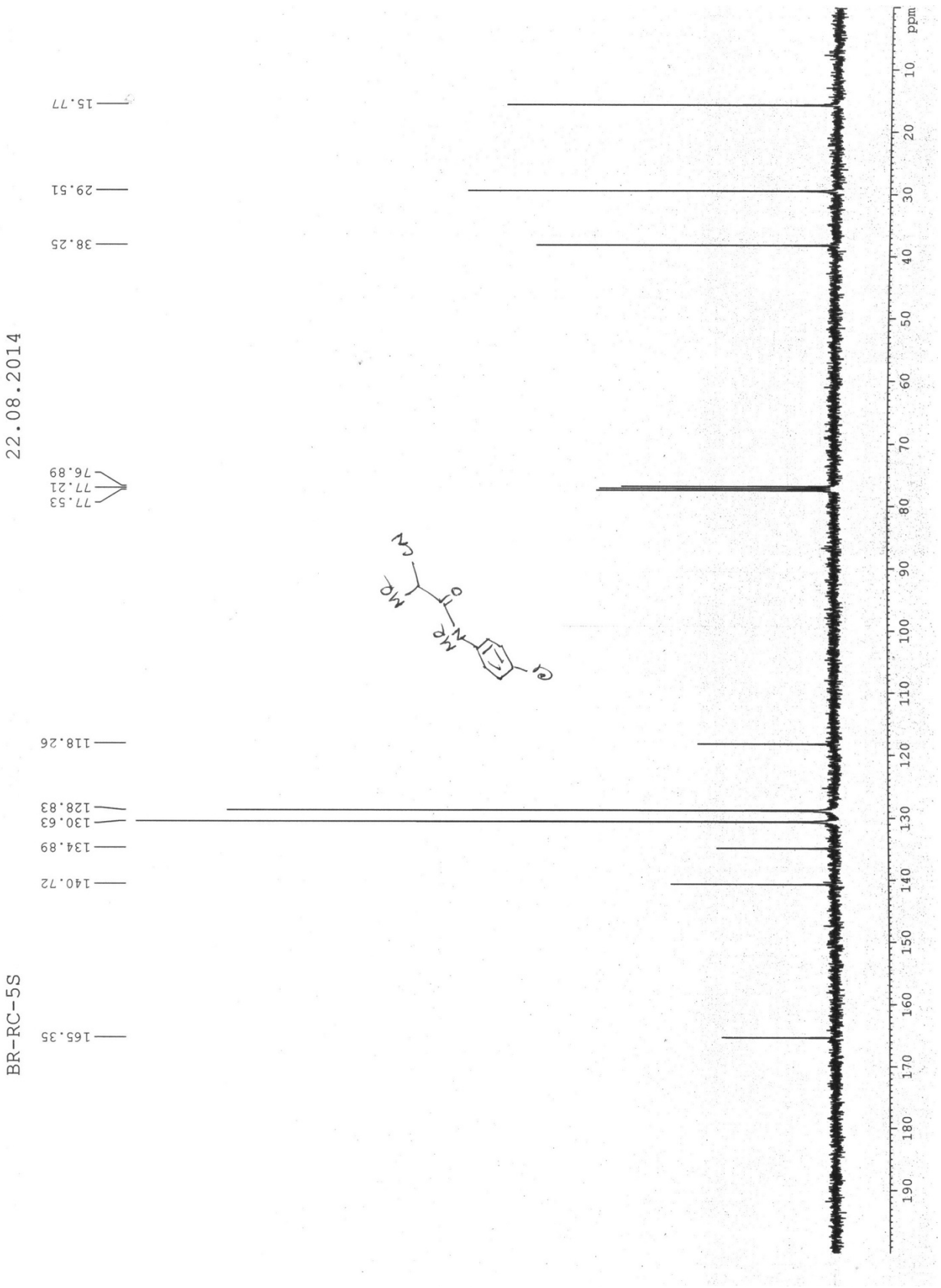
m/z
For Help, press F1

N-(4-chlorophenyl)-2-cyano-N-methylpropanamide (1e):

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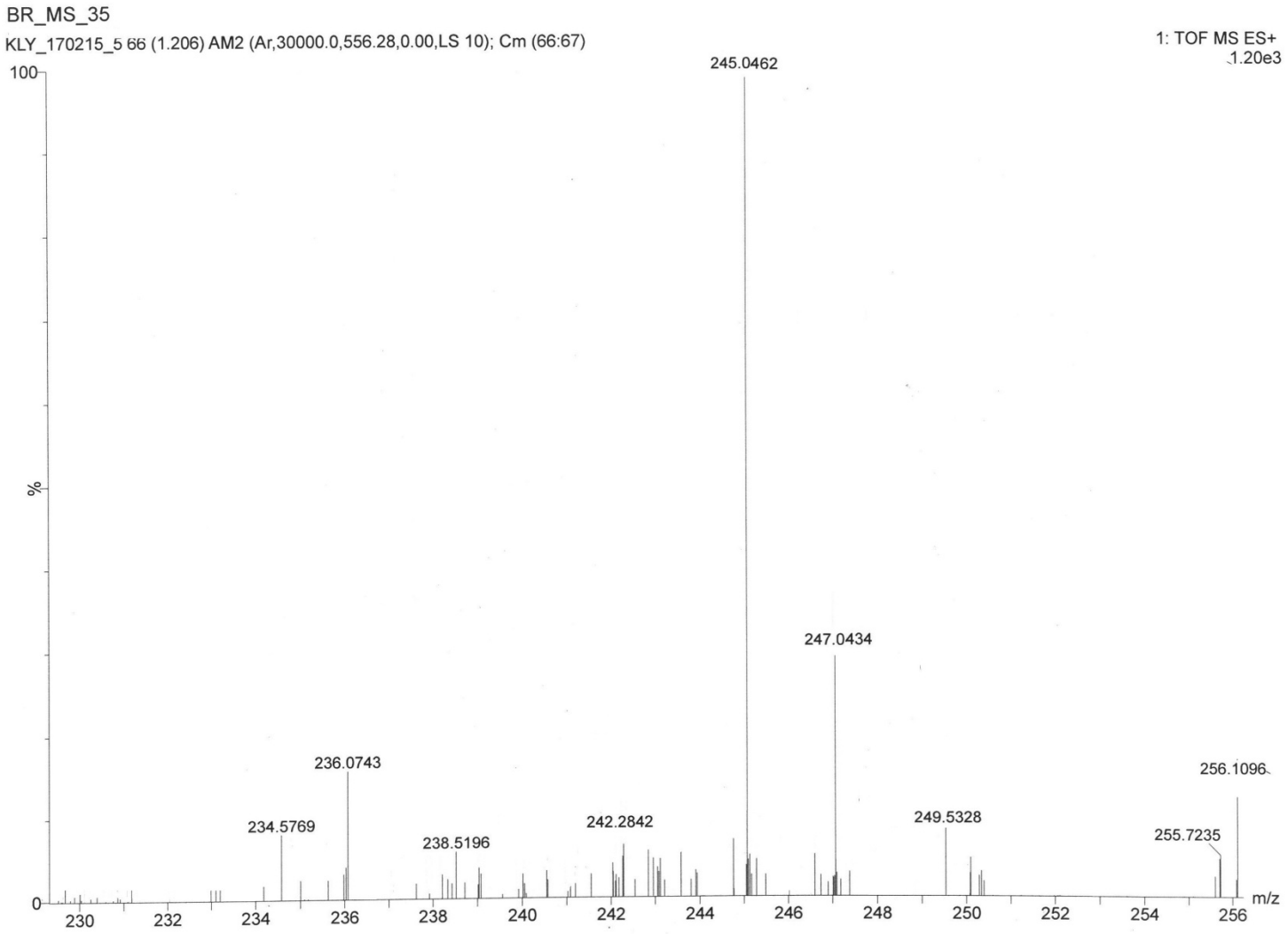
165.35
ppm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%
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242.2842
249.5328
255.7235
256.1096
230
232
234
236
238
240
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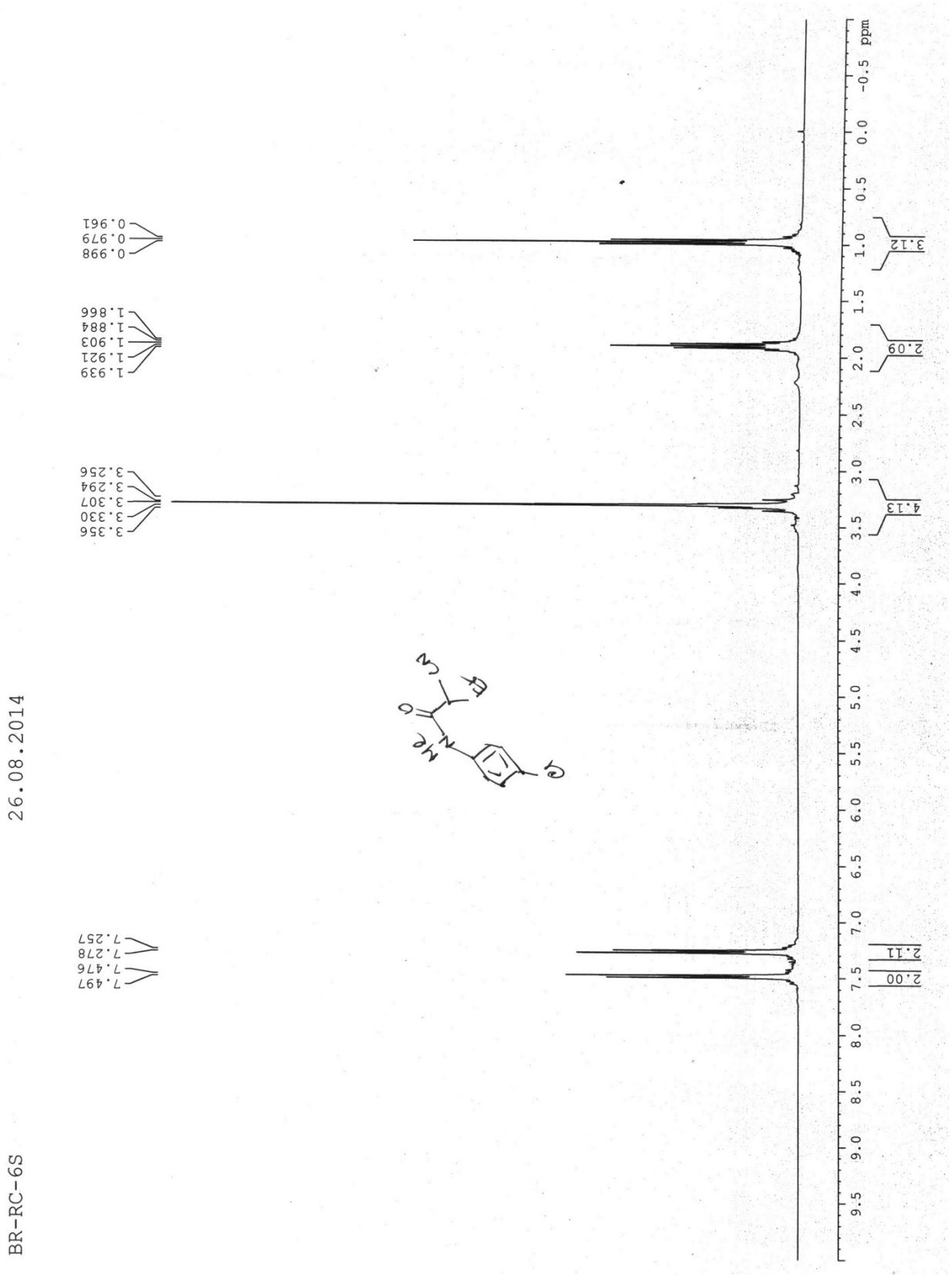
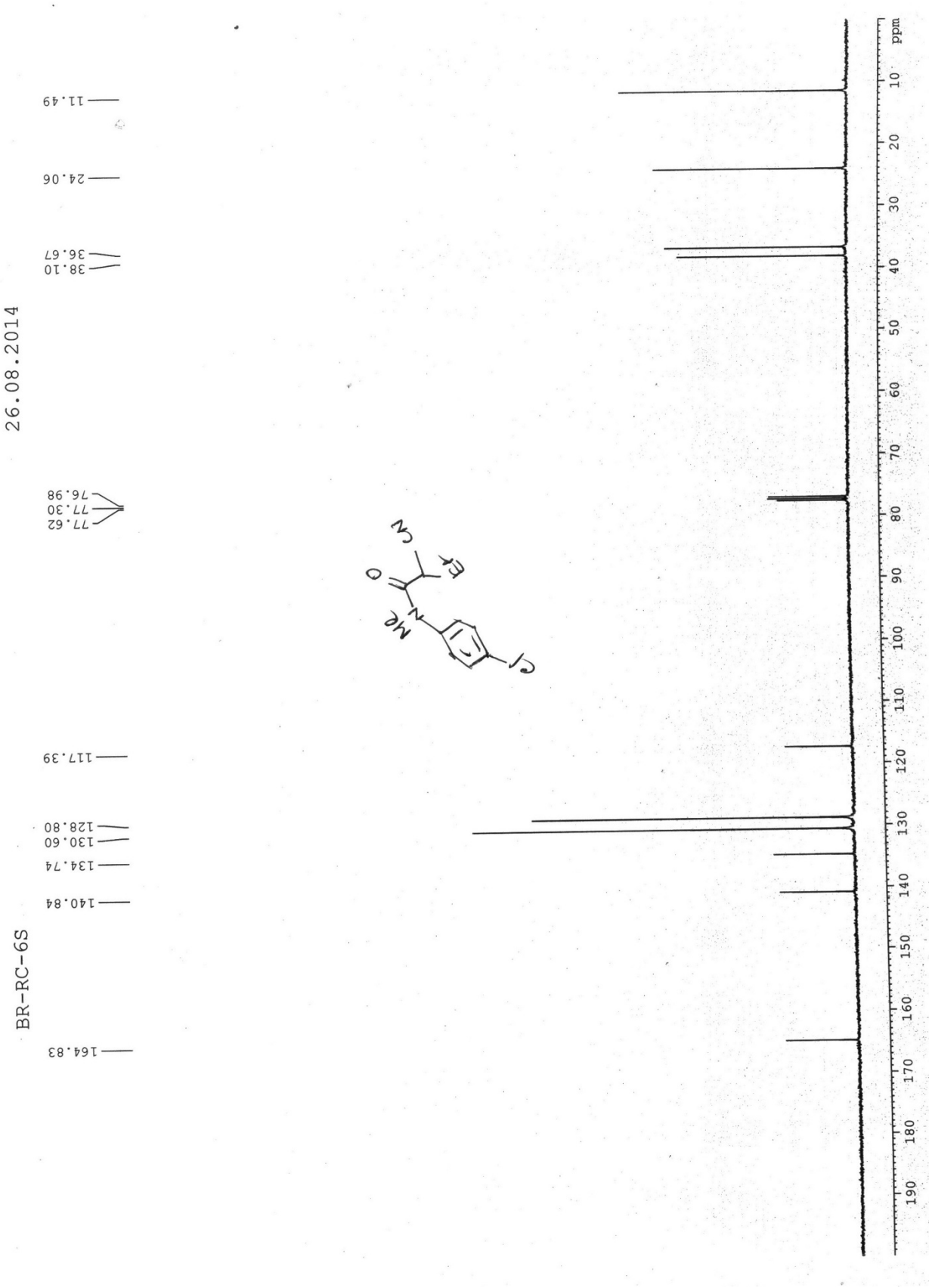
246
248
250
252
254
256
m/z

N-(4-chlorophenyl)-2-cyano-N-methylbutanamide (1f):

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26.08.2014
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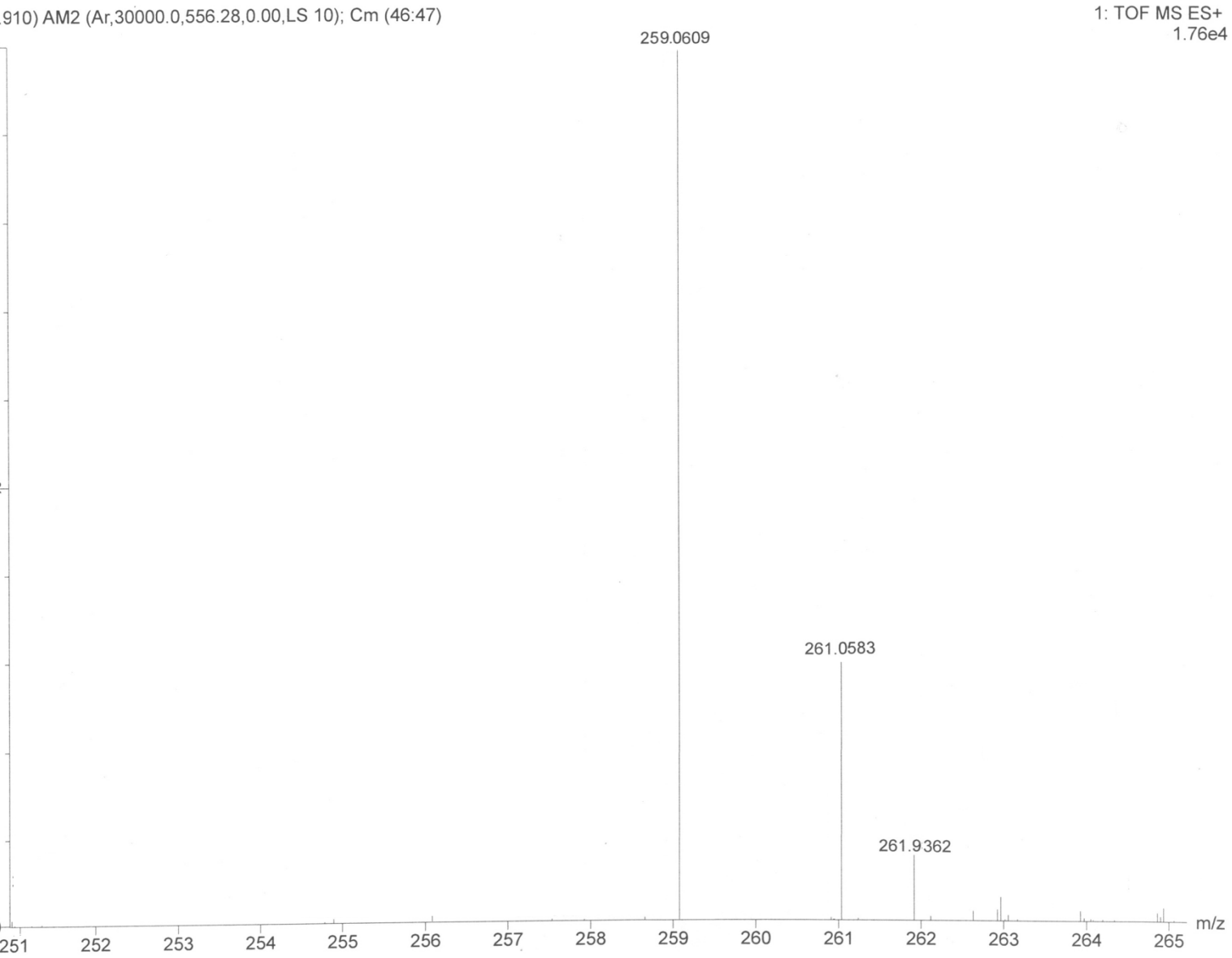
36.67
24.06
11.49
ppm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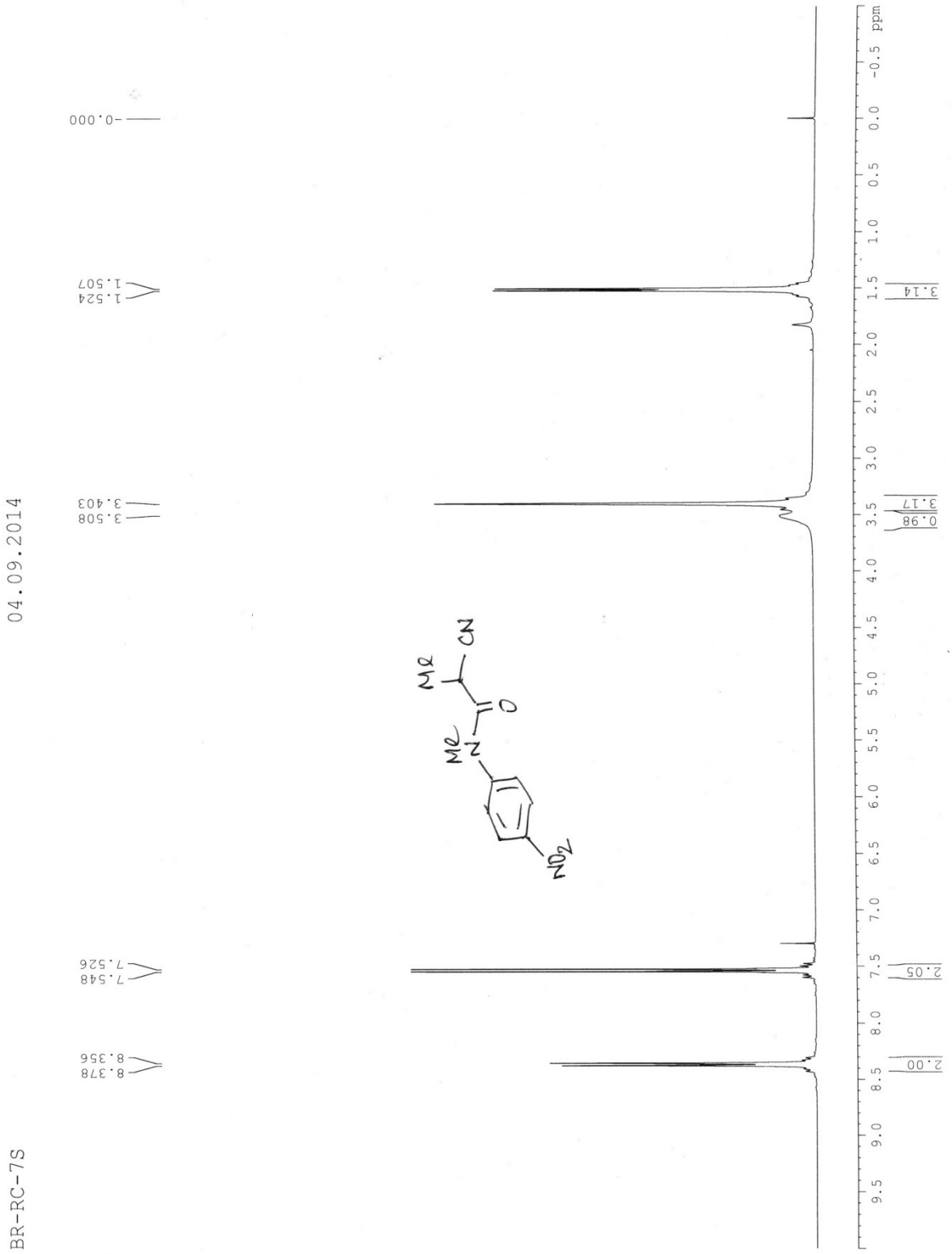
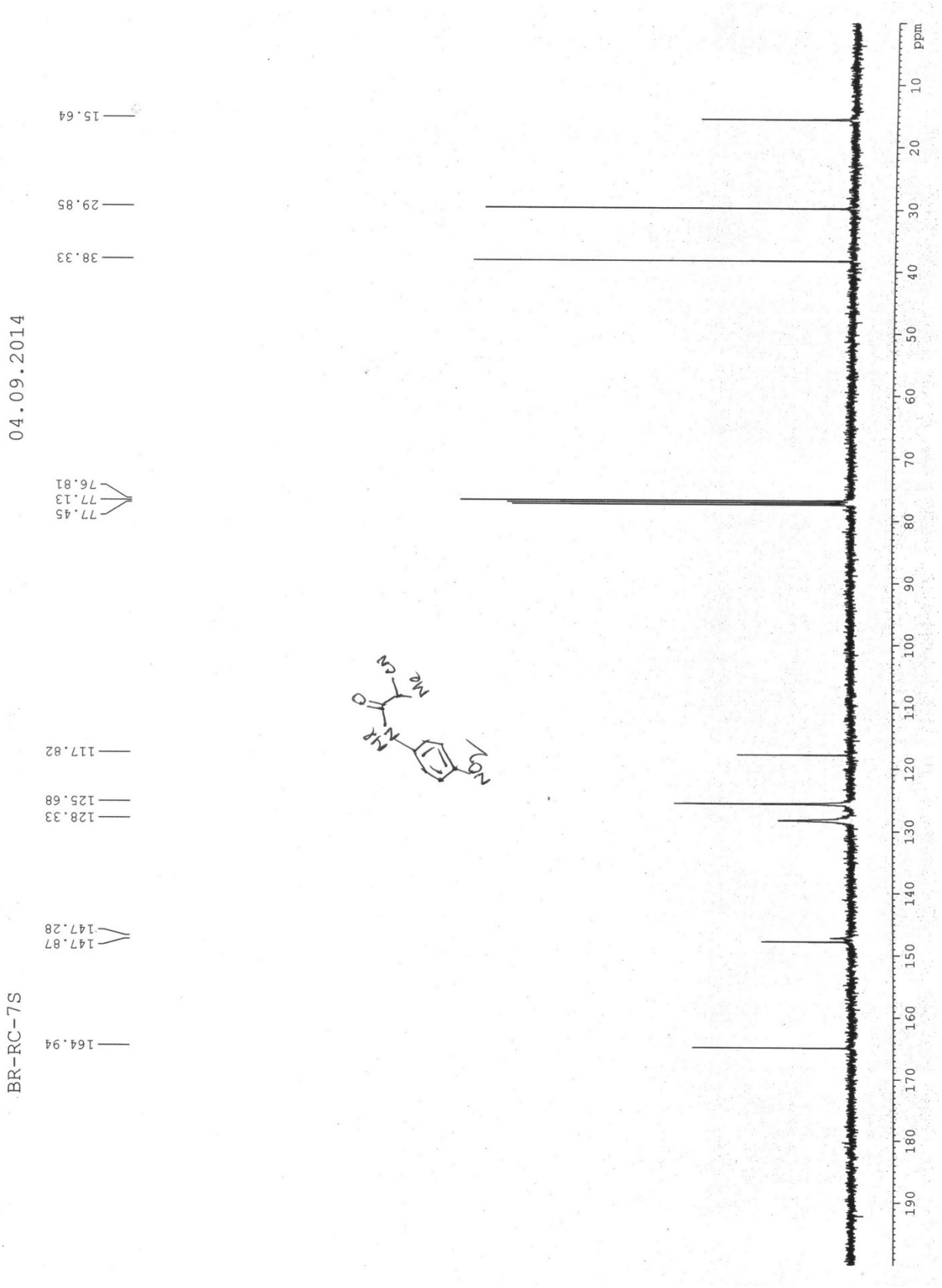
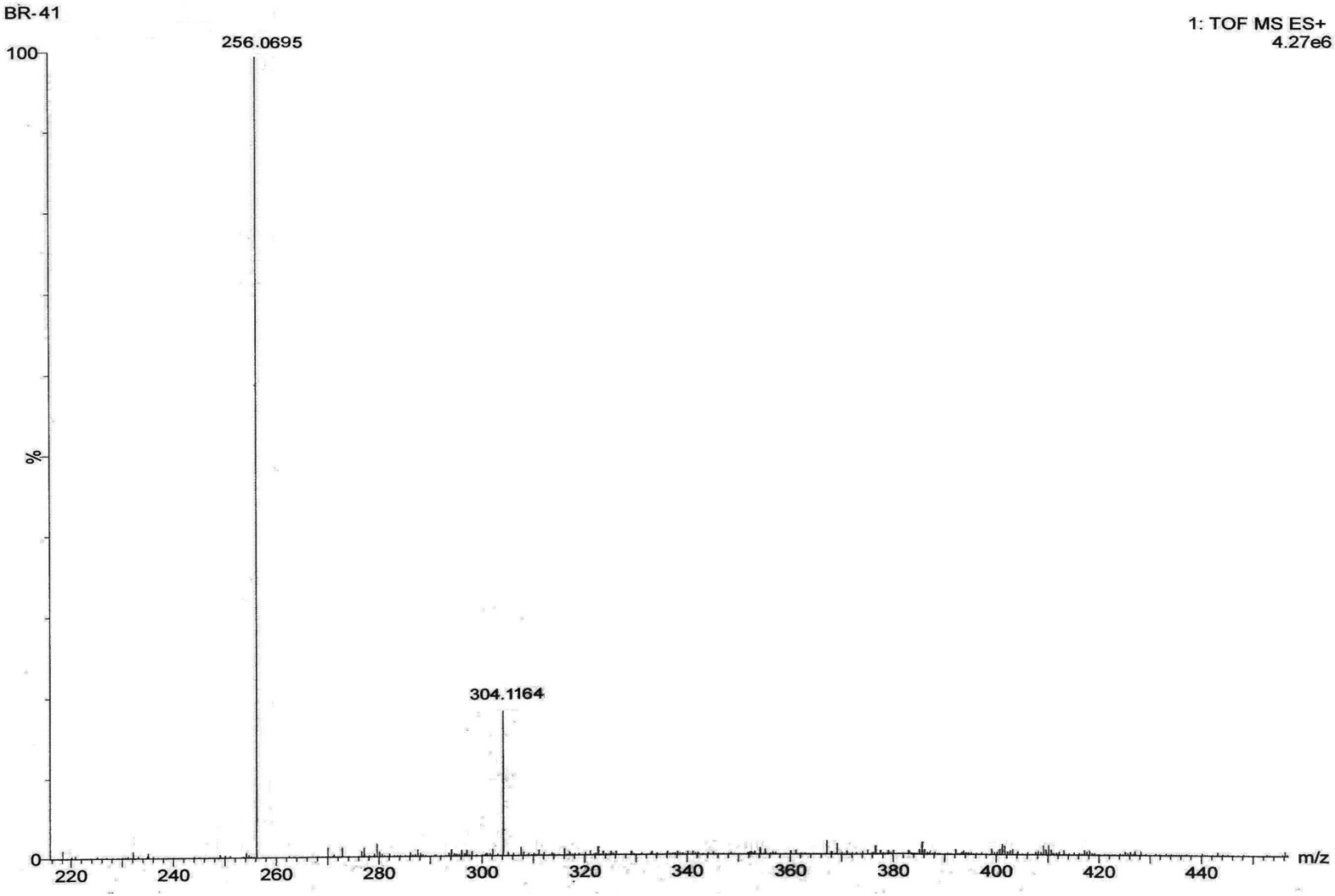
261.0583
261.9362
m/z
251
252
253
254
255
256
257
258
259
260
261
262
263
264
265

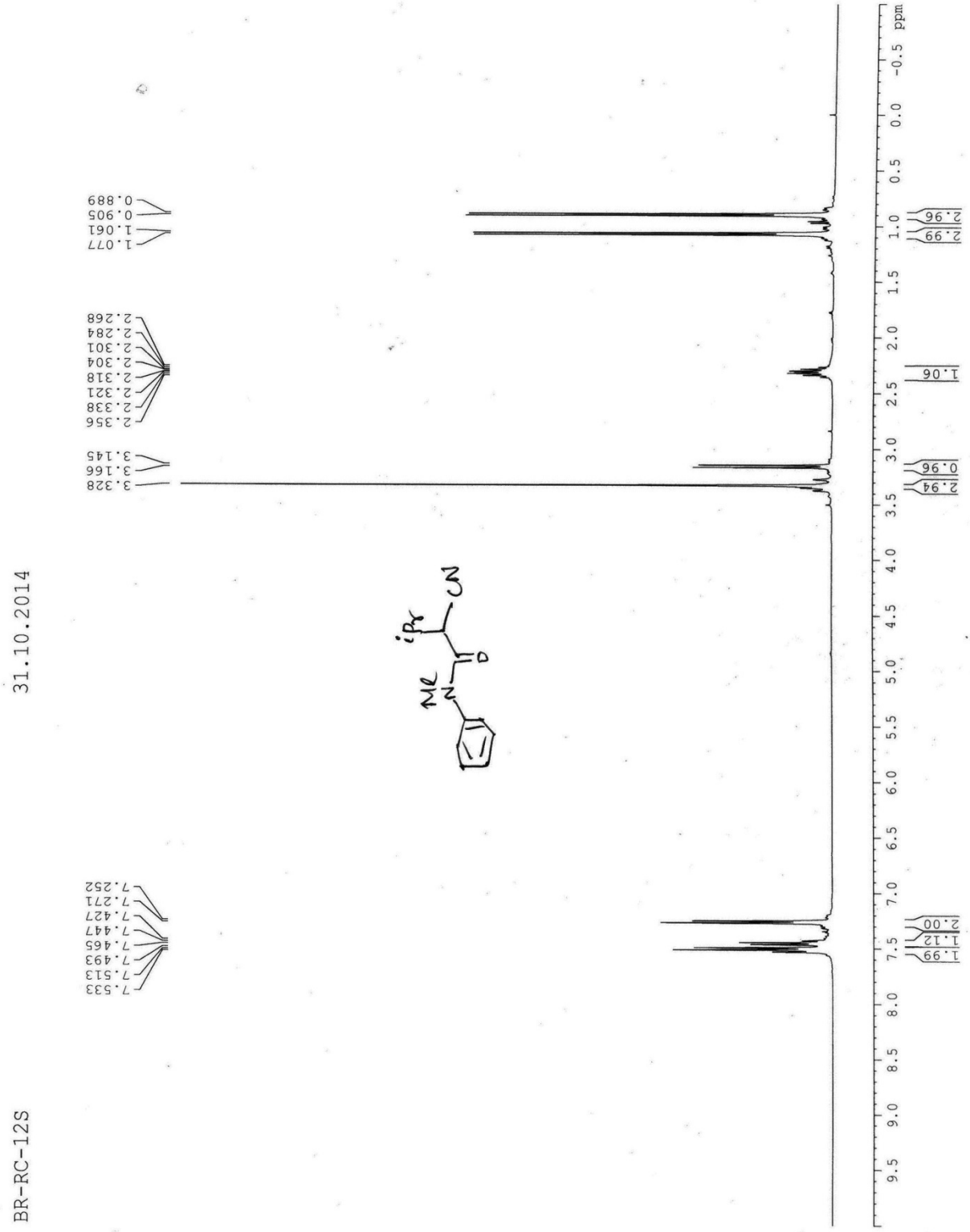
2-Cyano-N-methyl-N-(4-nitrophenyl)propanamide (1g):

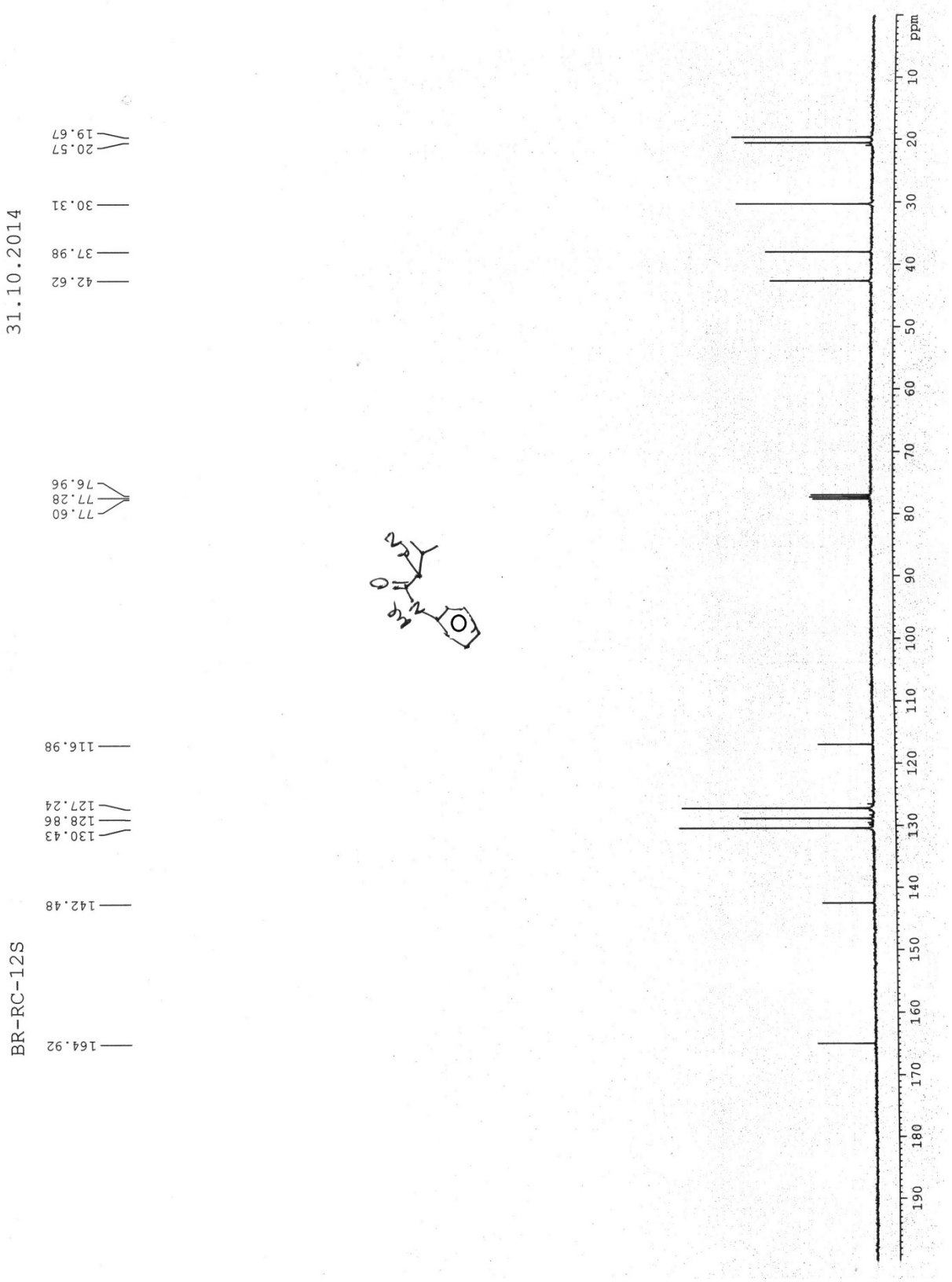


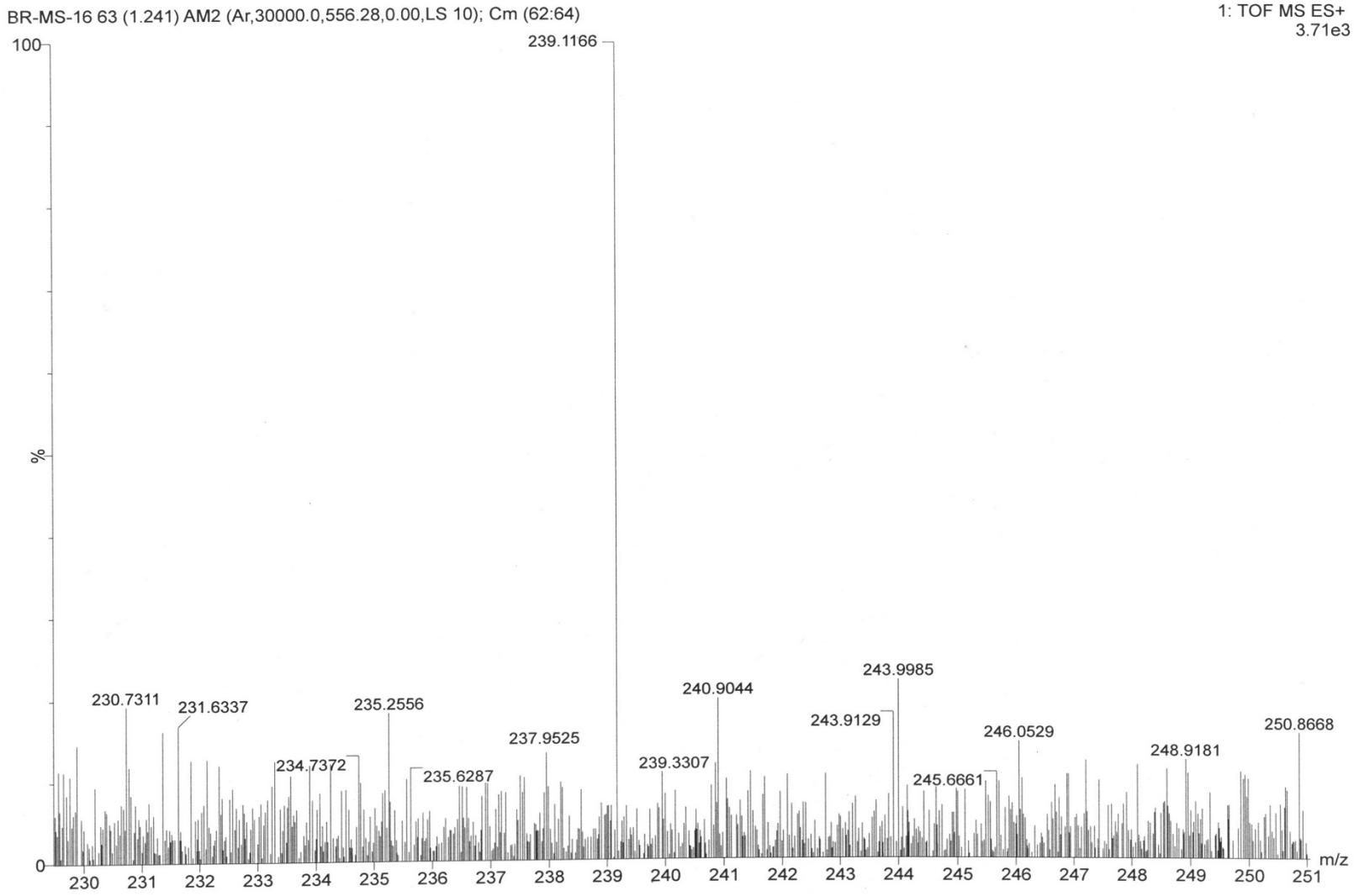

BR-RC-7S
04.09.2014
164.94
147.87
147.28
128.33
125.68
117.82
77.45
77.13
76.81
38.33
29.85
15.64
ppm


BR-41
1: TOF MS ES+
4.27e6
256.0695
304.1164
100
%
0
220
240
260
280
300
320
340
360
380
400
420
440
m/z

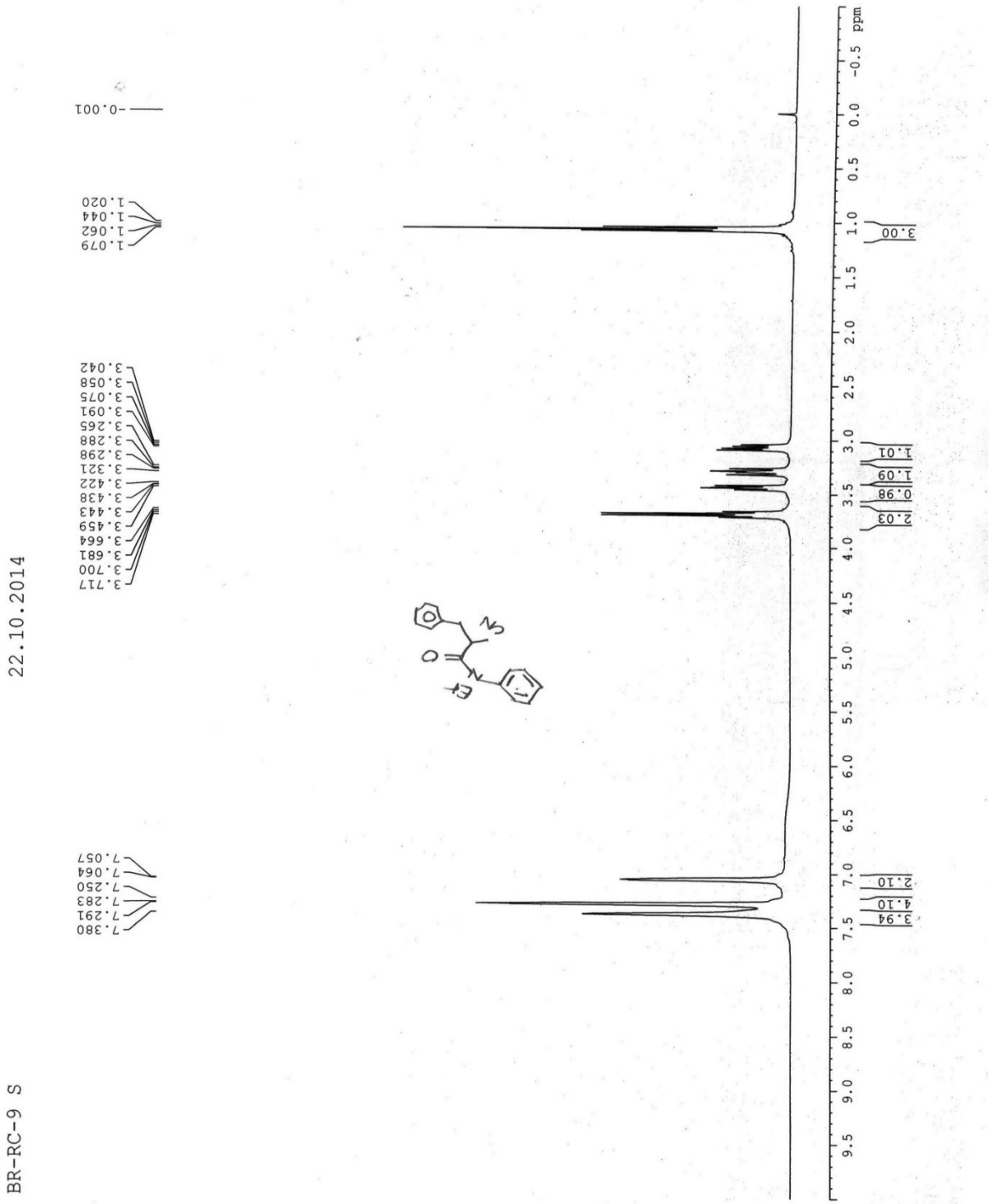
2-Cyano-N-ethyl-3-methyl-N-phenylbutanamide (1h):

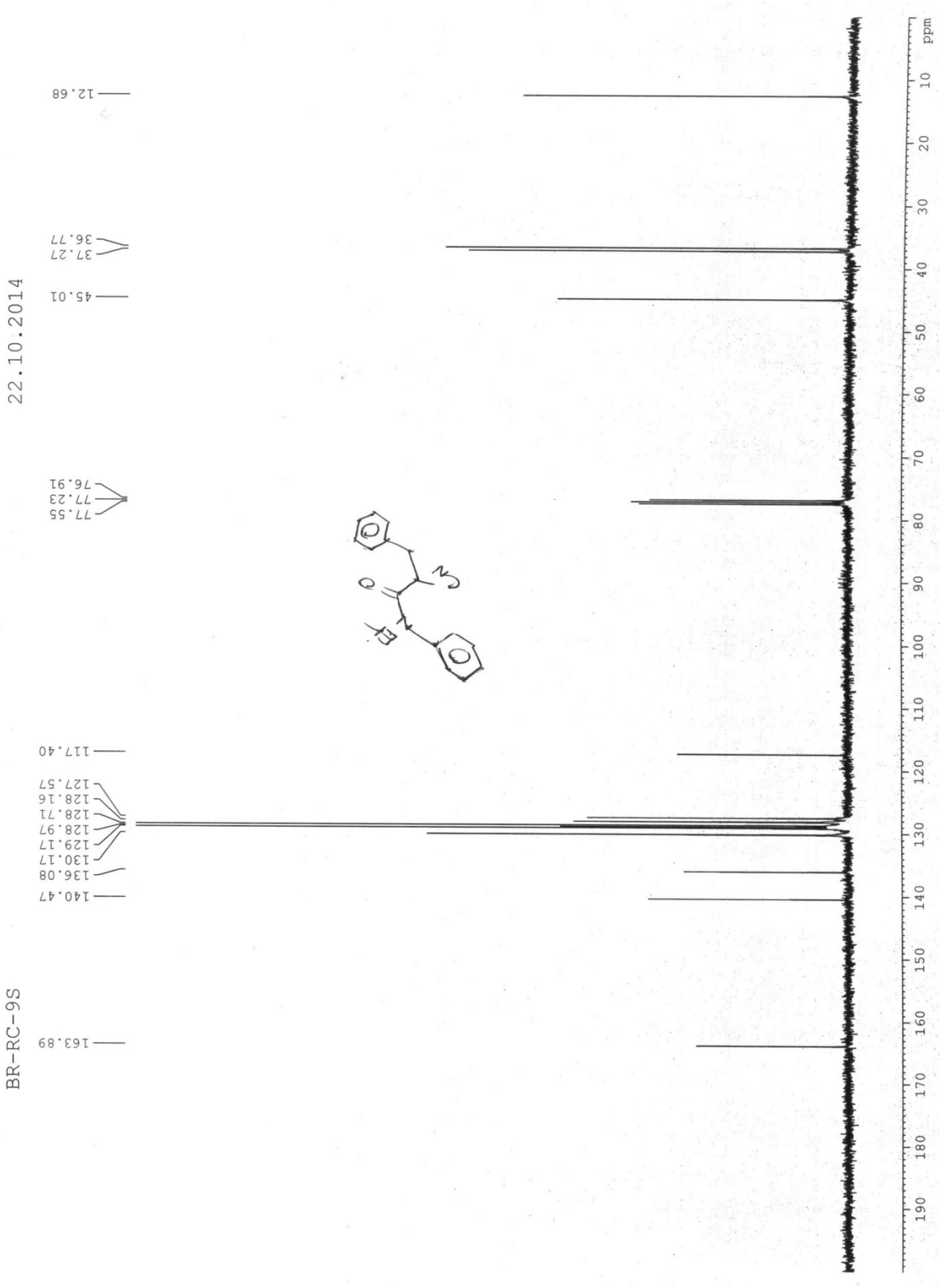


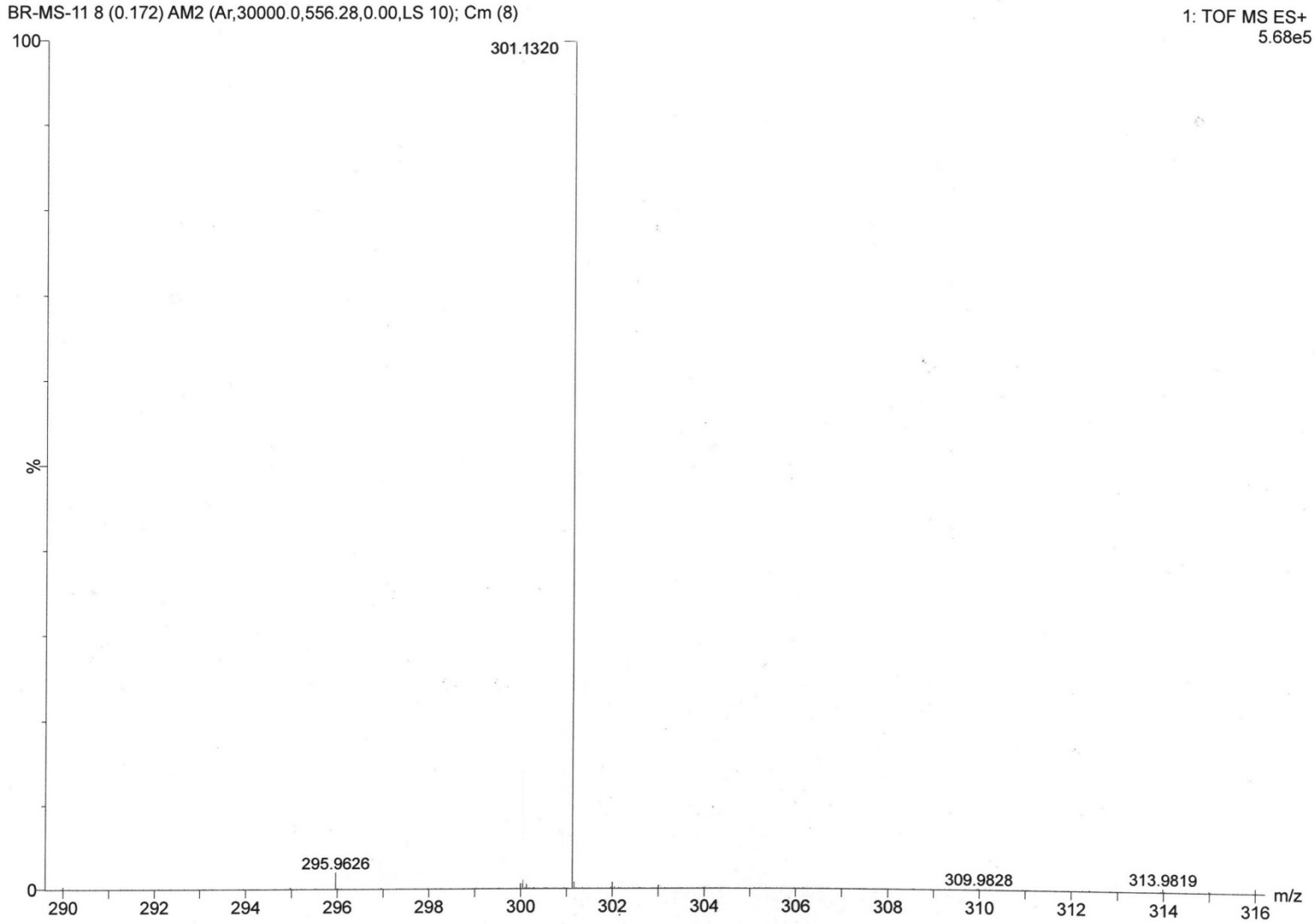

BR-RC-12S
31.10.2014
164.92
142.48
130.43
128.86
127.24
116.98
77.60
77.28
76.96
42.62
37.98
30.31
20.57
19.67
ppm


