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New Journal of Chemistry

Dibromination of Alkenes with LiBr and H_2O_2 Under Mild Conditions

*Nayara Silva Martins and Eduardo E. Alberto**

Department of Chemistry, Federal University of Minas Gerais, Belo Horizonte, MG, Brazil; Phone (+55) 31 3409–5696; E-mail: albertoee@ufmg.br

Supporting Information

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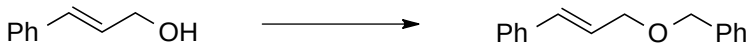
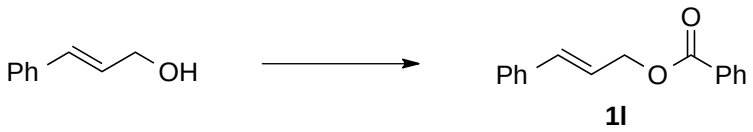
Procedures and Characterization Data for Starting Materials



1k

To a three-neck round-bottom flask under argon and at 0 ºC (ice bath) NaH (60% dispersion in mineral oil, 0.8 g, 20 mmol) was added to a solution of cinnamyl alcohol (98% purity, 2.74 g, 20 mmol) dissolved in dry THF (20 mL). After ten minutes, benzyl chloride (97% purity, 2.4 mL, 20 mmol) was added and the reaction mixture stirred overnight at room temperature. Then, MeOH (20 mL) was slowly added followed by distilled water (100 mL). The product was extracted with AcOEt (3 x 15 mL) and the combined organic phases were dried under $MgSO_4$, filtered and solvents removed under reduced pressure. Purification was performed by flash chromatography (hexanes/AcOEt = 98/2). Further purification (to remove remaining benzyl chloride) was accomplished by stirring the product at 60 ºC under high vacuum during 3 hours. Product **1n** was obtained as a pale yellow oil (2.13 g, 48% yield);[1] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.38-7.20 (m, 10H), 6.62 (d, *J* = 15.9 Hz, 1H), 6.35-6.28 (m, 1H), 4.55 (s, 2H), 4.18 (d, *J* = 6.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 138.6, 137.0, 132.8, 128.8, 128.7, 128.1, 127.95, 127.92, 126.8, 126.4, 72.5, 71.0.

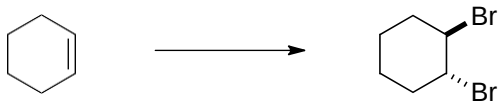


1l

To an one-neck round-bottom flask at 0 ºC (ice bath) benzoyl chloride (3.2 mL, 27.5 mmol) was dropwise added to a solution of cinnamyl alcohol (98% purity, 3.2 mL, 25 mmol) dissolved in DCM (80 mL). After one hour of reaction at room temperature, a solution of Et_3N (3.5 mL, 25 mmol) dissolved in DCM (20 mL) was added and the reaction stirred at room temperature overnight. The reaction mixture was washed with water (2 x 50 mL) and then with NaOH 0.2 M (2 x 50 mL). The organic phase was dried under $MgSO_4$, filtered and the solvent removed under reduced pressure. Purification by flash chromatography (hexanes/AcOEt = 95/5) gave product **1m** as a colorless oil (3.77 g, 63% yield);[2] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 8.09-8.07 (m, 2H), 7.56-7.52 (m, 1H), 7.45-7.40 (m, 4H), 7.33-7.23 (m, 3H), 6.73 (d, *J* = 15.9 Hz, 1H), 6.43-6.36 (m, 1H), 4.98

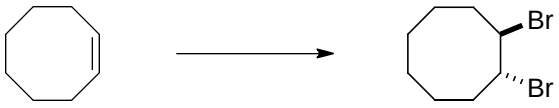
(d, *J* = 6.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 166.6, 136.5, 134.5, 133.2, 129.9, 128.9, 128.6, 128.3, 126.9, 123.5, 65.8.

Characterization of Brominated Products



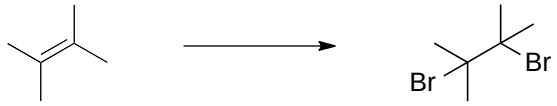
1a **2a**

The general procedure was followed using **1a** (102 μL, 1.0 mmol) as substrate, LiBr (208 mg, 2.4 mmol) and H_2O_2 8.8 M (136 μL, 1.2 mmol). At the end of the reaction the product was extracted with a 9/1 solution of hexanes/AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 98/2) to afford a pale yellow oil. **Condition A**: 1 hour of reaction, 193 mg, 80% yield; **Condition B**: 2 hours of reaction, 135 mg, 56% yield.[3] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 4.46 (br, 2H), 2.49-2.43 (m, 2H), 1.91-1.79 (m, 4H), 1.53-1.50 (m, 2H); ^{13}C NMR ($CDCl_3$, 400 MHz) δ = 55.1, 31.9, 22.3.



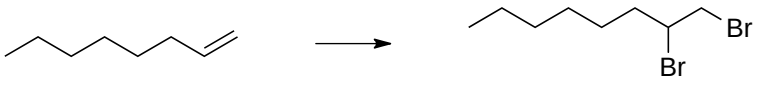
1b **2b**

The general procedure was followed using **1b** (95% purity, 137 μL, 1.0 mmol) as substrate, LiBr (208 mg, 2.4 mmol) and H_2O_2 8.8 M (136 μL, 1.2 mmol). At the end of the reaction the product was extracted with a 9/1 solution of hexanes/AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 98/2) to afford a pale yellow oil. **Condition A**: 1 hour of reaction, 234 mg, 87% yield; **Condition B**: 2 hours of reaction, 144 mg, 53% yield.[4] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 4.59-4.58 (m, 2H), 2.44-2.38 (m,
2H), 2.12-2.11 (m, 2H), 2.07-1.88 (m, 2H), 1.70-1.47 (m, 6H); ^{13}C NMR ($CDCl_3$, 400 MHz) δ = 61.8, 33.5, 26.2, 25.7.



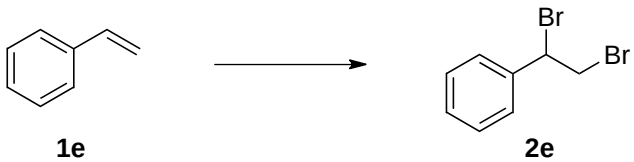
1c **2c**

The general procedure was followed using **1c** (95% purity, 121 μL, 1.0 mmol) as substrate, LiBr (350 mg, 4.0 mmol) and H_2O_2 8.8 M (227 μL, 2.0 mmol). At the end of the reaction the product was extracted with a 9/1 solution of hexanes/AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 98/2) to afford a white solid. **Condition A**: 1 hour of reaction, 183 mg, 75% yield; **Condition B**: 2 hours of reaction, 24 mg, 10% yield, m.p. = 70 ºC ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.95 (s). ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 74.1, 32.1; MS (EI, 70 eV): m/z (%) = 163/165 (100/97) $[M - Br]^+$, 83 (64) $[C_6H_{11}]^+$, 69 (55) $[C_5H_9]^+$, 55 (83) $[C_4H_7]^+$. Elem. analysis calculated: C = 29.54, H = 4.96, found: C = 29.39, H = 4.63.



1d **2d**

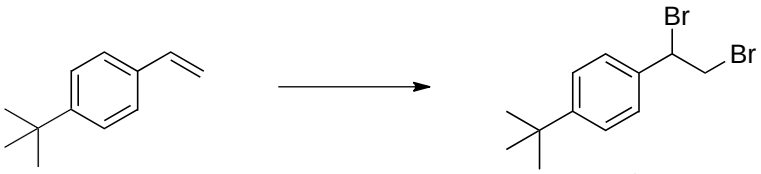
The general procedure was followed using **1d** (98% purity, 160 μL, 1.0 mmol) as substrate, LiBr (208 mg, 2.4 mmol) and H_2O_2 8.8 M (136 μL, 1.2 mmol). At the end of the reaction the product was extracted with a 9/1 solution of hexanes/AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 98/2) to afford a pale yellow oil. **Condition A**: 1 hour of reaction, 249 mg, 92% yield; **Condition B**: 2 hours of reaction, 143 mg, 53% yield.[4] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 4.20-4.14 (m, 1H), 3.85 (dd, J^1 = 10.2 Hz, J^2 = 4.4 Hz, 1H), 3.63 (t, J = 9.9 Hz, 1H), 2.18-2.09 (m, 1H), 1.83-1.173 (m, 1H), 1.62-1.51 (m, 1H), 1.51-1.30 (m, 7H), 0.91-0.88 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 53.1, 36.3, 36.0, 31.6, 28.5, 26.7, 22.5, 14.0.



1e **2e**

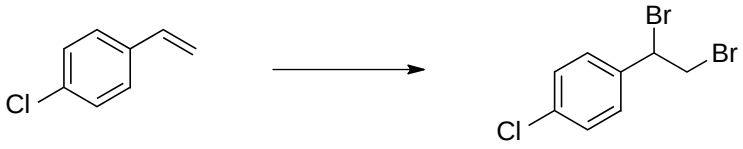
The general procedure was followed using **1e** (115 μL, 1.0 mmol) as substrate, LiBr (208 mg, 2.4 mmol) and H_2O_2 8.8 M (136 μL, 1.2 mmol). At the end of the reaction the product was extracted with a 9/1 solution of hexanes/AcOEt (3 x 10 mL) and purified by

silica gel cromatography (hexanes/AcOEt = 98/2) to afford a white solid. **Condition A**: 1 hour of reaction, 263 mg, >99% yield; **Condition B**: 2 hours of reaction, 161 mg, 61% yield, m.p. = 72–73 °C (literature = 71–73 ºC)[4]. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.31-7.22 (m, 5H), 5.04 (dd, J^1 = 16.1 Hz, J^2 = 10.6 Hz, 1H), 3.99-3.89 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 138.5, 129.1, 128.8, 127.6, 50.9, 35.0.



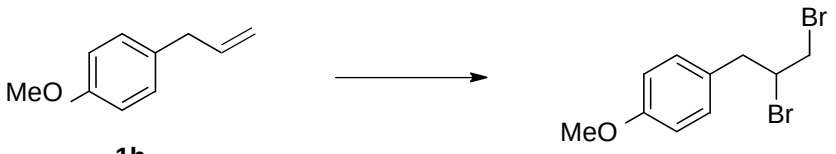
1f **2f**

The general procedure was followed using **1f** (95% purity, 193 µL, 1.0 mmol) as substrate, LiBr (208 mg, 2.4 mmol) and H_2O_2 8.8 M (136 µL, 1.2 mmol). At the end of the reaction the product was extracted with a 9/1 solution of hexanes/AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 98/2) to afford a white solid. **Condition A**: 1 hour of reaction, 320 mg, >99% yield; **Condition B**: 2 hours of reaction, 296 mg, 93% yield, m.p. = 48–49 °C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.40-7.31 (m, 4H), 5.15 (dd, J^1 = 10.2 Hz, J^2 = 5.8 Hz, 1H), 4.09-4.00 (m, 1H), 1.32 (s, 9H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 152.5, 135.8, 127.5, 126.1, 51.5, 35.4, 35.0, 31.5; HRMS (APCI, positive mode) *m/z* calculated for $C_{12}H_{16}{}^{79}Br$ $[M - Br]^+$ 239.0430, found: 239.0434.



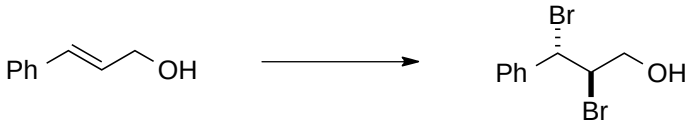
1g **2g**

The general procedure was followed using **1g** (97% purity, 132 µL, 1.0 mmol) as substrate, LiBr (208 mg, 2.4 mmol) and H_2O_2 8.8 M (136 µL, 1.2 mmol). At the end of the reaction the product was extracted with a 9/1 solution of hexanes/AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 98/2) to afford a white wax. **Condition A**: 1 hour of reaction, 228 mg, 77% yield; **Condition B**: 2 hours of reaction, 151 mg, 51% yield.[5] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.35-7.30 (m, 4H), 5.09 (dd, J^1 = 11.0 Hz, J^2 = 5.1 Hz, 1H), 4.04 (dd, J^1 = 10.3 Hz, J^2 = 5.1 Hz, 1H), 3.94 (t, J = 10.7Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 137.4, 135.2, 129.4, 129.3, 49.9, 35.0.



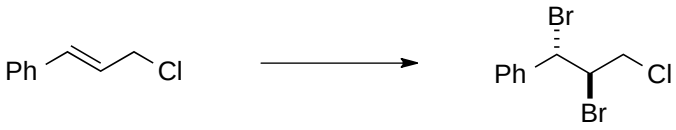
1h **2h**

The general procedure was followed using **1h** (98% purity, 157 μL, 1.0 mmol) as substrate, LiBr (208 mg, 2.4 mmol) and H_2O_2 8.8 M (136 μL, 1.2 mmol). At the end of the reaction the product was extracted with AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 98/2) to afford a pale yellow oil. **Condition A**: 2 hours of reaction, 297 mg, 96% yield; **Condition B**: 2 hours of reaction, 244 mg, 79% yield.[4] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.19 (d, *J* = 8.3 Hz, 2H), 6.86 (d, *J* = 8.3 Hz, 2H), 4.35-4.28 (m, 1H), 3.79 (s, 3H), 3.60 (t, *J* = 9.3 Hz, 1H), 3.40 (dd, J^1 = 14.7 Hz, J^2 = 4.7 Hz, 1H), 3.09 (dd, J^1 = 14.6 Hz, J^2 = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 158.7, 130.5, 128.7, 113.9, 55.2, 52.9, 41.0, 35.9.



1i **2i**

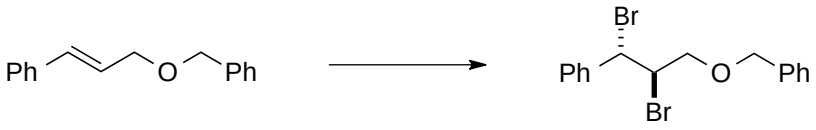
The general procedure was followed using **1i** (98% purity, 137 mg, 1.0 mmol) as substrate, LiBr (350 mg, 4.0 mmol) and H_2O_2 8.8 M (227 μL, 2.0 mmol). At the end of the reaction the product extracted with AcOEt and purified by silica gel cromatography (hexanes/AcOEt = 90/10) affording a white solid. **Condition A**: 6 hours of reaction, 239 mg, 81% yield; **Condition B**: 6 hours of reaction, 253 mg, 86% yield, m.p. = 69 °C (literature = 69 ºC).[3] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.38-7.34 (m, 5H), 5.27 (d, *J* = 11.0 Hz, 1H), 4.71-4.67 (m, 1H), 4.34-4.22 (m, 2H), 2.23 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 140.1, 129.2, 129.0, 128.1, 66.1, 59.5, 52.5.



1j **2j**

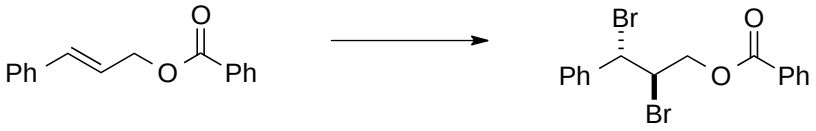
The general procedure was followed using **1j** (139 μL, 1.0 mmol) as substrate, LiBr (208 mg, 2.4 mmol) and H_2O_2 8.8 M (136 μL, 1.2 mmol). At the end of the reaction the product was extracted with a 9/1 solution of hexanes/AcOEt (3 x 10 mL) and purified by

silica gel cromatography (hexanes/AcOEt = 98/2) to afford a white solid. **Condition A**: 6 hours of reaction, 247 mg, 79% yield; **Condition B**: 6 hours of reaction, 275 mg, 88% yield, m.p. = 102-103 °C. The product was obtained as a 17/1 mixture of diastereoisomers (assessed by the examination of the ^{1}H NMR spectrum) ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.42-7.35 (m, 5H), 5.30 (d, *J* = 9.9 Hz, 1H), 4.79-4.75 (m, 1H), 4.38-4.34 (m, 1H), 4.09-4.06 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 139.0, 129.4, 129.0, 128.4, 55.1, 53.2, 49.5. HRMS (APCI, positive mode) *m/z* calculated for $C_9H_9{}^{79}BrCl$ $[M - Br]^+$ 230.9571, found: 230.9572.



1k **2k**

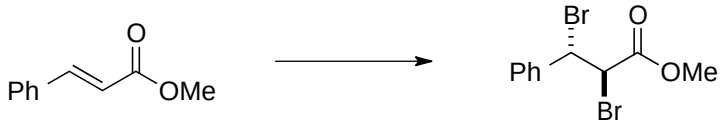
The general procedure was followed using **1k** (210 mg, 1.0 mmol) as substrate, LiBr (350 mg, 4.0 mmol) and H_2O_2 8.8 M (227 μL, 2.0 mmol). At the end of the reaction the product was extracted with AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 95/5) to afford a white solid. **Condition A**: 6 hours of reaction, 300 mg, 78% yield; **Condition B**: 6 hours of reaction, 265 mg, 69% yield, m.p. = 64-65 °C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.40-7.33 (m, 10H), 5.36 (d, *J* = 10.1 Hz, 1H), 4.67-4.63 (m, 3H), 4.21-4.17 (m, 1H), 4.00-3.97 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 139.9, 137.9, 129.1, 128.8, 128.7, 128.3, 128.14, 128.08, 73.8, 72.7, 55.0, 53.0; HRMS (APCI, positive mode) *m/z* calculated for $C_{16}H_{16}{}^{79}BrO$ $[M - Br]^+$ 303.0379, found: 303.0384.



1l **2l**

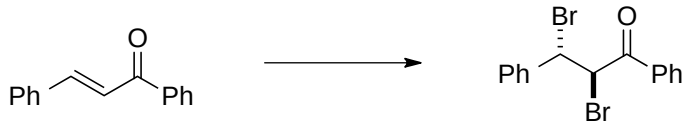
The general procedure was followed using **1l** (238 mg, 1.0 mmol) as substrate, LiBr (350 mg, 4.0 mmol) and H_2O_2 8.8 M (227 μL, 2.0 mmol). At the end of the reaction the product was extracted with AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 95/5) to afford a white solid. **Condition A**: 6 hours of reaction, 225 mg, 57% yield; **Condition B**: 6 hours of reaction, 311 mg, 78% yield, m.p. = 111-112 °C.^{1}H NMR ($CDCl_3$, 400 MHz) δ = 8.12 (d, *J* = 7.6 Hz, 2H), 7.63-7.59 (m, 1H), 7.51-7.34 (m, 7H), 5.28 (d, *J* = 10.3 Hz, 1H), 5.04-5.00 (m, 1H), 4.97-4.96 (m, 1H), 4.85-4.82 (m,

1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 166.1, 139.6, 133.6, 130.0, 129.8, 129.3, 129.0 128.8, 128.2, 67.5, 52.9, 52.8. HRMS (APCI, positive mode) *m/z* calculated for $C_{16}H_{14}{}^{79}BrO_2$ $[M - Br]^+$ 317.0172, found: 317.0173.



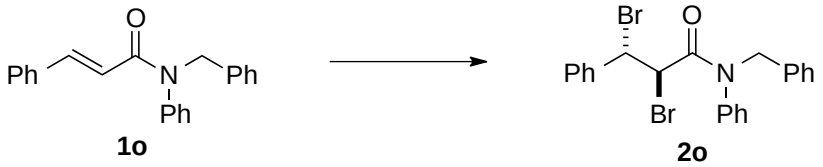
1m **2m**

The general procedure was followed using **1m** (162 mg, 1.0 mmol) as substrate, LiBr (350 mg, 4.0 mmol) and H_2O_2 8.8 M (227 μL, 2.0 mmol). At the end of the reaction the product was extracted with AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 90/10) to afford a white solid. **Condition A**: 48 hours of reaction, 269 mg, 84% yield; **Condition B**: 48 hours of reaction, 179 mg, 56% yield (NMR yield), m.p. = 112-113 °C (literature = 115-116 °C ºC).[6] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.39-7.37 (m, 5H), 5.34 (d, *J* = 11.7 Hz, 1H), 4.84 (d, *J* = 11.7 Hz, 1H), 3.90 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 168.6, 137.8, 129.6, 129.1, 128.3, 53.6, 50.9, 46.9.



1n **2n**

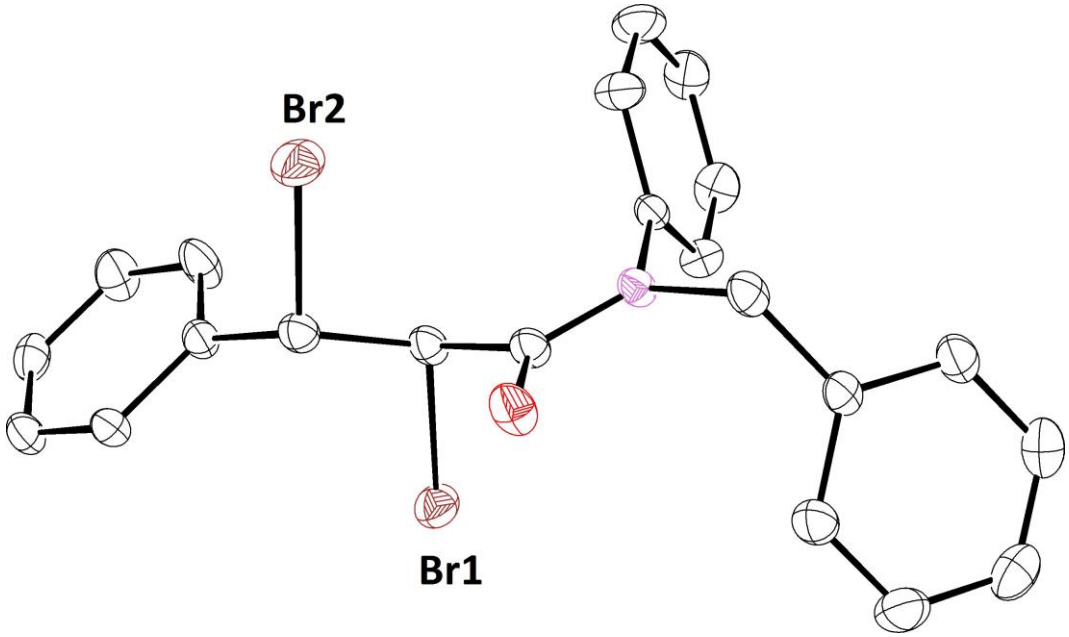
The general procedure was followed using **1n** (215 mg, 1.0 mmol) as substrate, LiBr (350 mg, 4.0 mmol) and H_2O_2 8.8 M (227 μL, 2.0 mmol). At the end of the reaction the product was extracted with AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 90/10) to afford a white solid. **Condition A**: 48 hours of reaction, 306 mg, 83% yield; **Condition B**: 48 hours of reaction, 147 mg, 40% yield, m.p. = 156-157 °C (literature = 157 ºC).[3] ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 8.10 (d, *J* = 7.3 Hz, 2H), 7.68-7.64 (m, 1H), 7.57-7.52 (m, 4H), 7.44-7.38 (m, 3H), 5.83 (d, *J* = 11.3 Hz, 1H), 5.65 (d, *J* = 11.3 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 191.4, 138.5, 134.7, 134.4, 129.5, 129.2, 129.13, 129.09, 128.6, 50.0, 47.1.



