Electronic Supplementary Material (ESI) for Inorganic Chemistry Frontiers.

Rapid Synthesis of Redox-Active Dodecaborane $B_{12}(OR)_{12}$ Clusters Under Ambient Conditions - SI

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Supporting Information (SI)

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Experimental Section

General considerations. Initial microwave synthesis reactions were carried out under an inert atmosphere of nitrogen using standard glovebox techniques. All post-microwave work-up and characterization was performed under ambient conditions. All reactions designated as "open-air" were carried out and worked up under ambient conditions. The "ambient conditions" for this manuscript refer to room temperature (20 - 25 °C) and uncontrolled laboratory air.

Materials. Deuterated solvents were purchased from Cambridge Isotope Laboratories and used as received. MilliQ water described in this manuscript refers to purified potable water with a resistivity at 25 °C of ≤18.2 MΩ·cm. $[NEt_3H]_2[B_{12}H_{12}]$ was purchased from Boron Specialties (USA). Ethanol (200 proof) was purchased from Decon Labs and used as received. $FeCl_3 \cdot 6H_2O$ (≥97%), $CsOH \cdot 1H_2O$ (≥99.5%), H_2O_2 (30% in H_2O), $[N^nBu_4]OH$ (40% in H_2O), bromoethane (≥99%), 6-bromo-1-hexane (98%) allyl bromide (99%), 4-nitrobenzyl bromide (99%), acetonitrile (≥99.9%), CH_2Cl_2 (≥99.5%), ethyl acetate (≥99.5%), hexanes (≥98.5%), methanol (≥99.8%), *N*,*N*-diisopropylethylamine (≥99%), and tetrabutylammonium hexafluorophosphate (≥99.0%, electrochemical grade and 98%, recrystallized from ethanol and dried under vacuum at 90 °C) were purchased from Sigma-Aldrich. Benzyl bromide (99%) and ethyl 4-bromobutyrate (98%) were purchased from Alfa Aesar, and 4-methylbenzyl bromide (98%) was purchased from Acros. 6-bromo-1-hexene (98%), undec-10-enyl bromide (95%), 4-trifluoromethylbenzyl bromide (99%), and 3,5-bis$(CF_3)_2$-benzyl bromide (97%) were purchased from Oakwood. All reagents were used as received unless otherwise indicated.

Instruments. Bruker AV400 and AV500 spectrometers were used to obtain ^{11}B, $^{13}C\{^1H\}$, ^1H, and ^{19}F NMR spectra and Bruker Topspin software was used to process the NMR data. $^{13}C\{^1H\}$ and

^{1}H NMR spectra were referenced to residual solvent resonances in deuterated solvents (due to high humidity H_2O resonances are often present). ^{11}B and ^{19}F NMR spectra were referenced to $BF_3{\cdot}Et_2O$ and $CFCl_3$ standards, respectively. A Bruker EMX EPR spectrometer was used to acquire EPR spectra, with all spectra collected in CH_2Cl_2 at ambient temperature. Mass spectrometry data was acquired using a Thermo Scientific^{TM} Q-Exactive^{TM} Plus instrument with a quadrupole mass filter and Orbitrap mass analyzer (compounds 2-13), and a Thermo Instruments Exactive Plus with IonSense ID-CUBE DART source instrument for compound 14. IR spectroscopy was acquired on solid samples using a PerkinElmer Spectrum Two FT-IR spectrometer equipped with a diamond universal ATR probe. X-ray photoelectron spectroscopy (XPS) data was acquired using an AXIS Ultra DLD instrument (Kratos Analytical Inc., Chestnut Ridge, NY, USA) with a monochromatic Al Kα X-ray source (10 mA for survey and high-resolution scans). A 300 x 700 nm oval spot size and ultrahigh vacuum (10^{-9} Torr) were used, with 160 eV pass energy for survey spectra and 20 eV for high-resolution spectra of B 1s using a 200 ms dwell time and 20 scans. All XPS peaks were externally referenced to the C 1s signal at 284.6 eV. The experimental setup and design of the infrared-spectroelectrochemistry (IR-SEC) cell has been published previously.[1,2]

X-ray data collection and processing parameters. For [**11**] and [**13**]$^{1-}$, a single crystal was mounted on a nylon loop using perfluoropolyether oil and cooled rapidly to 100 K with a stream of cold dinitrogen. Diffraction data were measured using a Bruker APEX-II CCD diffractometer using Mo-K_α radiation. The cell refinement and data reduction were carried out using Bruker SAINT and the structure was solved with SHELXS-97. All subsequent crystallographic calculations were performed using SHELXL-2013.

Cyclic voltammetry and IRSEC. Cyclic voltammetry was performed on [**11**] and [**14**] using a CH Instruments CHI630D potentiostat with a glassy carbon disc working electrode, platinum wire

counter electrode, and Ag/Ag^+ wire pseudoreference. All experiments were conducted in 0.1M $[N^nBu_4]PF_6/CH_2Cl_2$ with 0.5 mM analyte concentrations (11.2 mg in 10 mL for [**11**] and 3.7 mg in 10 mL for [**14**]). The CH_2Cl_2 was dried in house with a custom drying system running through two alumina columns prior to use. The solution was degassed by bubbling Ar, and the cyclic voltammetry was performed under Ar gas. For [**11**], a scan rate of 0.1 mV/s was used with Fc/Fc^+ as an external standard. For [**14**], a scan rate of 0.5 mV/s was used with Fc/Fc^+ as an internal standard.

IRSEC and cyclic voltammetry for [**13**] were performed using a Pine Instrument Company model AFCBP1 bipotentiostat and BAS Epsilon potentiostat, respectively. For IR-SEC, as the potential was scanned, thin-layer bulk electrolysis was monitored by Fourier-Transform Reflectance IR off the electrode surface. All experiments were conducted in 0.1 M $[N^nBu_4]PF_6/CH_2Cl_2$ solutions with analyte concentrations of ~5 mM (13.9 mg in 1 mL) prepared under a nitrogen atmosphere. The IR-SEC cell used a glassy carbon working electrode, Pt wire counter electrode, and Ag wire pseudoreference electrode. The anionic $[N^nBu_4]_2[\mathbf{13}]^{2-}$ was used for IRSEC, starting at resting potential and increasing to more oxidizing potentials stepwise.

Microwave Synthesis. Microwave reactions were performed using a CEM Discover SP microwave synthesis reactor. Except where noted otherwise, all reactions were performed in glass 10 mL microwave reactor vials purchased from CEM with silicone/PTFE caps. Flea micro PTFE-coated stir bars were used in the vials with magnetic stirring set to high and 15 seconds of premixing prior to the temperature ramping. All microwave reactions were carried out at 140 °C with the pressure release limit set to 250 psi (no reactions exceeded this limit to trigger venting) and the maximum wattage set to 250W (the power applied was dynamically controlled by the microwave instrument and did not exceed this limit for any reactions). Column chromatography

was performed using 2.0 - 2.25 cm inner diameter glass fritted chromatography columns with 20-30 cm of slurry-packed silica gel to ensure full separation of reagents and products. Unfiltered pressurized air was used to assist column chromatography.

Dicesium Dodecahydroxy-*closo*-dodecaborate Cs_2[1].

$CsOH{\cdot}H_2O$ (14.00 g, 83.4 mmol) was dissolved in methanol (130 mL) in a 300 mL glass round bottom flask. $[NEt_3H]_2[B_{12}H_{12}]$ (13.3758 g, 38.9 mmol) was added along with a PTFE-coated stir bar, and the reaction was left to stir vigorously for 18 h at ambient temperature. The cloudy suspension was then filtered through a 60 mL fritted glass funnel and washed with methanol (3 x 20 mL). The resulting white solid was dried on the frit for 1.5 h then left under high vacuum for 12 h and complete conversion to Cs_2[*closo*-$B_{12}H_{12}$] was confirmed by the absence of amine resonances in the ^{1}H NMR spectrum. Alternatively, commercially-obtained $Cs_2[B_{12}H_{12}]$ (98%, Strem) can be utilized for hydroxylation.

Note: The perhydroxylation procedure described herein should always be undertaken with caution and careful planning in order to ensure the $Cs_2[B_{12}H_{12}]$ reagent is pure and contains no organic contaminants. Blast shielding to contain any possible explosions should be utilized. Under no circumstances should the hydrogen peroxide used in the reaction come into contact with any organic material or solvents due to the possibility of an explosion. Synthesis of $Cs_2[B_{12}(OH)_{12}]$ and the ion exchange to produce $[N^nBu_4]_2[B_{12}(OH)_{12}]$ have been described elsewhere,[3,4] but will be reported here for convenience. $Cs_2[B_{12}H_{12}]$ (15.0 g, 36.8 mmol) was added to a glass three-necked round bottom flask with a water-cooled condensing coil in the top slot. The rear neck outlet contained a stopcock for venting pressure, and the front outlet was sealed with a glass stopper and secured with a plastic Keck clip. The apparatus was suspended in a silicone oil bath on a hot plate and secured, with a blast shield in front as a precaution against any potential explosion. The oil

bath was heated to 95 °C, and a 50 mm oval PTFE stir bar was added to the flask, and the reaction was initiated with the addition of H_2O_2 (50 mL, 30% in H_2O). The flask was stoppered and the mixture allowed to stir at that temperature for 2 h. After 2 h, additional H_2O_2 (12 mL) was added, with the flask being vented, the glass stopper removed, and upon completion of addition, re-stoppering the flask. This addition of H_2O_2 was repeated every 2 h until a total volume of 60 mL was added to the reaction mixture. Upon completion of the addition, the oil bath temperature was increased to 105 °C, and additional H_2O_2 aliquots (10-15 mL) were added every 2 - 3 days, cooling the solution in the flask by raising it out of the oil bath and leaving it to cool for 20 – 30 minutes prior to each addition. After 14 days, the progress of the reaction was assessed *via* ^{11}B NMR, with reaction completion indicated by the appearance of a broad resonance at -18.0 ppm corresponding to $Cs_2[B_{12}(OH)_{12}]$ and the disappearance of the resonance at -16 ppm corresponding to unreacted $Cs_2[B_{12}H_{12}]$. Once the reaction is complete (as assessed by ^{11}B NMR), the mixture was cooled to 2 – 8 °C and cold MilliQ water was used to transfer the solution and solid product to a 150 mL glass fritted filter funnel. The crude product was washed with additional MilliQ water prior to drying on the filter frit under water-aspirator vacuum for 6 – 12 hours. Yield: 18.8 g (85 %).

From this point, N^nBu_4 will be referred to as TBA. For the cation exchange, Dowex 50X8 (100-200 mesh, hydrogen form, Sigma-Aldrich) was washed with MilliQ water in a 500 mL glass beaker until neutral (decanting and discarding the wash fractions), and [TBA]OH (40% in H_2O) was added in 10 mL increments until basic with magnetic stirring using a 50 mm PTFE coated standard stir bar. After 30 minutes, the pH was assessed again, and if no longer basic, additional [TBA]OH was added to restore basicity and left to stir overnight covered with a watch glass. The resin was slurry-packed into a 5 x 30 cm column wrapped with heating tape (controller set to hold temperature at 50 °C), and washed with MilliQ water until neutral. $Cs_2[B_{12}(OH)_{12}]$ (5.996 g,

10.0 mmol) was dissolved in boiling water (600 mL), cooled to 50 °C and slowly added to column. The product was washed with an additional 750 mL of 50 °C water, and the product was concentrated *in vacuo* and lyophilized to produce pure TBA_2[**1**]. Yield: 6.90 g (85 %). TBA_2[**1**] is a white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.66 (s, 12H, O<u>H</u>), 3.08 (m, 8H, N-<u>CH_2</u>), 1.54 (m, 8H, N-CH_2<u>CH_2</u>), 1.25 (m, 8H, N-$(CH_2)_2$<u>CH_2</u>), 0.84 (m, 12H, N-$(CH_2)_3$<u>CH_3</u>). ^{11}B{^{1}H} NMR (128 MHz, D_2O): δ -17.9. *Note: TBA_2[**1**] is air-stable, but hygroscopic. Store under inert atmosphere or in a sealed desiccator to prevent excess absorption of water over extended periods of time under storage.*

General ether alkylation/benzylation of TBA_2[1] to $B_{12}(OR)_{12}$ microwave procedure

Reactions were performed using TBA_2[**1**] which was weighed and placed into a 10 mL glass microwave vial and transferred out of a nitrogen-filled glovebox, being opened to the air prior to synthesis. The acetonitrile solvent, base, and alkyl reagents were all used under ambient temperature and pressure conditions with no additional purification or drying. *Note: the initial inert-atmosphere trials mentioned in the main text for **2**, **3**, and **4** were prepared inside a nitrogen-filled glovebox using rigorously anhydrous solvent. Once the PTFE/silicone cap was placed on the microwave vial it was transferred out of the glovebox and the reactions were performed identically to the open-air reactions.* TBA_2[**1**] (50.0 mg, 0.061 mmol) was transferred to a 10 mL glass microwave vial containing a flea micro stir bar and dissolved in acetonitrile (1 mL). *N*,*N*-diisopropylethylamine (Hünig's base, 0.2 mL, 1.15 mmol) and alkyl halide (7.6 mmol) were added, and a PTFE/silicone cap was placed on the microwave vial. The mixture was heated to 140 °C with stirring in the microwave for 5 min to 8 hrs (depending upon the alkyl halide), with the progress of the reaction monitored *via* ^{11}B NMR spectroscopy. Multiple resonances between -14 and -16 ppm are first observed, indicating partial substitution of the 12 vertices. The reaction has

reached completion when these resonances coalesce to a broad singlet resonance between -14 and -16 ppm corresponding to the fully substituted $[B_{12}(OR)_{12}]^{2-}$ species. The color of the reaction mixture is typically a faint yellow initially, with the completed reaction mixture changing to a pink/purple, faint pink, or deep red/orange color indicative of the 1- species. Upon completion of the reaction, excess acetonitrile and base were removed *via* rotary evaporation. With the exception of **3**, **4**, **6,** and **14** the remaining reaction mixture containing product and unreacted alkyl halide were separated *via* column chromatography with silica gel. The unreacted alkyl halide (clear and colorless or slightly yellow/orange, UV active) was eluted first, followed by the elution of the remaining pink/purple product mixture consisting of 1-/2- species (note that the 2- species is colorless). The excess solvent was removed *via* a rotary evaporator, and the remaining 1-/2- product mixture was dissolved in a 90/5/5 ethanol/acetonitrile/MilliQ H_2O or 90/10 ethanol/acetonitrile mixture and transferred to a 50 mL round bottom flask. $FeCl_3 \cdot 6H_2O$ (0.3 g, 1.11 mmol) was added to the dissolved mixture, and subsequently stirred vigorously for 12-24 h at ambient temperature. The solvent was removed from the resulting dark orange/brown mixture *via* rotary evaporation, and the neutral [*hypercloso*-$B_{12}(OR)_{12}]^0$ or radical TBA[*hypocloso*-$B_{12}(OR)_{12}]^{1-}$ product was separated from the $FeCl_3 \cdot 6H_2O$ *via* column chromatography with silica gel. A dark orange, yellow-orange, or red-orange band consisting of neutral [*hypercloso*-$B_{12}(OR)_{12}]^0$ was eluted with CH_2Cl_2, with a pink/purple band containing charged 1-/2- species eluting next if any product was not fully oxidized. The red or orange fraction containing the desired neutral closomer was dried with rotary evaporation followed by high vacuum, and the above procedure for oxidation could be repeated on the remaining 1-/2- mixture to obtain additional neutral product if any non-fully oxidized product remains. *Note: if the final oxidized product*

appears to have any impurities (via ^{1}H NMR spectroscopy), eluting the product through an additional silica plug or short column with 1:1 CH_2Cl_2/hexanes should remove any contaminants.

Dodeca(benzyloxy)-*hypercloso*-dodecaborane [2]

TBA_2[**1**] (50.0 mg, 0.061 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 1 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.2 mL, 1.15 mmol) and benzyl bromide (1.74 mL, 14.7 mmol) were added along with a flea micro stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 15 min. The volatiles were removed *via* rotary evaporation, and the excess reagent was eluted through a slurry-packed silica gel column with 65/35 hexanes/ethyl acetate, and the pink/purple product mixture was eluted with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation, the remaining charged 1-/2- product mixture was dissolved in 5 mL 90/5/5 ethanol/acetonitrile/H_2O, $FeCl_3{\cdot}6H_2O$ (0.3 g, 1.11 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and an orange band containing the neutral product was separated from the $FeCl_3{\cdot}6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation and the final neutral product **2** was dried under high vacuum to obtain an isolated yield of 54.3 mg (63%). Compound **2** is a dark orange solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.08 - 7.19 (m, 60H, C_6H_5), 5.25 (s, 24H, O-$\underline{CH_2}$). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ 140.8, 128.4, 127.3, 73.4. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): δ 41.8. HRMS (Orbitrap): *m/z* calculated for $C_{84}H_{84}B_{12}O_{12}$ (M^-), 1414.7152 Da; found, 1414.7183 Da.

Dodeca(allyloxy)-*hypercloso*-dodecaborane [3]

Note: this reaction should be performed with minimal exposure to light. TBA_2[**1**] (100.0 mg, 0.122 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 2 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.4 mL, 2.30 mmol) and allyl bromide (1.28 mL, 14.68 mmol) were added along with a flea micro stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 15 min. The volatiles and excess reagent were removed *via* rotary evaporation and the purple 1-/2- product mixture was dissolved in 5 mL 90/10 ethanol/acetonitrile, $FeCl_3 \cdot 6H_2O$ (0.3 g, 1.11 mmol) was added and the mixture was left to stir for 12-24 h (wrapped in foil to avoid excessive light exposure). Following oxidation, the solvent mixture was removed *via* rotary evaporation, and a yellow band containing the neutral product was separated from the $FeCl_3 \cdot 6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation and the final neutral product **3** was dried under high vacuum to obtain an isolated yield of 76.8 mg (77%). Compound **3** is a dark yellow-orange viscous oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.91 – 5.99 (m, 12H, CH), 5.21 (dq, 12H, CH), 5.05 (dq, 12H, CH), 4.62 (m, 24H, O-$\underline{CH_2}$). $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 136.9, 114.2, 71.6. $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$): δ 41.1. HRMS (Orbitrap): *m/z* calculated for $C_{36}H_{60}B_{12}O_{12}$ (M^-), 814.5274 Da; found, 814.5333 Da. *Note: Compound **3** should be stored at -20 °C or used immediately.*

Dodeca(ethoxy)-*hypercloso*-dodecaborane [4]

TBA_2[**1**] (50.0 mg, 0.061 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 1 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.2 mL, 1.15 mmol) and bromoethane (1.65 mL, 22.1 mmol) were added along with a flea micro stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was

heated at 140 °C with stirring in the microwave for 30 min. The volatiles and excess reagent were removed *via* rotary evaporation, the remaining charged 1-/2- product mixture was dissolved in 4 mL 90/5/5 ethanol/acetonitrile/H_2O, $FeCl_3 \cdot 6H_2O$ (0.3 g, 1.11 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and an orange band containing the neutral product was separated from the $FeCl_3 \cdot 6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation and the final neutral product **4** was dried under high vacuum to obtain an isolated yield of 32.7 mg (80%). Compound **4** is a dark orange solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.09 (q, 24H, O-$\underline{CH_2}$), 1.24 (t, 36H, CH_3). $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 66.8, 17.8. $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$): δ 37.7. HRMS (Orbitrap): *m/z* calculated for $C_{24}H_{60}B_{12}O_{12}$ (M^-), 670.5274 Da; found, 670.5278 Da.

Dodeca(hexoxy)-*hypercloso*-dodecaborane [5]

TBA_2[**1**] (99.0 mg, 0.121 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 2 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.4 mL, 2.30 mmol) and 6-bromo-1-hexane (2.85 mL, 20.3 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 2 h. The volatiles were removed *via* rotary evaporation at 65 °C, the remaining charged 1-/2- product mixture was dissolved in 5 mL 90/5/5 ethanol/acetonitrile/H_2O, $FeCl_3 \cdot 6H_2O$ (0.3 g, 1.11 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and an orange band containing the neutral product was separated from the $FeCl_3 \cdot 6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation and the final neutral product **5** was dried under high vacuum to obtain an isolated yield of 91.4 mg

(56%). Compound **5** is a dark yellow-orange oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.02 (t, 24H, O-$\underline{CH_2}$), 1.54 (m, 24H, $CH_2\underline{CH_2}(CH_2)_3CH_3$), 1.31, (m, 72H, $CH_2CH_2(\underline{CH_2})_3CH_3$), 0.89 (m, 36H, CH_3). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 70.2, 32.2, 31.8, 25.9, 22.8, 14.1. ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ 42.0. HRMS (Orbitrap): *m/z* calculated for $C_{72}H_{156}B_{12}O_{12}$ (M^-), 1343.2786 Da; found, 1343.2838 Da.

Dodeca(6-hexeneoxy)-*hypercloso*-dodecaborane [6]

Note: this reaction should be performed with minimal exposure to light. TBA_2[**1**] 50.0 mg (0.061 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 1 mL acetonitrile. *N,N*-diisopropylethylamine (0.2 mL, 1.15 mmol) and 6-bromo-1-hexene (0.59 mL, 4.41 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 7 h. The volatiles and excess reagent were removed *via* rotary evaporation, and the remaining charged 1-/2- product mixture was dissolved in 5 mL 90/10 ethanol/acetonitrile, $FeCl_3{\cdot}6H_2O$ (0.3 g, 1.11 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and an orange band containing the neutral product was separated from the $FeCl_3{\cdot}6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation and the final neutral product **6** was dried under high vacuum to obtain an isolated yield of 35.0 mg (43%). Compound **6** is a dark yellow-orange oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.79 (m, 12H, CH), 4.95 (m, 24H, $\underline{CH_2}CH$), 4.02 (t, 24H, O-$\underline{CH_2}$), 2.05 (q, 24H, $(CH_2)_3\underline{CH_2}CH_2CH$), 1.56 (m, 24H, $CH_2\underline{CH_2}(CH_2)_3CH$), 1.43 (m, 24H, $(CH_2)_2\underline{CH_2}(CH_2)_2CH$). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 139.1, 114.2, 70.1, 33.6, 31.7, 25.5. ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ 41.6. HRMS (Orbitrap): *m/z* calculated for $C_{72}H_{132}B_{12}O_{12}$ (M^-), 1319.0908 Da; found, 1319.1003 Da.

Dodeca(11-undeceneoxy)-*hypercloso*-dodecaborane [7]

Note: this reaction should be performed with minimal exposure to light. TBA_2[**1**] (50.0 mg, 0.061 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 1 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.2 mL, 1.15 mmol) and undec-10-enyl bromide (1.54 mL, 7.33 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was reacted at 140 °C with stirring in the microwave for 8 h. The volatiles were removed *via* rotary evaporation, the excess reagent was eluted through a slurry-packed silica gel column with hexanes, and the product mixture yellow-orange and red-orange fractions were eluted with CH_2Cl_2 followed by a pink fraction with ethyl acetate. The CH_2Cl_2/ethyl acetate was removed *via* rotary evaporation, the remaining charged 1-/2- product mixture was dissolved in 9 mL 49/49/2 ethanol/CH_2Cl_2/acetonitrile, $FeCl_3 \cdot 6H_2O$ (0.5 g, 1.85 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and a dark brown/black band containing the neutral product was separated from the $FeCl_3 \cdot 6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation and the final neutral product **7** was dried under high vacuum to obtain an isolated yield of 37.1 mg (28%). Compound **7** is a dark, brown/black oil. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 5.81 (m, 12H, CH), 4.98 (m, 12H, *trans*-C$\underline{H_2}$CH), 4.91 (m, 12H, *cis*-C$\underline{H_2}$CH), 4.01 (t, 24H, O-$\underline{CH_2}$), 2.03 (m, 24H, O-CH$_2\underline{CH_2}$), 1.54 (m, 24H, O-(CH$_2$)$_2\underline{CH_2}$), 1.32 (m, 144H, O-(CH$_2$)$_3$($\underline{CH_2}$)$_6$CH$_2$CH. ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 139.3, 113.8, 70.4, 33.9, 32.3, 29.9, 29.7, 29.6, 29.3, 29.1, 26.2. ^{11}B{^{1}H} NMR (128 MHz, CD_2Cl_2): δ 41.5. *m/z* calculated for $C_{132}H_{252}B_{12}O_{12}$ (M^-), 2161.0332 Da; due to solubility issues, the molecule was incompatible with our M.S. instrument.

Dodeca(ethylbutyratoxy)-*hypercloso*-dodecaborane [8]

TBA_2[**1**] (100.0 mg, 0.122 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 2 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.4 mL, 2.30 mmol) and ethyl 4-bromobutyrate (1.08 mL, 7.55 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 1.5 h. The volatiles were removed *via* rotary evaporation, and the reaction mixture was eluted through Sephadex LH-20 size exclusion column with methanol, with the pink fraction containing the desired 1-/2- product collected. The methanol was removed *via* rotary evaporation, and the charged 1-/2- product mixture was oxidized by eluting through a slurry-packed silica gel column with 90/10 CH_2Cl_2/ethanol, collecting the orange fraction. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and the product was purified by eluting through a short (15 cm) silica gel column slurry-packed with 1:1 hexanes/ethyl ether, with ~50 mL 1:1 hexanes/ethyl ether followed by ~50 mL ethyl ether eluting an orange band. The volatiles were removed *via* rotary evaporation, and the final neutral product **8** was dried under high vacuum to obtain an isolated yield of 14.0 mg (7%). *Note: ~8% additional 1-/2- product was collected from the first silica column, and by repeating the oxidation step with the charged 1-/2- mixture additional* **8** can be isolated. Compound **8** is an orange solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.11 (q, 24H, O-$\underline{CH_2}$), 4.03 (t, 24H, COO$\underline{CH_2}$), 2.34 (t, 24H, $\underline{CH_2}$COO), 1.87 (m, 24H, O-$CH_2\underline{CH_2}$), 1.24 (t, 36H, CH_3). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ, 173.3, 69.7, 60.3, 30.9, 27.4, 14.2. ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$): δ 41.9. HRMS (Orbitrap): *m/z* calculated for $C_{72}H_{132}B_{12}O_{36}$ (M^-), 1702.9688 Da; found, 1702.9714 Da.

Dodeca(4-methylbenzyloxy)-*hypercloso*-dodecaborane [9]

TBA_2[**1**] (48.0 mg, 0.059 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 1 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.2 mL, 1.15 mmol) and 4-methylbenzyl bromide (1.362 g, 7.36 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 5 min. The volatiles were removed *via* rotary evaporation, and the excess reagent was eluted through a slurry-packed silica gel column with 90/10 hexanes/ethyl acetate, and the pink/purple product mixture was eluted with acetonitrile. The volatiles were removed *via* rotary evaporation, the remaining charged 1-/2- product mixture was dissolved in 5 mL 90/10 ethanol/acetonitrile, $FeCl_3 \cdot 6H_2O$ (0.3 g, 1.11 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and a dark orange band containing the neutral product was separated from the $FeCl_3 \cdot 6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed via rotary evaporation and the final neutral product **9** was dried under high vacuum to obtain an isolated yield of 30.9 mg (33%). Compound **9** is a brown/orange viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.98 (m, 48H, C_6H_4), 5.17 (s, 24H, CH_2), 2.31 (s, 36H, CH_3). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ 137.9, 136.4, 128. 7, 127.3, 72.8, 21.2. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): δ 41.6. HRMS (Orbitrap): *m/z* calculated for $C_{96}H_{108}B_{12}O_{12}$ (M^-), 1582.9030 Da; found, 1582.9058 Da.

Dodeca(4-bromobenzyloxy)-*hypercloso*-dodecaborane [10]

TBA_2[**1**] (50.0 mg, 0.061 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 1 mL acetonitrile. *N*,*N*-

diisopropylethylamine (0.2 mL, 1.15 mmol) and 4-bromobenzyl bromide (1.358 g, 7.36 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 30 min. The volatiles were removed *via* rotary evaporation, and the excess reagent was eluted through a slurry-packed silica gel column with hexanes, and the red-pink product mixture was eluted with CH_2Cl_2 followed by ethyl acetate. The volatiles were removed *via* rotary evaporation, the remaining charged 1-/2- product mixture was dissolved in 5 mL 90/10 ethanol/acetonitrile, $FeCl_3 \cdot 6H_2O$ (0.3 g, 1.11 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and a dark orange band containing the neutral product was separated from the $FeCl_3 \cdot 6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed via rotary evaporation and the final neutral product **10** was dried under high vacuum to obtain an isolated yield of 62.5 mg (43%). Compound **10** is a dark-orange solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 (d, 24H, *m*-$C_6\underline{H}_4$), 6.86 (d, 24H, *o*-$C_6\underline{H}_4$), 5.07 (s, 24H, O-$\underline{CH}_2$). $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ, 138.5, 131.6, 128.5, 121.6, 72.6. $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$): δ 41.4. HRMS (Orbitrap): *m/z* calculated for $C_{84}H_{72}B_{12}Br_{12}O_{12}$ (M^-), 2361.6291 Da; found, 2361.6311 Da.

Dodeca(4-trifluoromethylbenzyloxy)-*hypercloso*-dodecaborane [11]

TBA_2[**1**] (50.0 mg, 0.061 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 1 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.2 mL, 1.15 mmol) and 4-trifluoromethylbenzyl bromide (1.765 g, 7.5 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was reacted at 140 °C with stirring in the microwave for 30 min. The volatiles were removed *via* rotary evaporation, and the excess reagent was eluted through a slurry-packed silica

gel column with hexanes, and the pink/purple product mixture was eluted with acetonitrile. The acetonitrile was removed *via* rotary evaporation, the remaining charged 1-/2- product mixture was dissolved in 5 mL of 90/10 ethanol/acetonitrile, $FeCl_3 \cdot 6H_2O$ (0.4 g, 1.48 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and an orange band containing the neutral product was separated from the $FeCl_3 \cdot 6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation and the final neutral product **11** was dried under high vacuum to obtain an isolated yield of 89.6 mg (66%). Compound **11** is a red-orange solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38 - 7.48 (m, 24H, *m*-$C_6\underline{H}_4$), 7.06 - 7.15 (m, 24H, *o*-$C_6\underline{H}_4$), 5.27 (s, 24H, O-$\underline{CH}_2$). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ 143.0, 130.6, 126.6, 125.7, 125.0, 122.8, 72.9. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): δ 41.8. ^{19}F NMR (376 MHz, $CDCl_3$): δ -62.76 (s, 36F). HRMS (Orbitrap): *m/z* calculated for $C_{96}H_{72}B_{12}F_{36}O_{12}$ (M^-), 2231.5672 Da; found, 2231.5637 Da. Crystallized from $CDCl_3$ and pentane at room temperature for 1 week to obtain a single crystal for X-ray diffraction analysis.

Dodeca(4-nitrobenzyloxy)-*hypocloso*-dodecaborane [12]$^{1-}$

TBA_2[**1**] (50.0 mg, 0.061 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 1 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.2 mL, 1.15 mmol) and 4-nitrobenzyl bromide (1.585 g, 7.34 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 30 min. The volatiles were removed *via* rotary evaporation, and the excess reagent was eluted through a slurry-packed silica gel column with 50/50 hexanes/CH_2Cl_2, and the orange product mixture was eluted with acetonitrile. The volatiles were removed *via* rotary evaporation, the remaining charged 1-/2- product mixture was dissolved

in 5 mL 90/10 ethanol/acetonitrile, $FeCl_3 \cdot 6H_2O$ (0.3 g, 1.11 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and the product mixture was washed with 150 – 200 mL ethanol in a glass fritted 30 mL filter funnel to remove the remaining $FeCl_3 \cdot 6H_2O$. The remaining orange radical 1- product TBA[**12**]$^{1-}$ was removed from the funnel and dried under high vacuum to obtain an isolated yield of 89.0 mg (66%). Compound TBA[**12**]$^{1-}$ is an orange solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 - 7.41 (m, 48H, C_6H_4). *Note: The CH_2 signal is masked and all other peaks are quite broad due to the paramagnetic radical state of the molecule.* $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ 191.26, 147.47, 140.59, 130.55, 124.24. *Note: The CH_2 signal is masked and all other peaks are quite broad due to the paramagnetic radical state of the molecule.* No resonances are visible by ^{11}B NMR due to paramagnetic broadening (a trace resonance at 20.4 ppm is indicative of borates, which result from decomposition). HRMS (Orbitrap): *m/z* calculated for $C_{84}H_{72}B_{12}N_{12}O_{36}$ (M^-), 1954.5361 Da; found, 1954.5363 Da.

Dodeca(3,5-bis(trifluoromethyl)$_2$benzyloxy)-*hypocloso*-dodecaborane [13]$^{1-}$

TBA_2[**1**] (99.0 mg, 0.121 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 2 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.4 mL, 2.30 mmol) and 3,5-bis(trifluoromethyl)benzyl bromide (2.68 mL, 14.6 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 30 min. The volatiles were removed *via* rotary evaporation, and the excess reagent was eluted through a slurry-packed silica gel column with 65/35 hexanes/ethyl acetate, and the colorless/very light pink product mixture was eluted with CH_2Cl_2. After removal of the CH_2Cl_2 *via* rotary evaporation, compound TBA_2[**13**]$^{2-}$, a clear, colorless solid, was dried under high vacuum to obtain an isolated yield of

313.6 mg (73%). After spectroscopic characterization, the dianionic TBA_2[**13**]$^{2-}$ was dissolved in 5 mL 90/10 ethanol/acetonitrile, 0.3 g (1.11 mmol) $FeCl_3 \cdot 6H_2O$ was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and a red-purple band containing [**13**]$^{1-}$ was separated from the $FeCl_3 \cdot 6H_2O$ through a slurry-packed silica gel column with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation and the final isolated radical [**13**]$^{1-}$ was dried under high vacuum to obtain an isolated yield of 226.4 mg (56%). Compound [**13**]$^{1-}$ is a red-purple solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 – 8.74 (m, 36H, C_6H_3), 3.13 (m, 8H, N-$\underline{CH_2}$), 1.65 (m, 8H, N-$CH_2\underline{CH_2}$), 1.47 (m, 8H, N-$(CH_2)_2\underline{CH_2}$), 1.05 (m, 12H, N-$(CH_2)_3\underline{CH_3}$). *Note: The CH_2 signal for the cluster is masked and all other peaks are quite broad due to the paramagnetic radical state of the molecule.* ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 189.0, 132.7, 132.5, 129.4, 124.6, 122.5, 121.0, 68.0, 59.2, 59.2, 59.1, 30.9, 25.6, 23.8, 19.7, 19.7, 13.4. No resonances are visible by ^{11}B NMR, due to paramagnetic broadening. ^{19}F NMR (376 MHz, $CDCl_3$): δ -63.22 (s, 72F). HRMS (Orbitrap): *m/z* calculated for $C_{108}H_{60}B_{12}F_{72}O_{12}$ (M^-), 3047.4158 Da; found, 1523.7080 (z=2) Da. Crystallized from $CDCl_3$ and pentane at room temperature for 1 week to obtain a single crystal for X-ray diffraction analysis.

Benzyloxy undeca(ethoxy)-*hypercloso*-dodecaborane [14]

TBA_2[**1**] (100.0 mg, 0.122 mmol) was added to a 10 mL glass microwave vial and transferred out of a nitrogen filled glovebox, opened to the air, and dissolved in 2 mL acetonitrile. *N*,*N*-diisopropylethylamine (0.4 mL, 2.30 mmol), benzyl bromide (0.0204 g, 0.119 mmol) and bromoethane (0.8055g, 7.39 mmol) were added along with a stir bar, the vial was sealed with a PTFE/silicone cap, and the mixture was heated at 140 °C with stirring in the microwave for 30 min. The volatiles and excess bromoethane were removed *via* rotary evaporation, the remaining charged 1-/2- product mixture was dissolved in 5 mL 90/10 ethanol/acetonitrile, $FeCl_3 \cdot 6H_2O$ (0.3

g, 1.11 mmol) was added and the mixture was left to stir for 12-24 h. Following oxidation, the solvent mixture was removed *via* rotary evaporation, and the product mixture was separated from the $FeCl_3 \cdot 6H_2O$ with CH_2Cl_2. The CH_2Cl_2 was removed *via* rotary evaporation, and the product mixture was loaded onto a long (30 – 35 cm) silica gel column slurry-packed with 80/20 CH_2Cl_2/hexanes, and the products were separated by eluting fractions with 80/20 CH_2Cl_2/hexanes. The first orange band eluted contained randomly di-substituted $benzyl_2ethyl_{10}$ species, followed by an orange band with the neutral closomer **14**, with extra **4** in a third yellow-orange band eluting last. *Note: The fractions overlap, and due to the similar colors of the different products, thin layer chromatography (TLC) with 80/20 CH_2Cl_2/hexanes was performed on the fractions near the band edges to determine which fractions contained a mixture of products.* The fractions containing only a single species according to TLC that eluted after the di-substituted product and prior to the pure **4** bands were combined. The volatiles were removed *via* rotary evaporation, and the final neutral product **14** was dried under high vacuum to obtain an isolated yield of 15.7 mg (18%). Compound **14** is a yellow-orange solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 (m, 5H, C_6H_5), 5.07 (m, 2H, $\underline{CH_2}C_6H_5$), 4.10 (m, 22H, $\underline{CH_2}CH_3$), 1.22 (m, 33H, $CH_2\underline{CH_3}$).^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 141.2, 128.0, 126.6, 71.6, 66.8, 17.7. ^{11}B NMR (128 MHz, $CDCl_3$): δ 39.0, 35.2. HRMS (DART): *m/z* calculated for $C_{29}H_{62}B_{12}O_{12}$ (M^-), 732.5431 Da; found, 732.5464 Da.

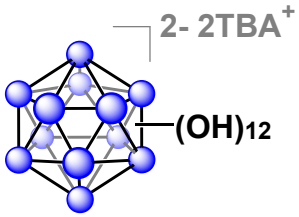
References

1 I. S. Zavarine and C. P. Kubiak, *J. Electroanal. Chem.*, 2001, **495**, 106–109.

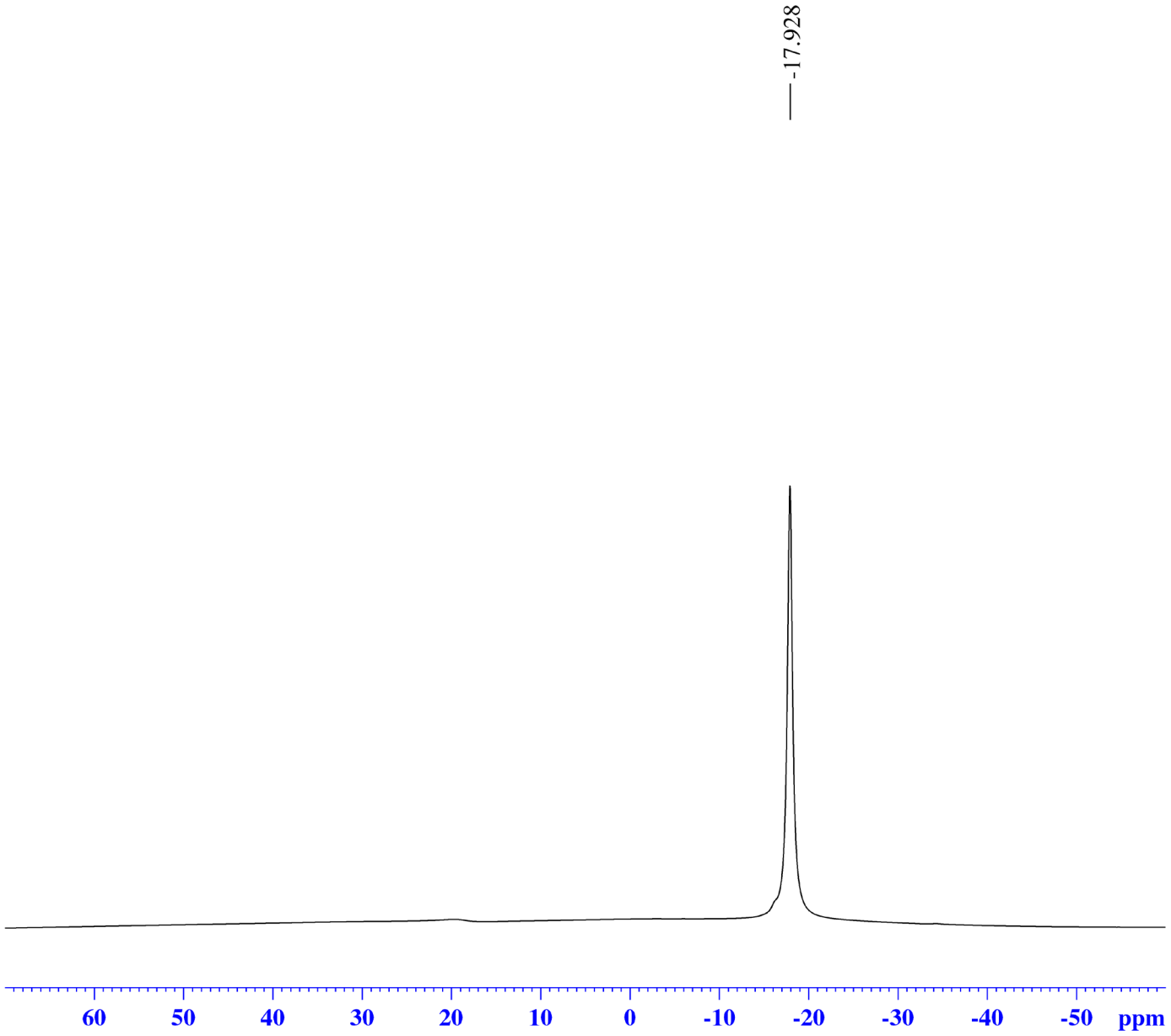
2 C. W. Machan, M. D. Sampson, S. A. Chabolla, T. Dang and C. P. Kubiak, *Organometallics*, 2014, **33**, 4550–4559.