BR-MS-16 63 (1.241) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (62:64)
1: TOF MS ES+
3.71e3
100
%
0
239.1166
230.7311
231.6337
234.7372
235.2556
235.6287
237.9525
239.3307
240.9044
243.9129
243.9985
245.6661
246.0529
248.9181
250.8668
230
231
232
233
234
235
236
237
238
239
240
241
242
243
244
245
246
247
248
249
250
251
m/z

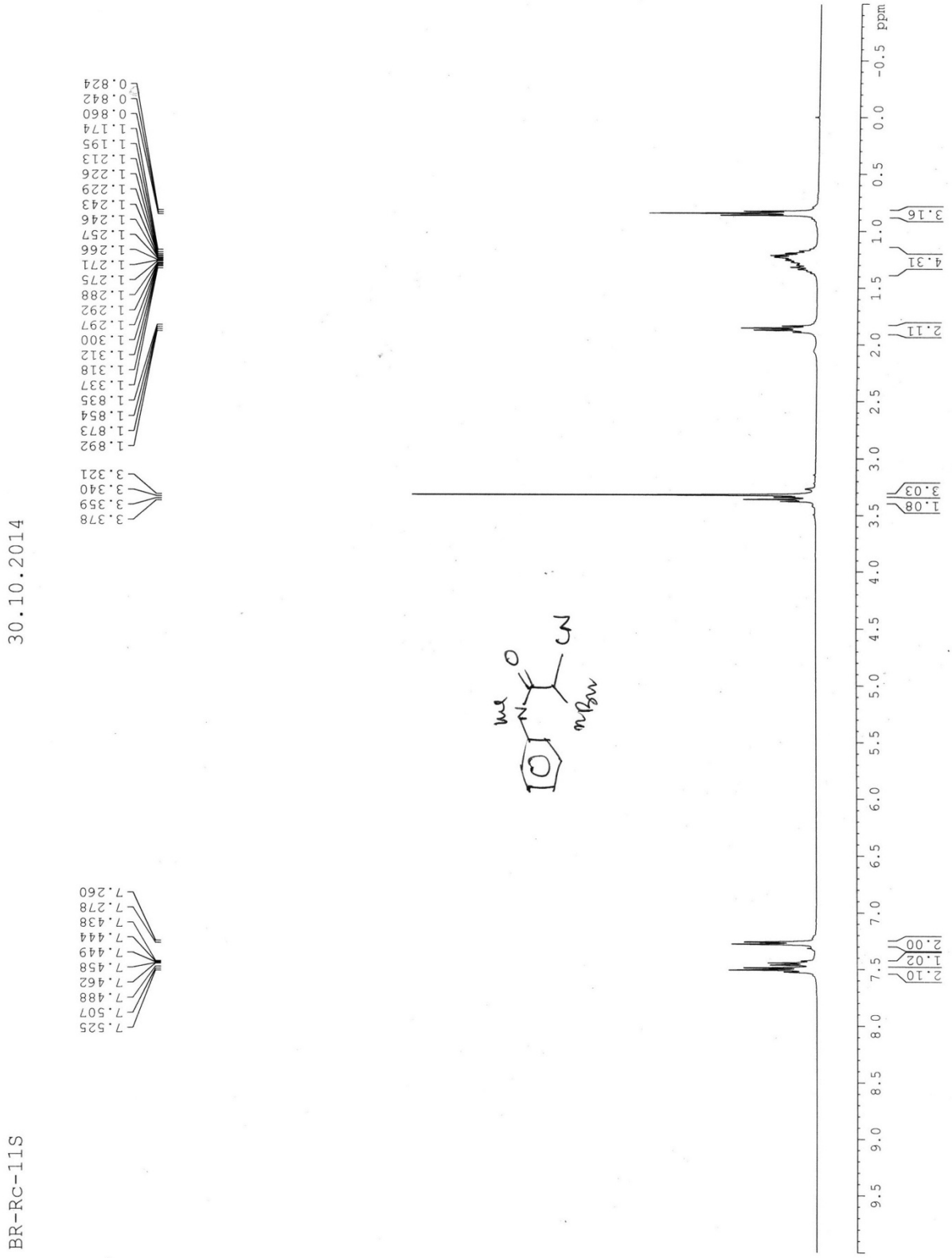
2-Cyano-N-ethyl-N,3-diphenylpropanamide (1i):

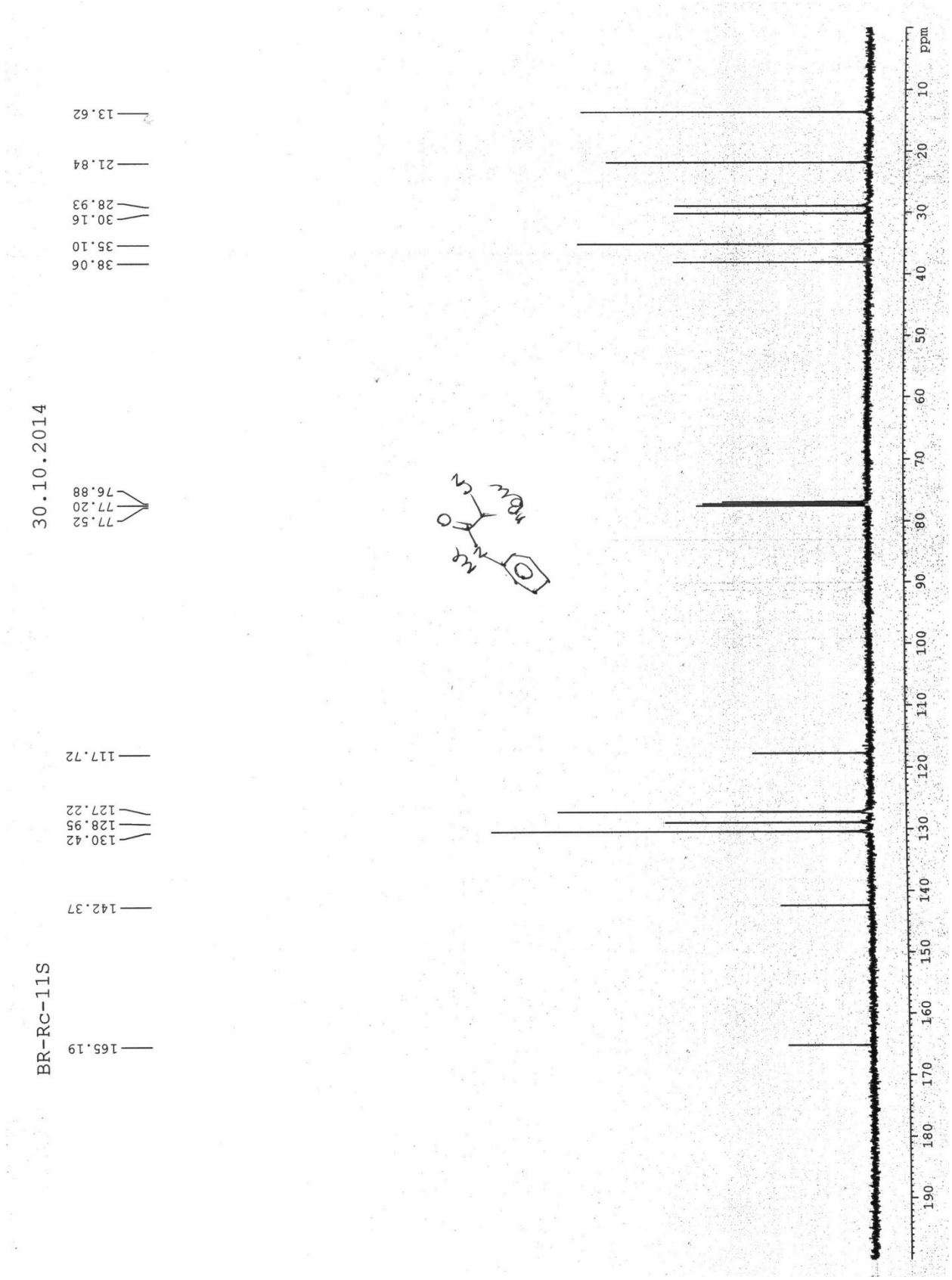


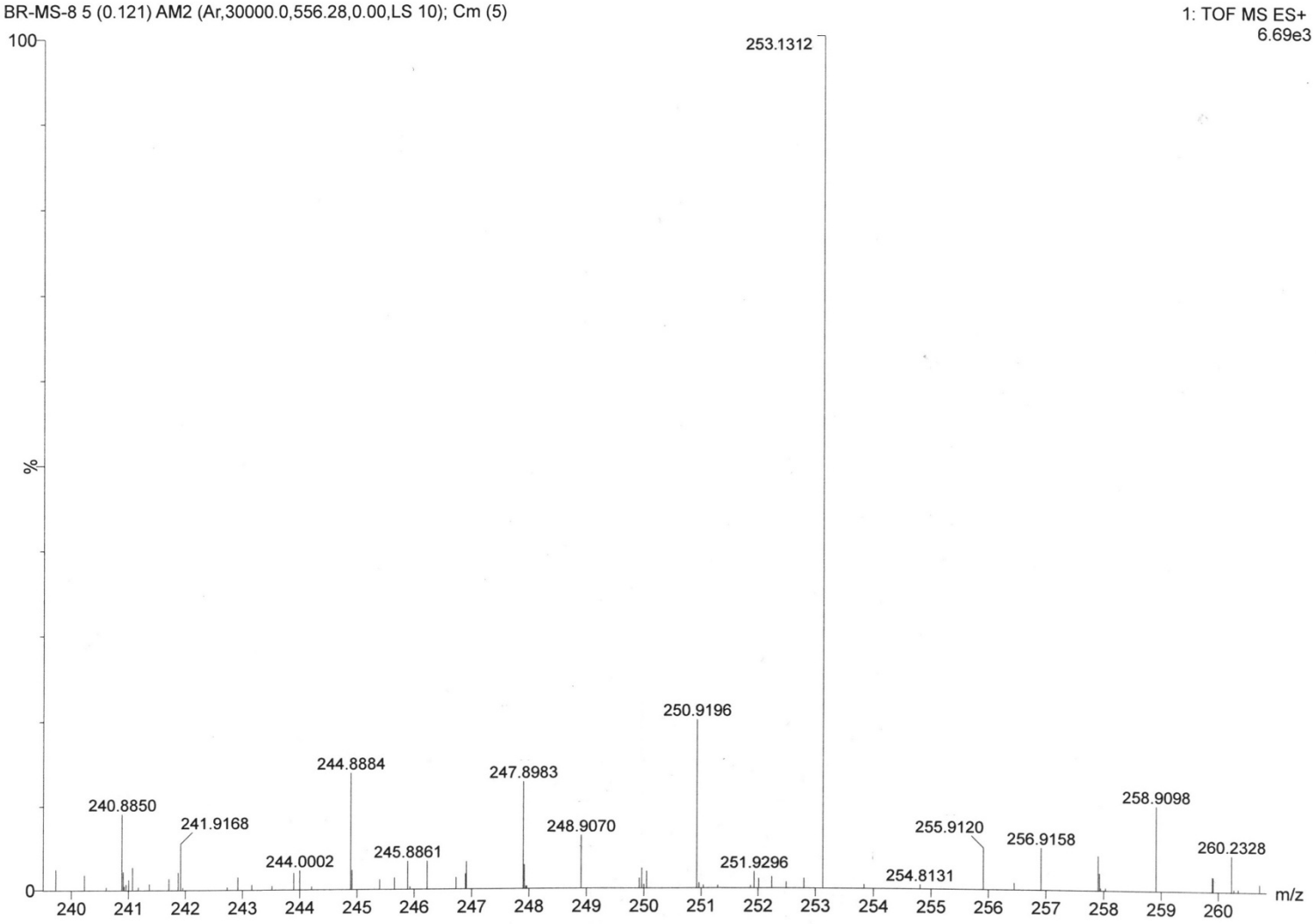

BR-RC-9S
22.10.2014
163.89
140.47
136.08
130.17
129.17
128.97
128.71
128.16
127.57
117.40
77.55
77.23
76.91
45.01
37.27
36.77
12.68
ppm


BR-MS-11 8 (0.172) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (8)
1: TOF MS ES+
5.68e5
100
%
0
301.1320
295.9626
309.9828
313.9819
290
292
294
296
298
300
302
304
306
308
310
312
314
316
m/z

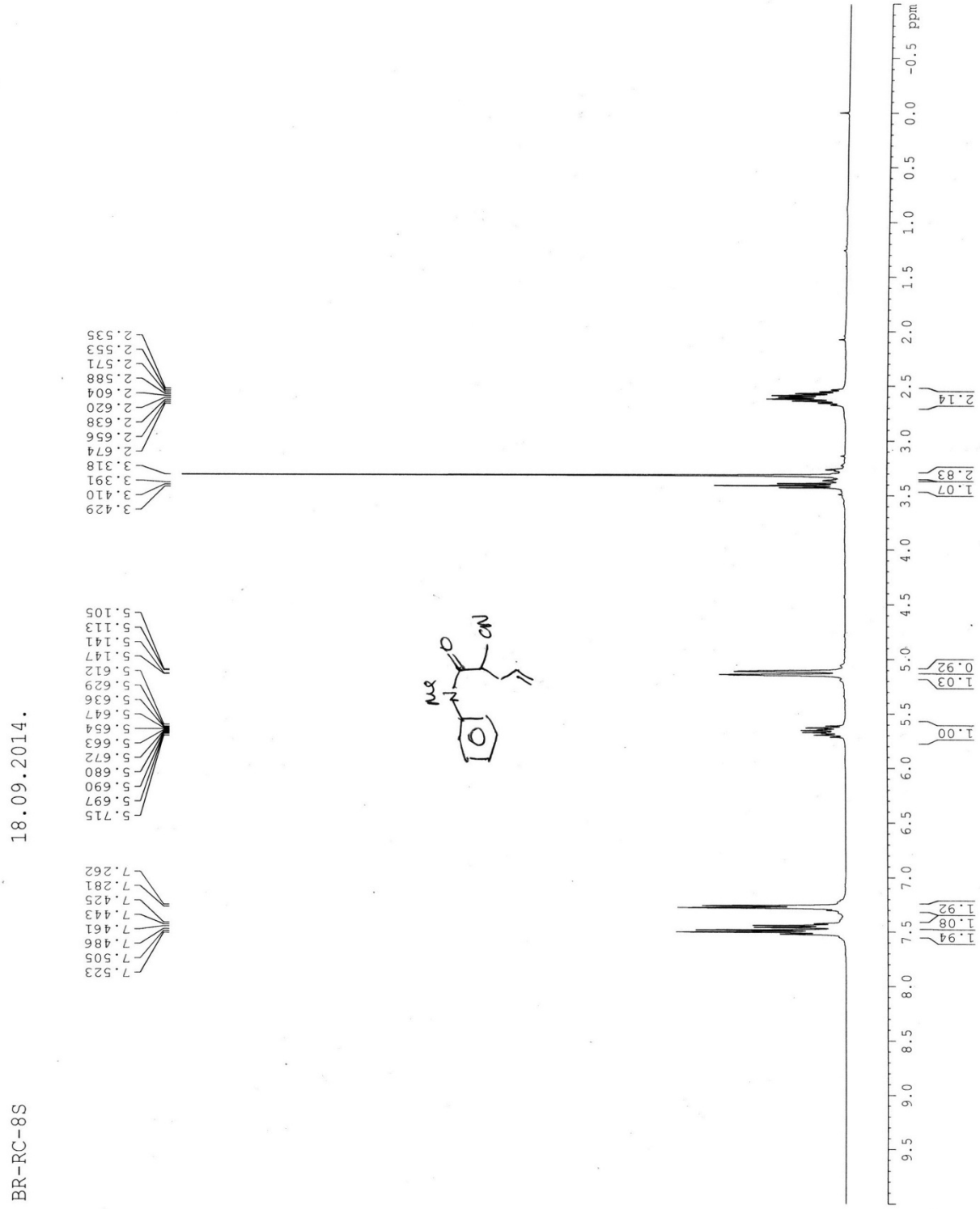
2-Cyano-N-ethyl-N-phenylhexanamide (1j):

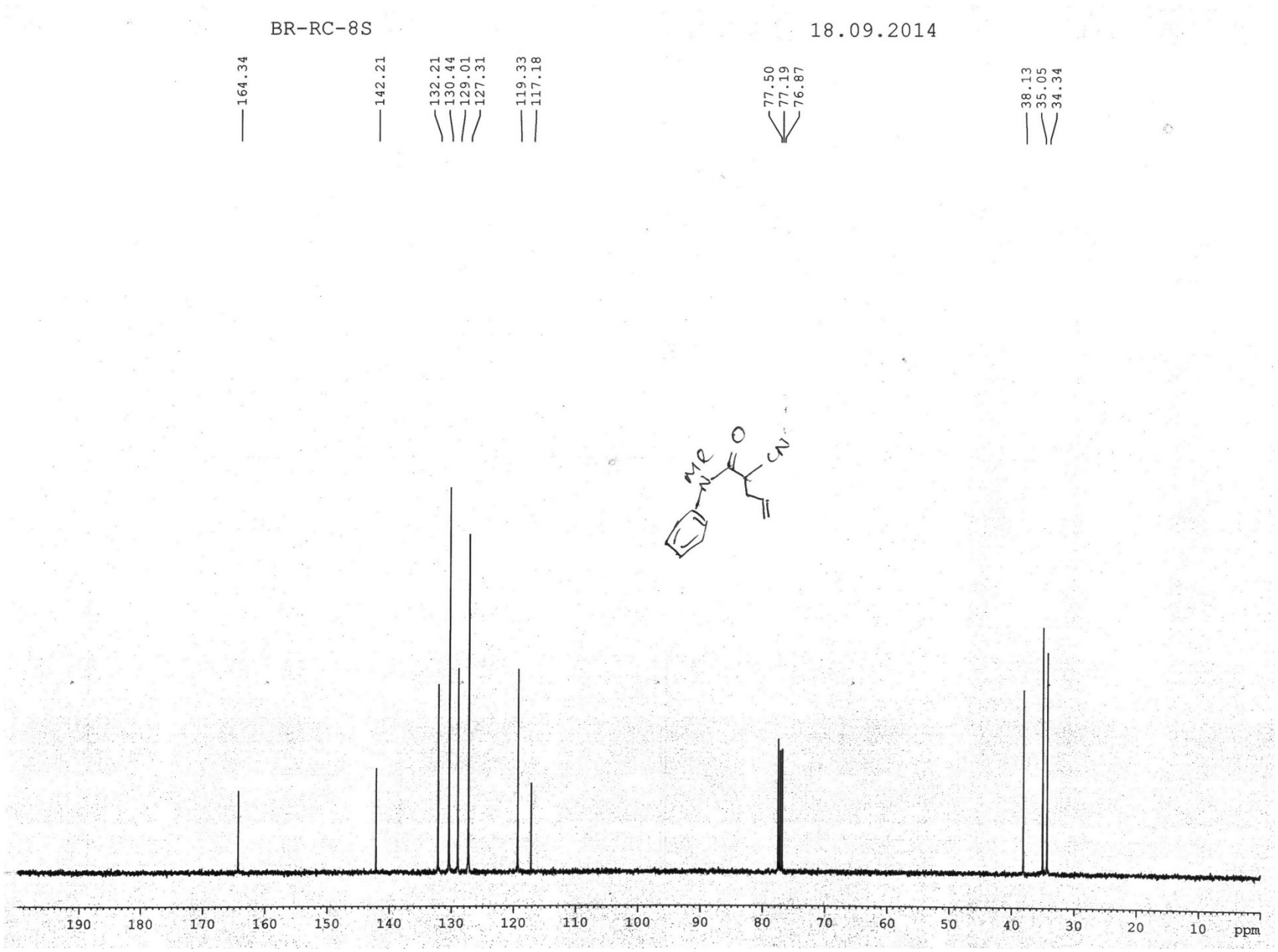


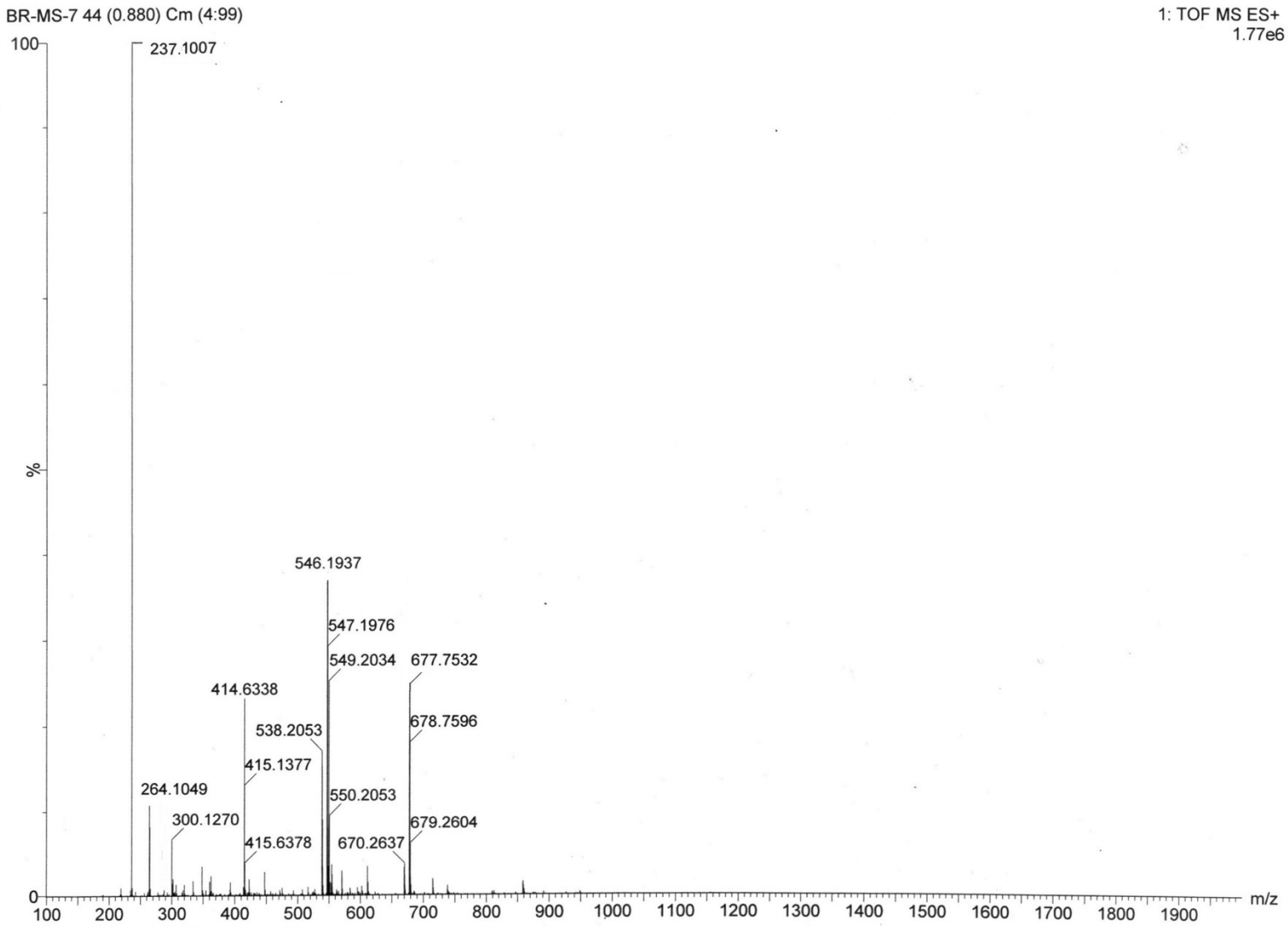

BR-Rc-11S
30.10.2014
165.19
142.37
130.42
128.95
127.22
117.72
77.52
77.20
76.88
38.06
35.10
30.16
28.93
21.84
13.62
ppm


BR-MS-8 5 (0.121) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (5)
1: TOF MS ES+
6.69e3
100
%
0
253.1312
250.9196
244.8884
247.8983
240.8850
241.9168
248.9070
244.0002
245.8861
251.9296
254.8131
255.9120
256.9158
258.9098
260.2328
240
241
242
243
244
245
246
247
248
249
250
251
252
253
254
255
256
257
258
259
260
m/z

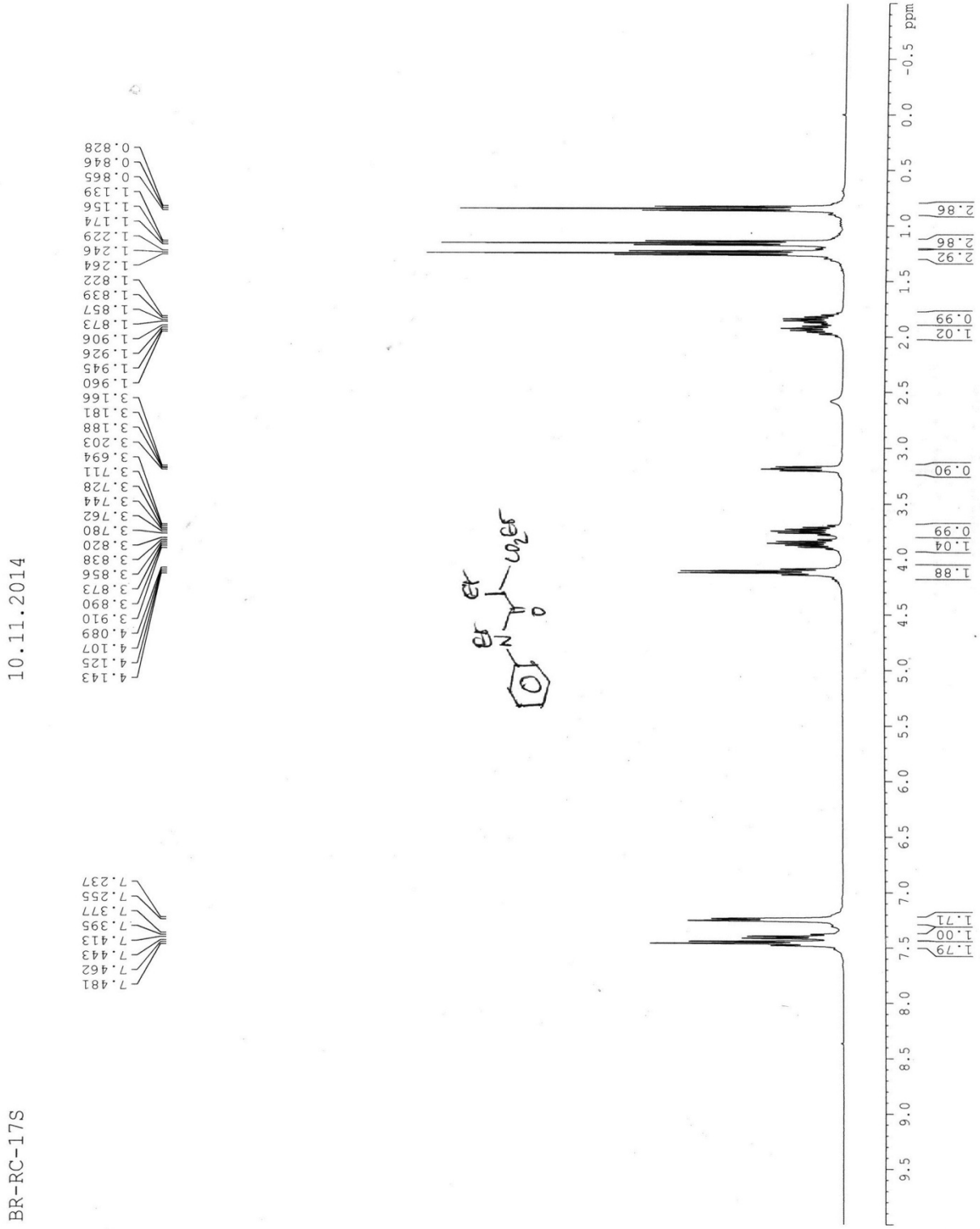
2-Cyano-N-ethyl-N-phenylpent-4-enamide (1k):




BR-RC-8S
18.09.2014
164.34
142.21
132.21
130.44
129.01
127.31
119.33
117.18
77.50
77.19
76.87
38.13
35.05
34.34
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

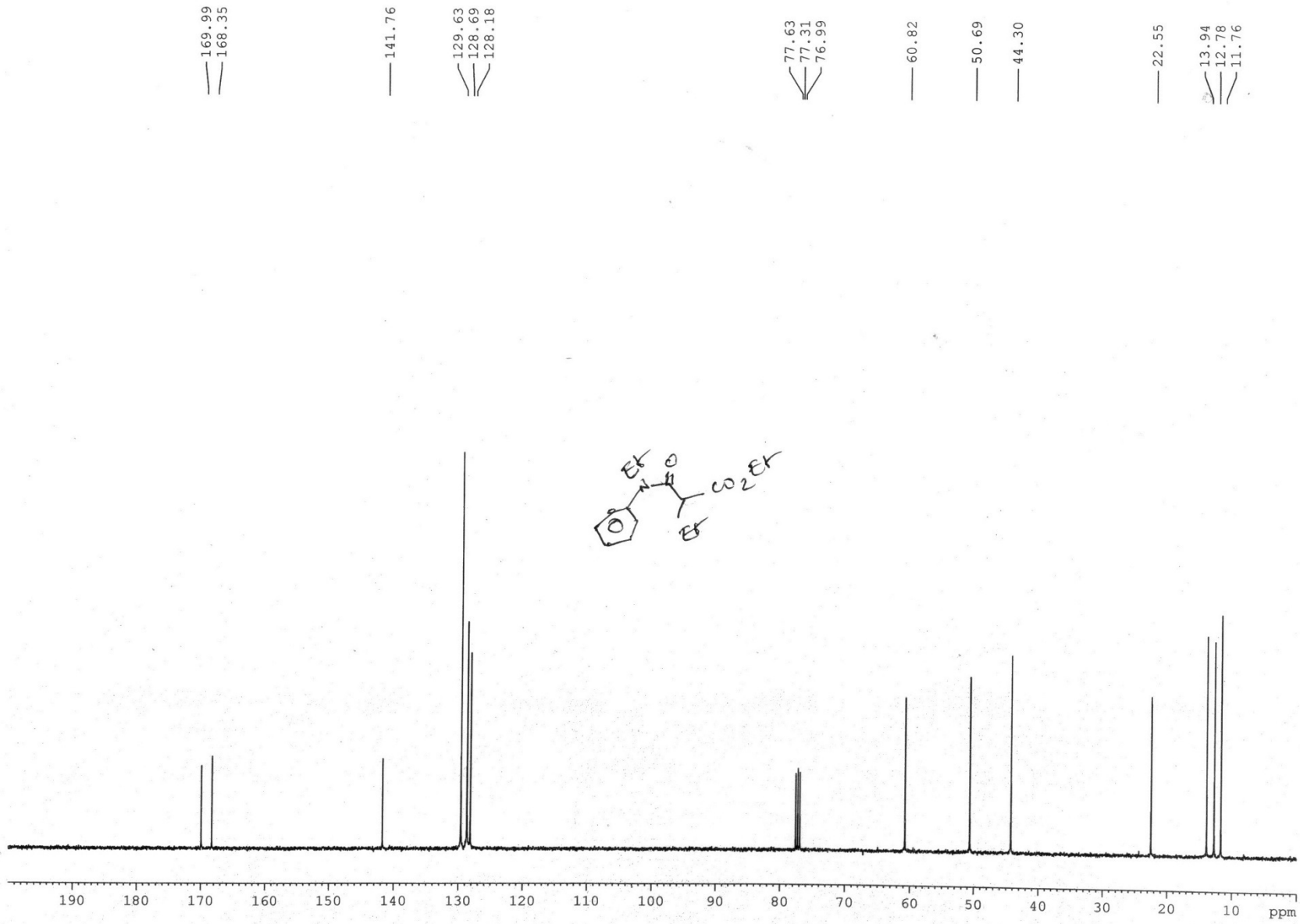

BR-MS-7 44 (0.880) Cm (4:99)
1: TOF MS ES+
1.77e6
100
%
0
237.1007
546.1937
547.1976
549.2034
677.7532
414.6338
678.7596
538.2053
415.1377
264.1049
550.2053
300.1270
679.2604
415.6378
670.2637
100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600 1700 1800 1900
m/z

Ethyl 2-(ethyl(phenyl)carbamoyl)butanoate (1l):



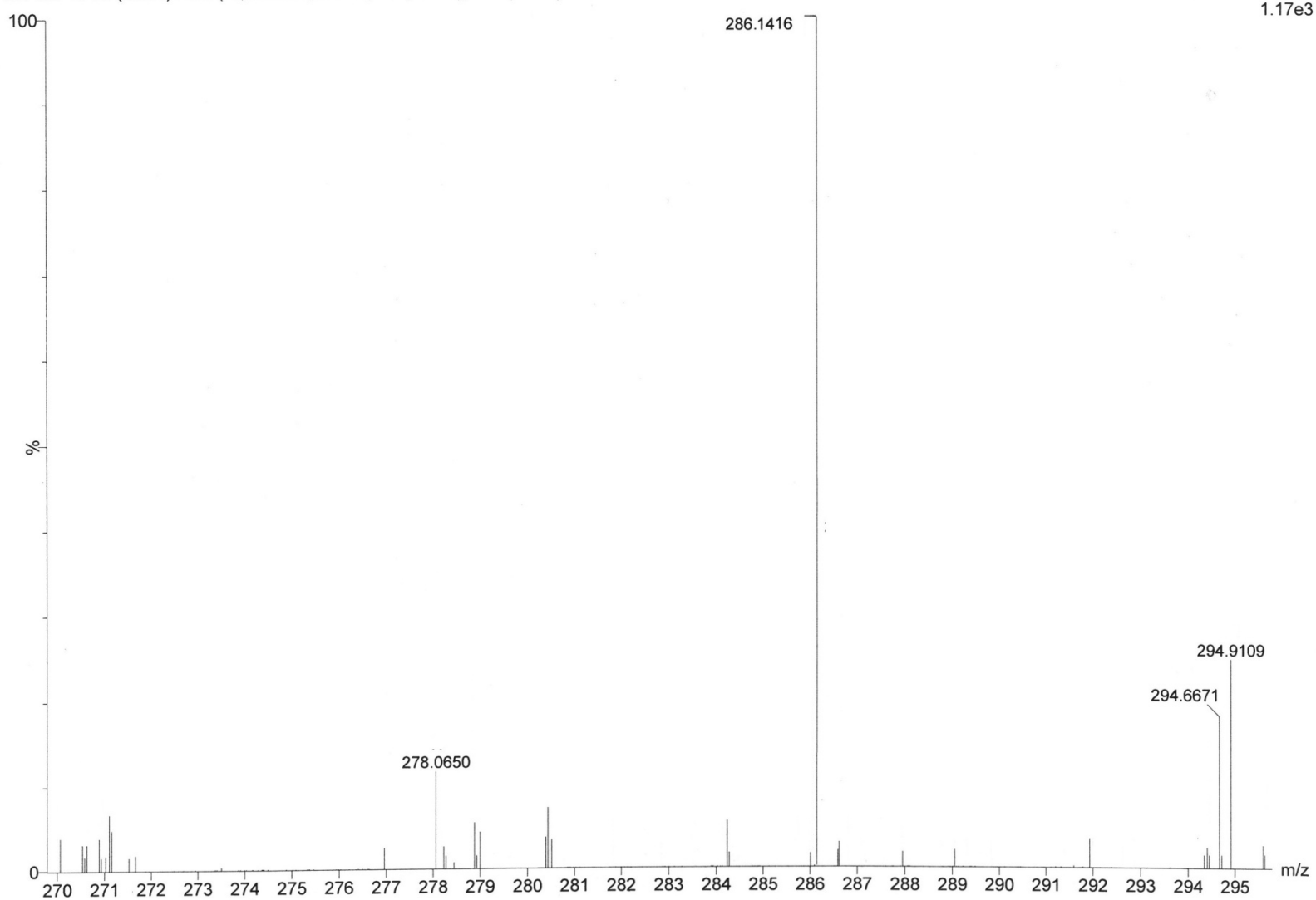
BR-RC-17S

10.11.2014

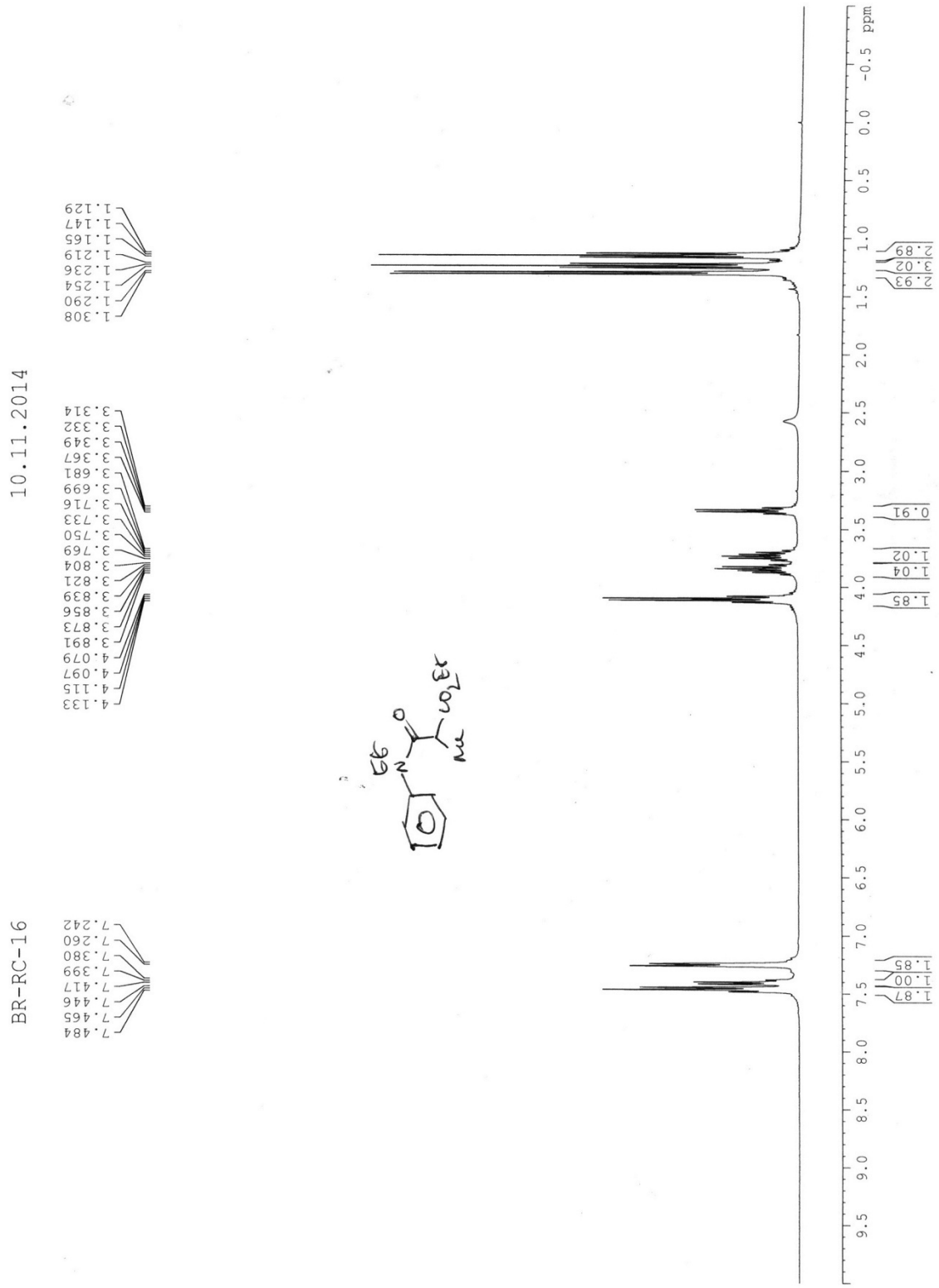


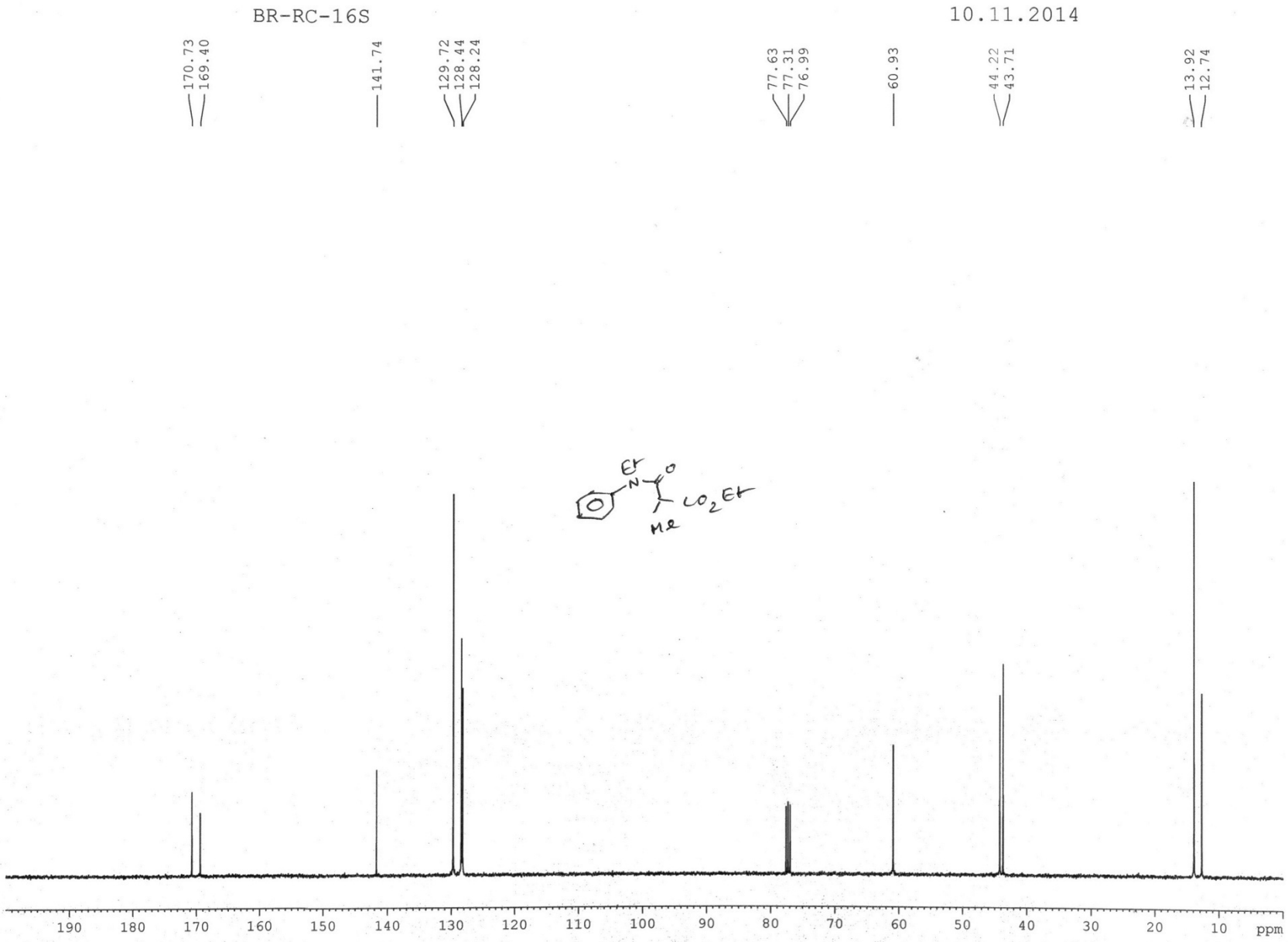
BR-MS-13 95 (1.861) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (95:96)