1o **2o**

The general procedure was followed using **1o** (313 mg, 1.0 mmol) as substrate, LiBr (350 mg, 4.0 mmol) and H_2O_2 8.8 M (227 μL, 2.0 mmol). At the end of the reaction the product was extracted with AcOEt (3 x 10 mL) and purified by silica gel cromatography (hexanes/AcOEt = 90/10) to afford a white solid. **Condition A**: 48 hours of reaction, 325 mg, 69% yield; **Condition B**: 48 hours of reaction, 322 mg, 68% yield, m.p. = 123-124 °C. Recrystallization was performed with DCM and hexanes. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.40 (br, 3H), 7.27-7.20 (m, 12H), 5.61 (d, *J* = 11.3 Hz, 1H), 5.06 (d, *J* = 14.2 Hz, 1H), 4.91 (d, *J* = 14.2 Hz, 1H), 4.71 (d, *J* = 11.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 167.3, 140.9, 138.4, 136.8, 130.1, 129.3, 129.2, 128.9, 128.7, 128.4, 127.9, 54.1, 52.4, 45.8. HRMS (ESI, positive mode) *m/z* calculated for $C_{22}H_{19}{}^{79}Br_2NNaO$ $[M + Na]^+$ 493.9726, found: 493.9710.

Crystallographic Data and Structure Refinement for Compound 2o:



$C_{22}H_{19}Br_2NO$

M_r = 473.2

Monoclinic, $P2_1/n$

$F(000)$ = 944

D_x = 1.545 Mg m^{-3}

Mo Ka radiation, l = 0.71073 Å

a = 9.8099 (5) Å	Cell parameters from 20953 reflections
b = 14.0372 (7) Å	q = 2.5–26.1°
c = 14.7788 (11) Å	m = 3.99 mm^{-1}
b = 90.878 (6)°	T = 298 K
V = 2034.9 (2) $Å^3$	Prism, colorless
Z = 4	0.53 × 0.30 × 0.30 mm

Data collection

Xcalibur, Atlas, Gemini ultra diffractometer	5499 independent reflections
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source	3667 reflections with $I > 2s(I)$
Graphite monochromator	R_{int} = 0.042
Detector resolution: 10.4186 pixels mm^{-1}	q_{max} = 29.6°, q_{min} = 2.5°
w scans	h = -13→13
Absorption correction: multi-scan *CrysAlis PRO* 1.171.38.43f (Rigaku Oxford Diffraction, 2015) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	k = -19→19
T_{min} = 0.597, T_{max} = 1.000	l = -20→20
70535 measured reflections	

Refinement

Refinement on F^2	0 restraints
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2s(F^2)]$ = 0.043	H-atom parameters constrained
$wR(F^2)$ = 0.106	$w = 1/[s^2(F_o^2) + (0.0351P)^2 + 2.1756P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(D/s)_{max}$ = 0.001
5499 reflections	$D\rho_{max}$ = 0.83 e $Å^{-3}$
235 parameters	$D\rho_{min}$ = -0.90 e $Å^{-3}$

References

[1] A. K. Chakraborti, S. V. Chankeshwara *J. Org. Chem.,* **2009**, *74*, 1367–1370.

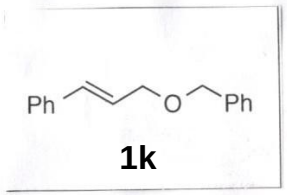
[2] S. S. Weng, C. S. Ke, F. K. Chen, Y. F. Lyu, G. Y. Lin, *Tetrahedron,* **2011**, *67*, 1640–1648.

[3] M. Stodulski, A. Goetzinger, S. V. Kohlhepp, T. Gulder, *Chem. Comm*., **2014**, *50*, 3435-3438.

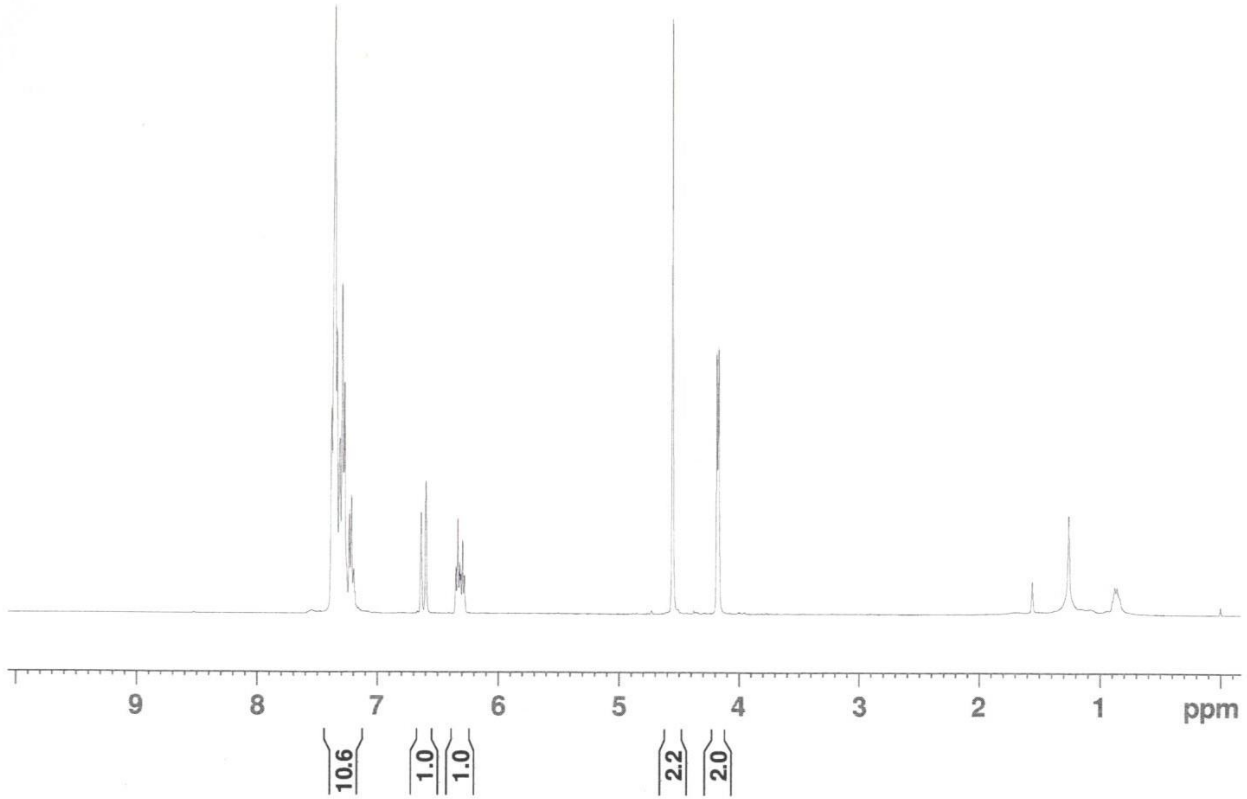
[4] M. Karki, J. Magolan, *J. Org. Chem.* **2015**, *80*, 3701−3707.

[5] S. Song, X. Li, X. Sun, Y. Yuana, N. Jiao, *Green Chem.,* **2015**, *17*, 3285–3289.

[6] R. E. Buckles, R. C. Johnson, W. J. Probst, *J. Org. Chem.,* **1957**, *22*, 55–59.



1k

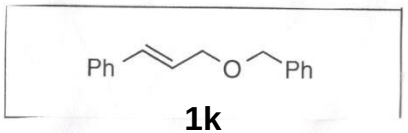



```
Current Data Parameters
NAME          I-NSM-147
EXPNO                 3
PROCNO                1

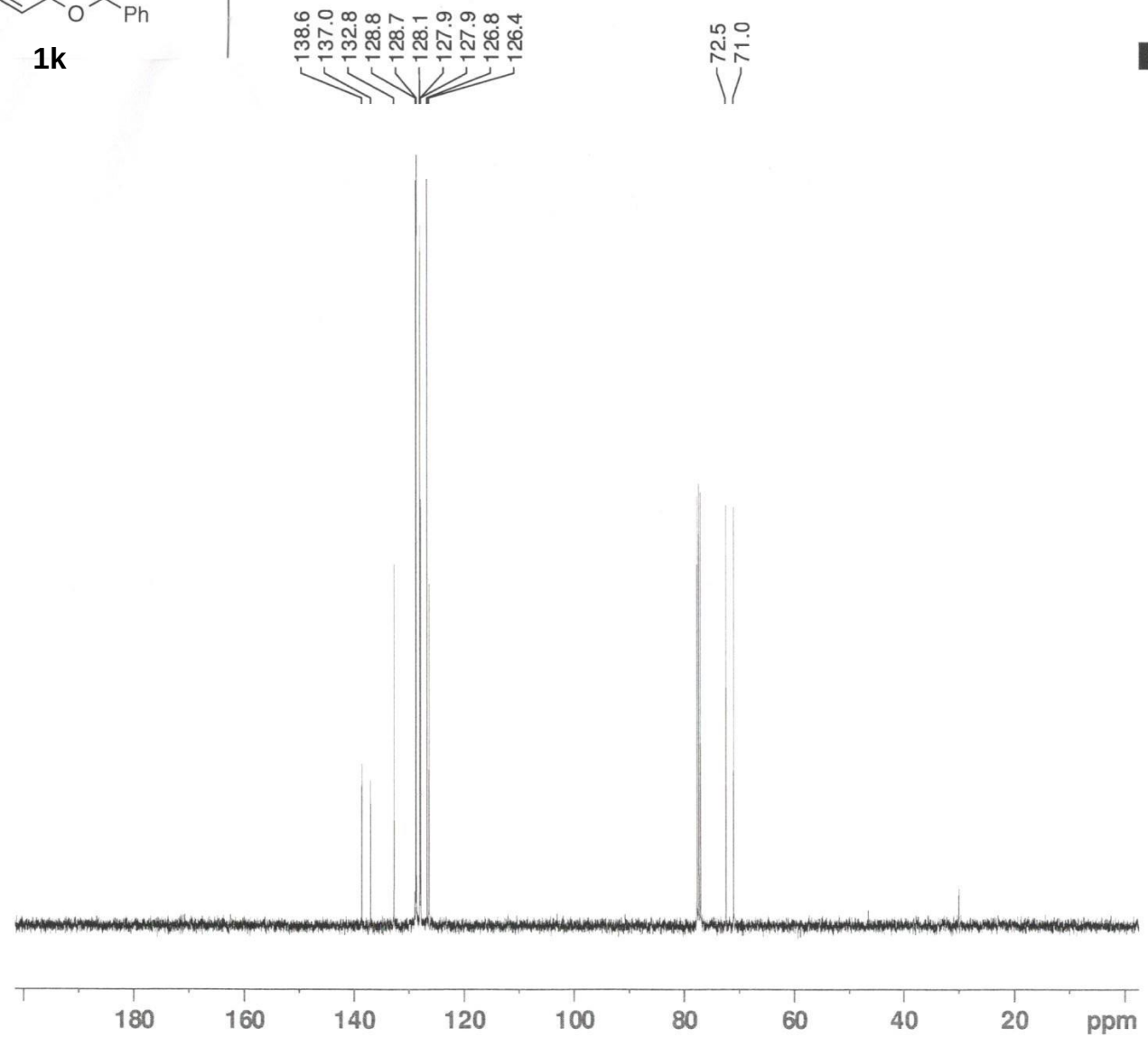
F2 - Acquisition Parameter
Date_          20170627
Time               8.17
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                35.05
DW               62.400 usec
DE               10.00 usec
TE                300.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1124007 MHz
NUC1                 1H
P1                11.75 usec
PLW1         11.19999981 W

F2 - Processing parameters
SI                65536
SF           400.1100391 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



1k



BRUKER

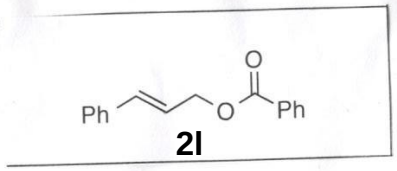
```
Current Data Parameters
NAME          I-NSM-147
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameter
Date_          20170627
Time               8.18
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                32768
SOLVENT           CDCl3
NS                   56
DS                    4
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ            0.6488064 sec
RG                197.86
DW              19.800 usec
DE              10.00 usec
TE                300.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

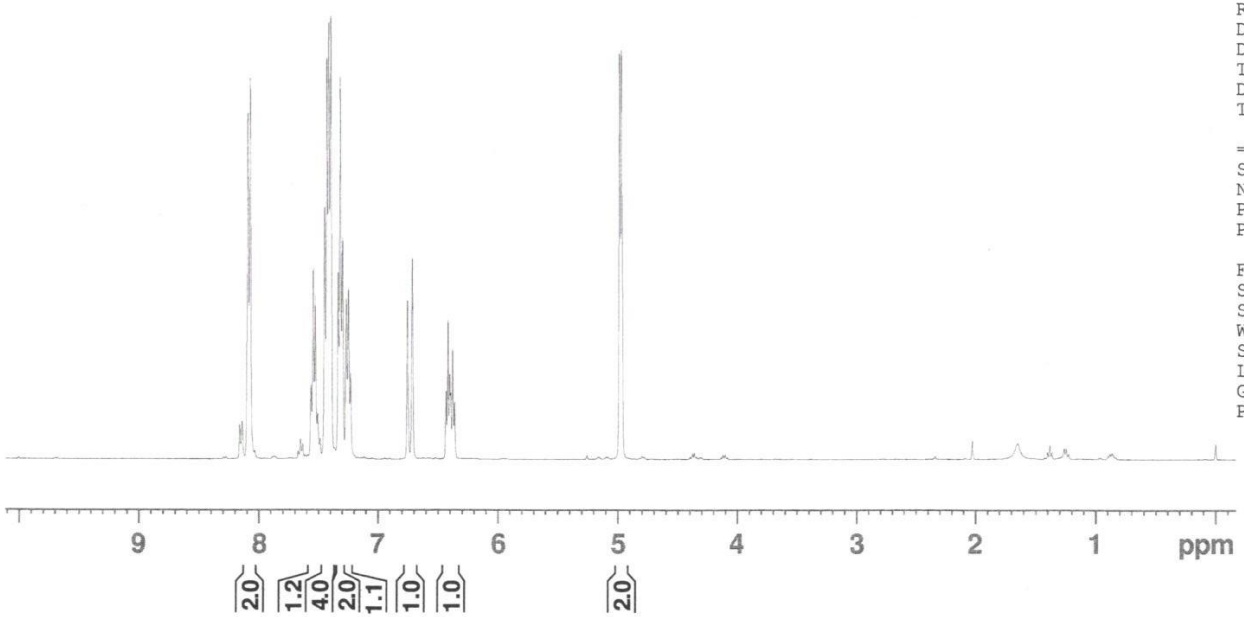
======== CHANNEL f1 ========
SFO1        100.6176997 MHz
NUC1                13C
P1                13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI                32768
SF          100.6077179 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



2l

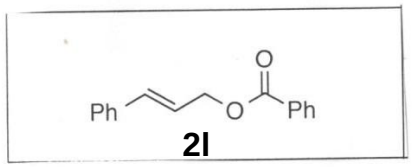



```
Current Data Parameters
NAME          I-NSM-133
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameter
Date_          20170522
Time               8.28
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    0
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                71.32
DW               62.400 usec
DE               10.00 usec
TE                300.9 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1124007 MHz
NUC1                   1H
P1                 11.75 usec
PLW1        11.19999981 W

F2 - Processing parameters
SI                 65536
SF          400.1100218 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2l

BRUKER

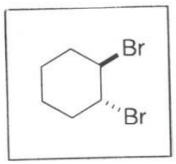
```
Current Data Parameters
NAME          I-NSM-133
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameter
Date_          20170522
Time               8.31
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                 104
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           0.6815744 sec
RG              197.86
DW              20.800 usec
DE              10.00 usec
TE              301.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

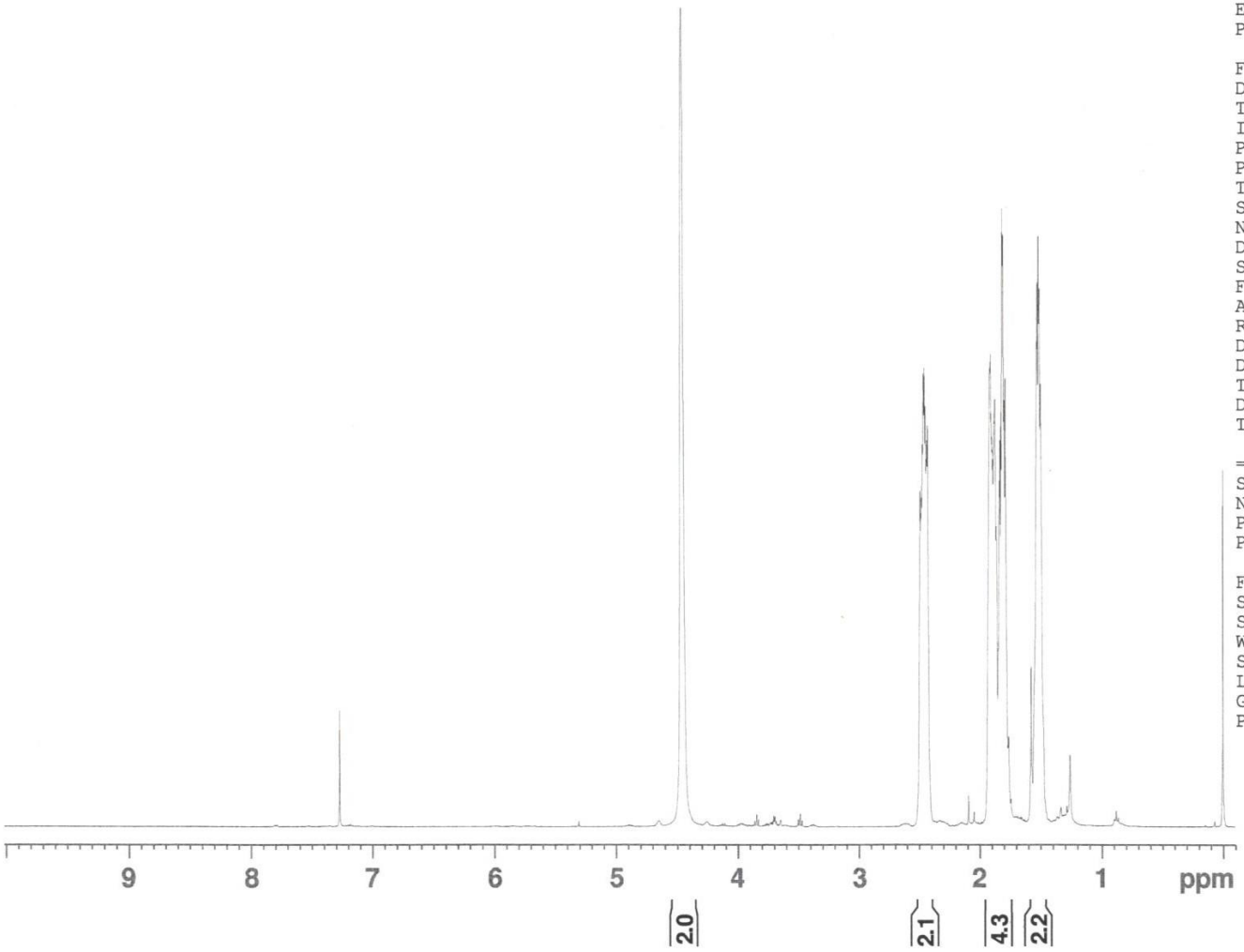
======== CHANNEL f1 ========
SFO1        100.6177998 MHz
NUC1               13C
P1               13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI                32768
SF          100.6077179 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```



2a



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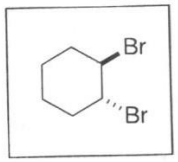
```
Current Data Parameters
NAME          I-NSM-96-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameter
Date_           20170222
Time               13.25
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    20
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 55.25
DW                62.400 usec
DE                10.00 usec
TE        0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1124708 MHz
NUC1                   1H
P1                 11.75 usec
PLW1         11.19999981 W

F2 - Processing parameters
SI                 65536
SF           400.1100059 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2a

BRUKER

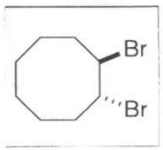
```
Current Data Parameters
NAME            I-NSM-96
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameter
Date_           20170220
Time               13.25
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                32768
SOLVENT            CDCl3
NS                   106
DS                     4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                197.86
DW               20.800 usec
DE               10.00 usec
TE        0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6177998 MHz
NUC1                 13C
P1                13.86 usec
PLW1         17.98900032 W

======== CHANNEL f2 ========
SFO2         400.1116004 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 use
PLW2         11.19999981 W
PLW12         0.19090000 W
PLW13         0.15463001 W

F2 - Processing parameters
SI                  32768
SF           100.6077492 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