3 T. Peymann, C. B. Knobler, S. I. Khan and M. F. Hawthorne, *J. Am. Chem. Soc.*, 2001, **123**, 2182–2185.

4 M. J. Bayer and M. F. Hawthorne, *Inorg. Chem.*, 2004, **43**, 2018–2020.



^{11}B {^{1}H} NMR



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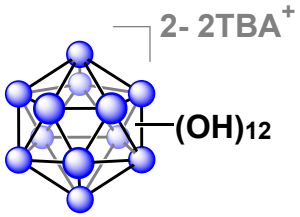

^{1}H NMR

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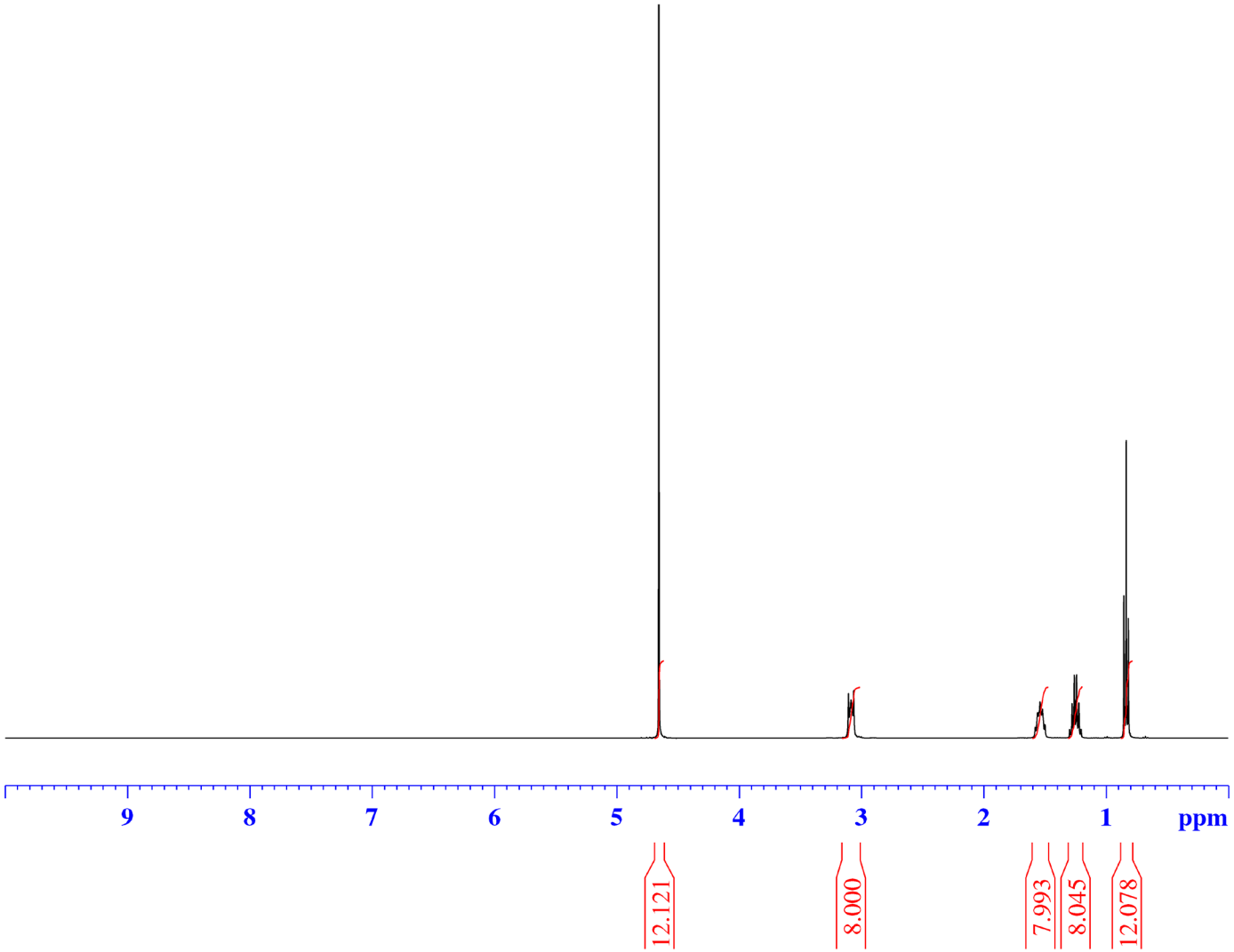

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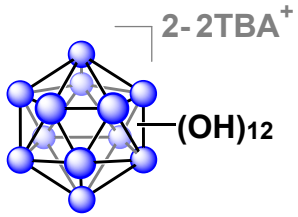

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Intensity/ a.u.

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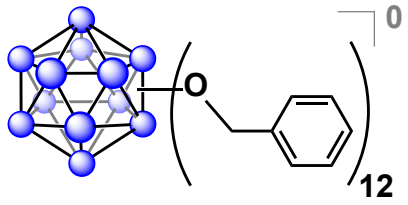
Binding energy/ eV



^{11}B {^{1}H} NMR

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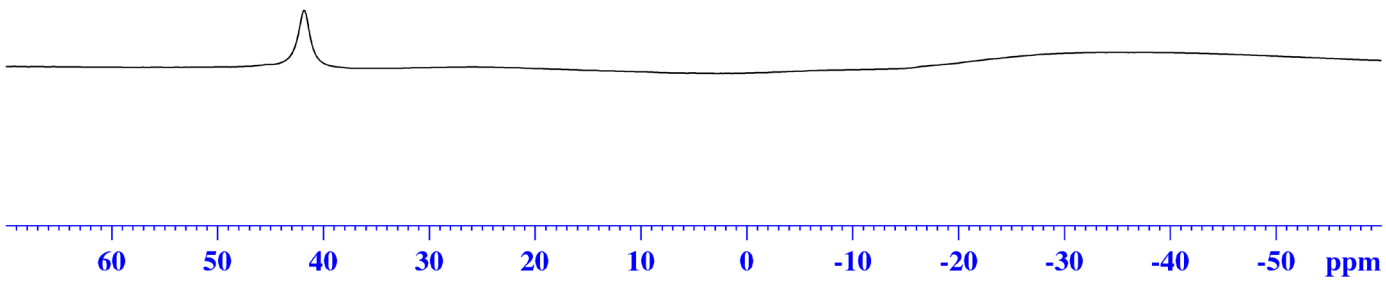

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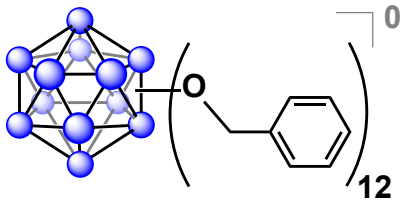
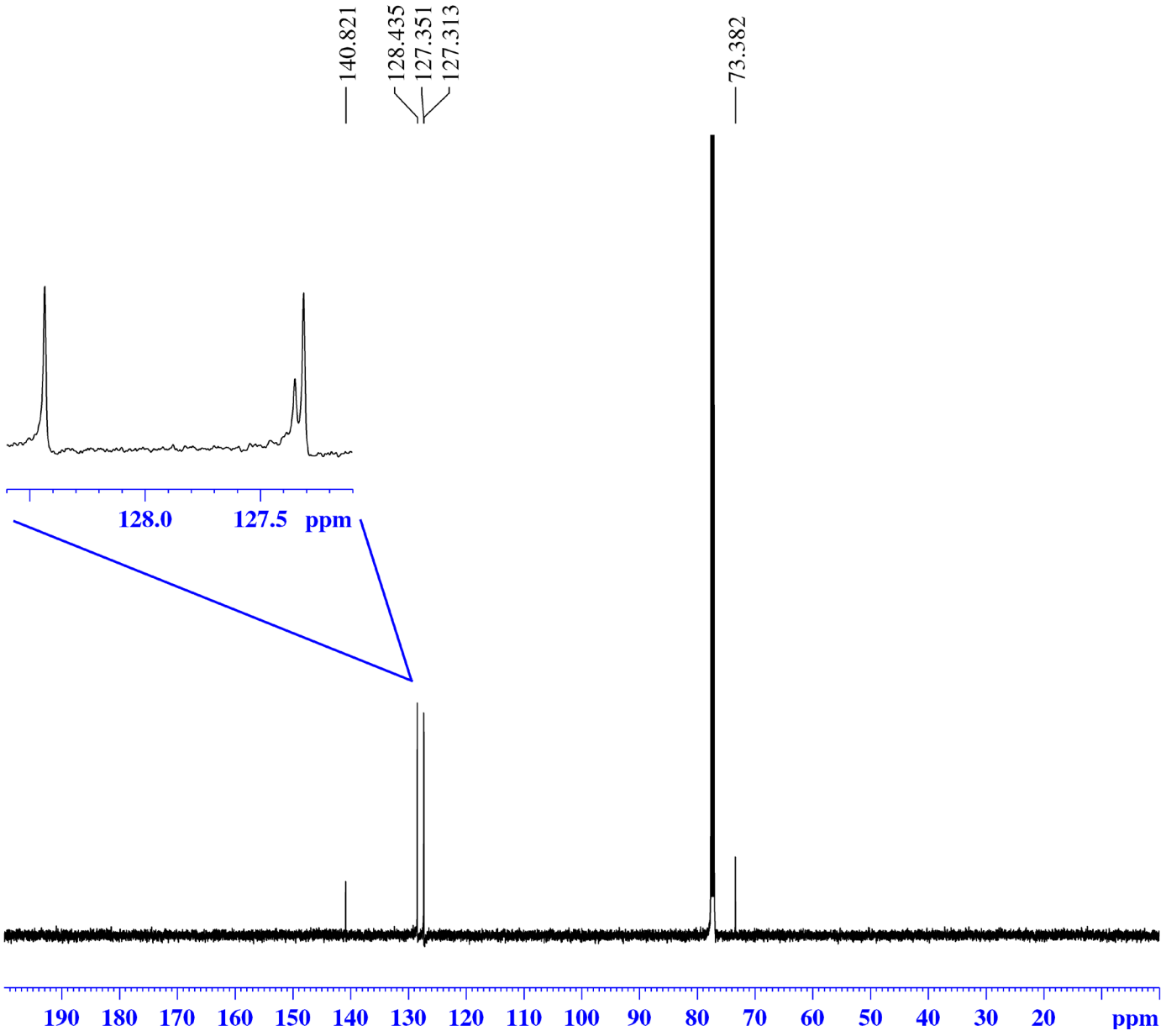


^{13}C NMR



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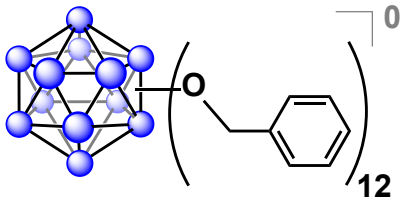
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

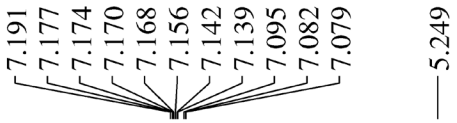
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

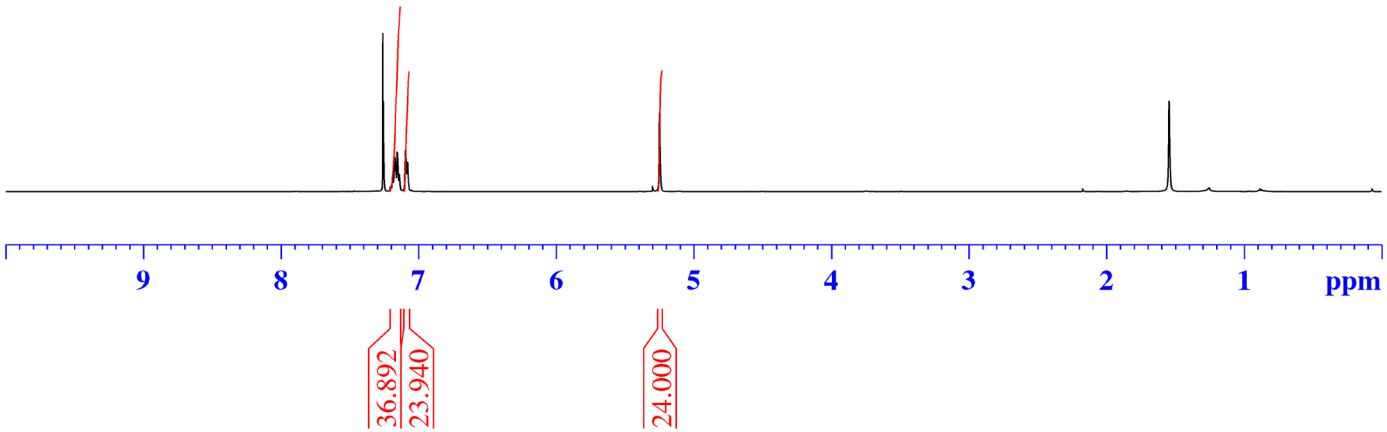
======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7574315 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR



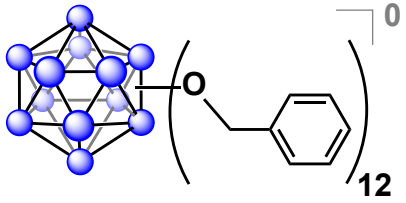


Current Data Parameters
NAME B12(O-Bn)12
EXPNO 101
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150407
Time 0.09
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CH3OH+D2O
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 63.27
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

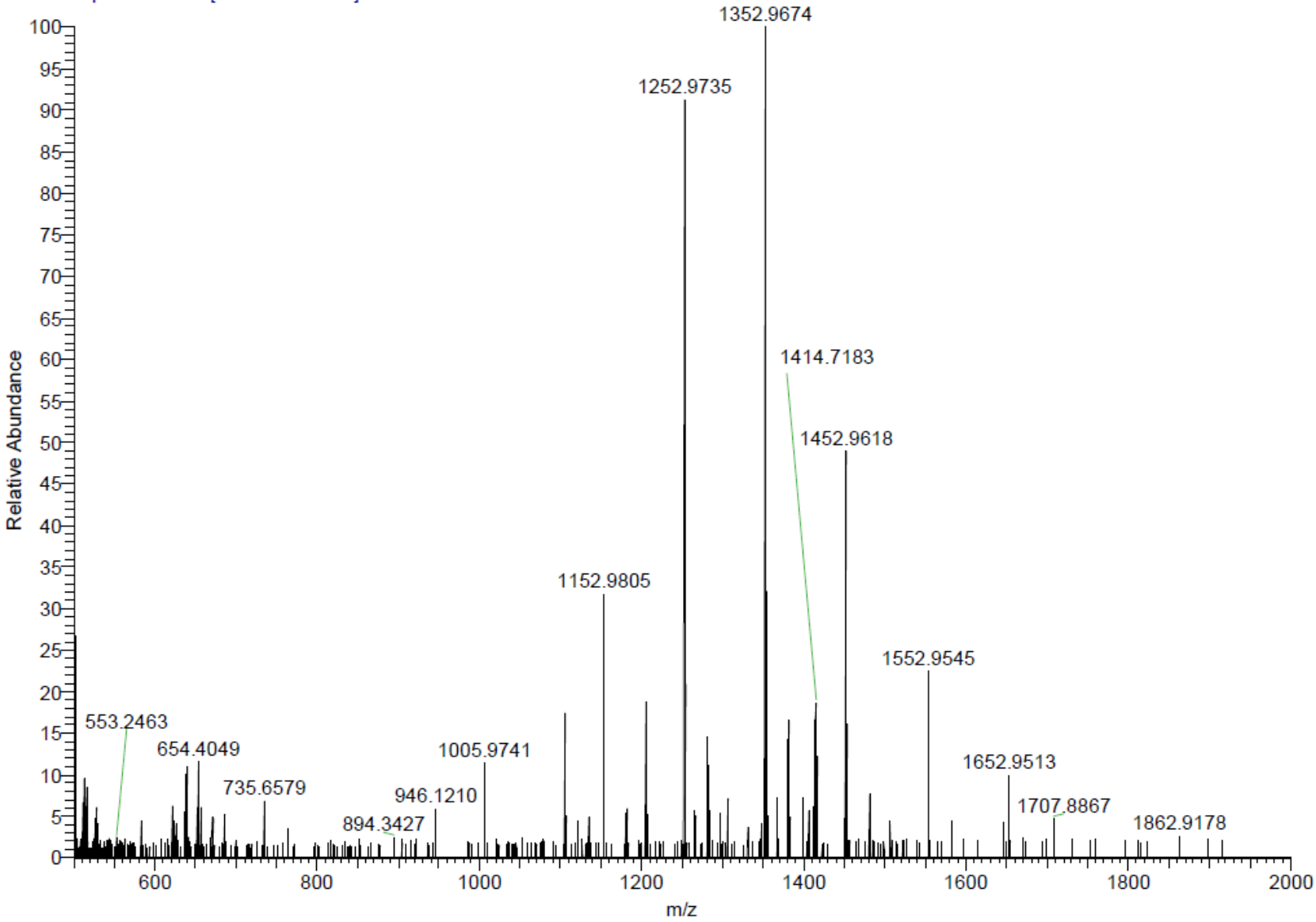
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

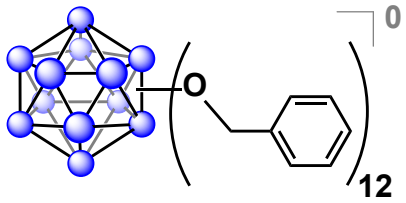
F2 - Processing parameters
SI 65536
SF 500.1287429 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



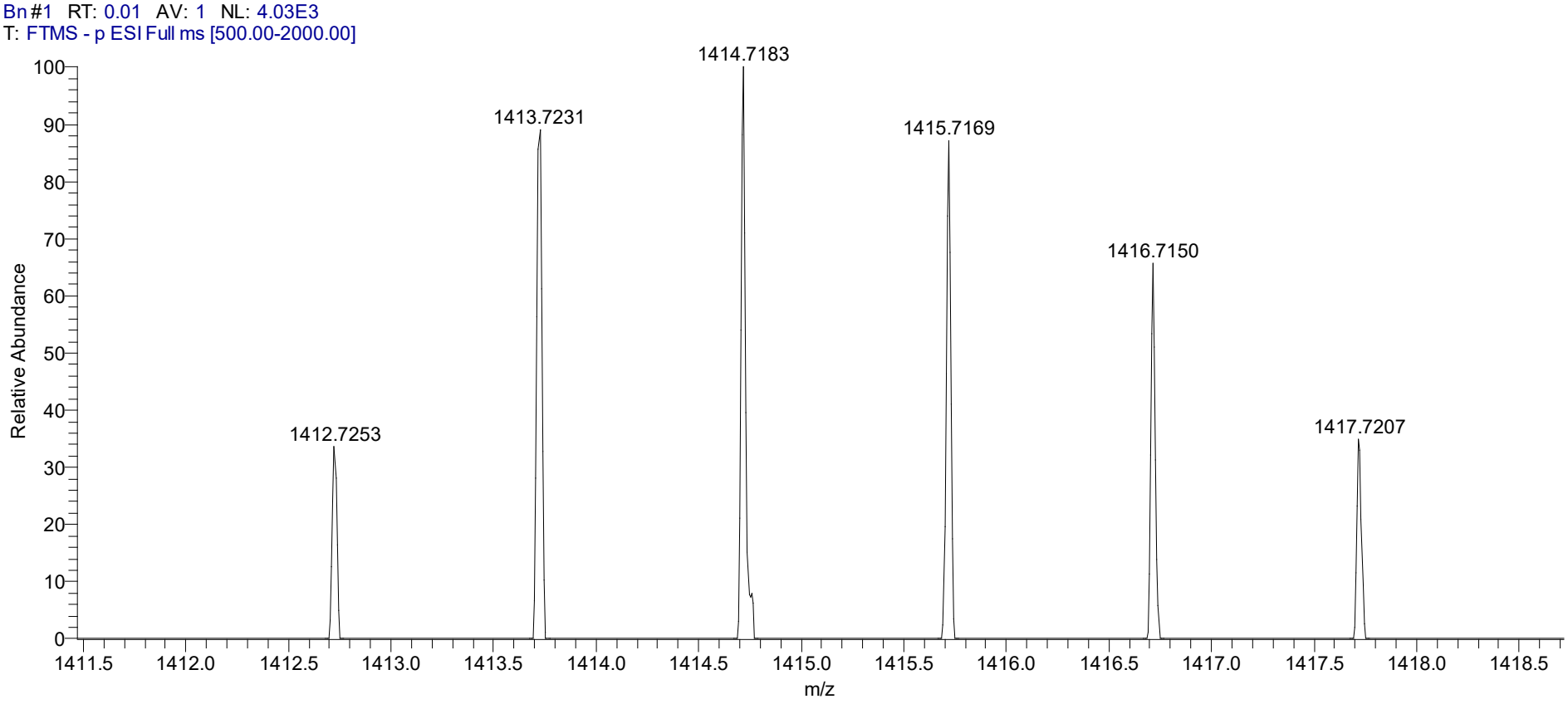
Q Exactive
High-Res Mass Spec

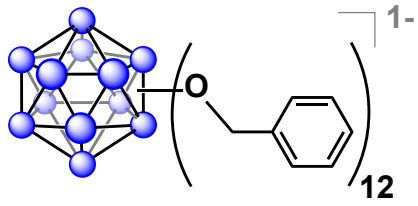
Bn #1 RT: 0.01 AV: 1 NL: 2.17E4
T: FTMS - p ESI Full ms [500.00-2000.00]



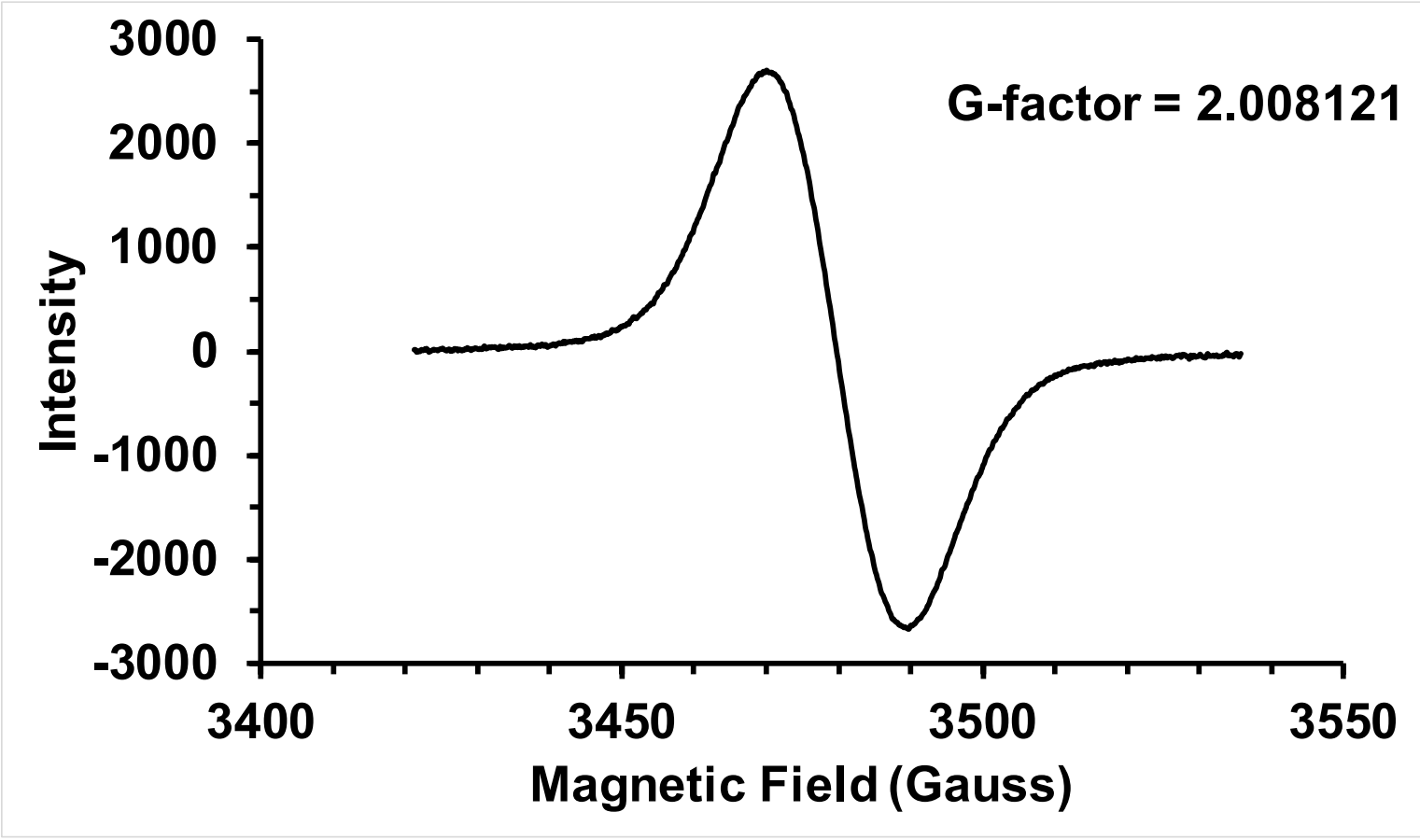


Q Exactive
High-Res Mass Spec





EPR



DOS	Format
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MIN	-2666.883789
MAX	2704.116211
JSS	0
GST	3421.274884
GSI	114.543004
JUN	G
JON	Bruker BioSpin GmbH
JDA	11/9/2015
JTM	14:44
JRE	c:\programfiles\bruker-emx\syscal\st0103.cal
JEX	field-sweep
JSD	1
HCF	3478.546386
HSW	114.543004
EMF	0
RCT	20.48
RTC	20.48
RRG	8.93E+03
RMA	4
MF	9.776766
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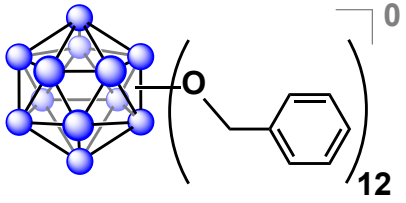
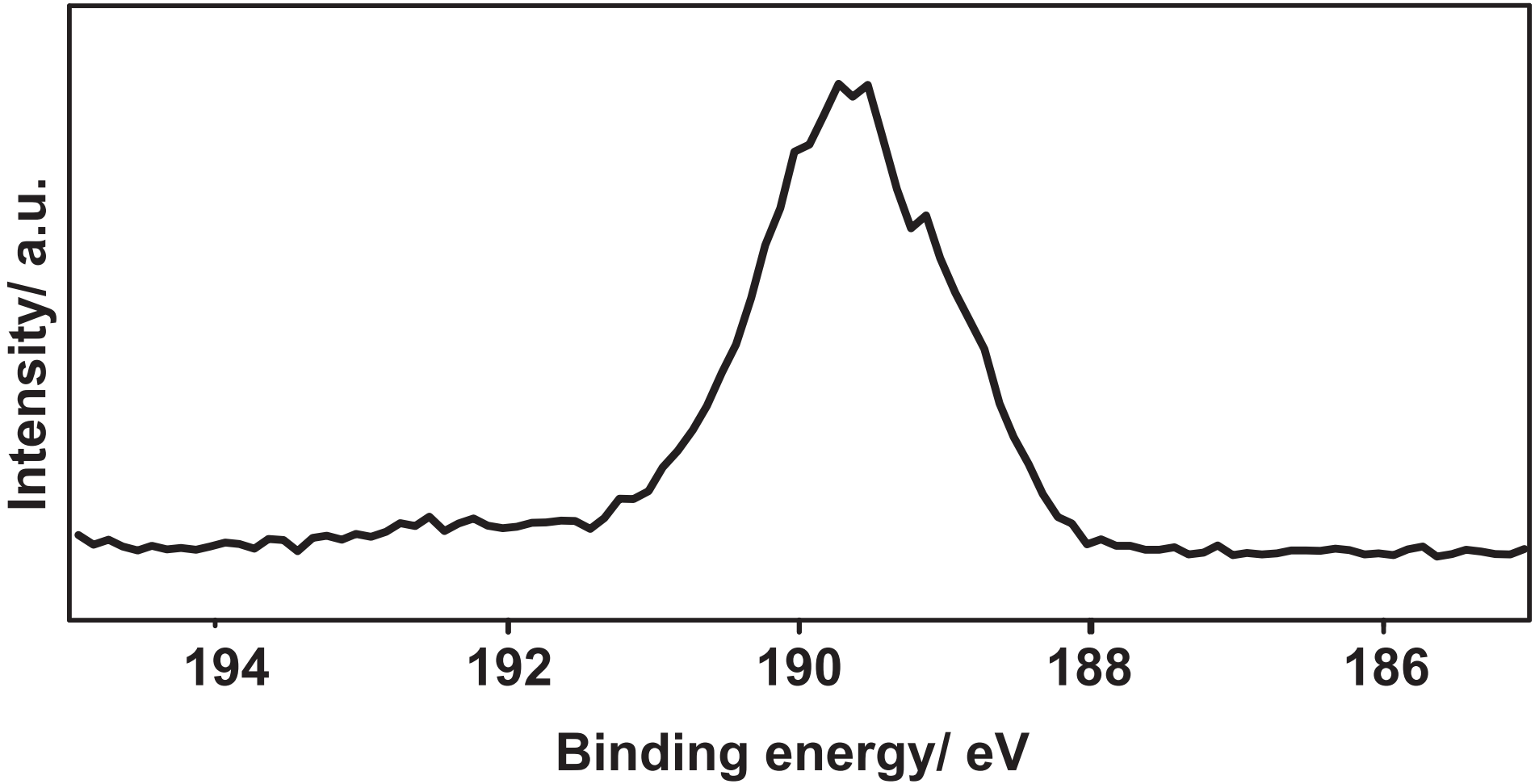
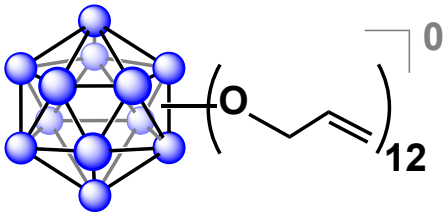
MPD	25



B 1s XPS

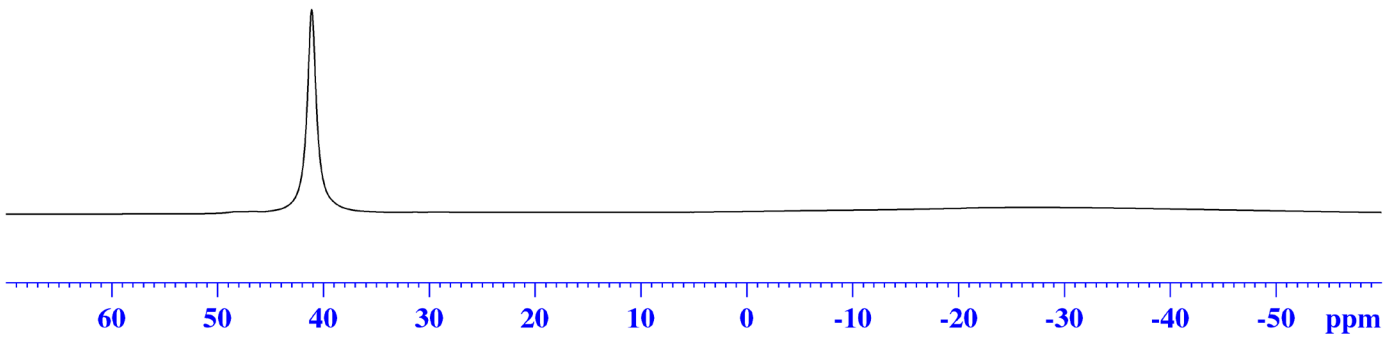
BRUKER





^{11}B {^{1}H} NMR

41.093



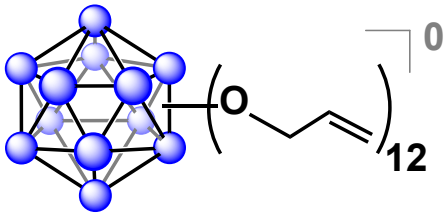
Current Data Parameters
NAME B12(O-Allyl)12
EXPNO 130
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150221
Time 17.36
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.0 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

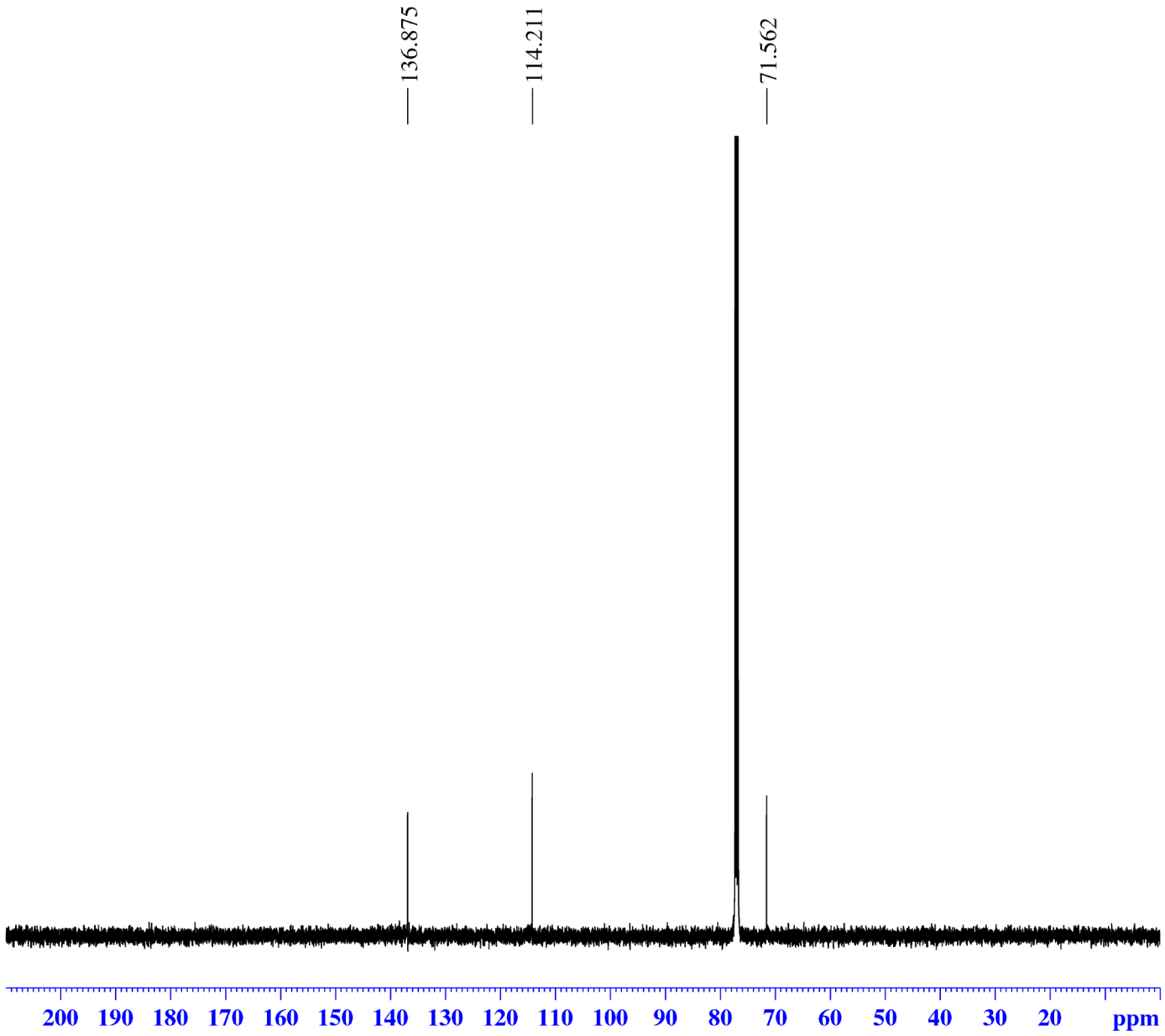
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40



^{13}C NMR



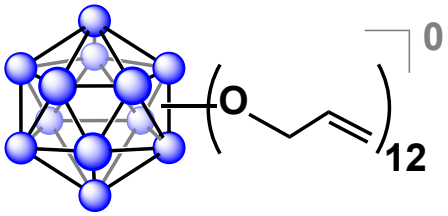
Current Data Parameters
NAME B12(O-Allyl)12
EXPNO 80
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150406
Time 23.59
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

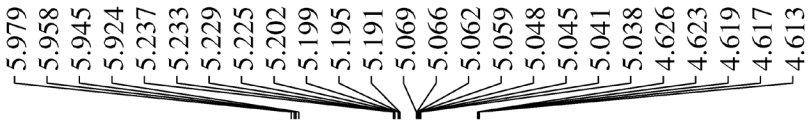
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

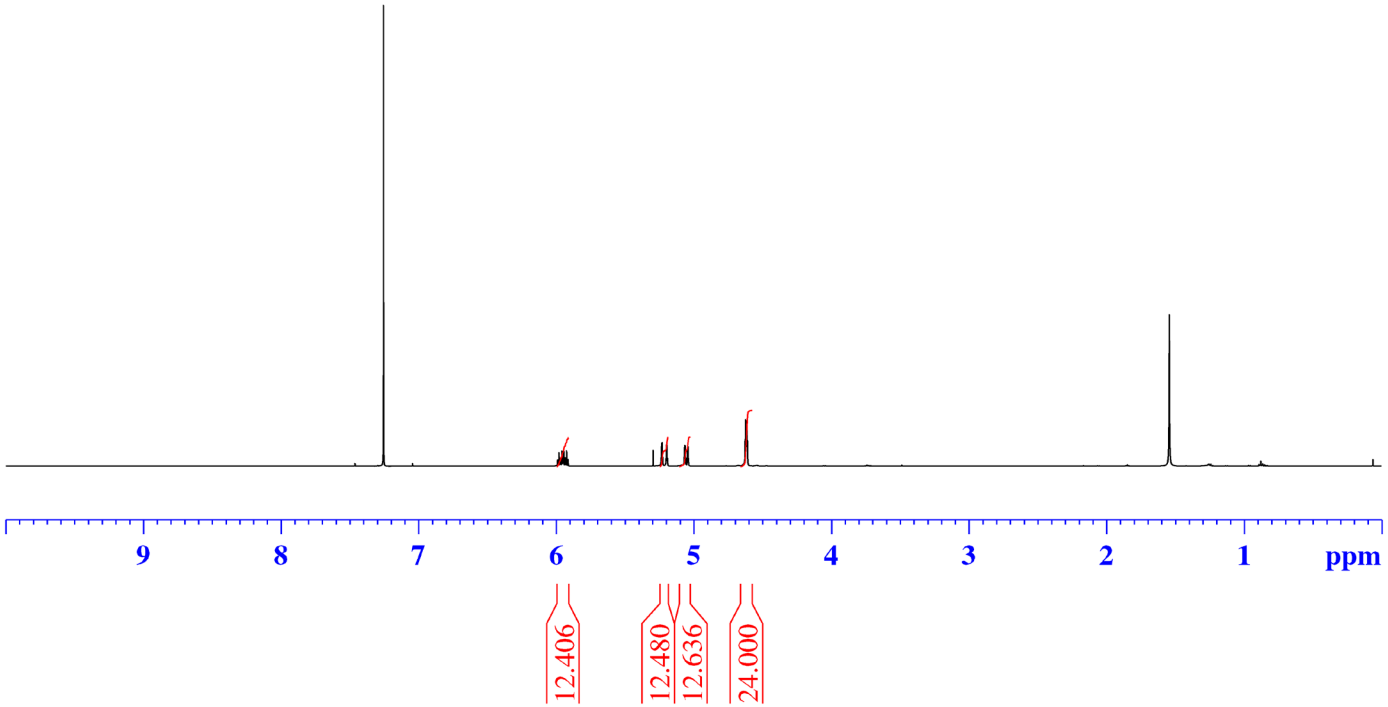
======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR