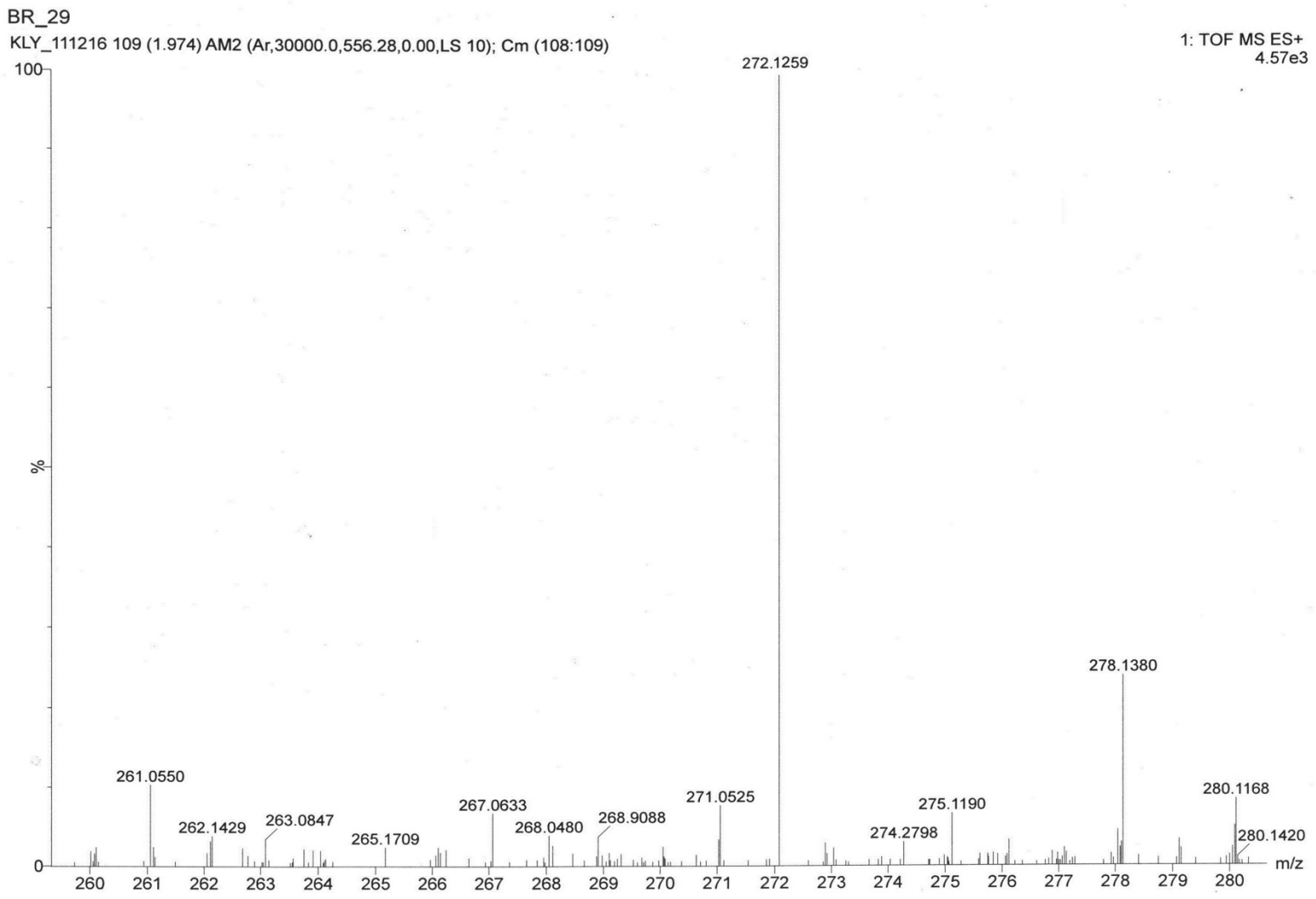
1: TOF MS ES+
1.17e3



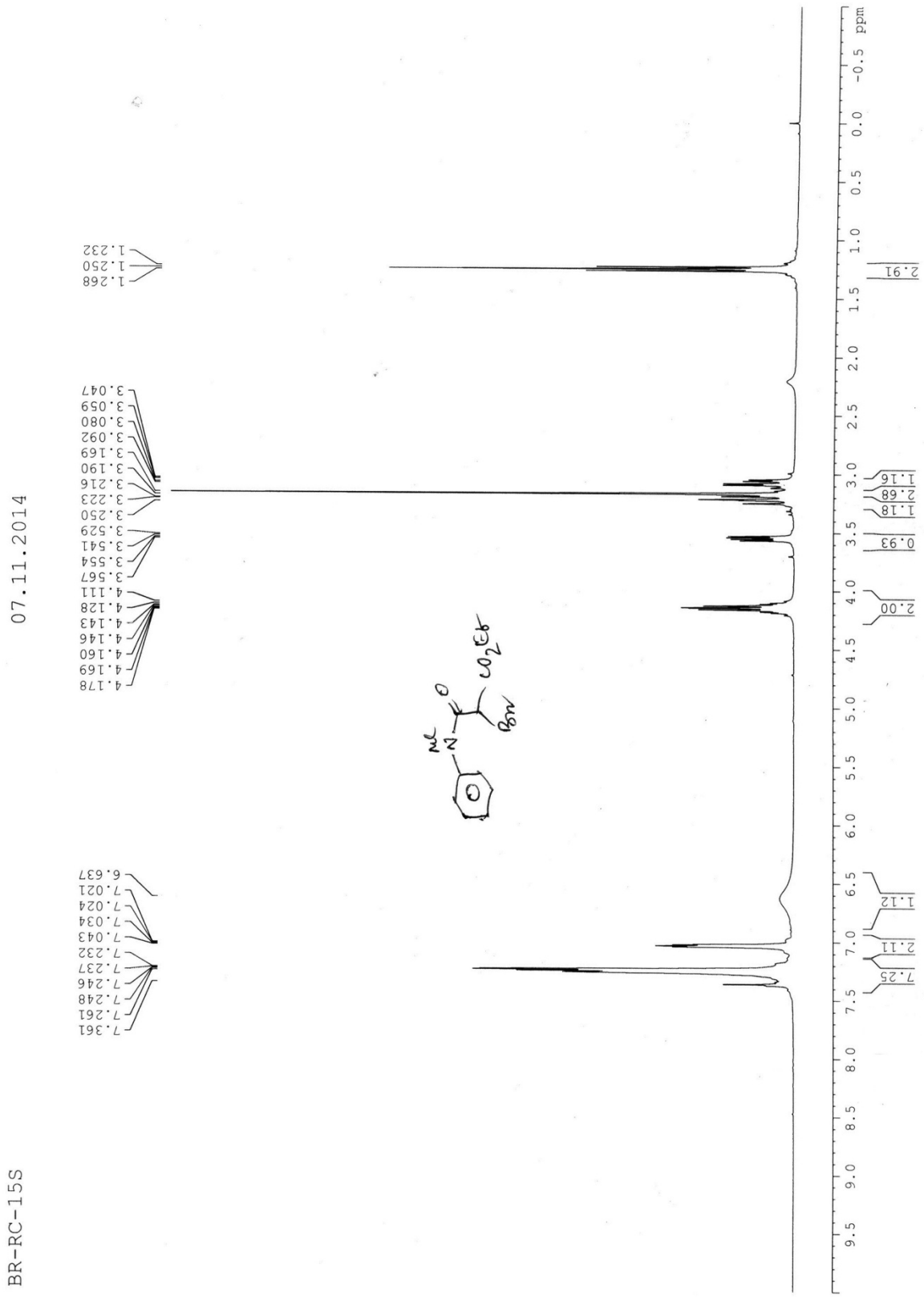
Ethyl 3-(ethyl(phenyl)amino)-2-methyl-3-oxopropanoate (1m):



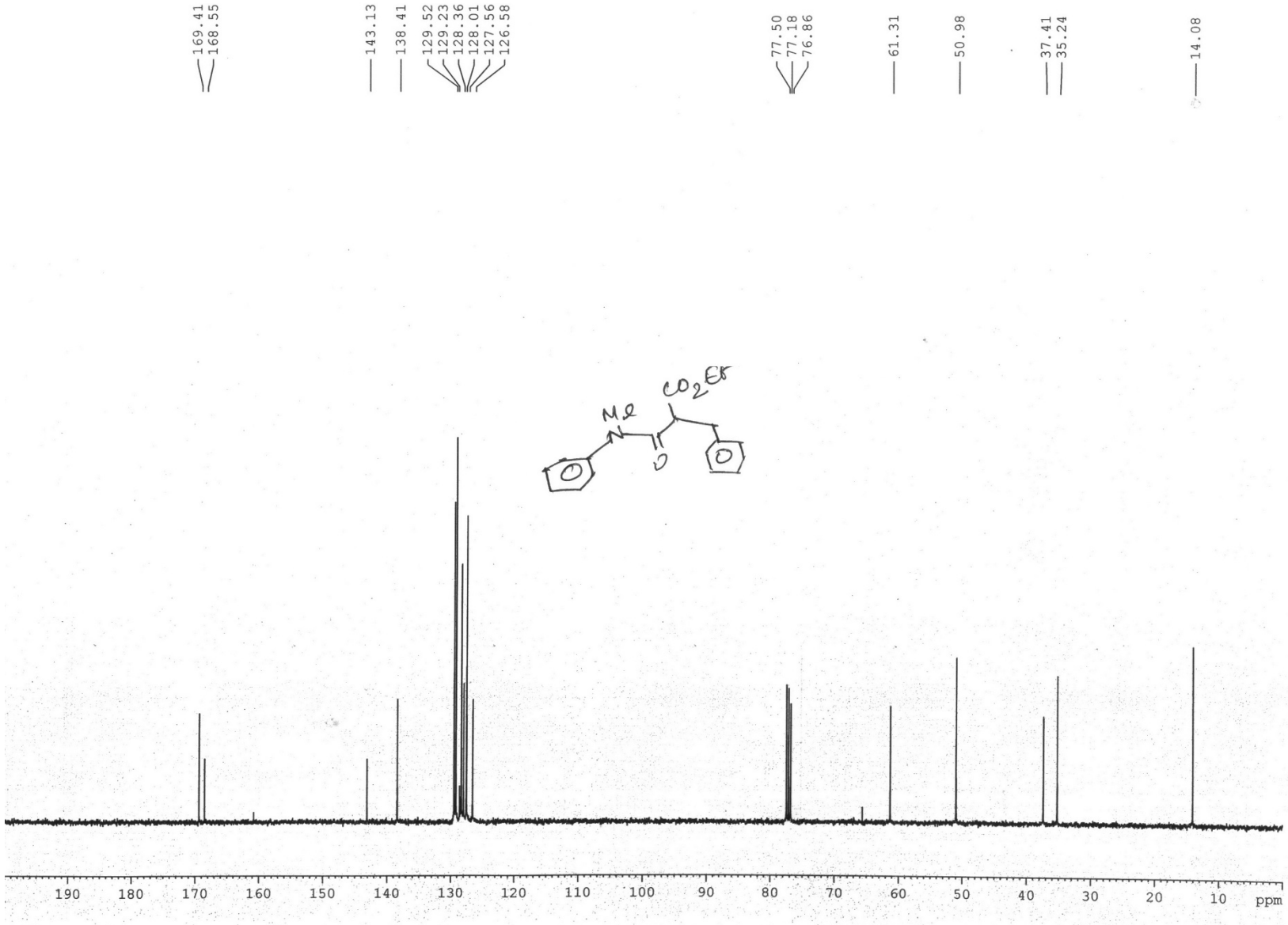

BR-RC-16S
10.11.2014
170.73
169.40
141.74
129.72
128.44
128.24
77.63
77.31
76.99
60.93
44.22
43.71
13.92
12.74
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


BR_29
KLY_111216 109 (1.974) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (108:109)
1: TOF MS ES+
4.57e3
100
%
0
272.1259
278.1380
261.0550
262.1429
263.0847
265.1709
267.0633
268.0480
268.9088
271.0525
274.2798
275.1190
280.1168
280.1420
m/z
260
261
262
263
264
265
266
267
268
269
270
271
272
273
274
275
276
277
278
279
280

Ethyl 2-benzyl-3-(methyl(phenyl)amino)-3-oxopropanoate (1n):

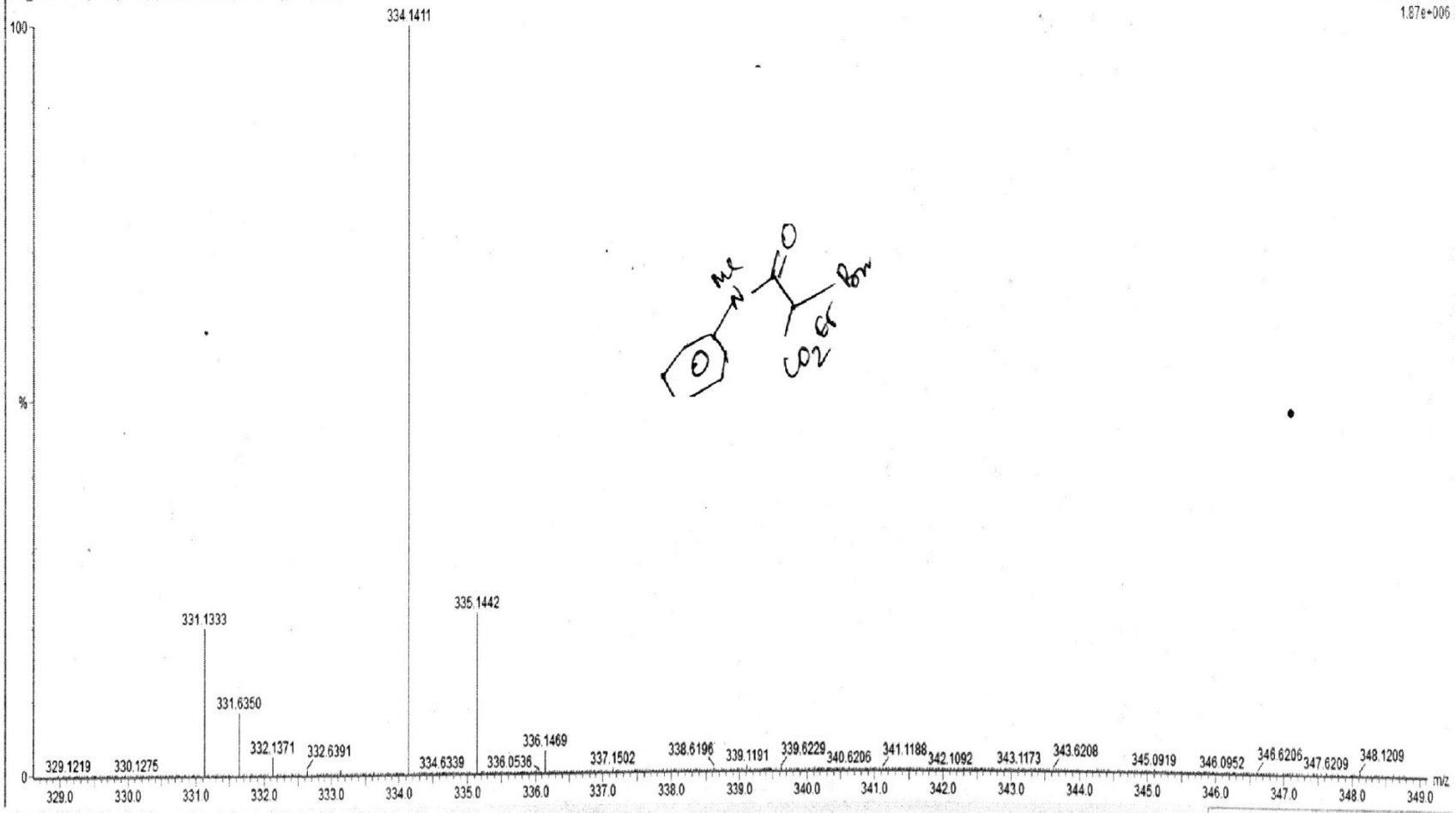


BR-RC-15 S 07.11.2014



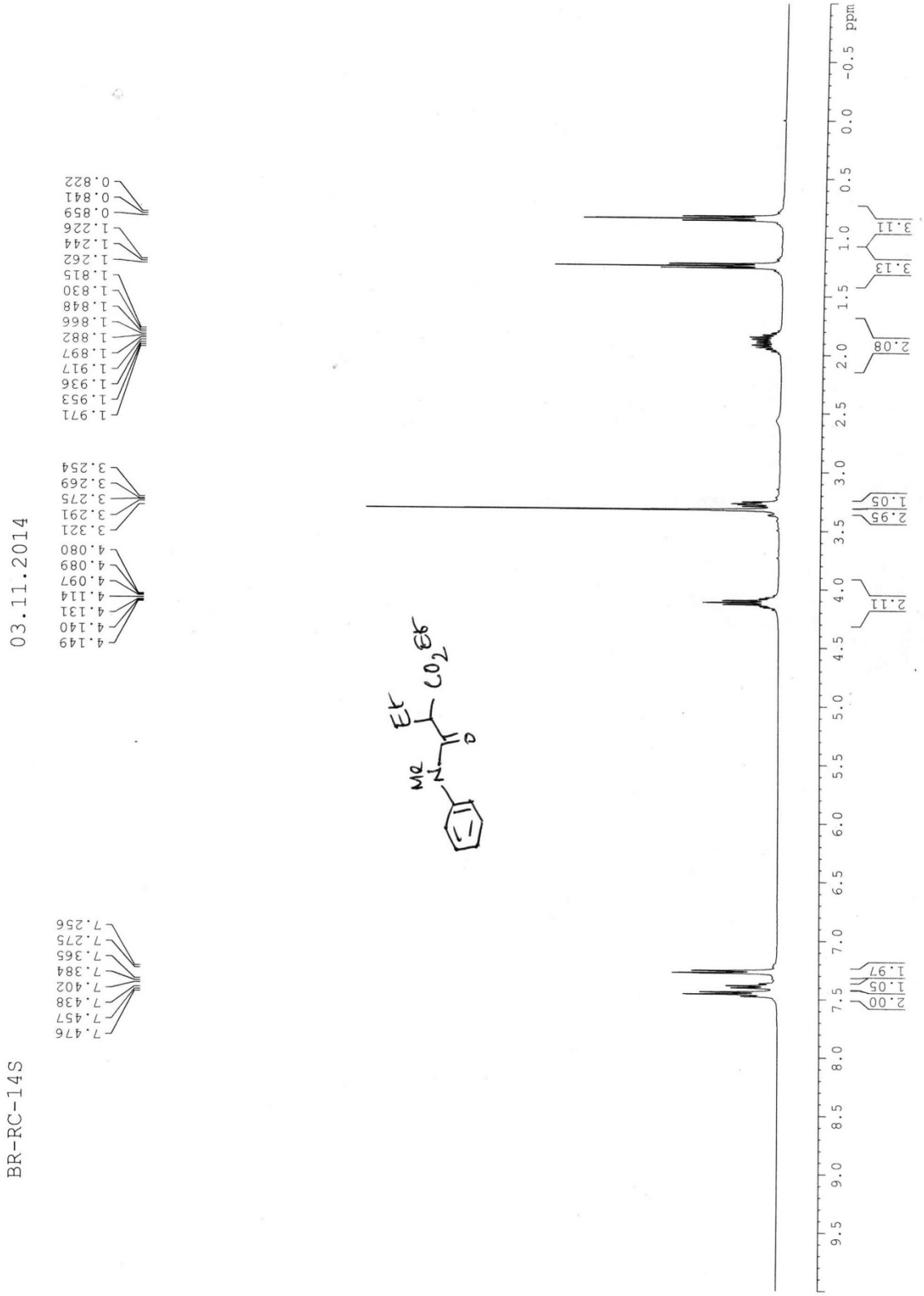
BR_MS_31
KLY_161219 34 (0.633) AM2 (Ar,30000.0,556.28,0.00,LS 2); Cm (23:34)

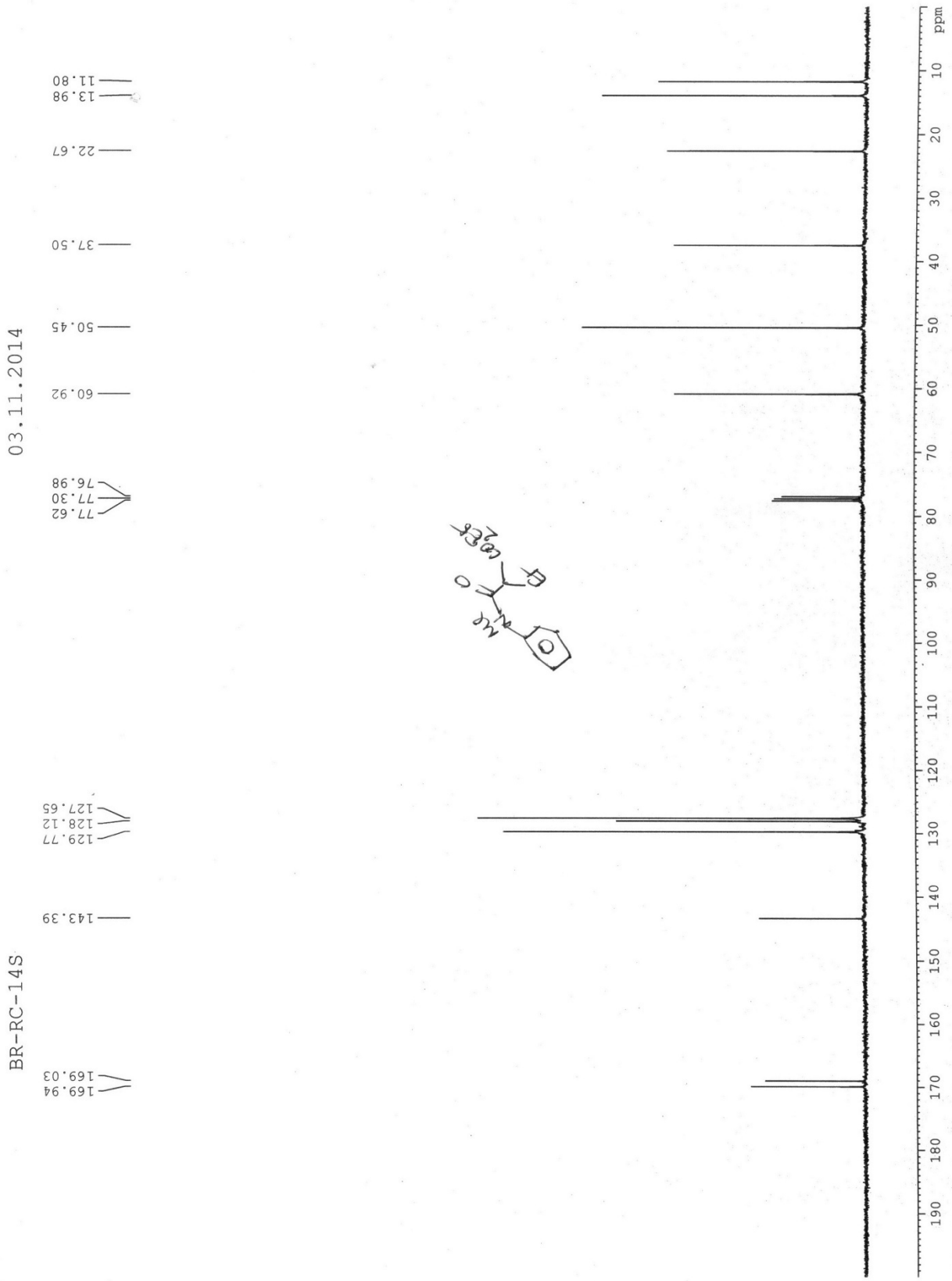
1: TOF MS ES+
1.87e+006

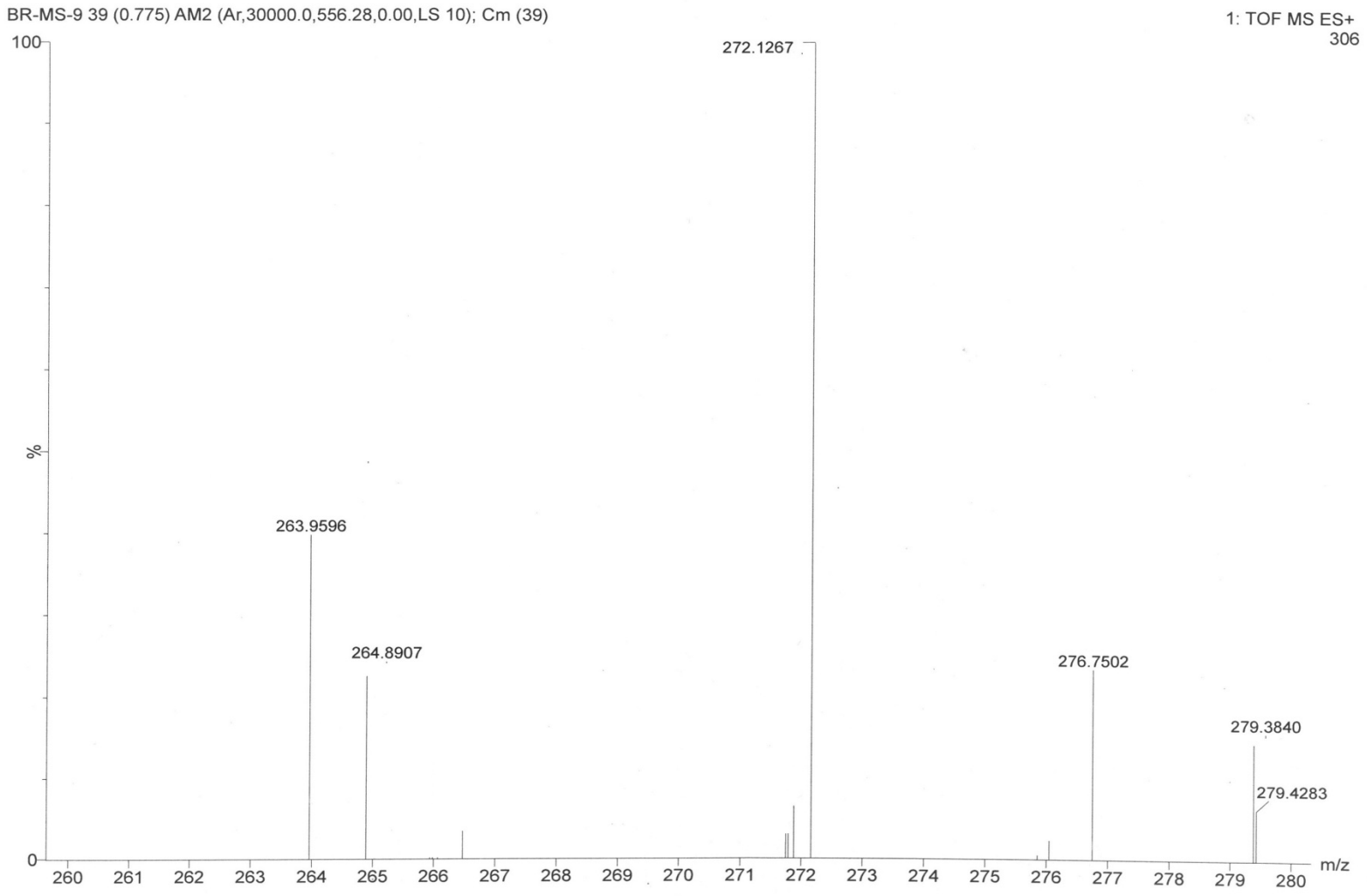


For Help, press F1

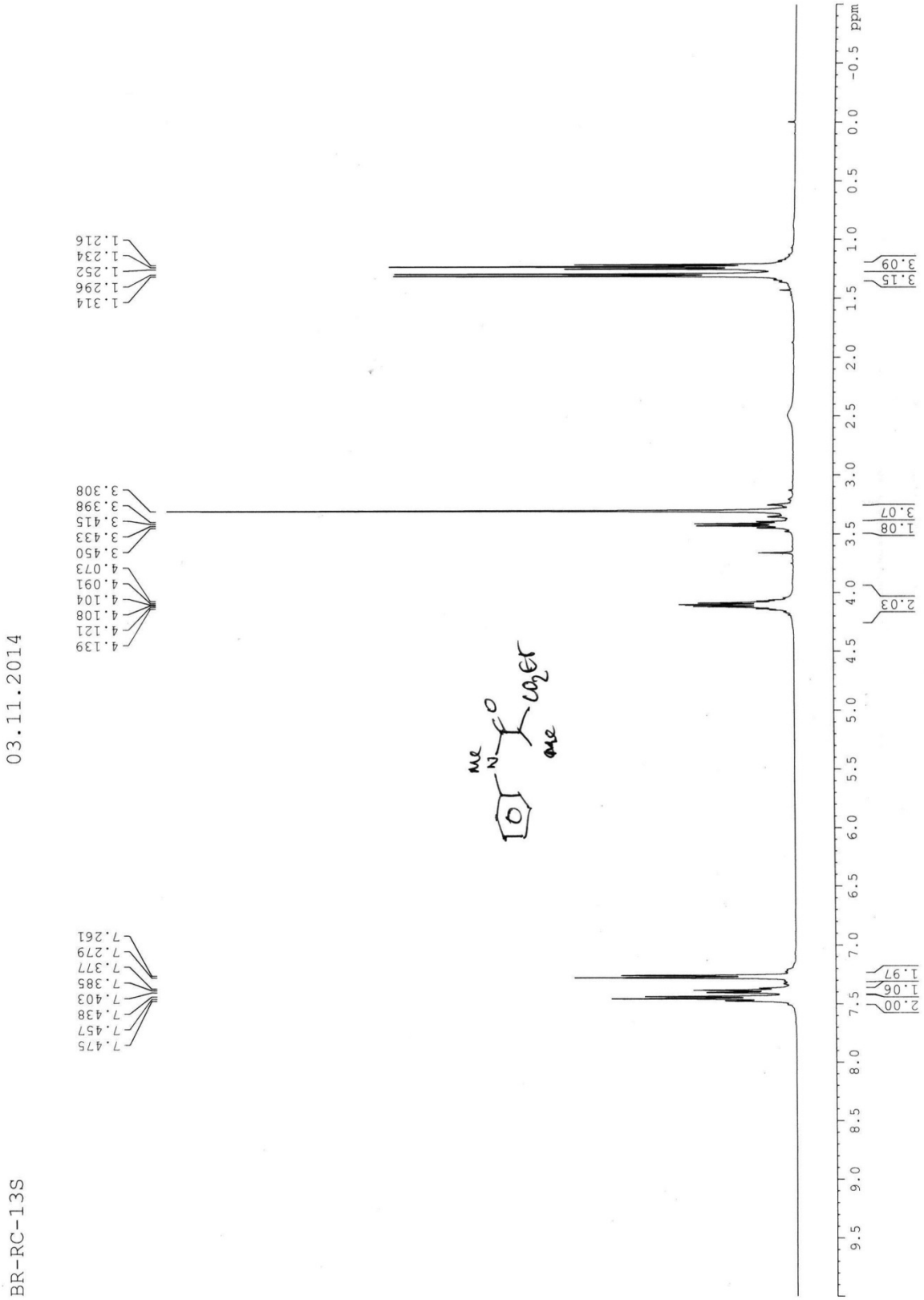
Ethyl 2-(methyl(phenyl)carbamoyl)butanoate (1o):

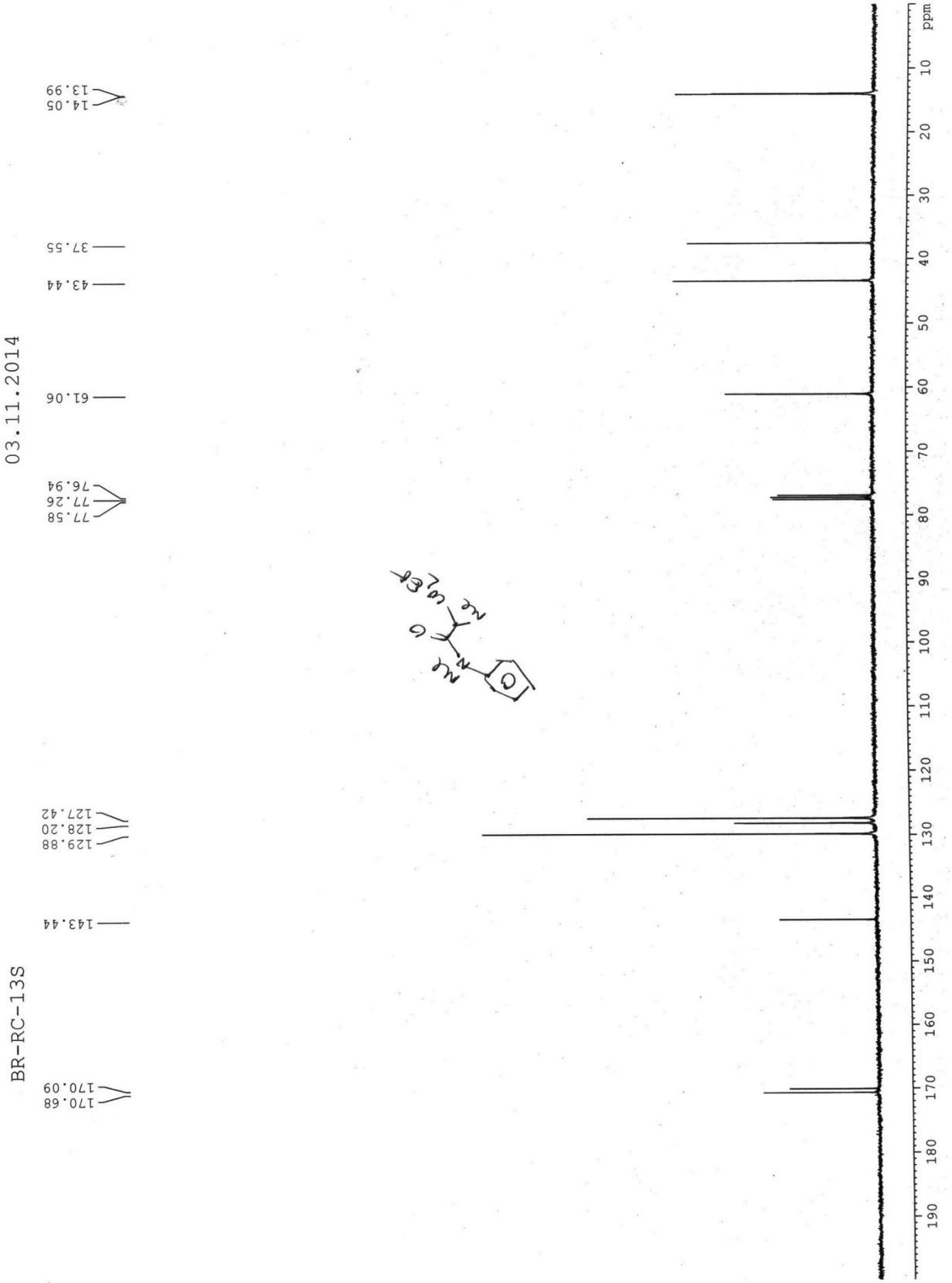


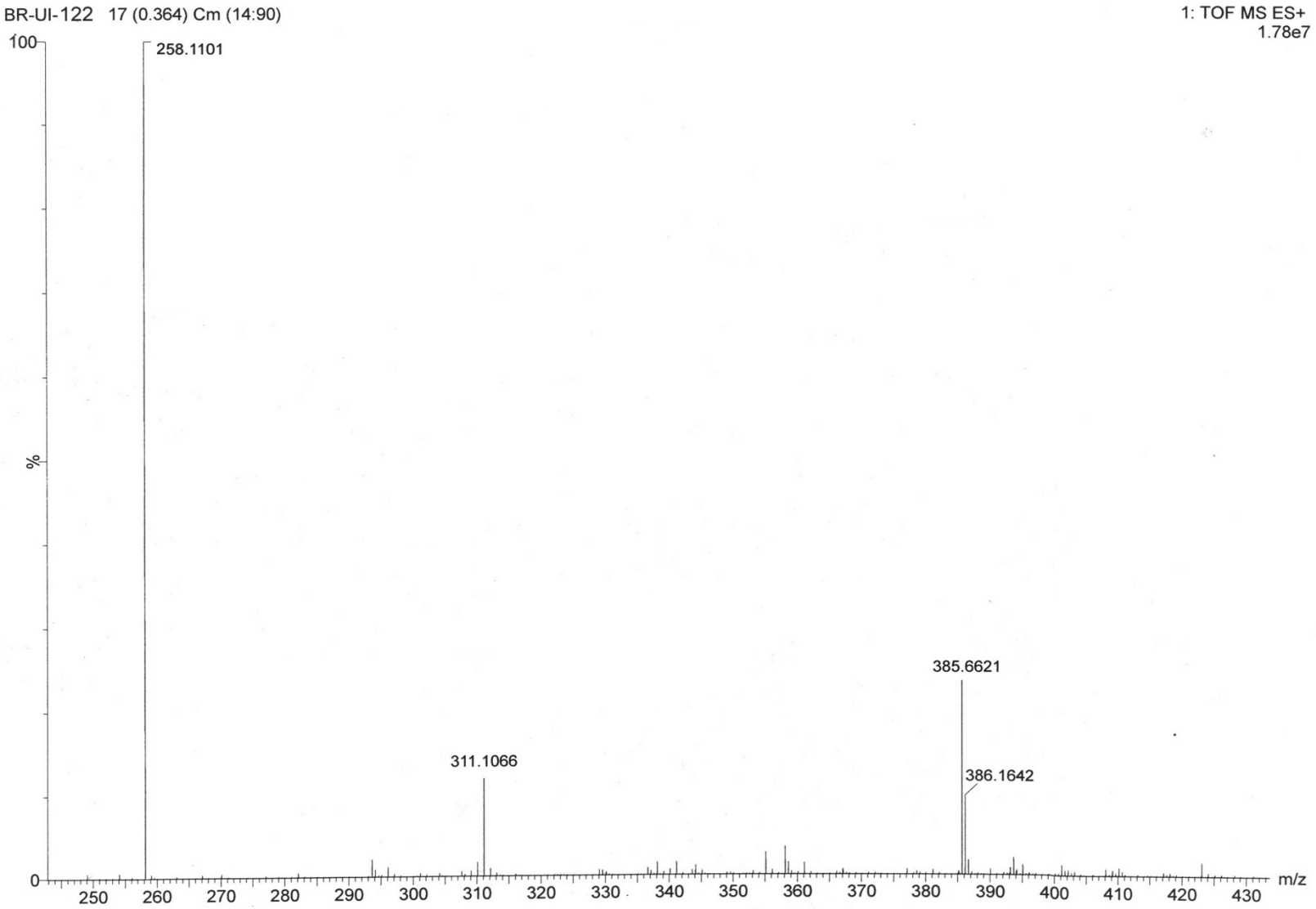

BR-RC-14S
03.11.2014
169.94
169.03
143.39
129.77
128.12
127.65
77.62
77.30
76.98
60.92
50.45
37.50
22.67
13.98
11.80
ppm


BR-MS-9 39 (0.775) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (39)
1: TOF MS ES+
306
100
%
0
272.1267
263.9596
264.8907
276.7502
279.3840
279.4283
260
261
262
263
264
265
266
267
268
269
270
271
272
273
274
275
276
277
278
279
280
m/z

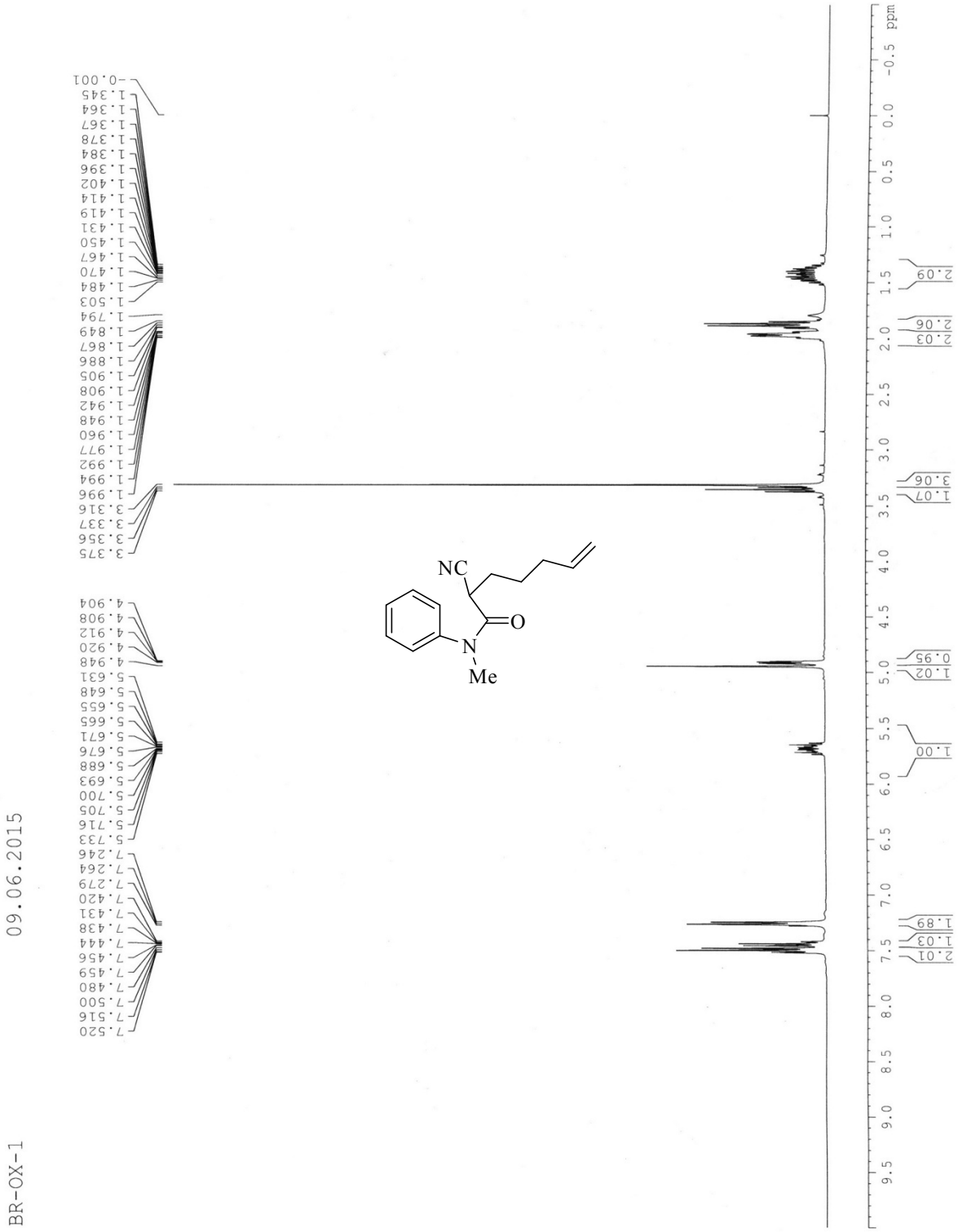
Ethyl 2-methyl-3-(methyl(phenyl)amino)-3-oxopropanoate (1p):




BR-RC-13S
03.11.2014
170.68
170.09
143.44
129.88
128.20
127.42
77.58
77.26
76.94
61.06
43.44
37.55
14.05
13.99
ppm

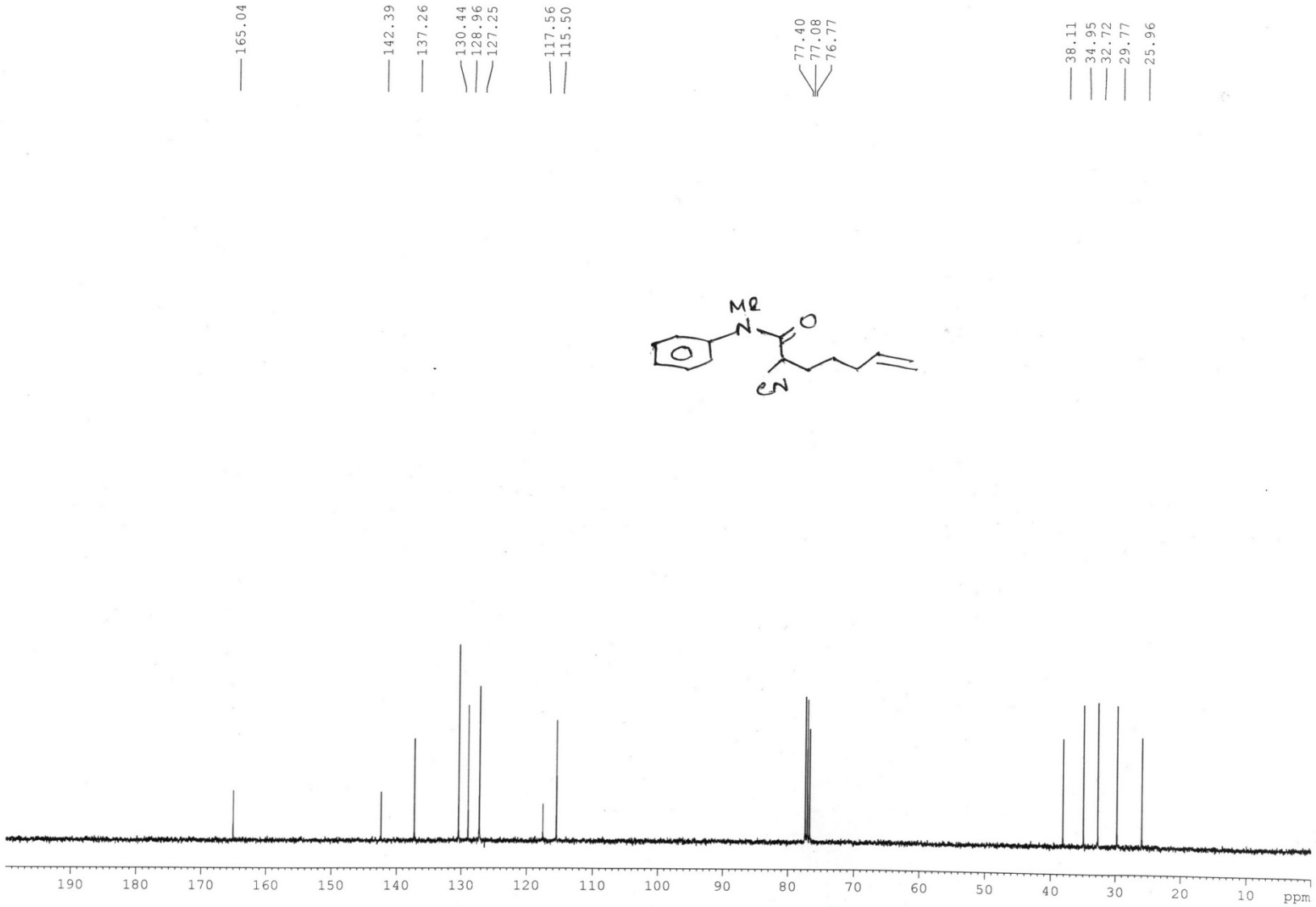

BR-UI-122 17 (0.364) Cm (14:90)
1: TOF MS ES+
1.78e7
100
%
0
258.1101
311.1066
385.6621
386.1642
250
260
270
280
290
300
310
320
330
340
350
360
370
380
390
400
410
420
430
m/z

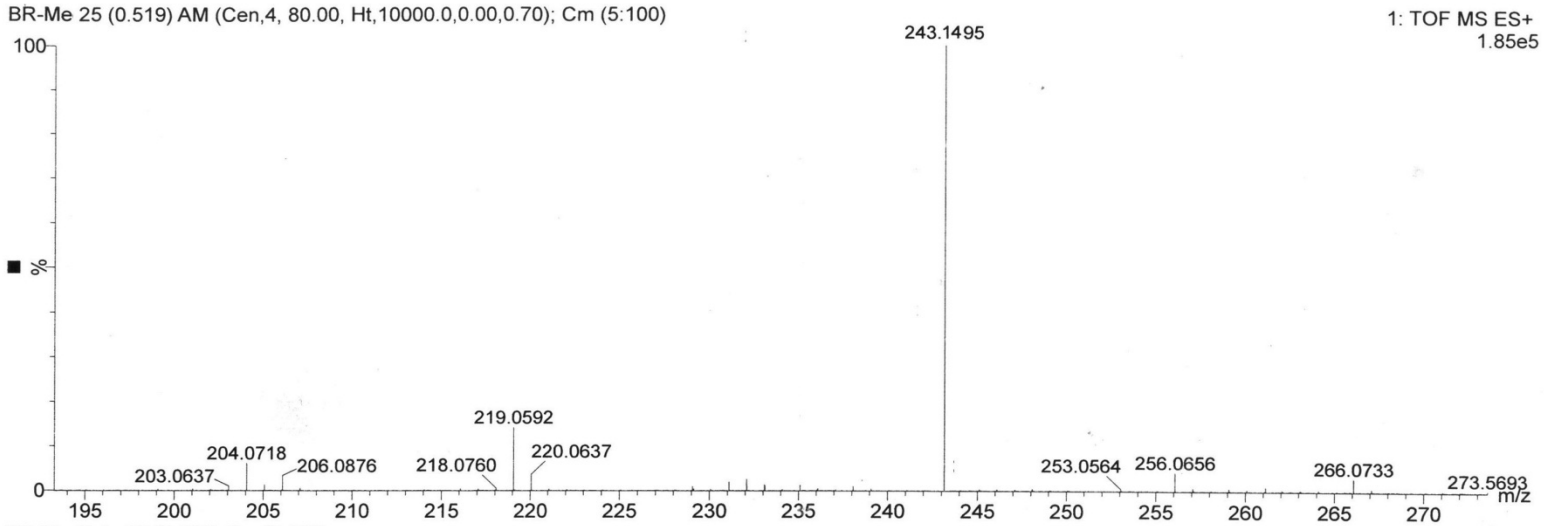
2-Cyano-N-methyl-N-phenylhept-6-enamide (1q):