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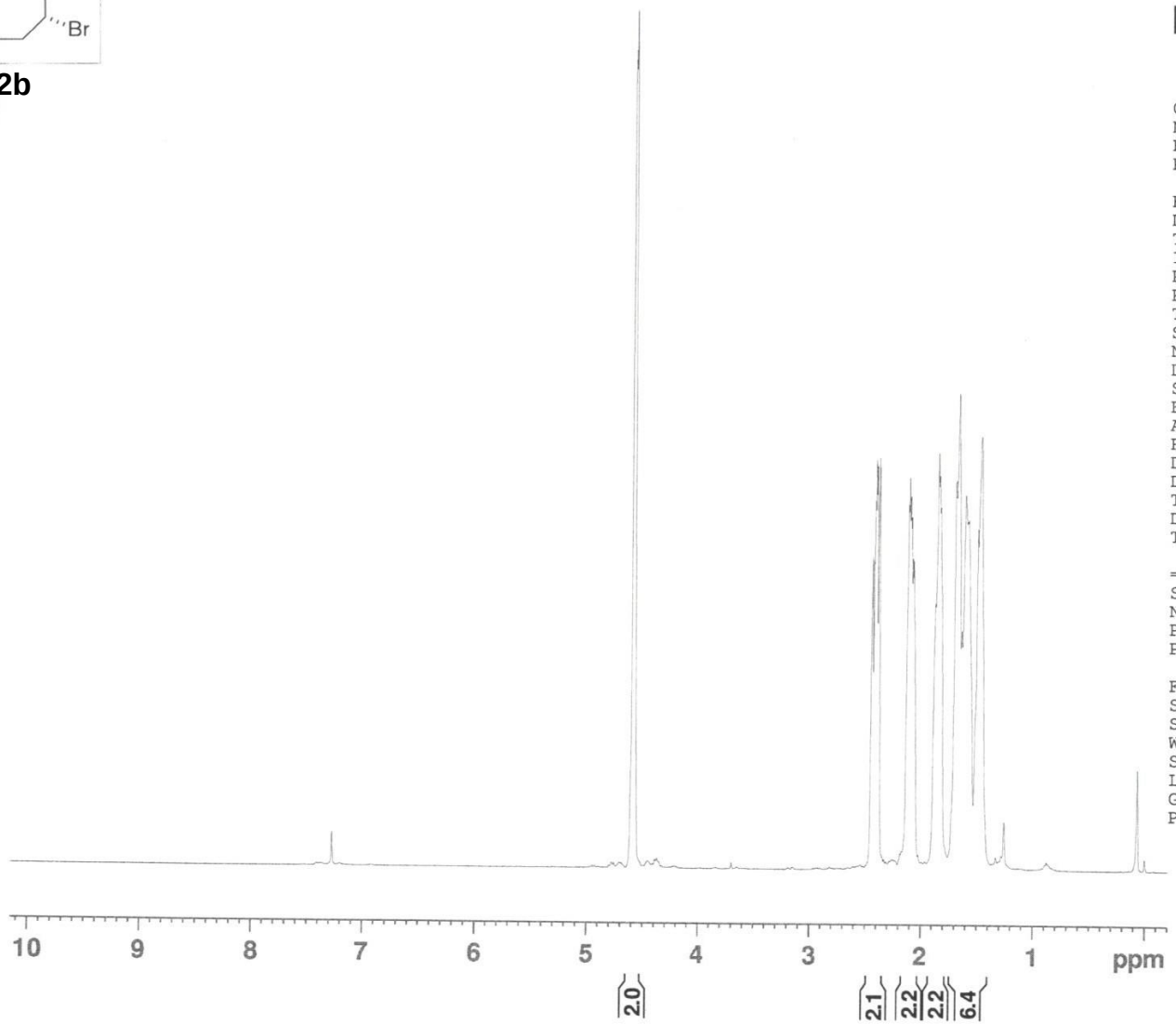

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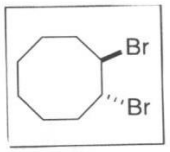
```
Current Data Parameters
NAME          I-NSM-151
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameter
Date_          20170626
Time               8.44
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                   20
DS                    0
SWH            8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                35.05
DW               62.400 usec
DE               10.00 usec
TE                300.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1124007 MHz
NUC1                  1H
P1               11.75 usec
PLW1        11.19999981 W

F2 - Processing parameters
SI                65536
SF          400.1100039 MHz
WDW                  EM
SSB       0
LB             0.30 Hz
GB        0
PC                1.00
```



2b

BRUKER

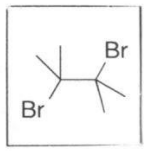
```
Current Data Parameters
NAME          I-NSM-151
EXPNO                 5
PROCNO                1

F2 - Acquisition Parameter
Date_          20170626
Time               8.45
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                  82
DS                   4
SWH          25252.525 Hz
FIDRES         0.770646 Hz
AQ           0.6488064 sec
RG              197.86
DW             19.800 usec
DE              10.00 usec
TE              300.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

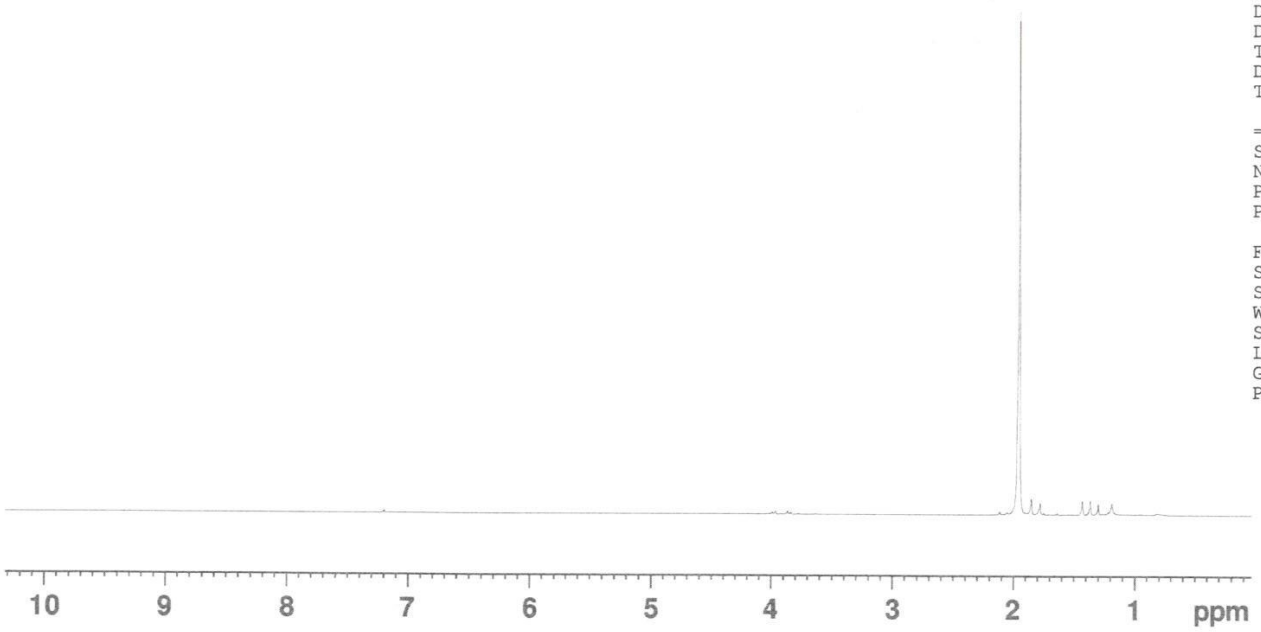
======== CHANNEL f1 ========
SFO1        100.6176997 MHz
NUC1               13C
P1              13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI              32768
SF          100.6077179 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```



2c




```
Current Data Parameters
NAME          I-NSM-121
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameter
Date_          20170510
Time               8.26
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    0
SWH            8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                63.68
DW               62.400 usec
DE               10.00 usec
TE               299.7 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1124007 MHz
NUC1                 1H
P1                11.75 usec
PLW1         11.19999981 W

F2 - Processing parameters
SI                65536
SF           400.1100354 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
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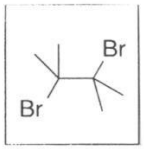
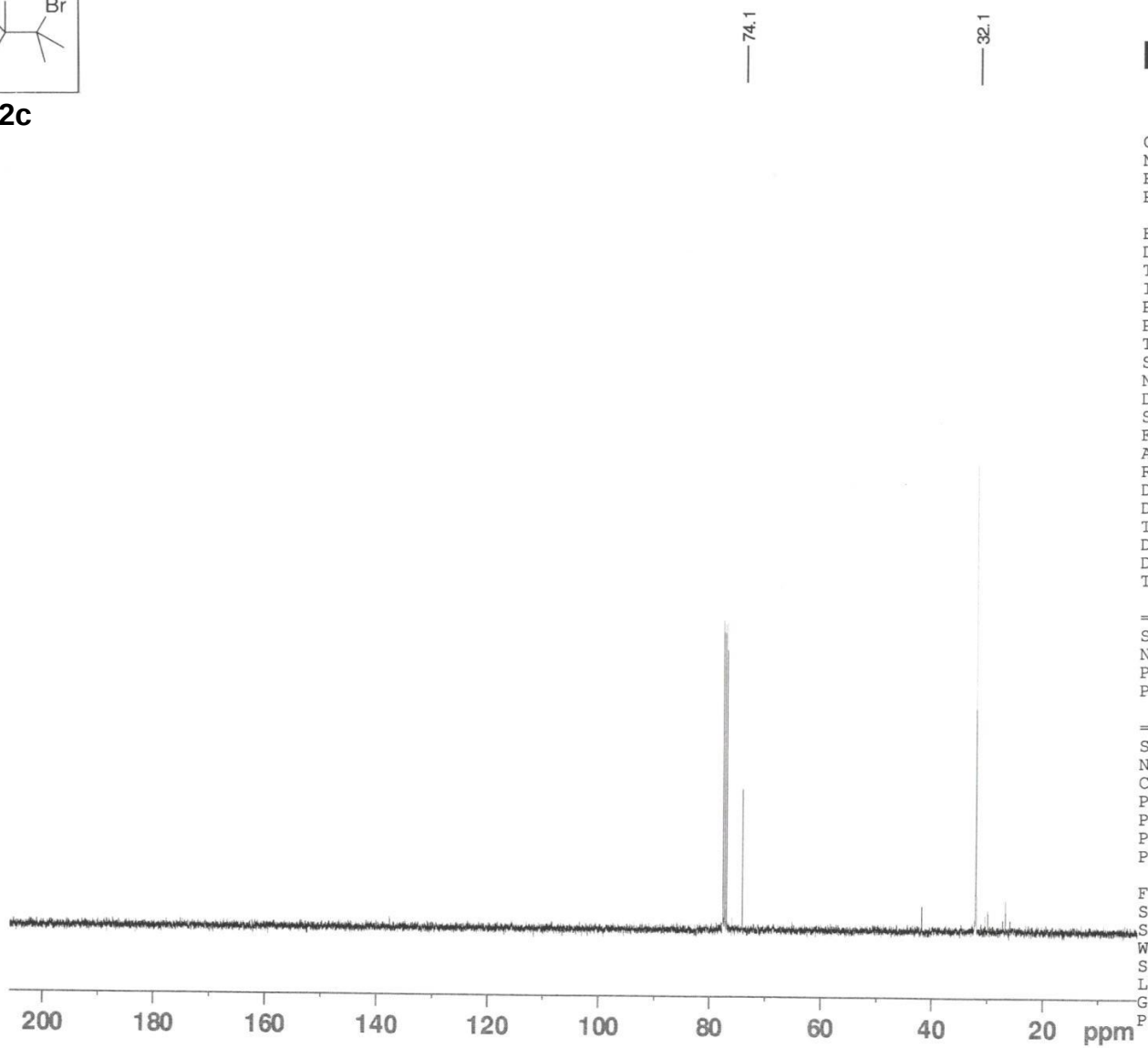


2c



BRUKER

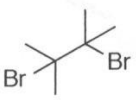
```
Current Data Parameters
NAME          I-NSM-121
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameter
Date_          20170510
Time               8.20
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              32768
SOLVENT          CDC13
NS                 100
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           0.6815744 sec
RG              197.86
DW            20.800 usec
DE            10.00 usec
TE             300.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6177998 MHz
NUC1              13C
P1              13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2              1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

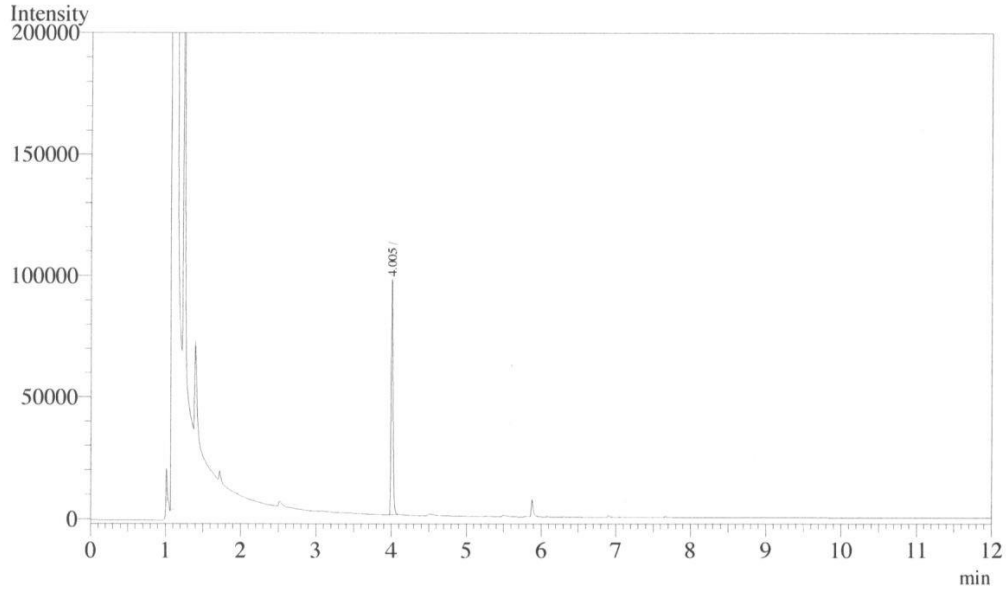
F2 - Processing parameters
SI              32768
SF         100.6077179 MHz
WDW                EM
SSB      0
LB              1.00 Hz
GB       0
PC              1.40
```



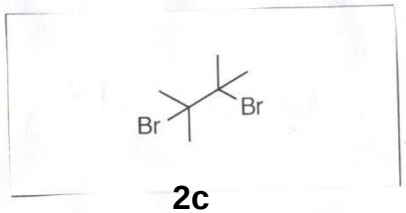
2c

Analysis Date & Time	: 22/08/2017 10:24:16
User Name	: Admin
Vial#	: 3
Sample Name	: I-NSM125'
Sample ID	:
Sample Type	: Unknown
Injection Volume	: 1.00
ISTD Amount	:

Data Name : C:\GCsolution\Data\GC3\Eduardo\Nayara\Massas\I-NSM125'.gcd
Method Name : C:\GCsolution\Data\GC3\Eduardo\Metodos\Ciclooctено.gcm

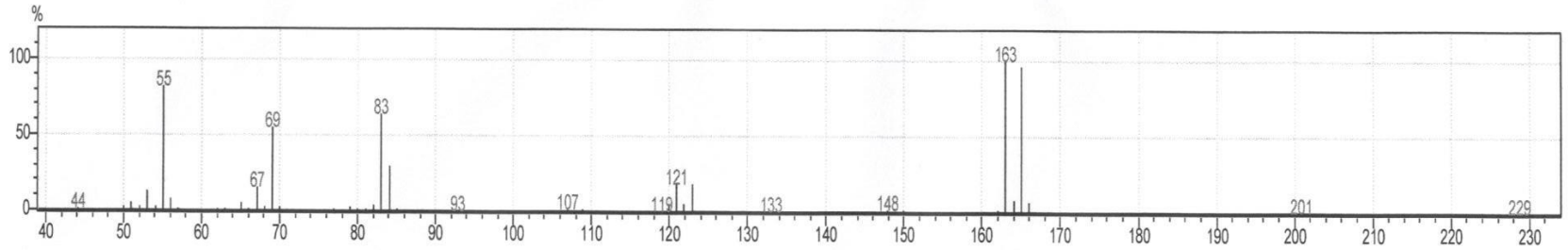


Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	4.005	158452	96976	100.000				
Total		158452	96976					



2c

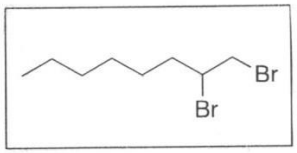
Event#1:Scan Ret.Time : [3.317] Scan# : [399]



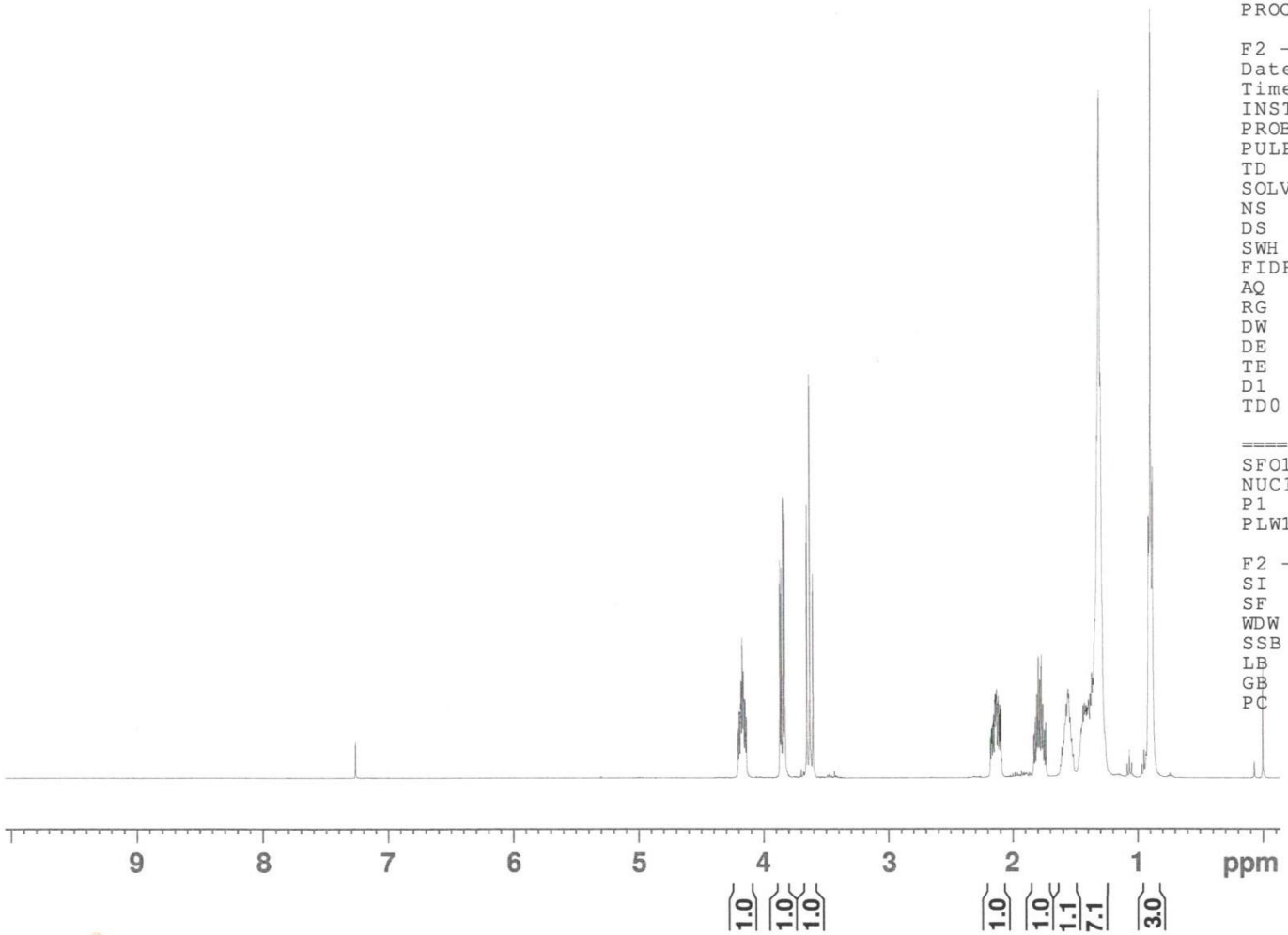
[MS Spectrum] # of Peaks 57

Raw Spectrum 3.317 (scan : 399) Base Peak m/z 163.00 (Inten : 834,743)

m/z	Relative Intensity (%)
53.05	13.36
55.05	82.54
67.05	15.19
69.05	55.54
83.05	64.17
84.05	29.65
163.00	100
165.00	96.55



2d

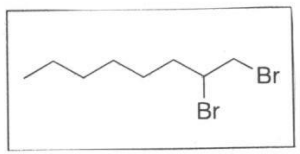



```
Current Data Parameters
NAME            I-NSM-97
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameter
Date_           20170220
Time               13.36
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    20
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 35.05
DW                62.400 usec
DE                10.00 usec
TE        0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1124708 MHz
NUC1                   1H
P1                 11.75 usec
PLW1         11.19999981 W

F2 - Processing parameters
SI                 65536
SF           400.1100077 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2d

BRUKER

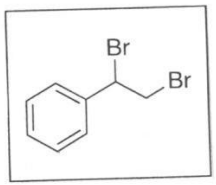
```
Current Data Parameters
NAME          I-NSM-97
EXPNO                2
PROCNO               1

F2 - Acquisition Parameter
Date_         20170220
Time             13.41
INSTRUM          spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                 120
DS                   4
SWH          24038.461 Hz
FIDRES         0.733596 Hz
AQ           0.6815744 sec
RG               197.86
DW              20.800 usec
DE              10.00 usec
TE       0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