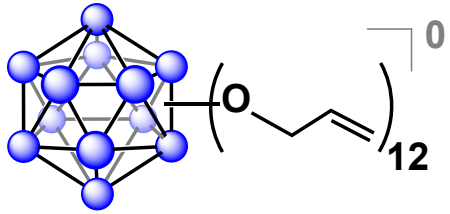


Current Data Parameters
NAME B12(O-Allyl)12
EXPNO 81
PROCNO 1

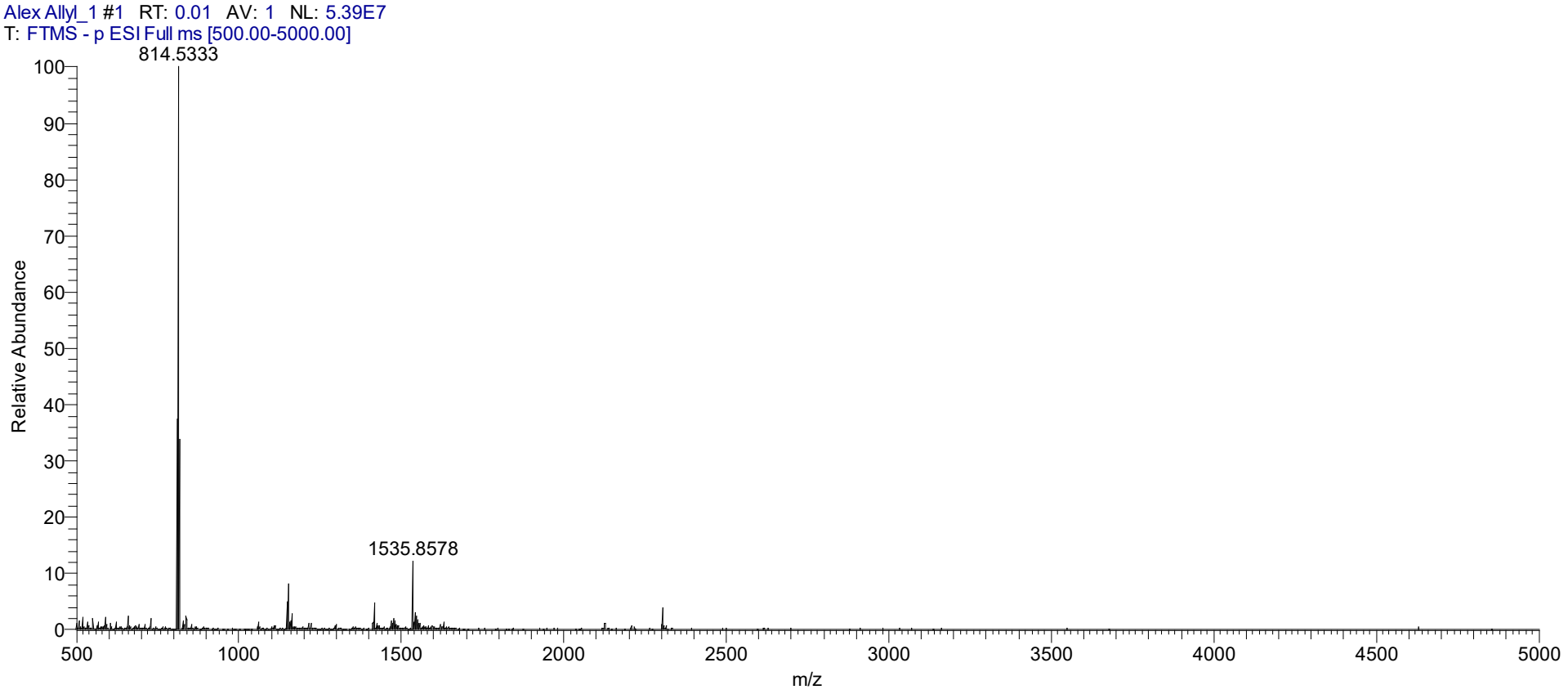
F2 - Acquisition Parameters
Date_ 20150407
Time 0.01
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 63.27
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

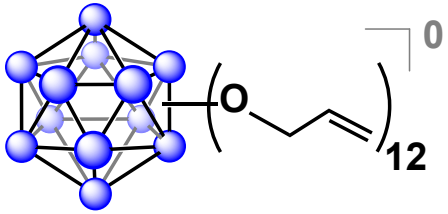
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

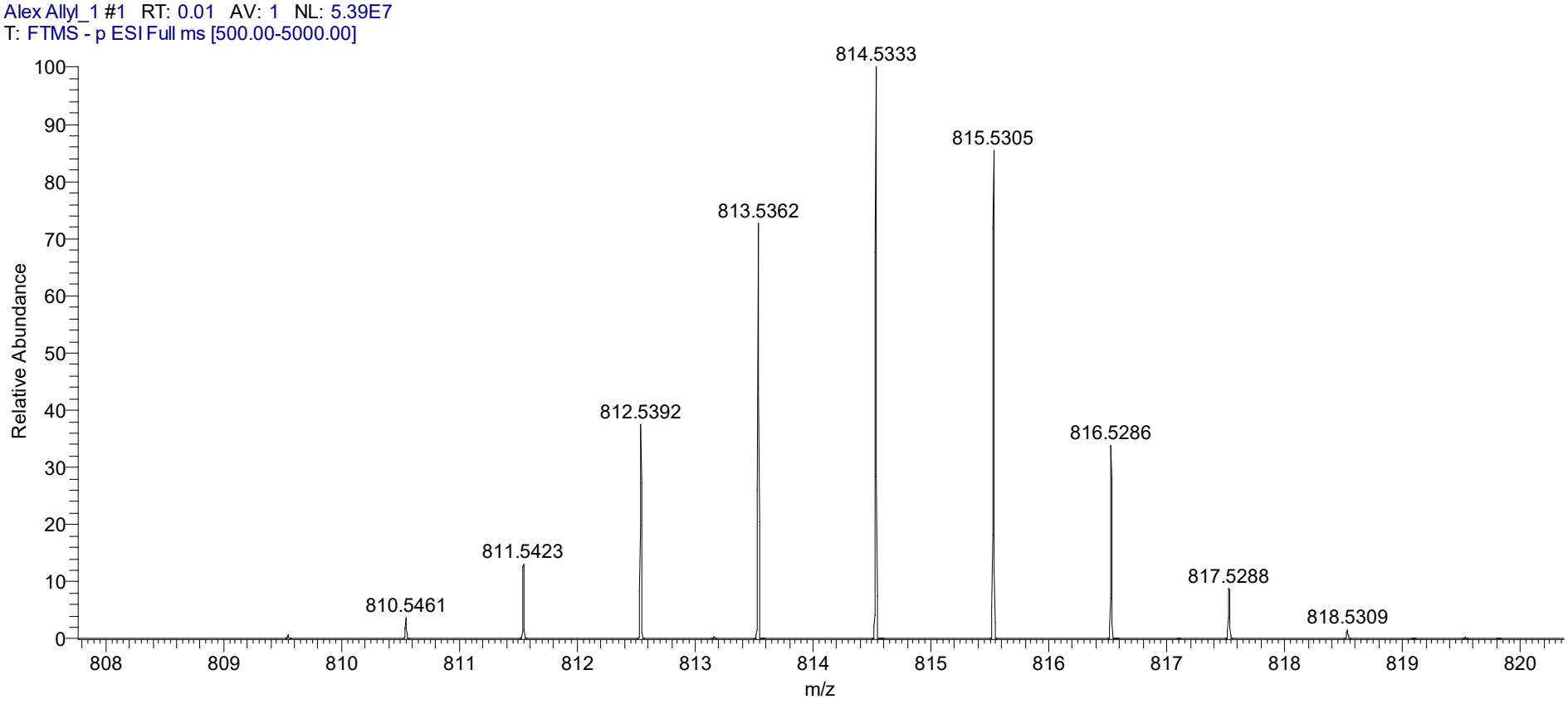


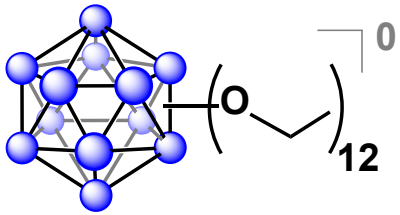
Q Exactive
High-Res Mass Spec





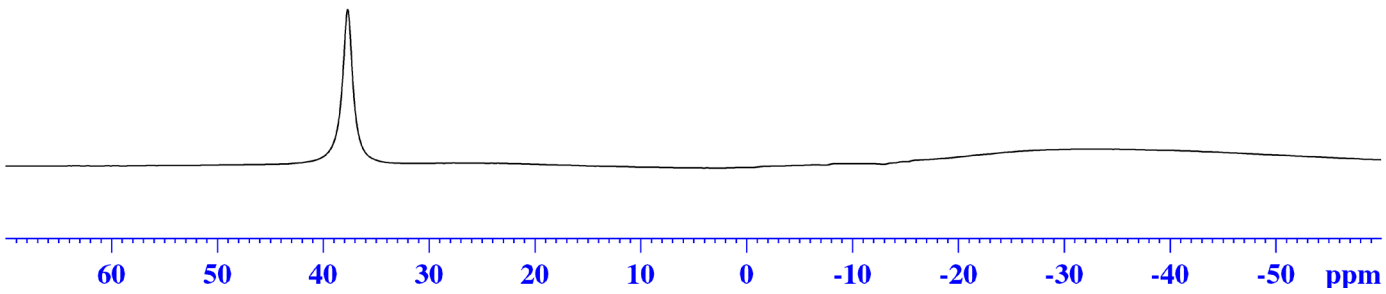
Q Exactive
High-Res Mass Spec





^{11}B {^{1}H} NMR

37.670



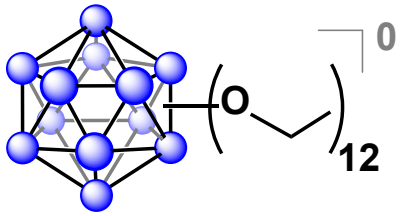
Current Data Parameters
NAME B12(O-Et)12
EXPNO 100
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150412
Time 19.11
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.1 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

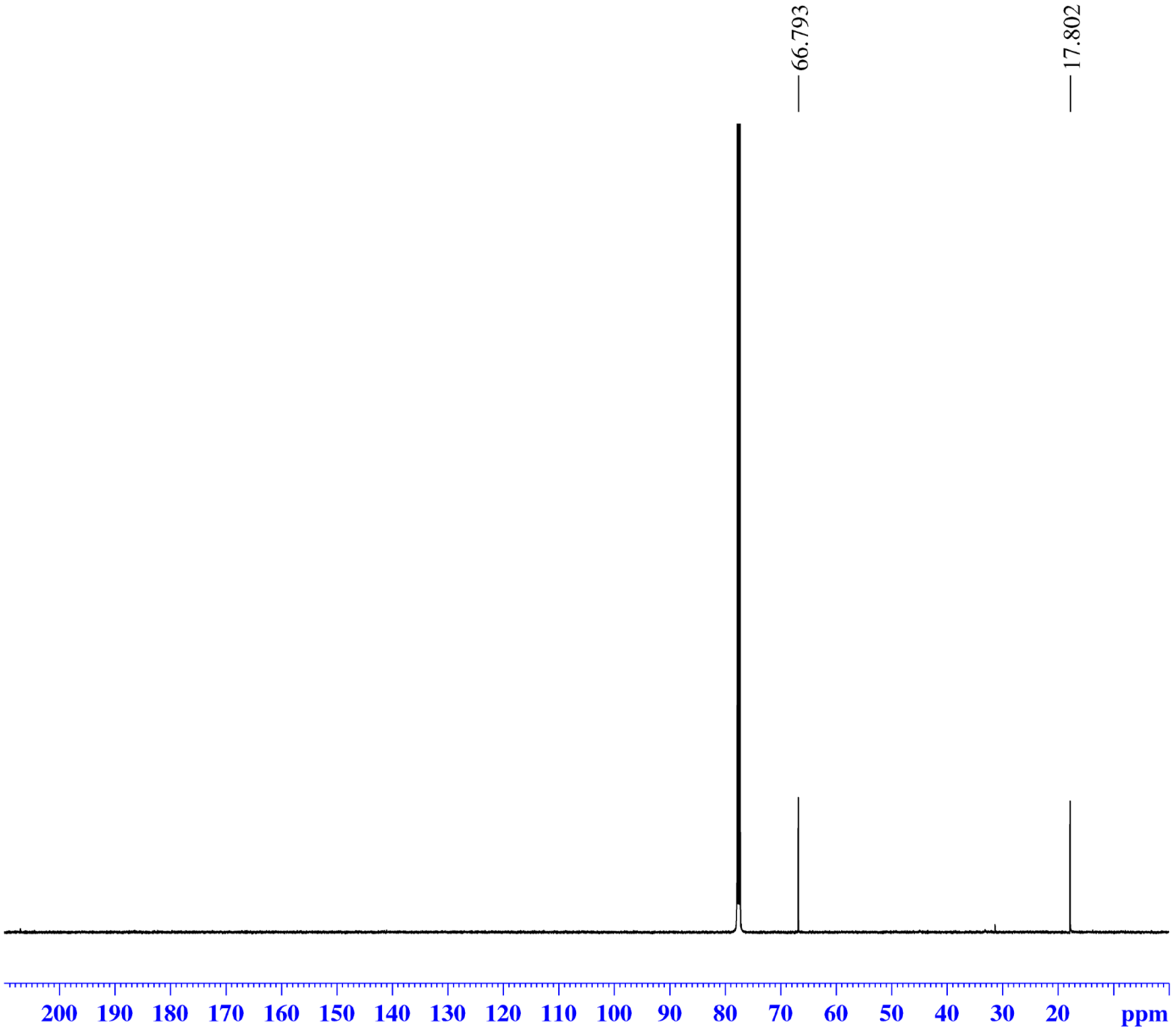
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40



^{13}C NMR



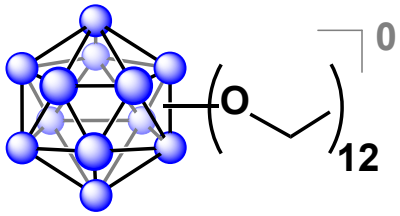
Current Data Parameters
NAME B12(O-Et)12
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150420
Time 21.23
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

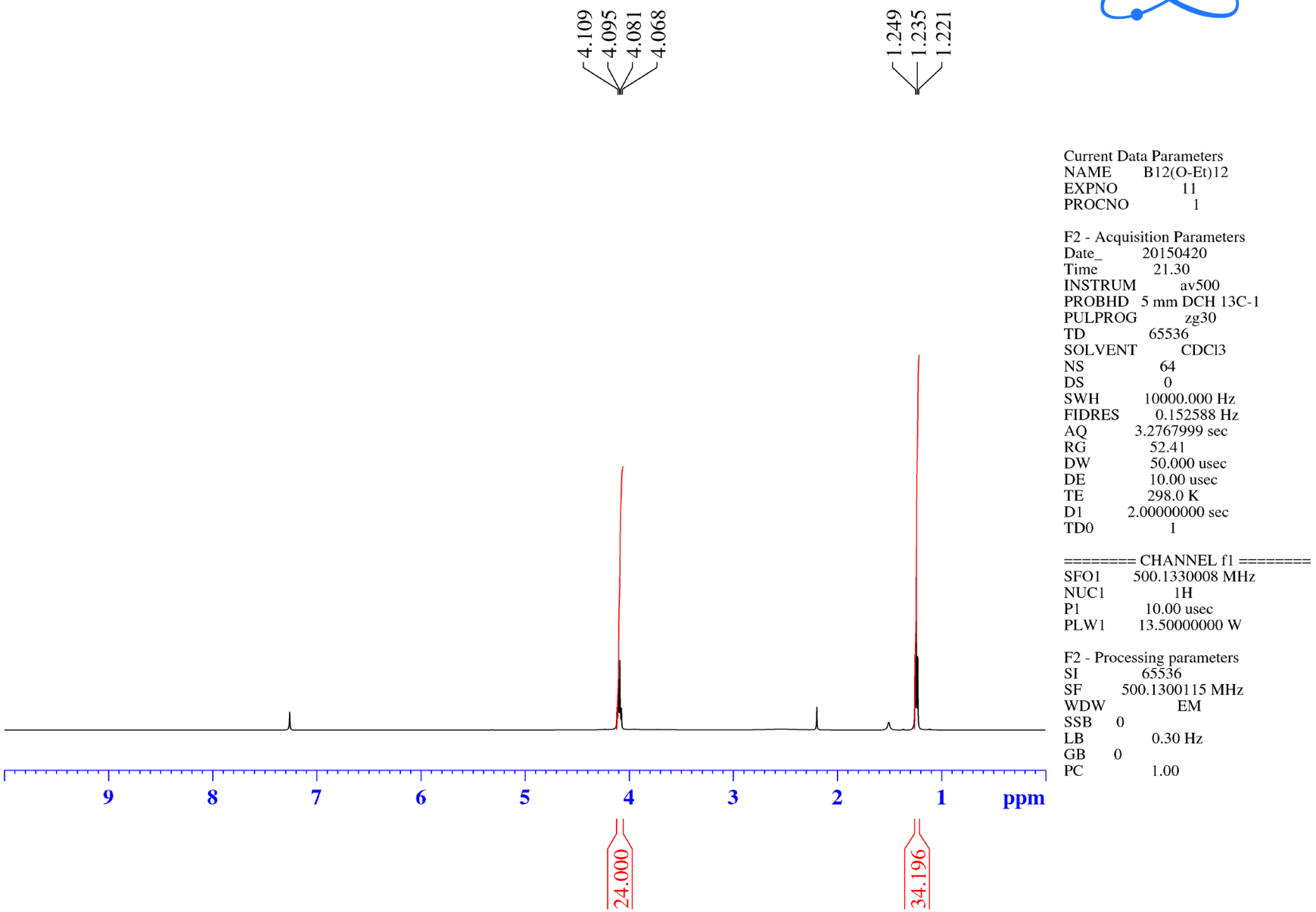
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR

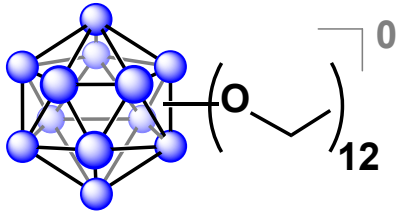


Current Data Parameters
NAME B12(O-Et)12
EXPNO 11
PROCNO 1

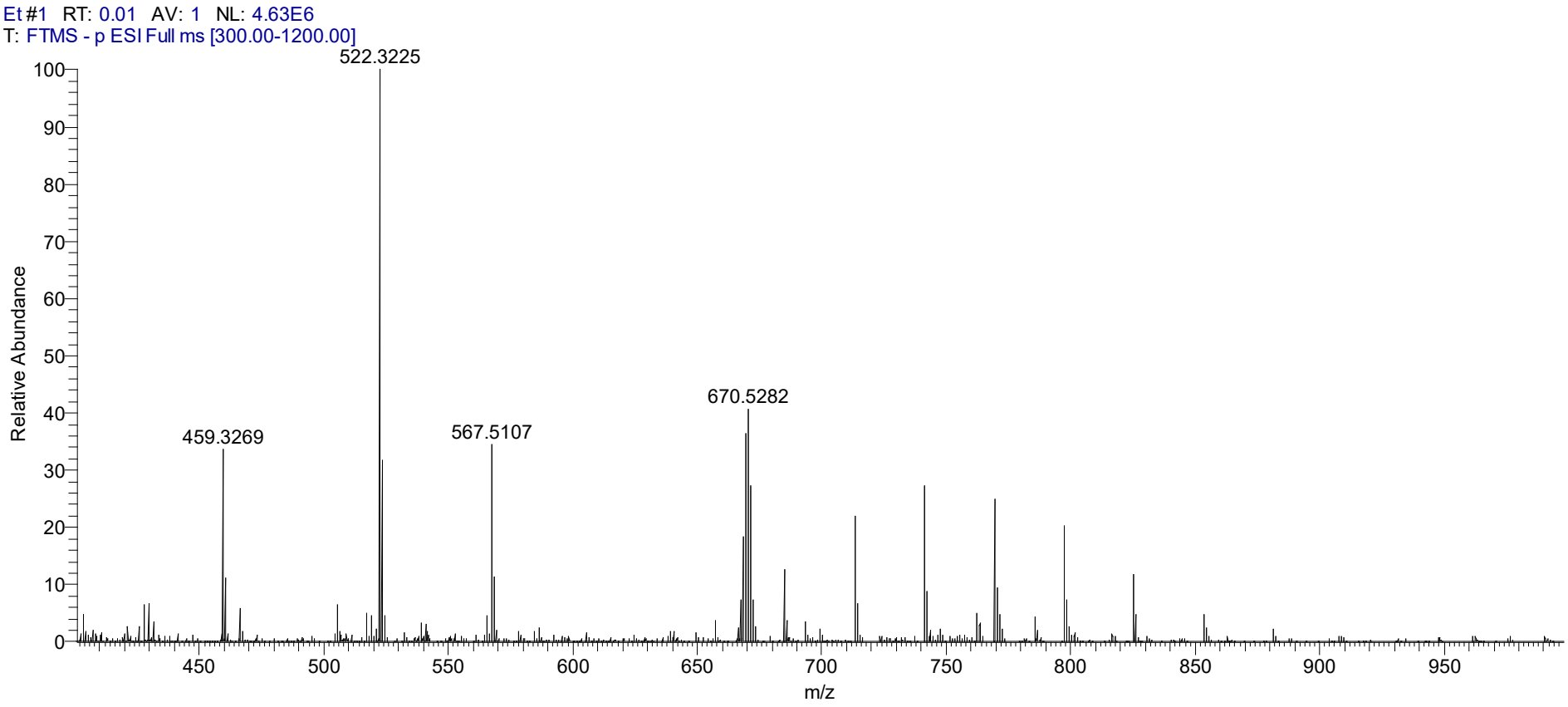
F2 - Acquisition Parameters
Date_ 20150420
Time 21.30
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 52.41
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

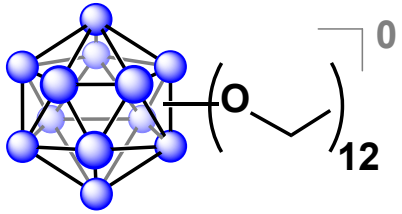
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

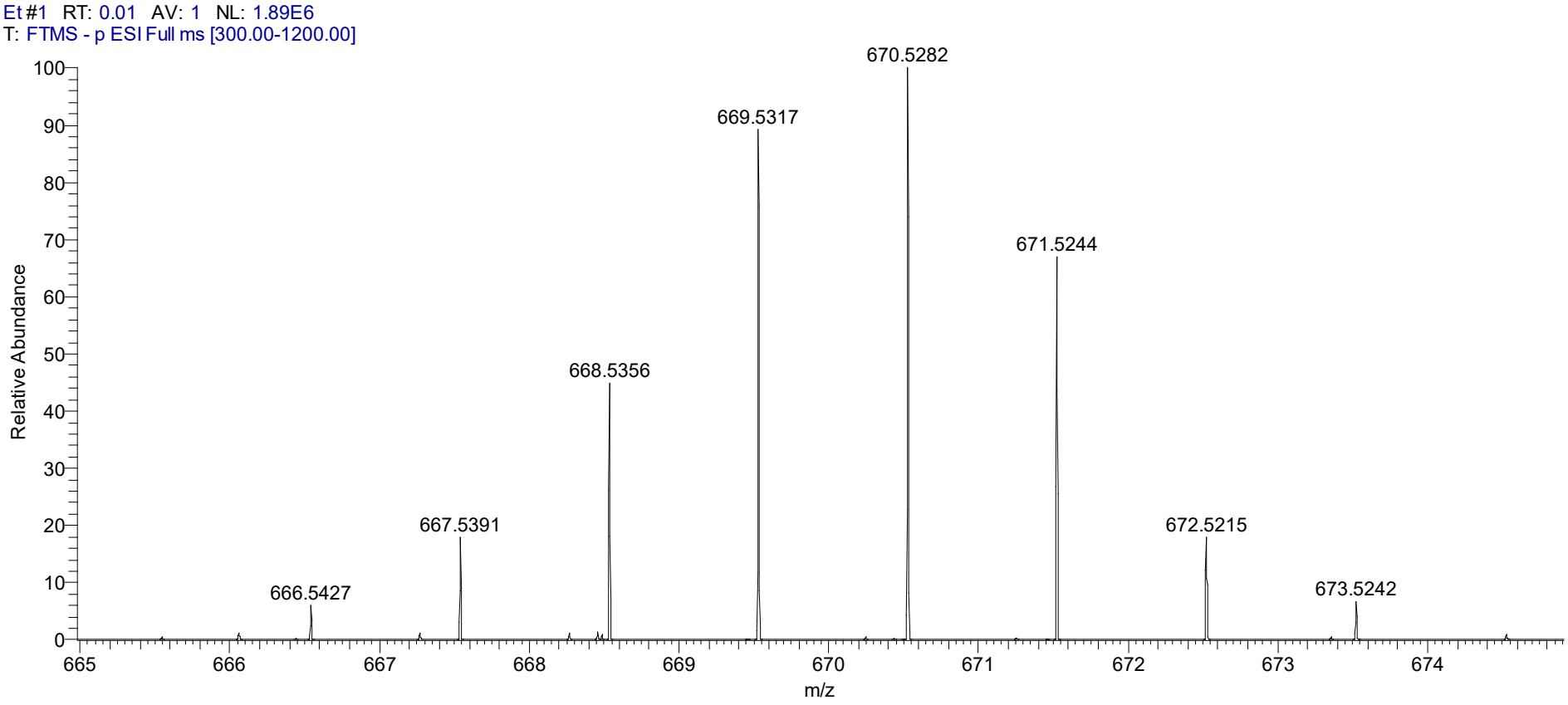


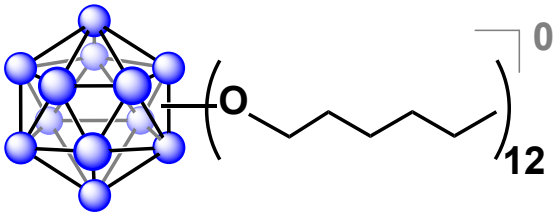
Q Exactive
High-Res Mass Spec





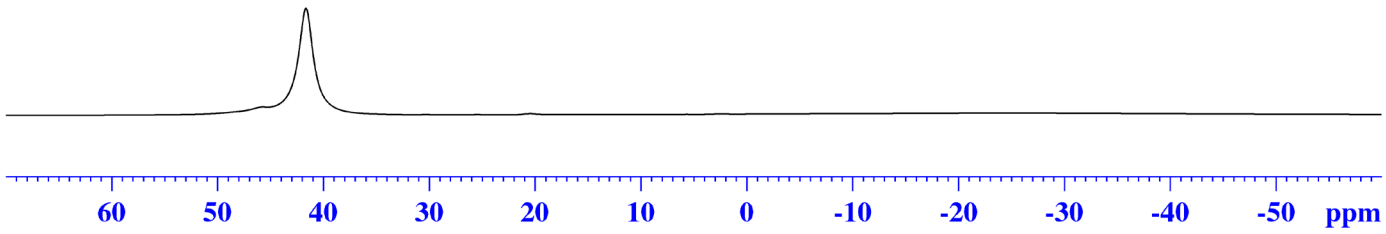
Q Exactive
High-Res Mass Spec





^{11}B {^{1}H} NMR

41.983



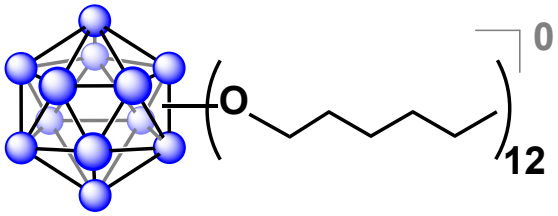
Current Data Parameters
NAME B12(O-Hexyl)12
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150610
Time 15.09
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.2 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

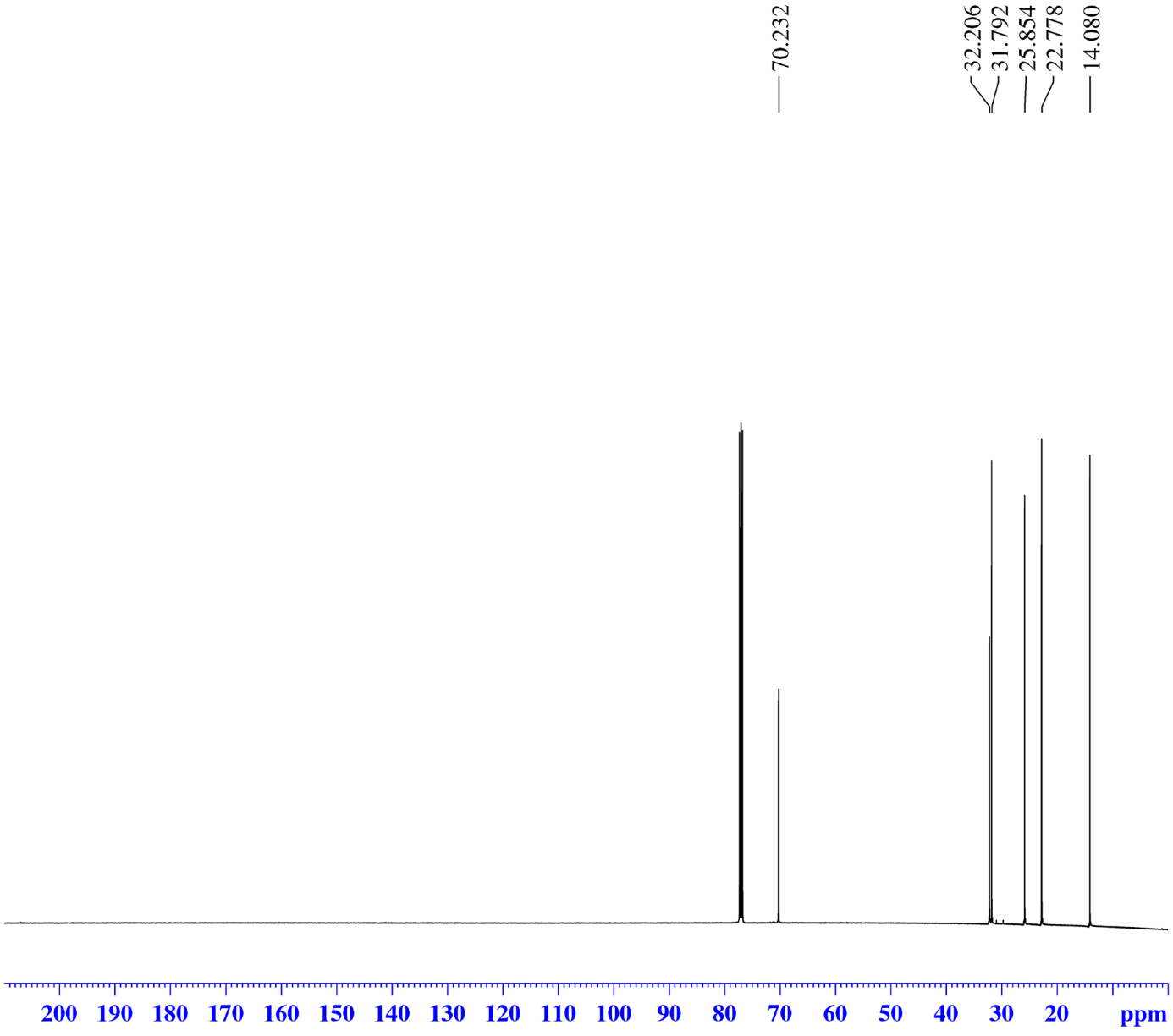
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40



^{13}C NMR



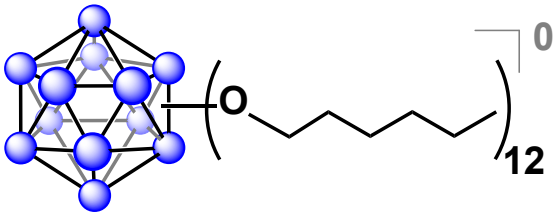
Current Data Parameters
NAME B12(O-Hexyl)12
EXPNO 200
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150829
Time 20.52
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

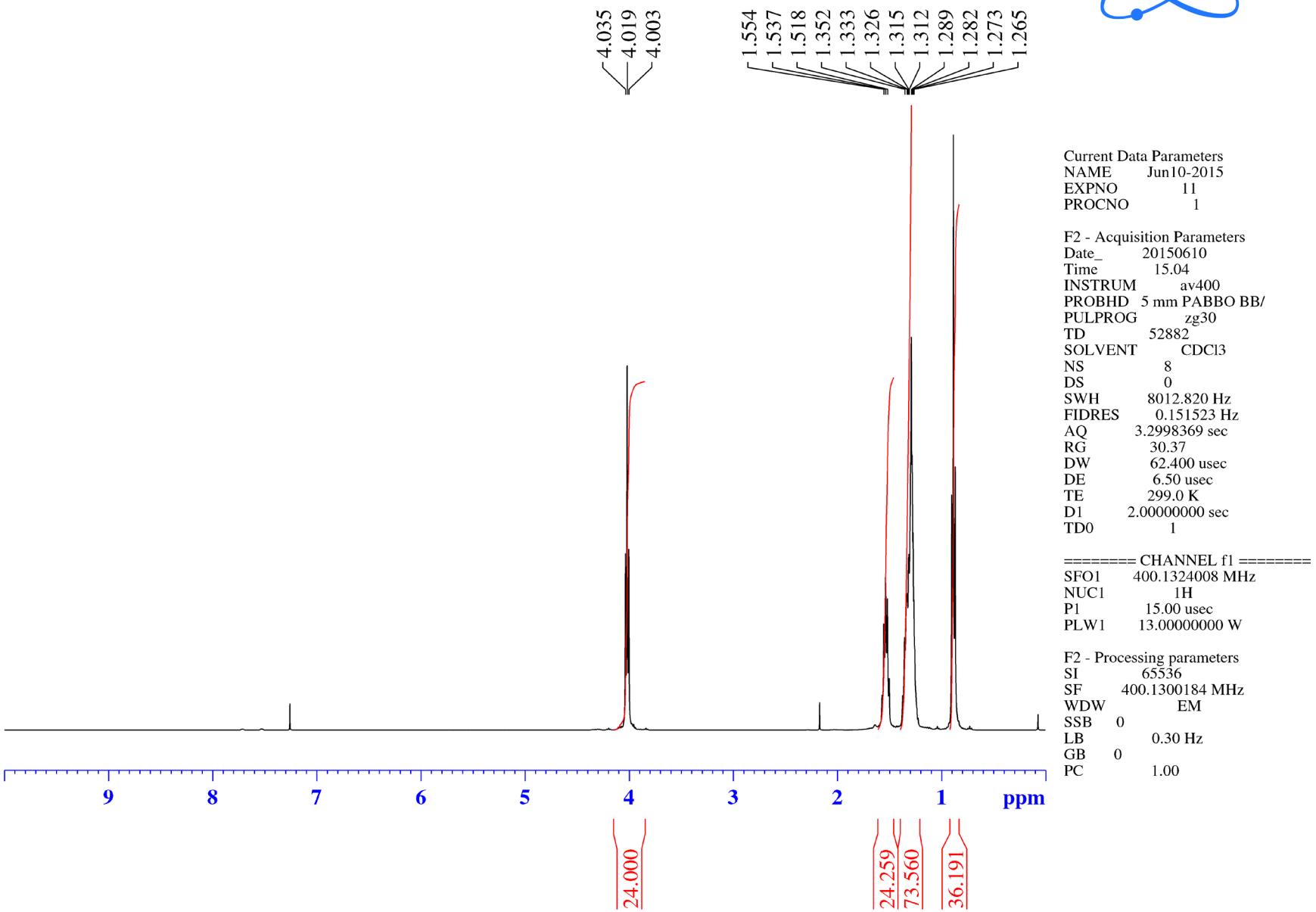
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR

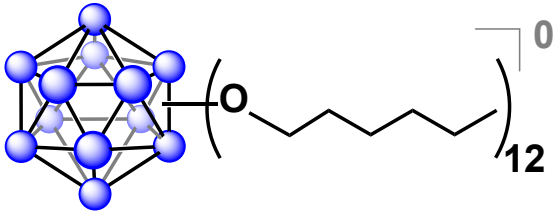


Current Data Parameters
NAME Jun10-2015
EXPNO 11
PROCNO 1

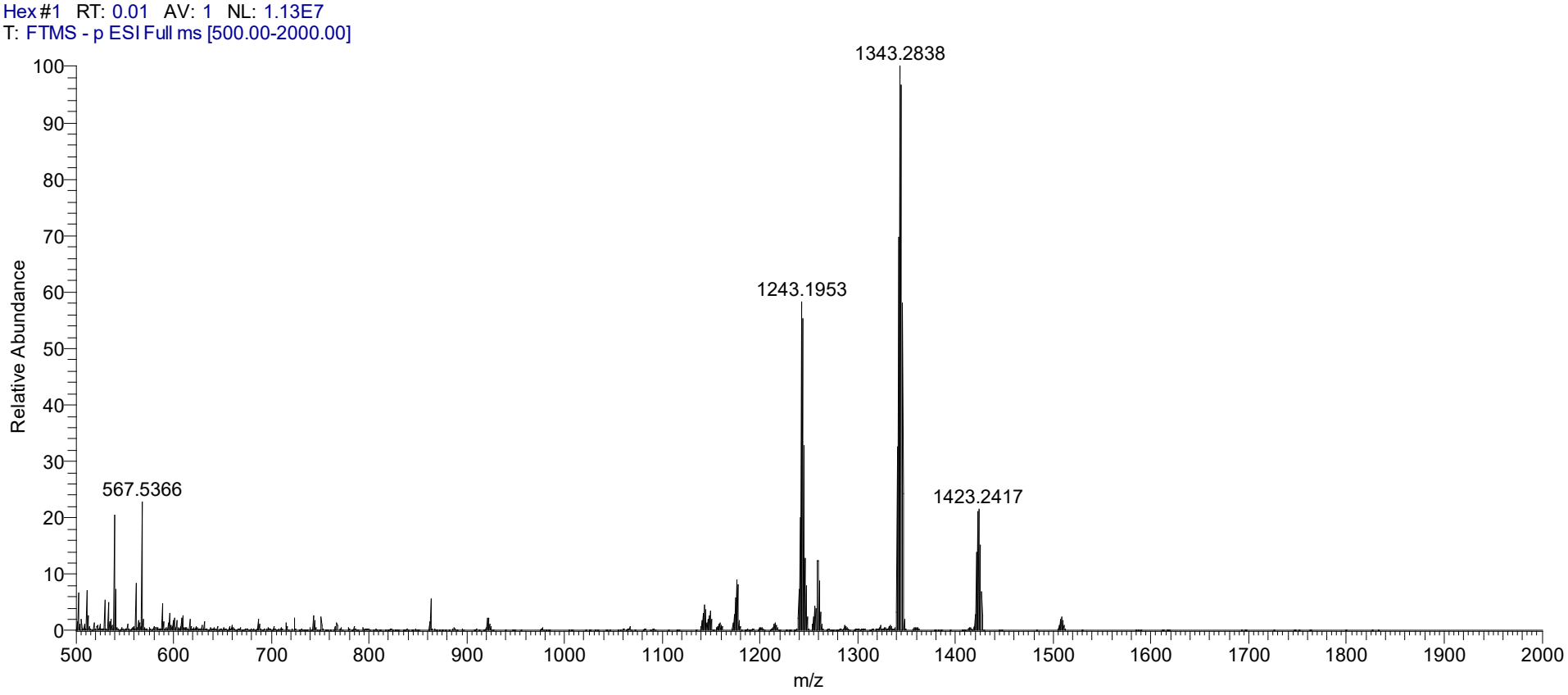
F2 - Acquisition Parameters
Date_ 20150610
Time 15.04
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 30.37
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