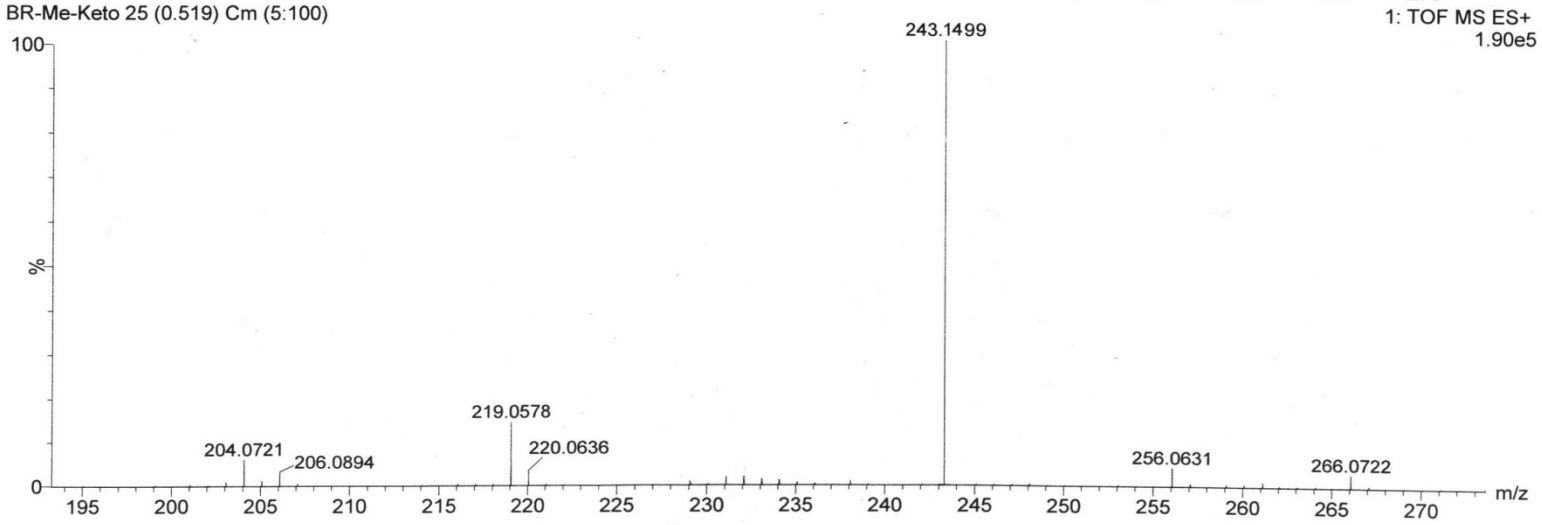


BR-OX-1

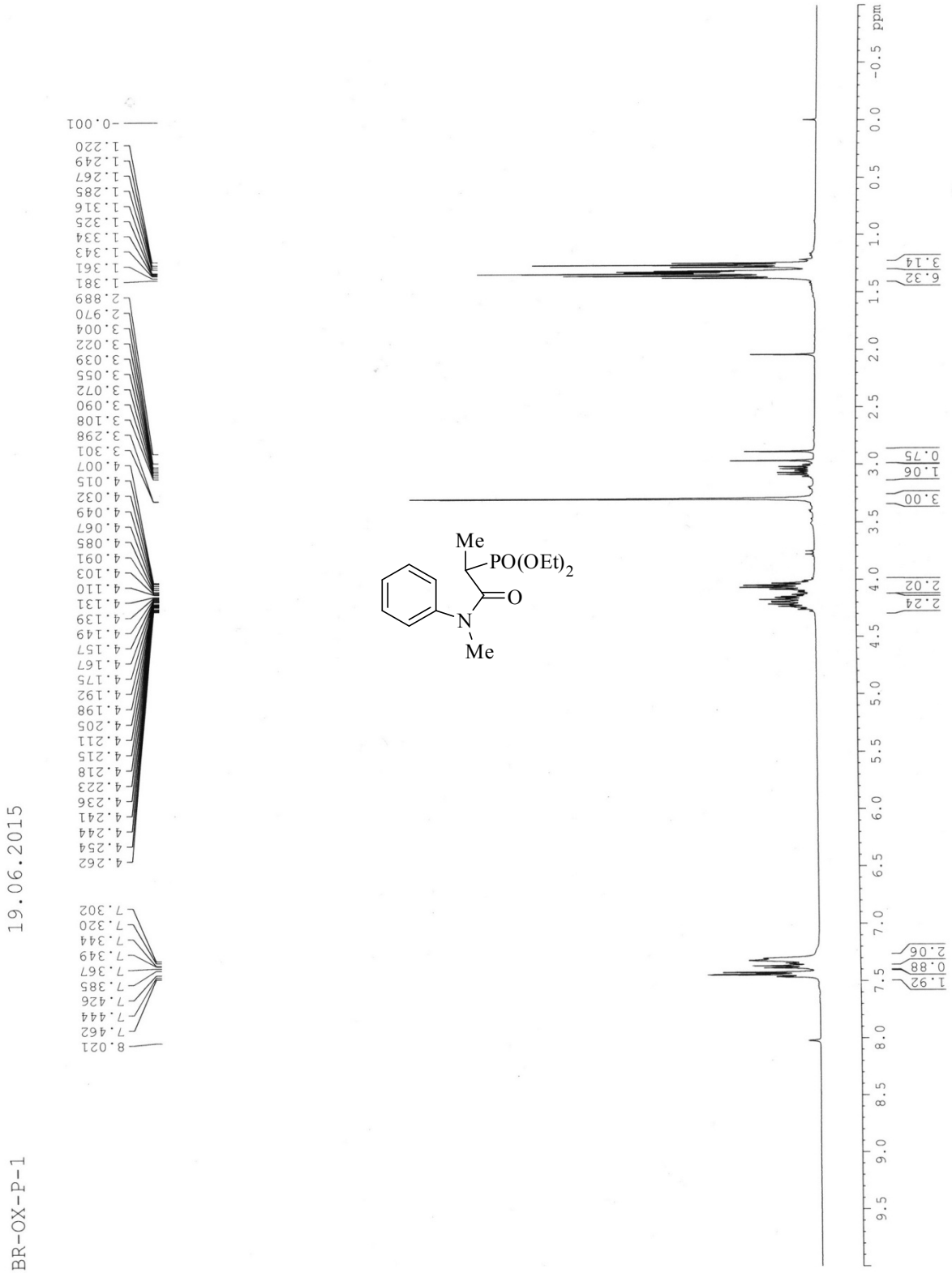
09.06.2015

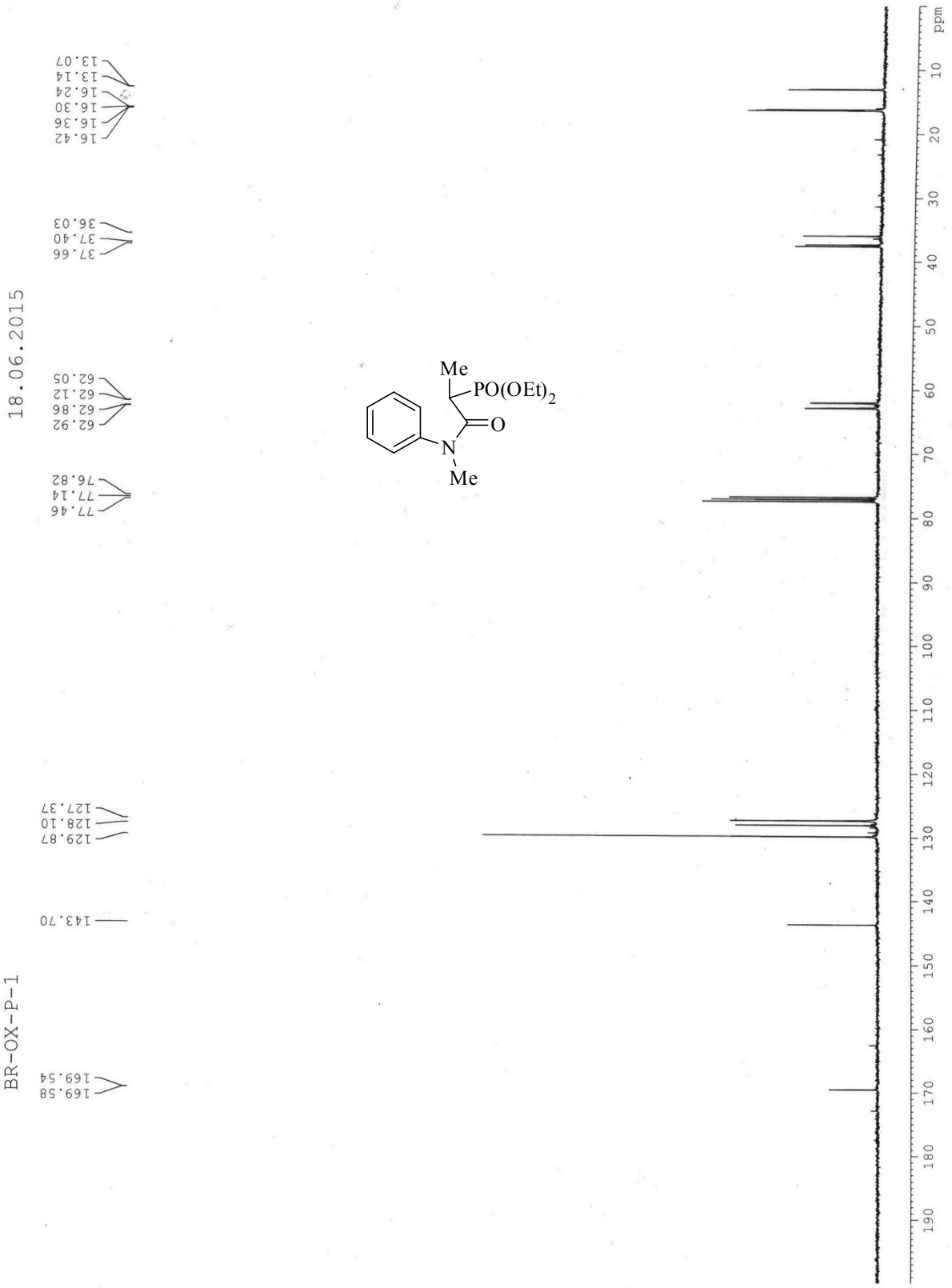


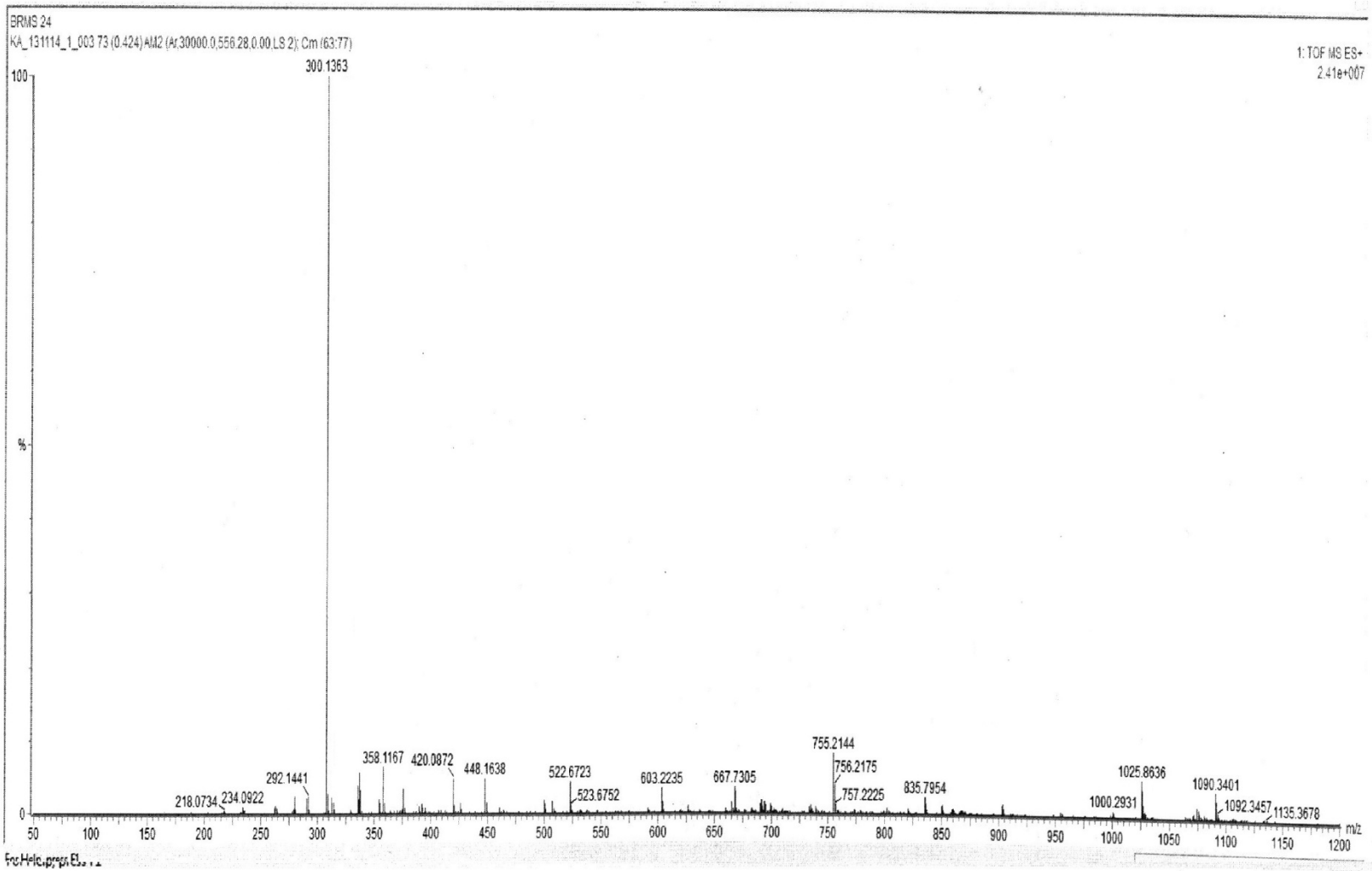

BR-Me 25 (0.519) AM (Cen,4, 80.00, Ht,10000.0,0.00,0.70); Cm (5:100)
1: TOF MS ES+
1.85e5
243.1495
219.0592
204.0718
206.0876
203.0637
218.0760
220.0637
253.0564
256.0656
266.0733
273.5693
100
%
0
195 200 205 210 215 220 225 230 235 240 245 250 255 260 265 270
m/z


BR-Me-Keto 25 (0.519) Cm (5:100)
1: TOF MS ES+
1.90e5
243.1499
219.0578
204.0721
206.0894
220.0636
256.0631
266.0722
100
%
0
195 200 205 210 215 220 225 230 235 240 245 250 255 260 265 270
m/z

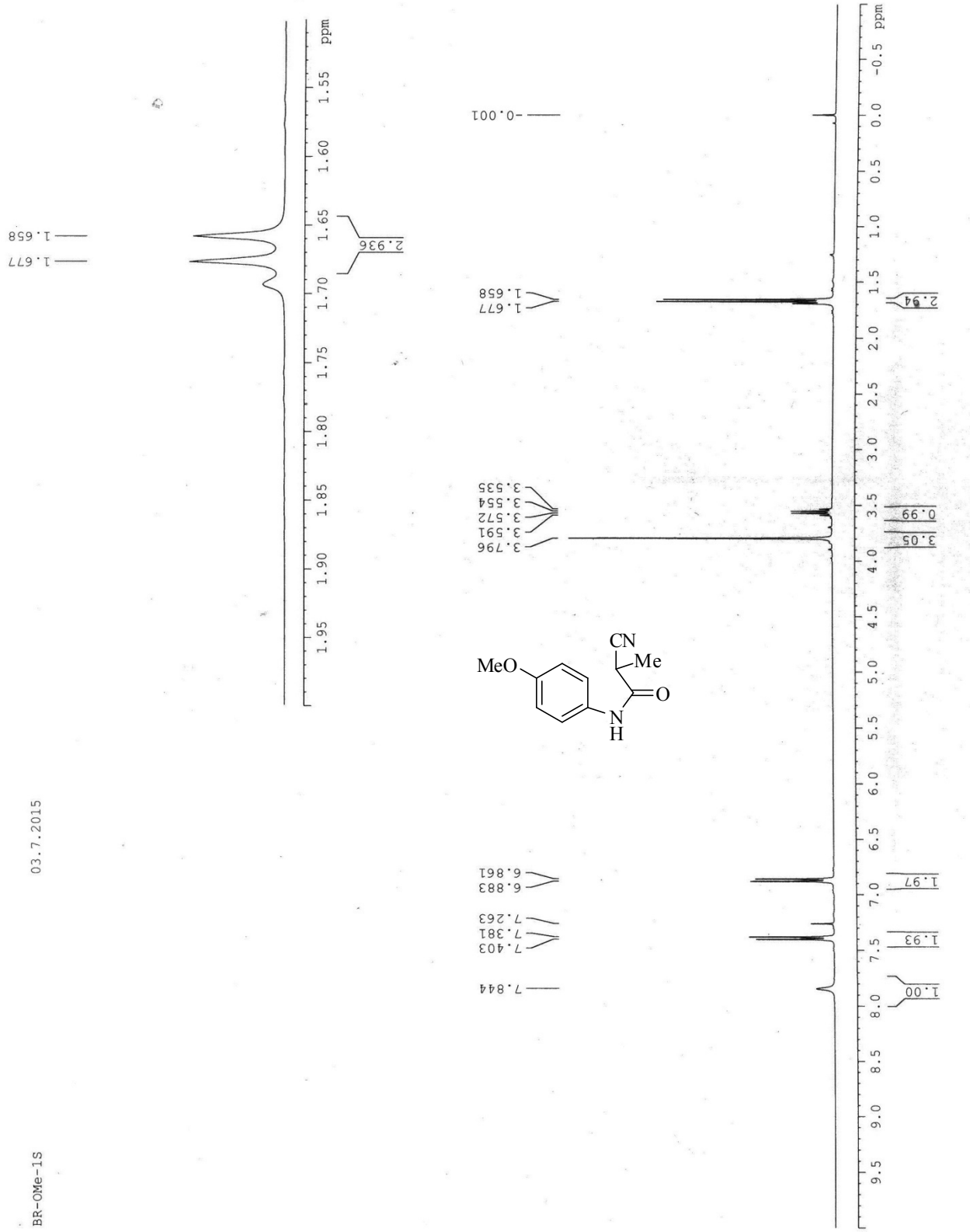
Diethyl 1-(methyl(phenyl)amino)-1-oxopropan-2-ylphosphonate (1r):

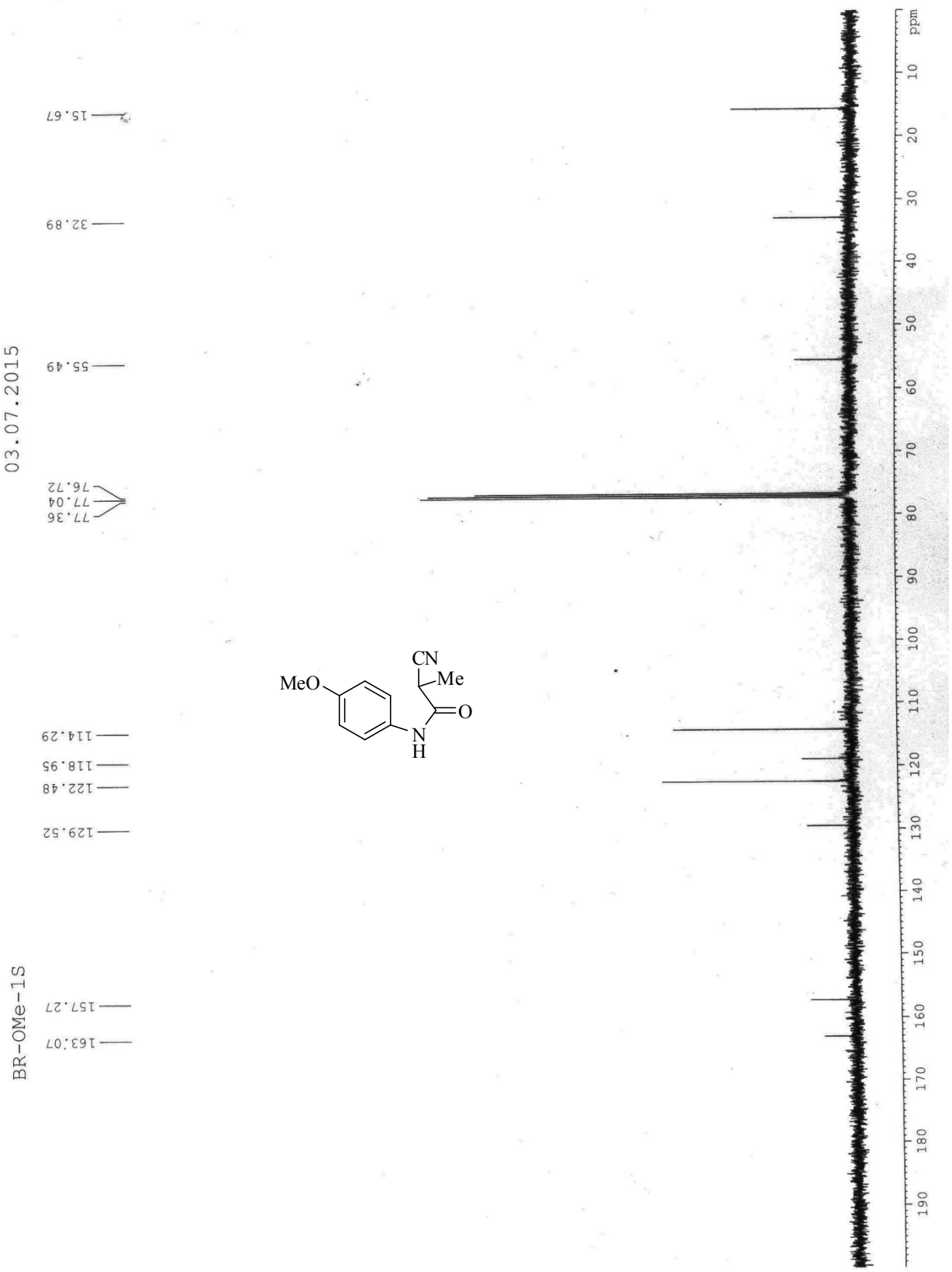



BR-OX-P-1
18.06.2015
169.58
169.54
143.70
129.87
128.10
127.37
77.46
77.14
76.82
62.92
62.86
62.12
62.05
37.66
37.40
36.03
16.42
16.36
16.30
16.24
13.14
13.07
Me
PO(OEt)2
O
N
Me
ppm


BRMS 24
KA_131114_1_003 73 (0.424) AM2 (Ar,30000.0,556.28,0.00,LS 2); Cm (63:77)
1: TOF MS ES+
2.41e+007
100
%
0
300.1363
218.0734
234.0922
292.1441
358.1167
420.0872
448.1638
522.6723
523.6752
603.2235
667.7305
755.2144
756.2175
757.2225
835.7954
1000.2931
1025.8636
1090.3401
1092.3457
1135.3678
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
950
1000
1050
1100
1150
1200
m/z

2-Cyano-N-(4-methoxyphenyl)propanamide (1s):

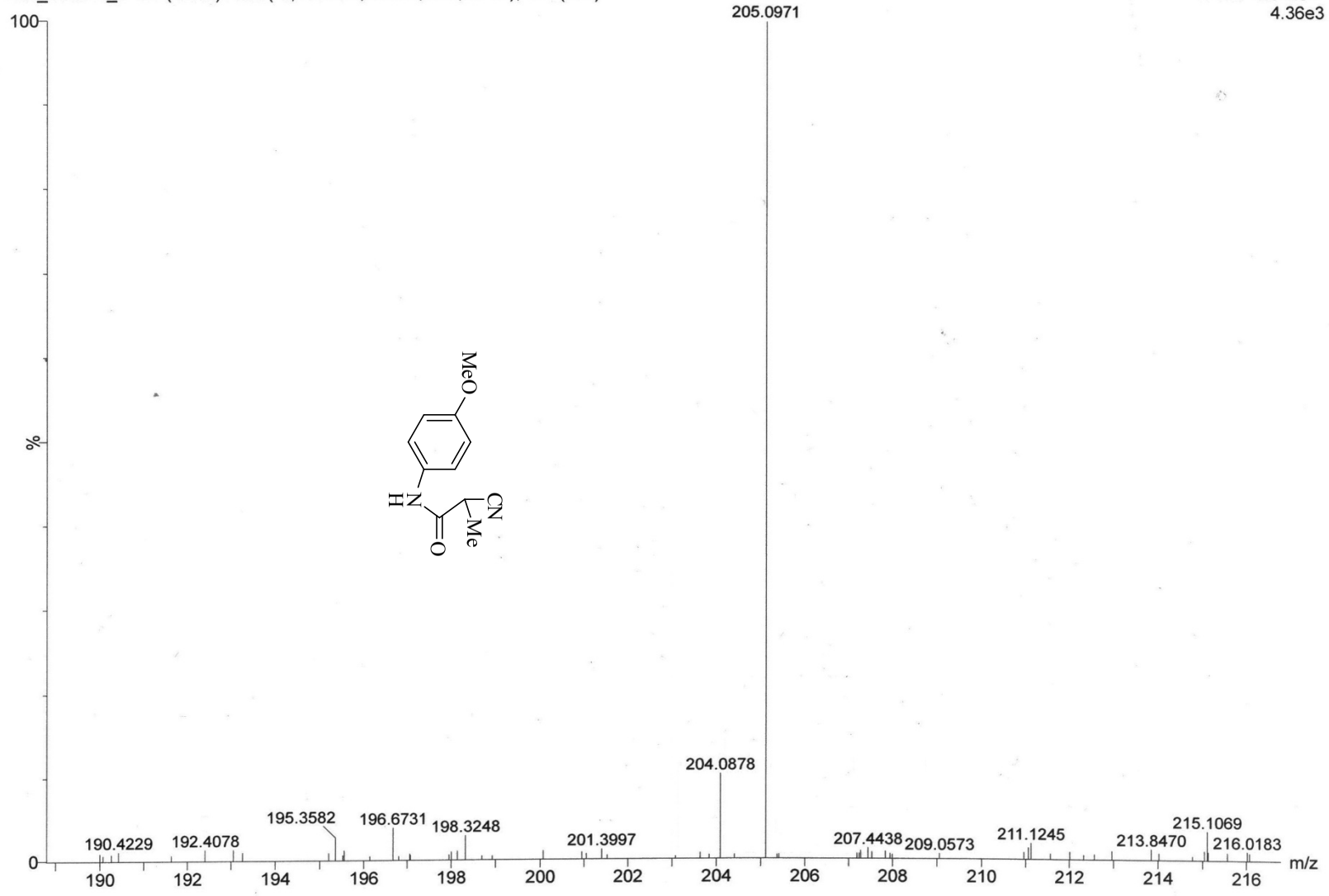



BR-OMe-1S
03.07.2015
163.07
157.27
129.52
122.48
118.95
114.29
77.36
77.04
76.72
55.49
32.89
15.67
MeO
CN
Me
O
N
H
ppm

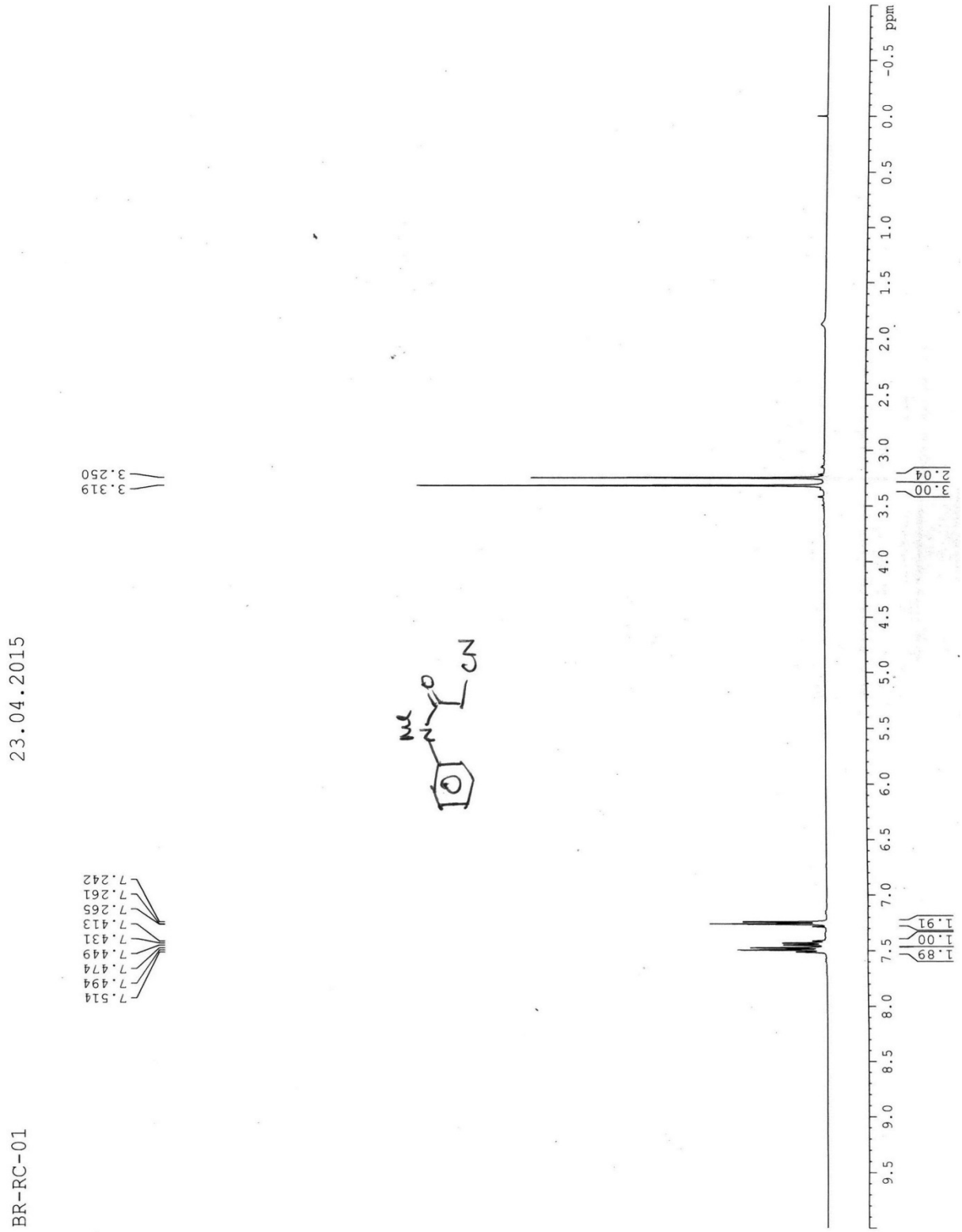
BR_MS_34

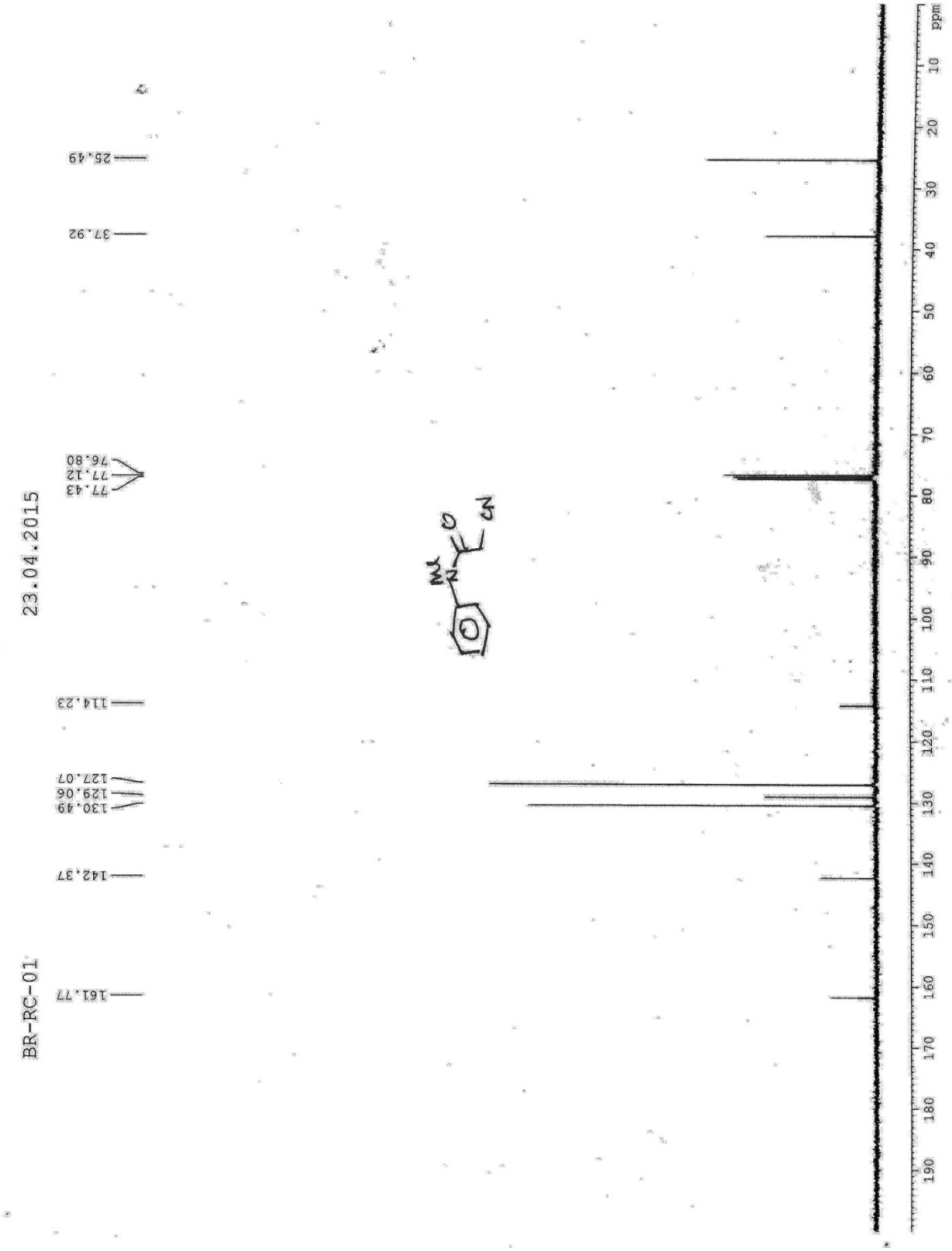
KLY_100215_6 107 (1.940) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (107)

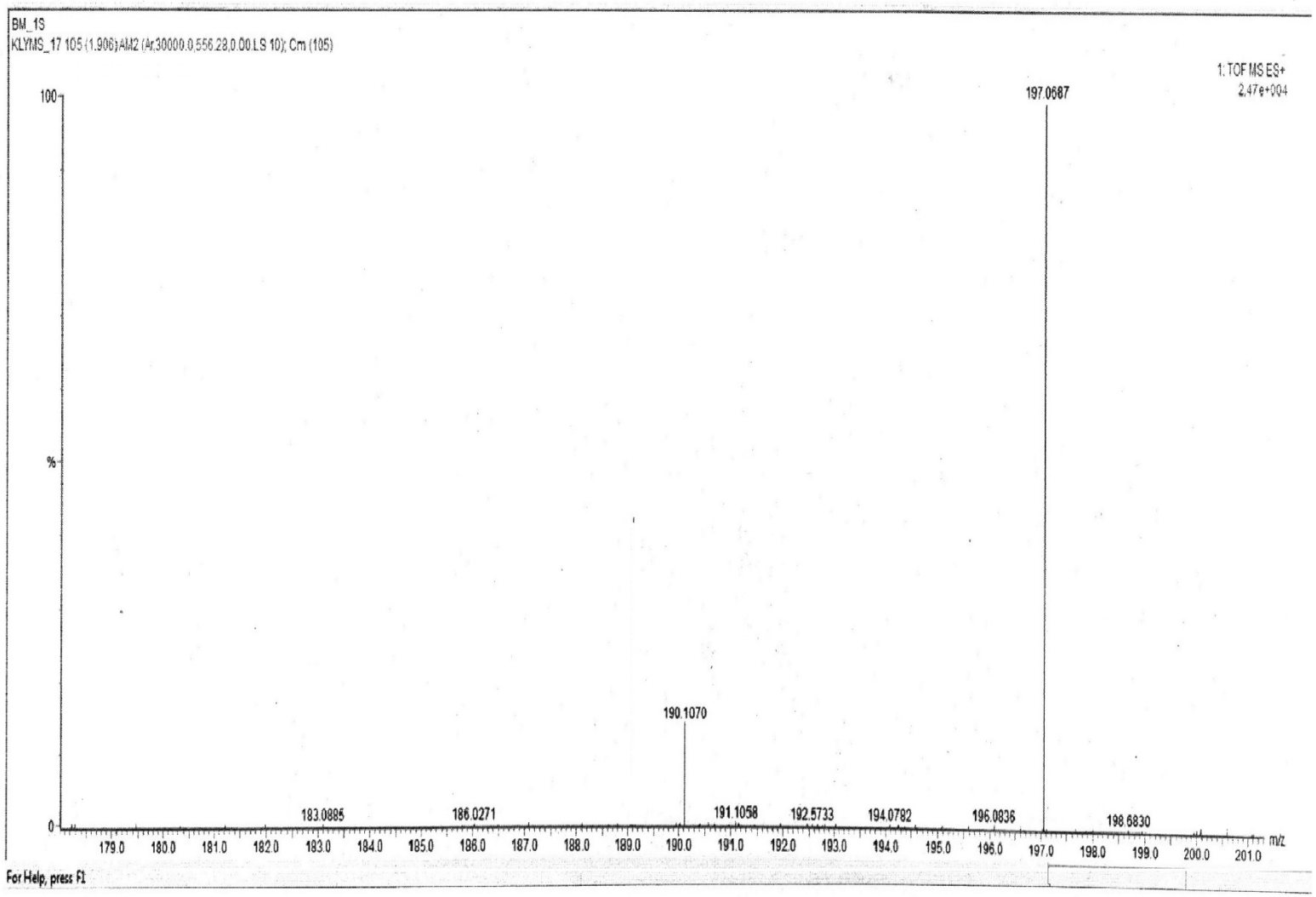
1: TOF MS ES+
4.36e3



2-Cyano-N-methyl-N-phenylacetamide (1a'):

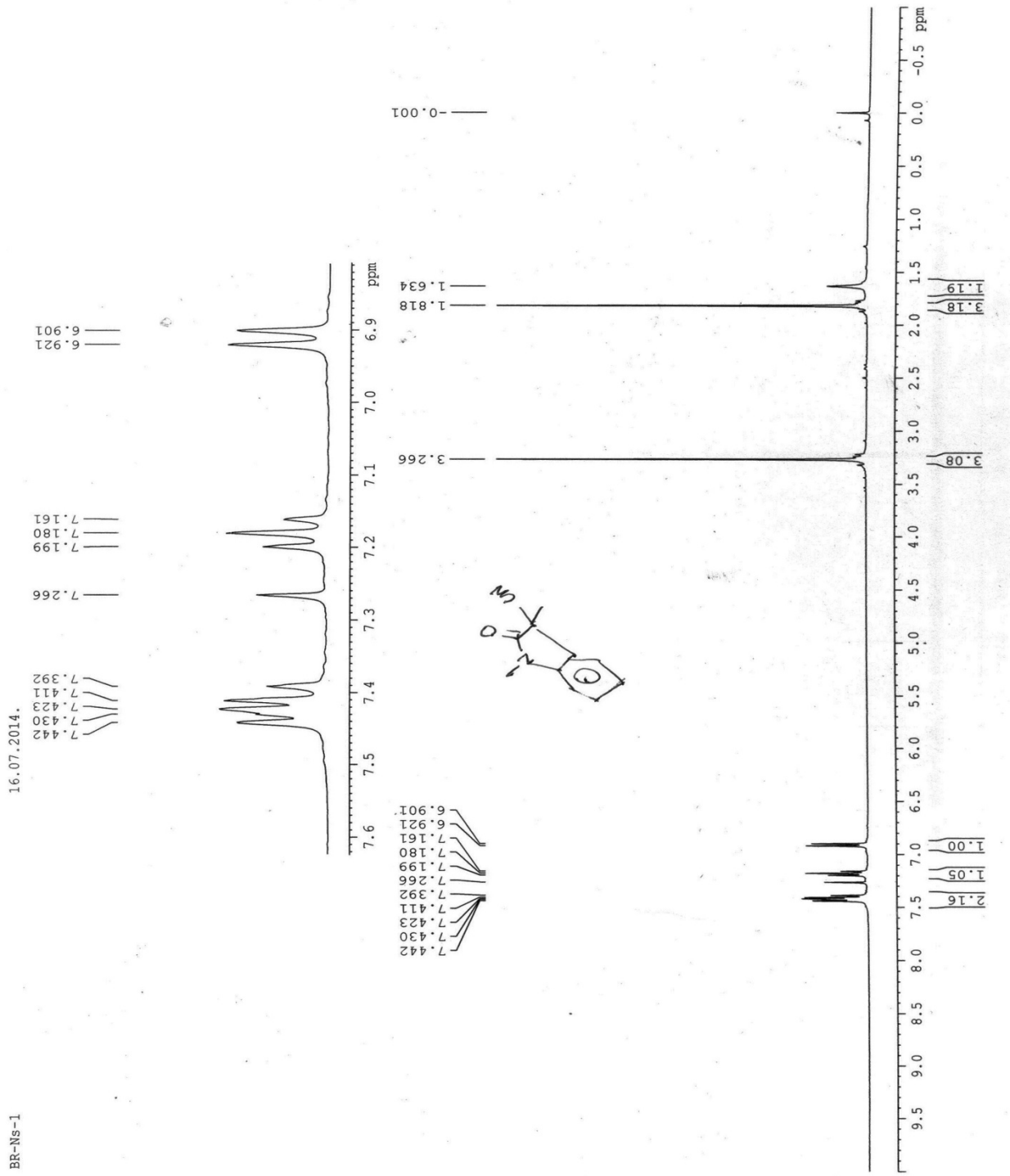


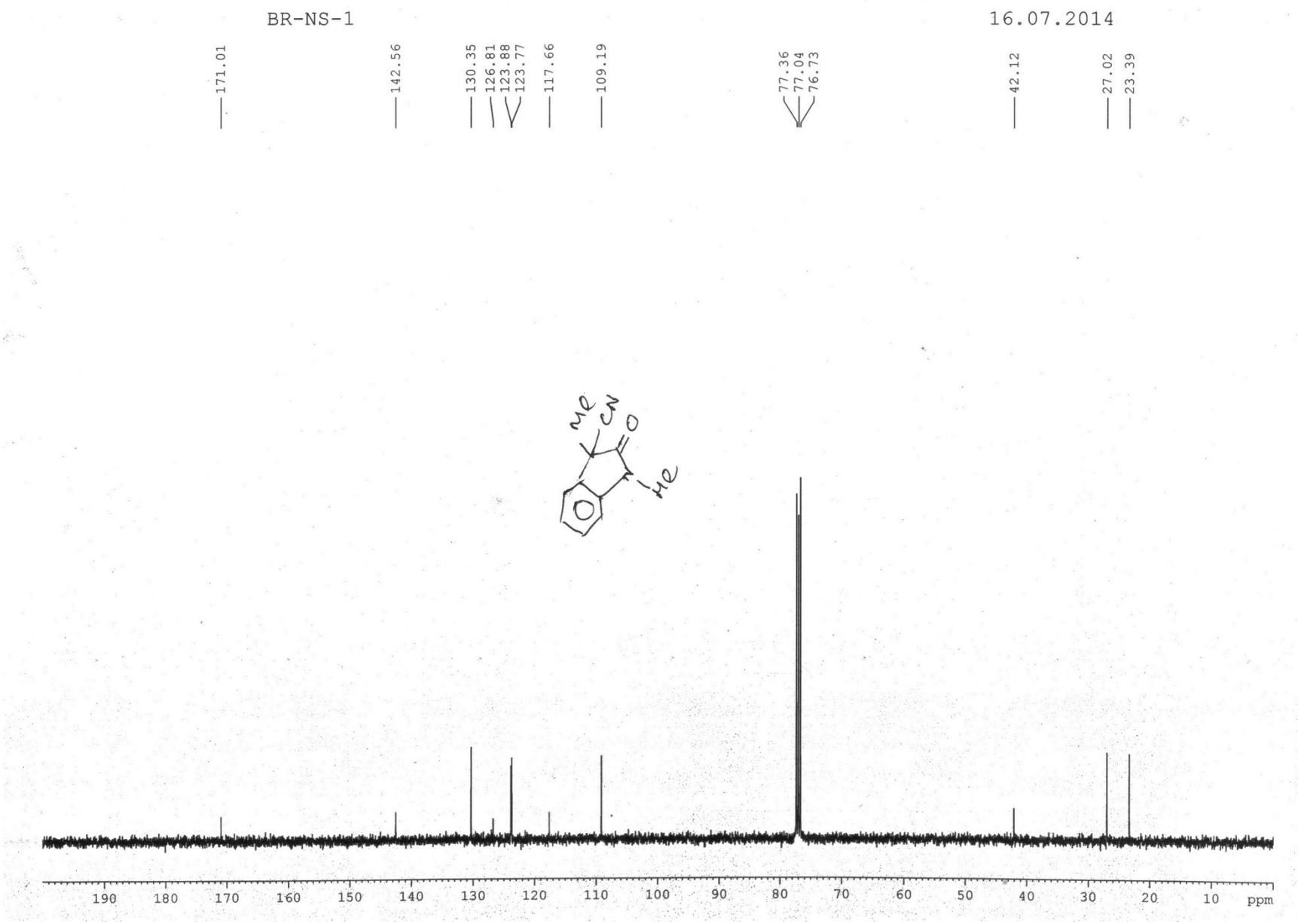

BR-RC-01
23.04.2015
161.77
142.37
130.49
129.06
127.07
114.23
77.43
77.12
76.80
37.92
25.49
ppm

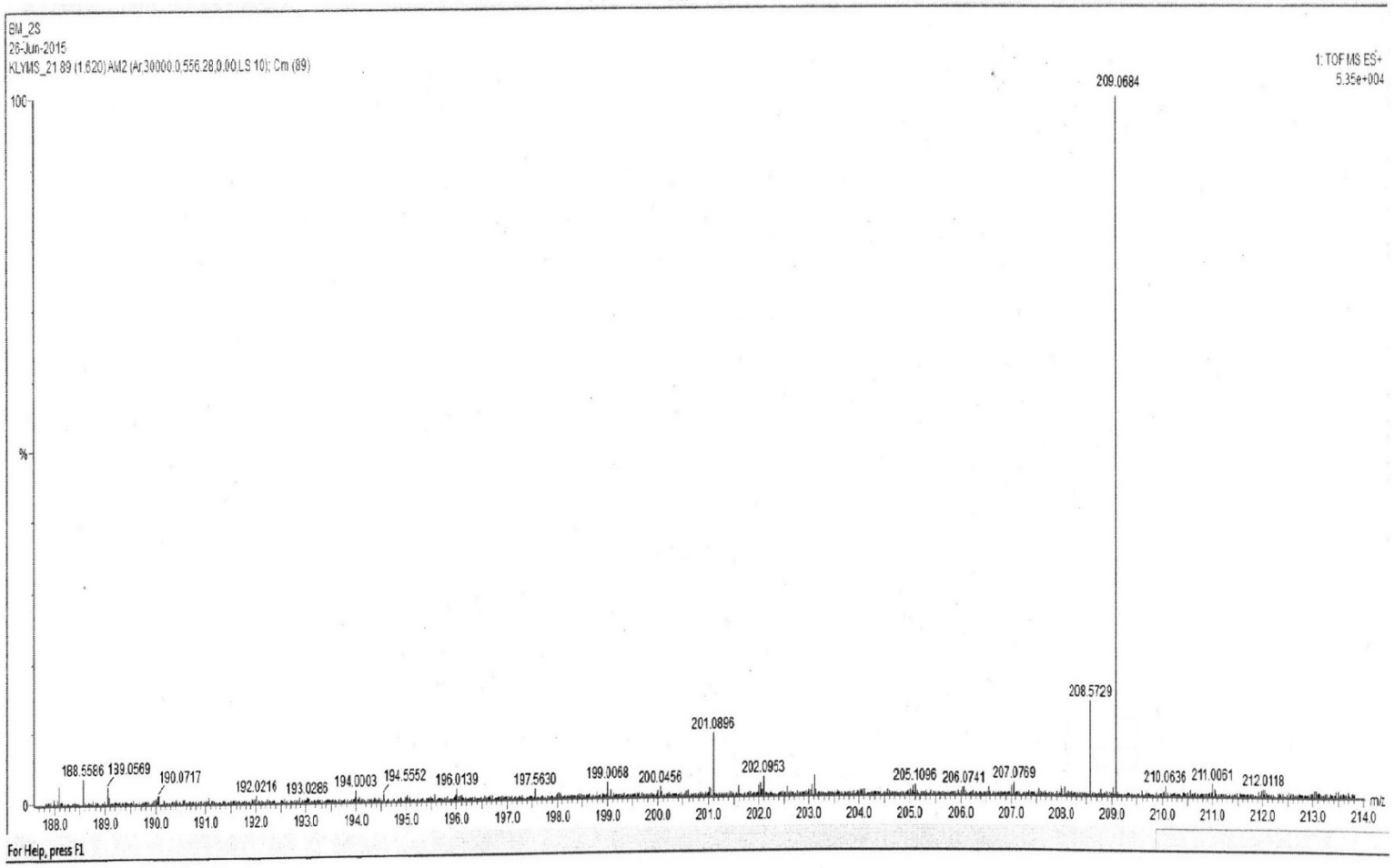

BM_1S
KLYNS_17 105 (1.906);AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (105)
1: TOF MS ES+
2.47e+004
100
%
0
197.0687
190.1070
183.0885
186.0271
191.1058
192.5733
194.0782
196.0836
198.6830
179.0
180.0
181.0
182.0
183.0
184.0
185.0
186.0
187.0
188.0
189.0
190.0
191.0
192.0
193.0
194.0
195.0
196.0
197.0
198.0
199.0
200.0
201.0
m/z
For Help, press F1

Copies of NMR (^{1}H & ^{13}C) spectra of products:

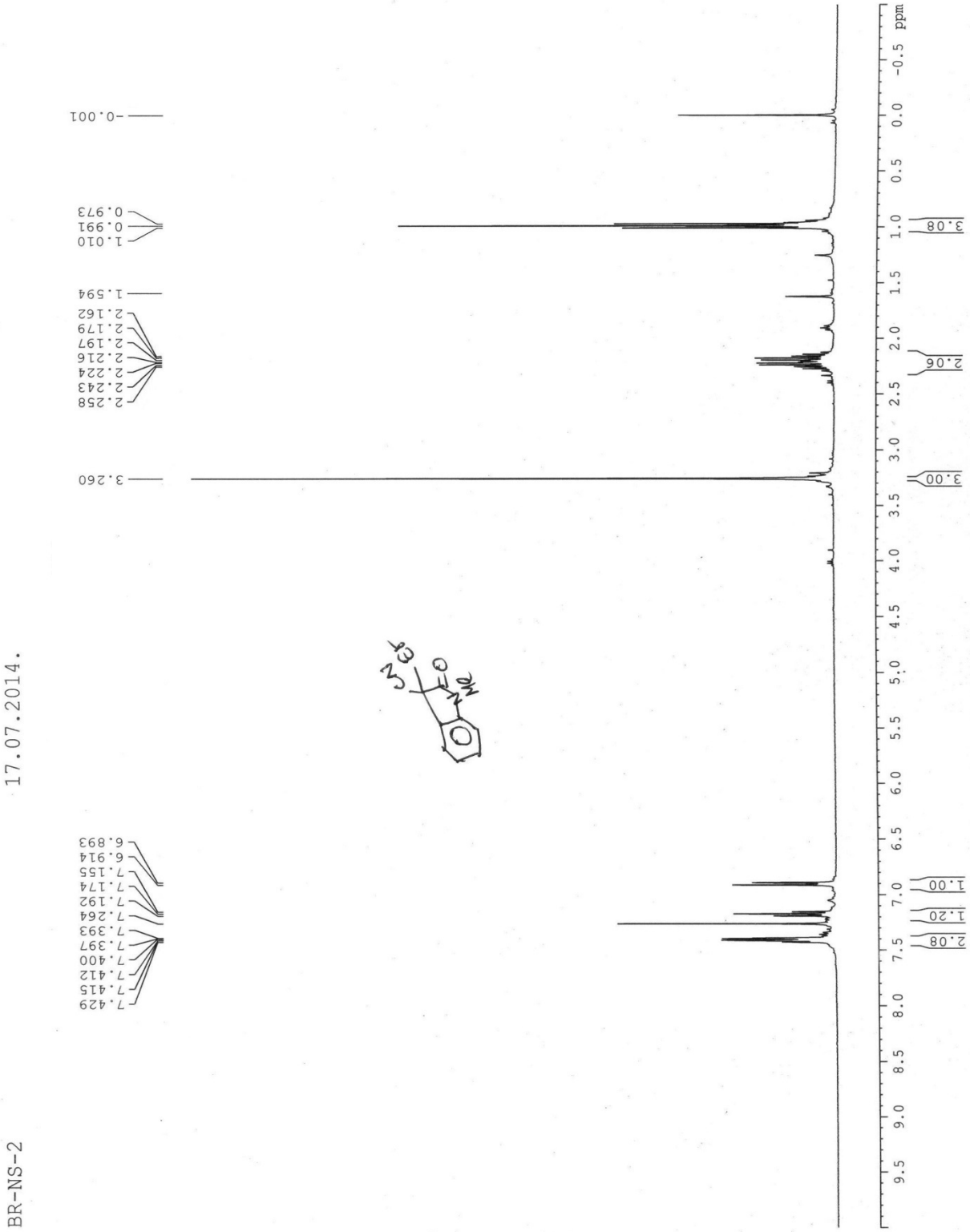
1,3-Dimethyl-2-oxoindoline-3-carbonitrile (2a):