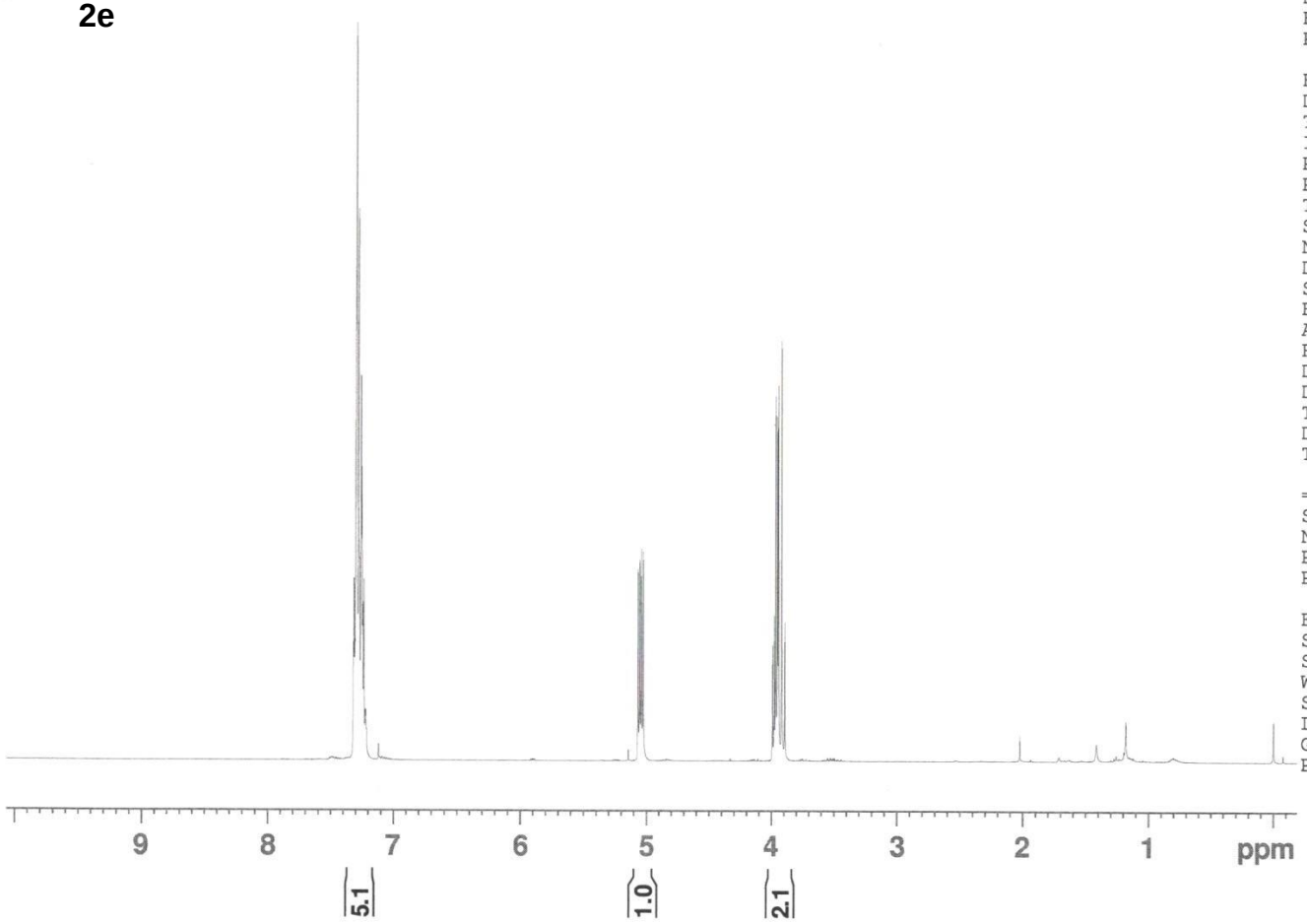
======== CHANNEL f1 ========
SFO1        100.6177998 MHz
NUC1               13C
P1              13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI              32768
SF          100.6077448 MHz
WDW                EM
SSB      0
LB              1.00 Hz
GB       0
PC              1.40
```



2e

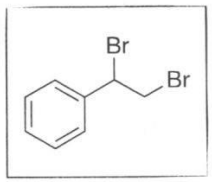



```
Current Data Parameters
NAME          I-NSM-95-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameter
Date_          20170222
Time             13.17
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    0
SWH           8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                35.05
DW               62.400 usec
DE               10.00 usec
TE        0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1124708 MHz
NUC1               1H
P1               11.75 usec
PLW1        11.19999981 W

F2 - Processing parameters
SI                65536
SF          400.1100669 MHz
WDW                EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2e

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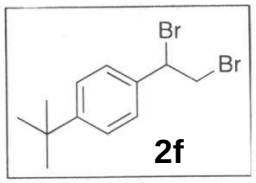
```
Current Data Parameters
NAME                 I-NSM-95
EXPNO                       2
PROCNO                      1

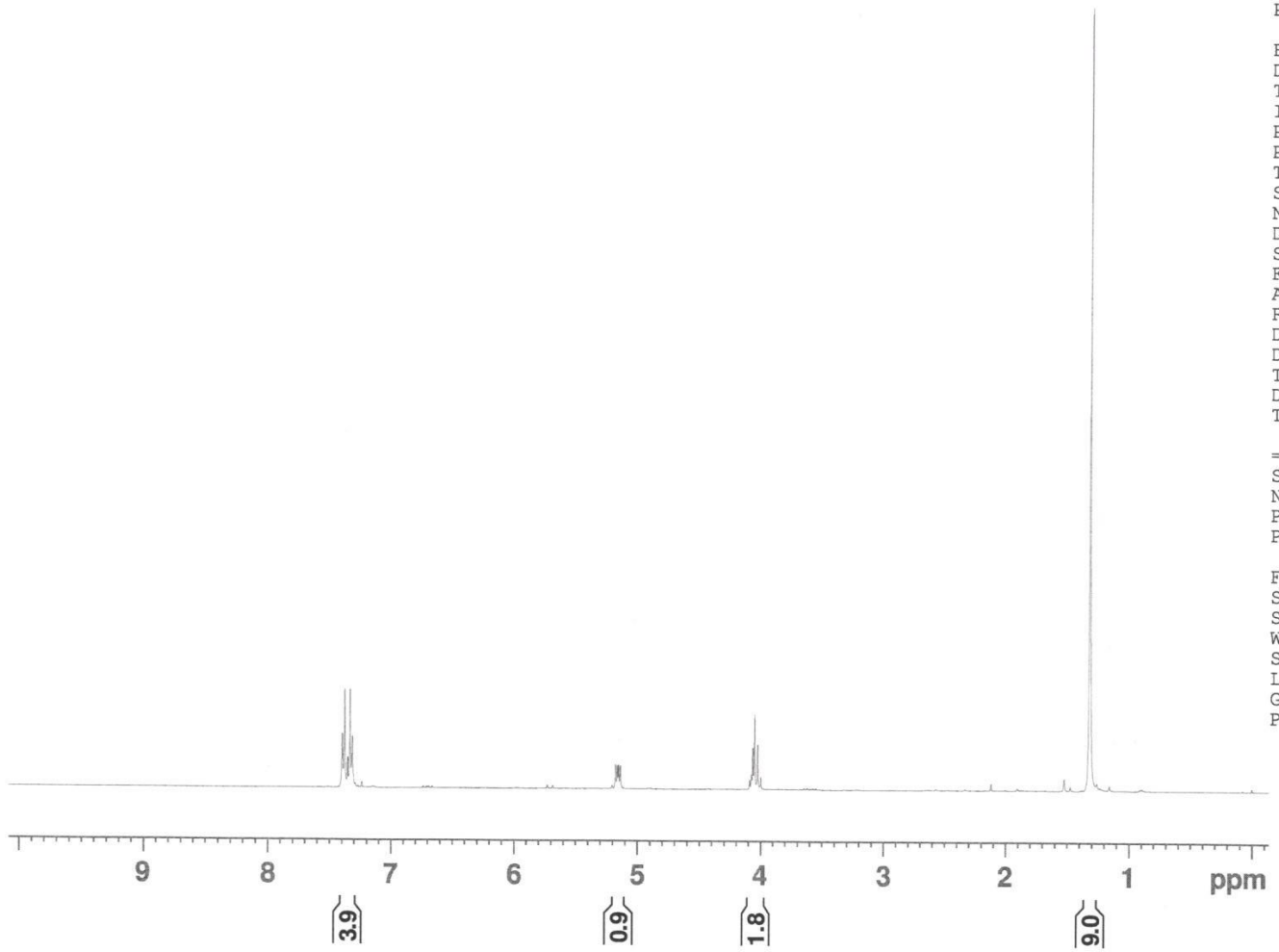
F2 - Acquisition Parameter
Date_               20170220
Time                   13.15
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                zgpg30
TD                     32768
SOLVENT                CDCl3
NS                       109
DS                         4
SWH                24038.461 Hz
FIDRES              0.733596 Hz
AQ                 0.6815744 sec
RG                     197.86
DW                   20.800 usec
DE                   10.00 usec
TE                 0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               100.6177998 MHz
NUC1                       13C
P1                   13.86 usec
PLW1               17.98900032 W

======== CHANNEL f2 ========
SFO2               400.1116004 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                90.00 usec
PLW2               11.19999981 W
PLW12               0.19090000 W
PLW13               0.15463001 W

F2 - Processing parameters
SI                     32768
SF                 100.6077526 MHz
WDW                        EM
SSB                0
LB                   1.00 Hz
GB                 0
PC                   1.40
```



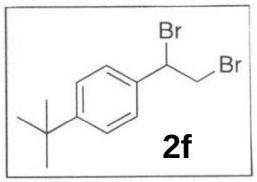



```
Current Data Parameters
NAME                  I-NSM-99
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameter
Date_                 20170309
Time                     15.48
INSTRUM                  spect
PROBHD   5 mm DUL 13C-1
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         20
DS                          0
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                   4.0894465 sec
RG                       55.25
DW                     62.400 usec
DE                     10.00 usec
TE              0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               400.1124708 MHz
NUC1                        1H
P1                     11.75 usec
PLW1               11.19999981 W

F2 - Processing parameters
SI                      65536
SF                 400.1100190 MHz
WDW                        EM
SSB             0
LB                     0.30 Hz
GB              0
PC                     1.00
```



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```
Current Data Parameters
NAME            I-NSM-99
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameter
Date_           20170309
Time               15.53
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  100
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG                197.86
DW              20.800 usec
DE              10.00 usec
TE        0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6177998 MHz
NUC1                13C
P1               13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 use
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI                32768
SF          100.6077179 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

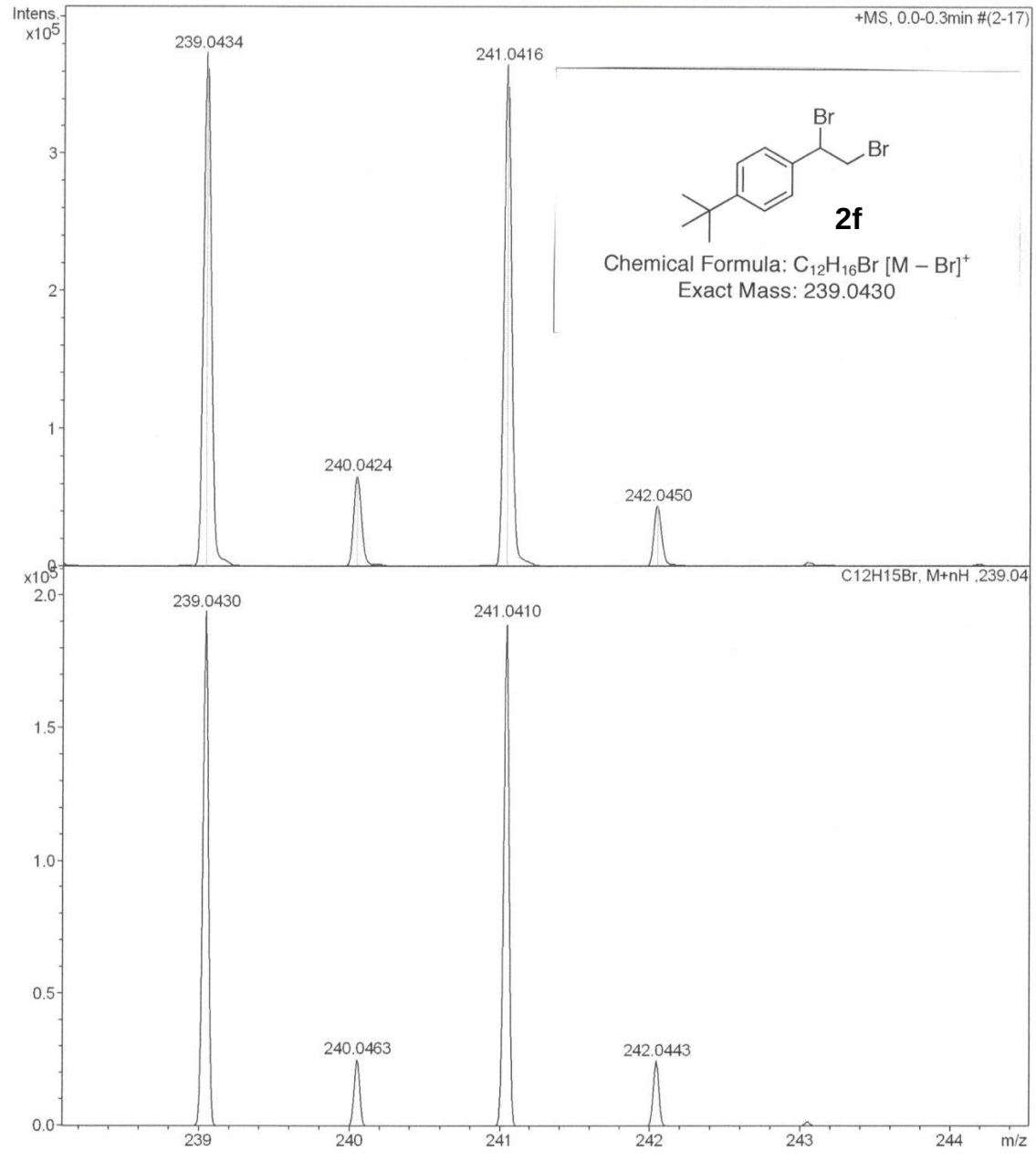
Display Report

Analysis Info

Analysis Name	C:\Data\LabSelen 13.09.17 APCI\I-NSM-99000003.d	Acquisition Date	13/9/2017 10:48:08
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	I-NSM-99	Instrument	micrOTOF-Q 10243
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	250 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	1.5 l/min
Scan End	1000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Source



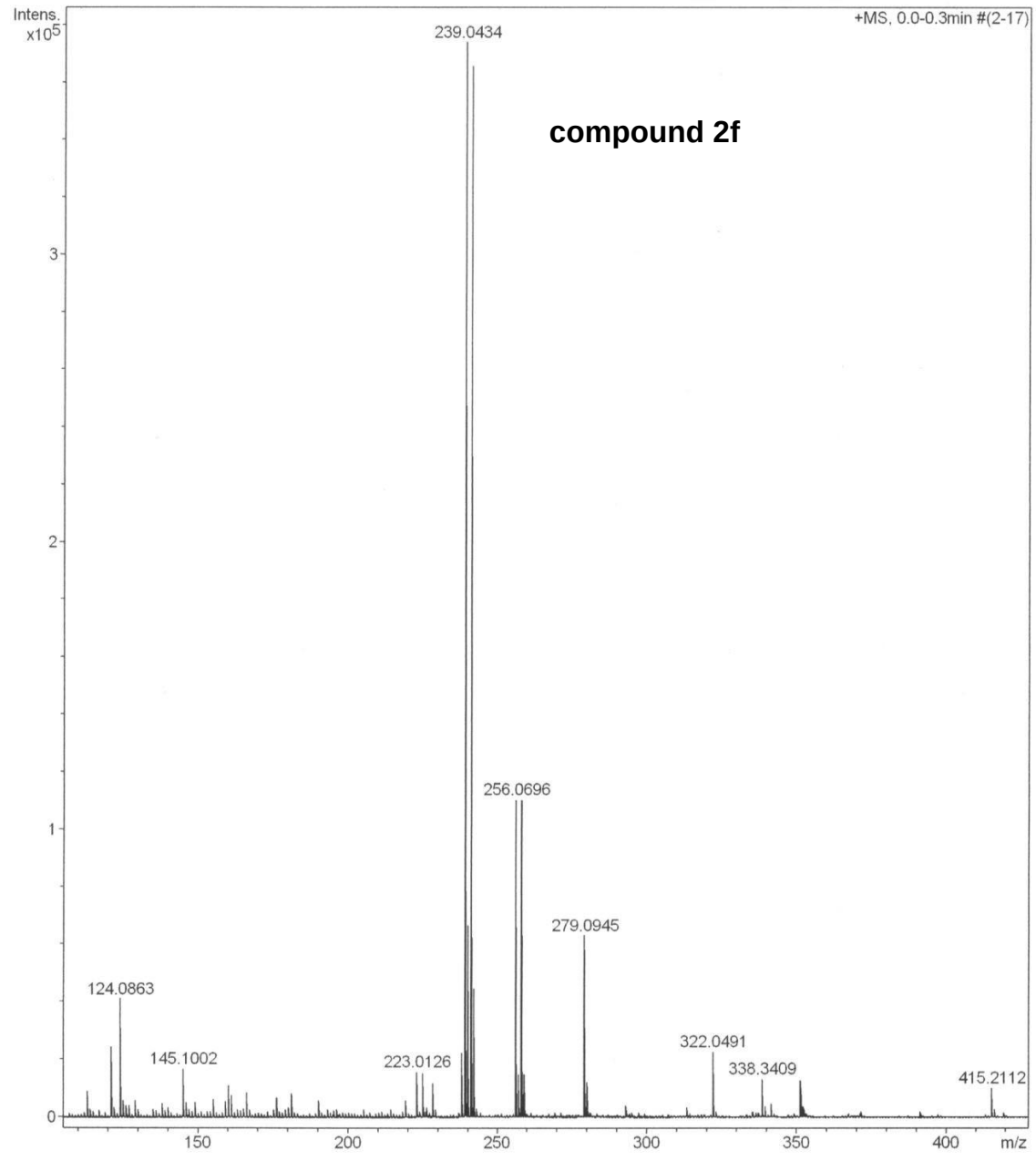
Display Report

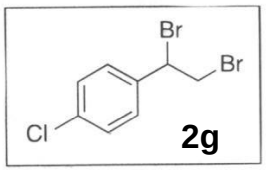
Analysis Info

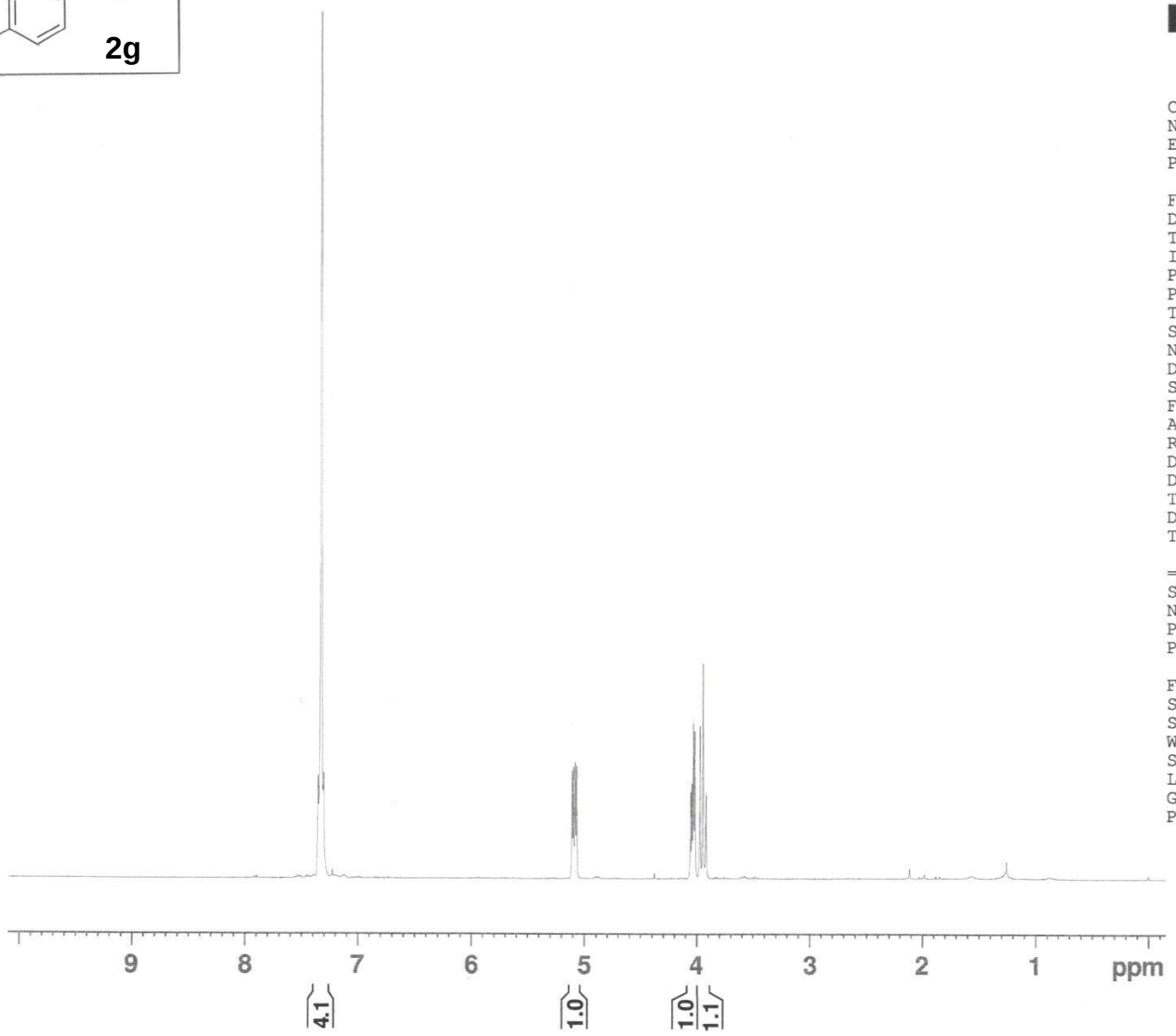
Analysis Name	C:\Data\LabSelen 13.09.17 APCI\I-NSM-99000003.d	Acquisition Date	13/9/2017 10:48:08
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	I-NSM-99	Instrument	micrOTOF-Q 10243
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	250 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	1.5 l/min
Scan End	1000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Source







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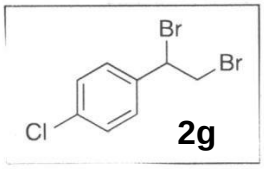
```
Current Data Parameters
NAME          I-NSM-100
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameter
Date_          20170314
Time              12.58
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                65536
SOLVENT           CDC13
NS                   20
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                35.05
DW               62.400 usec
DE               10.00 usec
TE        0 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1124708 MHz
NUC1                 1H
P1                11.75 usec
PLW1         11.19999981 W

F2 - Processing parameters
SI                65536
SF           400.1100222 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2g

BRUKER

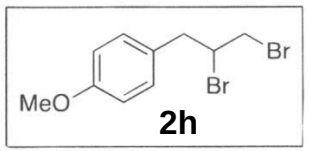
```
Current Data Parameters
NAME          I-NSM-100
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameter
Date_          20170314
Time              12.59
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                   53
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG                197.86
DW               20.800 usec
DE               10.00 usec
TE        0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