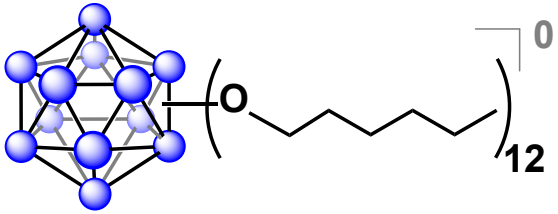
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300184 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



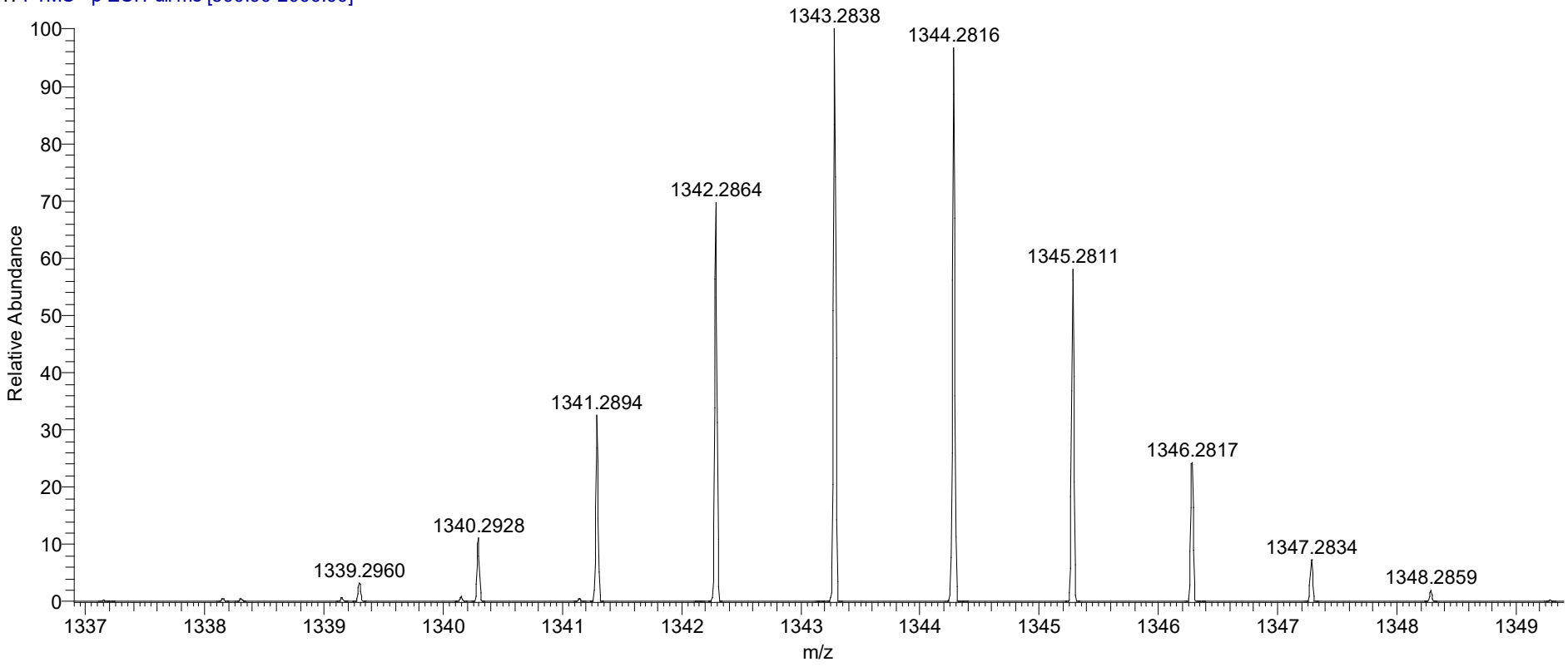
Q Exactive
High-Res Mass Spec

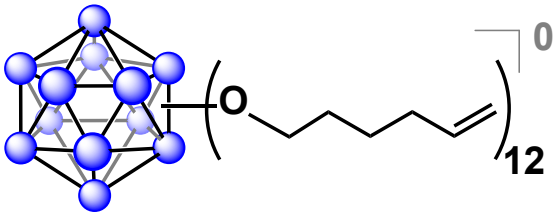




Q Exactive
High-Res Mass Spec

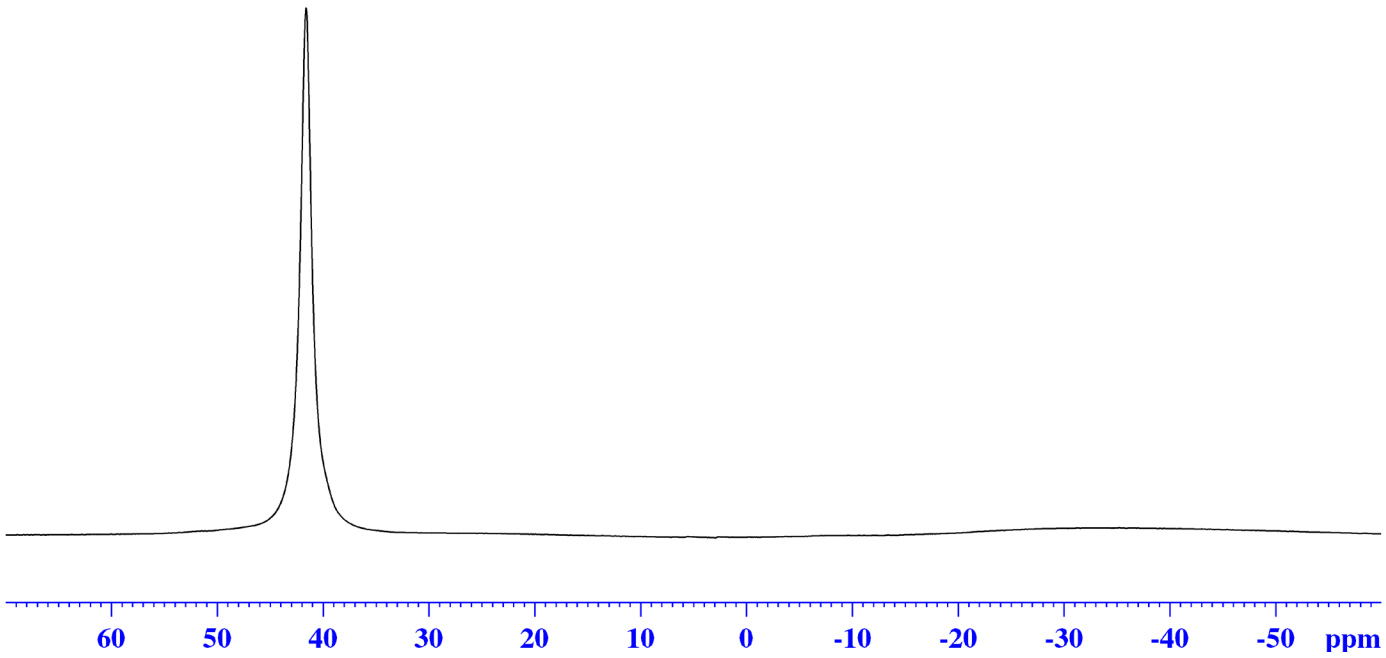
Hex #1 RT: 0.01 AV: 1 NL: 1.13E7
T: FTMS - p ESI Full ms [500.00-2000.00]





^{11}B {^{1}H} NMR

41.607



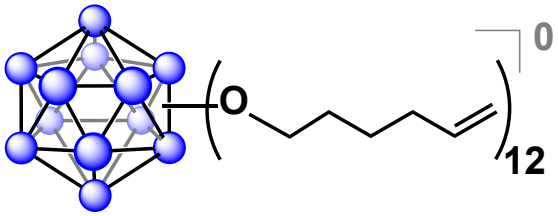
Current Data Parameters
NAME Oct05-2015
EXPNO 51
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151005
Time 15.01
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.1 K
D1 0.05000000 sec
D11 0.03000000 sec
TD0 1

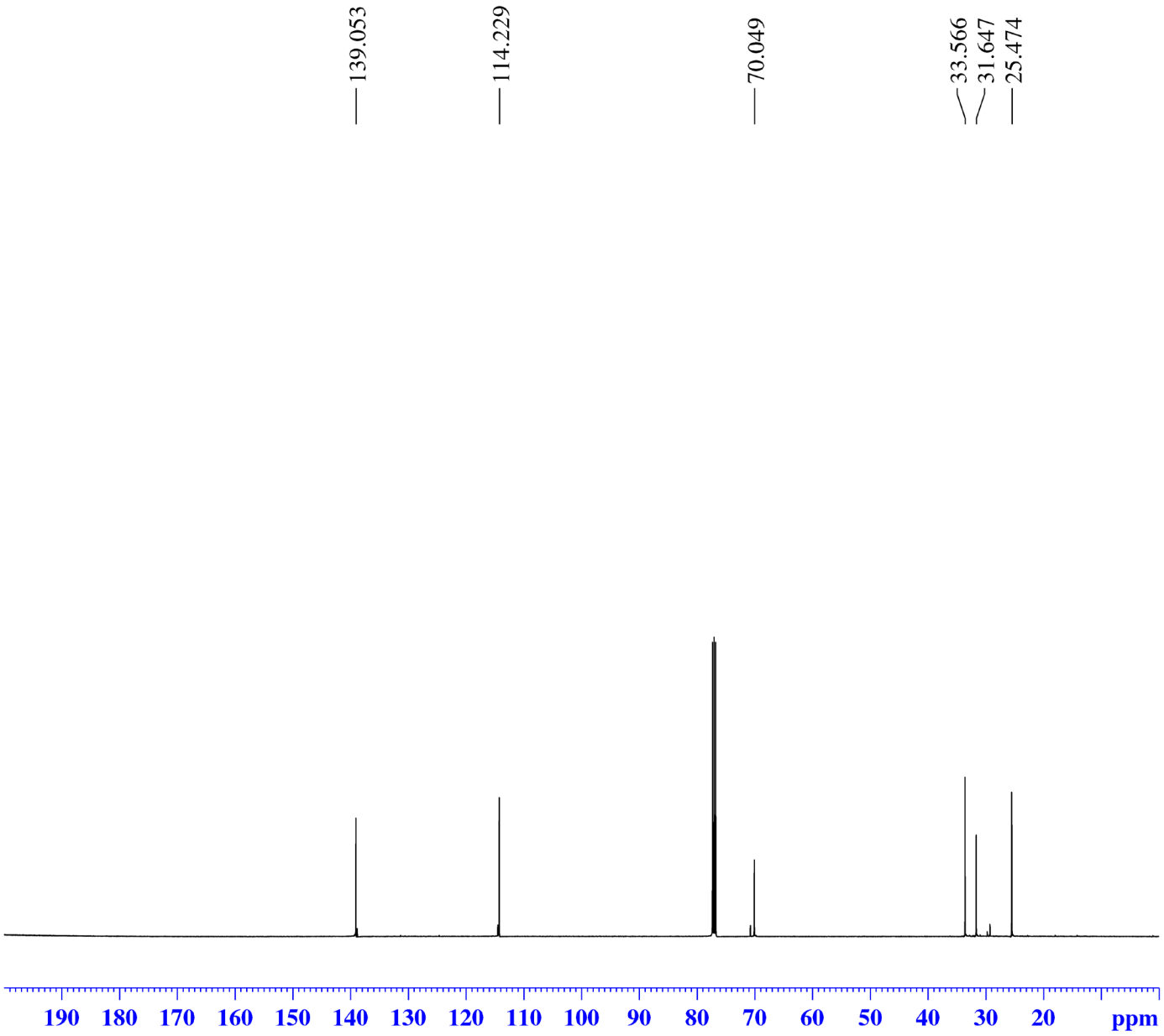
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776161 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 1.40



^{13}C NMR



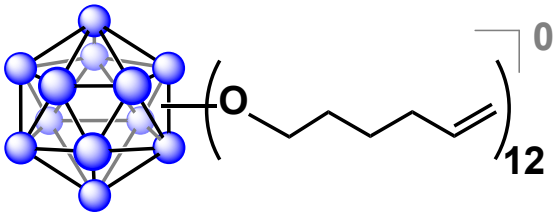
Current Data Parameters
NAME B12(O-Hexene)12
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151021
Time 17.44
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

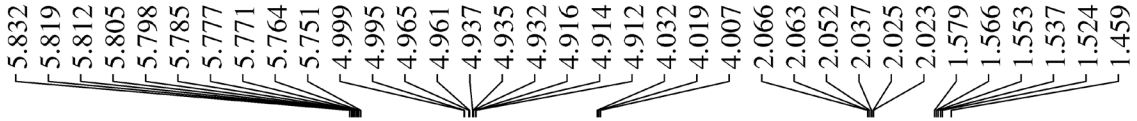
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

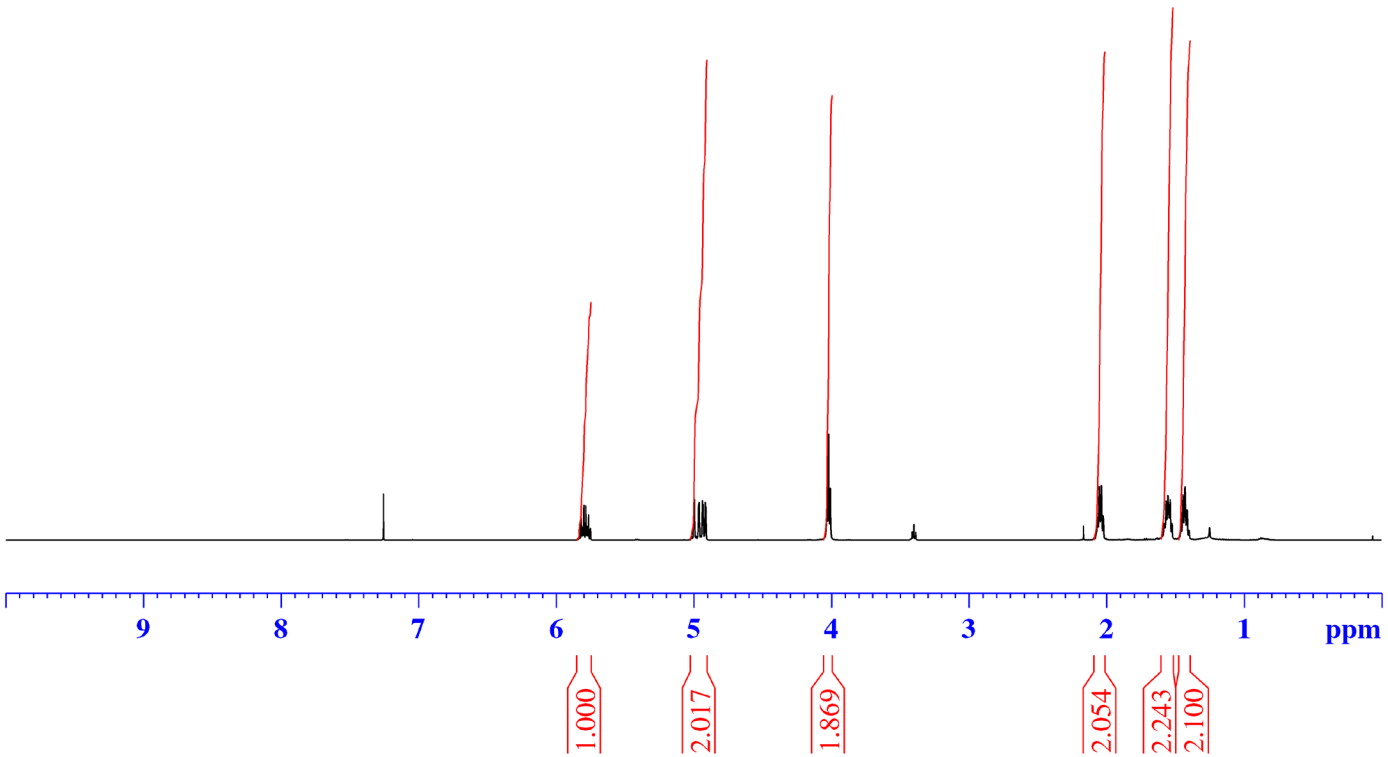
======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR



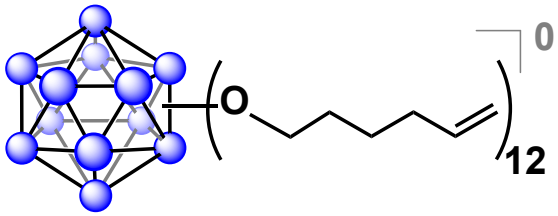


Current Data Parameters
NAME B12(O-Hexene)12
EXPNO 21
PROCNO 1

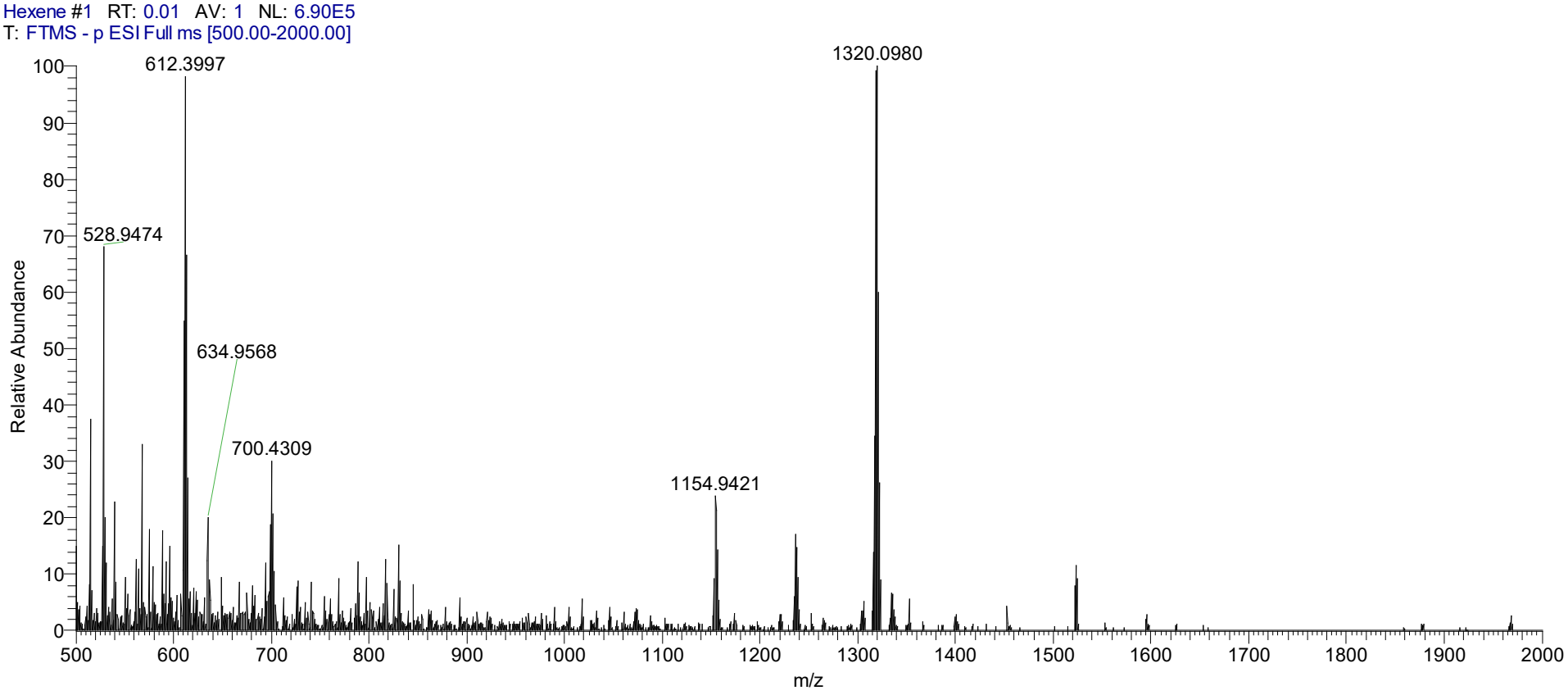
F2 - Acquisition Parameters
Date_ 20151021
Time 17.48
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 33.91
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

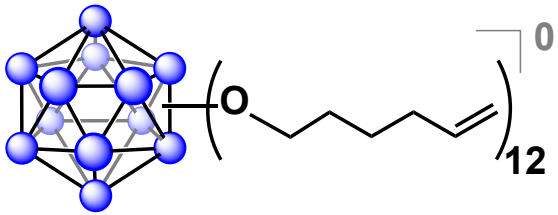
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



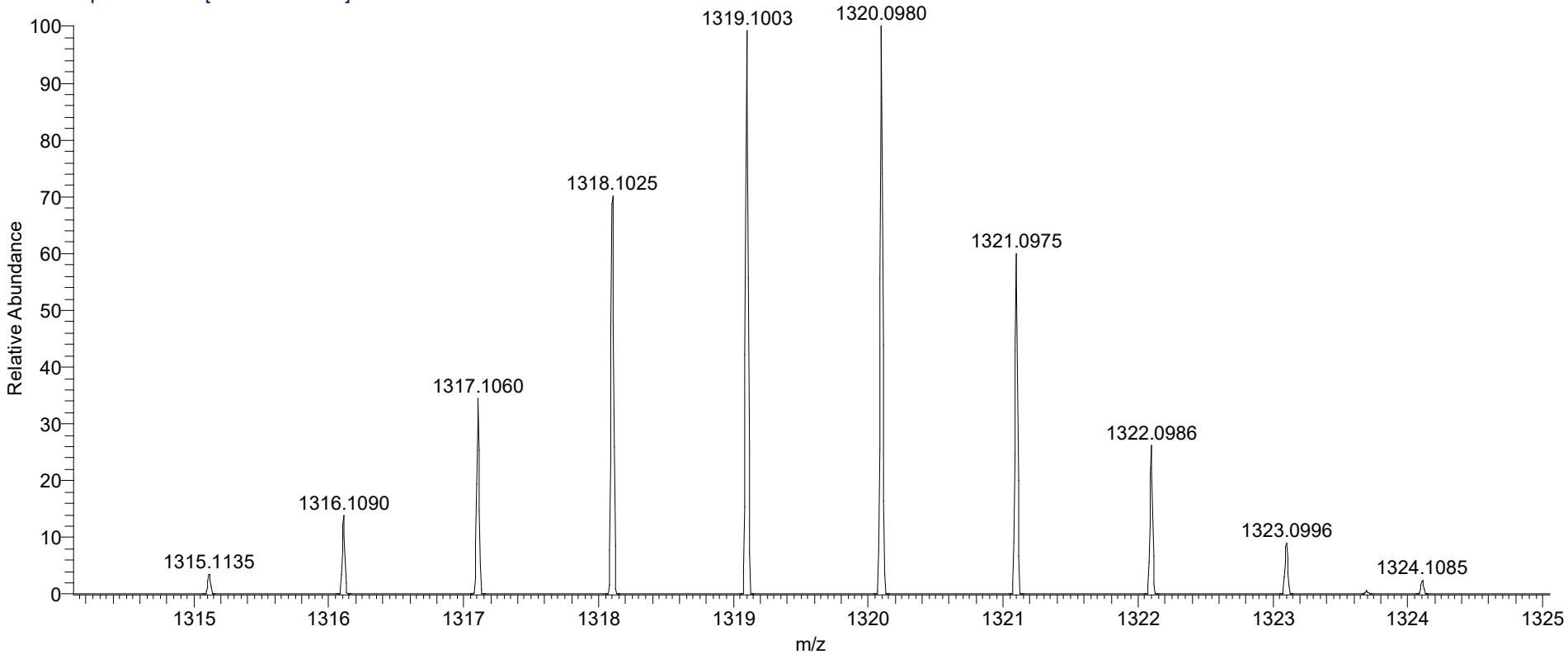
Q Exactive
High-Res Mass Spec

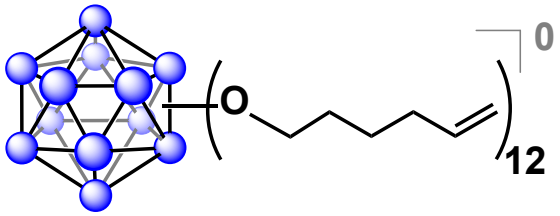




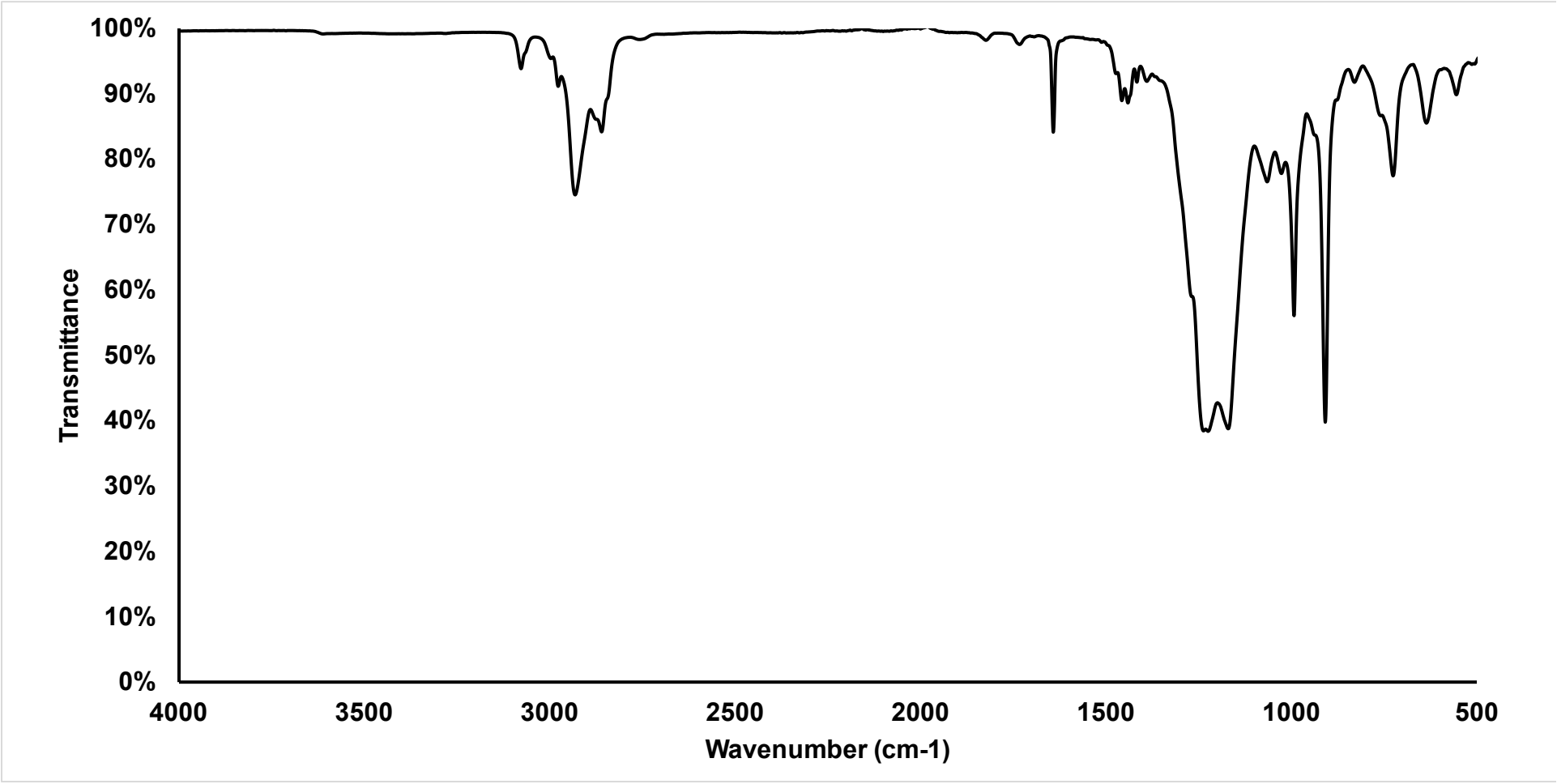
Q Exactive
High-Res Mass Spec

Hexene #1 RT: 0.01 AV: 1 NL: 6.90E5
T: FTMS - p ESI Full ms [500.00-2000.00]




O
12
0

IR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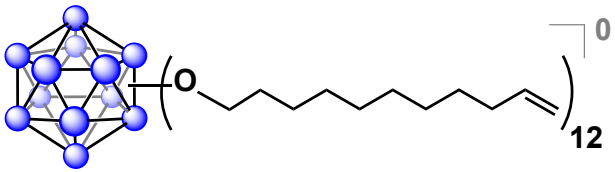
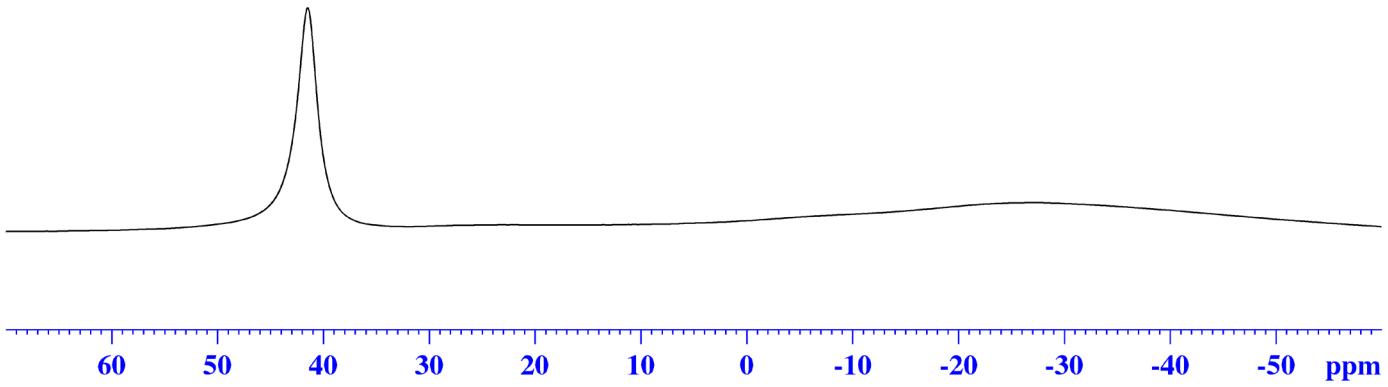
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
Transmittance
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)



^{11}B {^{1}H} NMR

41.474



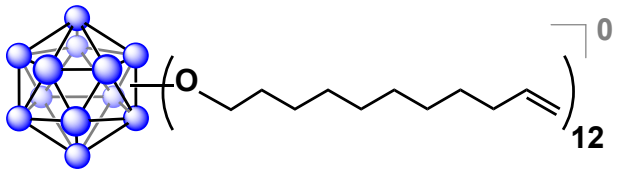
Current Data Parameters
NAME B12(O-1-undecene)12
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150626
Time 12.16
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CD2Cl2
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.1 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

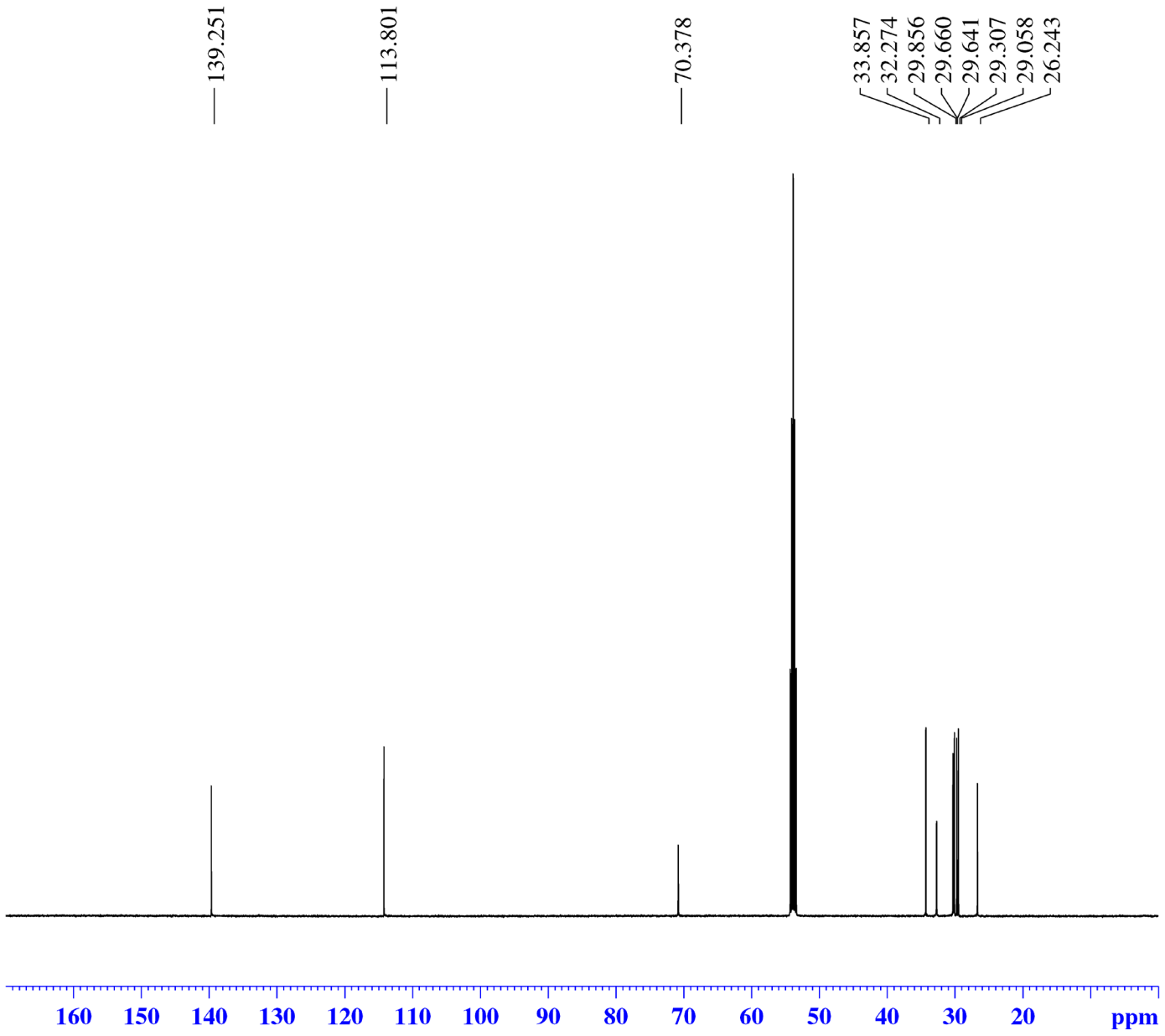
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40



^{13}C NMR



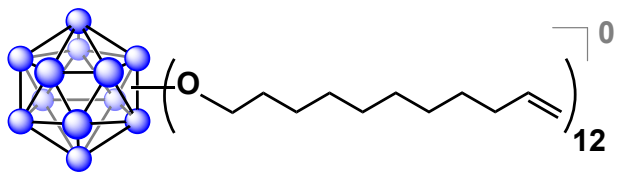
Current Data Parameters
NAME Jun26-2015
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150626
Time 14.05
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

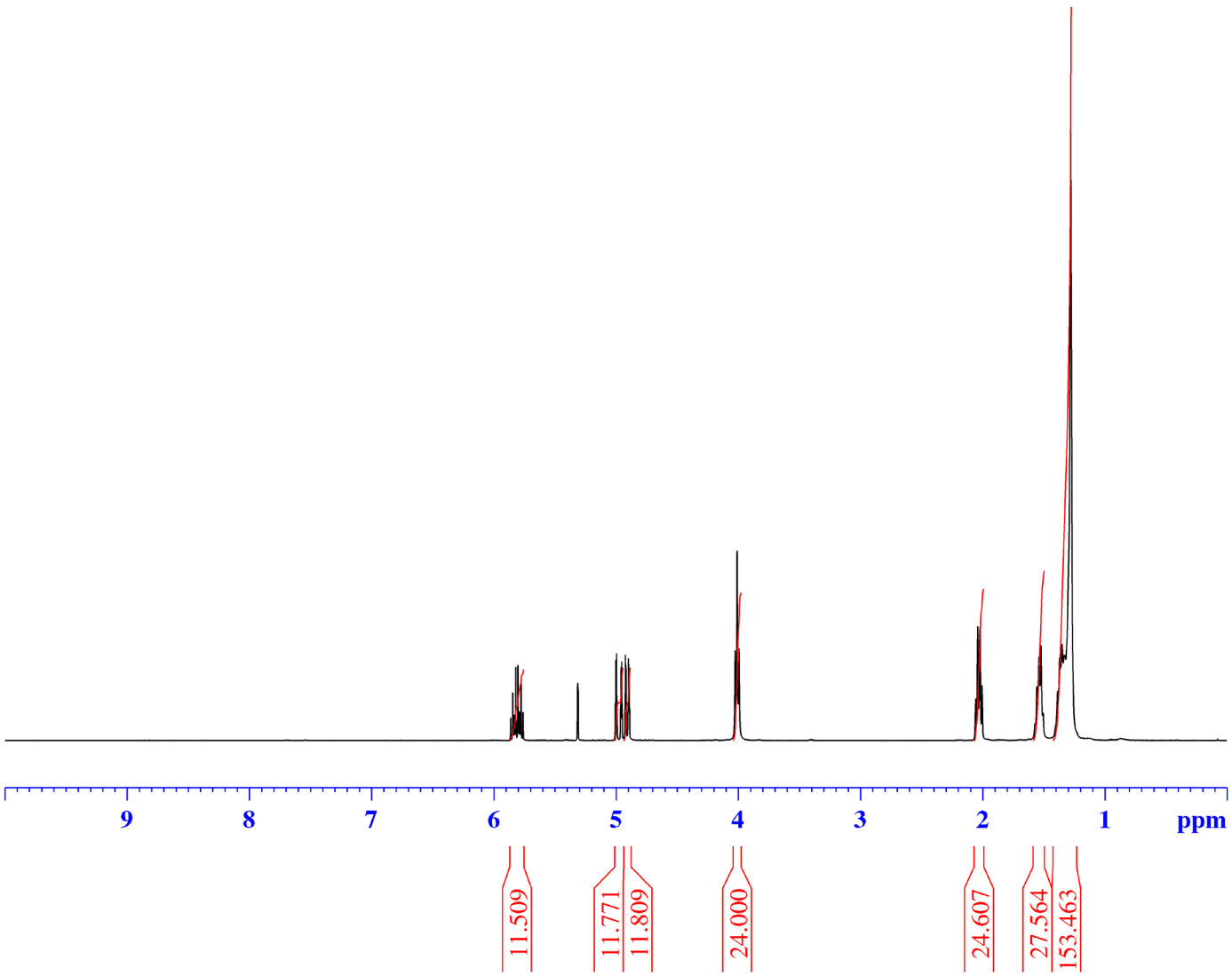
F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR

BRUKER

5.845 5.819 5.802 5.777 5.315 5.312 5.310 5.005 5.001 4.996 4.992 4.962 4.958 4.953 4.949 4.926 4.923 4.920 4.918 4.915 4.901 4.898 4.895 4.892 4.024 4.008 3.992 2.057 2.040 2.023 2.021 2.007 2.004 1.556 1.539 1.521 1.386 1.368 1.350 1.334 1.328 1.280