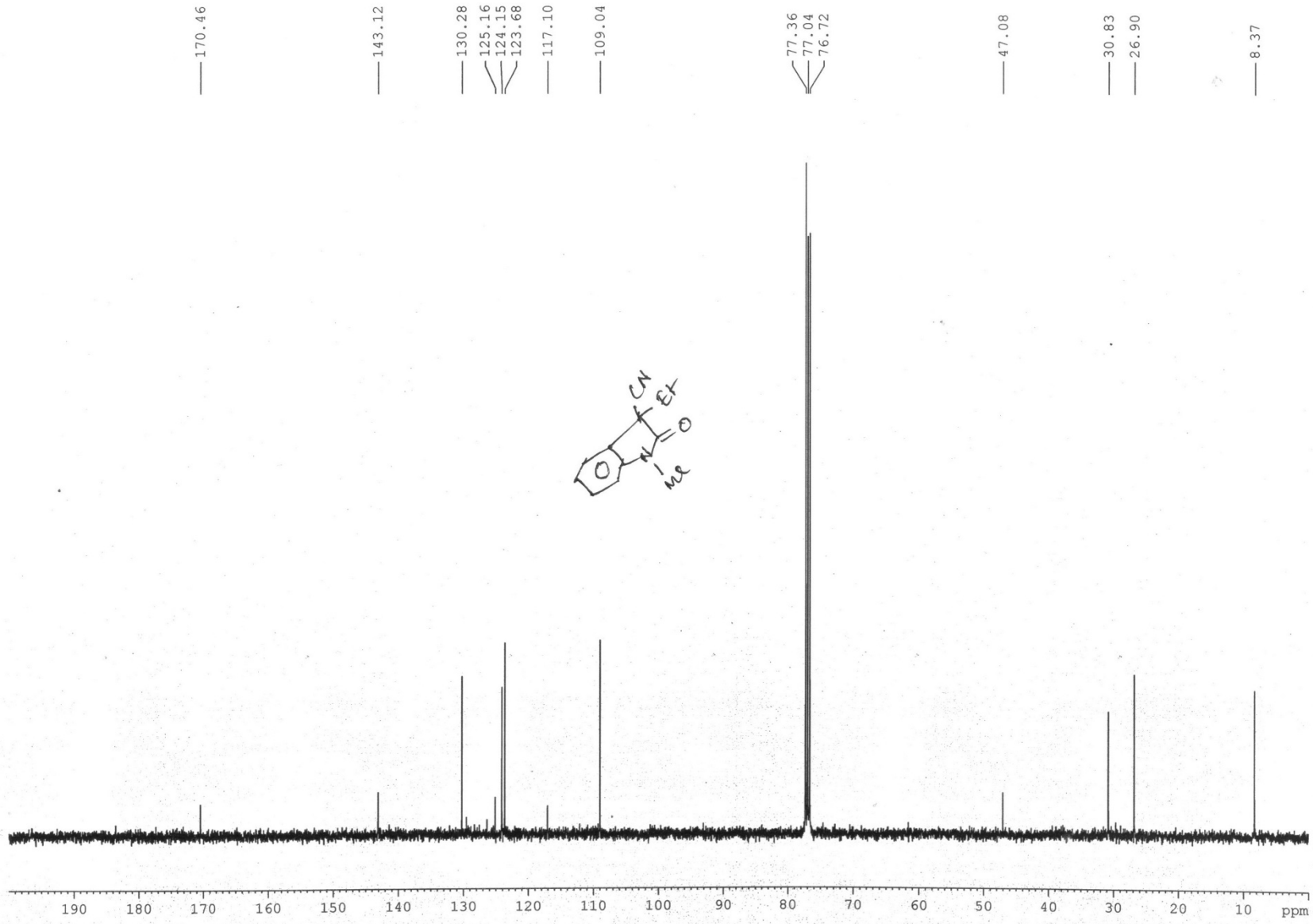

BR-NS-1
16.07.2014
171.01
142.56
130.35
126.81
123.88
123.77
117.66
109.19
77.36
77.04
76.73
42.12
27.02
23.39
Me
CN
O
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


BM_2S
26-Jun-2015
KLYMS_21 89 (1.620) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (89)
1: TOF MS ES+
5.35e+004
209.0684
208.5729
201.0896
188.5586
189.0569
190.0717
192.0216
193.0286
194.0003
194.5552
196.0139
197.5630
199.0068
200.0456
202.0953
205.1096
206.0741
207.0769
210.0636
211.0061
212.0118
100
%
0
m/z
For Help, press F1

3-Ethyl-1-methyl-2-oxoindoline-3-carbonitrile (2b):



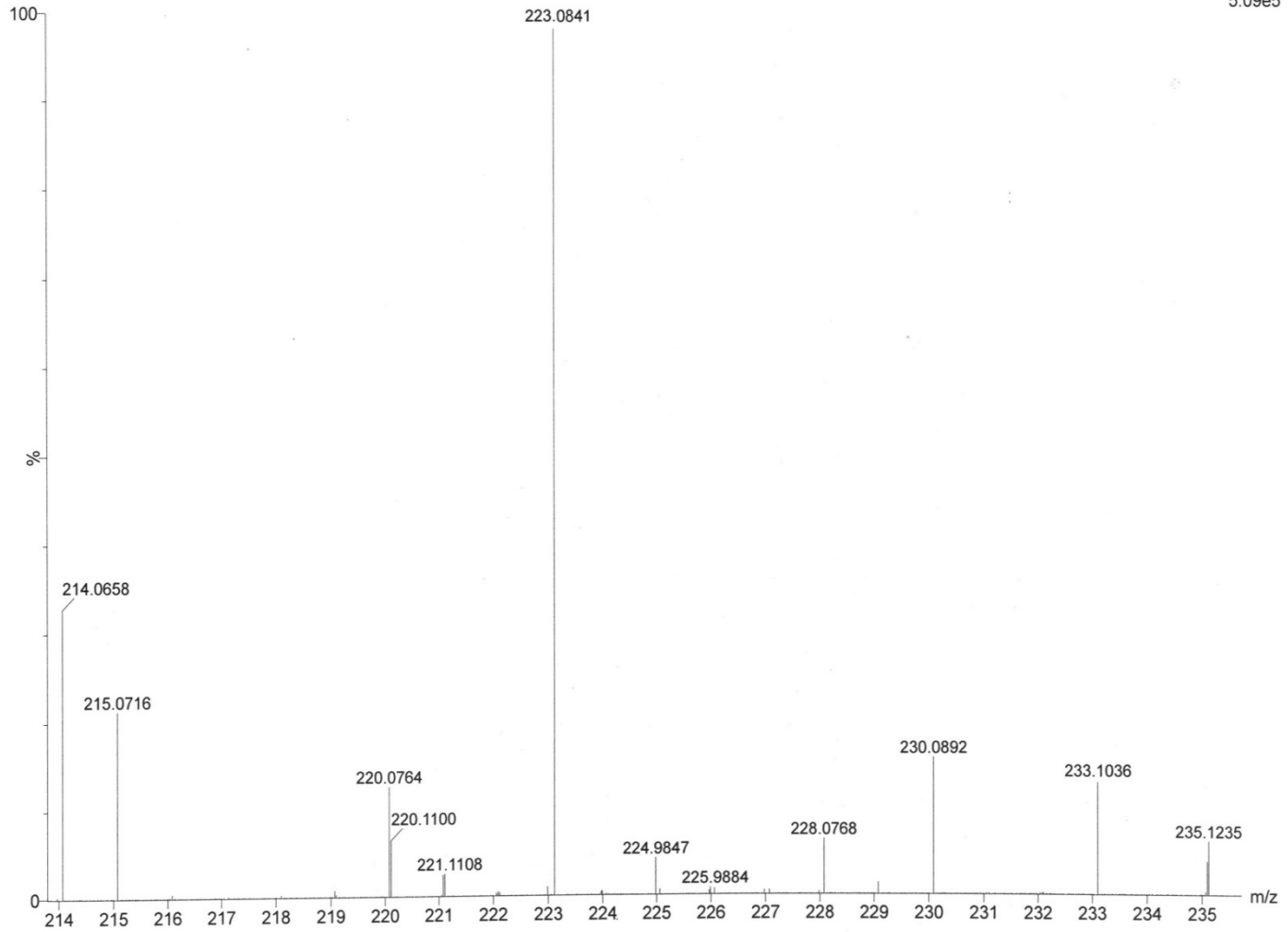
BR-NS-2 18.07.2014



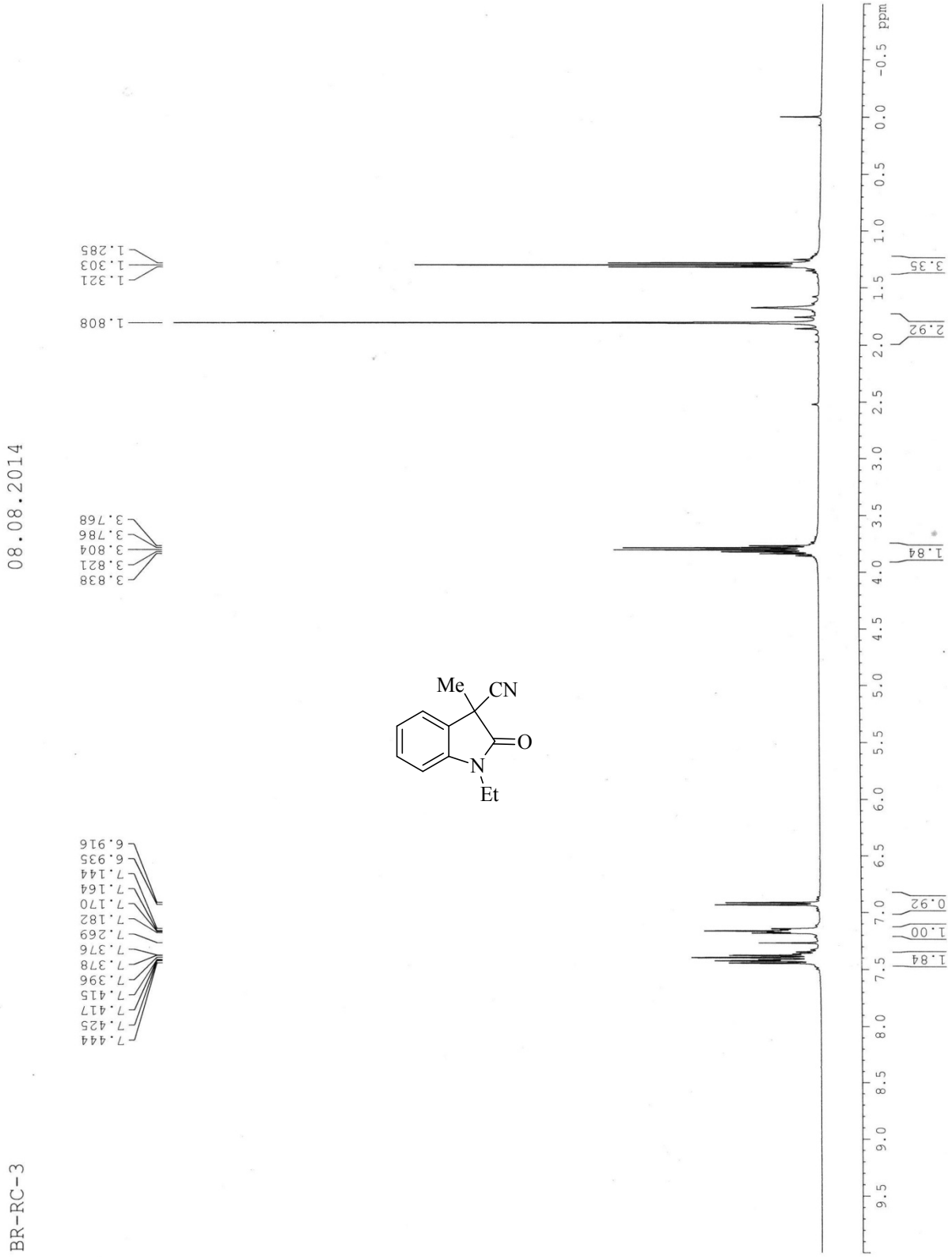
BRMS 213

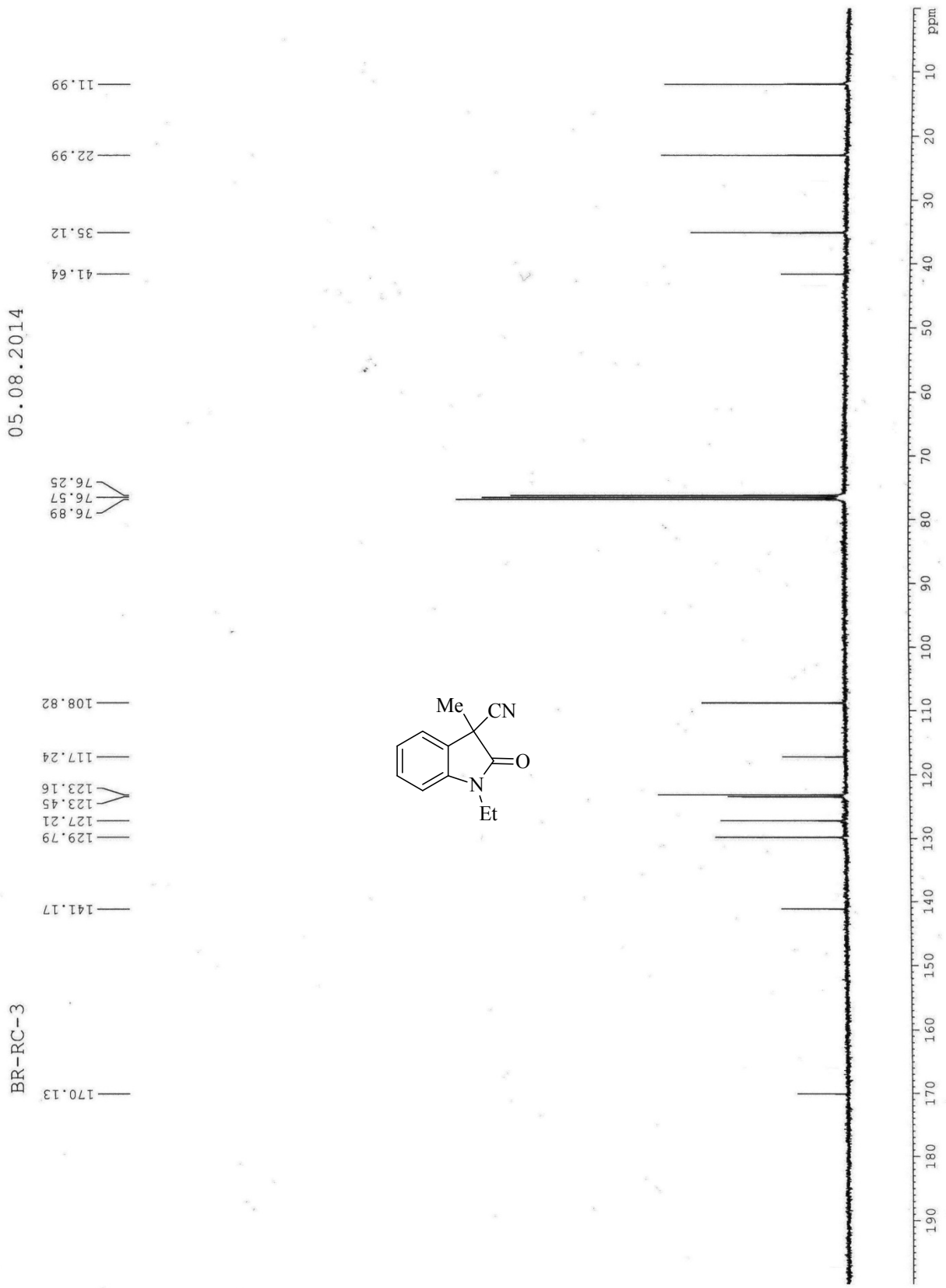
KA_131114_1_001 347 (1.932) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (347:353)

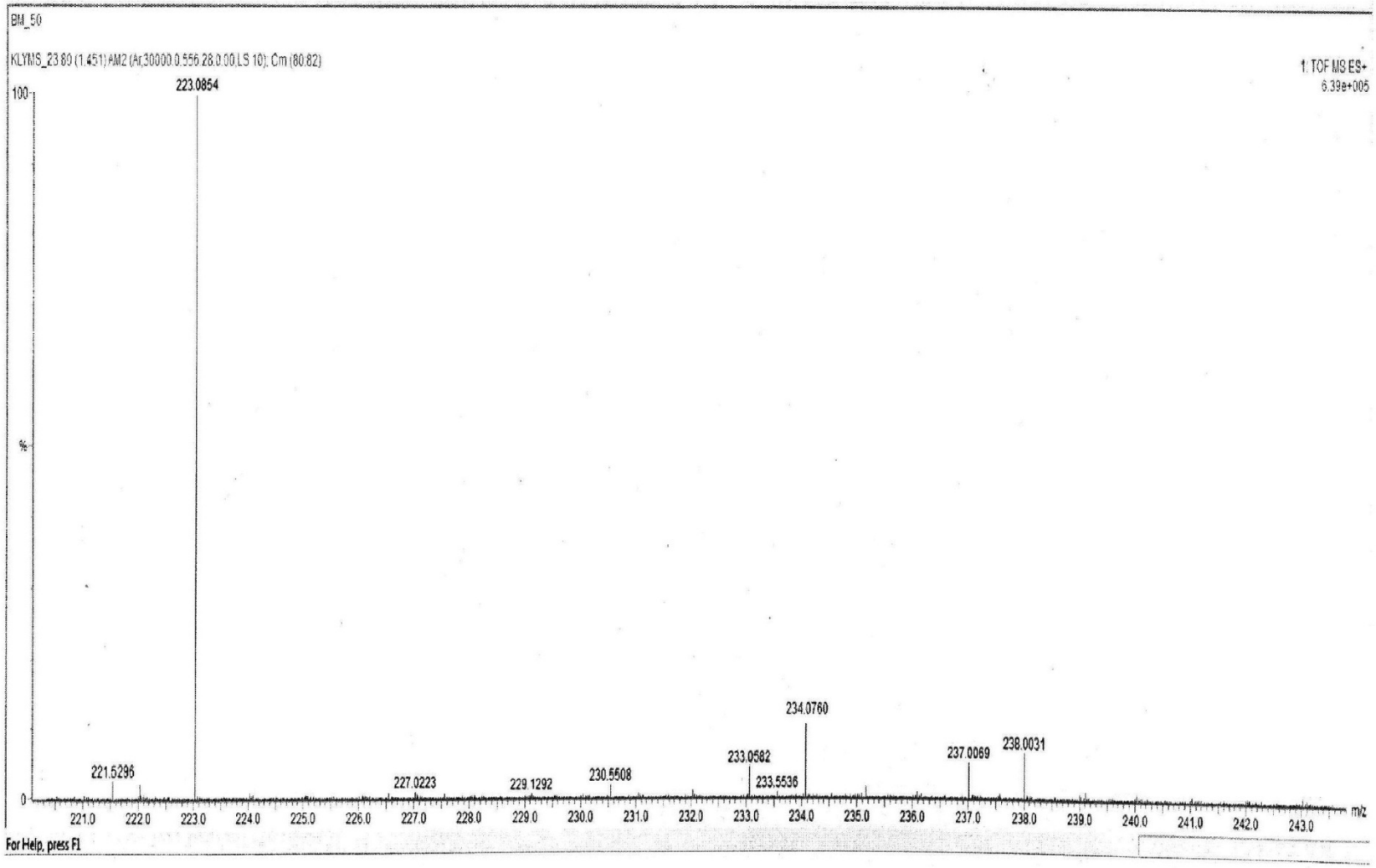
1: TOF MS ES+
5.09e5



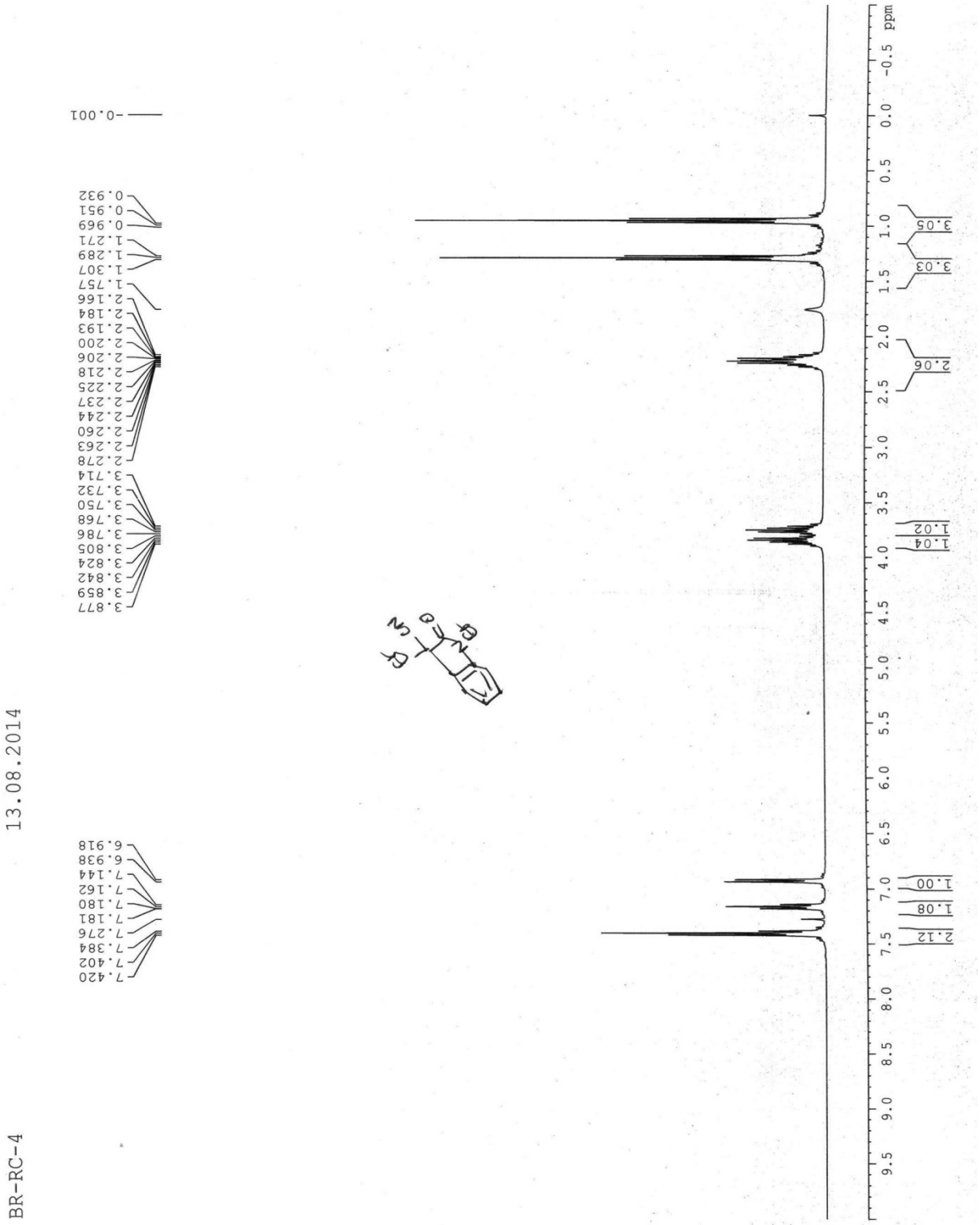
1-Ethyl-3-methyl-2-oxoindoline-3-carbonitrile (2c):

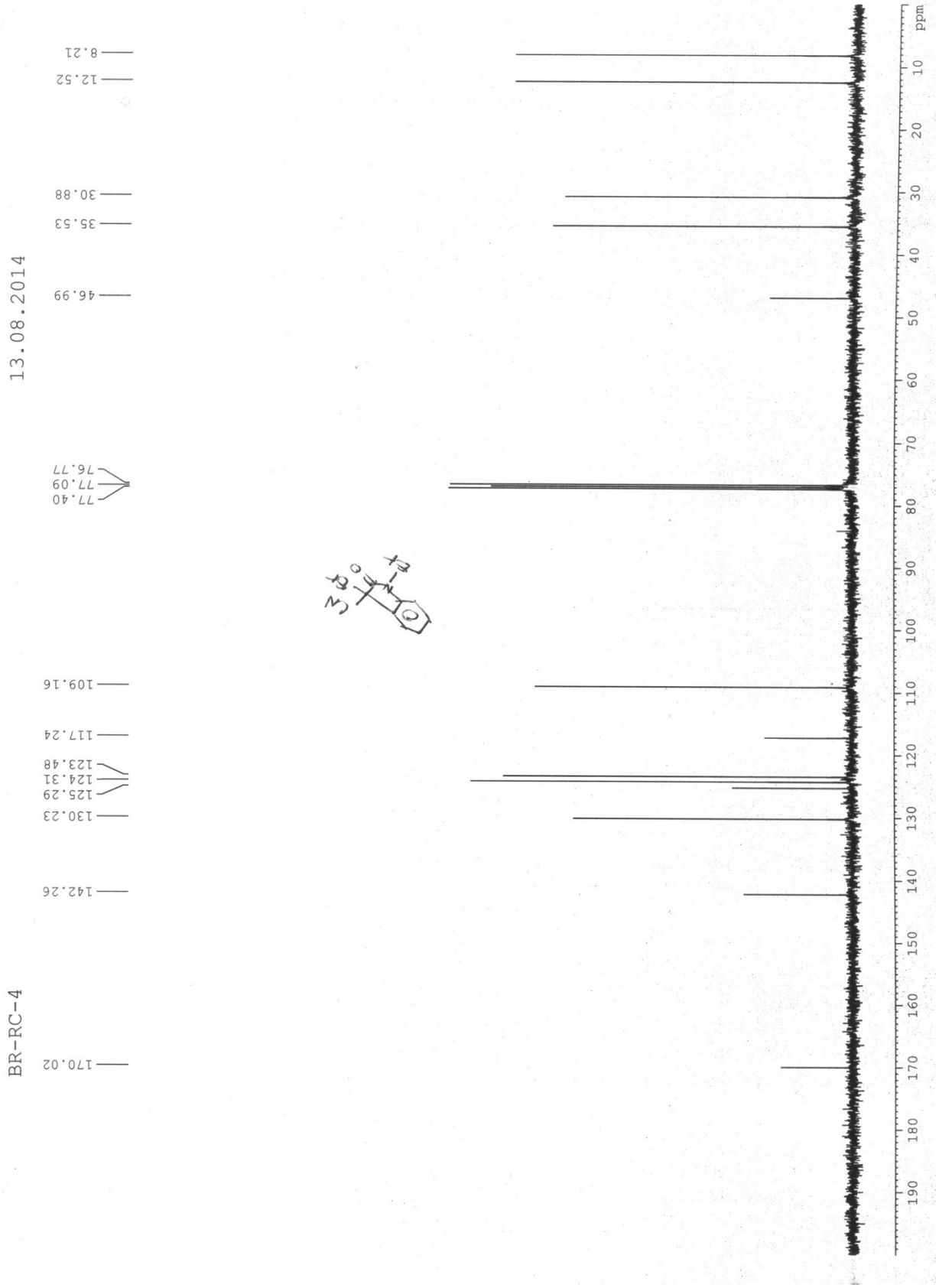


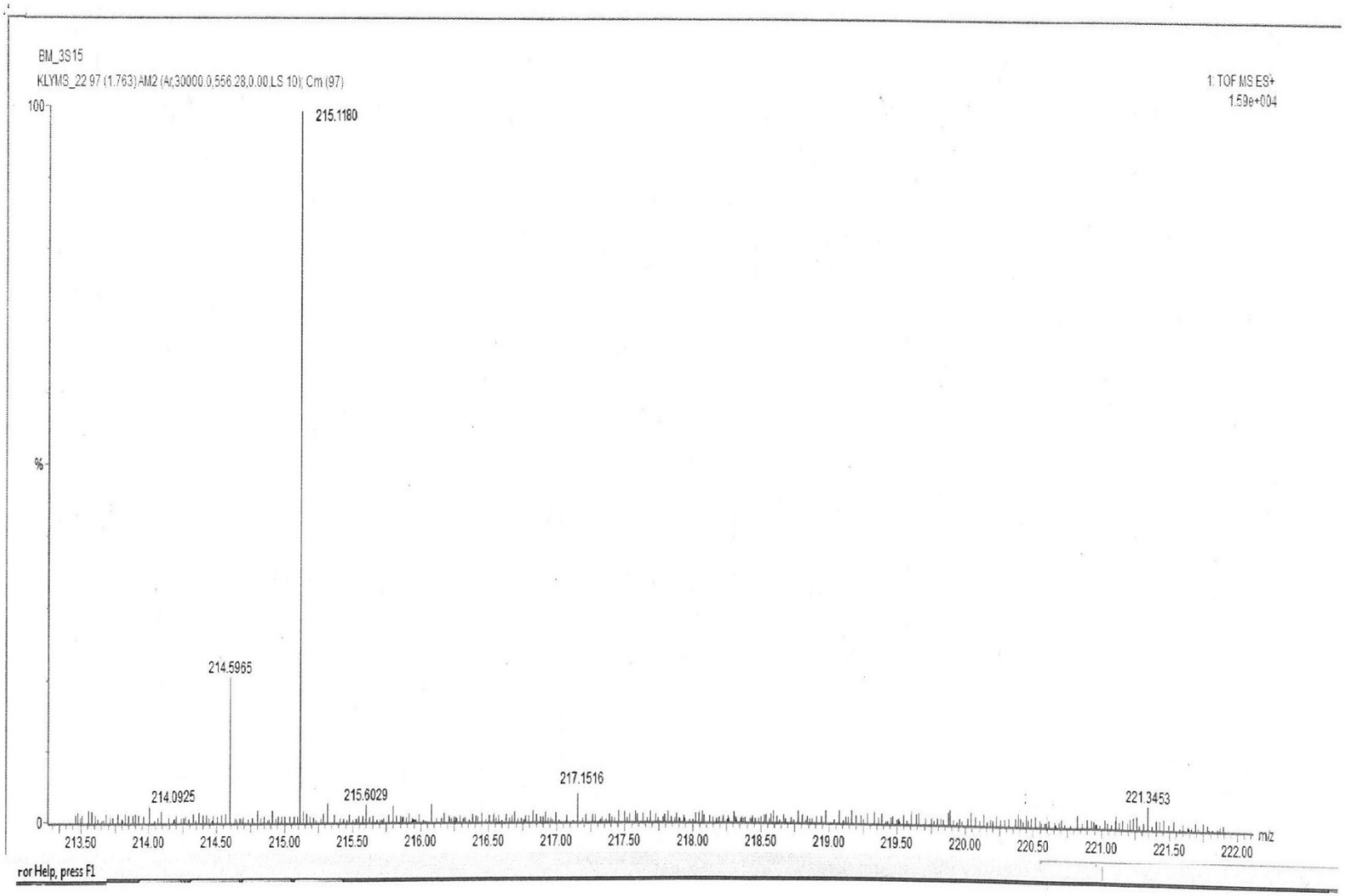

BR-RC-3
05.08.2014
170.13
141.17
129.79
127.21
123.45
123.16
117.24
108.82
76.89
76.57
76.25
41.64
35.12
22.99
11.99
Me
CN
O
N
Et
ppm


BM_50
KLYMS_23 80 (1.451) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (80:82)
1: TOF MS ES+
6.39e+005
100
%
0
223.0854
221.5296
227.0223
229.1292
230.5508
233.0582
233.5536
234.0760
237.0069
238.0031
221.0
222.0
223.0
224.0
225.0
226.0
227.0
228.0
229.0
230.0
231.0
232.0
233.0
234.0
235.0
236.0
237.0
238.0
239.0
240.0
241.0
242.0
243.0
m/z
For Help, press F1

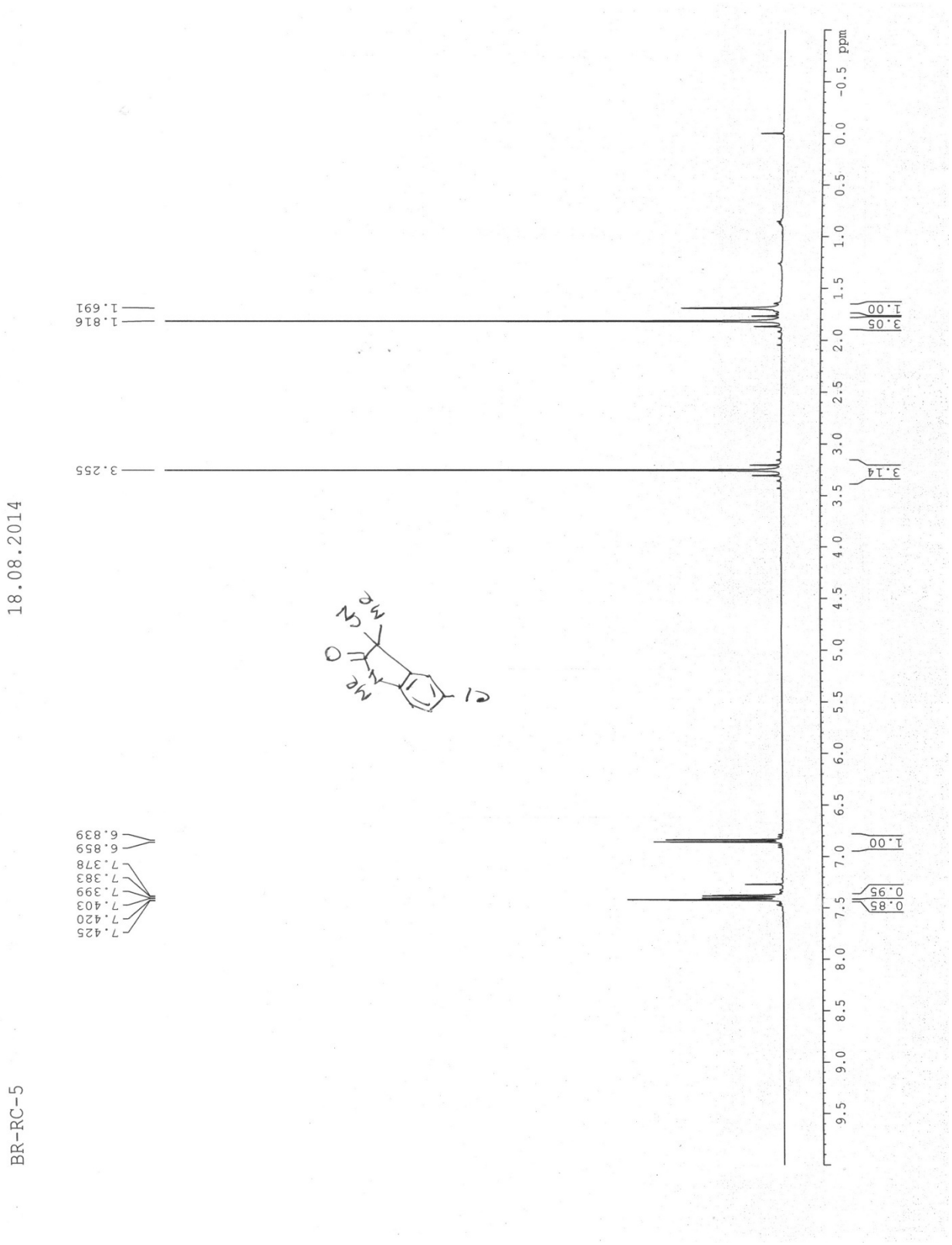
1,3-Diethyl-2-oxoindoline-3-carbonitrile (2d):

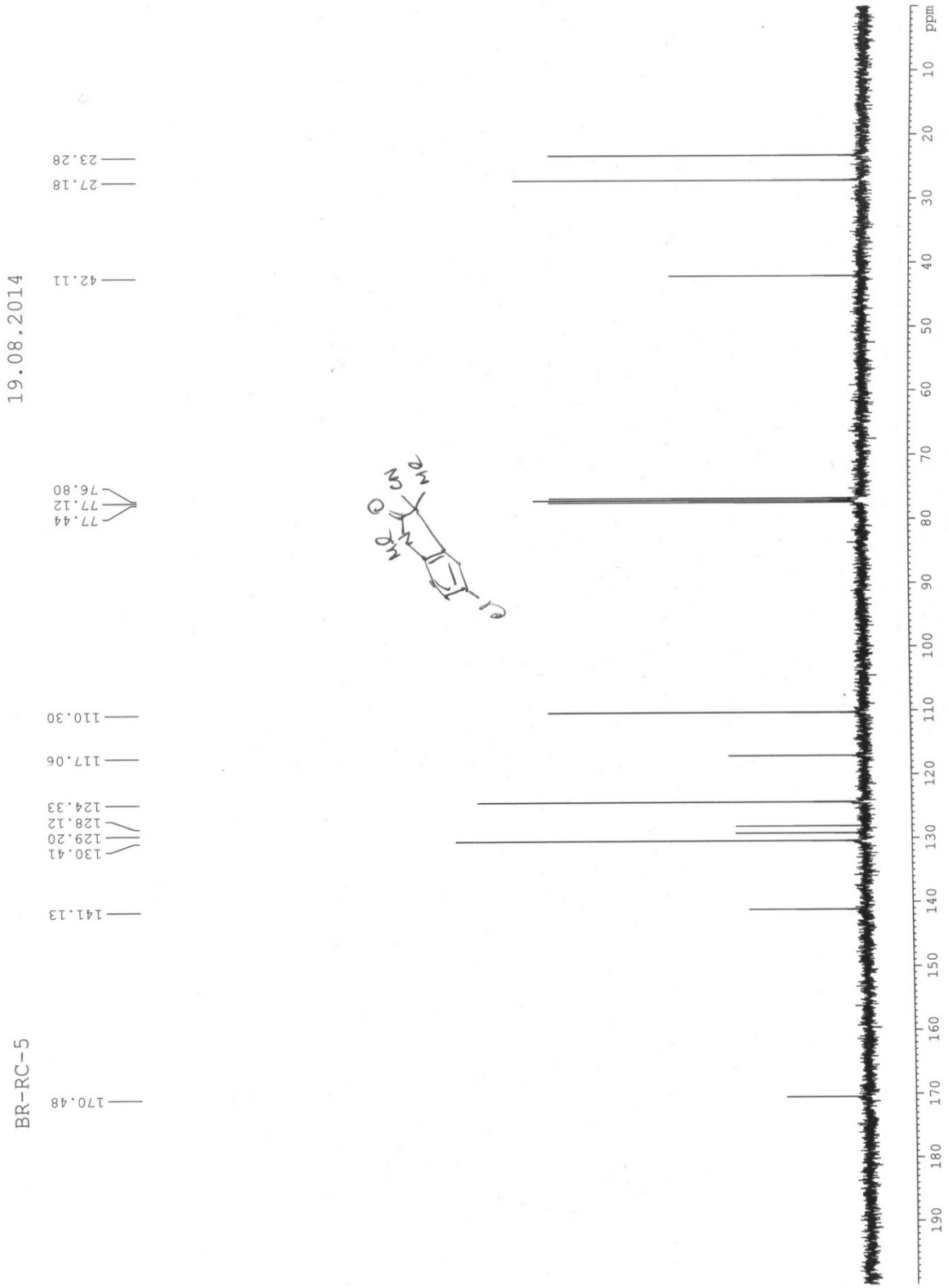



BR-RC-4
13.08.2014
170.02
142.26
130.23
125.29
124.31
123.48
117.24
109.16
77.40
77.09
76.77
46.99
35.53
30.88
12.52
8.21
ppm


BM_3S15
KLYMS_22 97 (1.763) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (97)
1: TOF MS ES+
1.59e+004
100
%
0
215.1180
214.5965
214.0925
215.6029
217.1516
221.3453
213.50
214.00
214.50
215.00
215.50
216.00
216.50
217.00
217.50
218.00
218.50
219.00
219.50
220.00
220.50
221.00
221.50
222.00
m/z
For Help, press F1

5-Chloro-1,3-dimethyl-2-oxoindoline-3-carbonitrile (2e):

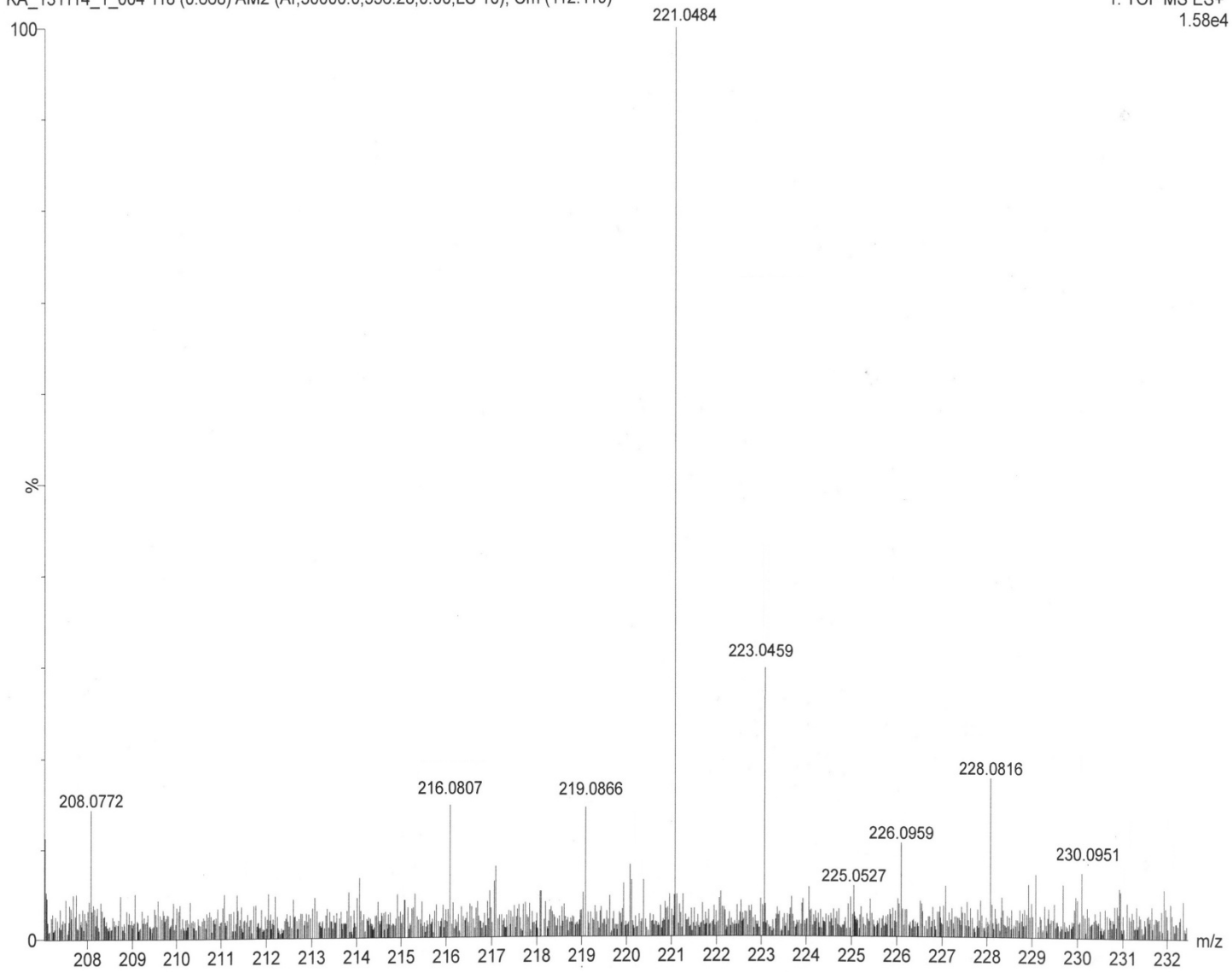



BR-RC-5
19.08.2014
170.48
141.13
130.41
129.20
128.12
124.33
117.06
110.30
77.44
77.12
76.80
42.11
27.18
23.28
ppm

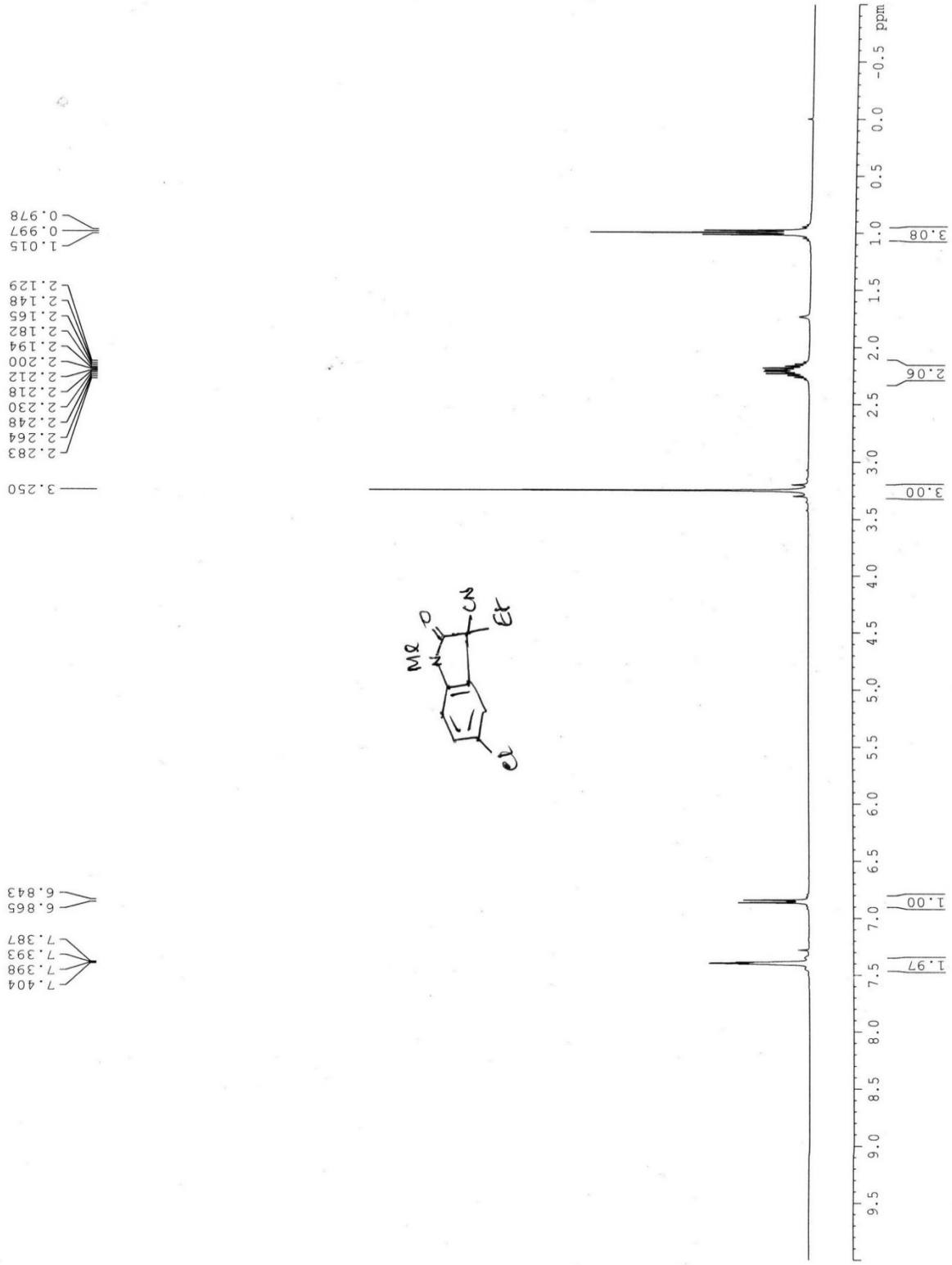
BRMS 25

KA_131114_1_004 118 (0.668) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (112:119)