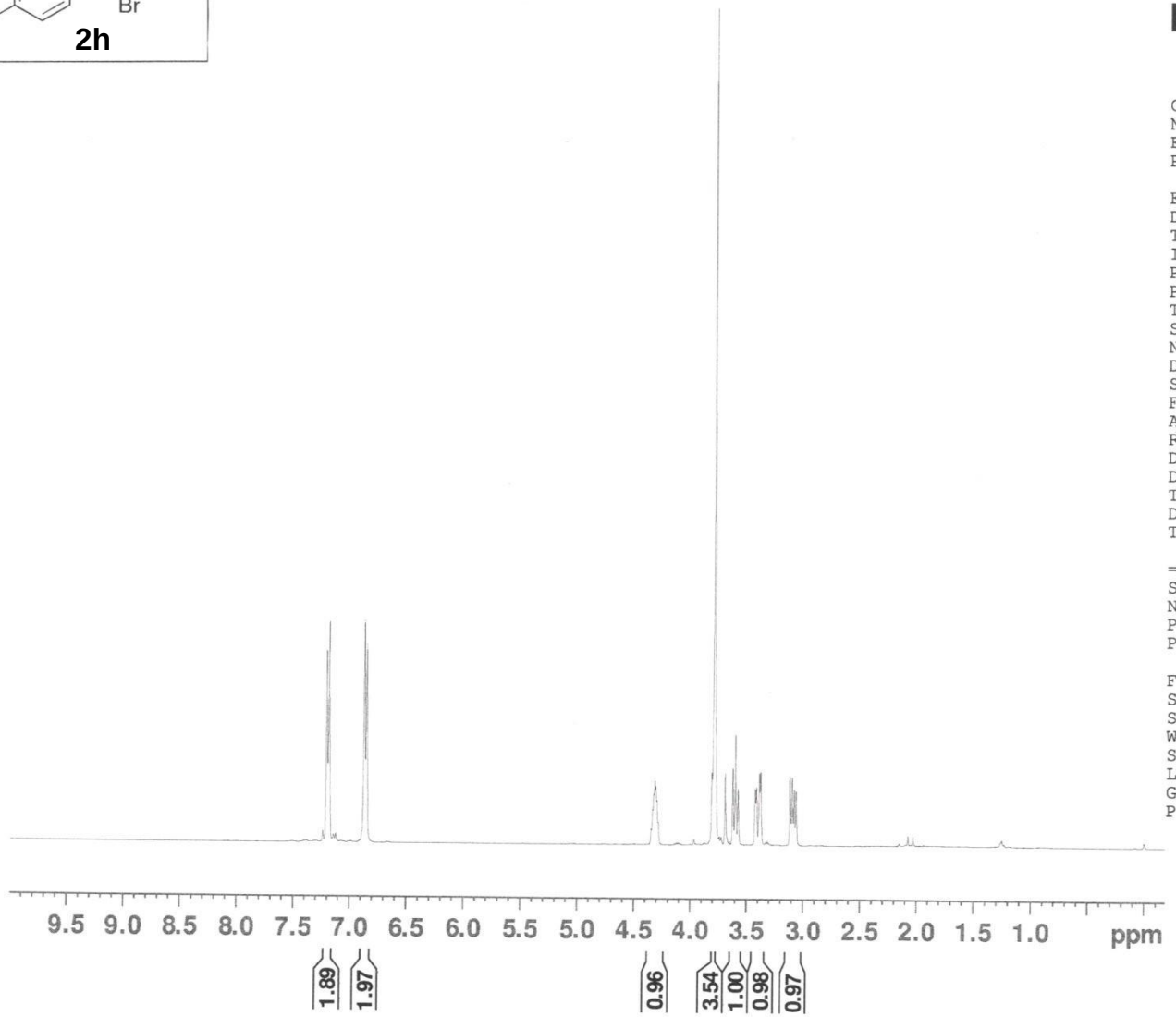
======== CHANNEL f1 ========
SFO1        100.6177998 MHz
NUC1                 13C
P1                13.86 usec
PLW1         17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 use
PLW2         11.19999981 W
PLW12         0.19090000 W
PLW13         0.15463001 W

F2 - Processing parameters
SI                32768
SF           100.6077179 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



2h



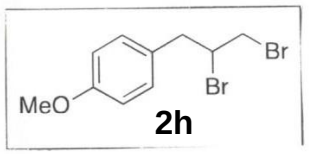
BRUKER

```
Current Data Parameters
NAME          I-NSM-134
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameter
Date_          20170518
Time              12.17
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    0
SWH            8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                63.68
DW              62.400 usec
DE              10.00 usec
TE              300.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1124007 MHz
NUC1                 1H
P1                11.75 usec
PLW1         11.19999981 W

F2 - Processing parameters
SI                65536
SF           400.1100182 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2h

BRUKER

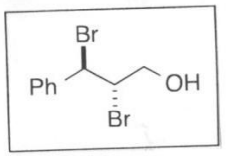
```
Current Data Parameters
NAME          I-NSM-103
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameter
Date_          20170223
Time               8.38
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                  75
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           0.6815744 sec
RG              197.86
DW             20.800 usec
DE              10.00 usec
TE        0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

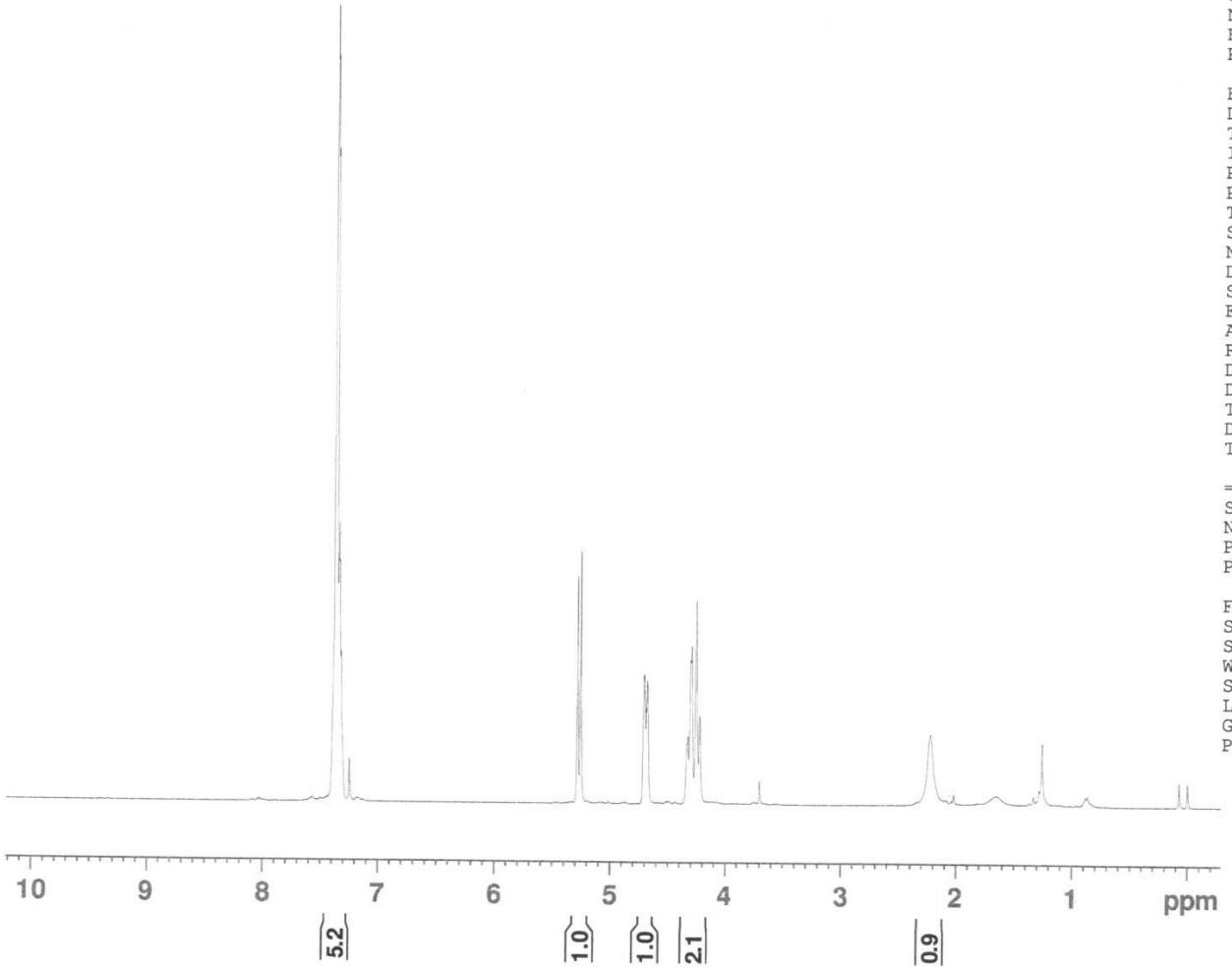
======== CHANNEL f1 ========
SFO1        100.6177998 MHz
NUC1               13C
P1              13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI              32768
SF         100.6077456 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```



2i

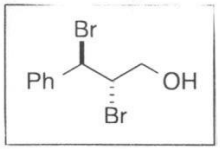



```
Current Data Parameters
NAME          I-NSM-143-2
EXPNO                   1
PROCNO                  1

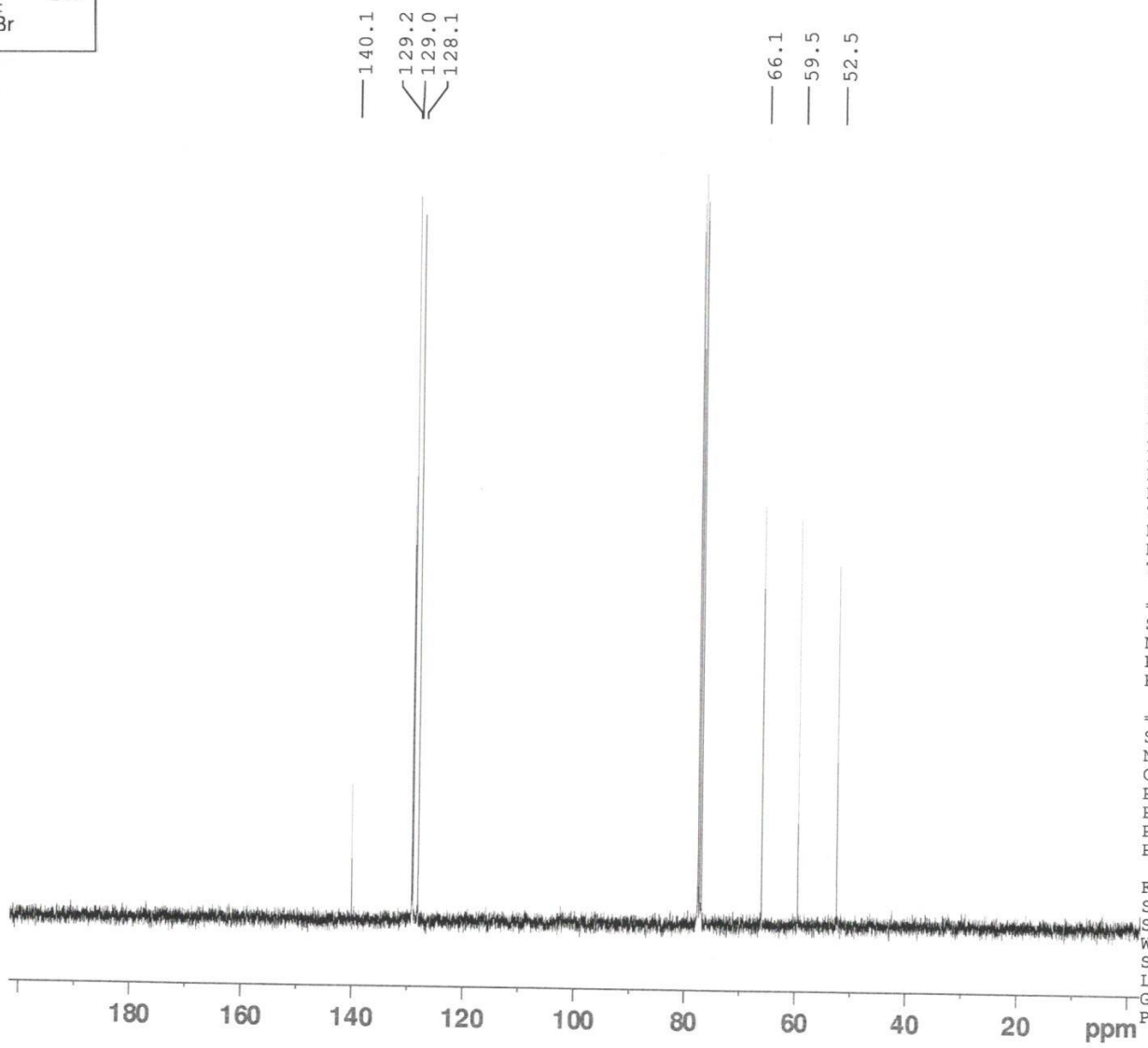
F2 - Acquisition Parameter
Date_           20170718
Time                8.07
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    20
DS                     0
SWH             8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 110.48
DW                 62.400 usec
DE                 10.00 usec
TE                 300.3 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1124007 MHz
NUC1                  1H
P1                 11.75 usec
PLW1          11.19999981 W

F2 - Processing parameters
SI                 65536
SF            400.1100136 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2i



BRUKER

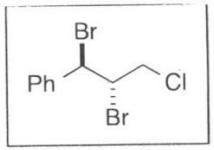
```
Current Data Parameters
NAME          I-NSM-143-2
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameter
Date_          20170718
Time               8.13
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                 141
DS                   4
SWH          25252.525 Hz
FIDRES        0.770646 Hz
AQ           0.6488064 sec
RG              197.86
DW              19.800 usec
DE               10.00 usec
TE               300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

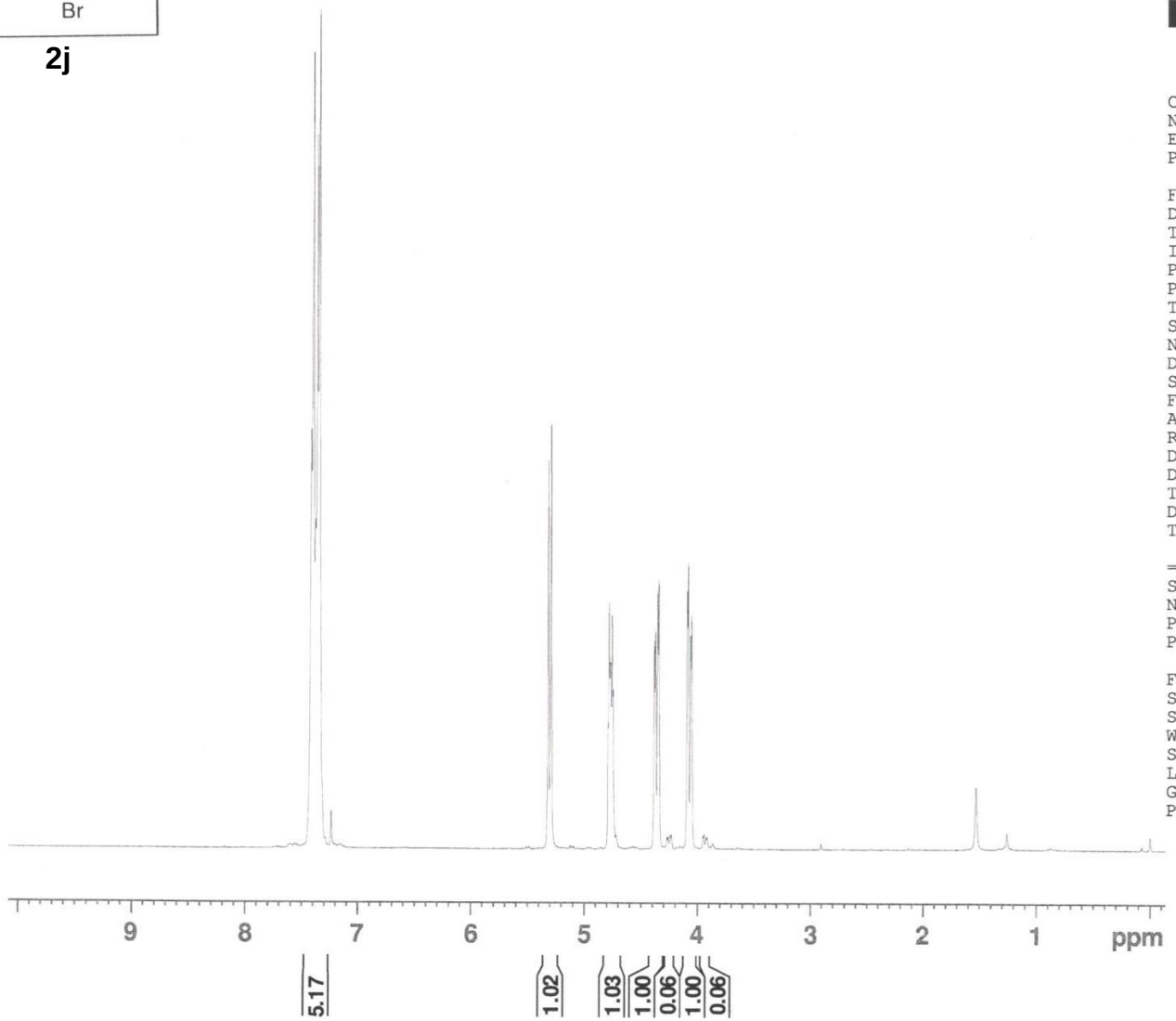
======== CHANNEL f1 ========
SFO1        100.6176997 MHz
NUC1              13C
P1              13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2              1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI               32768
SF          100.6077179 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```



2j



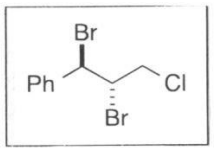
BRUKER

```
Current Data Parameters
NAME          I-NSM-114
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameter
Date_          20170404
Time              14.24
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                65536
SOLVENT           CDC13
NS                   20
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                78.22
DW               62.400 usec
DE               10.00 usec
TE        0 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1124708 MHz
NUC1                  1H
P1                11.75 usec
PLW1        11.19999981 W

F2 - Processing parameters
SI                65536
SF          400.1100192 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2j

BRUKER

```
Current Data Parameters
NAME          I-NSM-114
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameter
Date_          20170404
Time              14.14
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                  85
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           0.6815744 sec
RG              197.86
DW              20.800 usec
DE              10.00 usec
TE        0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6177998 MHz
NUC1              13C
P1              13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2              1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI                32768
SF          100.6077179 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

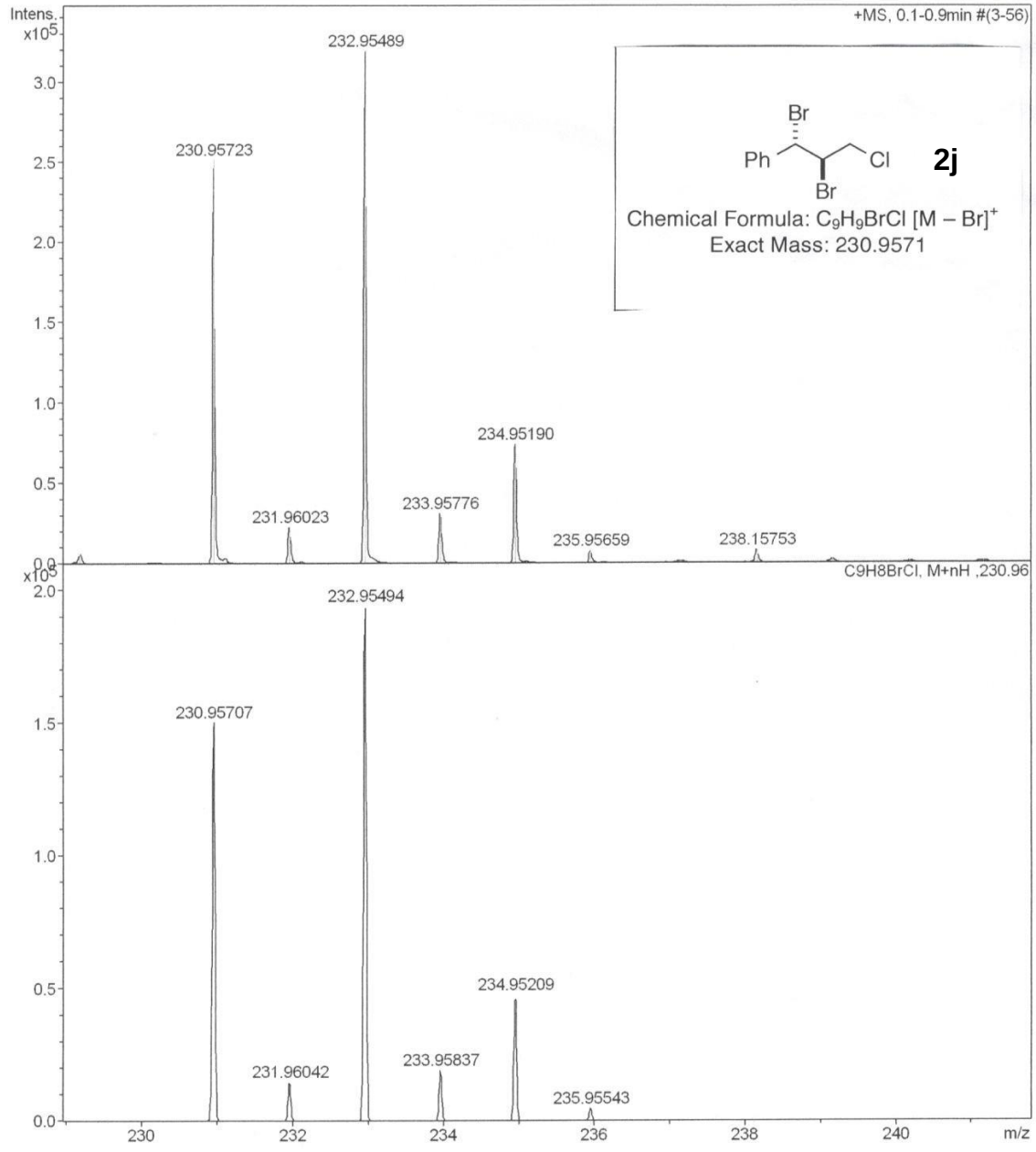
Display Report

Analysis Info

Analysis Name	C:\Data\LabSelen 13.09.17 APCI\I-NSM-114000002.d	Acquisition Date	13/9/2017 11:41:11
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	I-NSM-114	Instrument	micrOTOF-Q 10243
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Active	Set Capillary	4000 V	Set Dry Heater	250 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	1.5 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Source