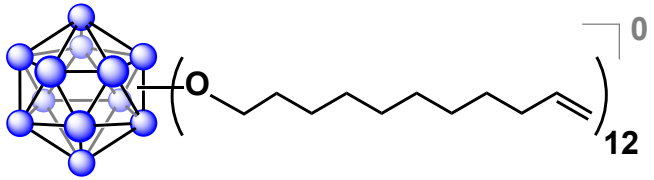


Current Data Parameters
NAME B12(O-1-undecene)12
EXPNO 21
PROCNO 1

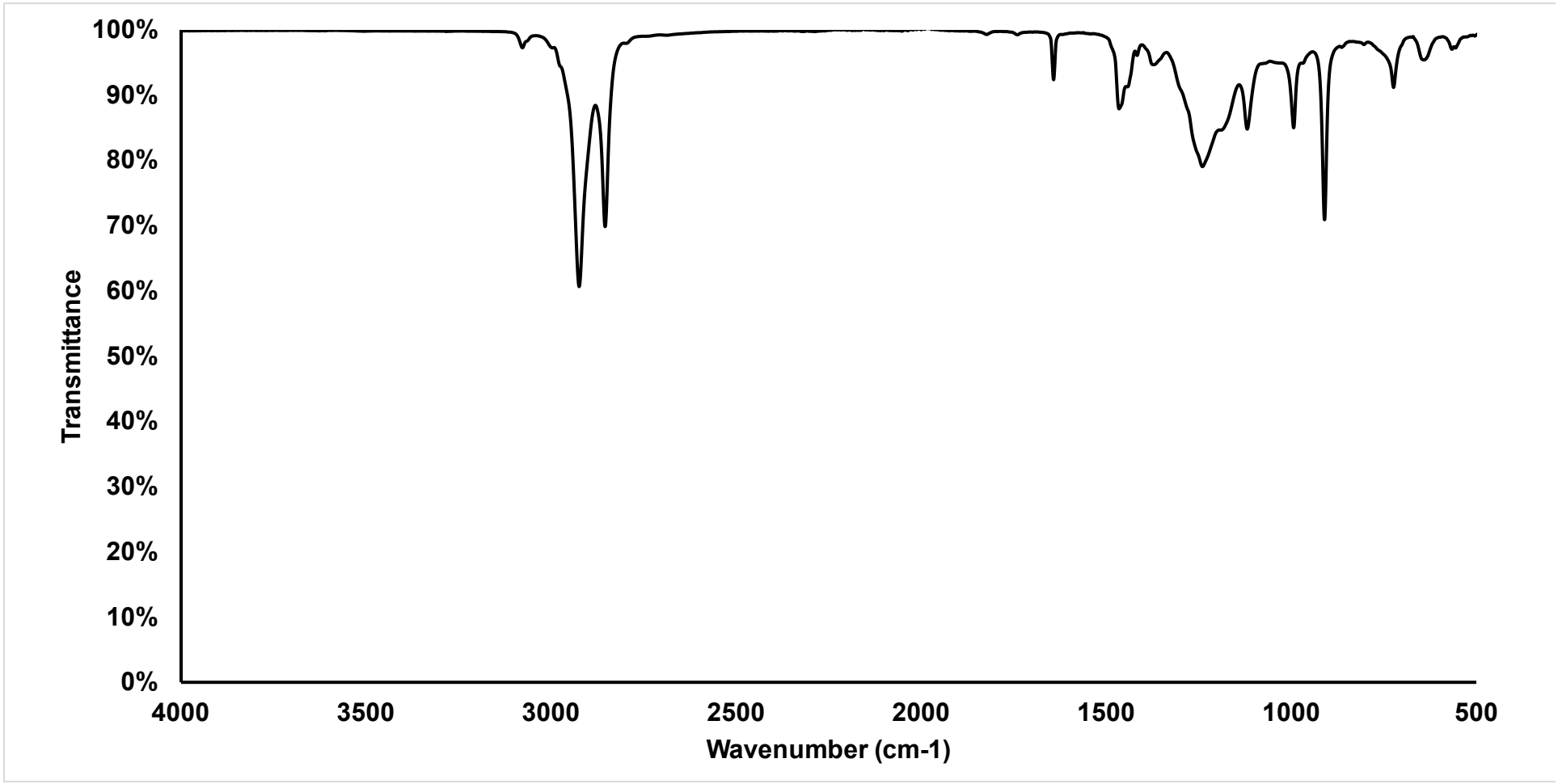
F2 - Acquisition Parameters
Date_ 20150626
Time 12.18
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CD2Cl2
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 83.63
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300184 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


O
12
0

IR


100%
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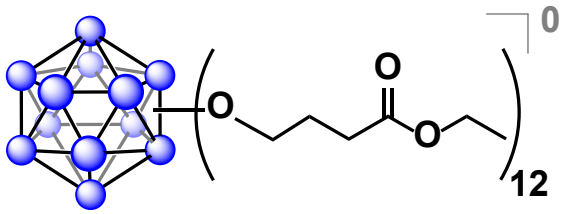
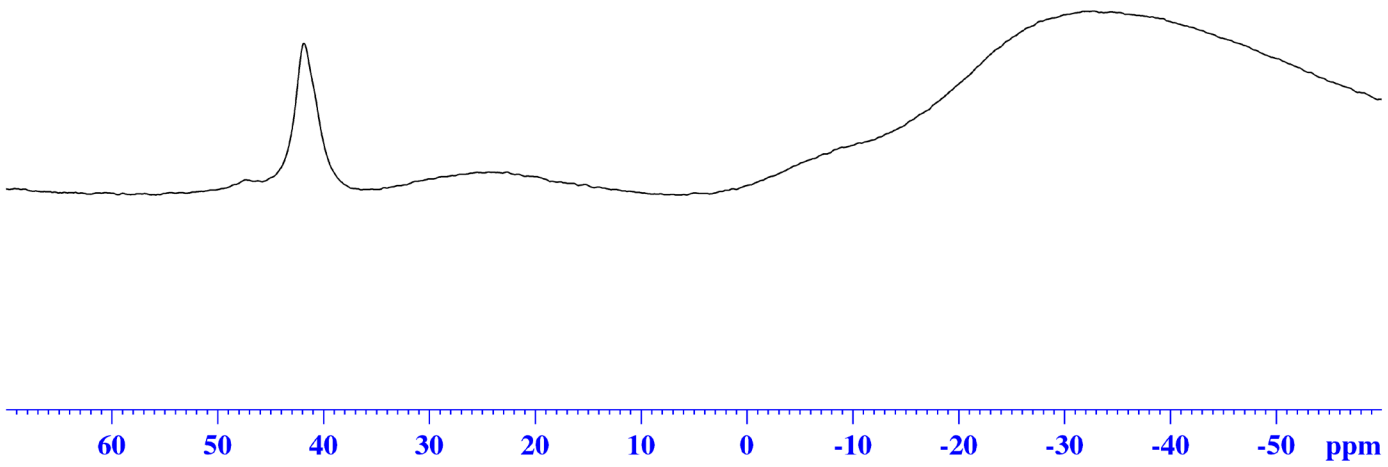
80%
70%
60%
50%
40%
30%
20%
10%
0%
Transmittance
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)



^{11}B {^{1}H} NMR

41.862



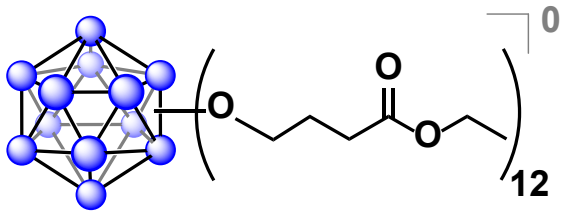
Current Data Parameters
NAME B12(O-EtButyr)12
EXPNO 110
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150412
Time 19.19
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.1 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

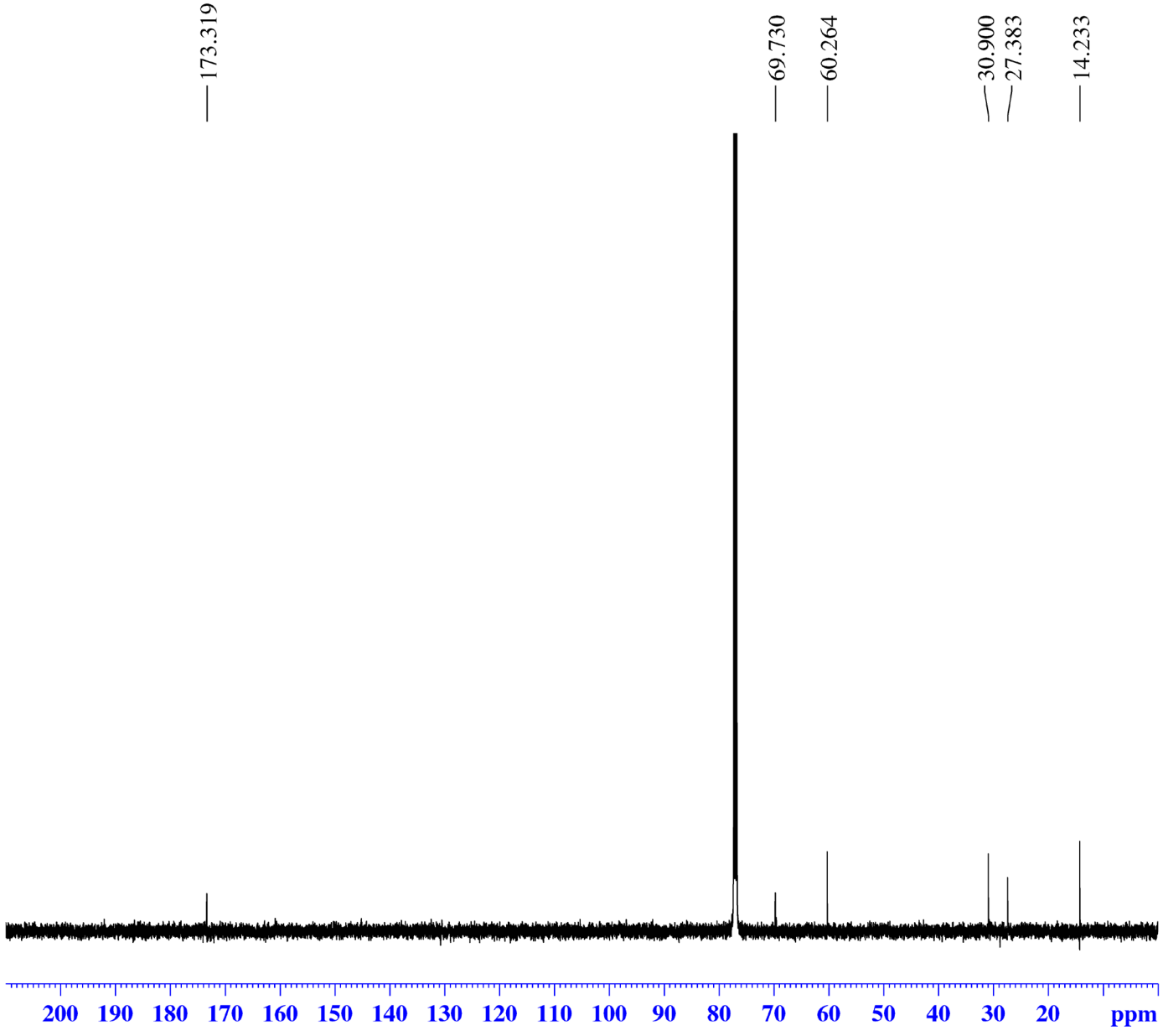
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40



^{13}C NMR



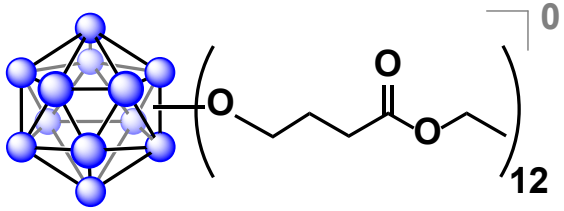
Current Data Parameters
NAME B12(O-EtButyr)12
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150406
Time 23.42
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

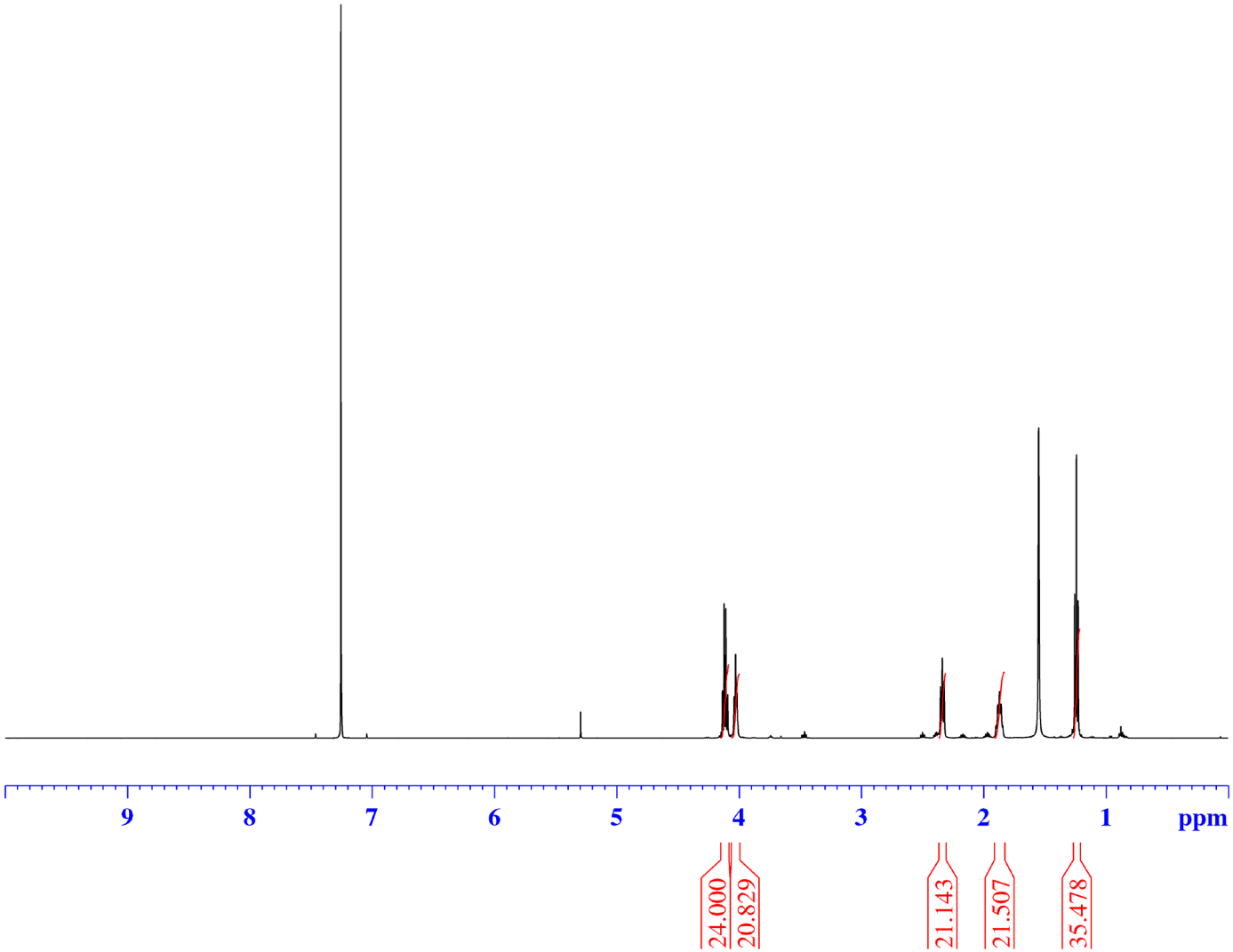
F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR

BRUKER

7.462 7.255 7.044 5.295 4.146 4.135 4.121 4.107 4.093 4.040 4.028 4.015 3.464 3.451 2.499 2.485 2.392 2.385 2.377 2.370 2.353 2.338 2.323 2.170 1.982 1.968 1.954 1.899 1.886 1.870 1.856 1.844 1.550 1.274 1.256 1.241 1.227 1.217 1.202 0.891 0.878 0.864

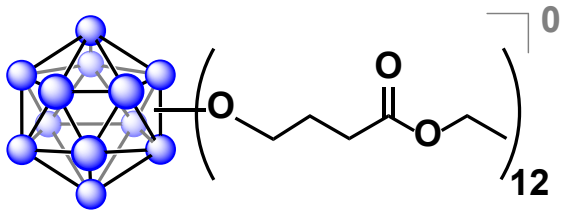


Current Data Parameters
NAME B12(O-EtButyr)12
EXPNO 61
PROCNO 1

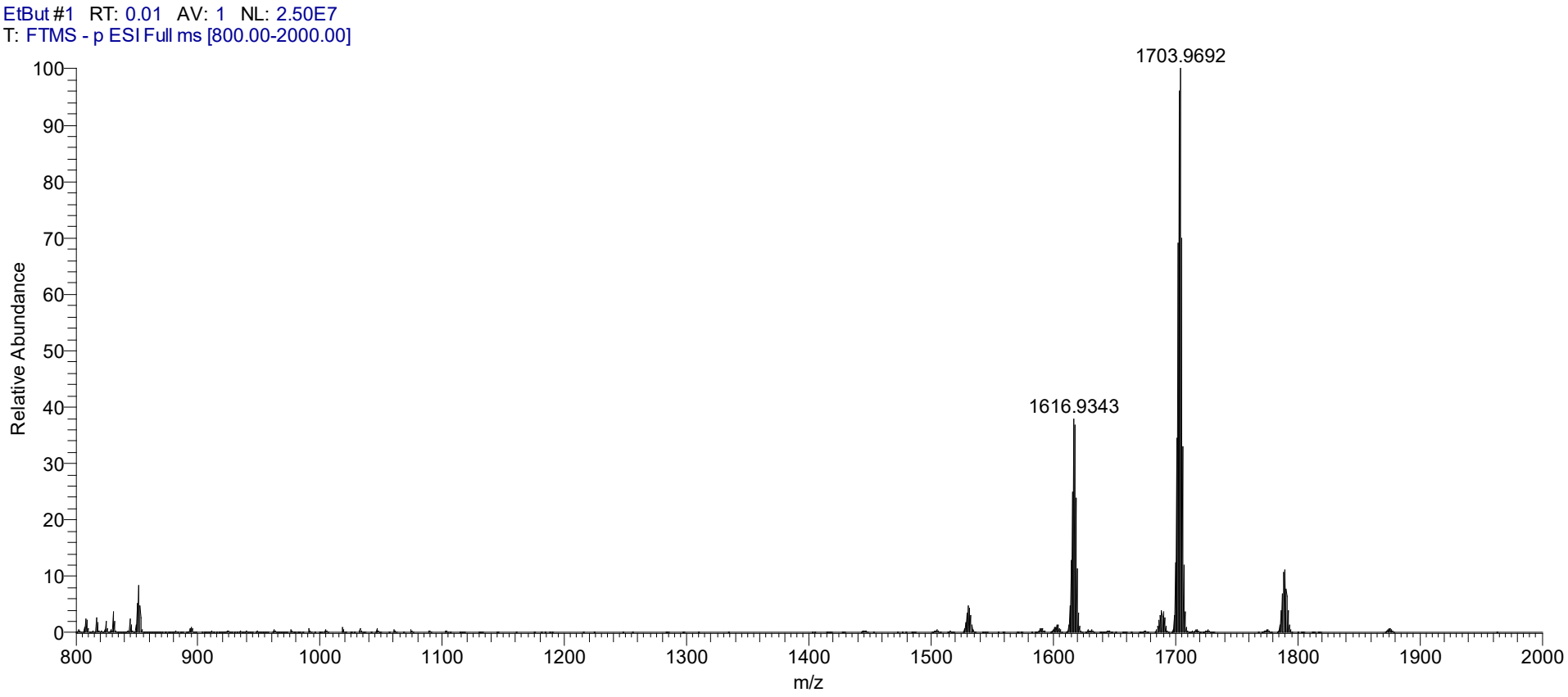
F2 - Acquisition Parameters
Date_ 20150406
Time 23.44
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 59.34
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

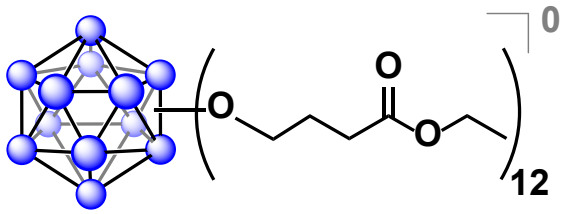
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

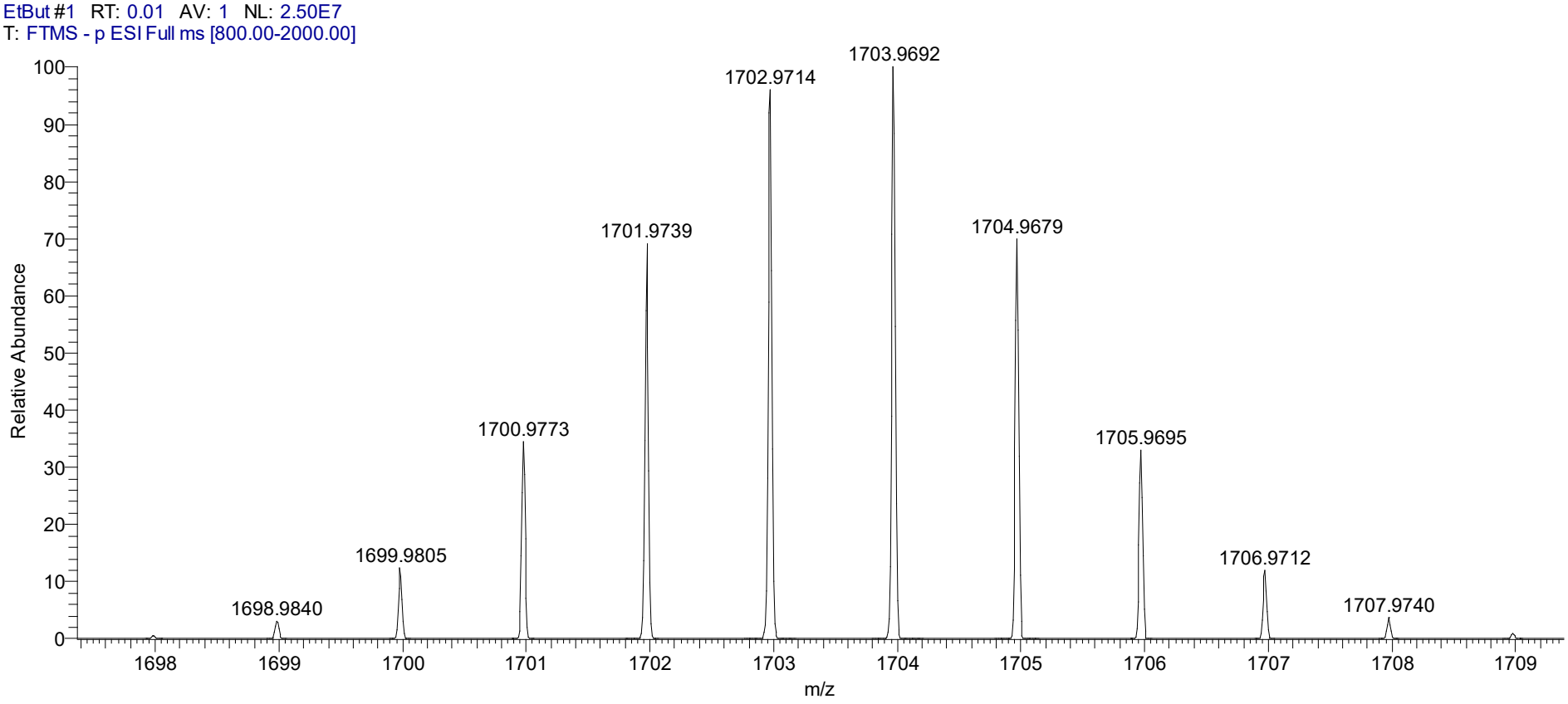


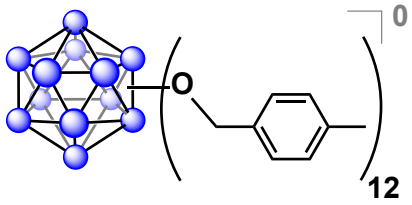
Q Exactive
High-Res Mass Spec





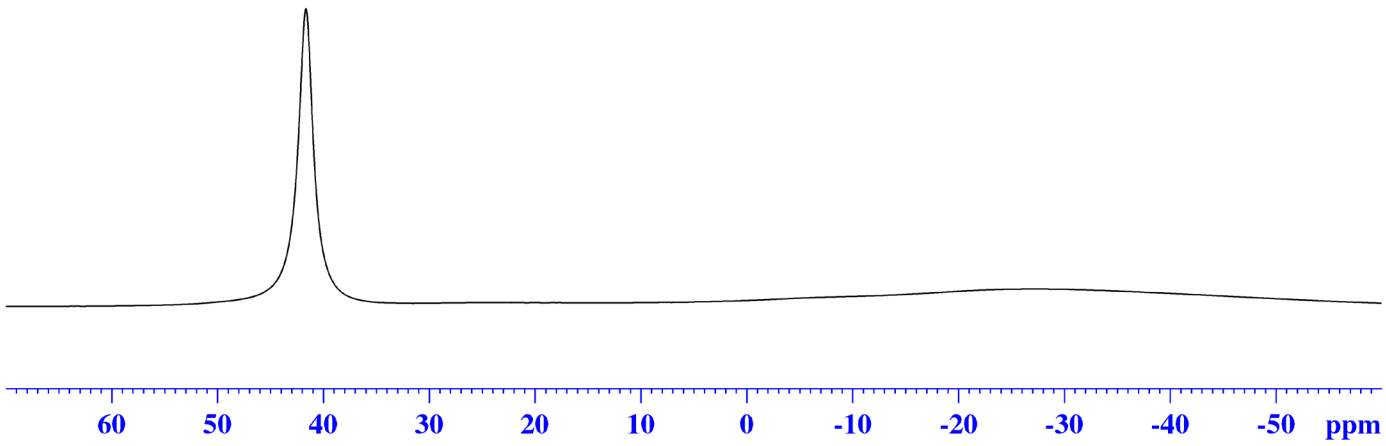
Q Exactive
High-Res Mass Spec





^{11}B {^{1}H} NMR

41.643



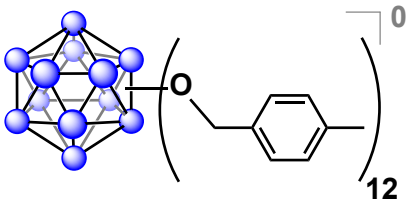
Current Data Parameters
NAME Aug29-2015
EXPNO 160
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150829
Time 21.02
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.3 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

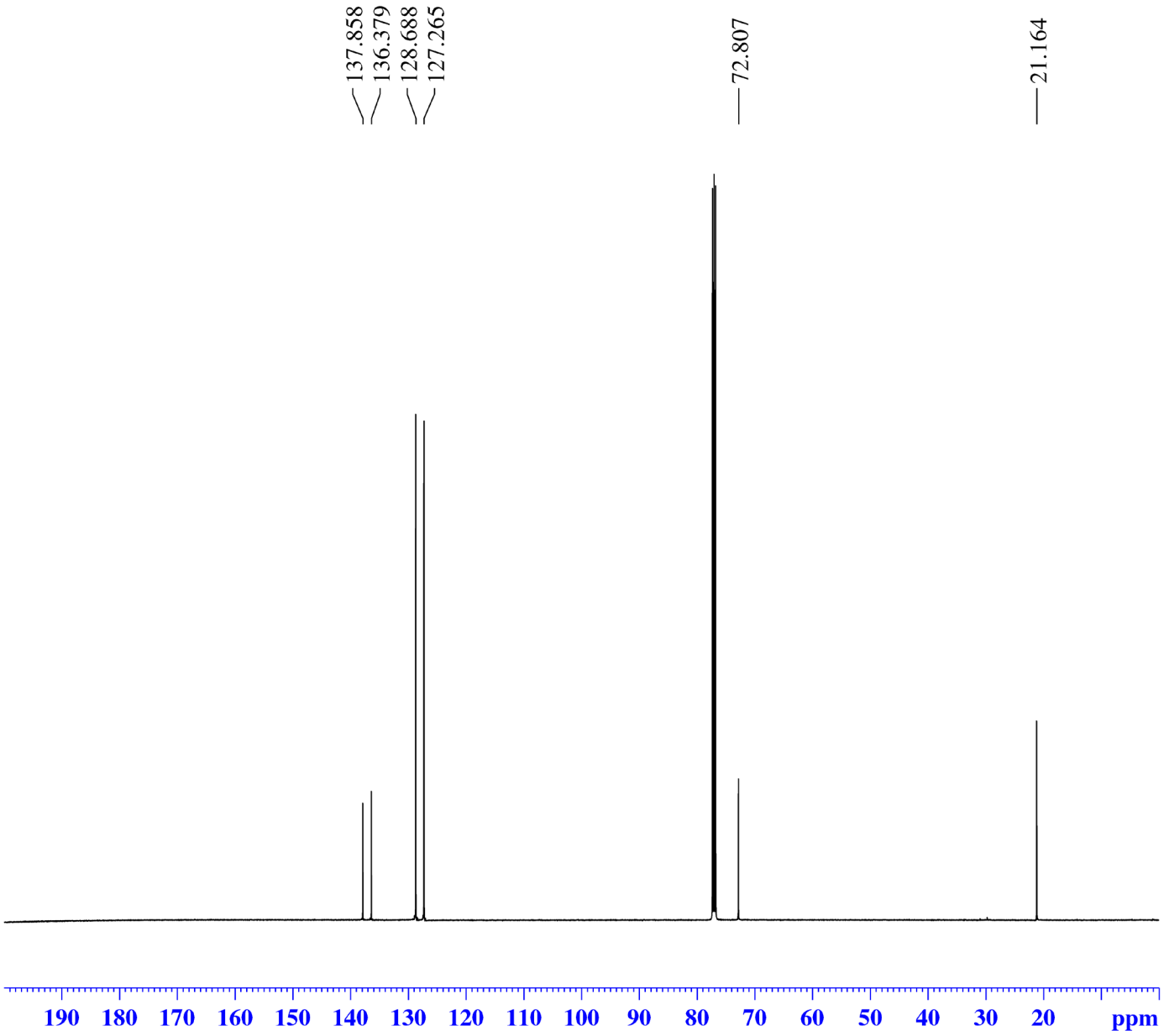
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40



^{13}C NMR



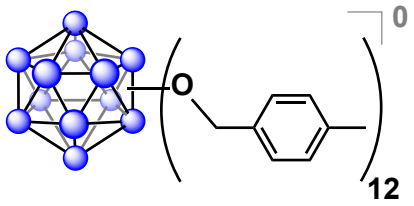
Current Data Parameters
NAME B12(O-4-MeBn)12
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150829
Time 22.04
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

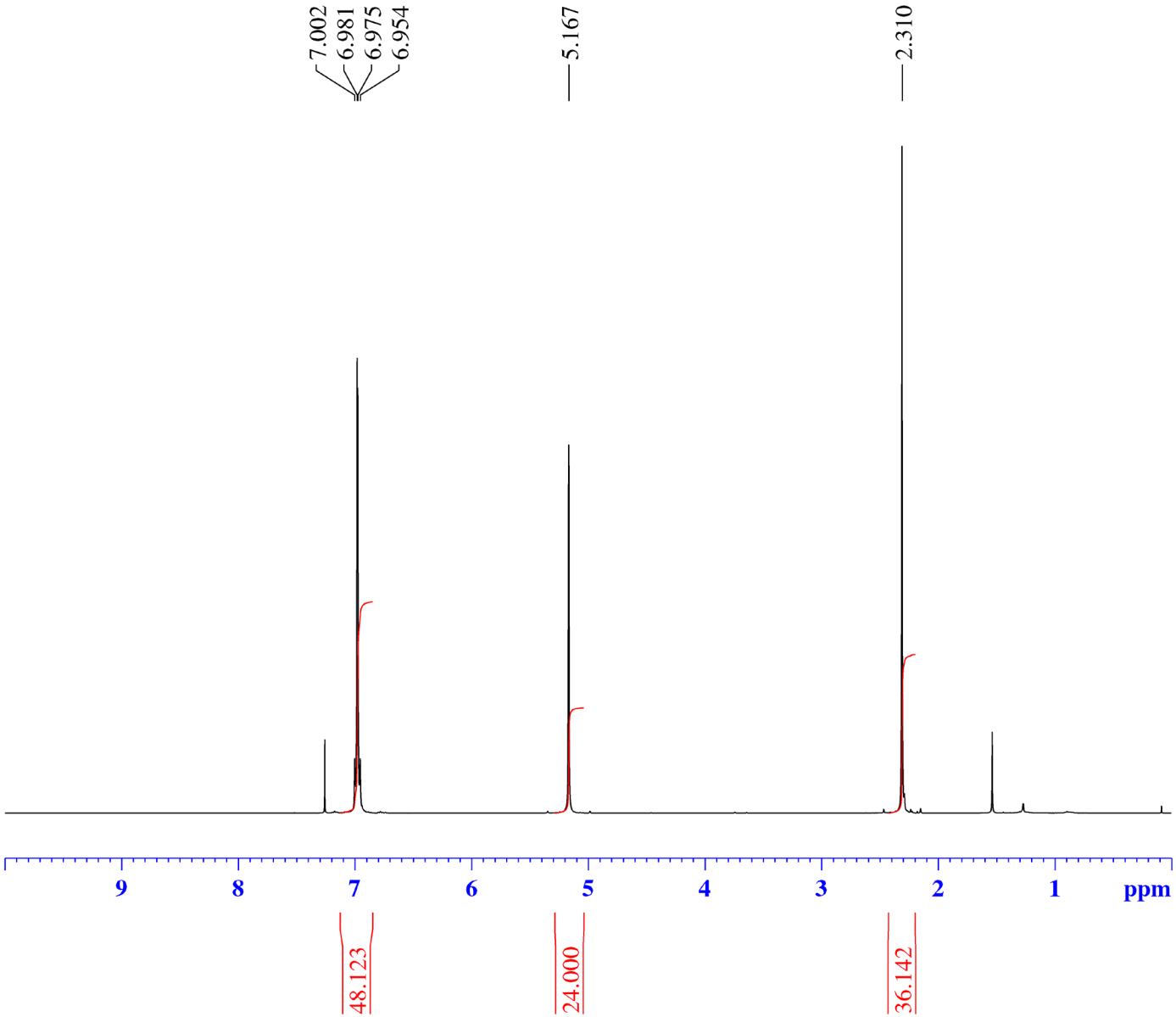
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR

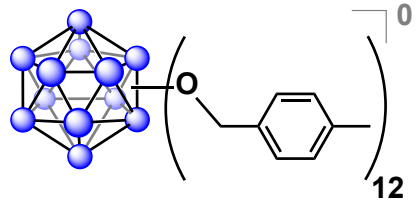


Current Data Parameters
NAME B12(O-4-MeBn)12
EXPNO 161
PROCNO 1

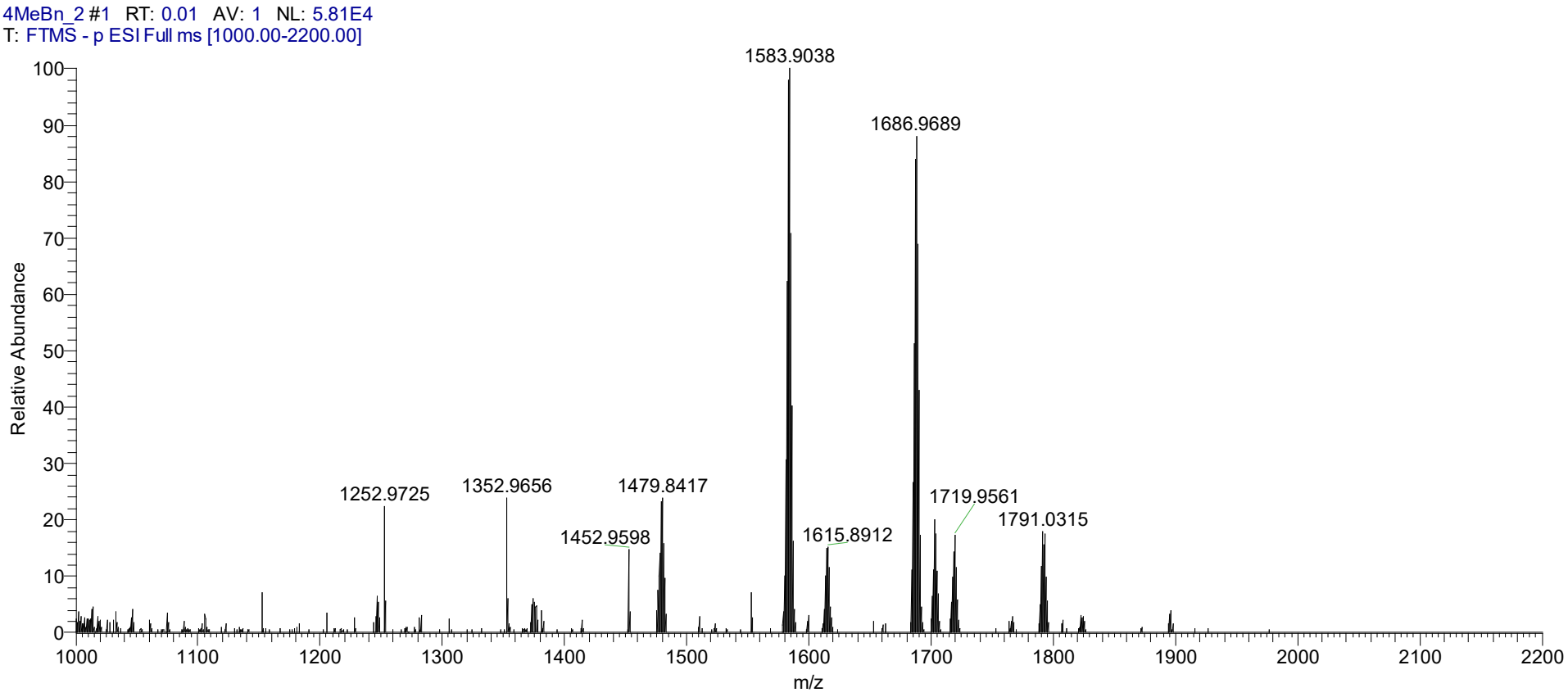
F2 - Acquisition Parameters
Date_ 20150829
Time 21.05
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 32
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

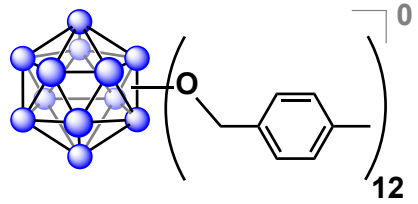
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300184 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