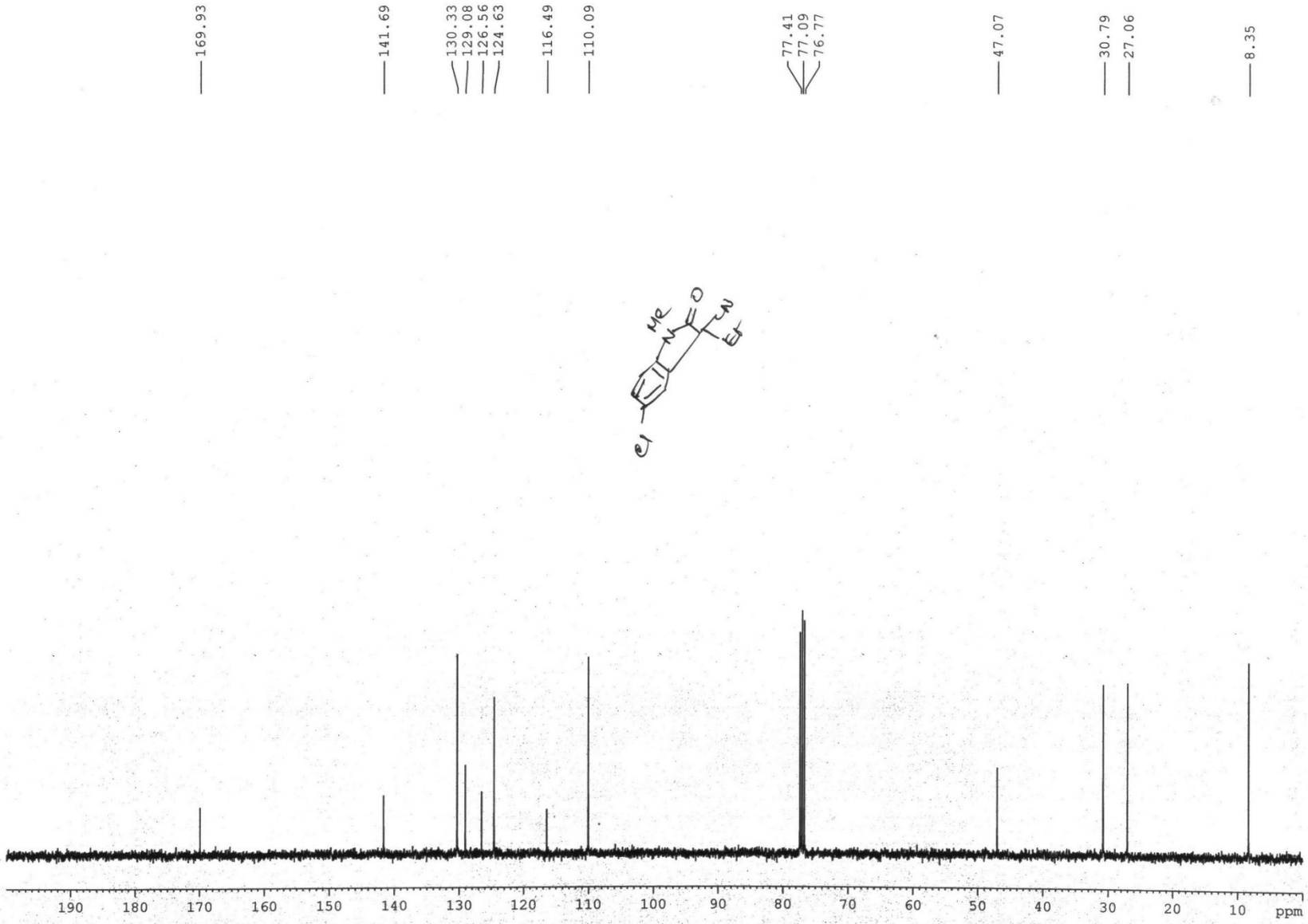
1: TOF MS ES+
1.58e4

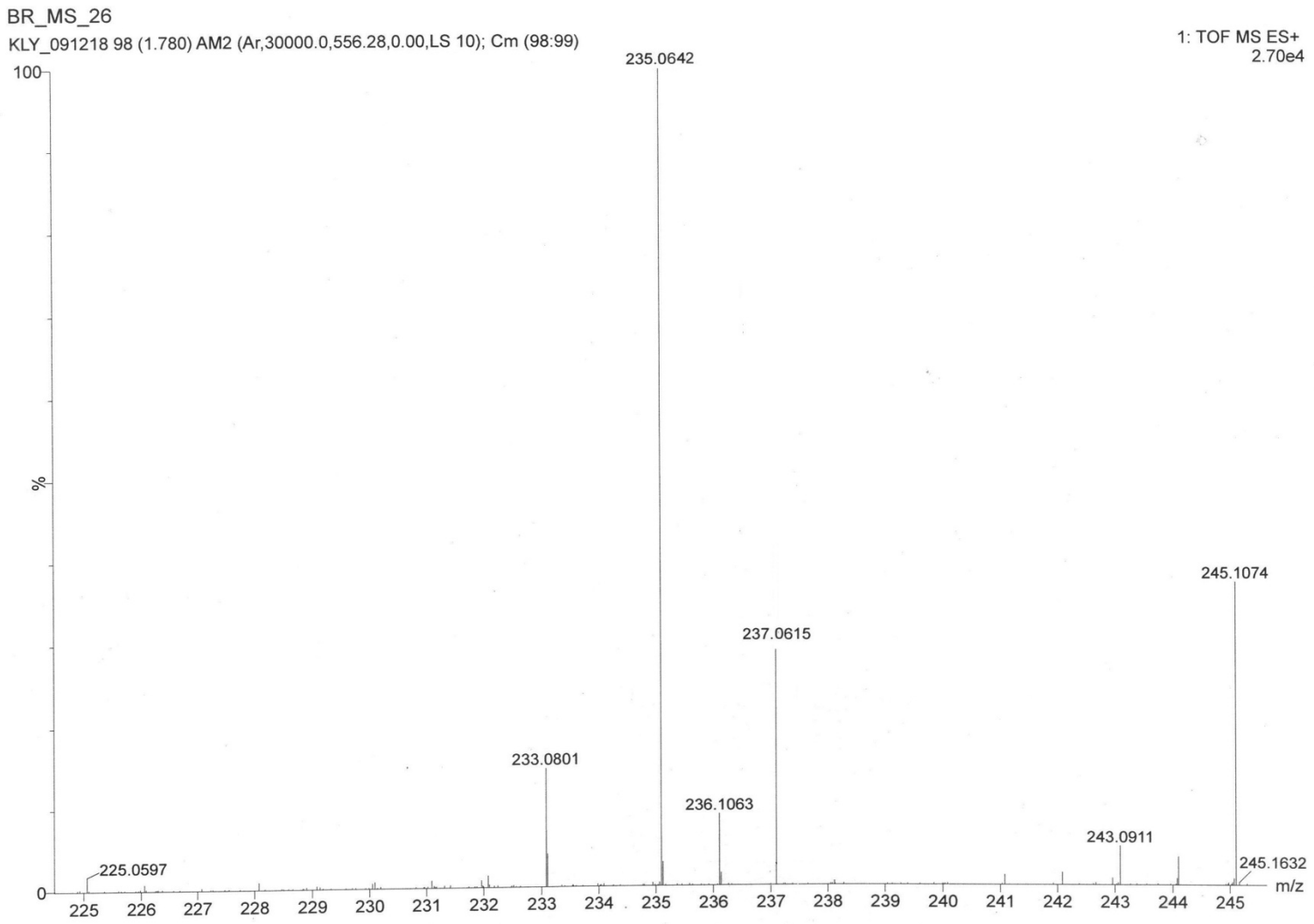


5-Chloro-3-ethyl-1-methyl-2-oxoindoline-3-carbonitrile (2f):

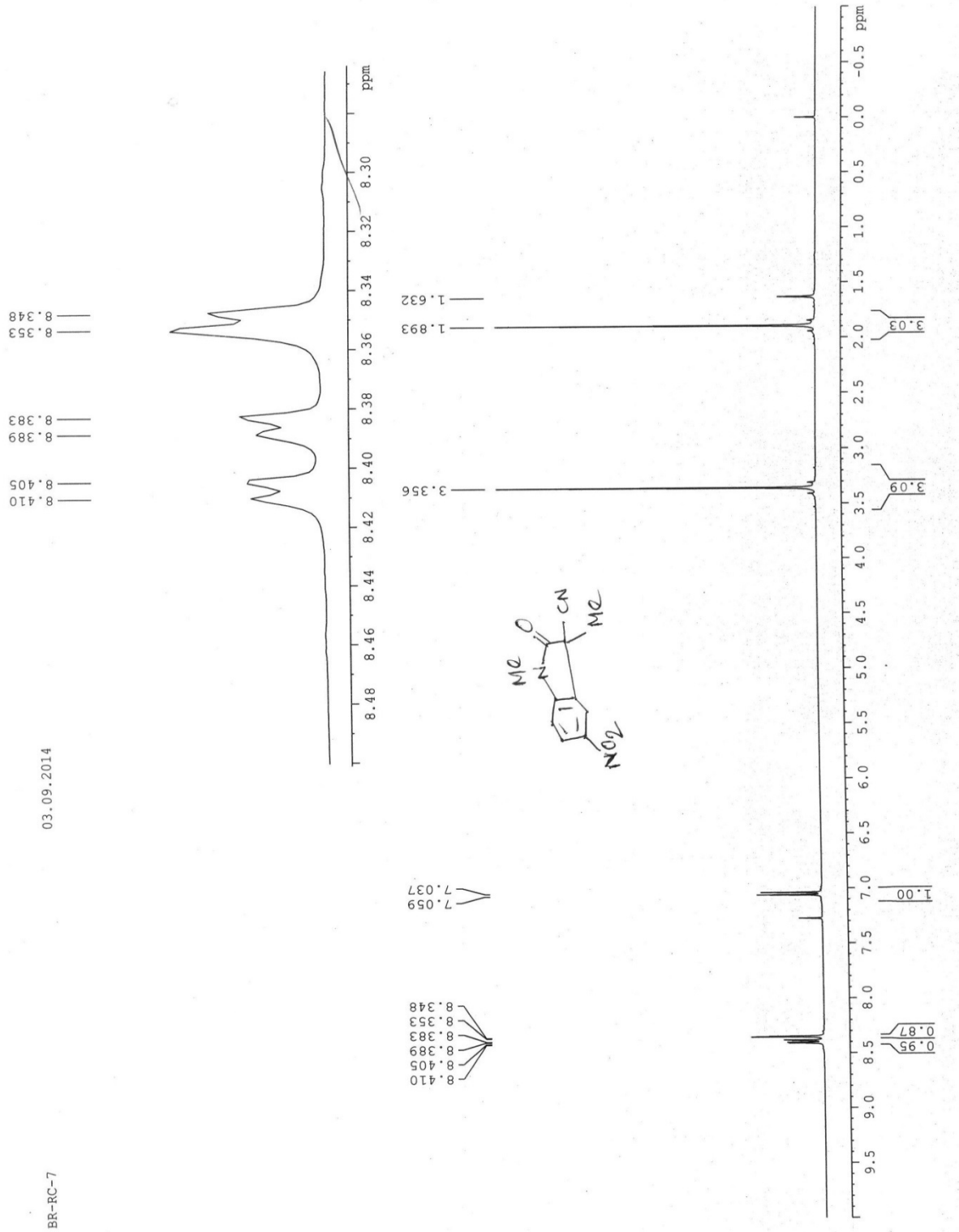


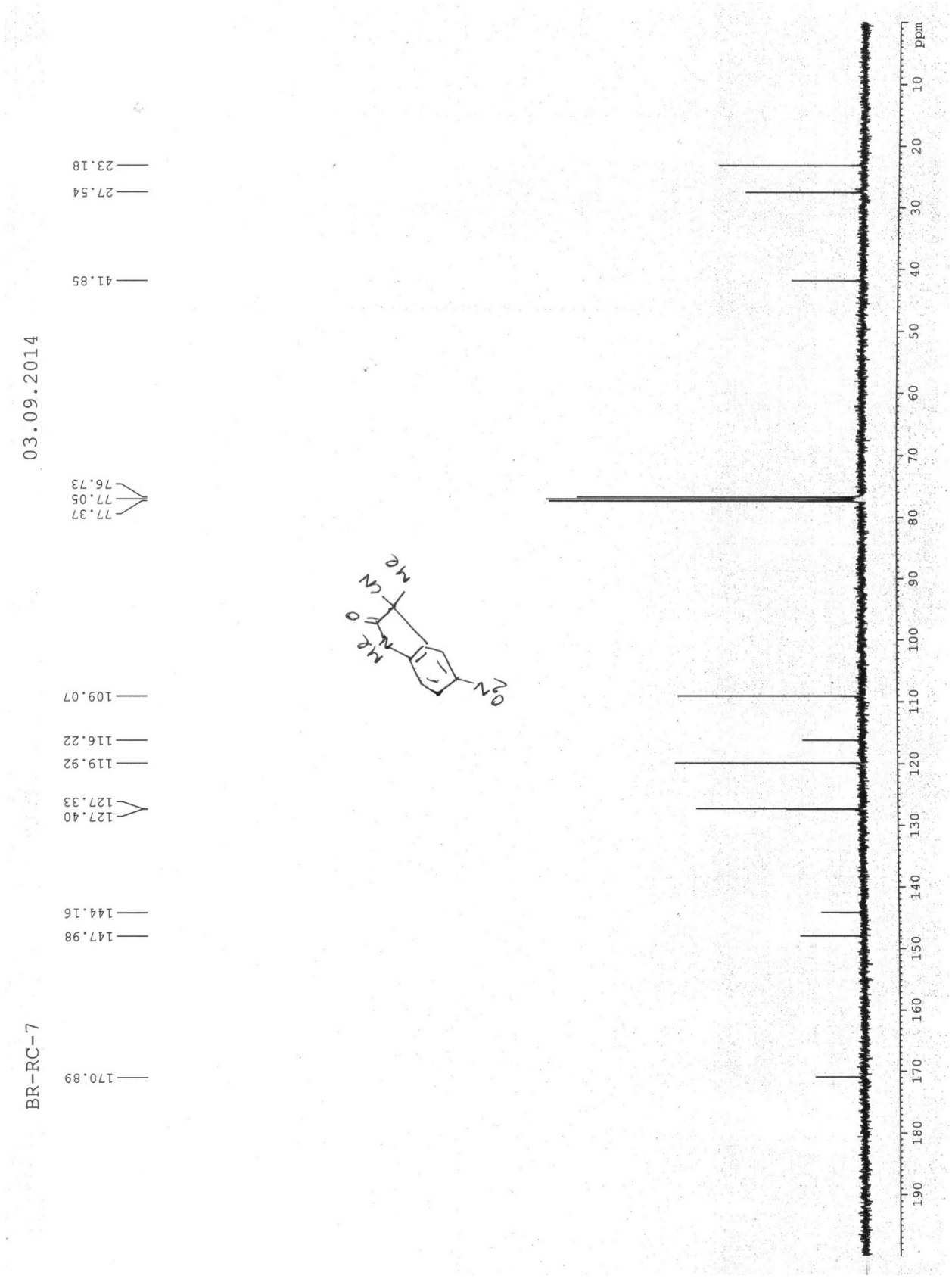
BR-RC-6 20.08.2014

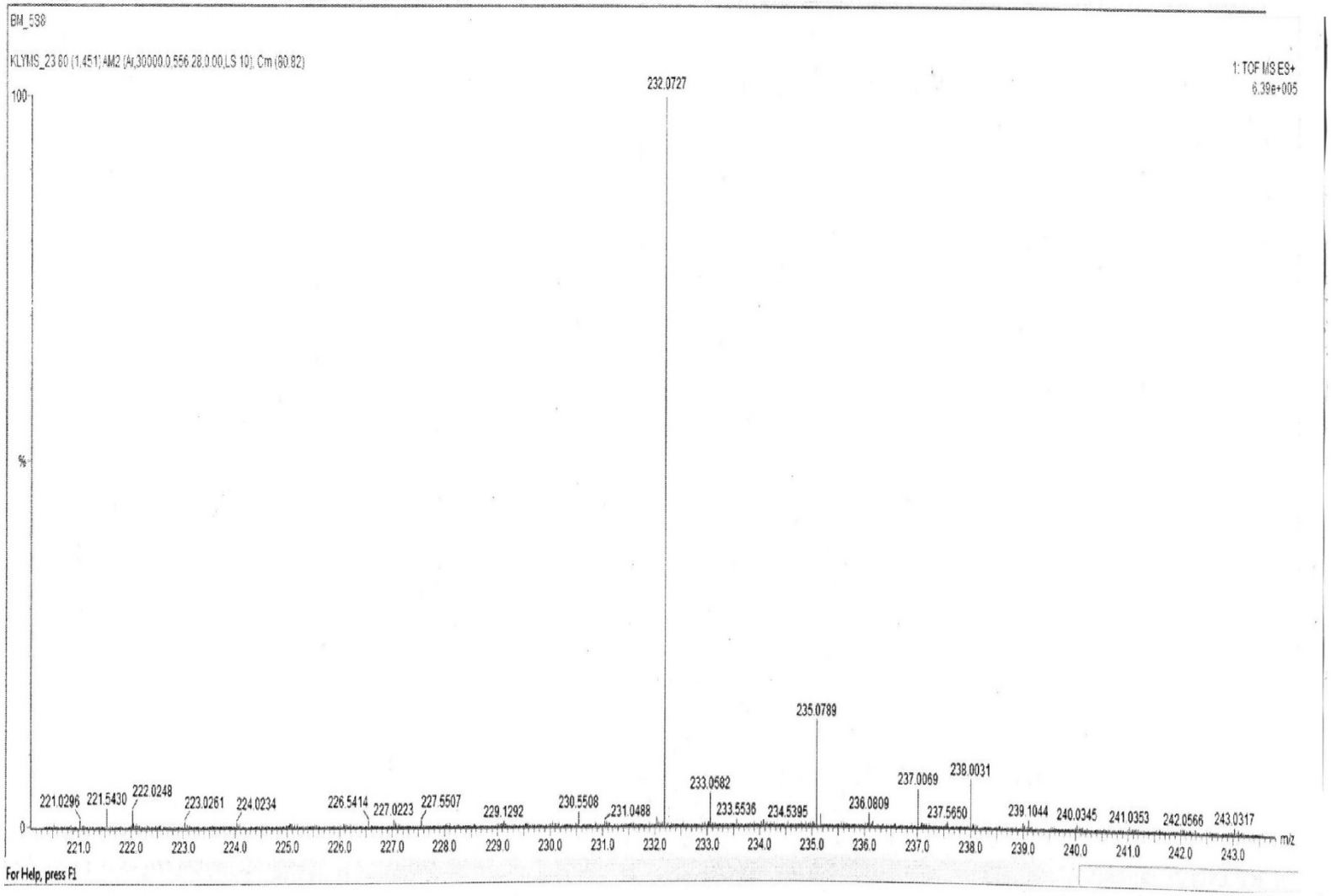



BR_MS_26
KLY_091218 98 (1.780) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (98:99)
1: TOF MS ES+
2.70e4
100
%
0
235.0642
245.1074
237.0615
233.0801
236.1063
243.0911
225.0597
245.1632
m/z
225 226 227 228 229 230 231 232 233 234 235 236 237 238 239 240 241 242 243 244 245

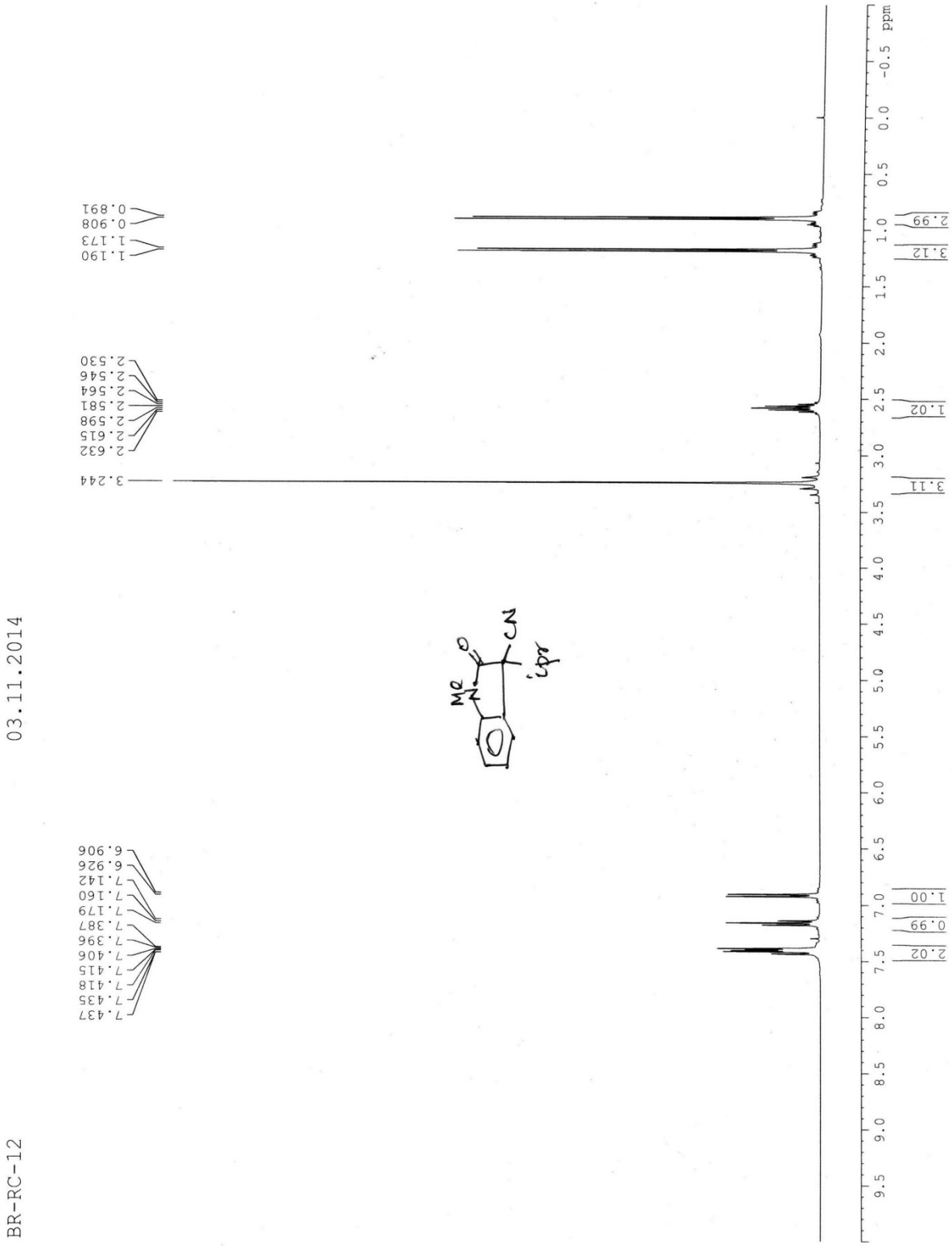
1,3-Dimethyl-5-nitro-2-oxoindoline-3-carbonitrile (2g):

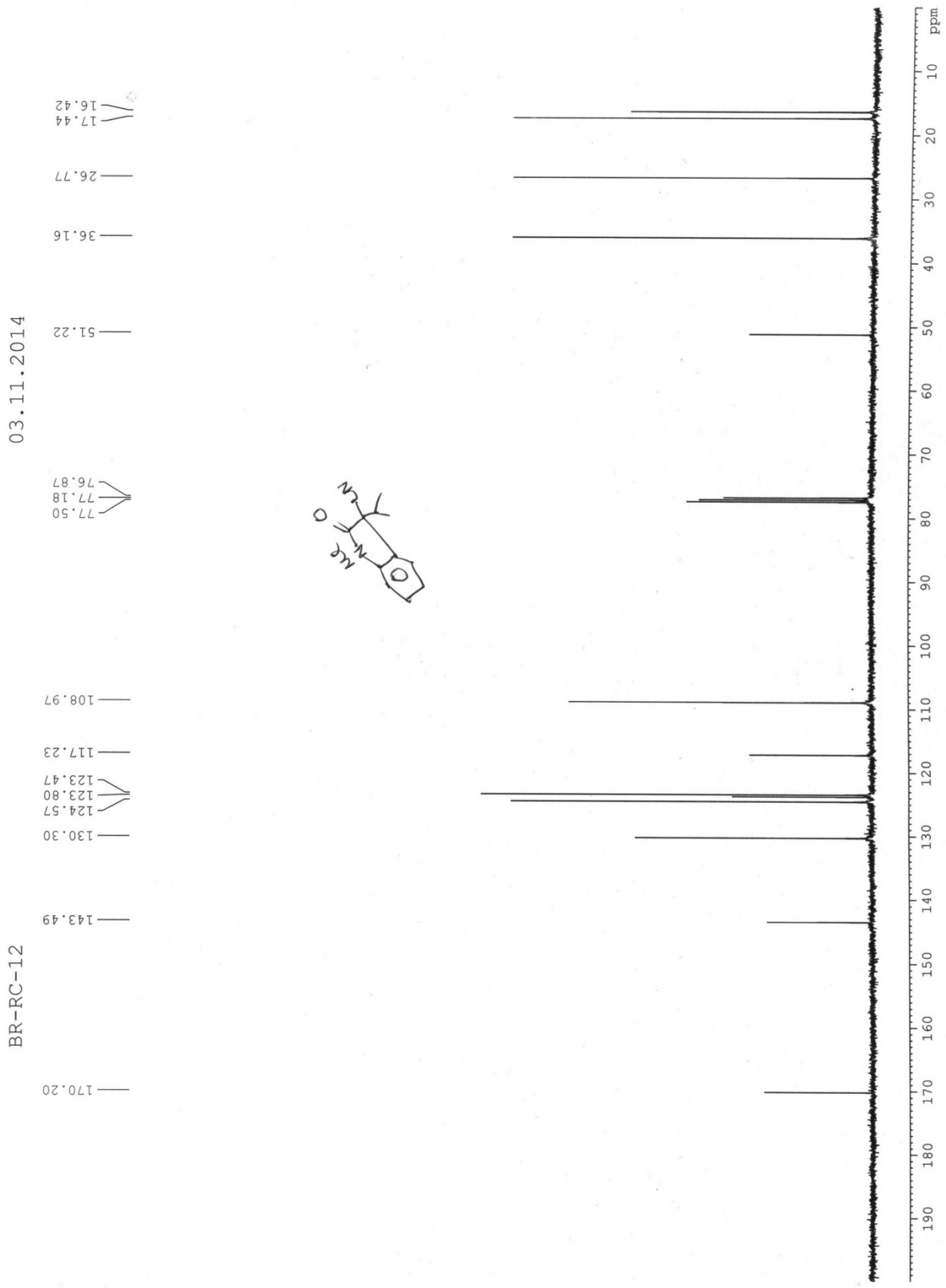


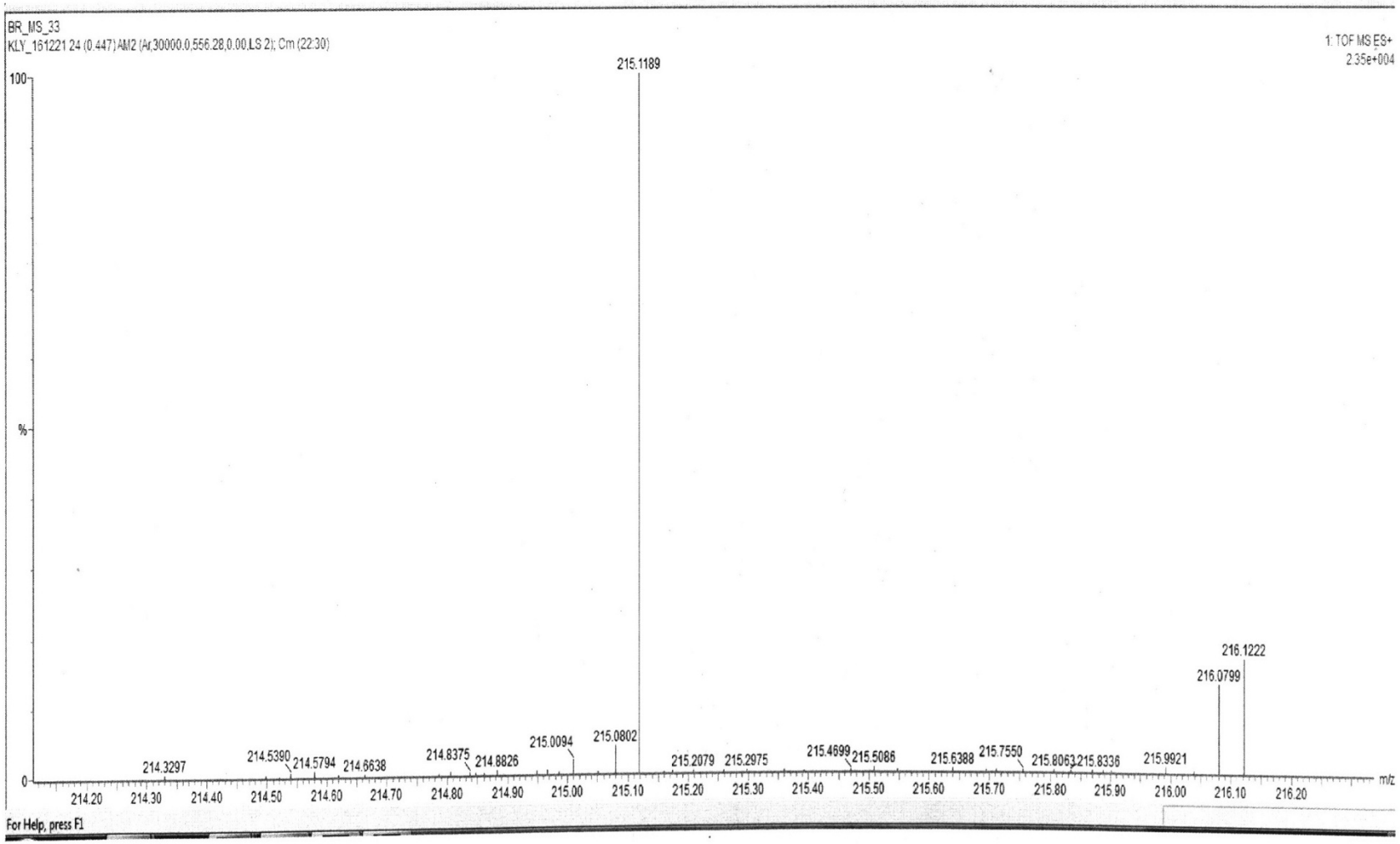

BR-RC-7
03.09.2014
170.89
147.98
144.16
127.40
127.33
119.92
116.22
109.07
77.37
77.05
76.73
41.85
27.54
23.18
ppm


BM_538
KLYNS_23 80 (1.451); AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (60:82)
1: TOF MS ES+
6.39e+005
100
%
0
232.0727
235.0789
238.0031
237.0069
233.0582
221.0296
221.5430
222.0248
223.0261
224.0234
226.5414
227.0223
227.5507
229.1292
230.5508
231.0488
233.5536
234.5395
236.0809
237.5650
239.1044
240.0345
241.0353
242.0566
243.0317
221.0
222.0
223.0
224.0
225.0
226.0
227.0
228.0
229.0
230.0
231.0
232.0
233.0
234.0
235.0
236.0
237.0
238.0
239.0
240.0
241.0
242.0
243.0
m/z
For Help, press F1

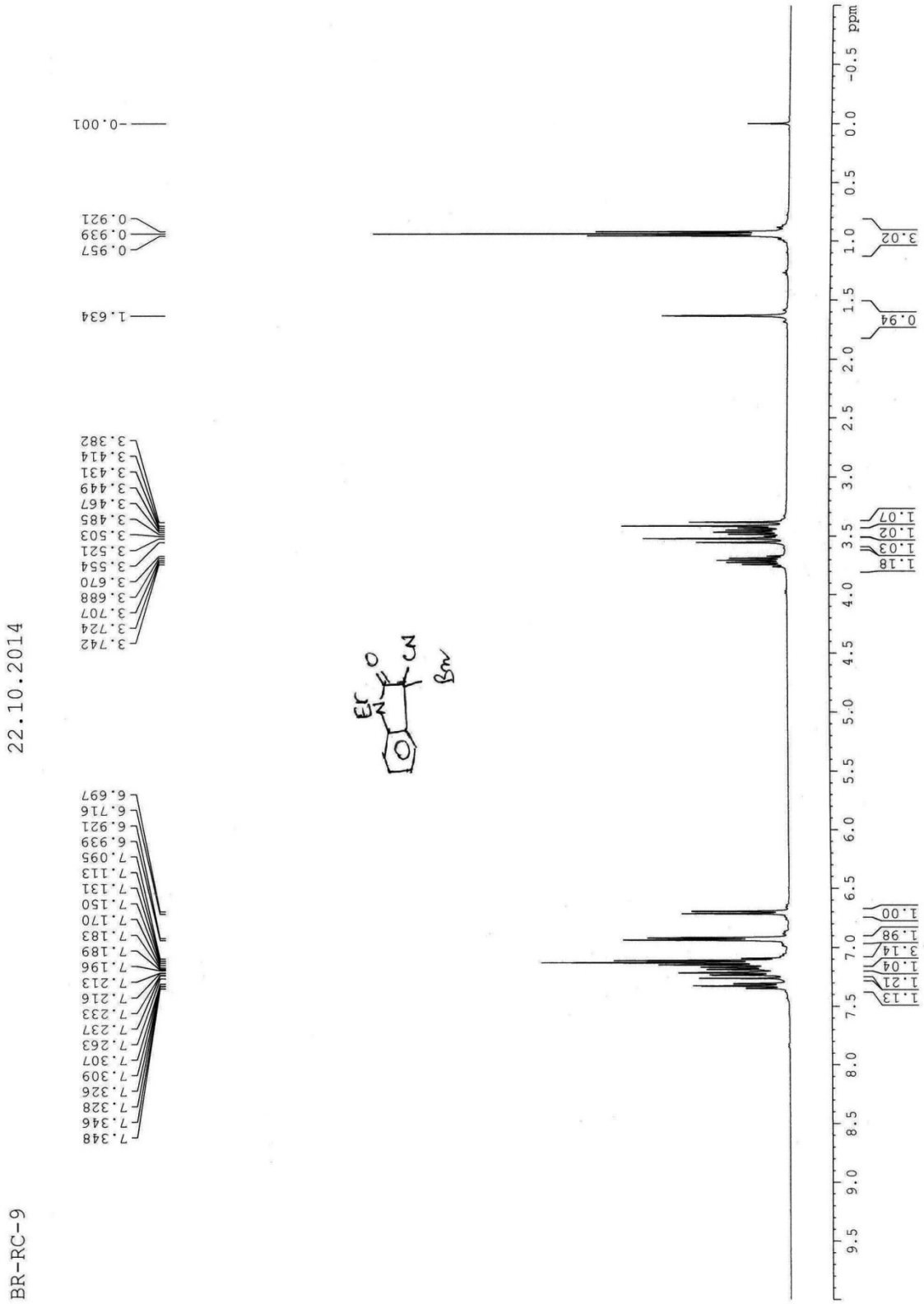
1-Ethyl-3-isopropyl-2-oxoindoline-3-carbonitrile (2h):

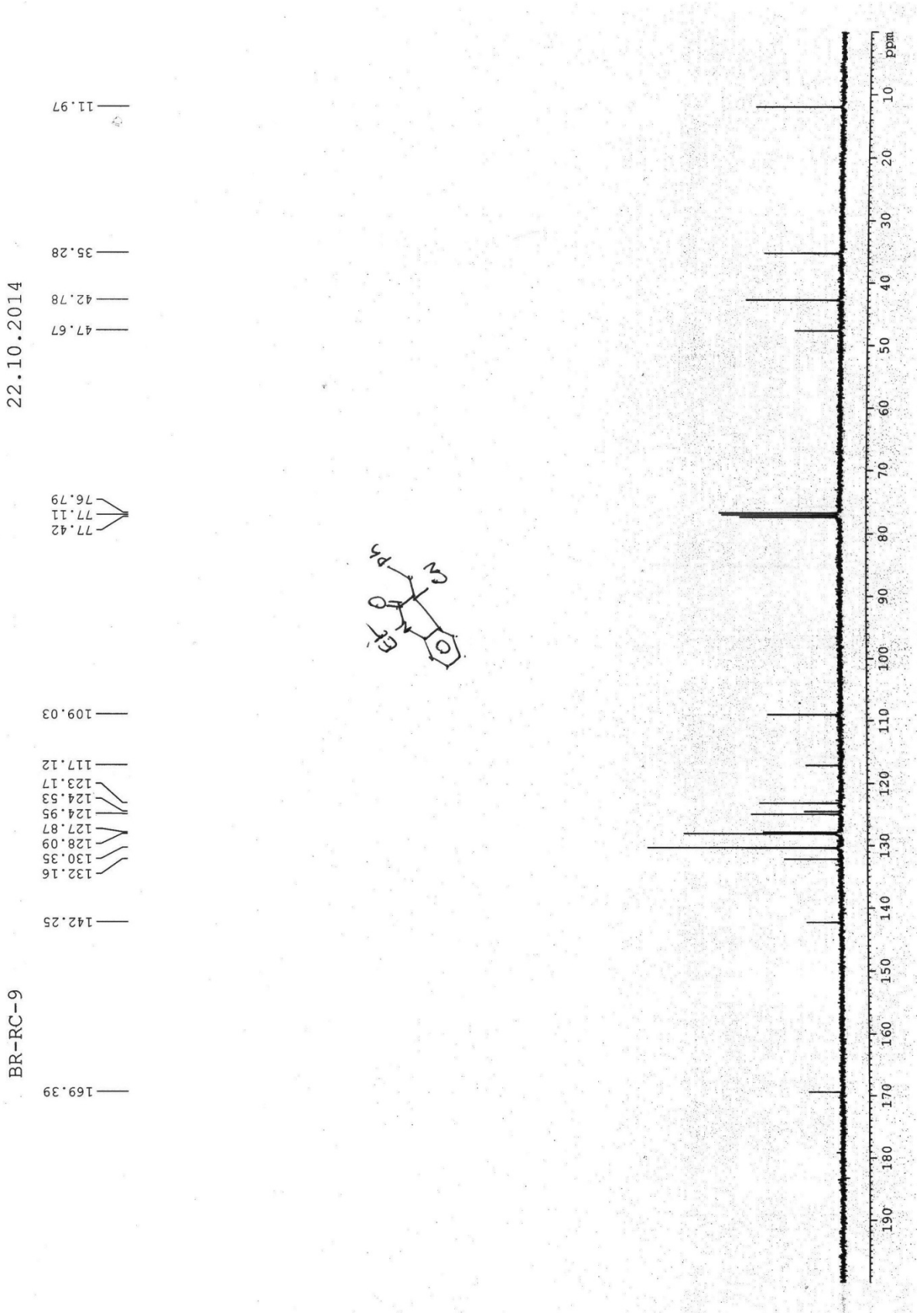


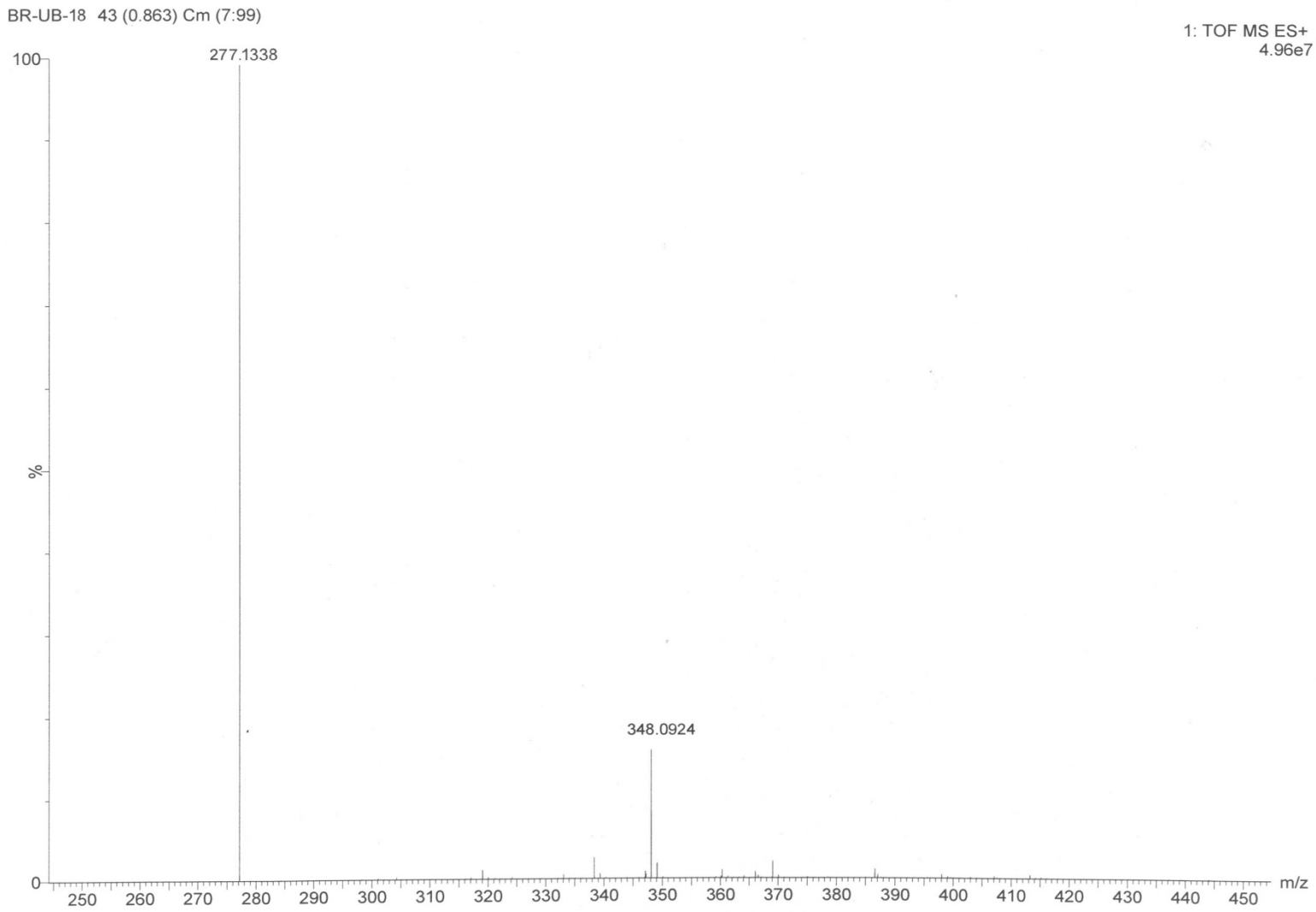

BR-RC-12
03.11.2014
170.20
143.49
130.30
124.57
123.80
123.47
117.23
108.97
77.50
77.18
76.87
51.22
36.16
26.77
17.44
16.42
ppm


BR_MS_33
KLY_161221 24 (0.447) AM2 (Ar,30000.0,556.28,0.00,LS 2); Cm (22:30)
1: TOF MS ES+
2.35e+004
100
%
0
215.1189
216.1222
216.0799
215.0094
215.0802
214.3297
214.5390
214.5794
214.6638
214.8375
214.8826
215.2079
215.2975
215.4699
215.5086
215.6388
215.7550
215.8063
215.8336
215.9921
214.20
214.30
214.40
214.50
214.60
214.70
214.80
214.90
215.00
215.10
215.20
215.30
215.40
215.50
215.60
215.70
215.80
215.90
216.00
216.10
216.20
m/z
For Help, press F1

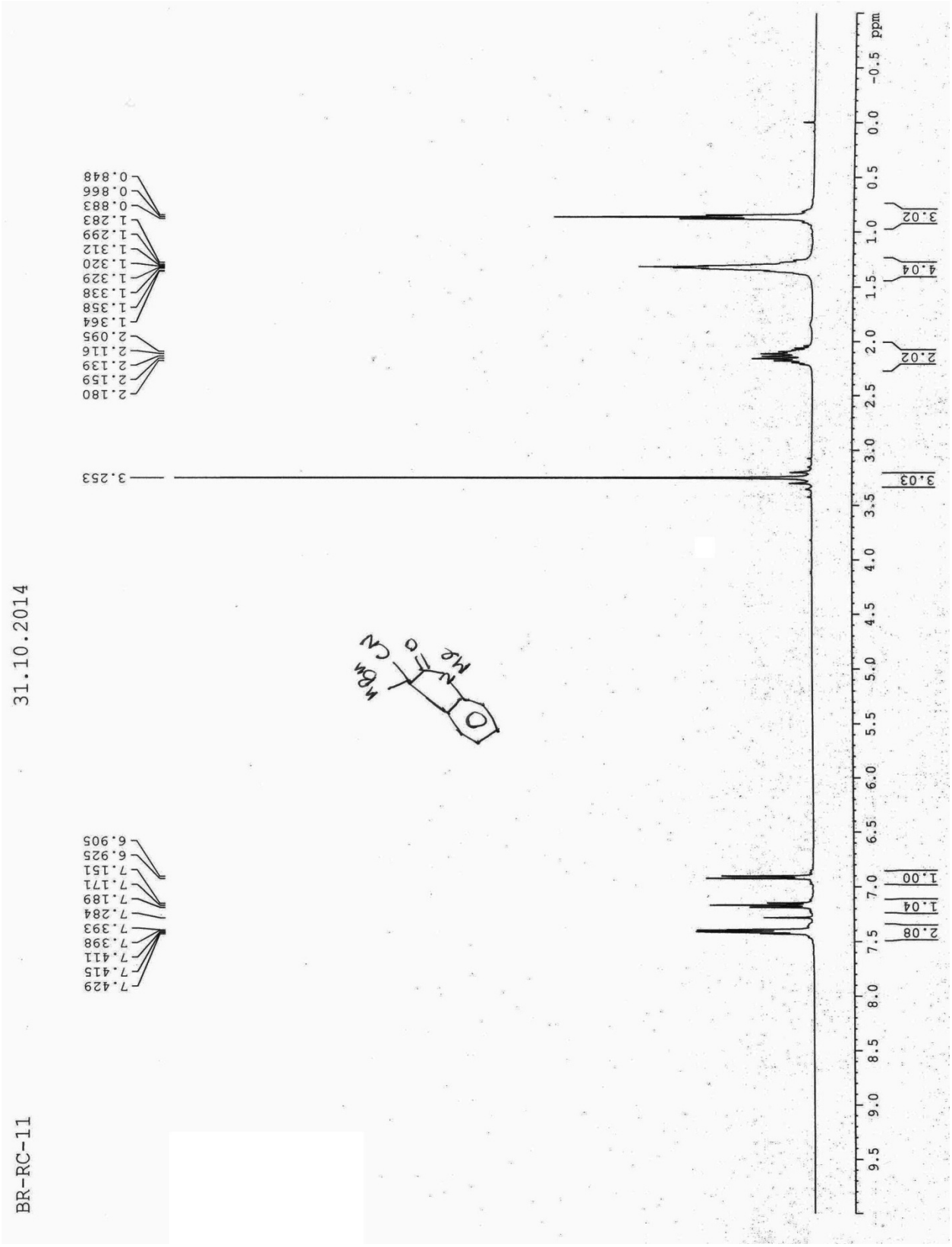
3-Benzyl-1-ethyl-2-oxoindoline-3-carbonitrile (2i):



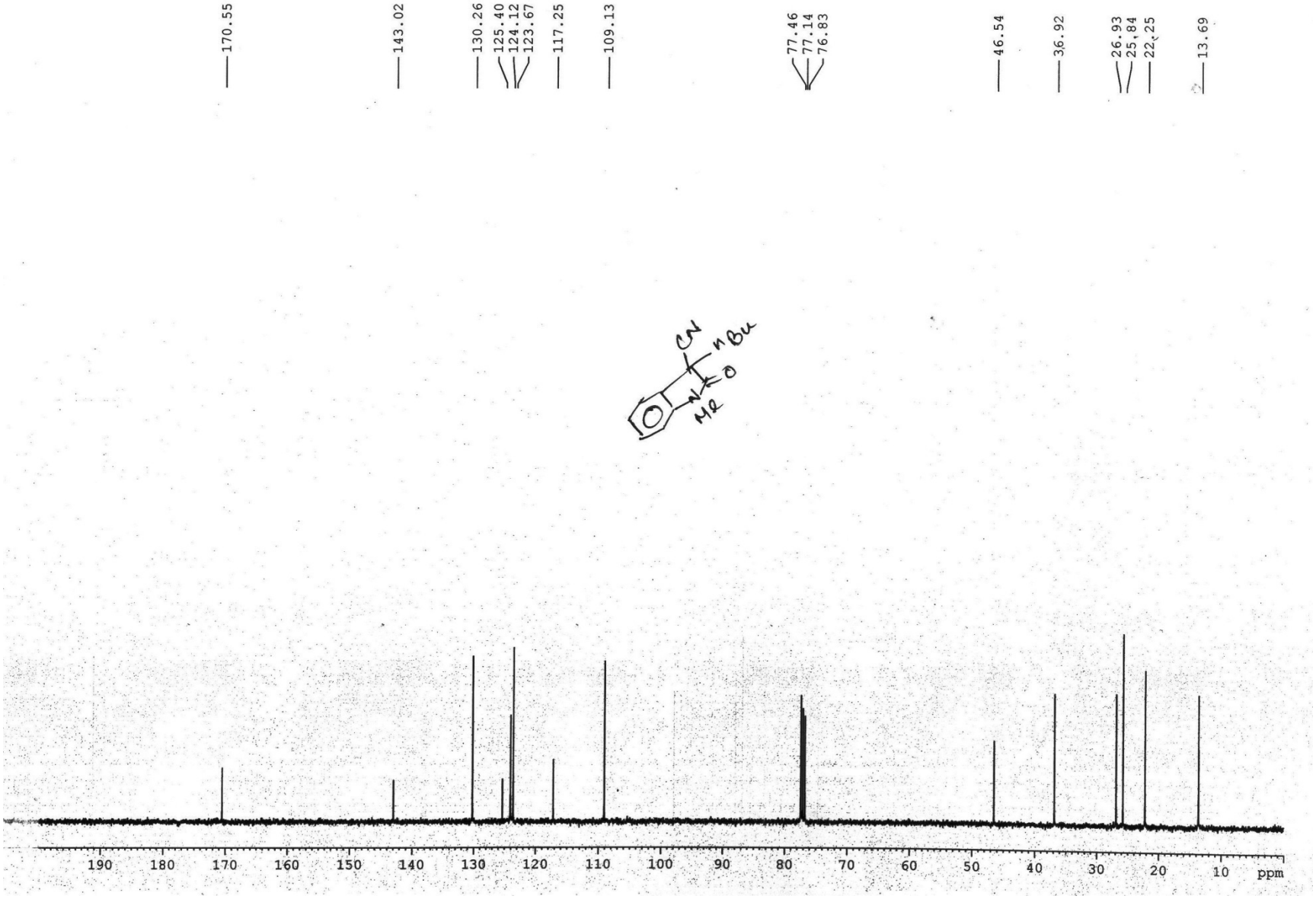

BR-RC-9
22.10.2014
169.39
142.25
132.16
130.35
128.09
127.87
124.95
124.53
123.17
117.12
109.03
77.42
77.11
76.79
47.67
42.78
35.28
11.97
ppm

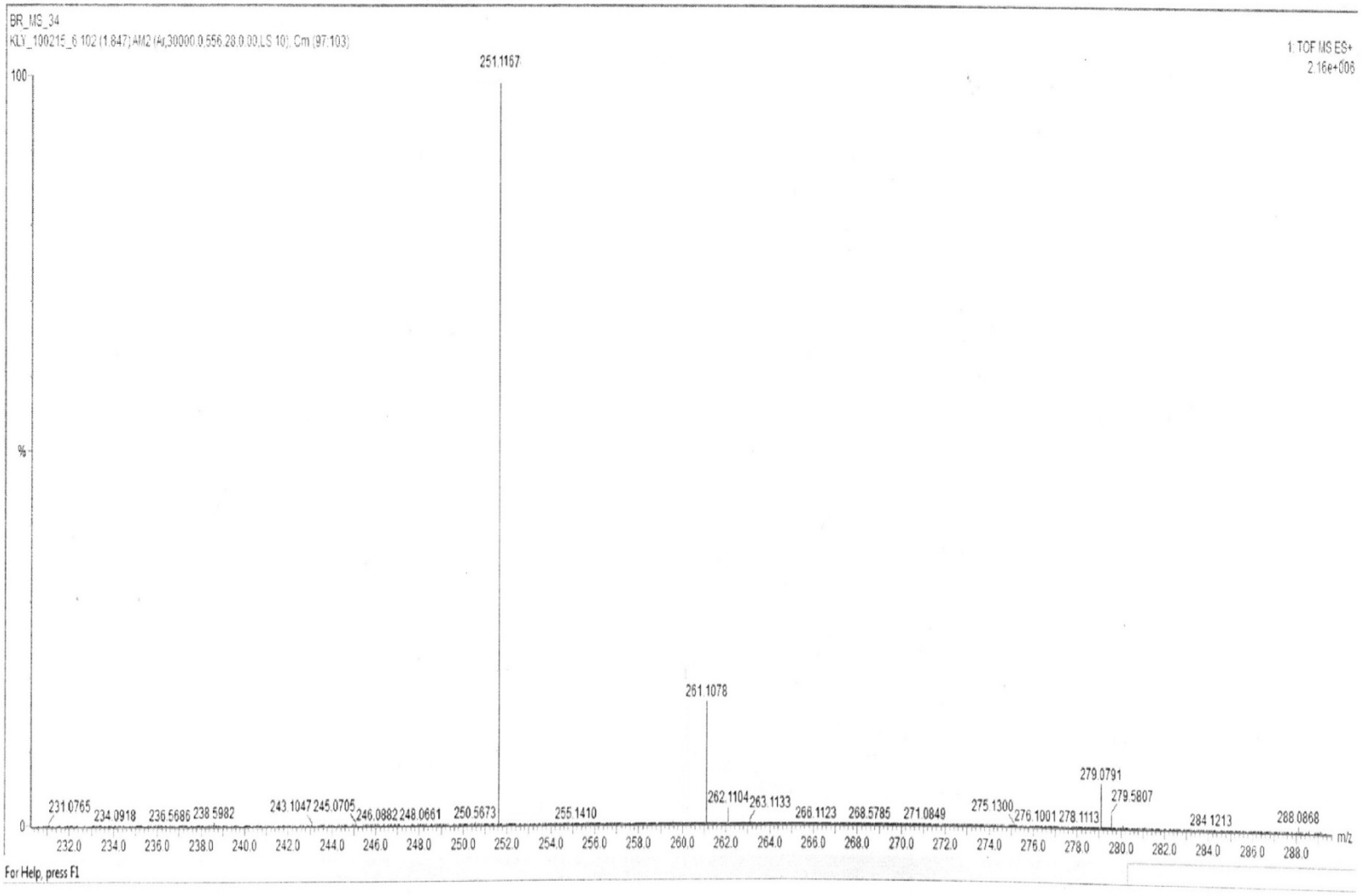

BR-UB-18 43 (0.863) Cm (7:99)
1: TOF MS ES+
4.96e7
277.1338
348.0924
100
%
0
250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450
m/z