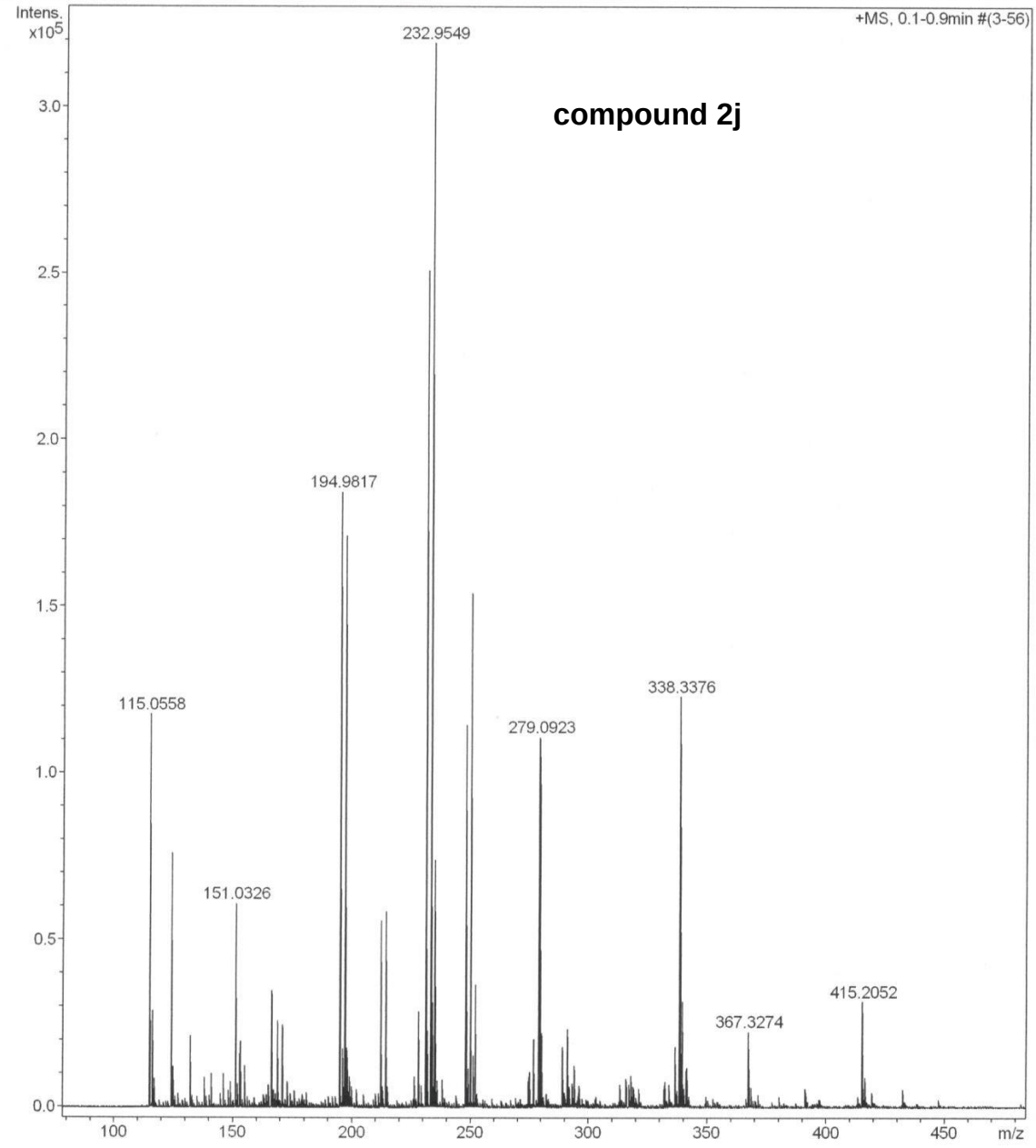
Display Report

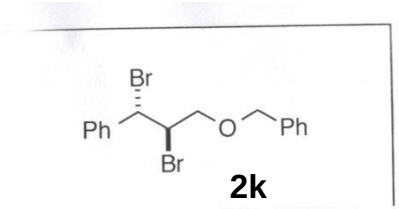
Analysis Info

Analysis Name	C:\Data\LabSelen 13.09.17 APCI\I-NSM-114000002.d	Acquisition Date	13/9/2017 11:41:11
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	I-NSM-114	Instrument	micrOTOF-Q 10243
Comment			

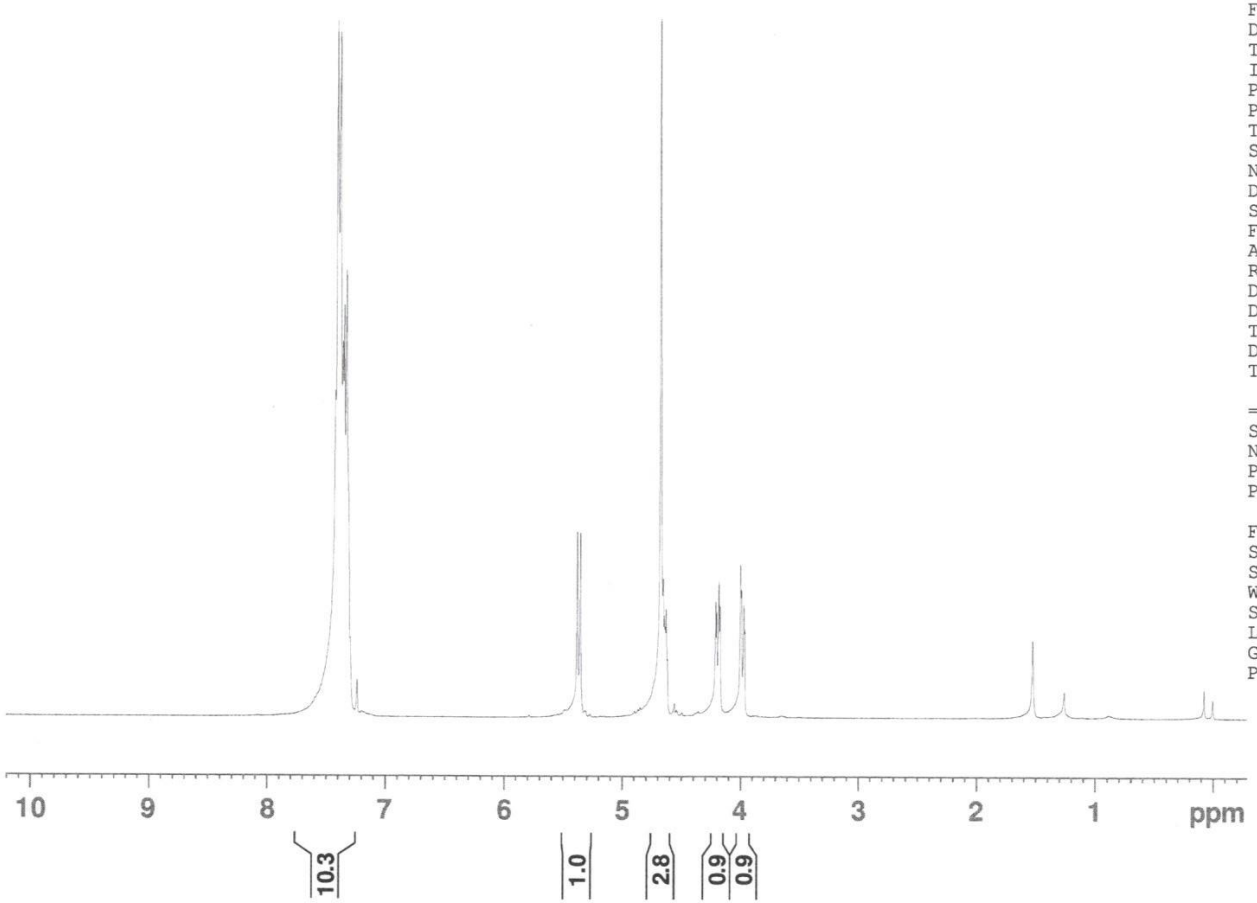
Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Active	Set Capillary	4000 V	Set Dry Heater	250 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	1.5 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Source





2k



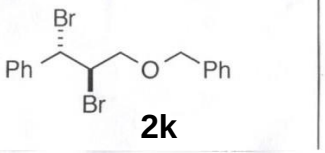
BRUKER

```
Current Data Parameters
NAME          I-NSM-149
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameter
Date_          20170915
Time              13.05
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    0
SWH            8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                97.17
DW               62.400 usec
DE               10.00 usec
TE                300.4 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1124007 MHz
NUC1                 1H
P1                11.75 usec
PLW1         11.19999981 W

F2 - Processing parameters
SI                65536
SF           400.1100200 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2k

BRUKER

```
Current Data Parameters
NAME          I-NSM-149
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameter
Date_          20170914
Time               9.33
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                32768
SOLVENT           CDC13
NS                  500
DS                    4
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ            0.6488064 sec
RG                197.86
DW              19.800 usec
DE              10.00 usec
TE                300.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6176997 MHz
NUC1                13C
P1               13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI                32768
SF          100.6077179 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

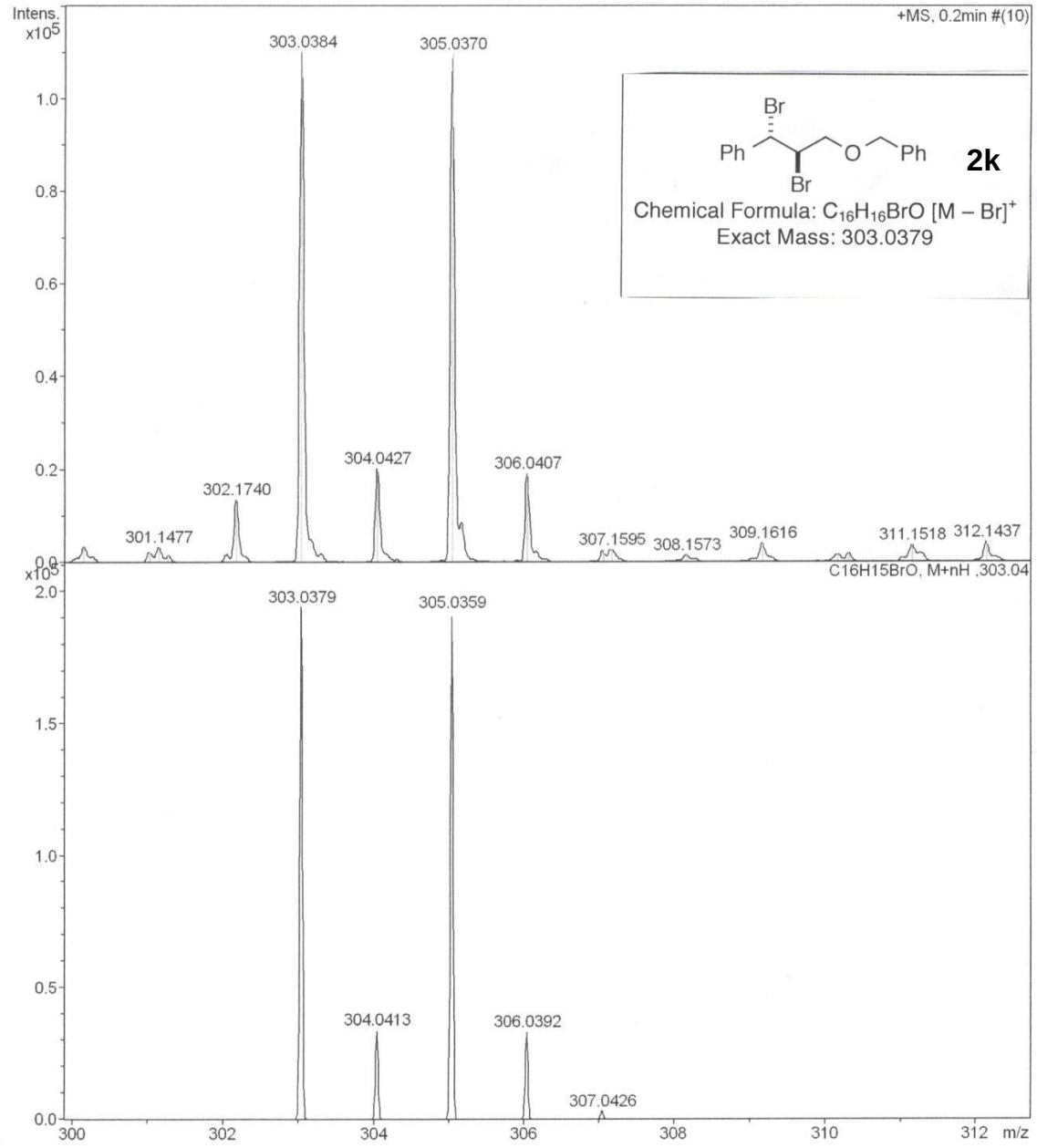
Display Report

Analysis Info

Analysis Name	C:\Data\LabSelen 13.09.17 APCI\I-NSM-149000004.d	Acquisition Date	13/9/2017 11:20:50
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	I-NSM-149	Instrument	micrOTOF-Q 10243
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	2000 V	Set Dry Heater	250 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	1.5 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Source



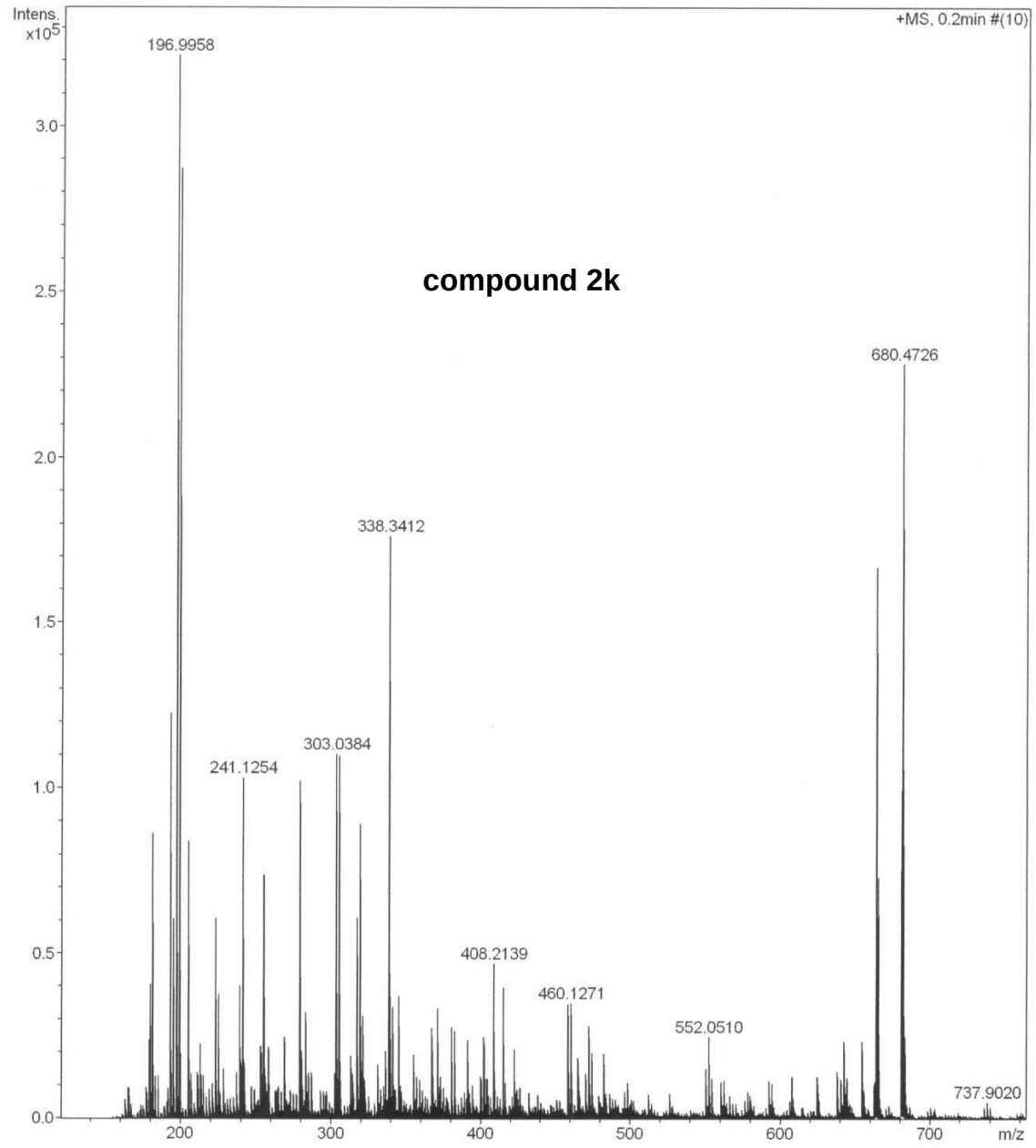
Display Report

Analysis Info

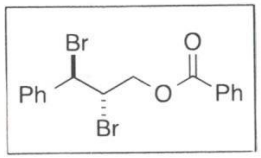
Analysis Name	C:\Data\LabSelen 13.09.17 APCI\I-NSM-149000004.d	Acquisition Date	13/9/2017 11:20:50
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	I-NSM-149	Instrument	micrOTOF-Q 10243
Comment			

Acquisition Parameter

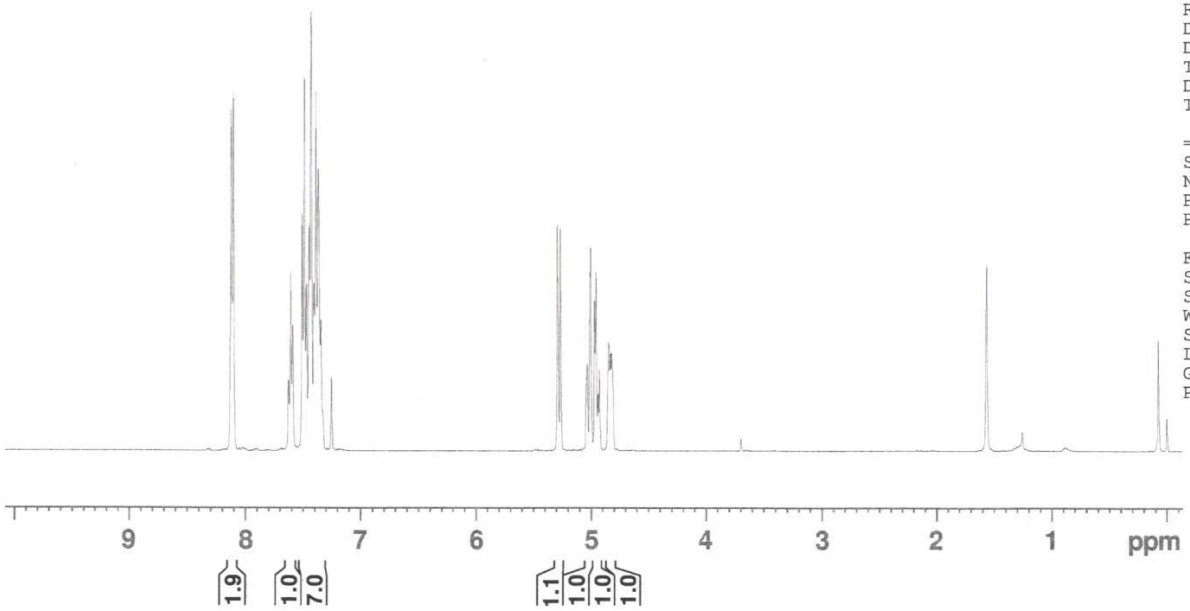
Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	2000 V	Set Dry Heater	250 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	1.5 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Source



compound 2k



2l

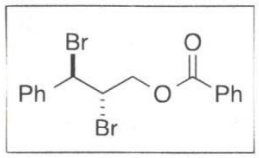



```
Current Data Parameters
NAME          I-NSM-137
EXPNO                 3
PROCNO                1

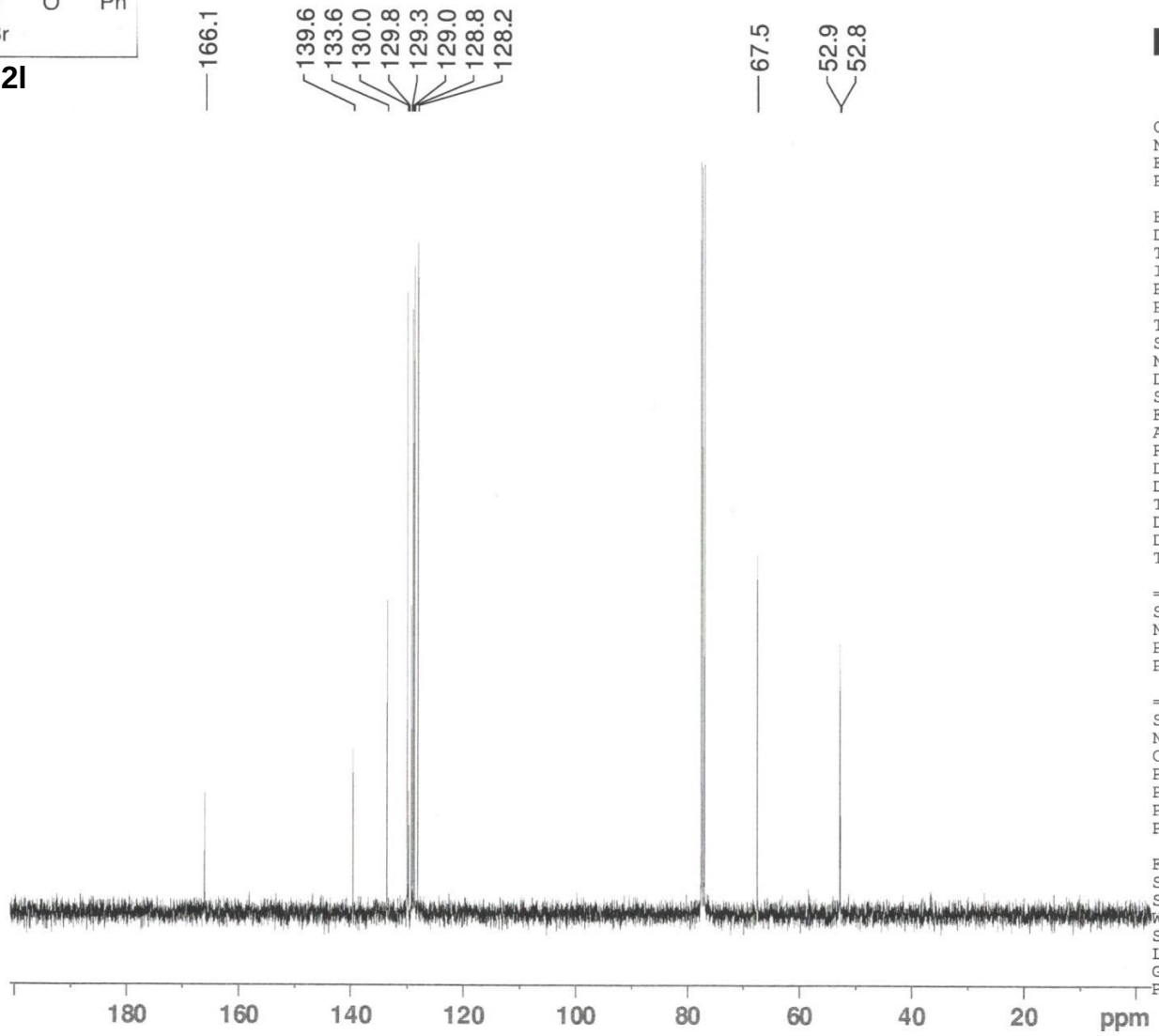
F2 - Acquisition Parameter
Date_          20170609
Time              12.16
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  20
DS                   0
SWH           8012.820 Hz
FIDRES         0.122266 Hz
AQ           4.0894465 sec
RG                154.14
DW              62.400 usec
DE              10.00 usec
TE              300.3 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1124007 MHz
NUC1                1H
P1              11.75 usec
PLW1        11.19999981 W

F2 - Processing parameters
SI               65536
SF          400.1100131 MHz
WDW                 EM
SSB      0
LB              0.30 Hz
GB       0
PC              1.00
```