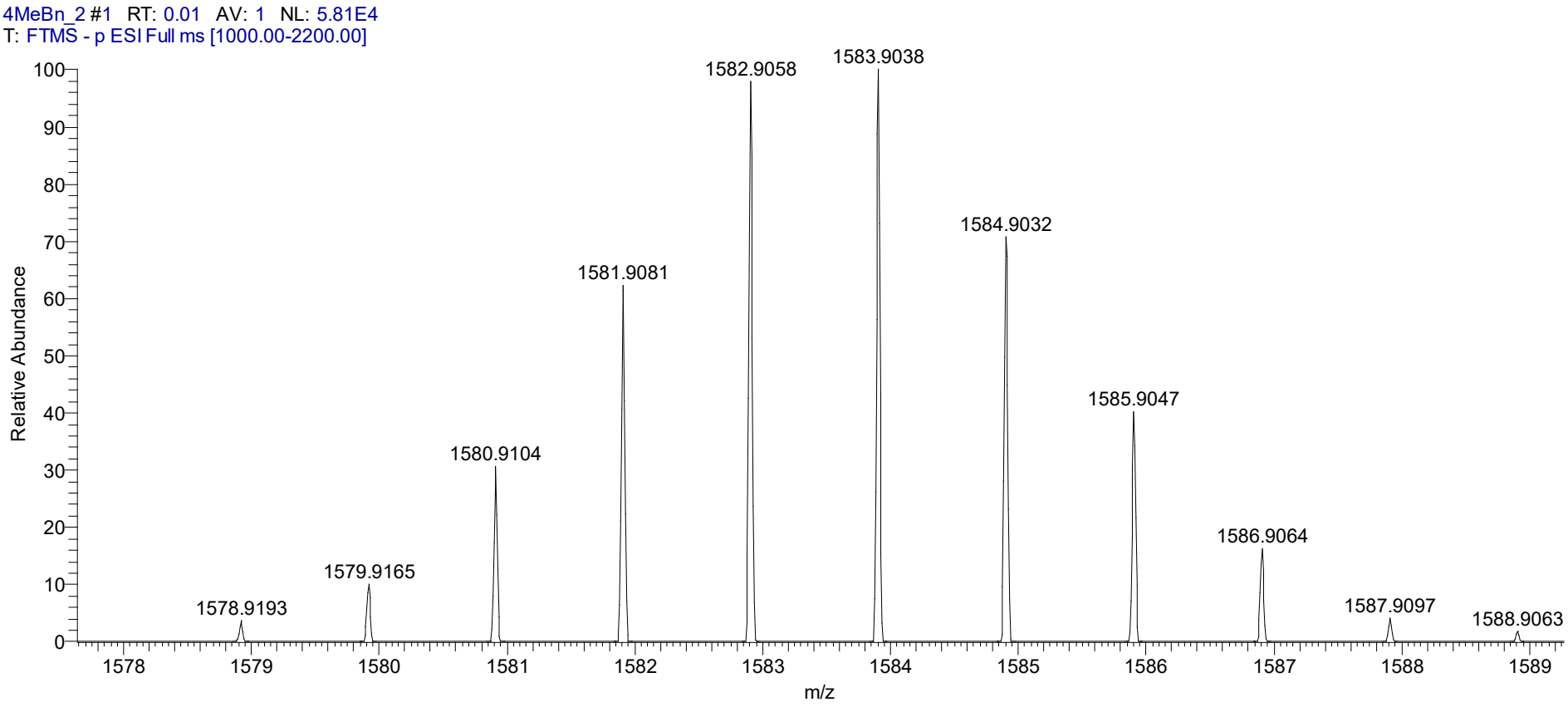


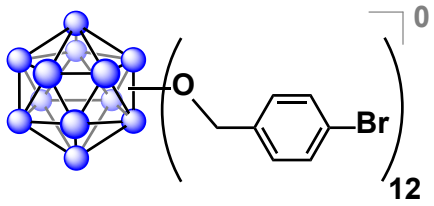
Q Exactive
High-Res Mass Spec





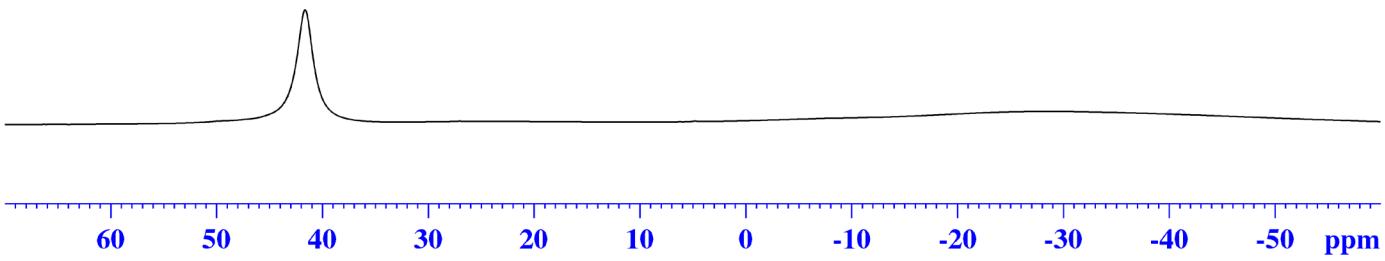
Q Exactive
High-Res Mass Spec





^{11}B {^{1}H} NMR

41.399



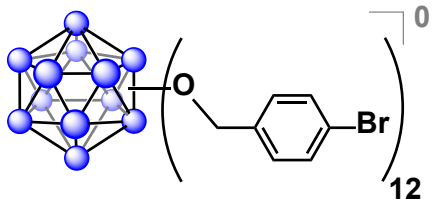
Current Data Parameters
NAME B12(O-4-BrBn)12
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150624
Time 10.50
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.1 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

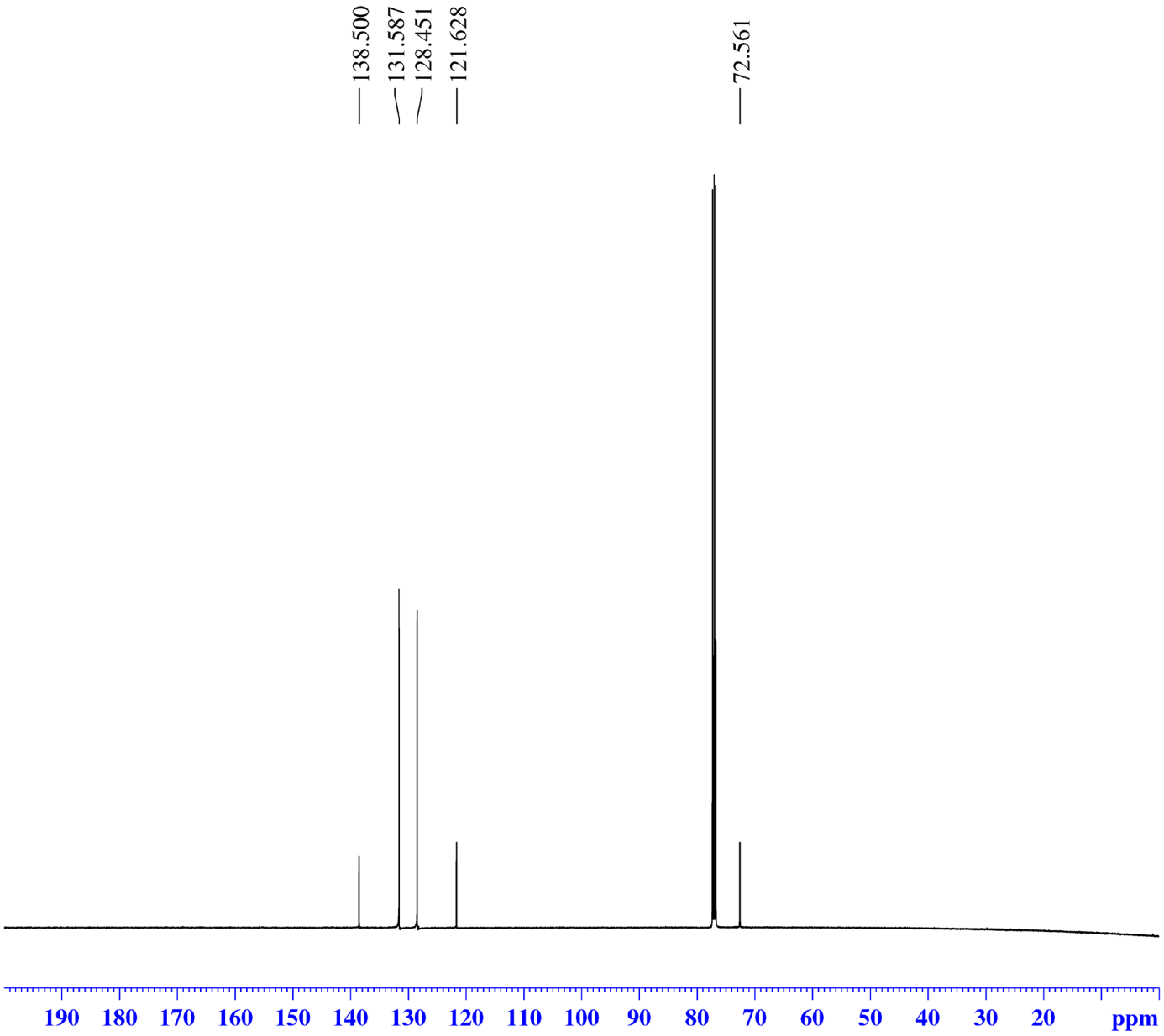
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40



^{13}C NMR



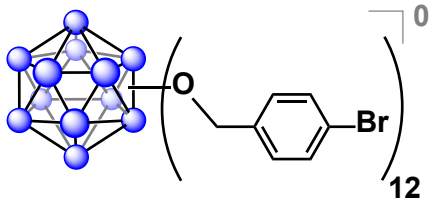
Current Data Parameters
NAME Jan13-2016-awixtrom
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160113
Time 15.37
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

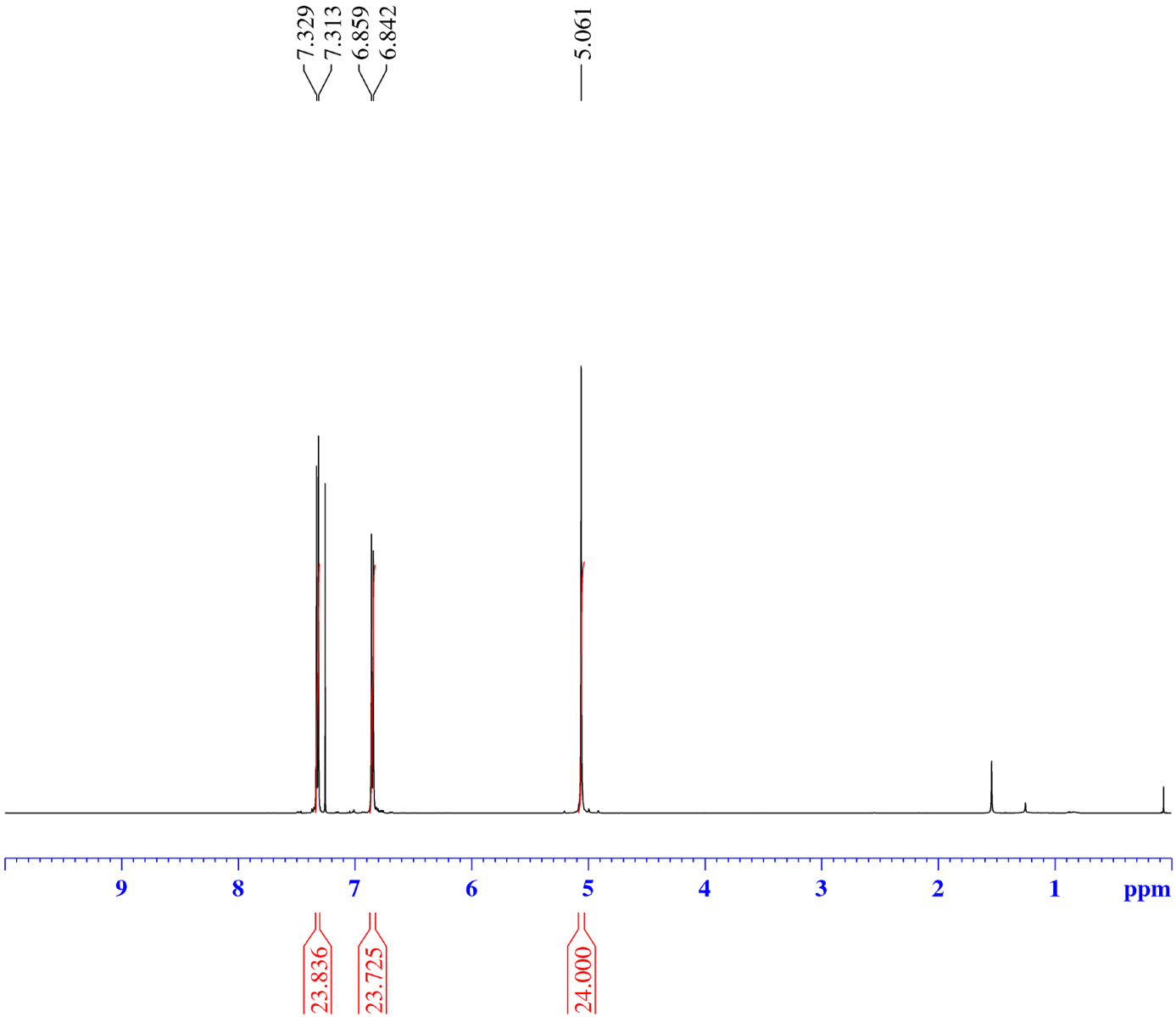
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR

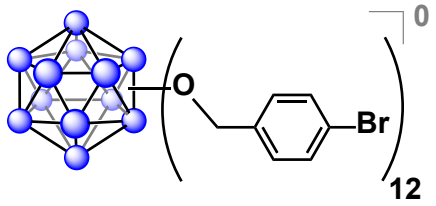


Current Data Parameters
NAME Jan13-2016-awixtrom
EXPNO 11
PROCNO 1

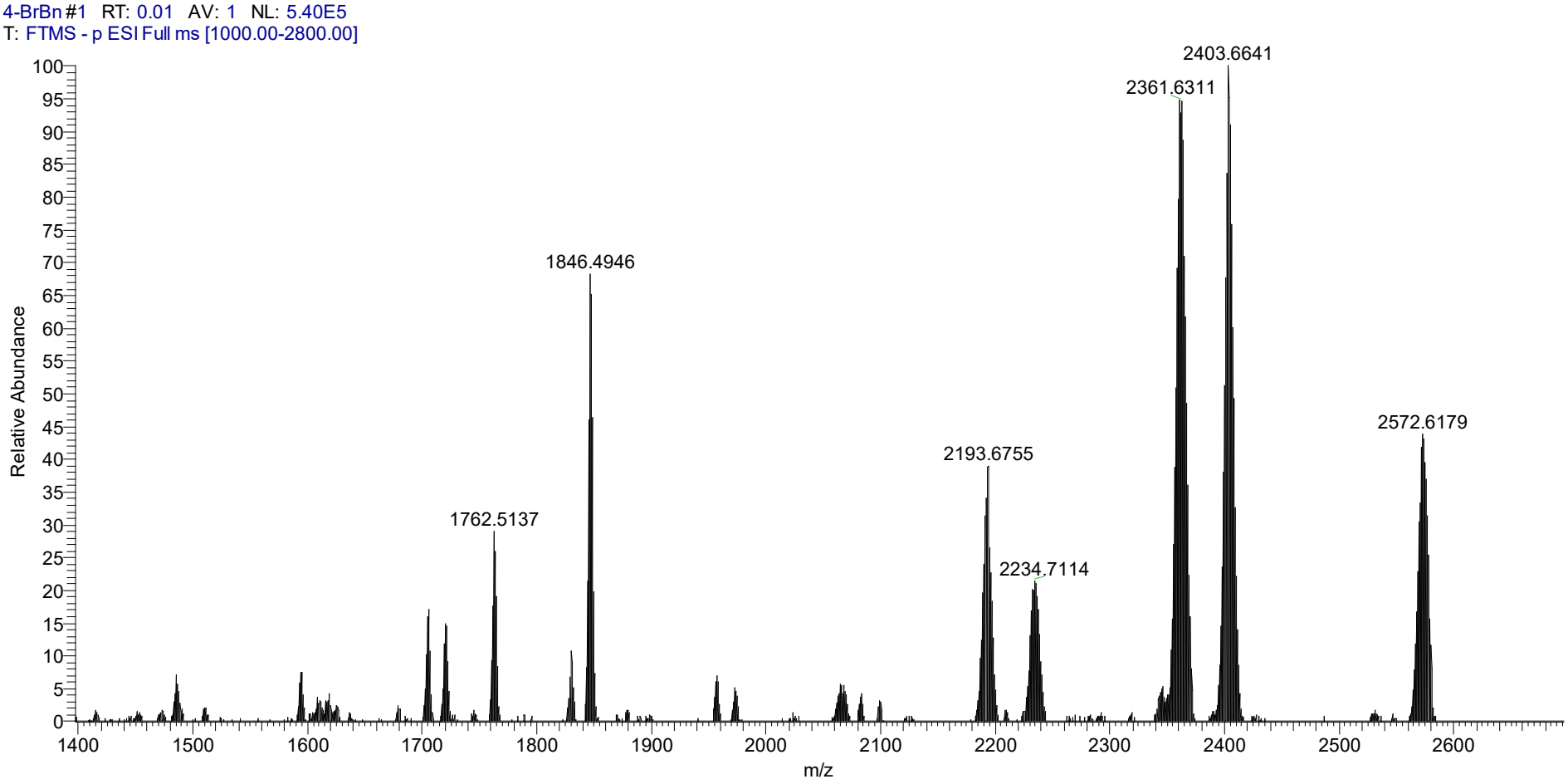
F2 - Acquisition Parameters
Date_ 20160113
Time 15.40
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 52.41
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

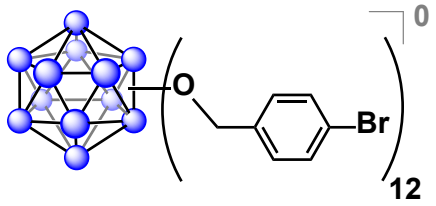
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

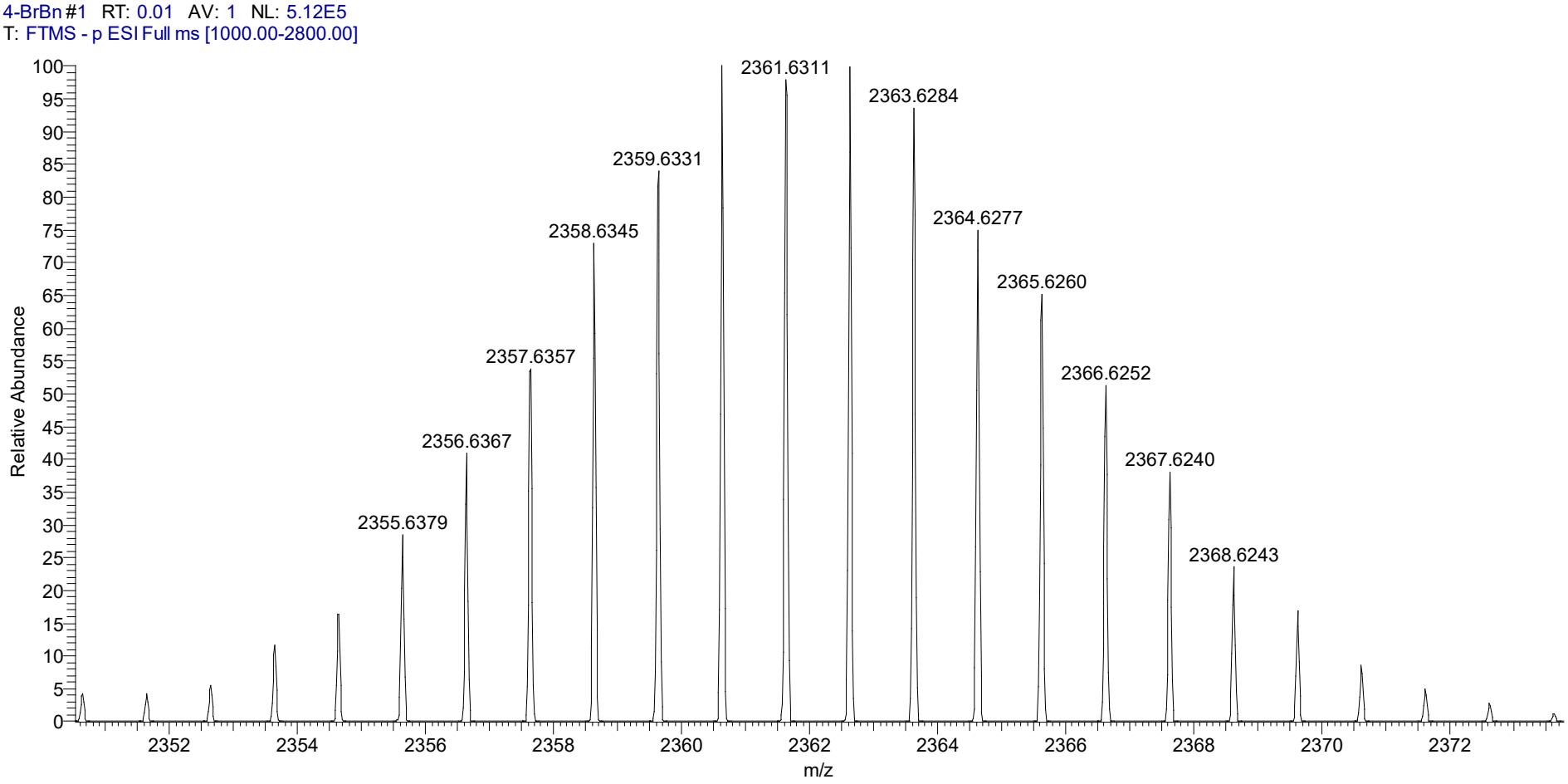


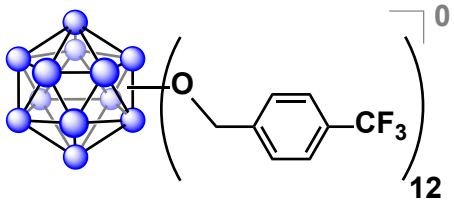
Q Exactive
High-Res Mass Spec





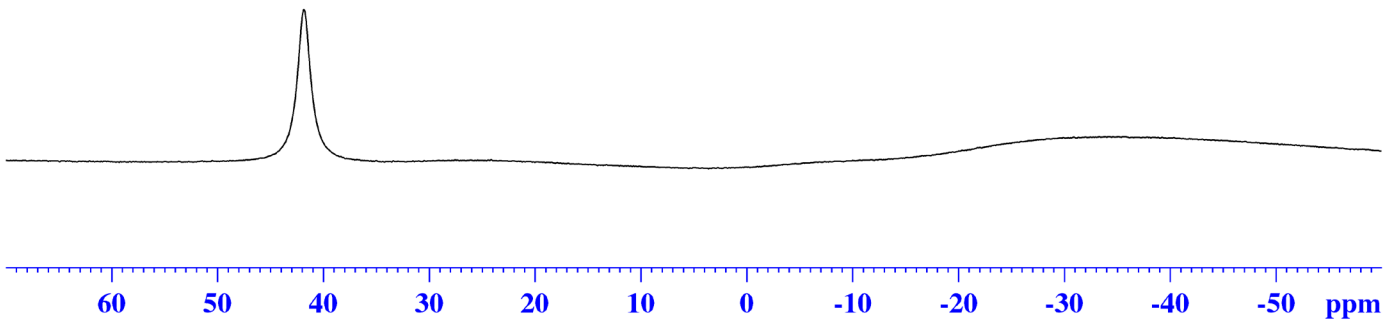
Q Exactive
High-Res Mass Spec





^{11}B {^{1}H} NMR

41.831



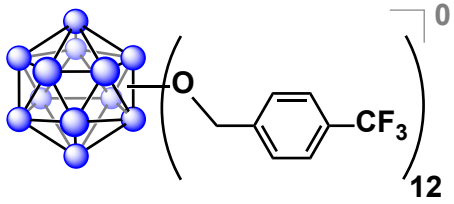
Current Data Parameters
NAME B12(O-4-TFMBn)12
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150927
Time 14.58
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.3 K
D1 0.05000000 sec
D11 0.03000000 sec
TD0 1

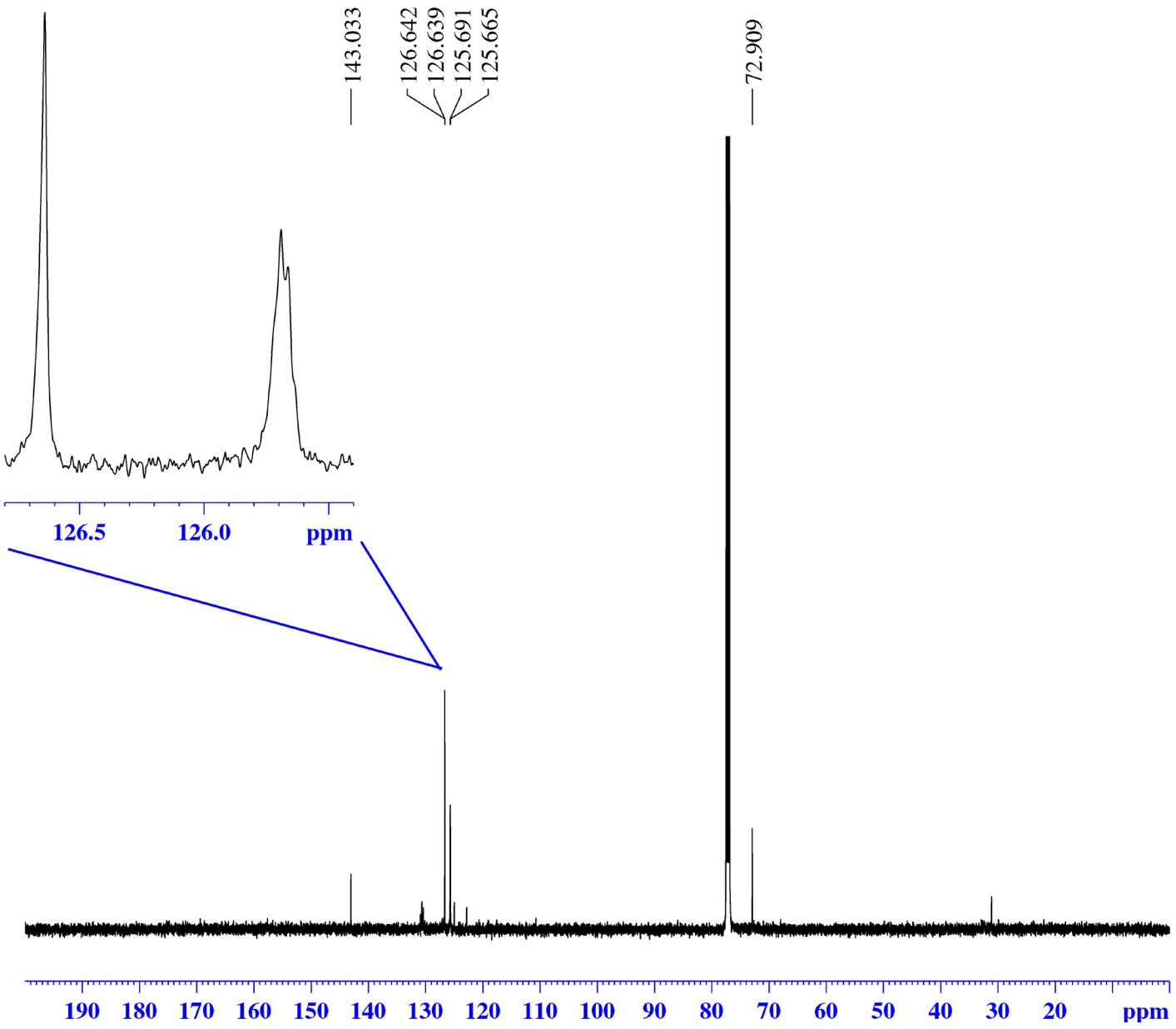
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776161 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 1.40



^{13}C NMR



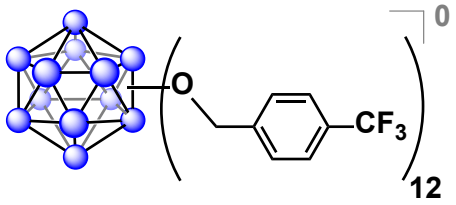
Current Data Parameters
NAME B12(O-4-TFMBn)12
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150928
Time 13.45
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

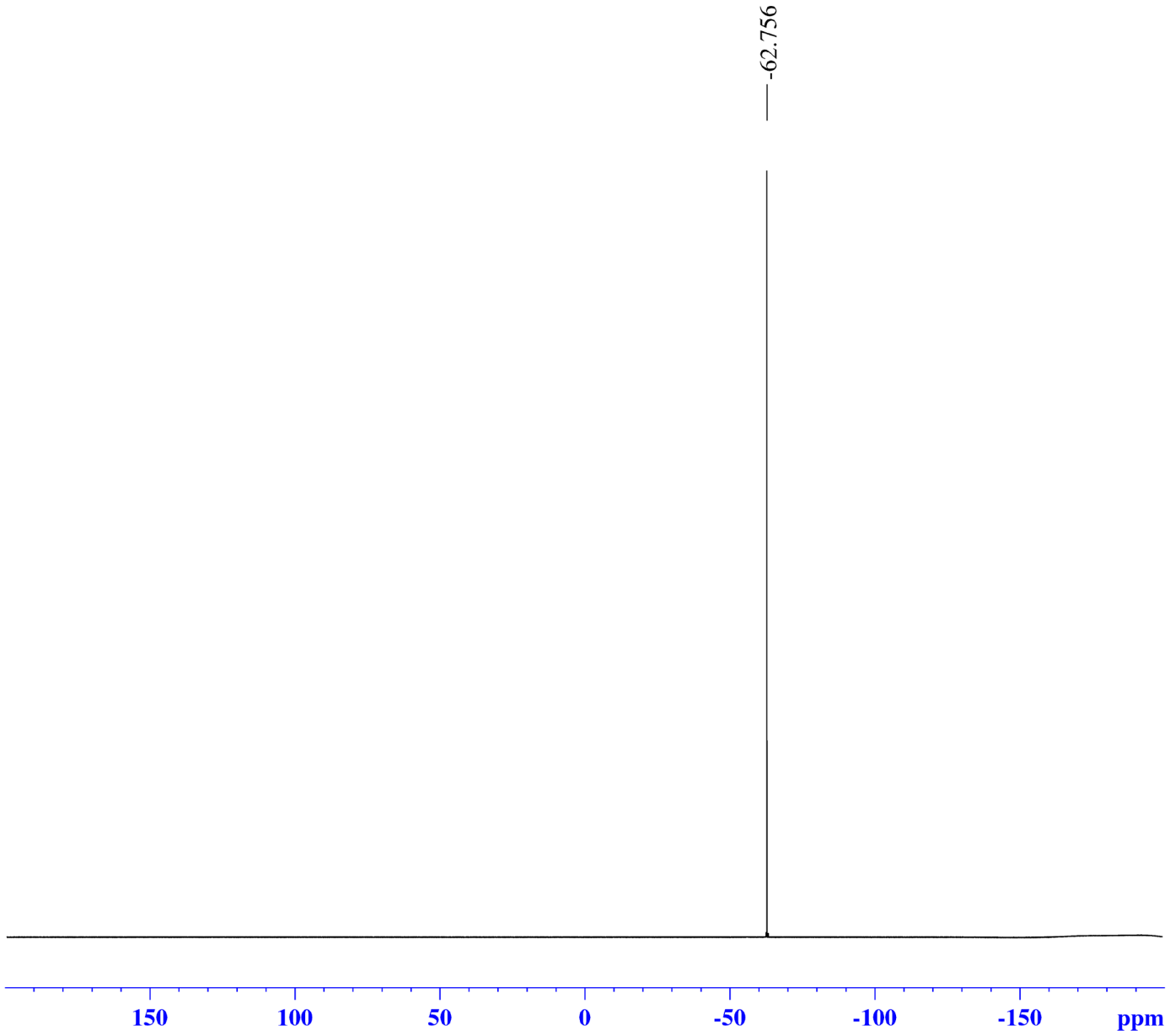
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{19}F NMR

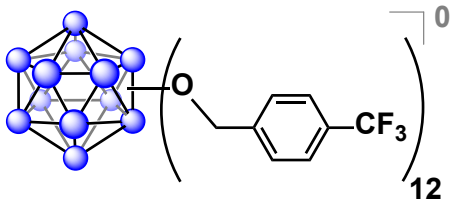


Current Data Parameters
NAME B12(O-4-TFMBn)12
EXPNO 42
PROCNO 1

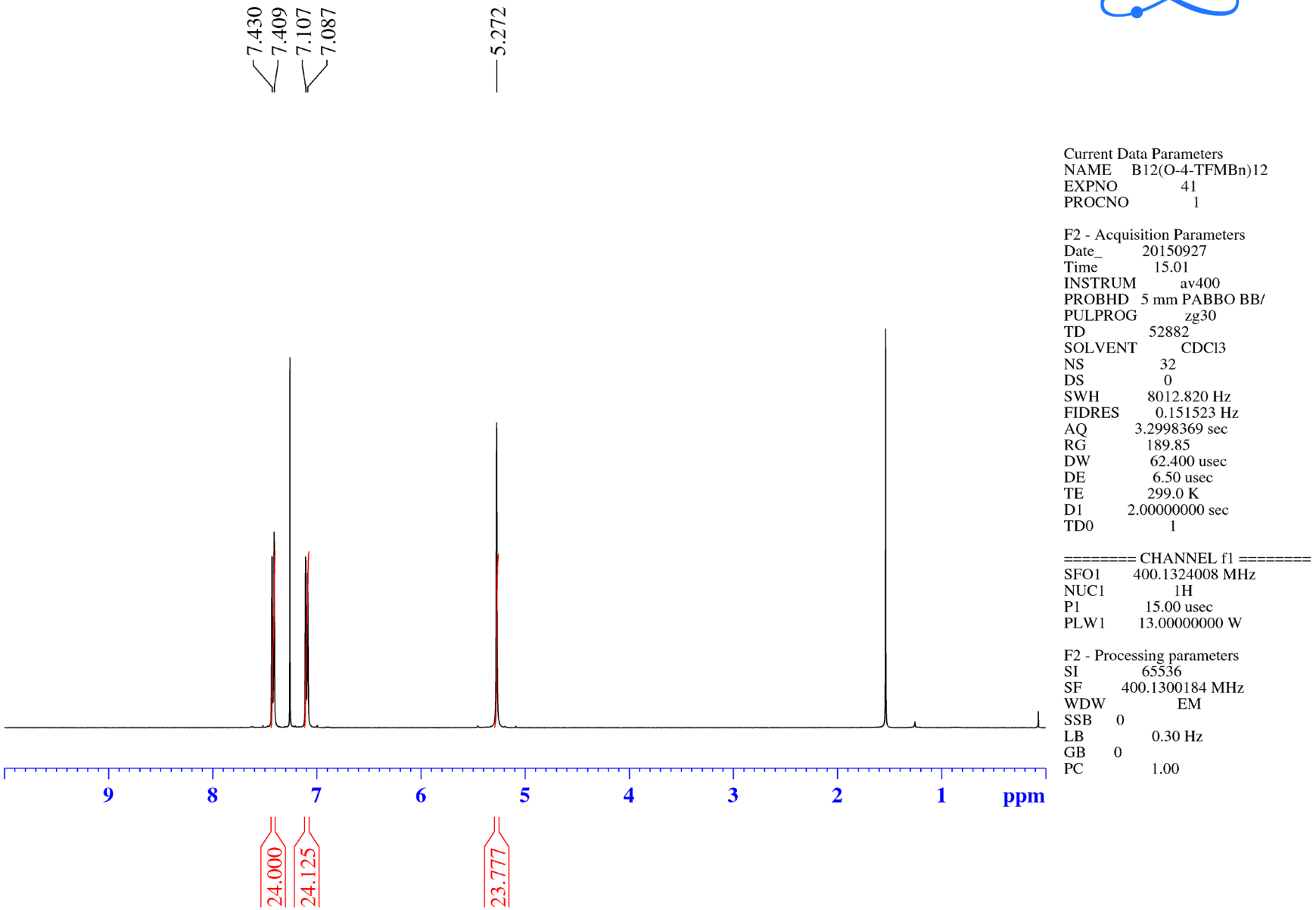
F2 - Acquisition Parameters
Date_ 20150927
Time 15.05
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgflqn30
TD 262144
SOLVENT CDCl3
NS 64
DS 0
SWH 150000.000 Hz
FIDRES 0.572205 Hz
AQ 0.8738133 sec
RG 189.85
DW 3.333 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 376.4983660 MHz
NUC1 19F
P1 14.50 usec
PLW1 17.00000000 W

F2 - Processing parameters
SI 262144
SF 376.4983660 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



^{1}H NMR

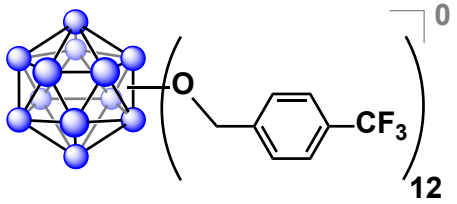


Current Data Parameters
NAME B12(O-4-TFMBn)12
EXPNO 41
PROCNO 1

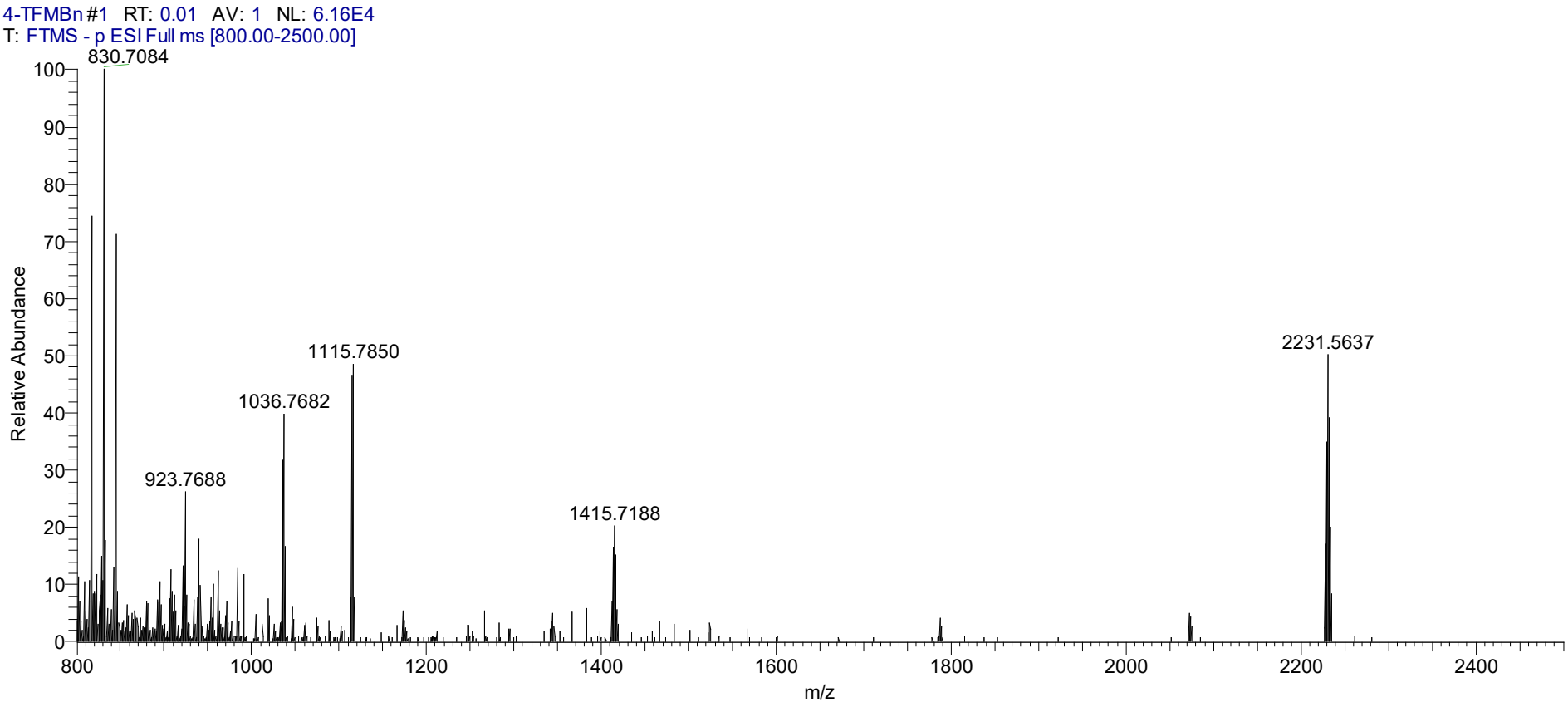
F2 - Acquisition Parameters
Date_ 20150927
Time 15.01
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 32
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 189.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

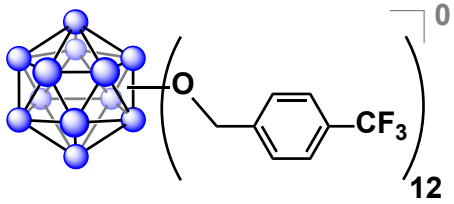
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300184 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