3-Butyl-1-ethyl-2-oxoindoline-3-carbonitrile (2j):

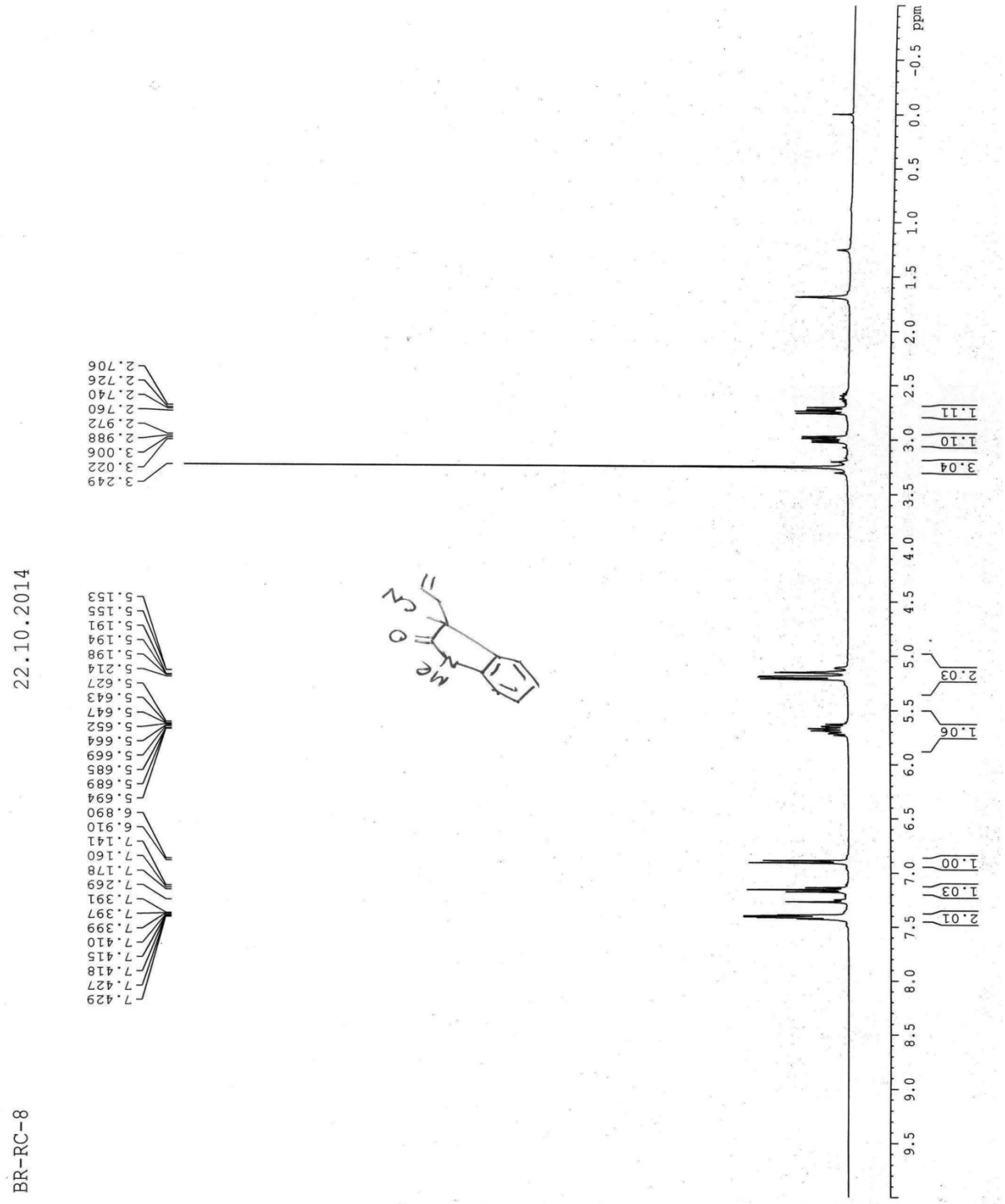


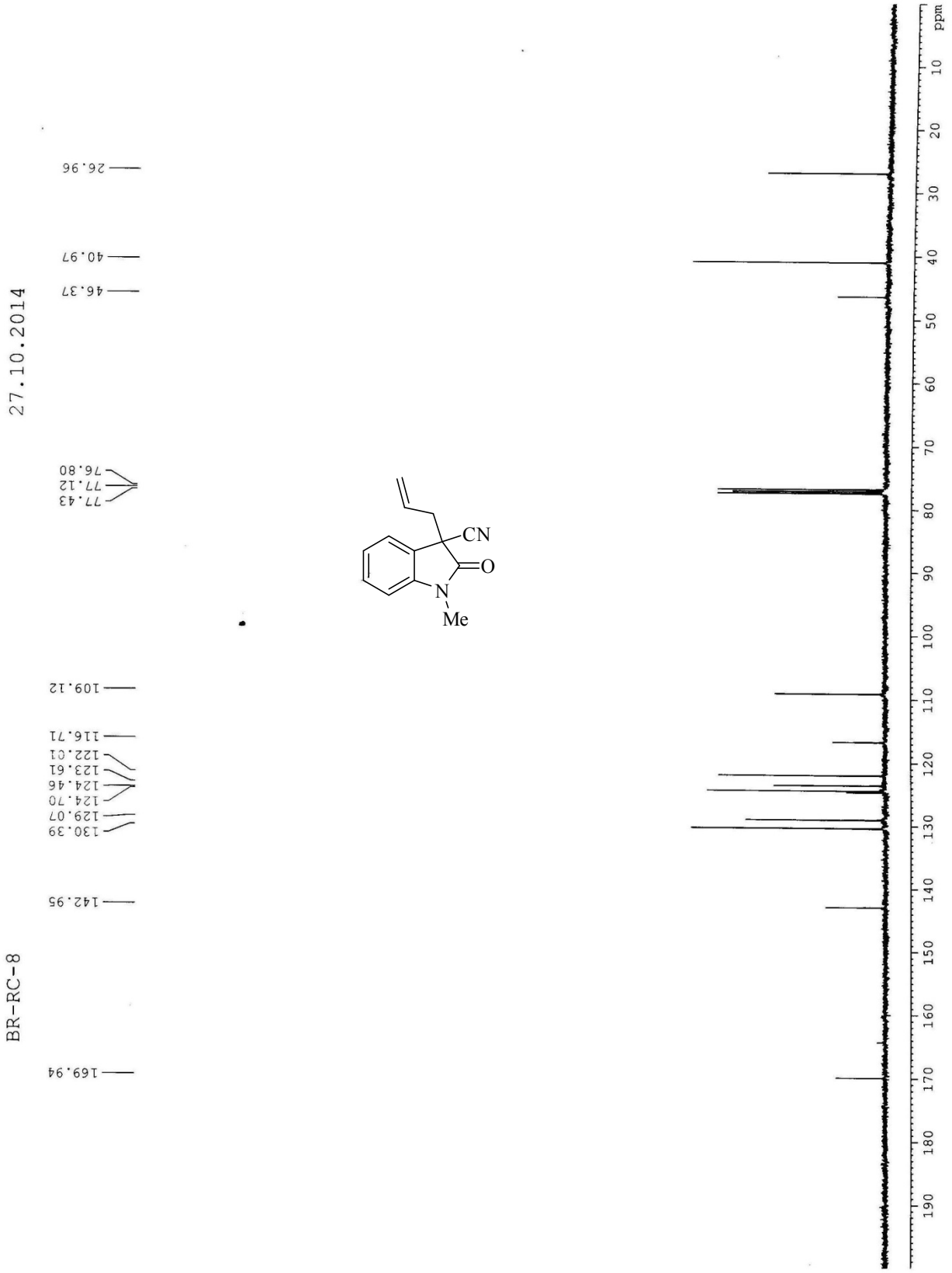
BR-RC-11 31.10.2014

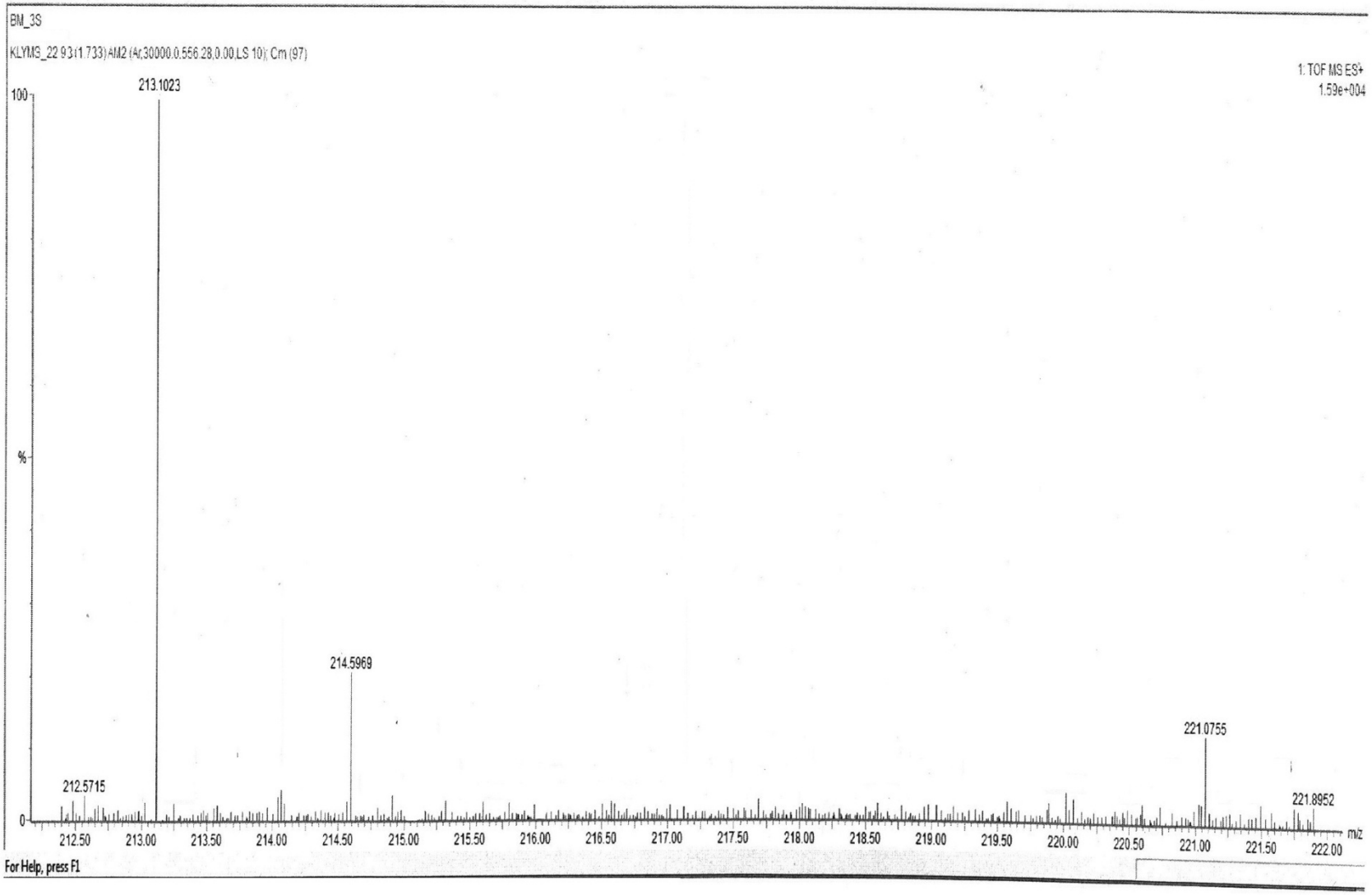



BR_MS_34
KLY_100215_6 102 (1.847) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (97:103)
1: TOF MS ES+
2.16e+006
100
%
0
251.1167
261.1078
279.0791
279.5807
231.0765
234.0918
236.5686
238.5982
243.1047
245.0705
246.0882
248.0661
250.5673
255.1410
262.1104
263.1133
266.1123
268.5785
271.0849
275.1300
276.1001
278.1113
284.1213
288.0868
232.0
234.0
236.0
238.0
240.0
242.0
244.0
246.0
248.0
250.0
252.0
254.0
256.0
258.0
260.0
262.0
264.0
266.0
268.0
270.0
272.0
274.0
276.0
278.0
280.0
282.0
284.0
286.0
288.0
m/z
For Help, press F1

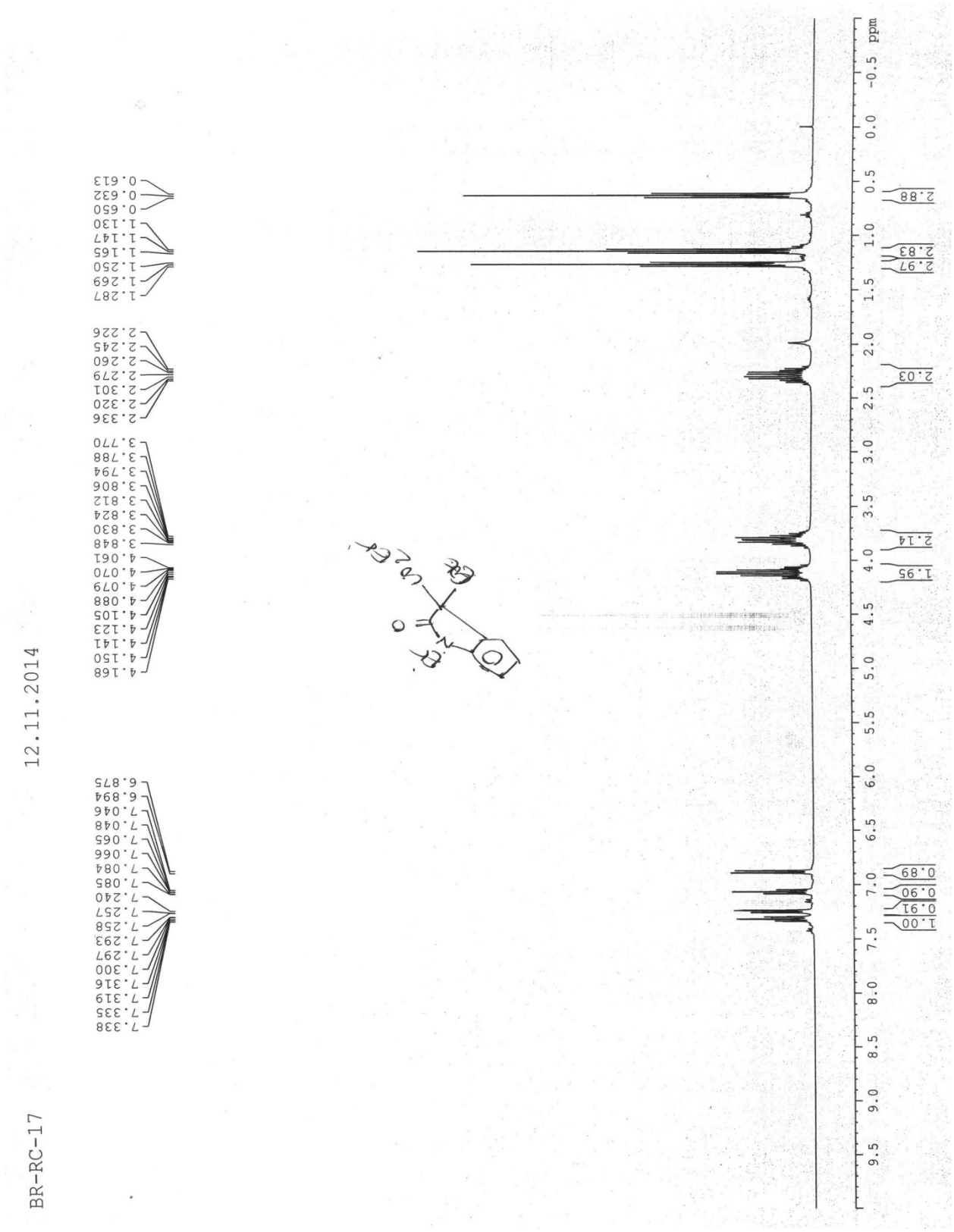
3-Allyl-1-ethyl-2-oxoindoline-3-carbonitrile (2k):

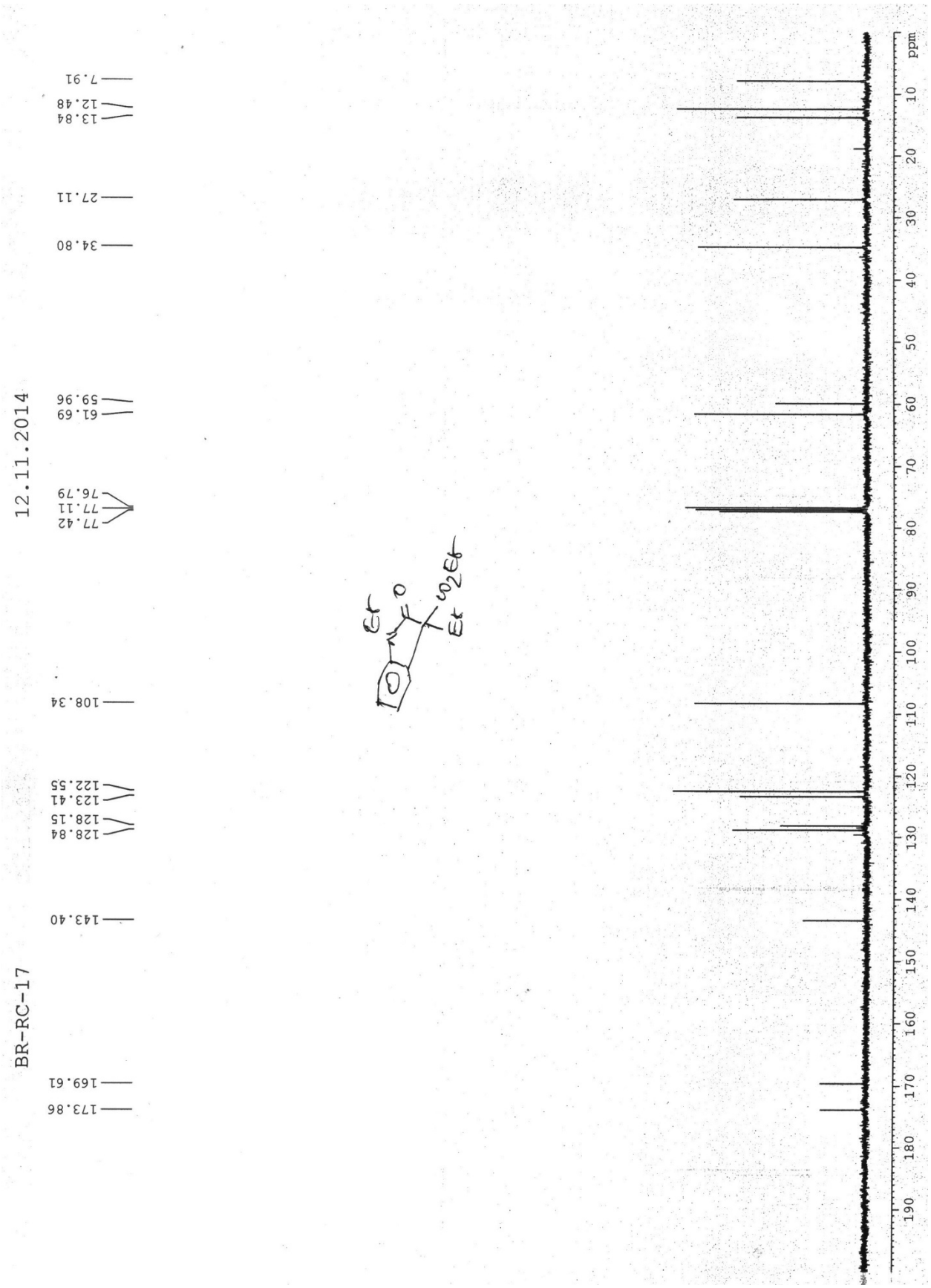


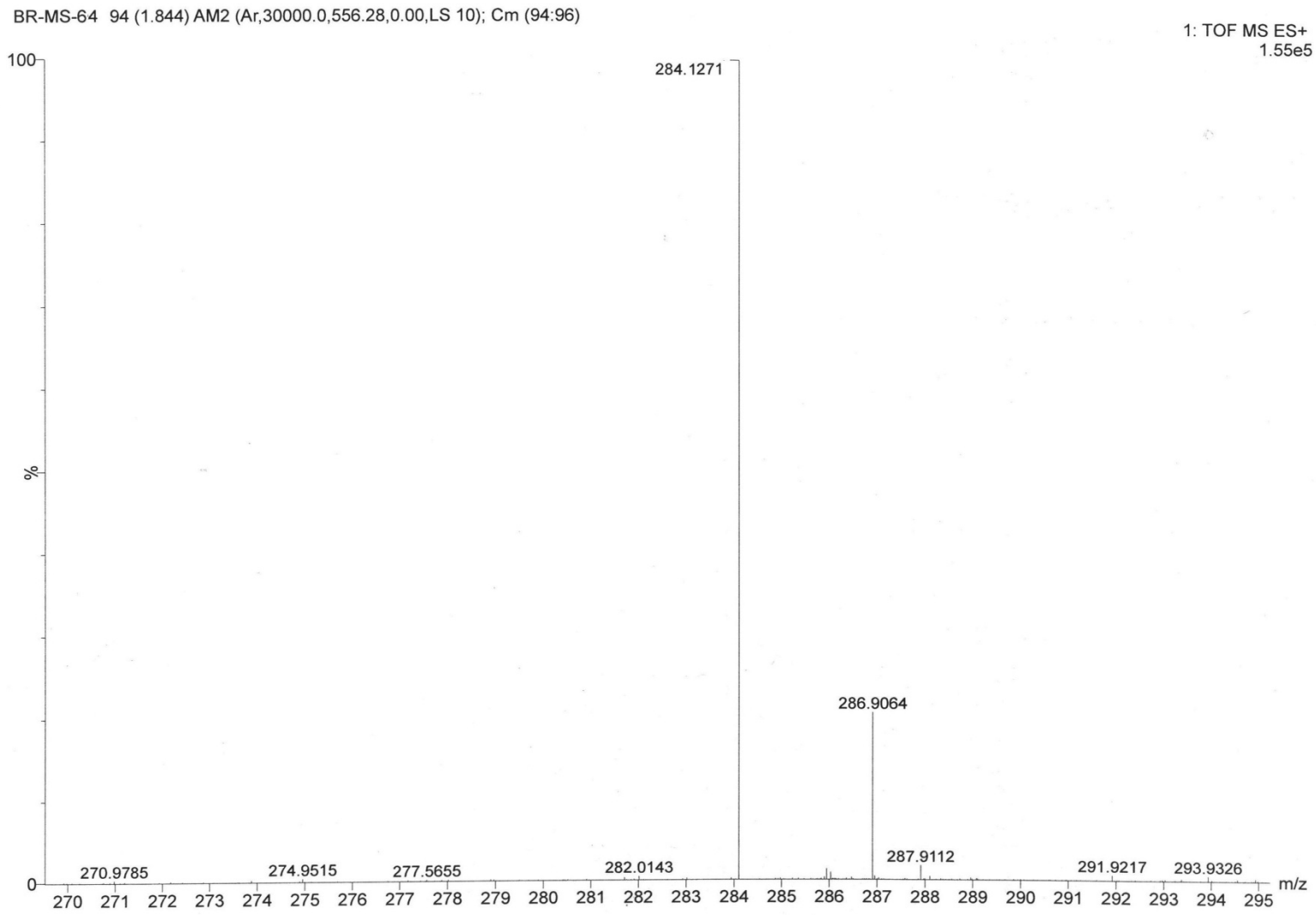

BR-RC-8
27.10.2014
169.94
142.95
130.39
129.07
124.70
124.46
123.61
122.01
116.71
109.12
77.43
77.12
76.80
46.37
40.97
26.96
CN
O
N
Me
ppm


BM_3S
KLYMS_22 93(1.733)AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (97)
1: TOF MS ES+
1.59e+004
100
%
0
213.1023
214.5969
212.5715
221.0755
221.8952
212.50
213.00
213.50
214.00
214.50
215.00
215.50
216.00
216.50
217.00
217.50
218.00
218.50
219.00
219.50
220.00
220.50
221.00
221.50
222.00
m/z
For Help, press F1

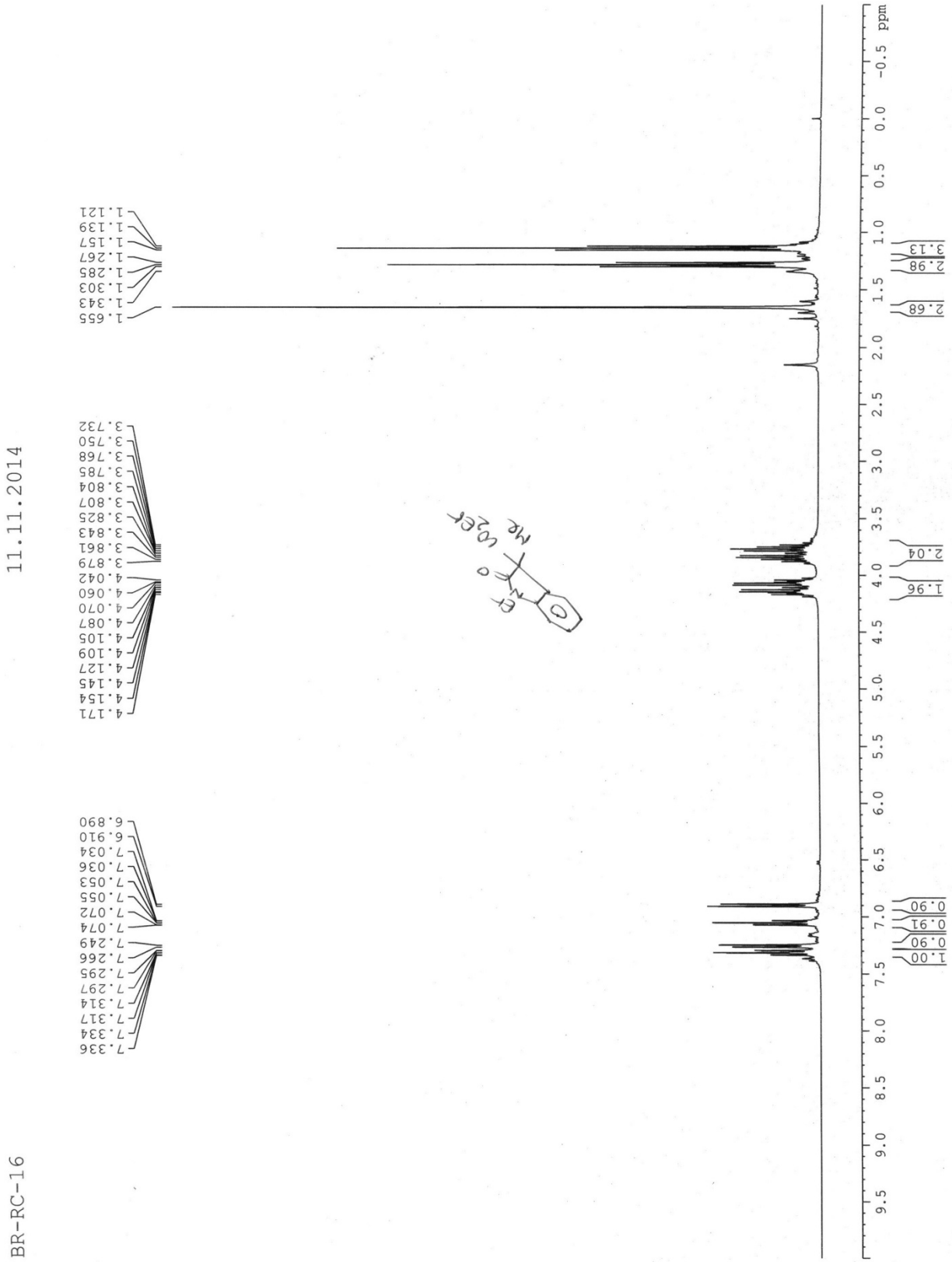
Ethyl 1,3-diethyl-2-oxoindoline-3-carboxylate (2l):

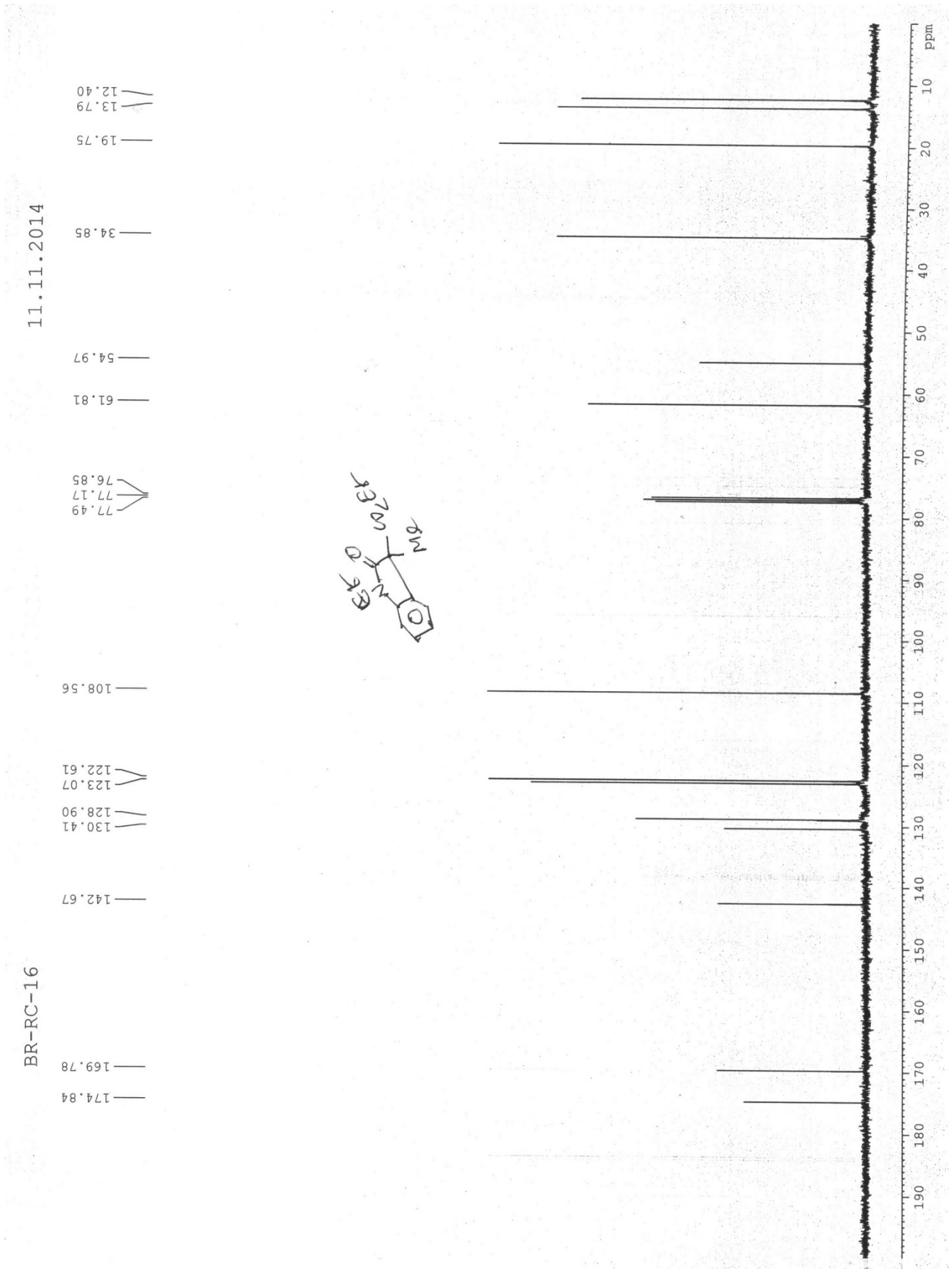


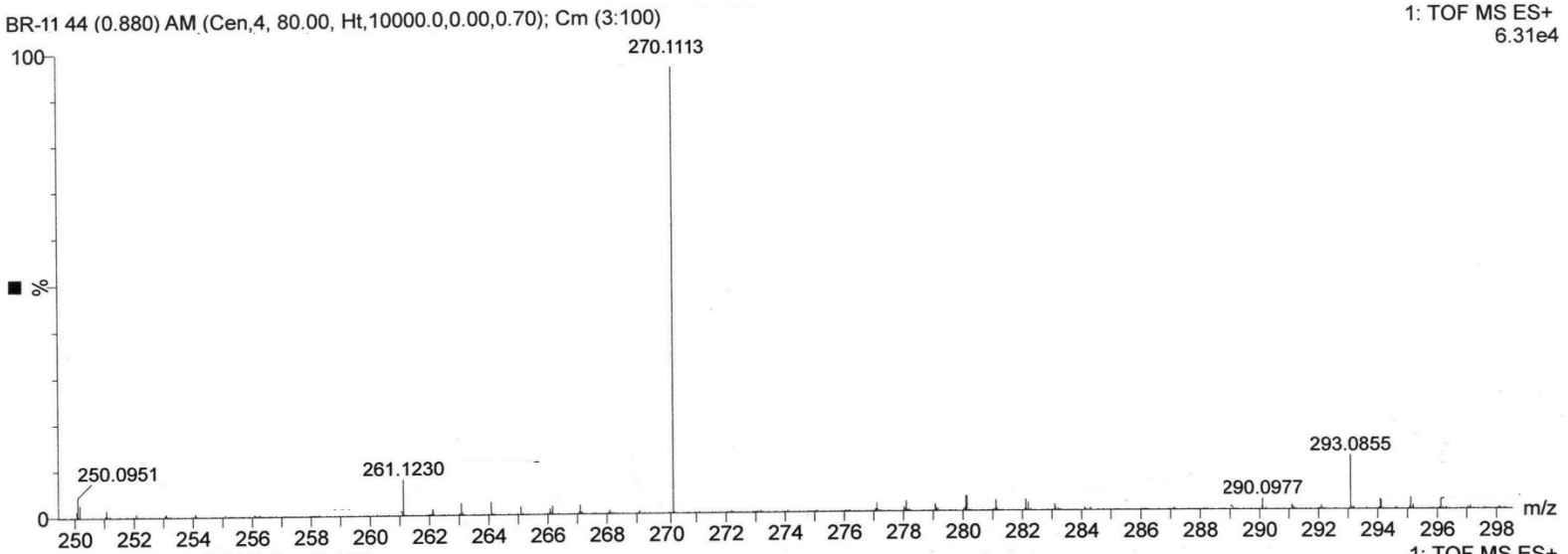

BR-RC-17
12.11.2014
173.86
169.61
143.40
128.84
128.15
123.41
122.55
108.34
77.42
77.11
76.79
61.69
59.96
34.80
27.11
13.84
12.48
7.91
ppm

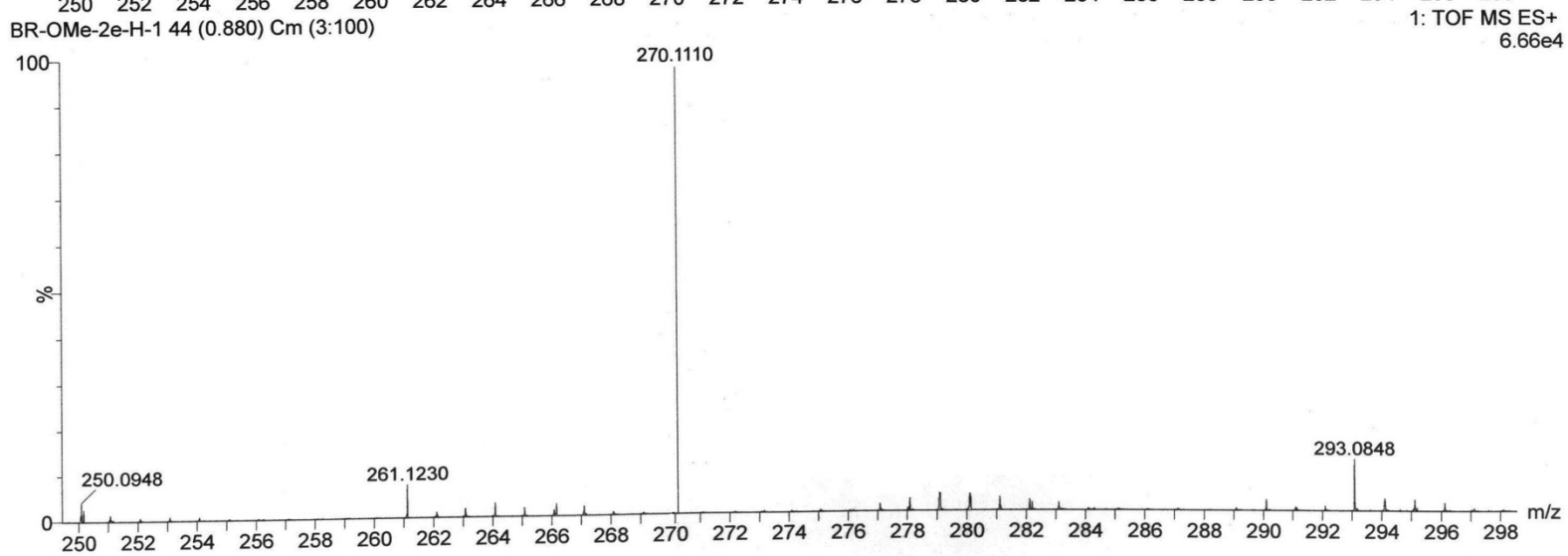

BR-MS-64 94 (1.844) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (94:96)
1: TOF MS ES+
1.55e5
100
%
0
284.1271
286.9064
287.9112
270.9785
274.9515
277.5655
282.0143
291.9217
293.9326
270 271 272 273 274 275 276 277 278 279 280 281 282 283 284 285 286 287 288 289 290 291 292 293 294 295
m/z

Ethyl 1-ethyl-3-methyl-2-oxoindoline-3-carboxylate (2m):

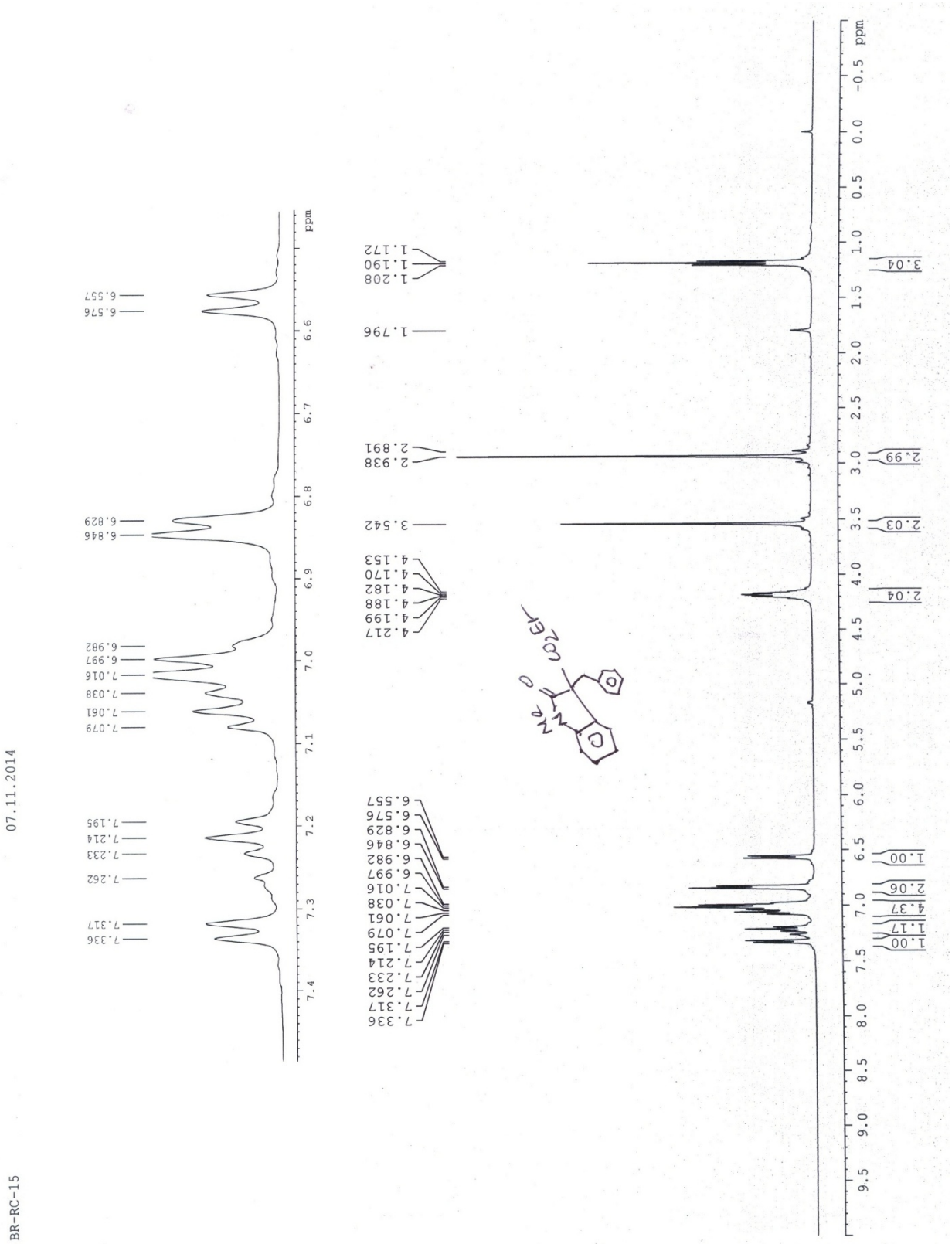



BR-RC-16
11.11.2014
174.84
169.78
142.67
130.41
128.90
123.07
122.61
108.56
77.49
77.17
76.85
61.81
54.97
34.85
19.75
13.79
12.40
ppm

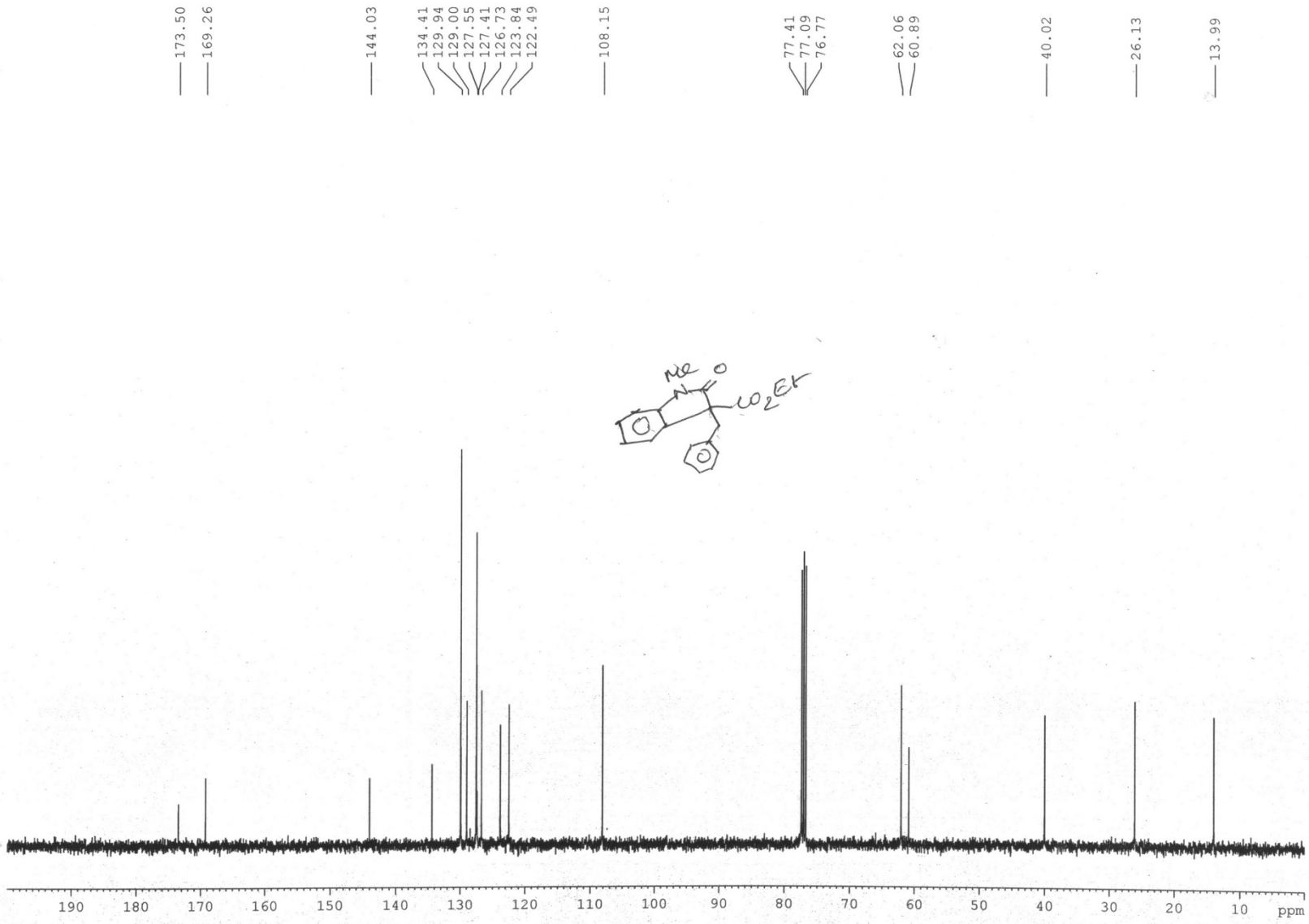

BR-11 44 (0.880) AM (Cen,4, 80.00, Ht,10000.0,0.00,0.70); Cm (3:100)
1: TOF MS ES+
6.31e4
100
%
0
270.1113
250.0951
261.1230
290.0977
293.0855
m/z
250 252 254 256 258 260 262 264 266 268 270 272 274 276 278 280 282 284 286 288 290 292 294 296 298

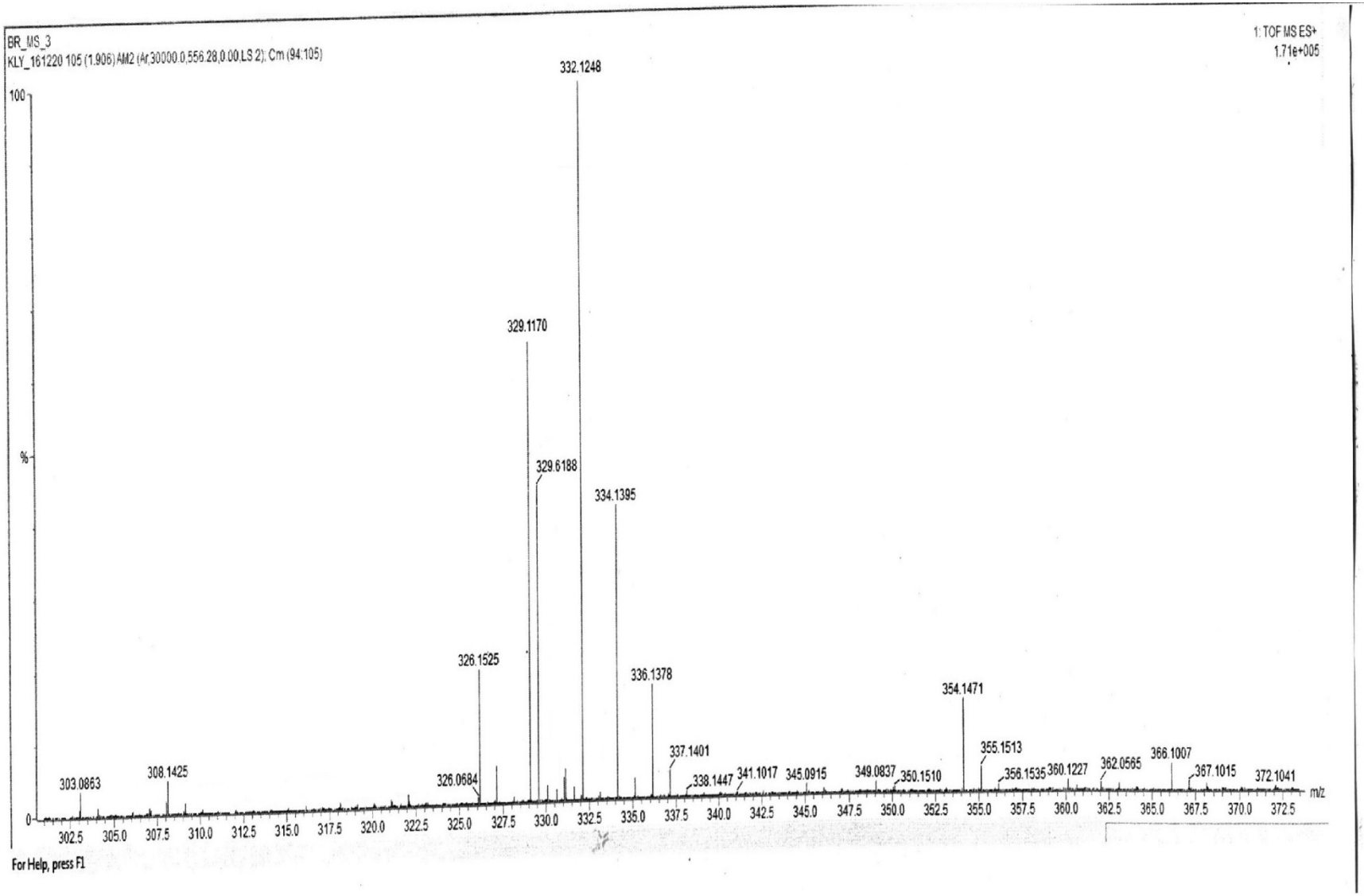

BR-OMe-2e-H-1 44 (0.880) Cm (3:100)
1: TOF MS ES+
6.66e4
100
%
0
270.1110
250.0948
261.1230
293.0848
m/z
250 252 254 256 258 260 262 264 266 268 270 272 274 276 278 280 282 284 286 288 290 292 294 296 298