2l




```
Current Data Parameters
NAME          I-NSM-137
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameter
Date_          20170606
Time              16.25
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  169
DS                    4
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ            0.6488064 sec
RG                197.86
DW              19.800 usec
DE              10.00 usec
TE                300.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6176997 MHz
NUC1                 13C
P1                13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 use
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI                32768
SF          100.6077179 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

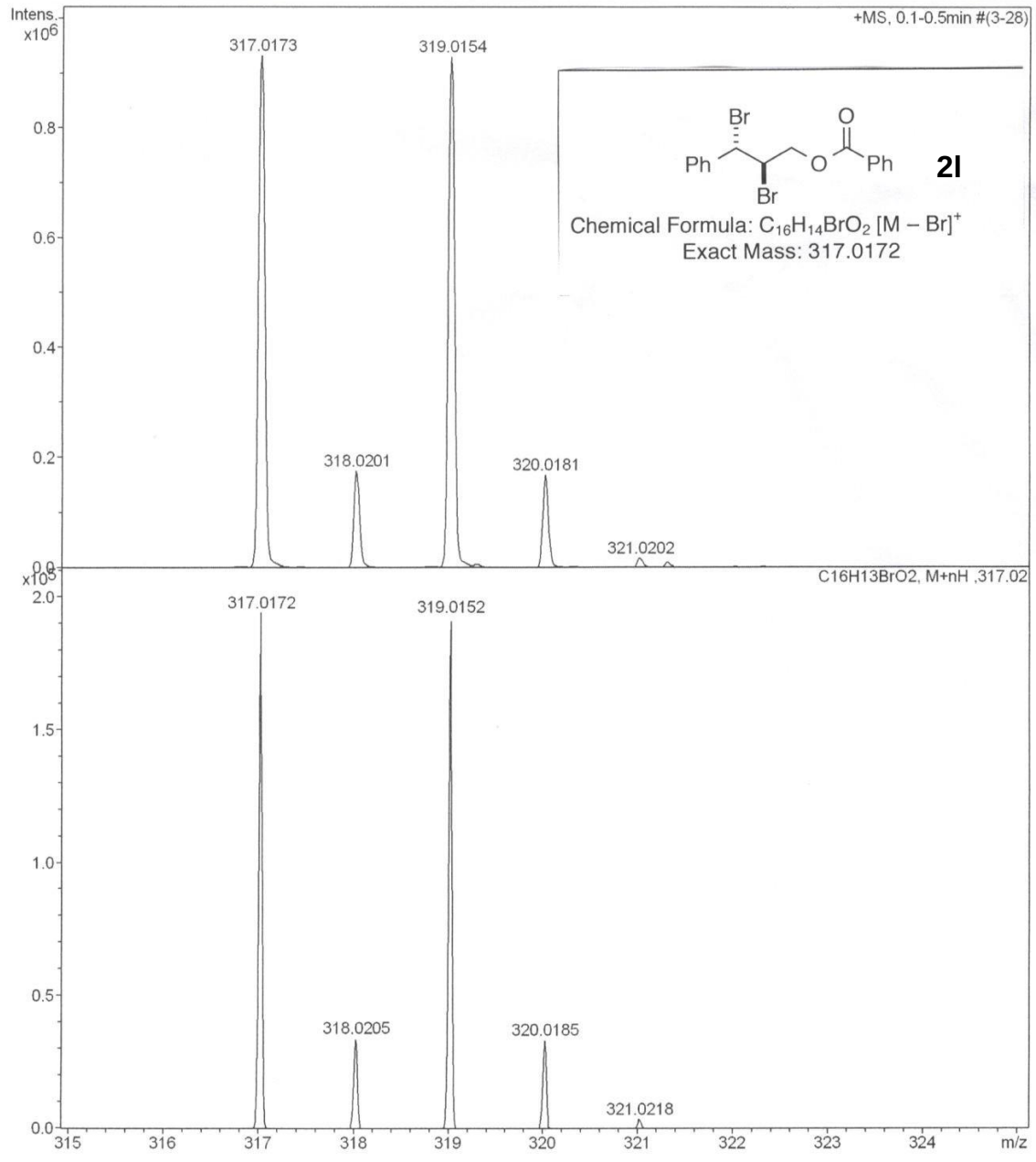
Display Report

Analysis Info

Analysis Name	C:\Data\LabSelen 13.09.17 APCI\I-NSM-137000003.d	Acquisition Date	13/9/2017 11:04:44
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	I-NSM-137	Instrument	micrOTOF-Q 10243
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	250 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	1.5 l/min
Scan End	1000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Source



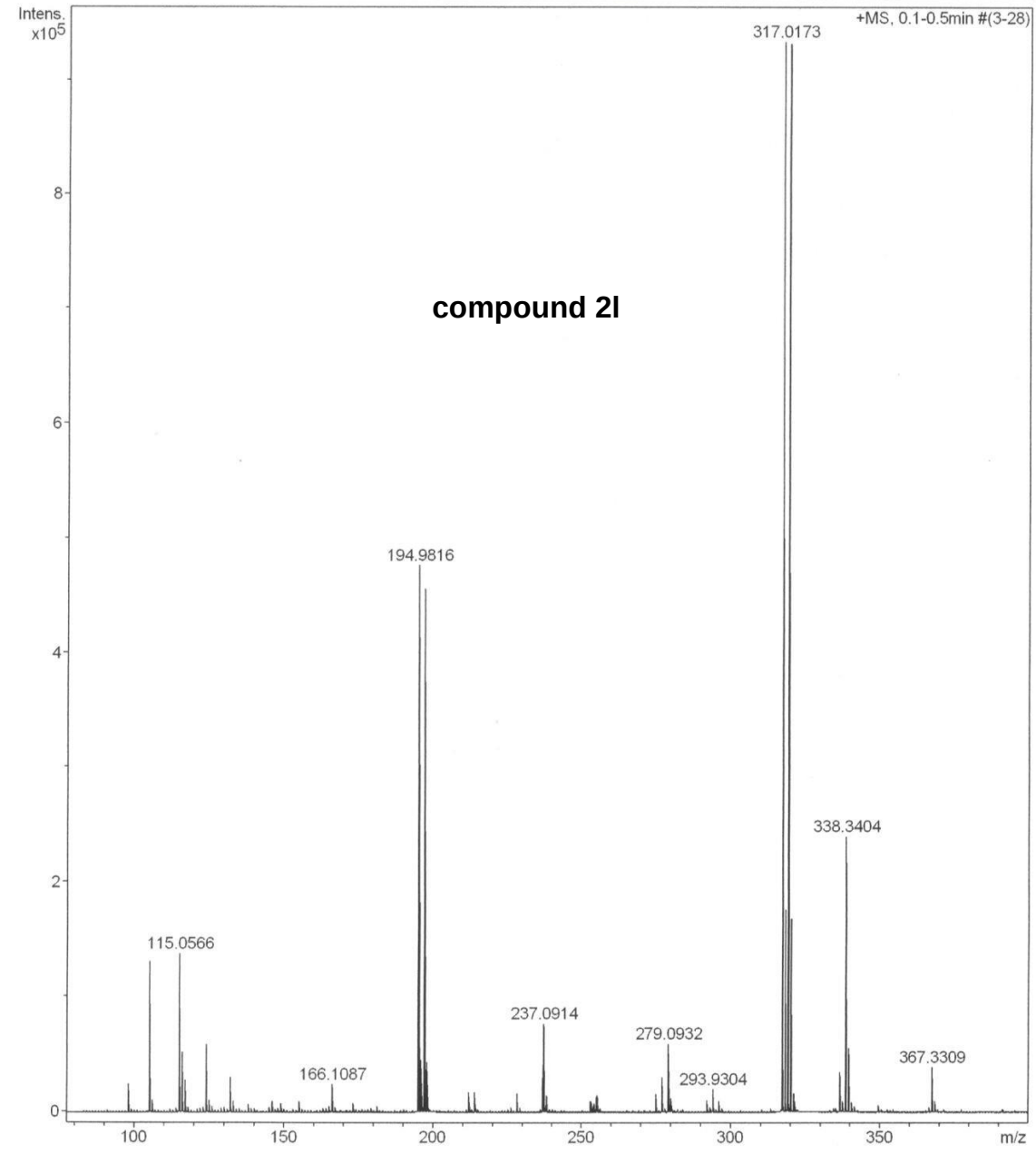
Display Report

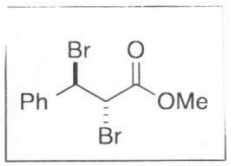
Analysis Info

Analysis Name	C:\Data\LabSelen 13.09.17 APCI\I-NSM-137000003.d	Acquisition Date	13/9/2017 11:04:44
Method	tune_low_APCI.m	Operator	Bruker
Sample Name	I-NSM-137	Instrument	micrOTOF-Q 10243
Comment			

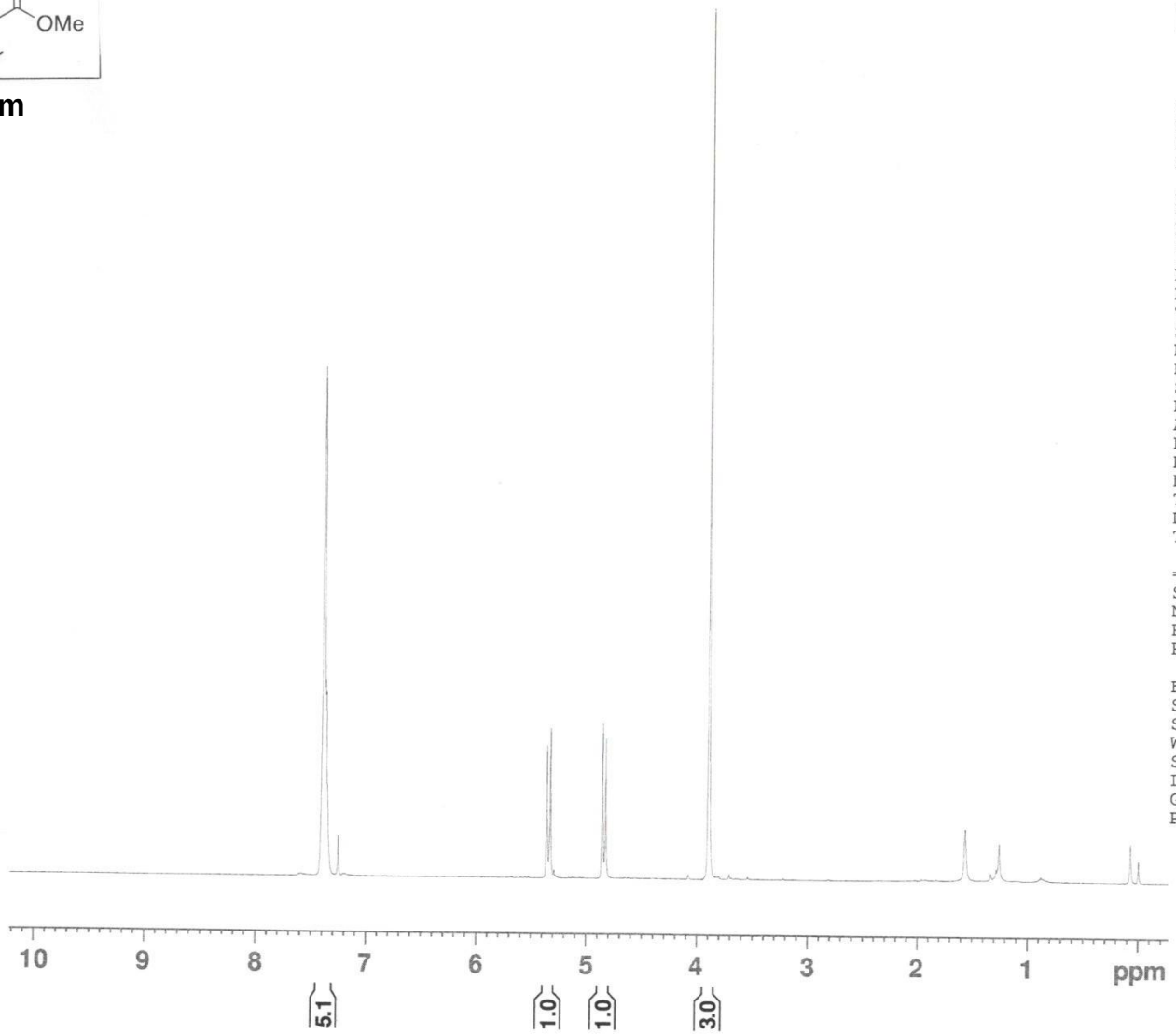
Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	250 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	1.5 l/min
Scan End	1000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Source





2m



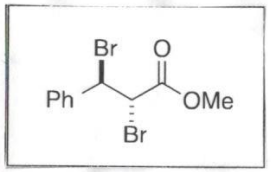
BRUKER

```
Current Data Parameters
NAME          I-NSM-146-2
EXPNO                   1
PROCNO                  1

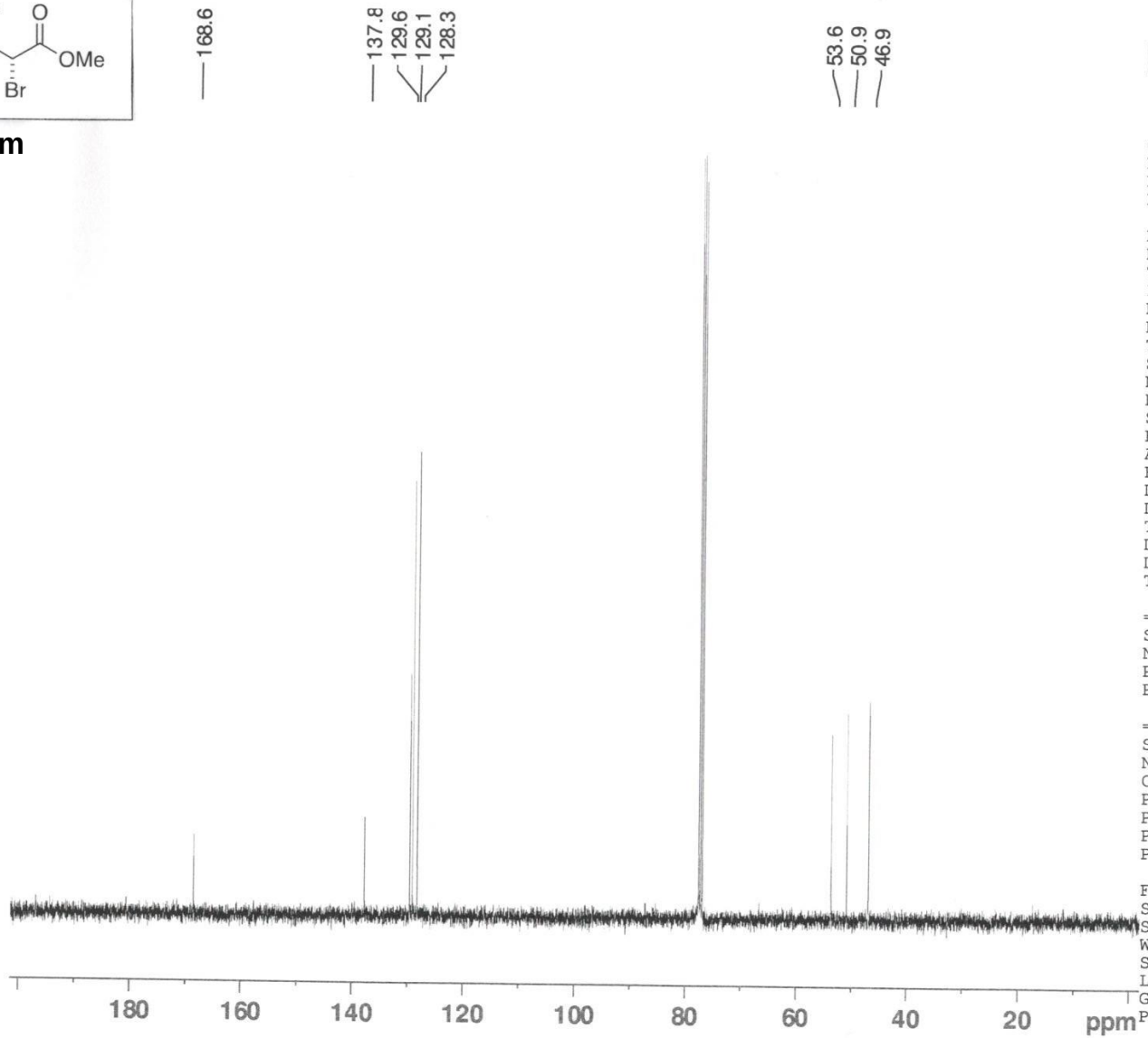
F2 - Acquisition Parameter
Date_          20170718
Time               8.20
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    0
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG                154.14
DW             62.400 usec
DE             10.00 usec
TE                300.9 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1124007 MHz
NUC1                   1H
P1              11.75 usec
PLW1         11.19999981 W

F2 - Processing parameters
SI                 65536
SF          400.1100114 MHz
WDW                   EM
SSB       0
LB              0.30 Hz
GB        0
PC                 1.00
```



2m



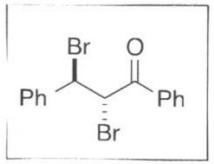

```
Current Data Parameters
NAME          I-NSM-146-2
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameter
Date_          20170718
Time               8.24
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                 130
DS                   4
SWH          25252.525 Hz
FIDRES        0.770646 Hz
AQ           0.6488064 sec
RG              197.86
DW              19.800 usec
DE              10.00 usec
TE              300.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

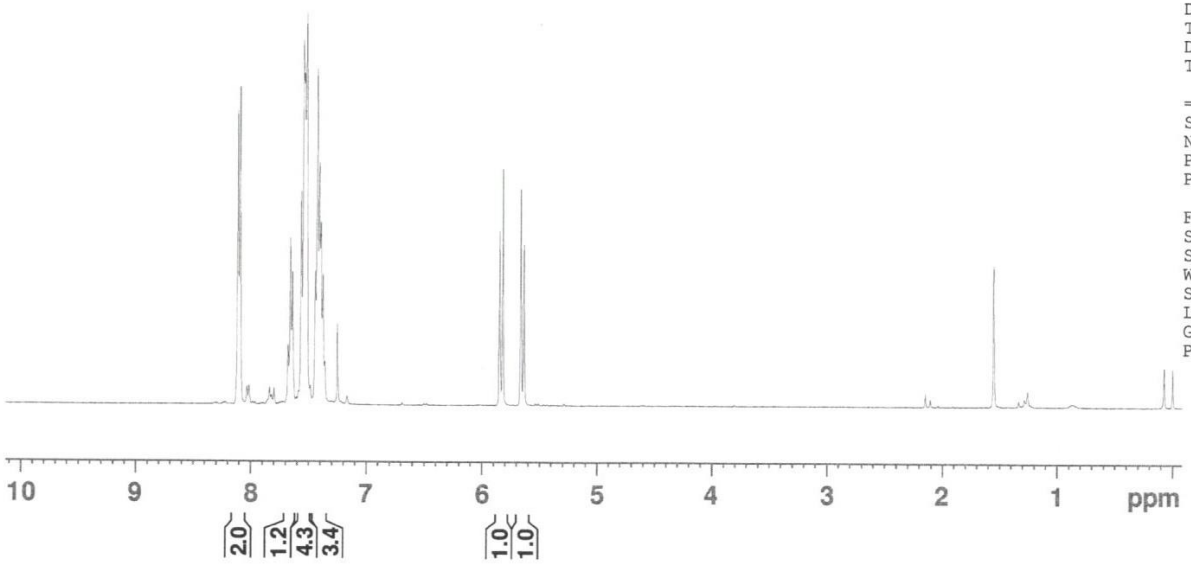
======== CHANNEL f1 ========
SFO1        100.6176997 MHz
NUC1              13C
P1              13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2              1H
CPDPRG[2        waltz16
PCPD2            90.00 use
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI                32768
SF          100.6077179 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```



2n



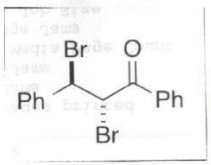
BRUKER

```
Current Data Parameters
NAME          I-NSM-138
EXPNO                 3
PROCNO                1

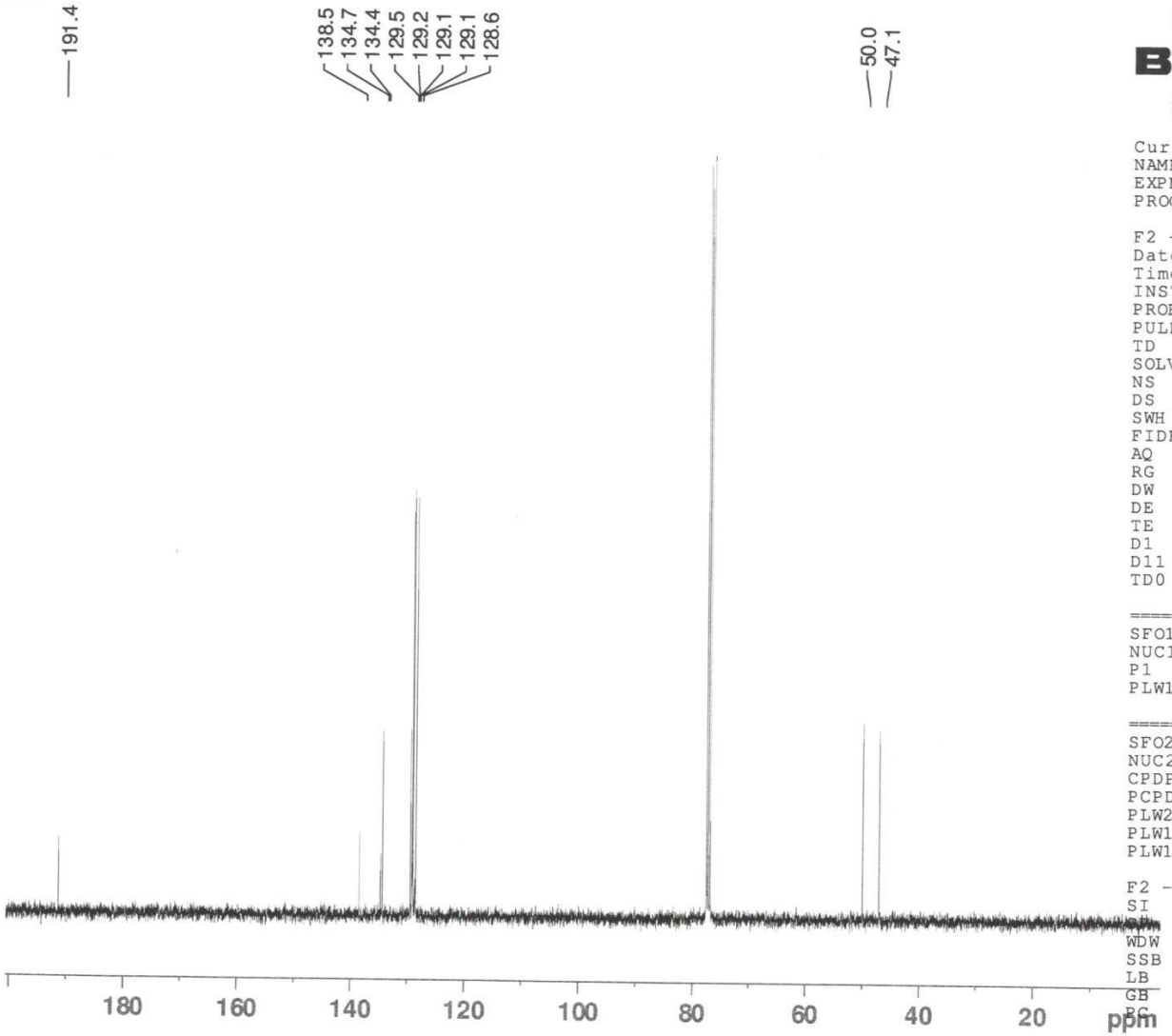
F2 - Acquisition Parameter
Date_          20170621
Time               8.33
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    0
SWH            8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG               175.09
DW               62.400 usec
DE               10.00 usec
TE               300.3 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1124007 MHz
NUC1                   1H
P1                 11.75 usec
PLW1        11.19999981 W

F2 - Processing parameters
SI                 65536
SF          400.1100130 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2n



BRUKER

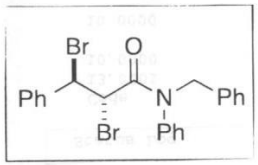
```
Current Data Parameters
NAME          I-NSM-138
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameter
Date_          20170621
Time               8.35
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  225
DS                    4
SWH           25252.525 Hz
FIDRES         0.770646 Hz
AQ            0.6488064 sec
RG                197.86
DW              19.800 usec
DE              10.00 usec
TE                300.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

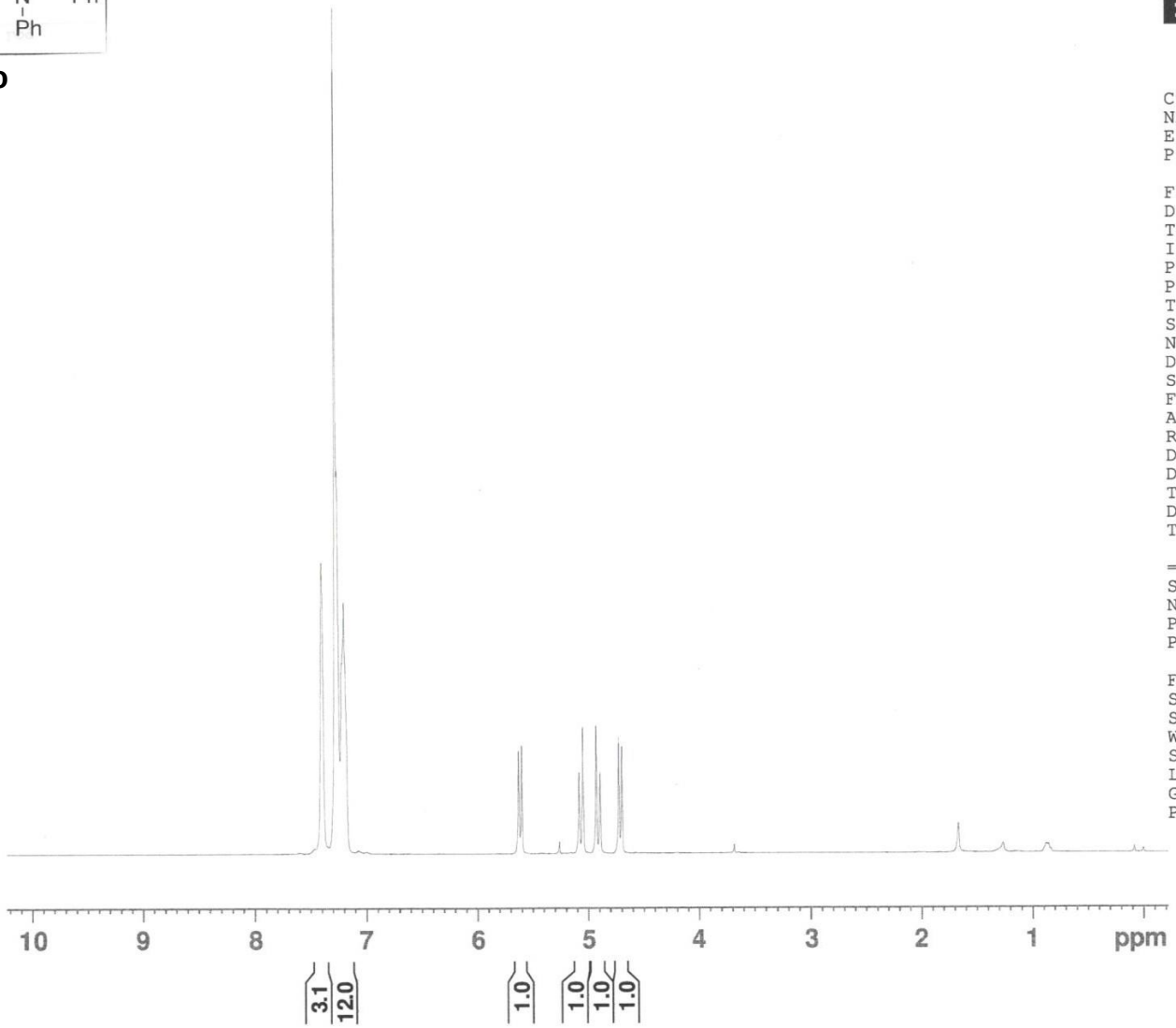
======== CHANNEL f1 ========
SFO1        100.6176997 MHz
NUC1                13C
P1                13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI                 32768
SF          100.6077179 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



2o



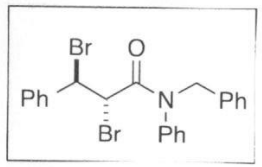
BRUKER

```
Current Data Parameters
NAME          I-NSM-142
EXPNO                 4
PROCNO                1

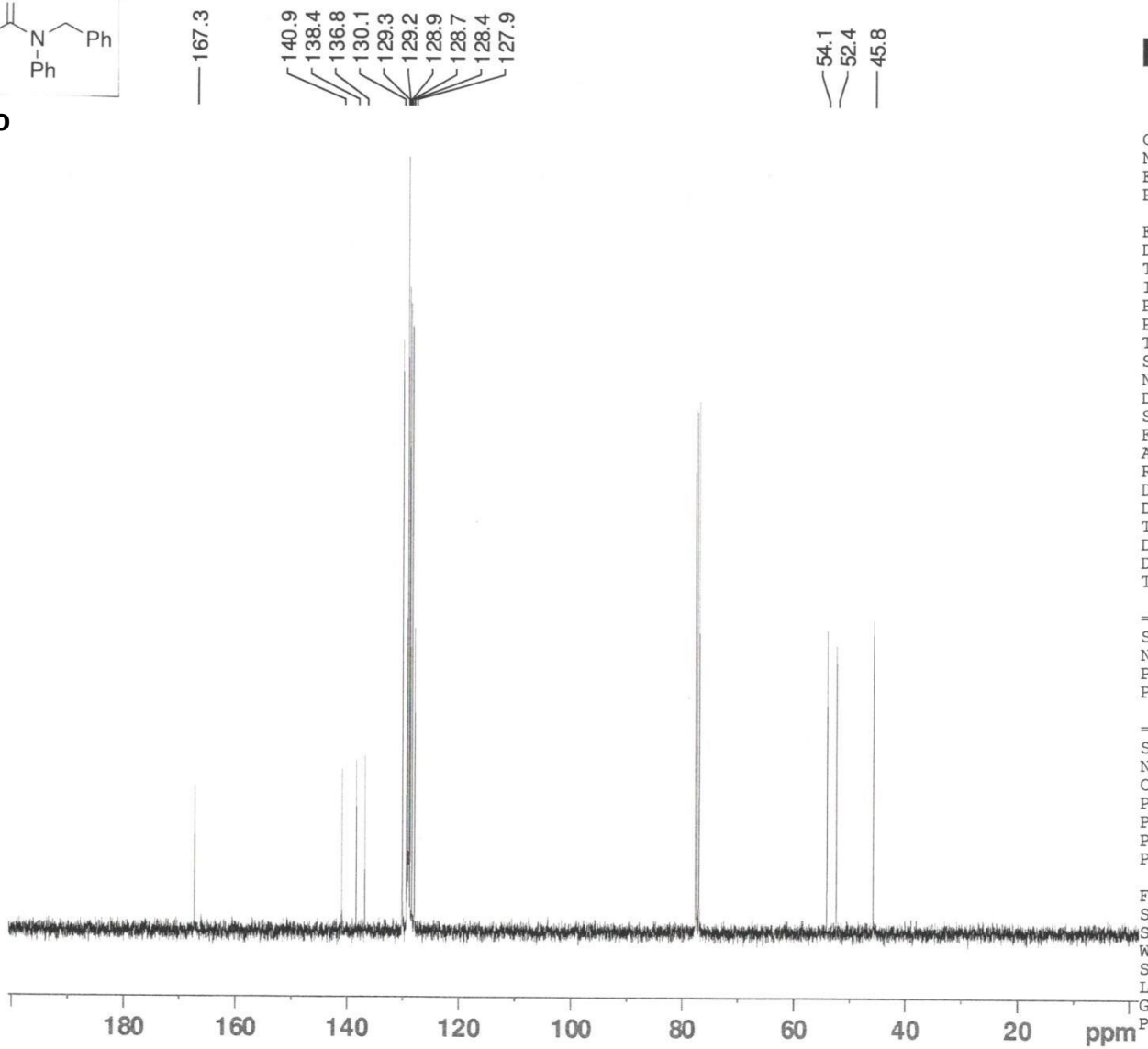
F2 - Acquisition Parameter
Date_          20170626
Time               8.16
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   20
DS                    0
SWH           8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                71.32
DW               62.400 usec
DE               10.00 usec
TE                301.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1124007 MHz
NUC1                   1H
P1                11.75 usec
PLW1        11.19999981 W

F2 - Processing parameters
SI                 65536
SF          400.1100188 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2o



BRUKER

```
Current Data Parameters
NAME          I-NSM-142
EXPNO                 5
PROCNO                1

F2 - Acquisition Parameter
Date_          20170626
Time               8.17
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                  75
DS                   4
SWH          25252.525 Hz
FIDRES         0.770646 Hz
AQ            0.6488064 sec
RG               197.86
DW             19.800 usec
DE             10.00 usec
TE               301.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6176997 MHz
NUC1                 13C
P1              13.86 usec
PLW1        17.98900032 W

======== CHANNEL f2 ========
SFO2        400.1116004 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 use
PLW2        11.19999981 W
PLW12        0.19090000 W
PLW13        0.15463001 W

F2 - Processing parameters
SI              32768
SF         100.6077179 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

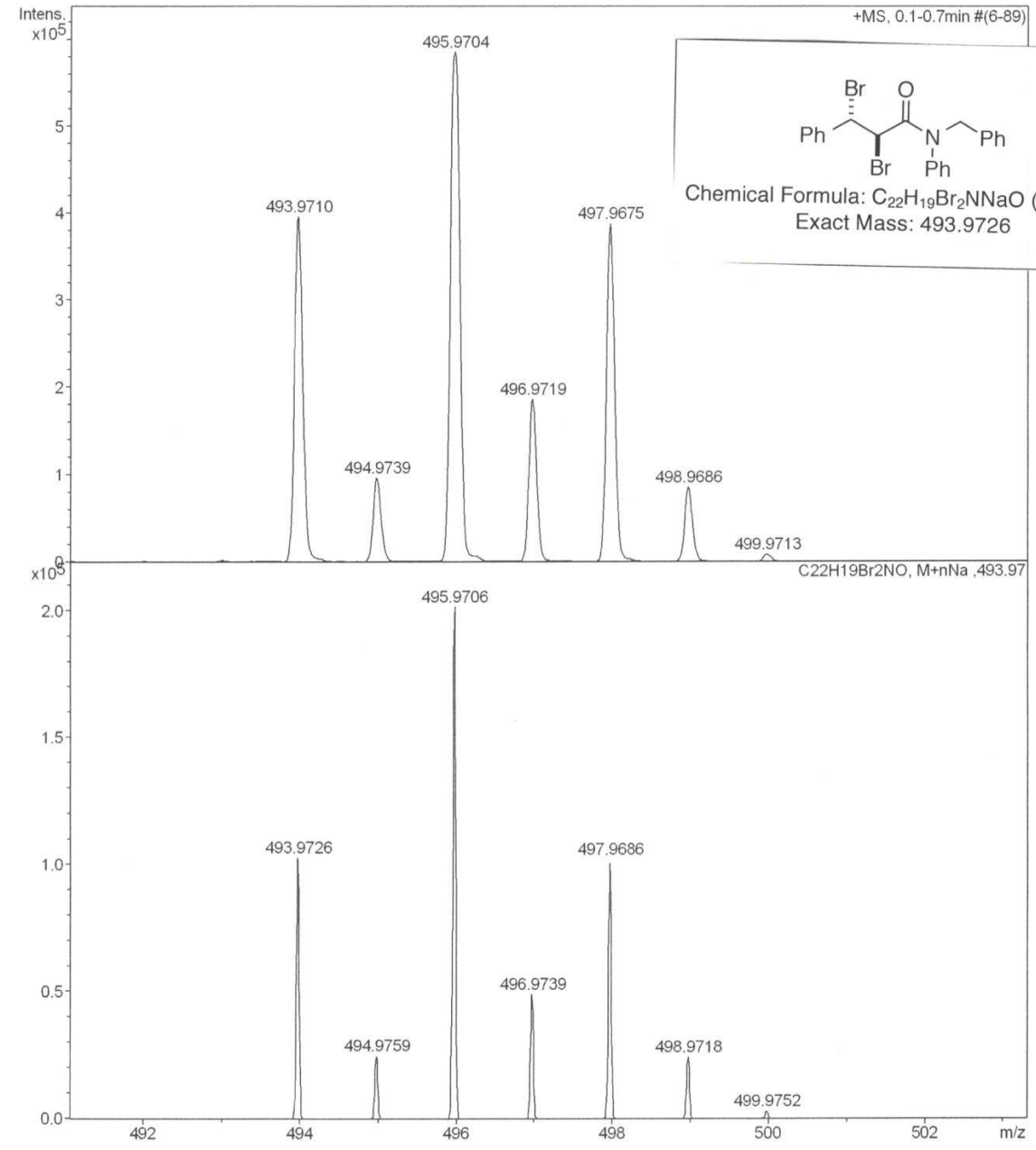
Display Report

Analysis Info

Analysis Name	C:\Data\LabSelen 01.08.17\I-NSM-142000001.d	Acquisition Date	1/8/2017 09:35:32
Method	tune_low_POS_internalcalibration INJEÇÃO.m	Operator	Bruker
Sample Name	I-NSM-142	Instrument	micrOTOF-Q 10243
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste



2o

Chemical Formula: $C_{22}H_{19}Br_2NNaO$ $(M + Na)^+$
Exact Mass: 493.9726

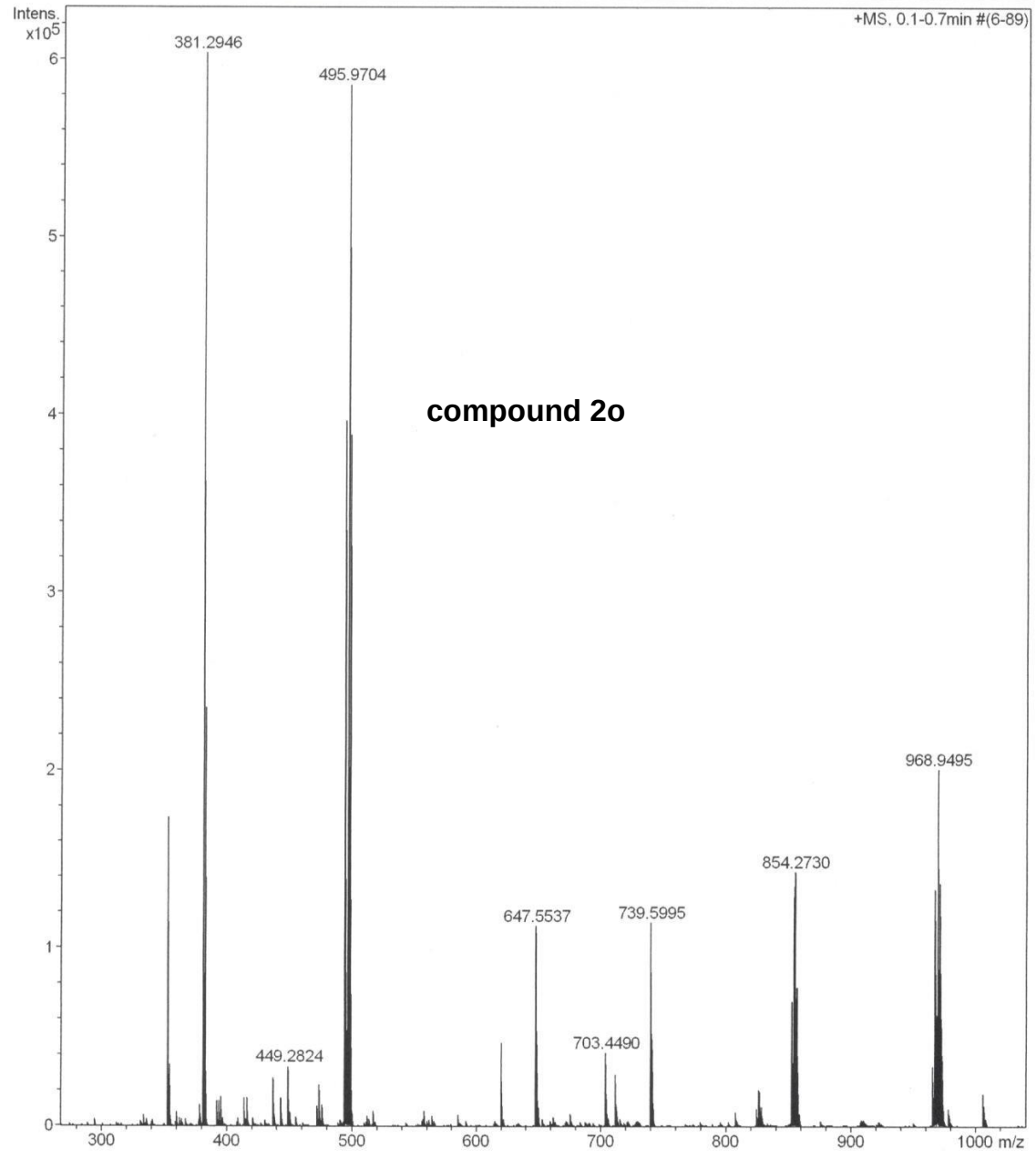
Display Report

Analysis Info

Analysis Name	C:\Data\LabSelen 01.08.17\I-NSM-142000001.d	Acquisition Date	1/8/2017 09:35:32
Method	tune_low_POS_internalcalibration INJEÇÃO.m	Operator	Bruker
Sample Name	I-NSM-142	Instrument	micrOTOF-Q 10243
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste



compound 2o