Ethyl 3-benzyl-1-methyl-2-oxoindoline-3-carboxylate (2n):

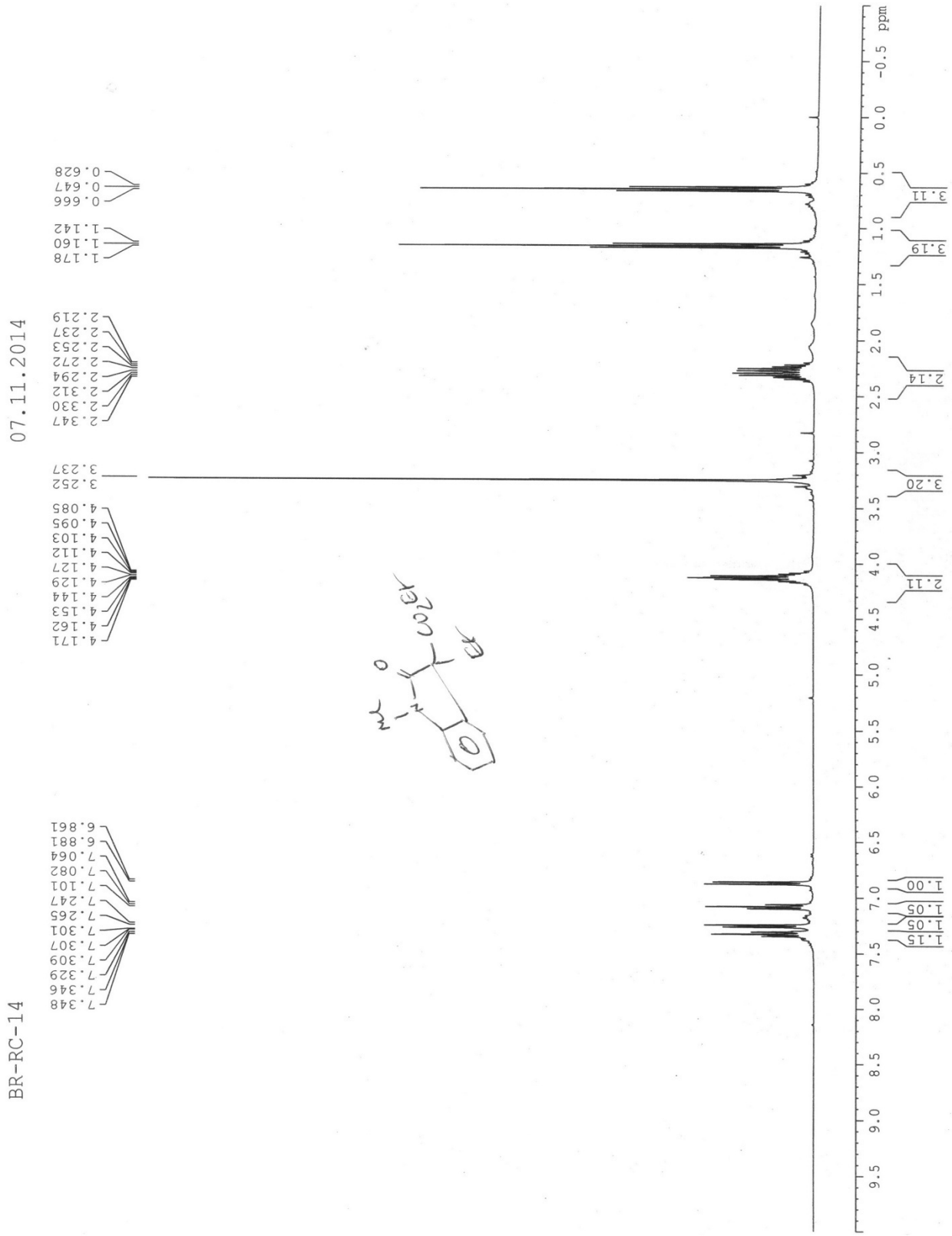


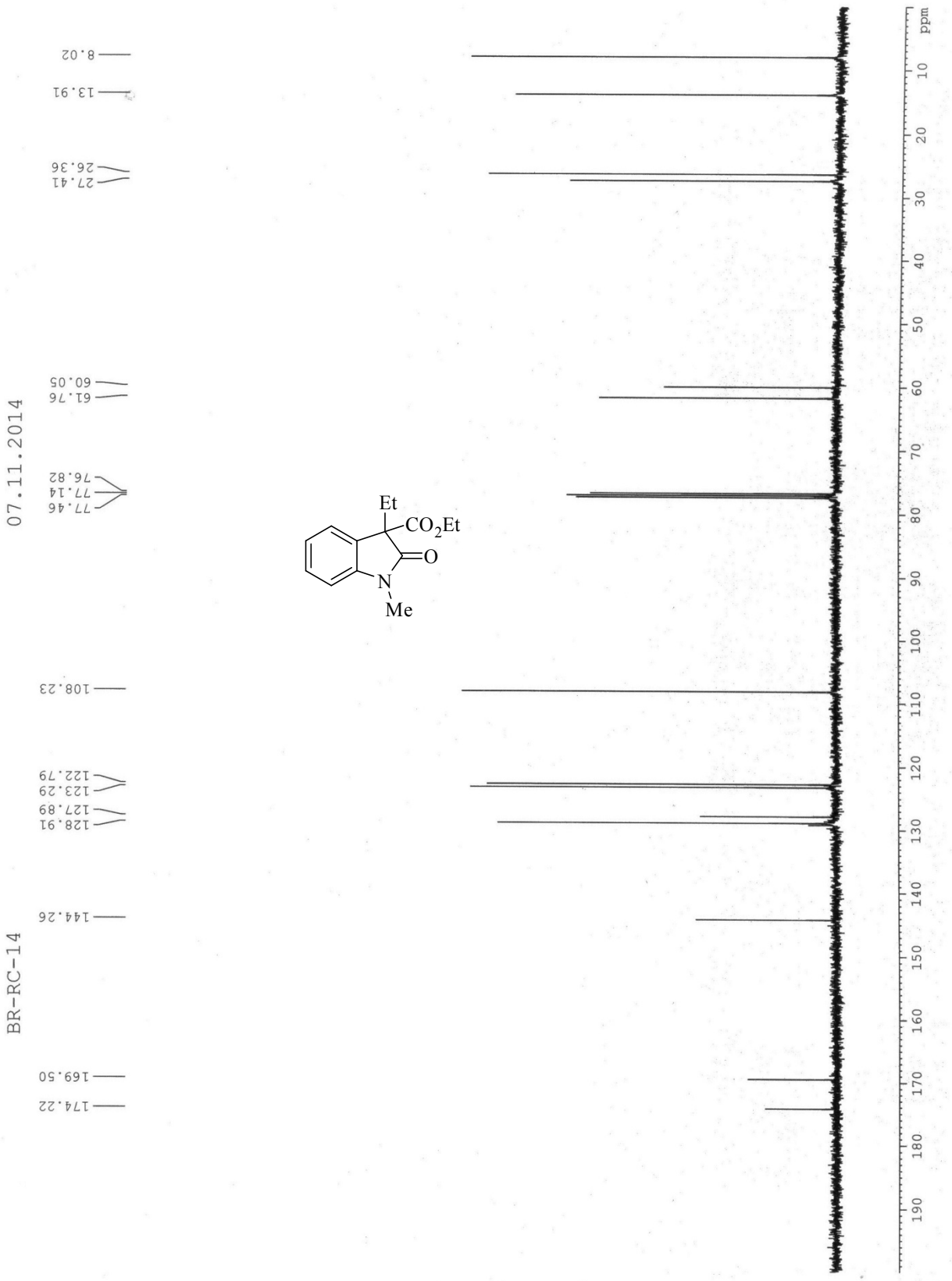
BR-RC-15 07.11.2014

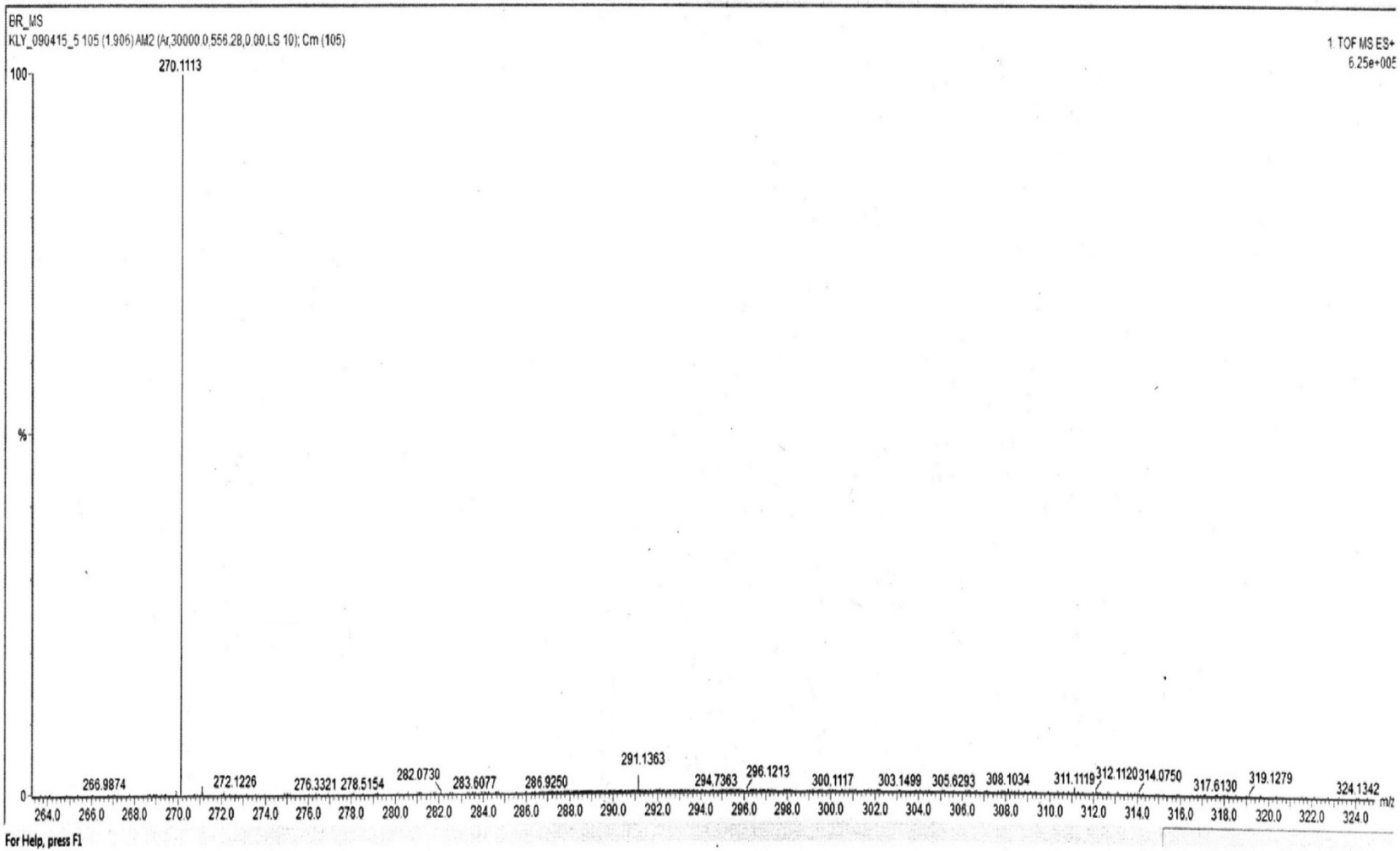



BR_MS_3
KLY_161220 105 (1.906) AM2 (Ar,30000.0,556.28,0.00,LS 2); Cm (94:105)
1: TOF MS ES+
1.71e+005
100
%
0
332.1248
329.1170
329.6188
334.1395
326.1525
336.1378
354.1471
337.1401
355.1513
303.0863
308.1425
326.0684
338.1447
341.1017
345.0915
349.0837
350.1510
356.1535
360.1227
362.0565
366.1007
367.1015
372.1041
m/z
302.5 305.0 307.5 310.0 312.5 315.0 317.5 320.0 322.5 325.0 327.5 330.0 332.5 335.0 337.5 340.0 342.5 345.0 347.5 350.0 352.5 355.0 357.5 360.0 362.5 365.0 367.5 370.0 372.5
For Help, press F1

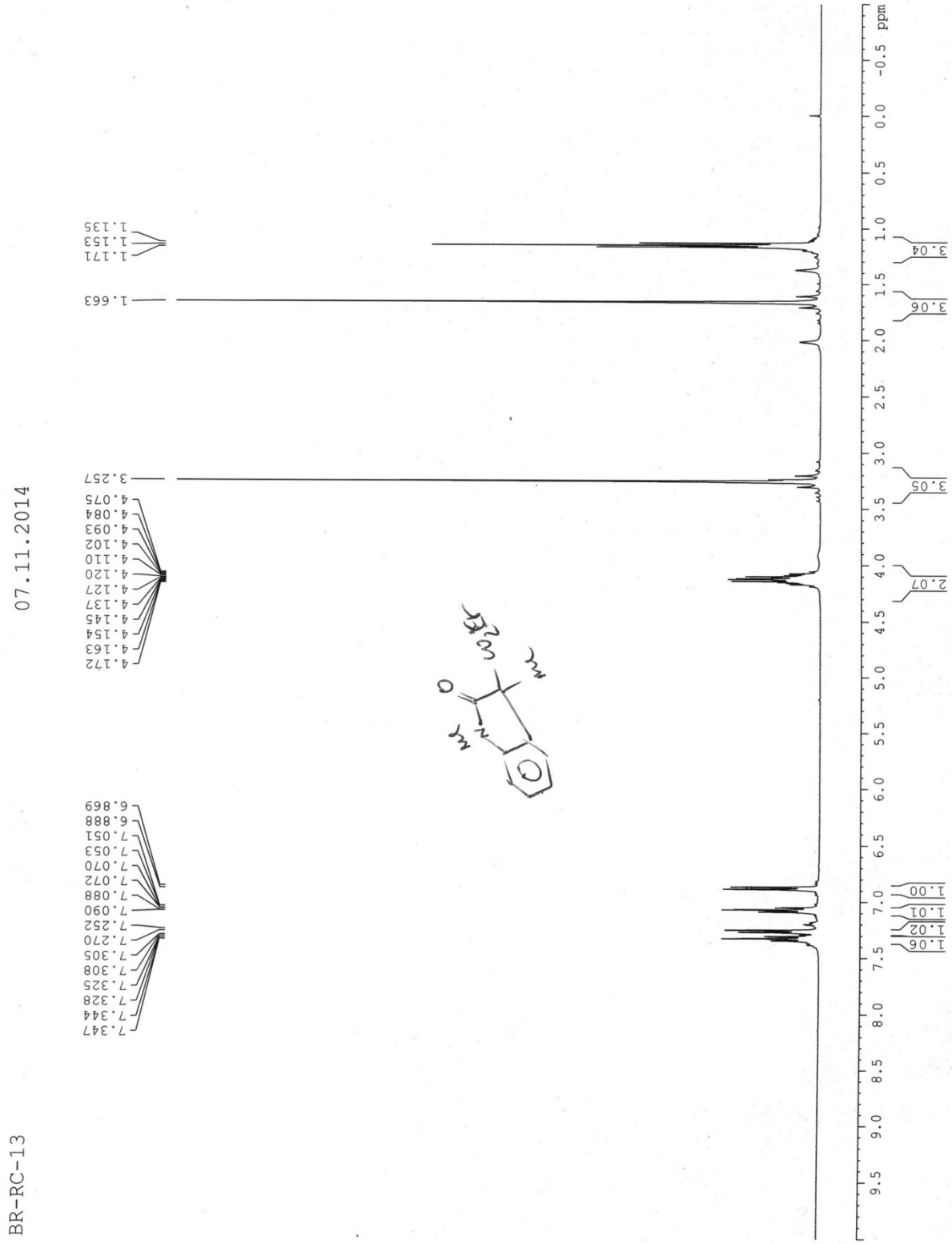
Ethyl 3-ethyl-1-methyl-2-oxoindoline-3-carboxylate (2o):

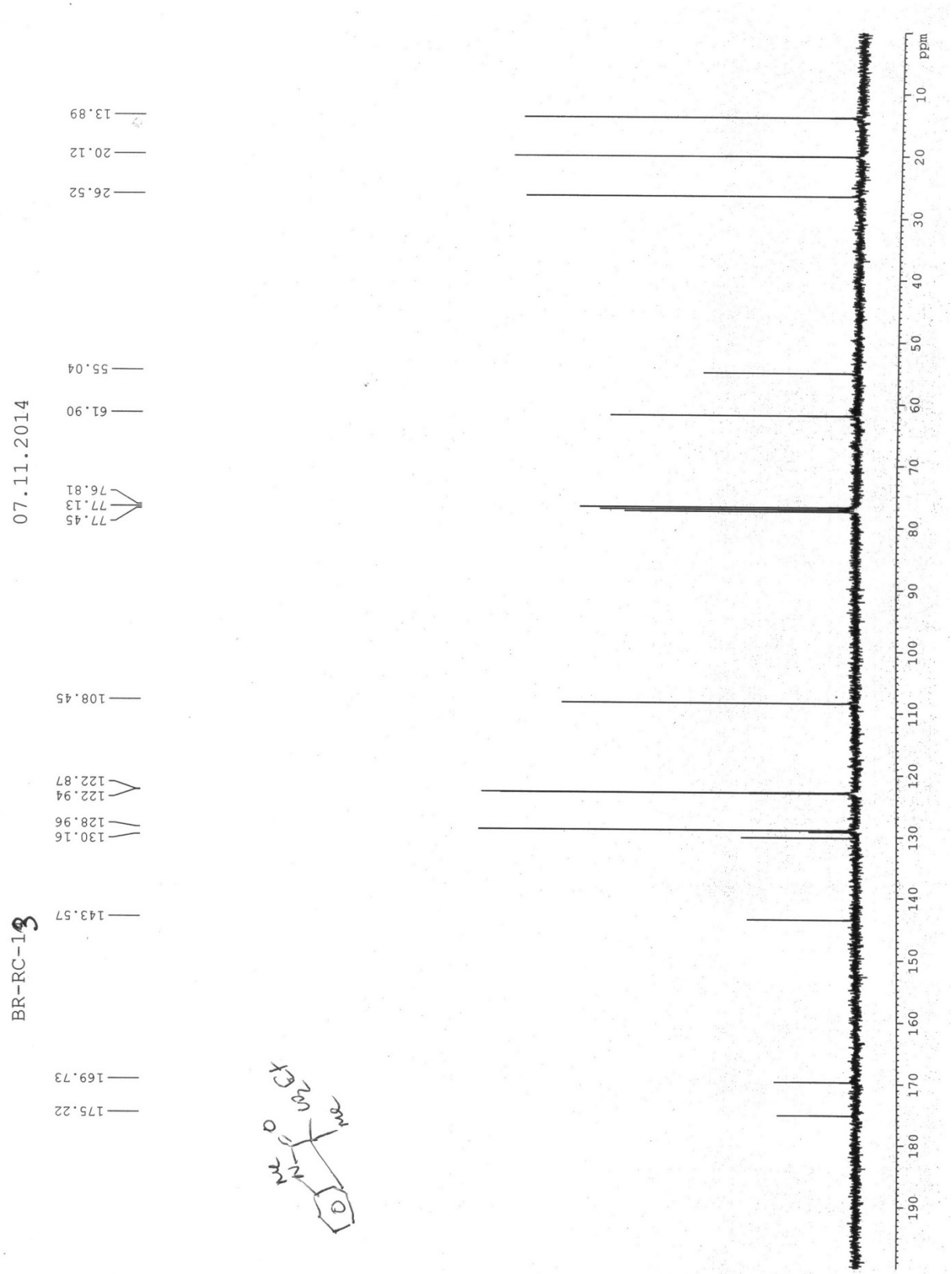


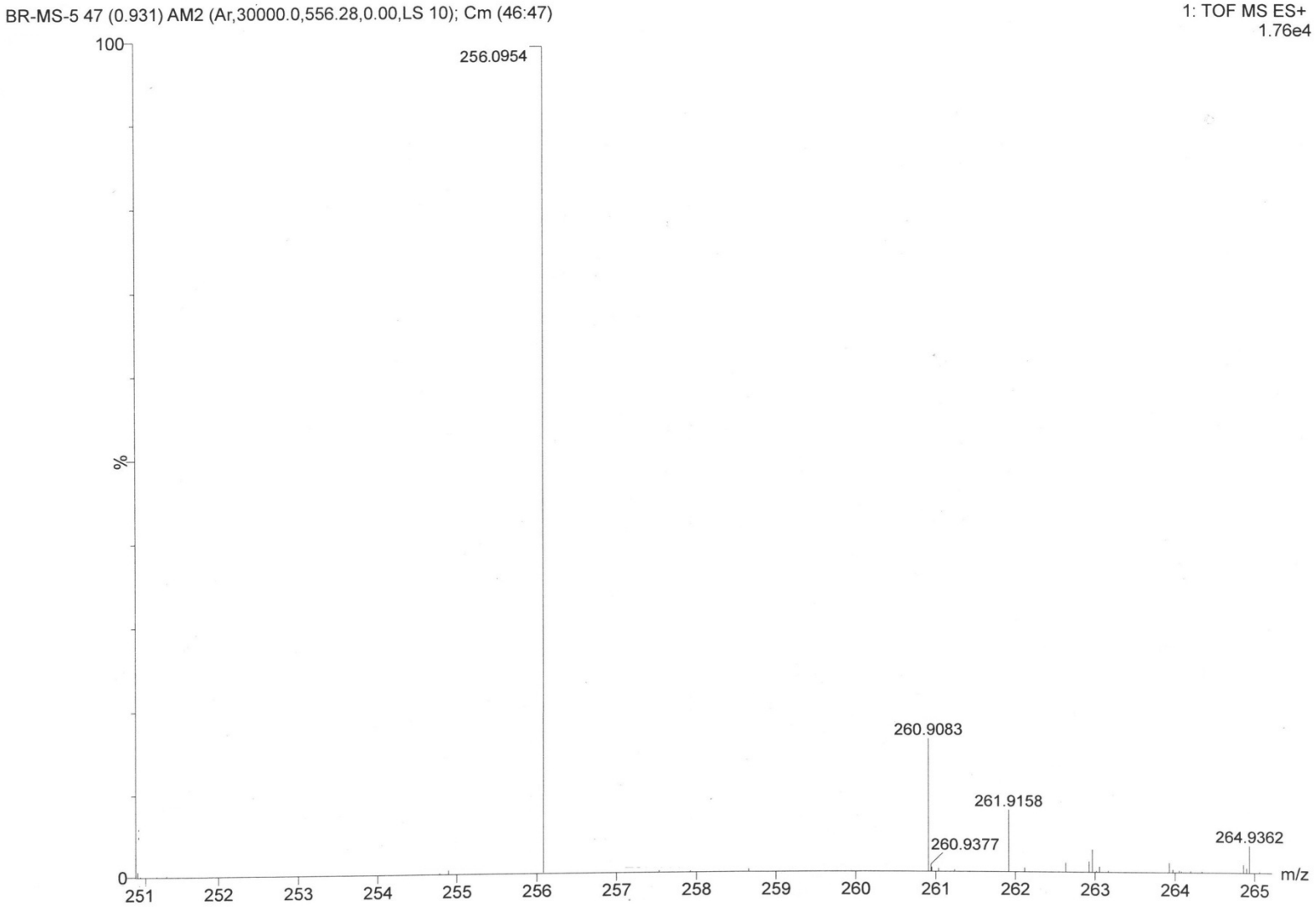

BR-RC-14
07.11.2014
174.22
169.50
144.26
128.91
127.89
123.29
122.79
108.23
77.46
77.14
76.82
61.76
60.05
27.41
26.36
13.91
8.02
Et
CO2Et
O
N
Me
ppm


BR_MS
KLY_090415_5 105 (1.906) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (105)
1: TOF MS ES+
6.25e+005
100
%
0
270.1113
266.9874
272.1226
276.3321
278.5154
282.0730
283.6077
286.9250
291.1363
294.7363
296.1213
300.1117
303.1499
305.6293
308.1034
311.1119
312.1120
314.0750
317.6130
319.1279
324.1342
264.0 266.0 268.0 270.0 272.0 274.0 276.0 278.0 280.0 282.0 284.0 286.0 288.0 290.0 292.0 294.0 296.0 298.0 300.0 302.0 304.0 306.0 308.0 310.0 312.0 314.0 316.0 318.0 320.0 322.0 324.0
m/z
For Help, press F1

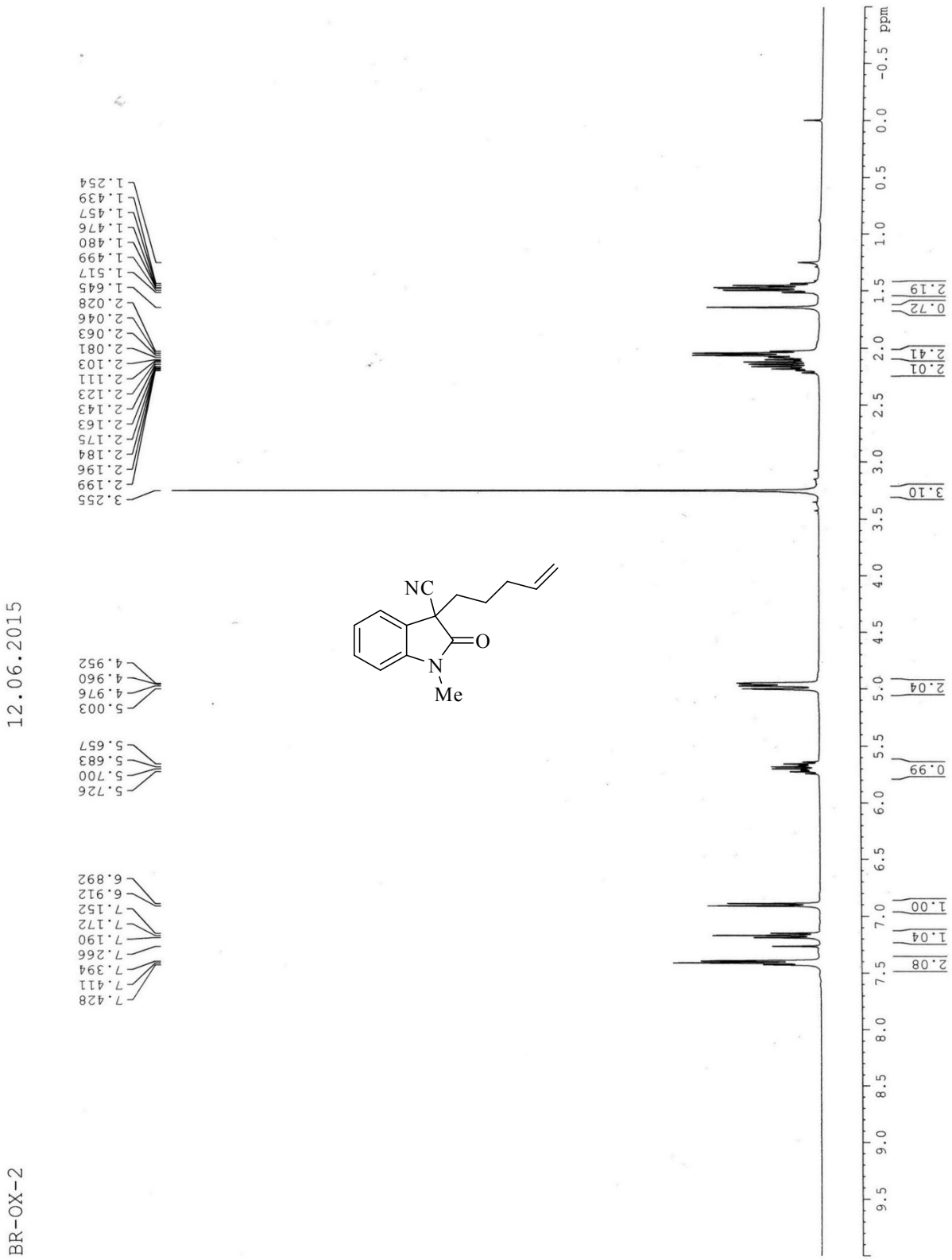
Ethyl 1,3-dimethyl-2-oxoindoline-3-carboxylate (2p):

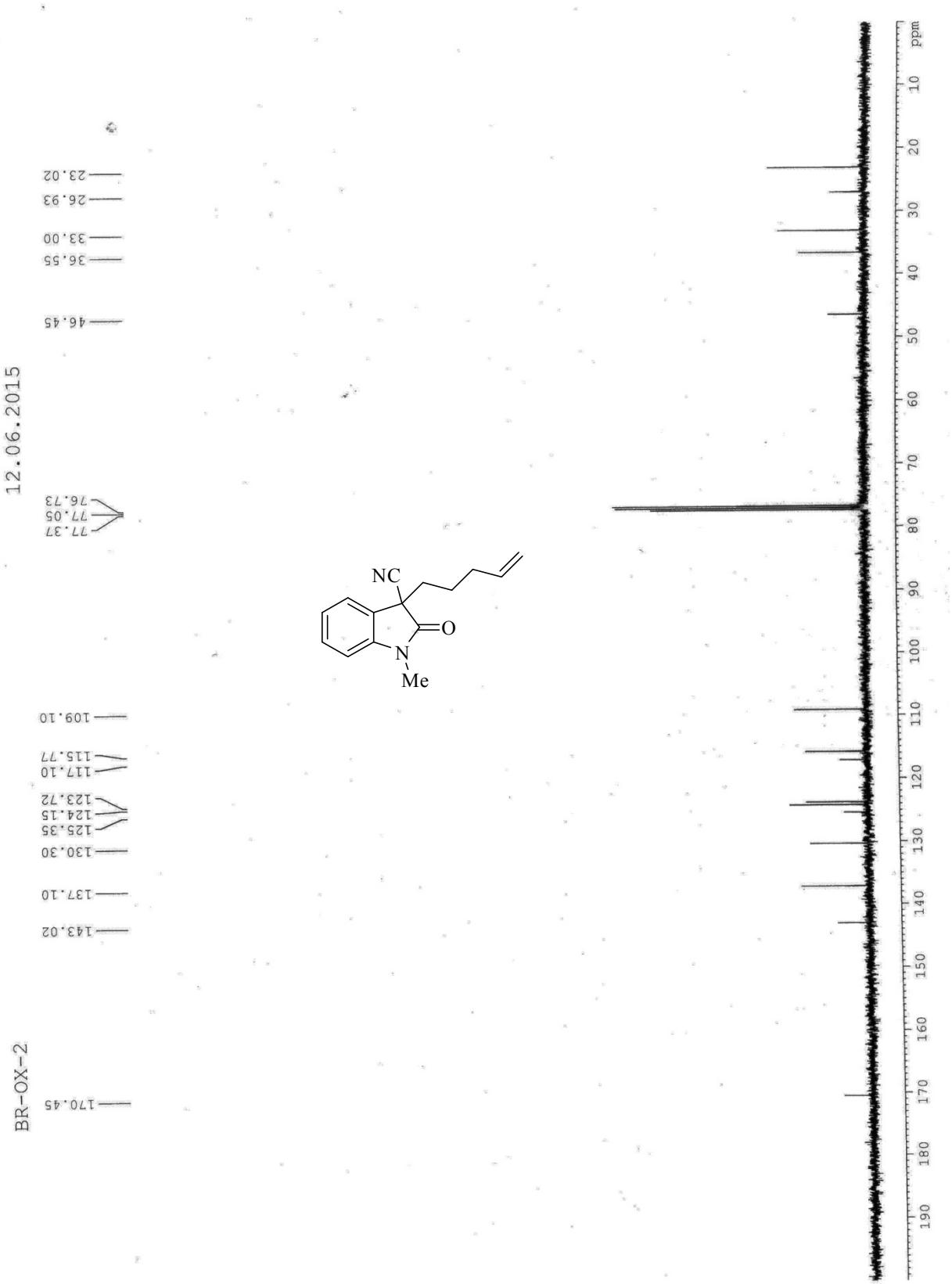


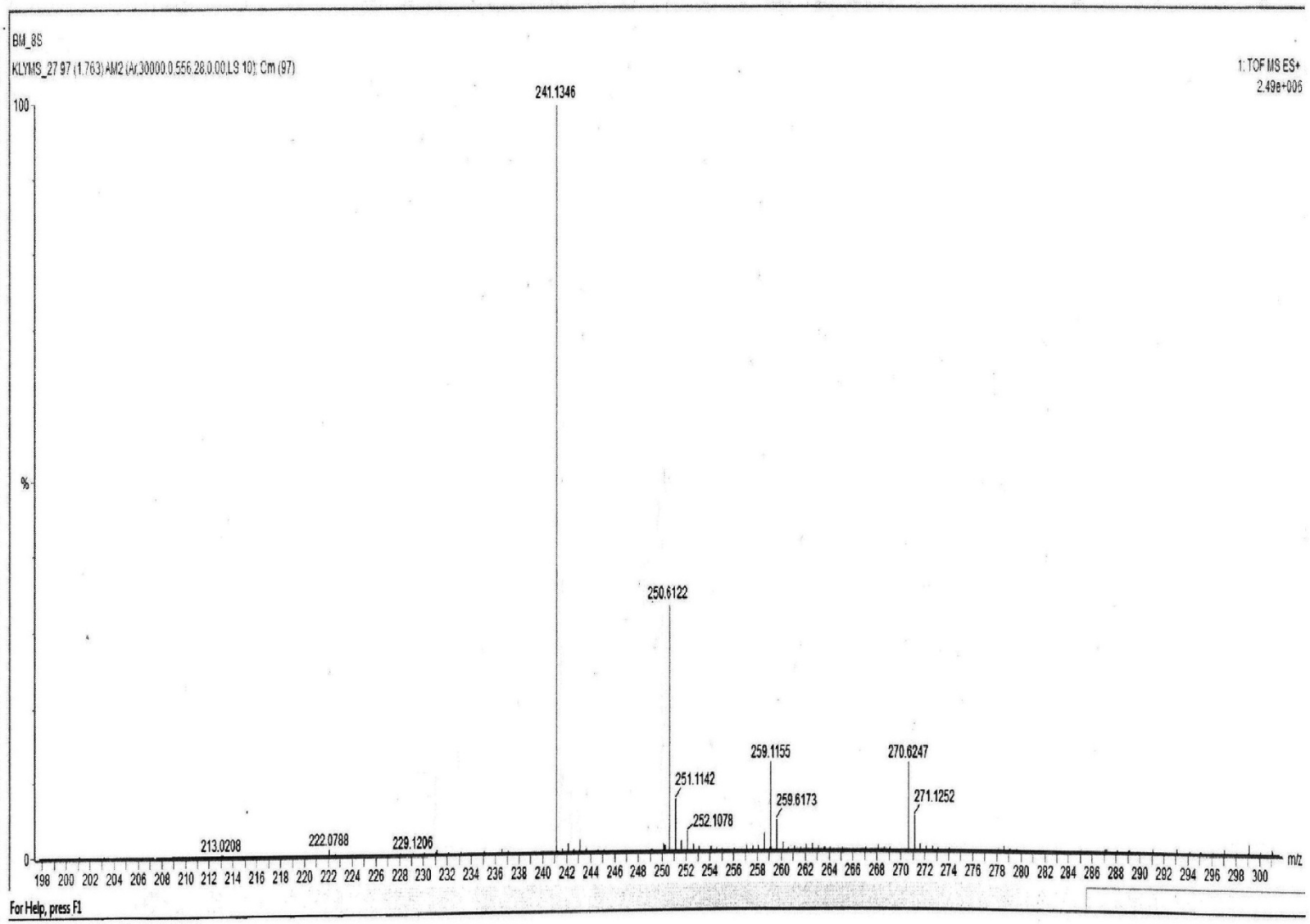

BR-RC-13
07.11.2014
175.22
169.73
143.57
130.16
128.96
122.94
122.87
108.45
77.45
77.13
76.81
61.90
55.04
26.52
20.12
13.89
ppm


BR-MS-5 47 (0.931) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (46:47)
1: TOF MS ES+
1.76e4
100
%
0
256.0954
260.9083
261.9158
260.9377
264.9362
251
252
253
254
255
256
257
258
259
260
261
262
263
264
265
m/z

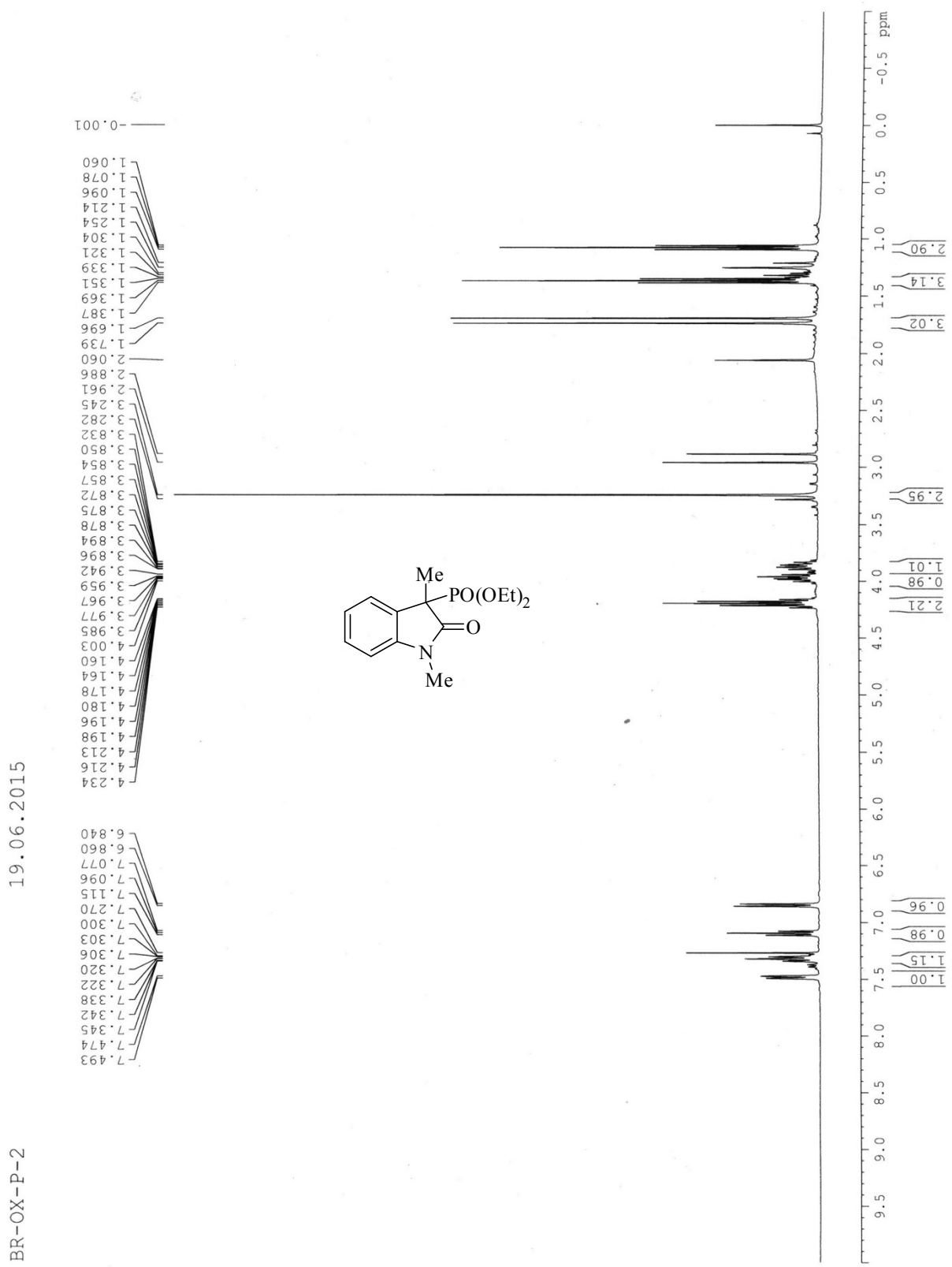
1-Methyl-2-oxo-3-(pent-4-enyl)indoline-3-carbonitrile (2q):

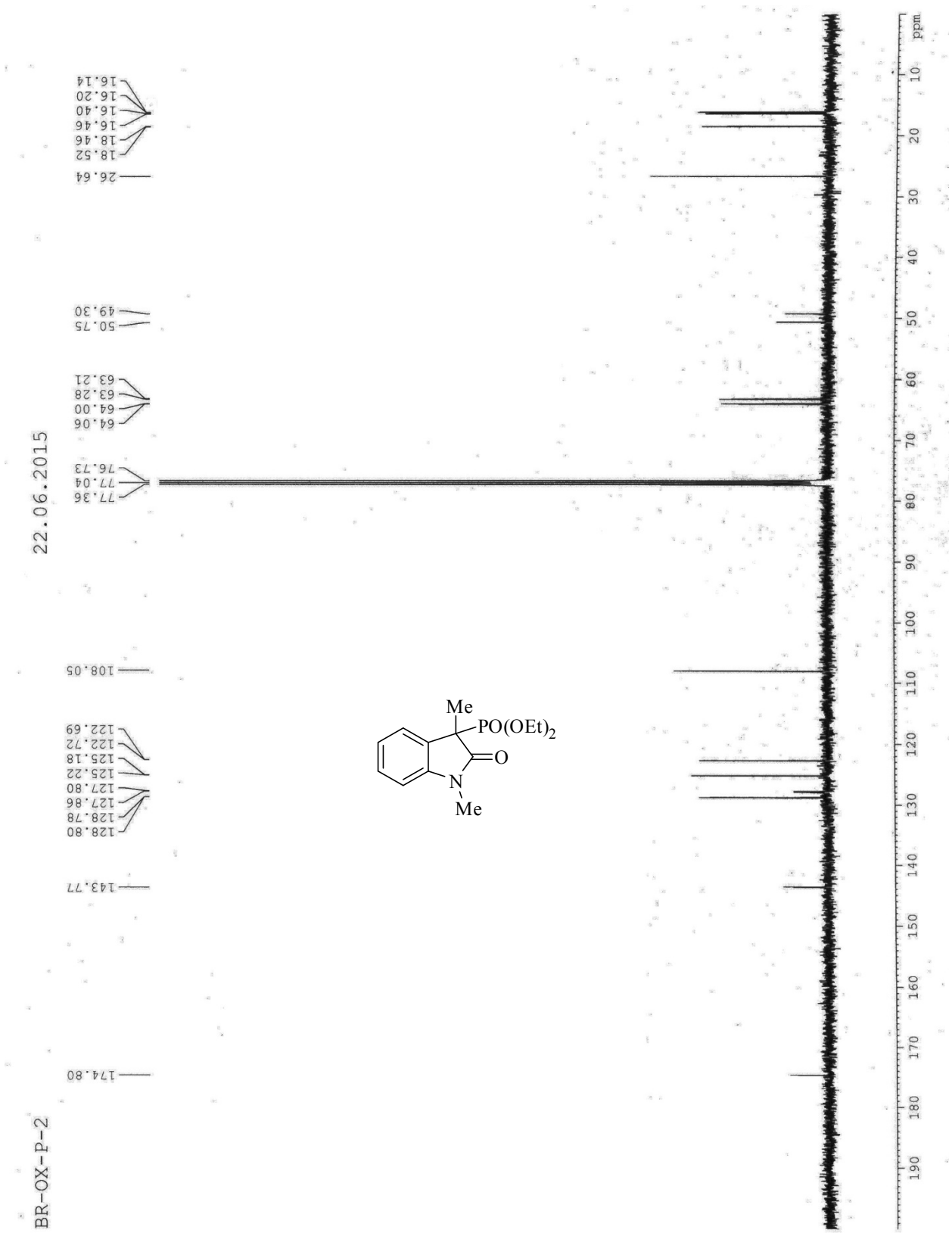


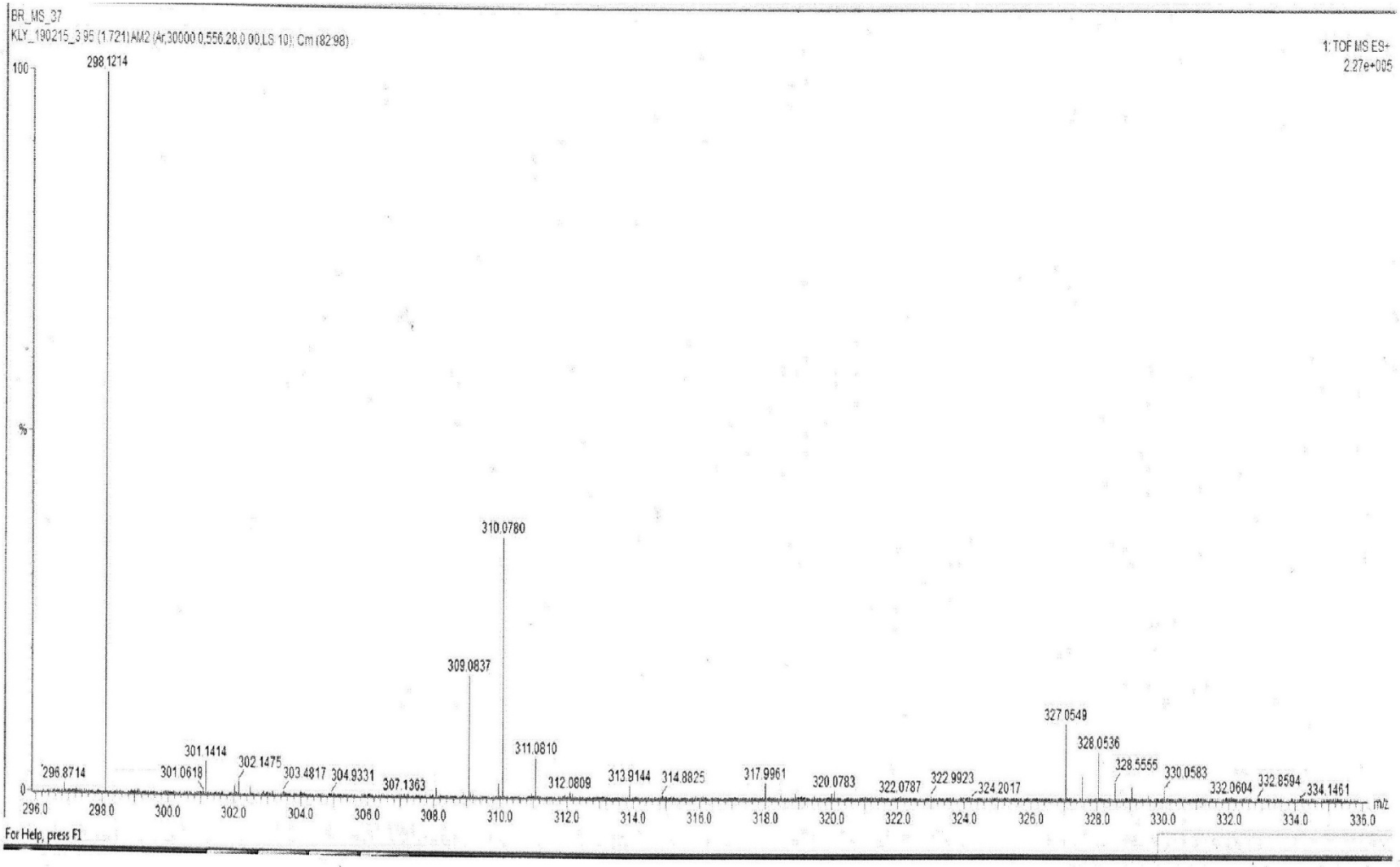

BR-OX-2
12.06.2015
170.45
143.02
137.10
130.30
125.35
124.15
123.72
117.10
115.77
109.10
77.37
77.05
76.73
46.45
36.55
33.00
26.93
23.02
NC
O
N
Me
ppm


BM_8S
KLYMS_27 97 (1.763) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (97)
1: TOF MS ES+
2.49e+005
100
%
0
241.1346
250.6122
251.1142
252.1078
259.1155
259.6173
270.6247
271.1252
213.0208
222.0788
229.1206
m/z
For Help, press F1

Diethyl 1,3-dimethyl-2-oxoindolin-3-ylphosphonate (2r):




BR-OX-P-2
22.06.2015
174.80
143.77
128.80
128.78
127.86
127.80
125.22
125.18
122.72
122.69
108.05
77.36
77.04
76.73
64.06
64.00
63.28
63.21
50.75
49.30
26.64
18.52
18.46
16.46
16.40
16.20
16.14
Me
PO(OEt)2
O
N
Me
ppm


BR_MS_37
KLY_190215_3 95 (1.721) AM2 (Ar,30000.0,556.28,0.00,LS 10); Cm (82:98)
1: TOF MS ES+
2.27e+005
100
%
0
298.1214
310.0780
309.0837
311.0810
327.0549
328.0536
328.5555
330.0583
332.0604
332.8594
334.1461
301.1414
302.1475
303.4817
304.9331
307.1363
312.0809
313.9144
314.8825
317.9961
320.0783
322.0787
322.9923
324.2017
296.8714
301.0618
296.0
298.0
300.0
302.0
304.0
306.0
308.0
310.0
312.0
314.0
316.0
318.0
320.0
322.0
324.0
326.0
328.0
330.0
332.0
334.0
336.0
m/z
For Help, press F1