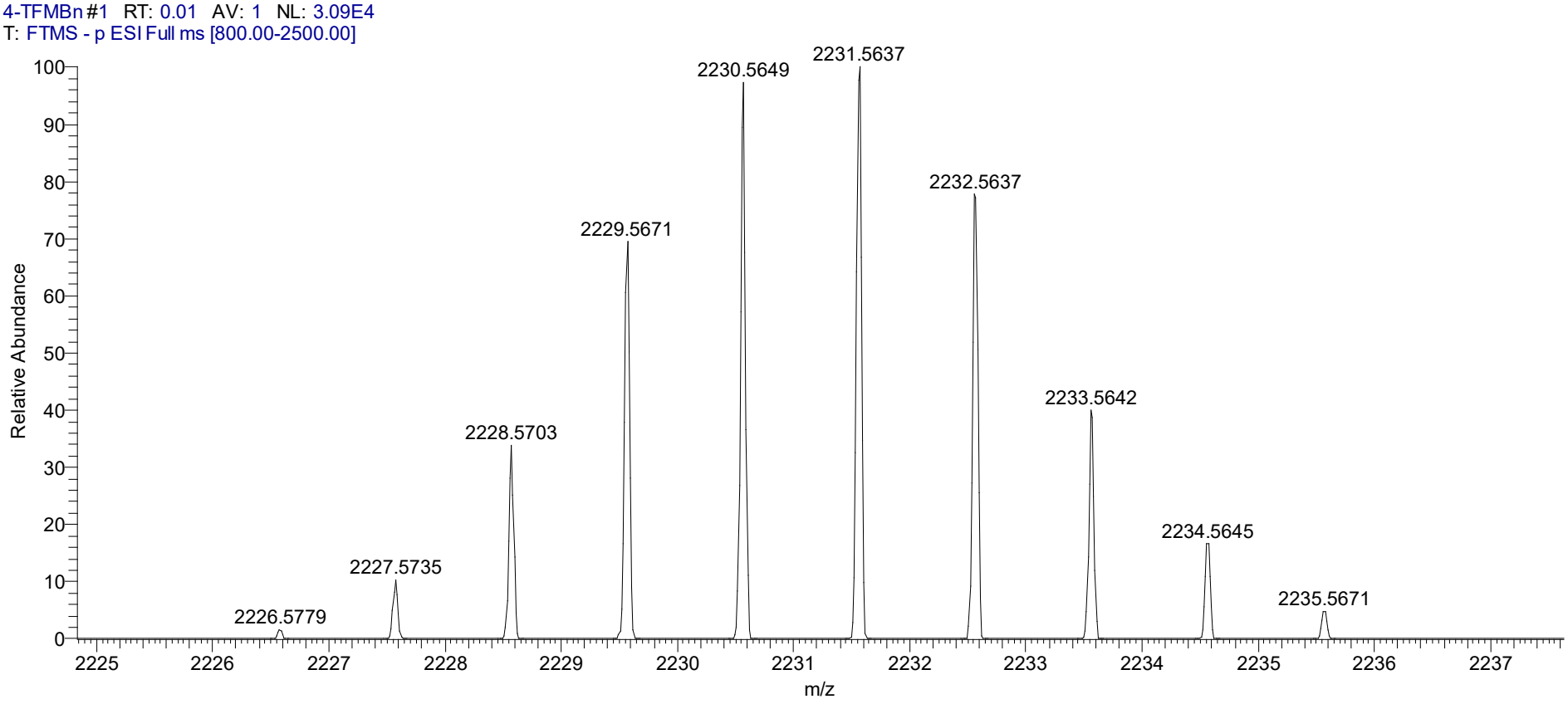


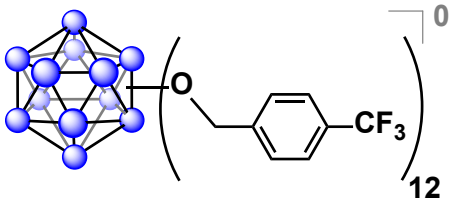
Q Exactive
High-Res Mass Spec



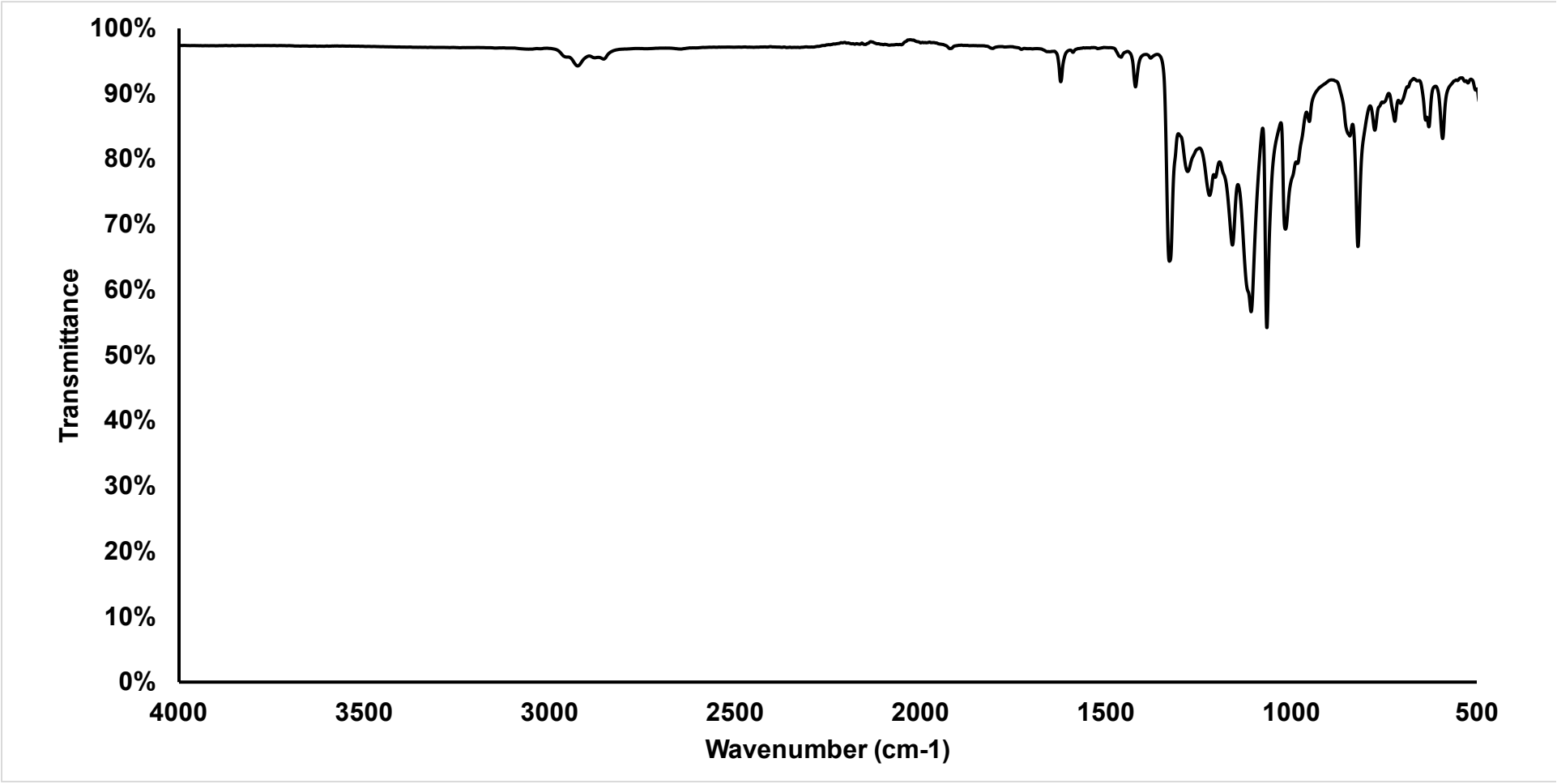


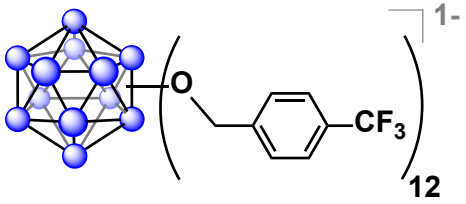
Q Exactive
High-Res Mass Spec



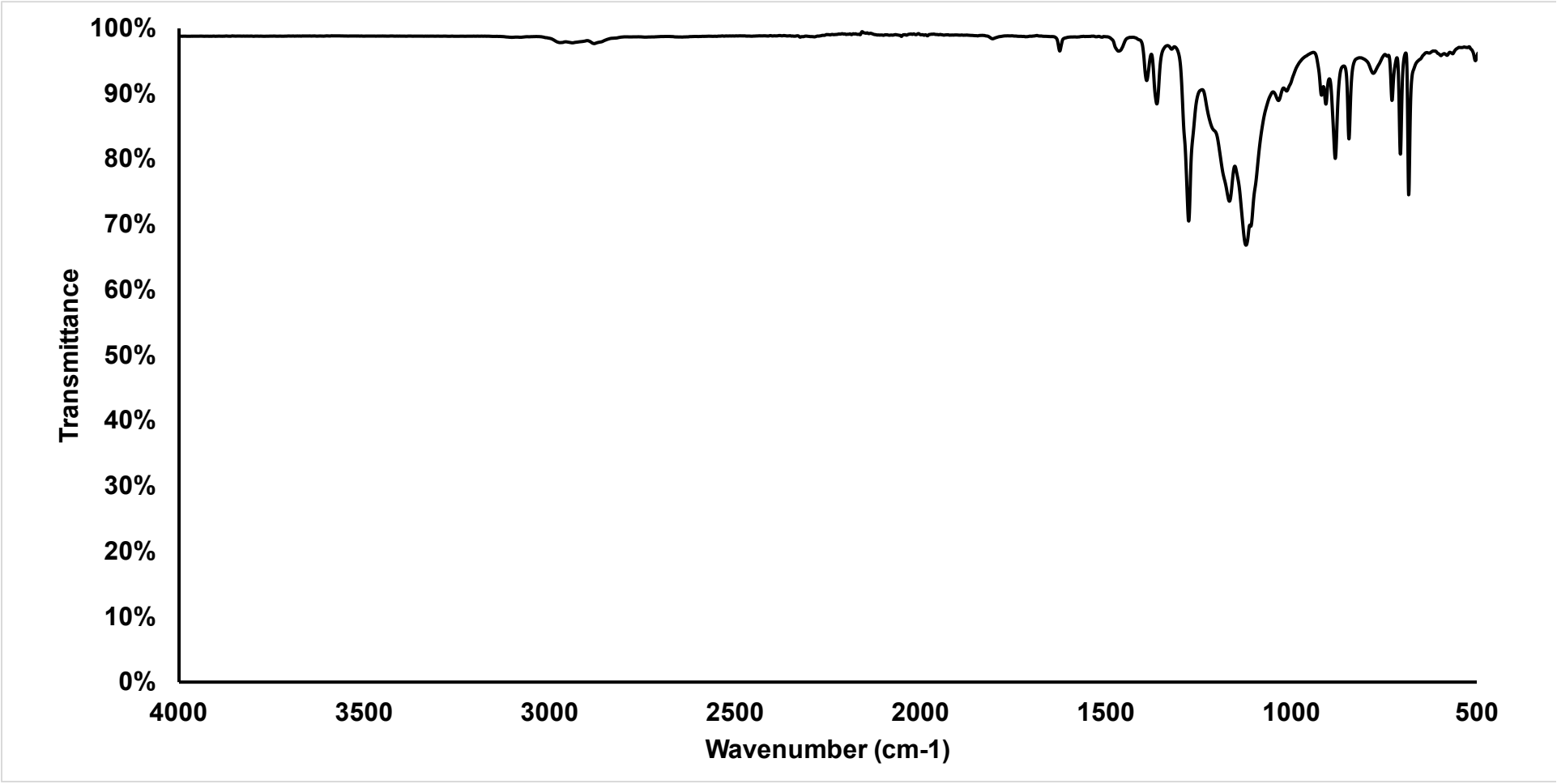

0
O
CF3
12

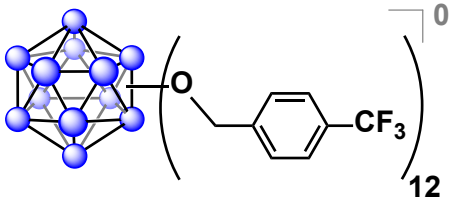
IR


100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
Transmittance
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)

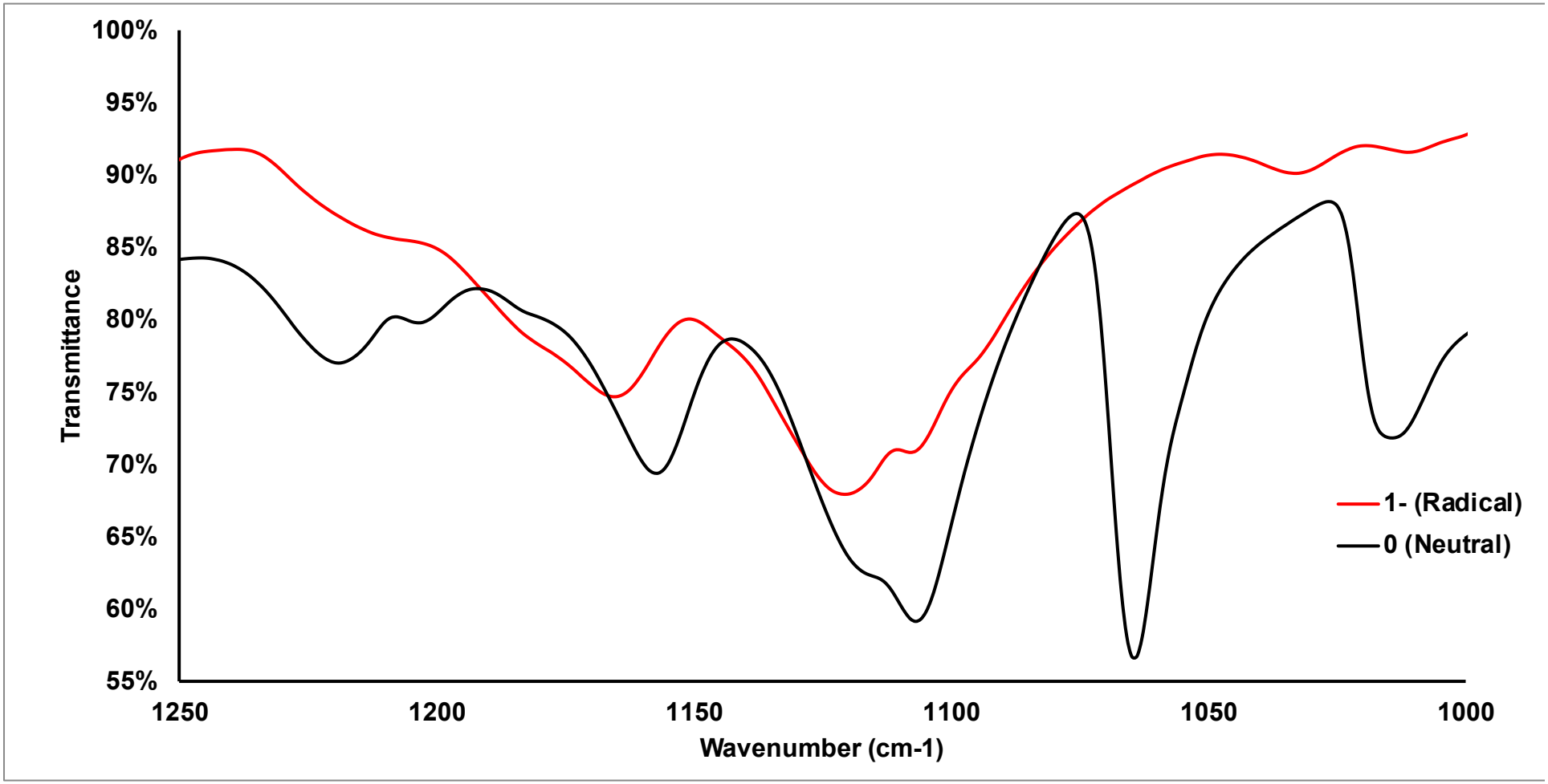

1-
O
CF3
12

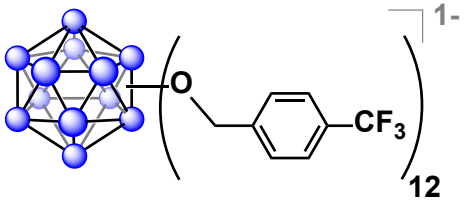
IR


100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
Transmittance
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)

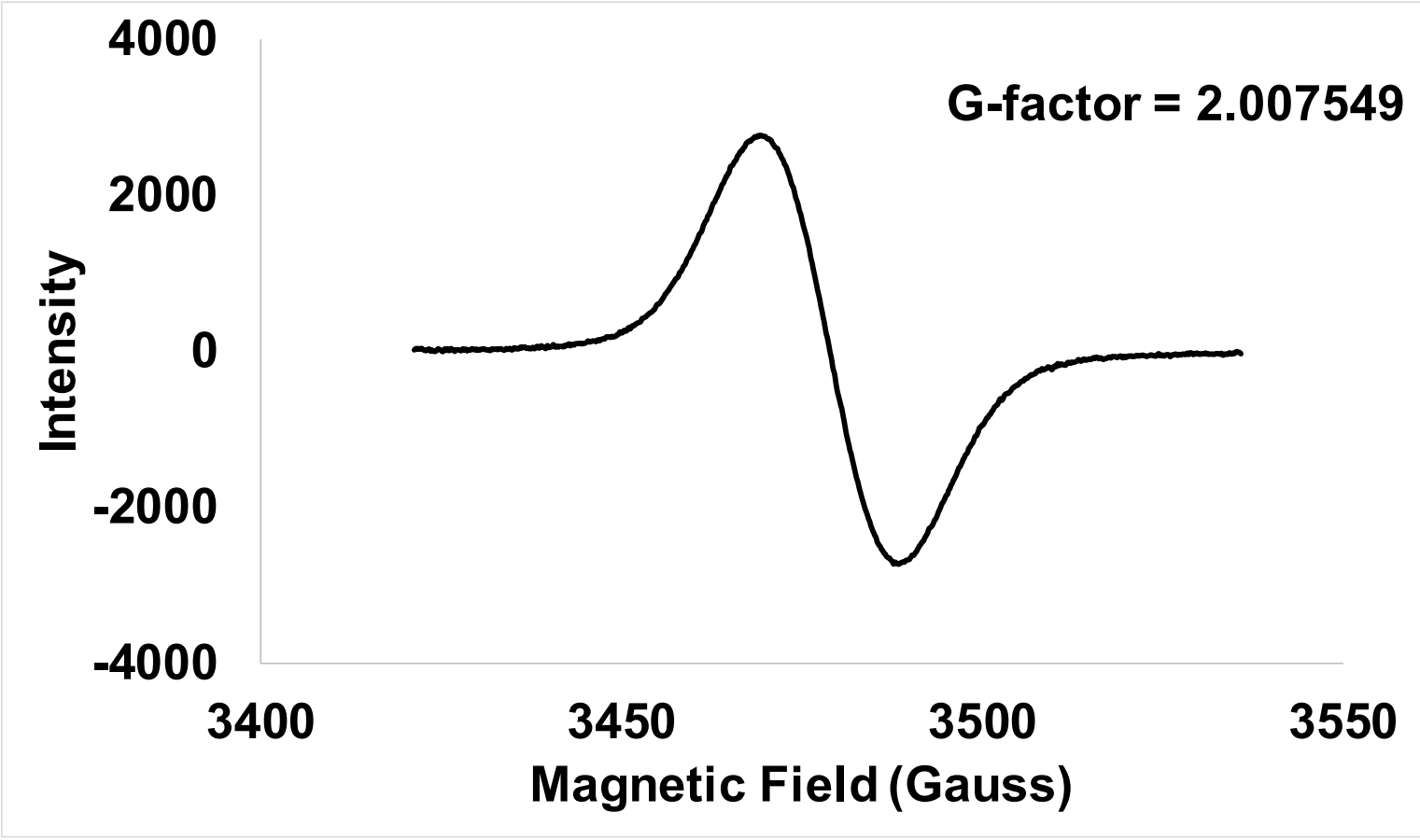

O
CF3
12
0

IR

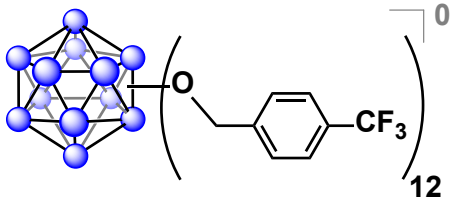

100%
95%
90%
85%
80%
75%
70%
65%
60%
55%
Transmittance
1250
1200
1150
1100
1050
1000
Wavenumber (cm-1)
1- (Radical)
0 (Neutral)



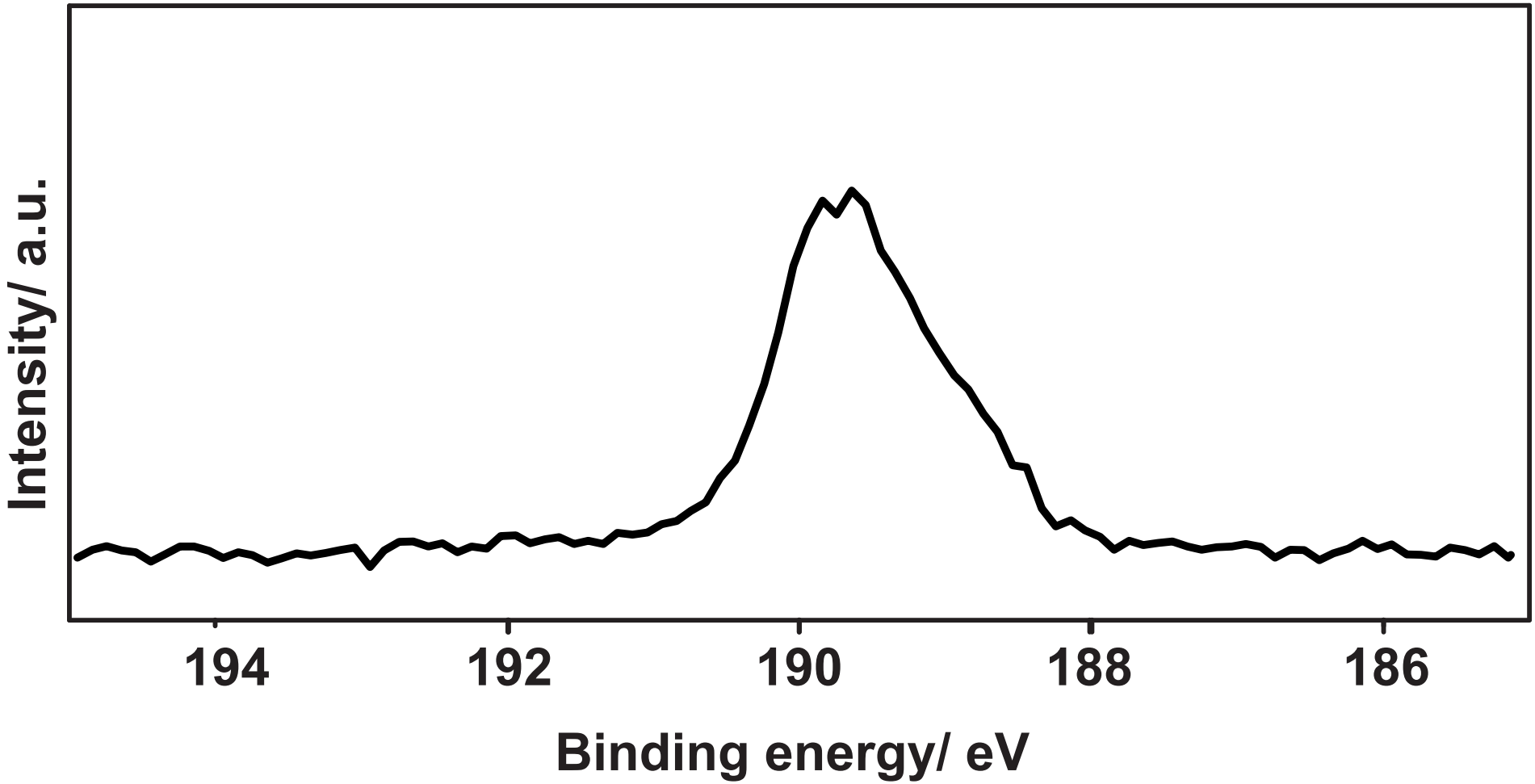
EPR

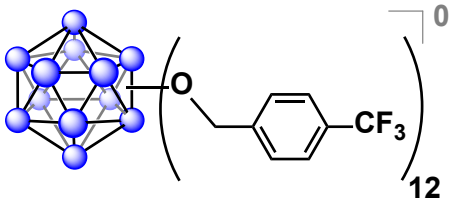


DOS	Format
ANZ	1024
MIN	-2733.186523
MAX	2772.813477
JSS	0
GST	3421.274884
GSI	114.543004
JUN	G
JON	Bruker BioSpin GmbH
JDA	9/25/2015
JTM	13:33
JRE	c:\programfiles\bruker-emx\syscal\st0103.cal
JEX	field-sweep
JSD	1
HCF	3478.546386
HSW	114.543004
EMF	0
RCT	20.48
RTC	20.48
RRG	8.93E+03
RMA	4
MF	9.773981
MP	6.38E-01
MPD	25

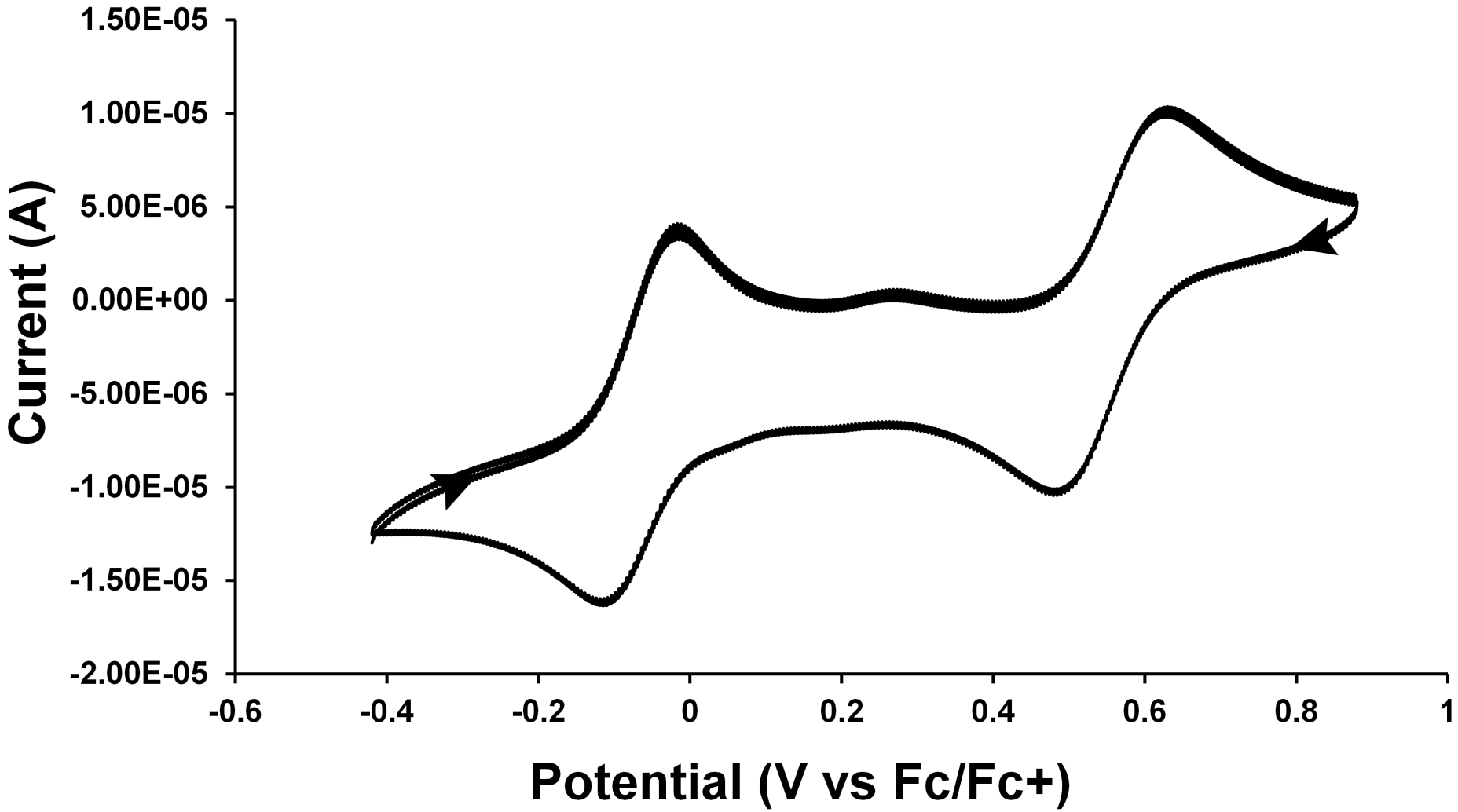


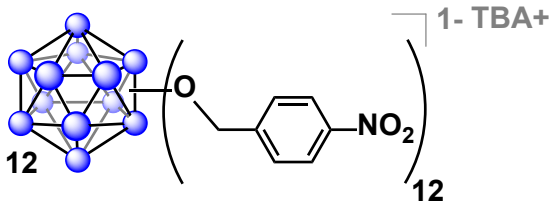
B 1s XPS



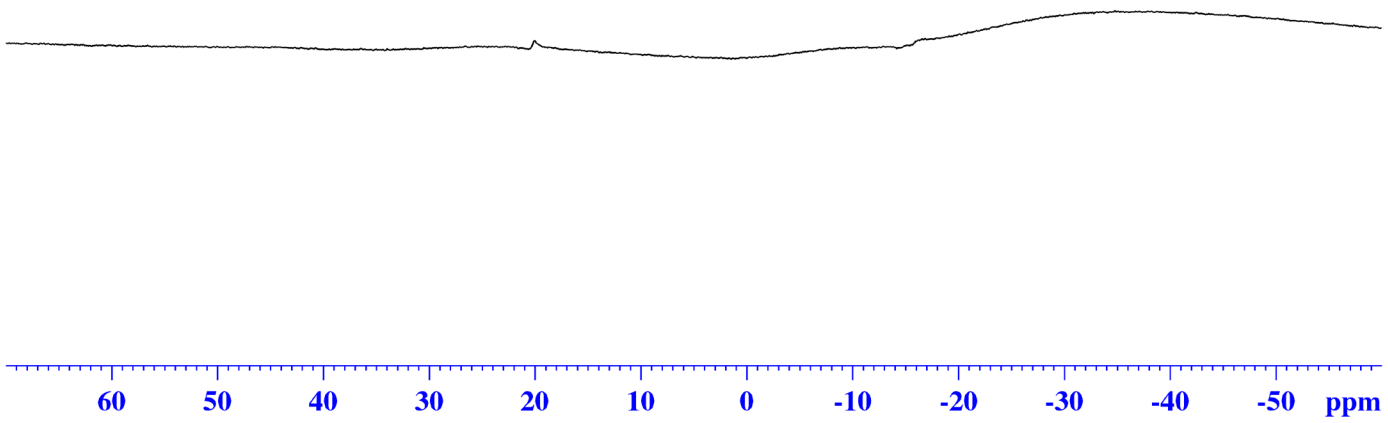


Cyclic voltammetry





^{11}B {^{1}H} NMR



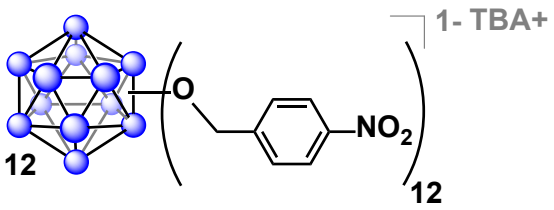
Current Data Parameters
NAME B12(O-4-NO2Bn)12
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151101
Time 19.40
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT Acetone
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.0 K
D1 0.05000000 sec
D11 0.03000000 sec
TD0 1

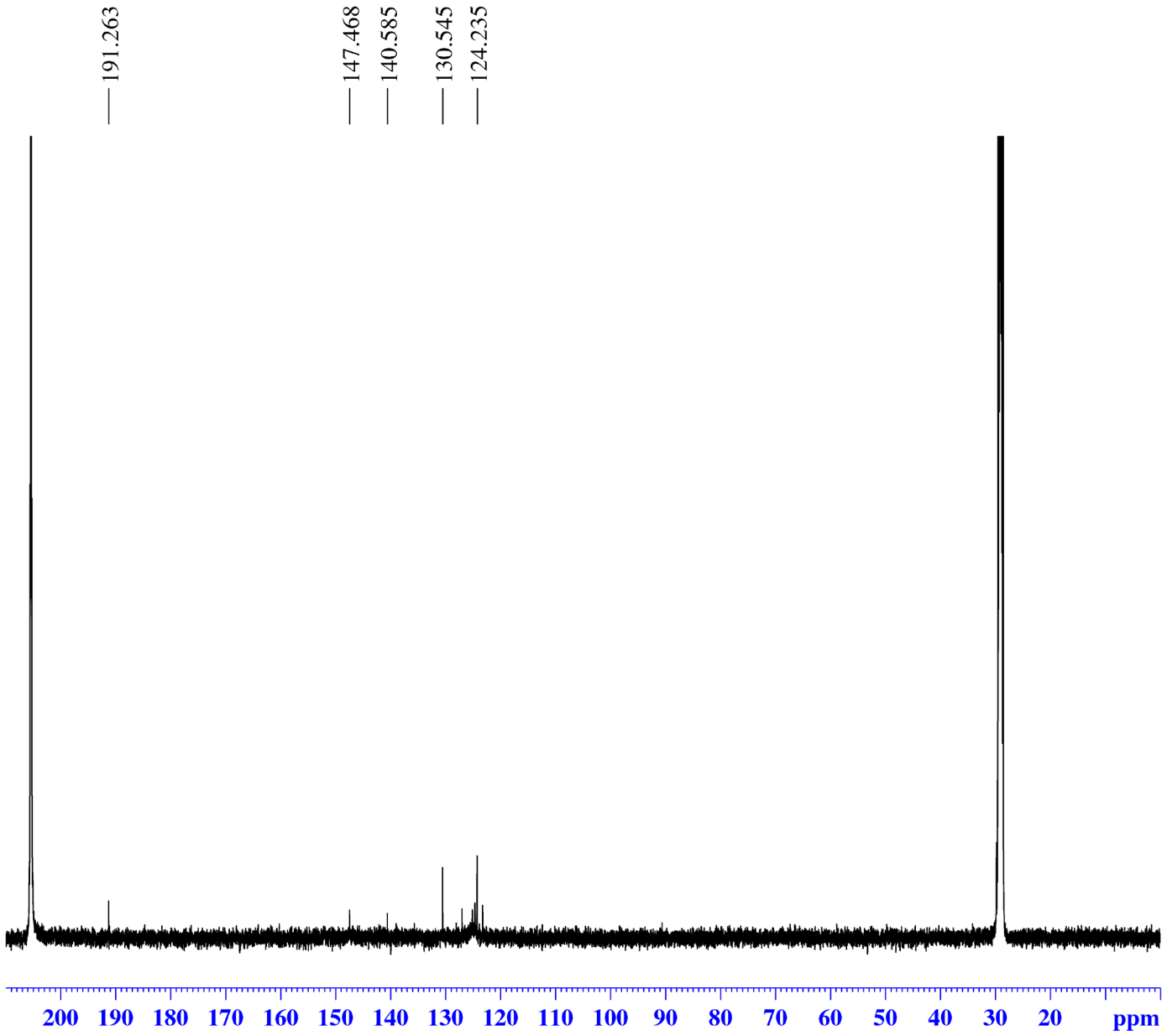
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776161 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 1.40



^{13}C NMR



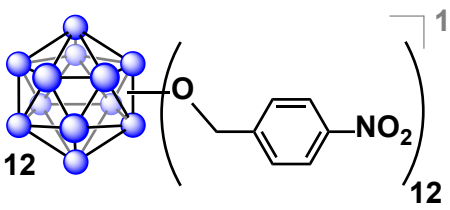
Current Data Parameters
NAME B12(O-4-NO2Bn)12
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151103
Time 11.12
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 128
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

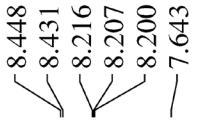
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

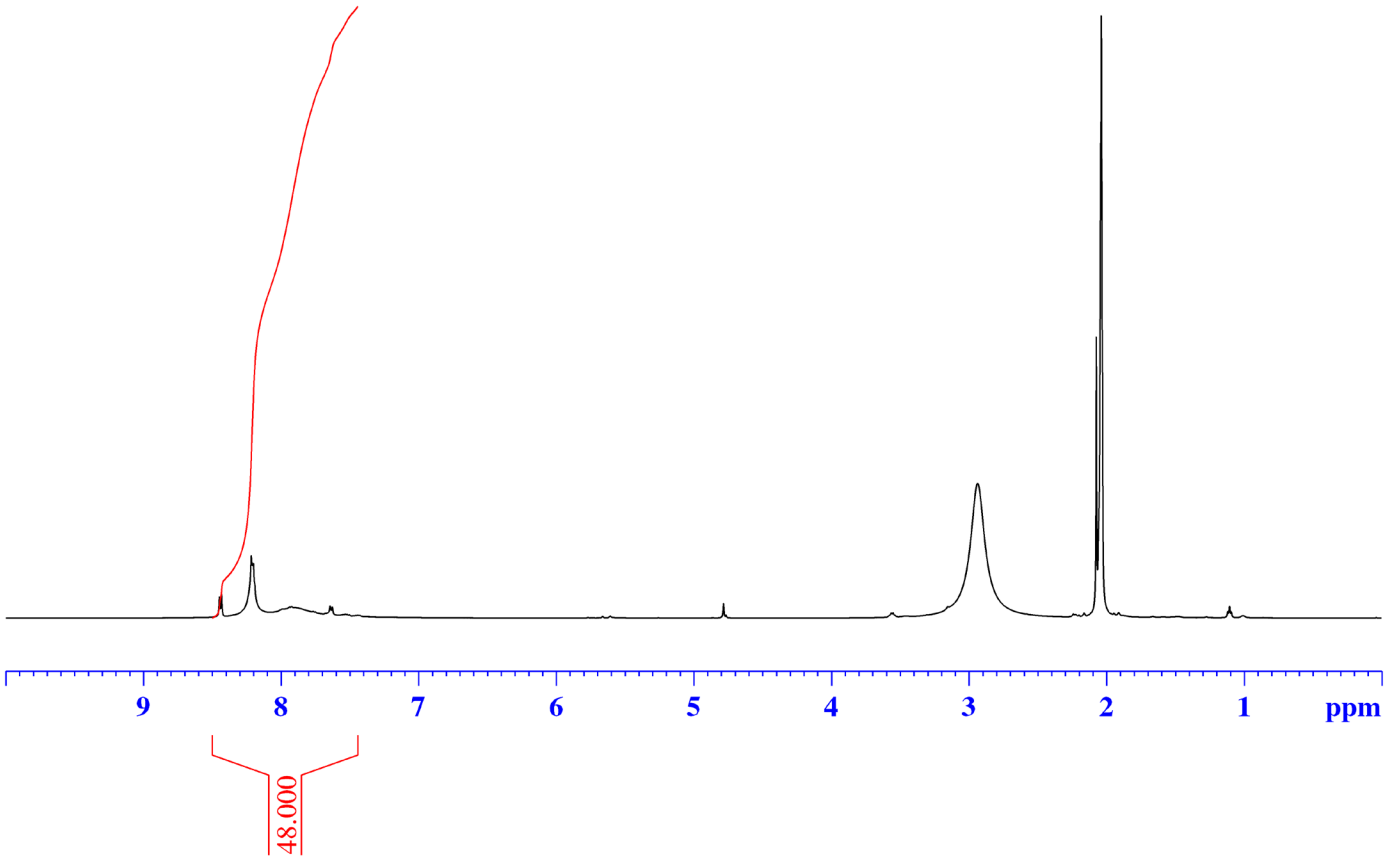
======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR



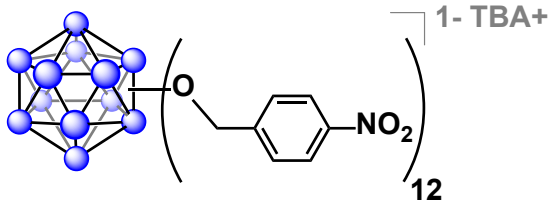


Current Data Parameters
NAME B12(O-4-NO2Bn)12
EXPNO 11
PROCNO 1

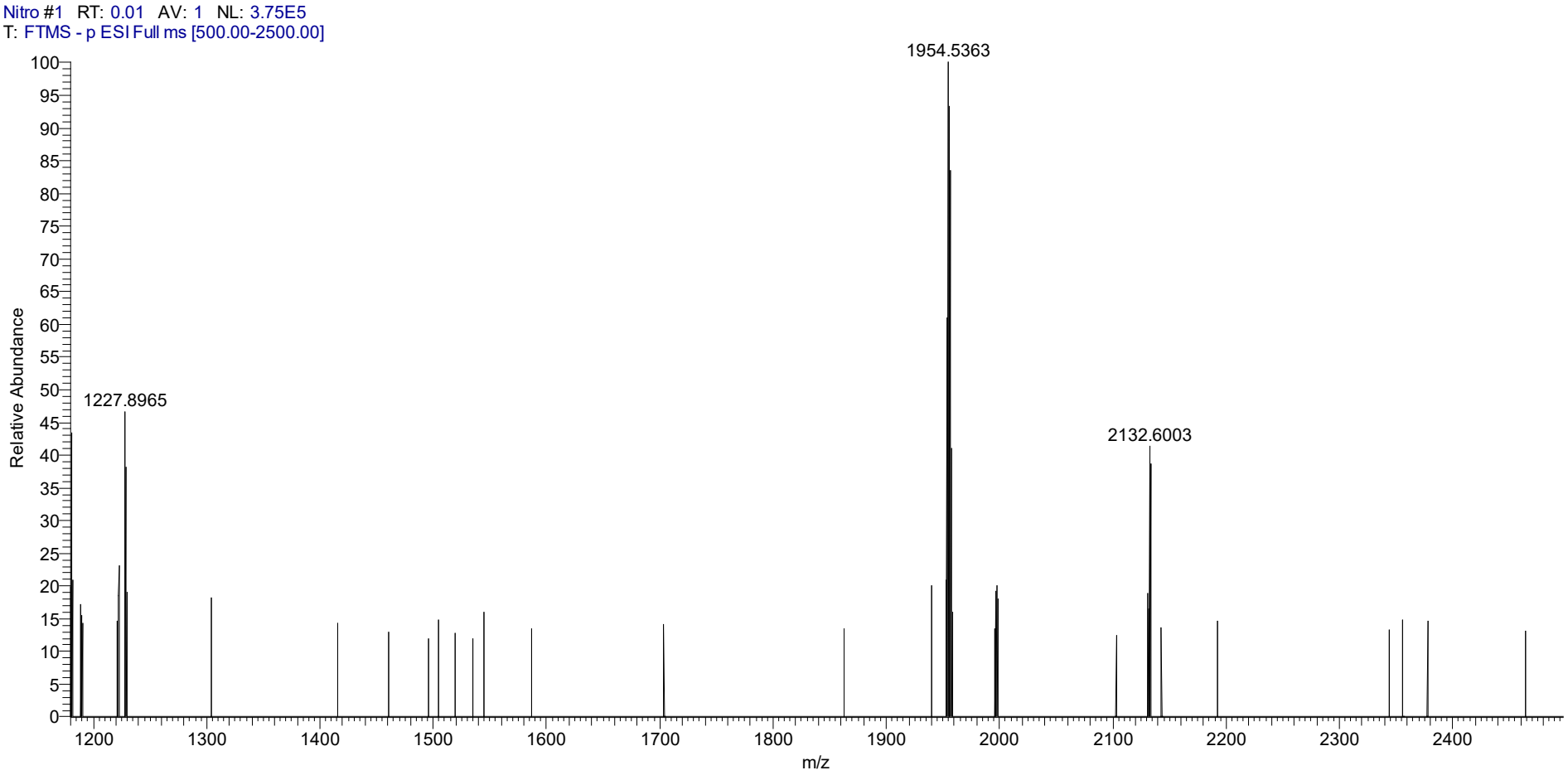
F2 - Acquisition Parameters
Date_ 20151103
Time 11.16
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 32
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 52.41
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

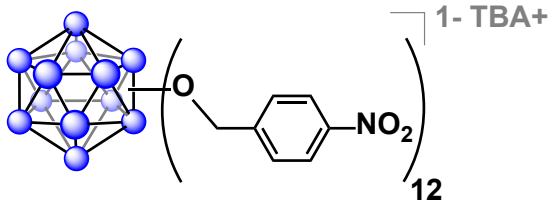
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

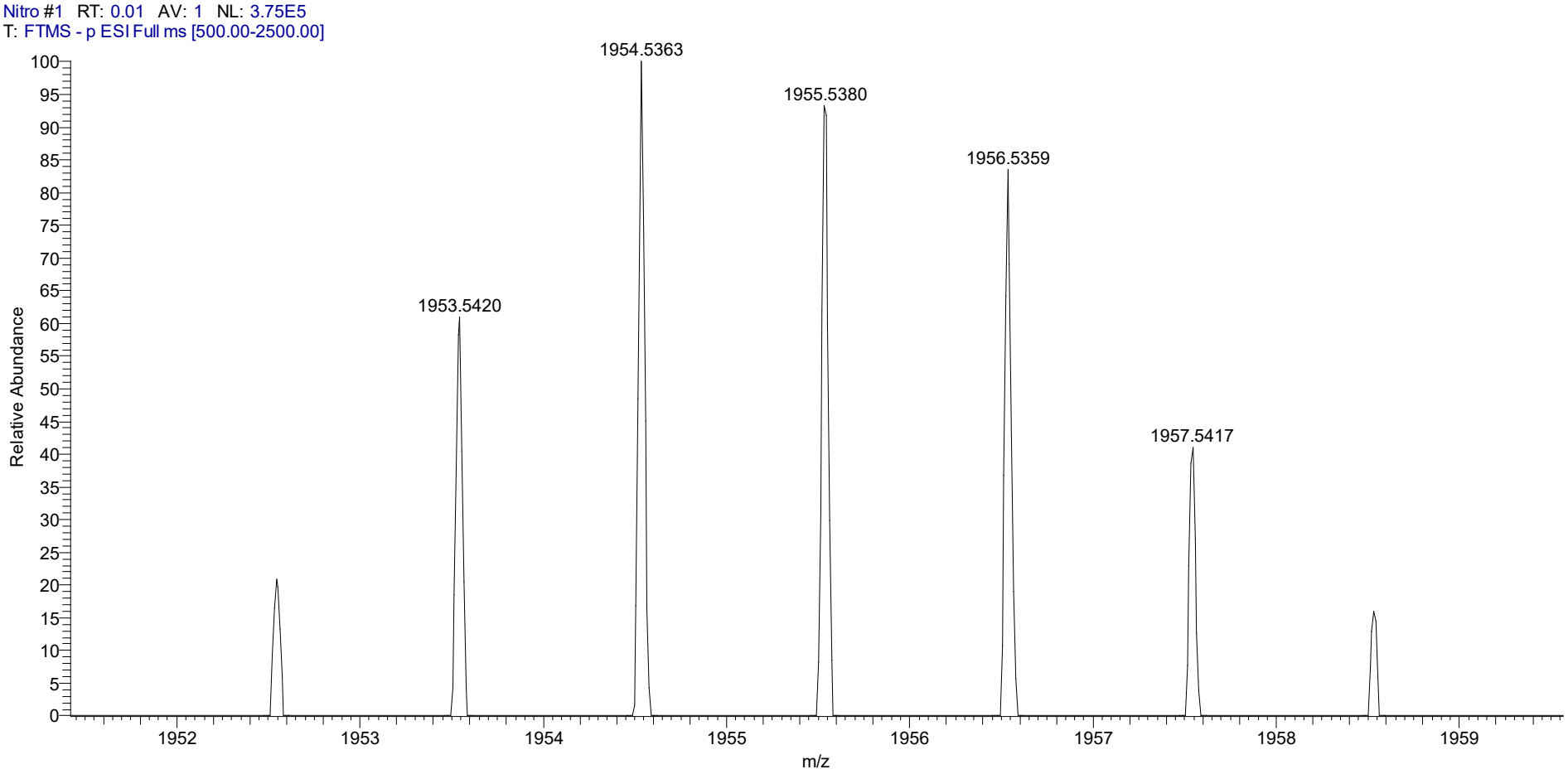


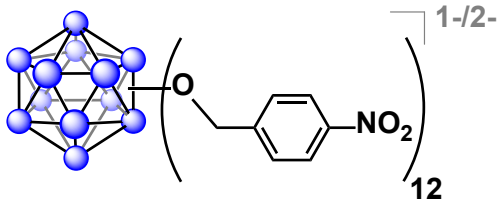
Q Exactive
High-Res Mass Spec



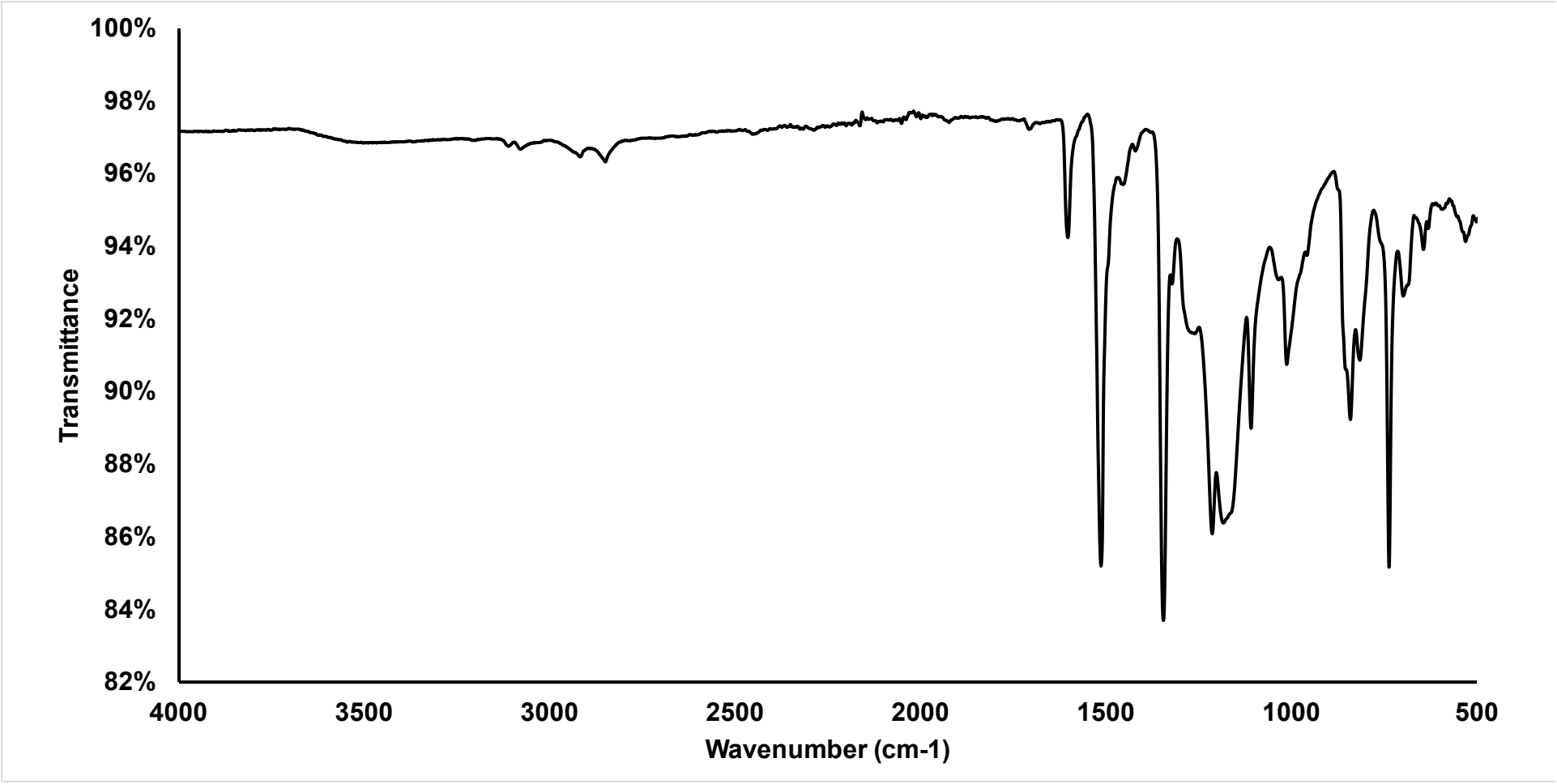


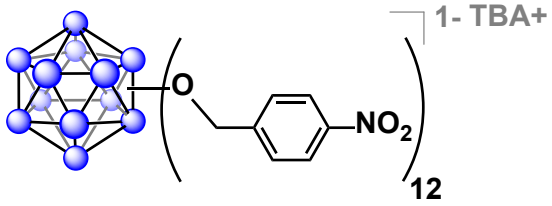
Q Exactive
High-Res Mass Spec



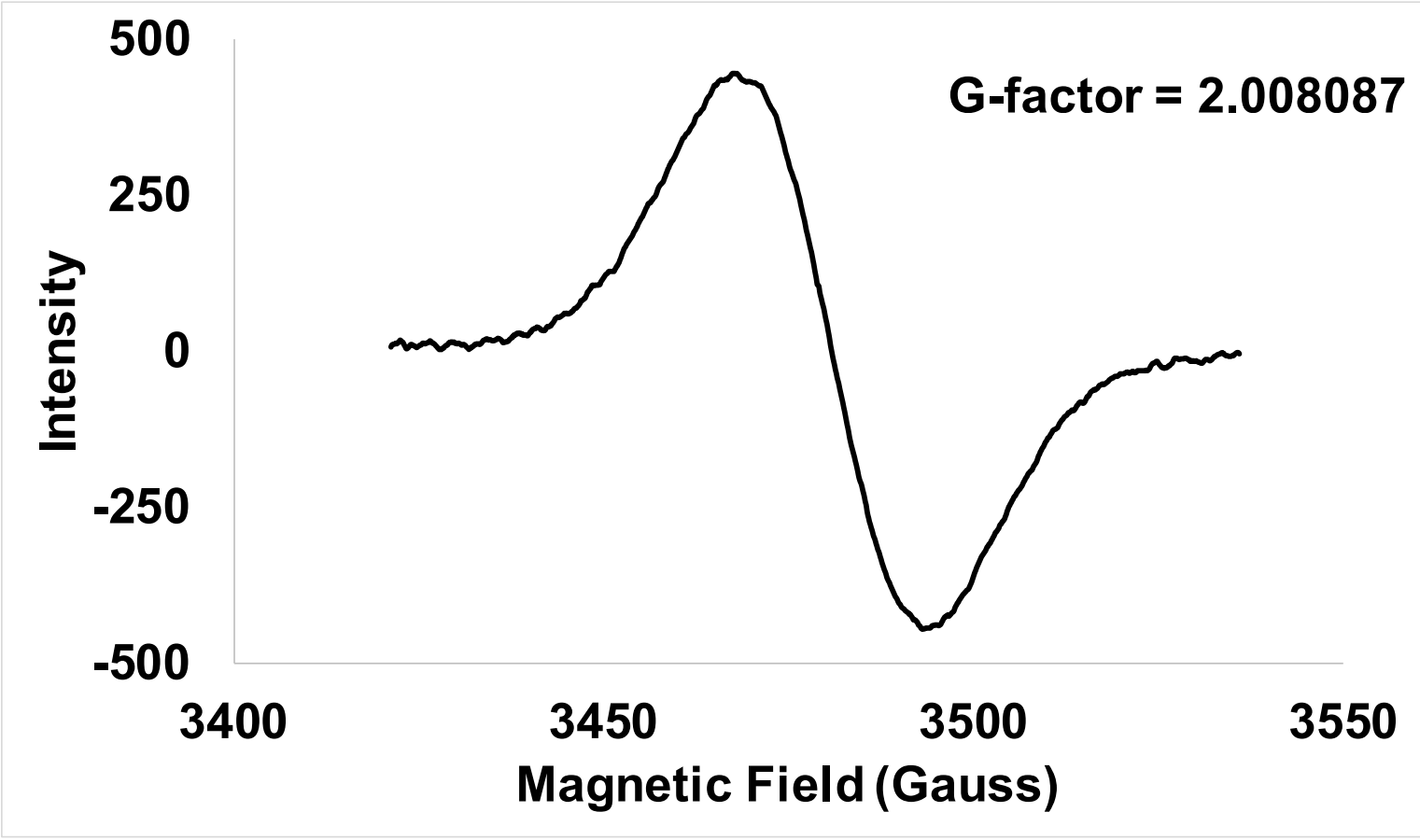

1-/2-
O
NO2
12

IR

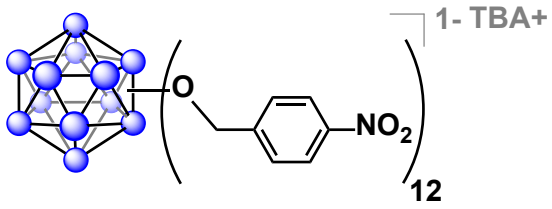

100%
98%
96%
94%
92%
90%
88%
86%
84%
82%
Transmittance
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)



EPR

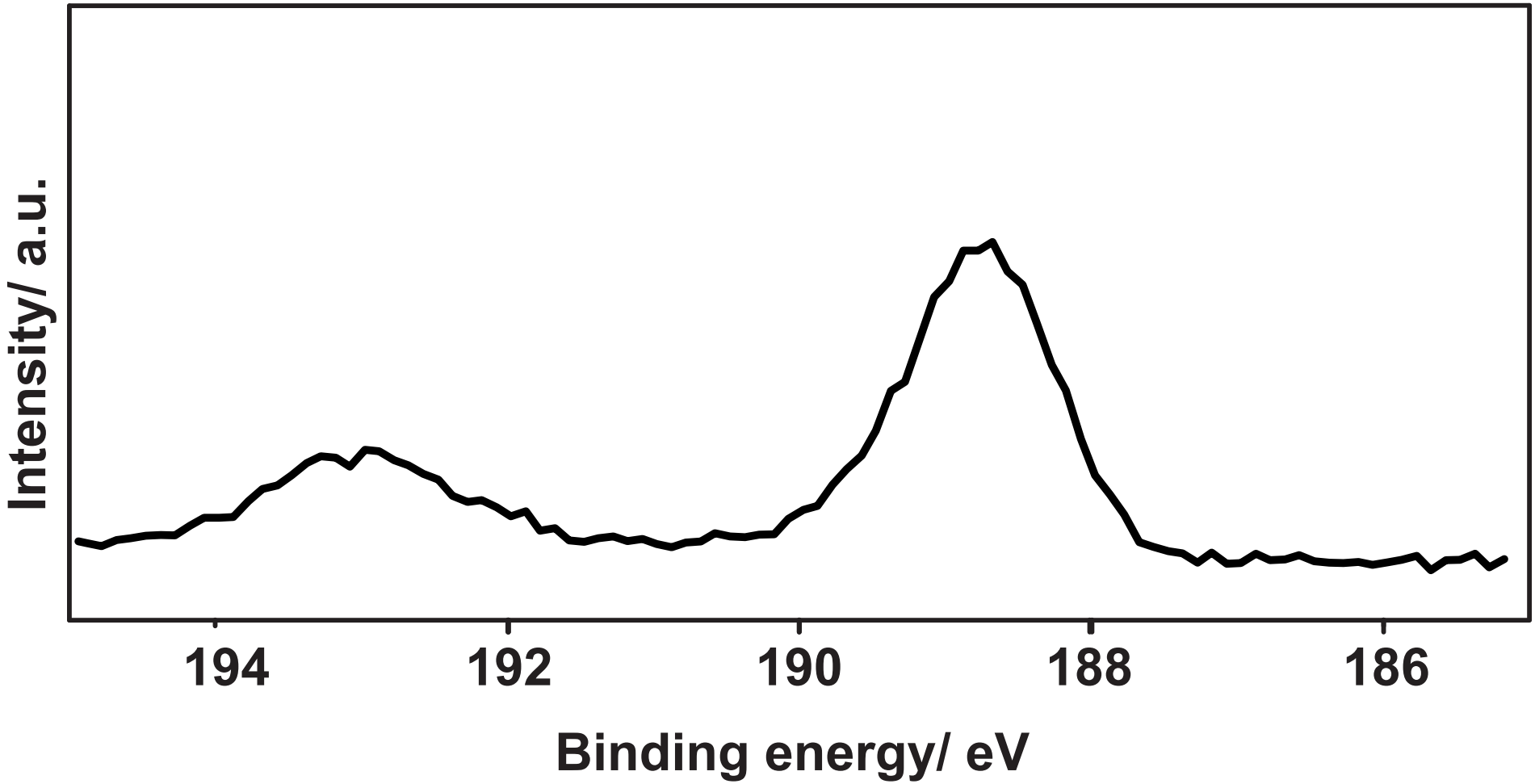


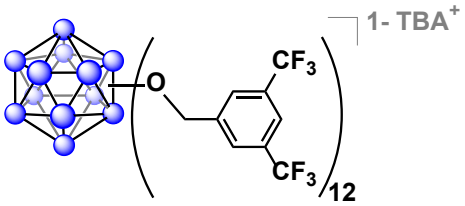
DOS	Format
ANZ	1024
MIN	-445.813477
MAX	444.186523
JSS	0
GST	3421.28
GSI	114.54
JUN	G
JON	Bruker BioSpin GmbH
JDA	9/29/2015
JTM	16:26
JRE	c:\programfiles\bruker-emx\syscal\st0103.cal
JEX	field-sweep
JSD	1
HCF	3478.55
HSW	114.54
EMF	0
RCT	20.48
RTC	163.84
RRG	8.93E+03
RMA	4
MF	9.776609
MP	6.38E-01
MPD	25



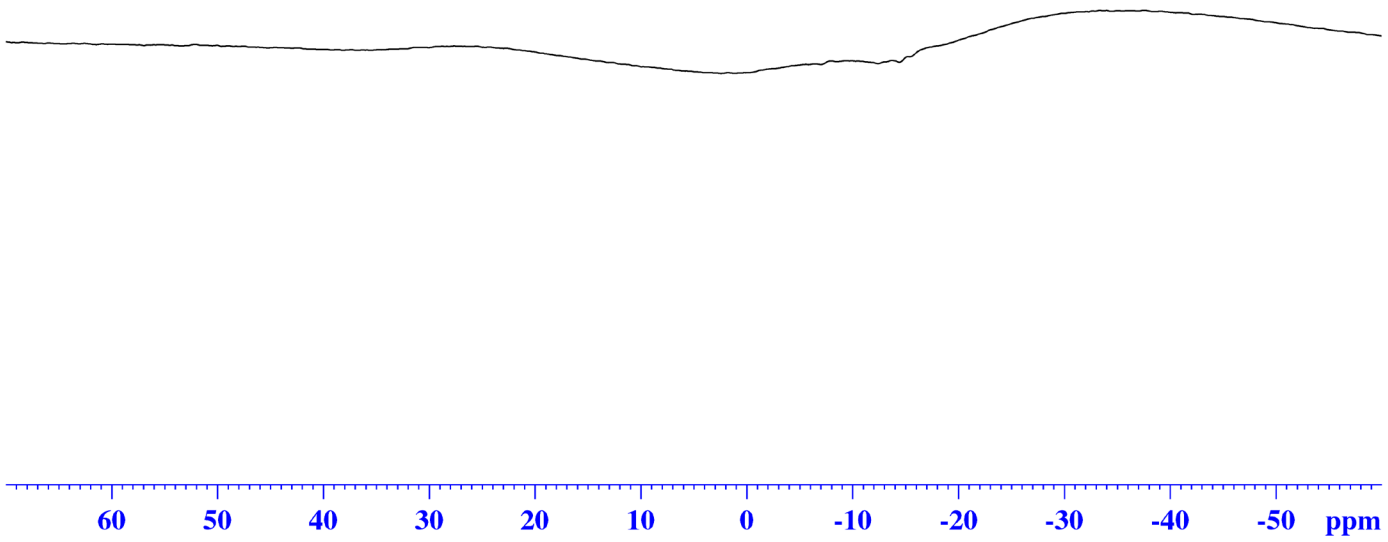
B 1s XPS

BRUKER





^{11}B {^{1}H} NMR



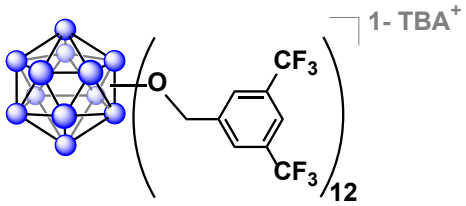
Current Data Parameters
NAME B12(O-3,5-bisTFMBn)12
EXPNO 210
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150824
Time 20.51
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.2 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

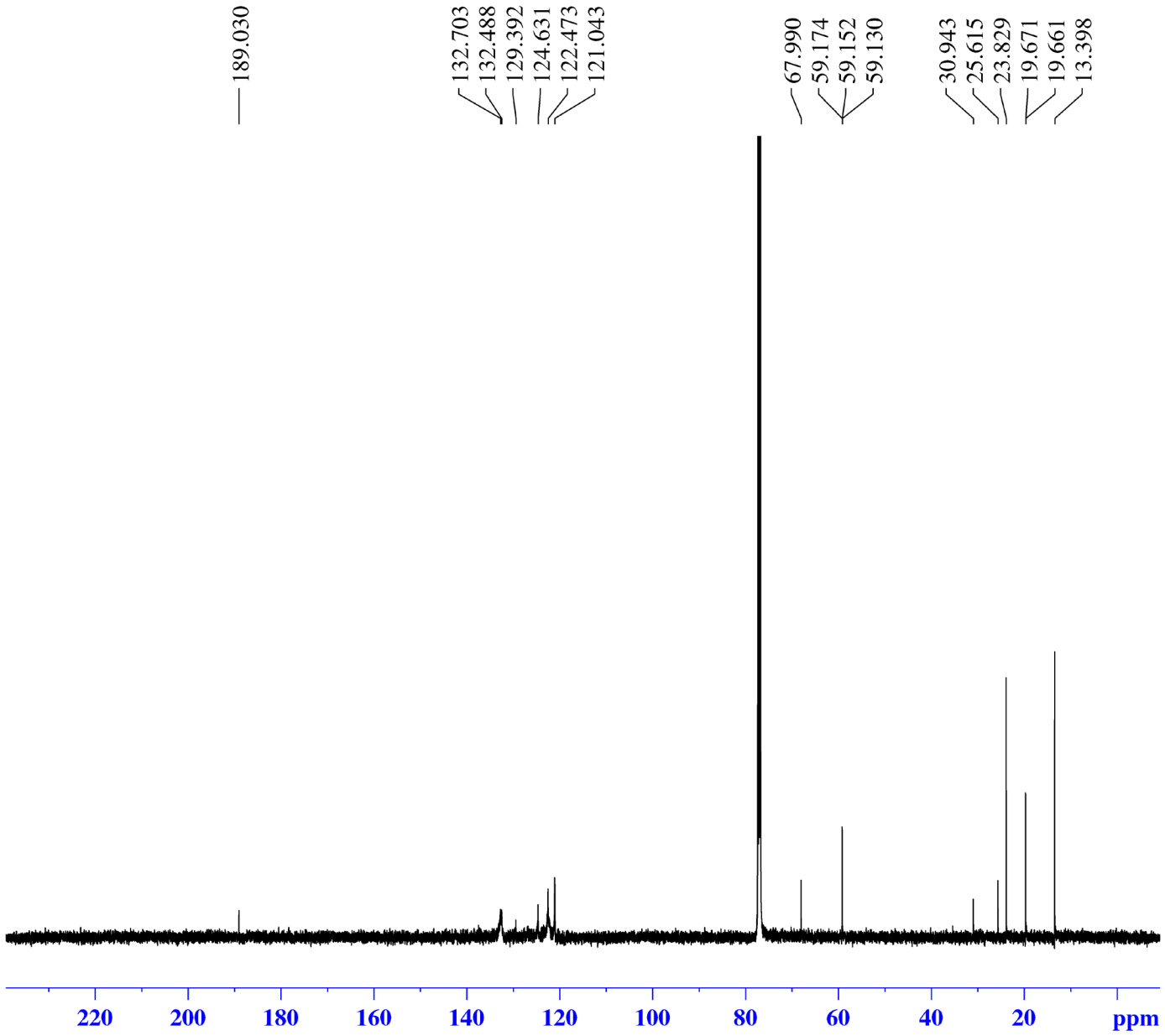
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40



^{13}C NMR



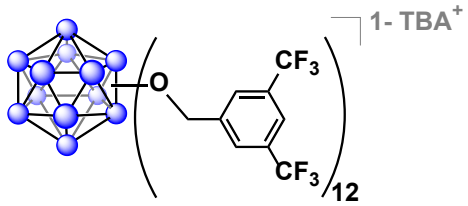
Current Data Parameters
NAME B12(O-3,5-bisTFMBn)12
EXPNO 300
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150829
Time 21.11
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

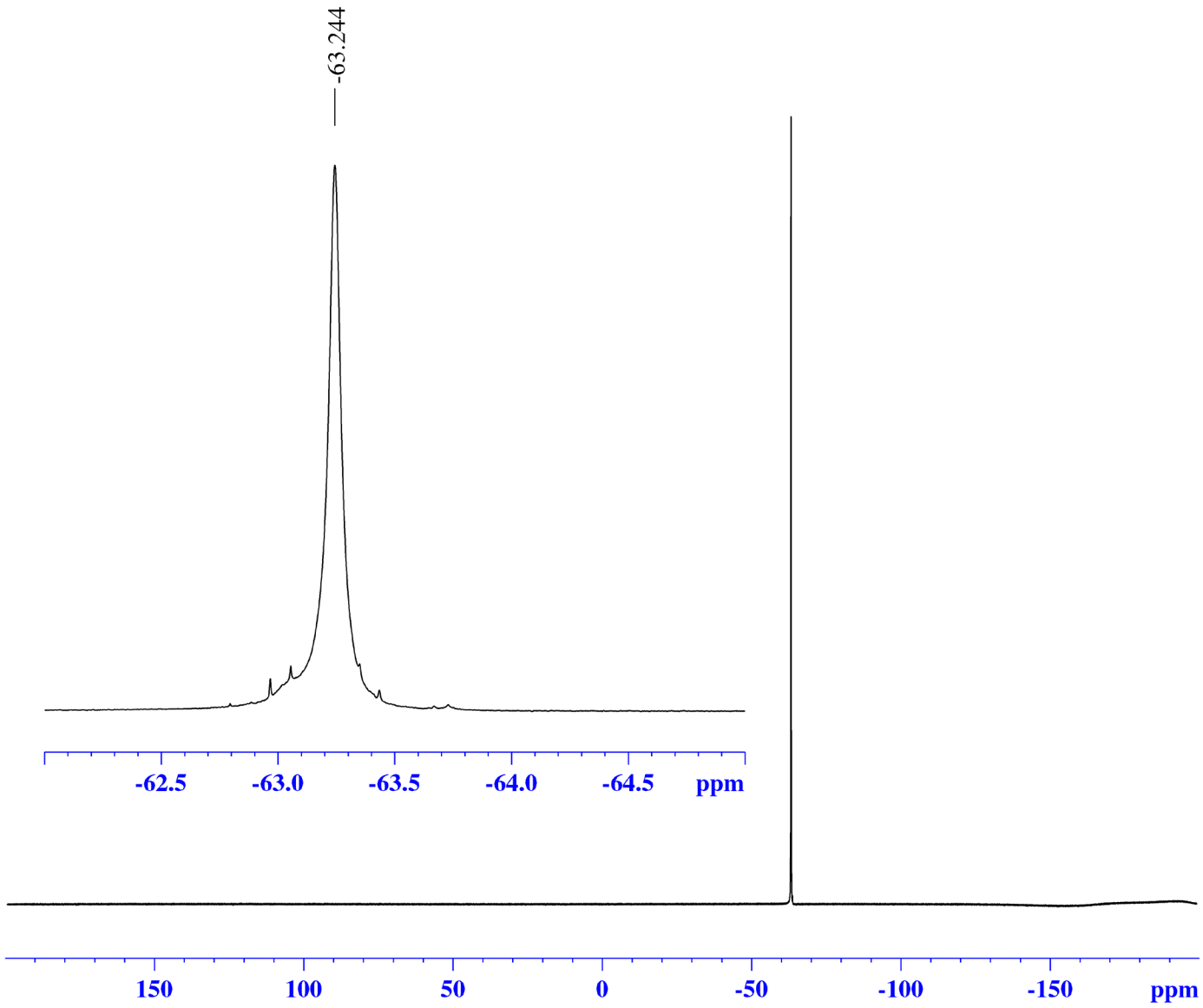
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{19}F NMR

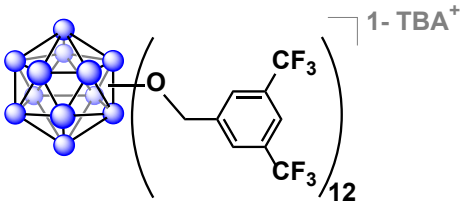


Current Data Parameters
NAME B12(O-3,5-bisTFMBn)12
EXPNO 212
PROCNO 1

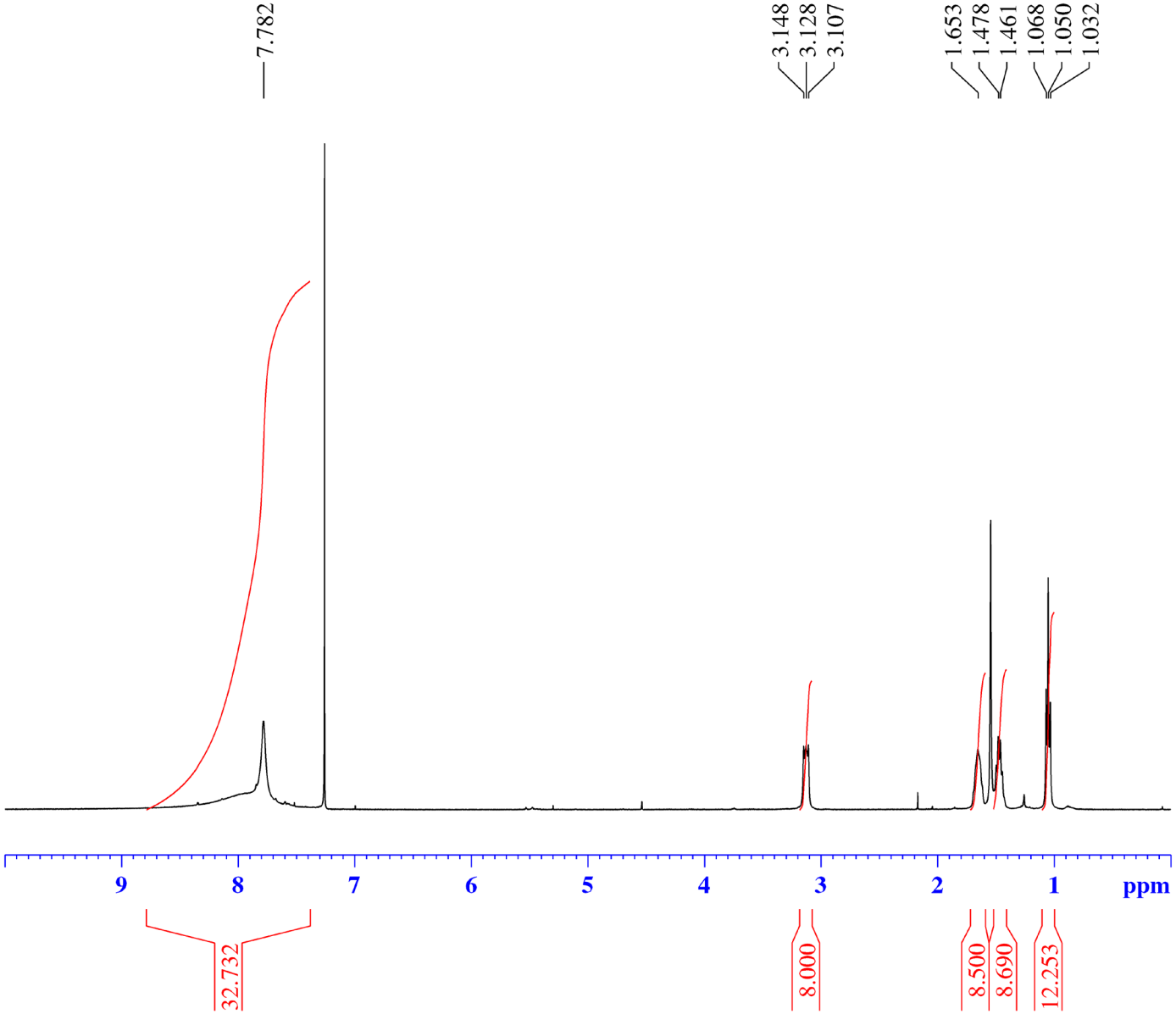
F2 - Acquisition Parameters
Date_ 20150824
Time 21.00
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgflqn30
TD 262144
SOLVENT CDCl3
NS 64
DS 0
SWH 150000.000 Hz
FIDRES 0.572205 Hz
AQ 0.8738133 sec
RG 189.85
DW 3.333 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 376.4983660 MHz
NUC1 19F
P1 14.50 usec
PLW1 17.00000000 W

F2 - Processing parameters
SI 262144
SF 376.4983660 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



^{1}H NMR

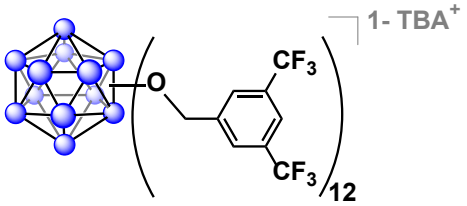


Current Data Parameters
NAME B12(O-3,5-bisTFMBn)12
EXPNO 211
PROCNO 1

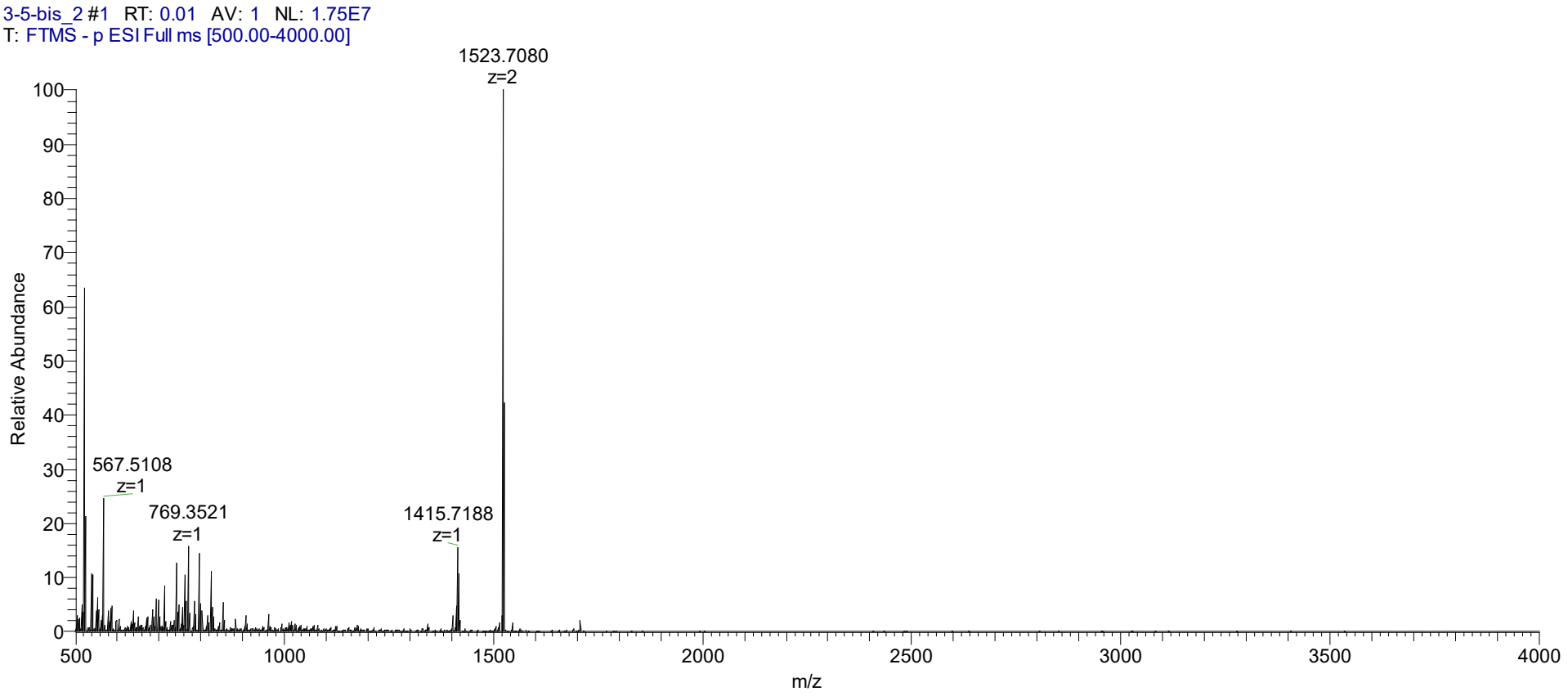
F2 - Acquisition Parameters
Date_ 20150824
Time 20.54
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 32
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 189.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

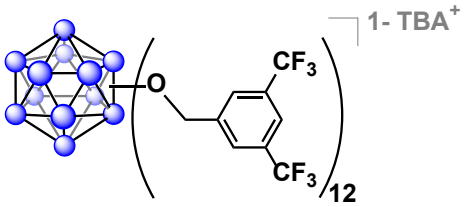
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300184 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



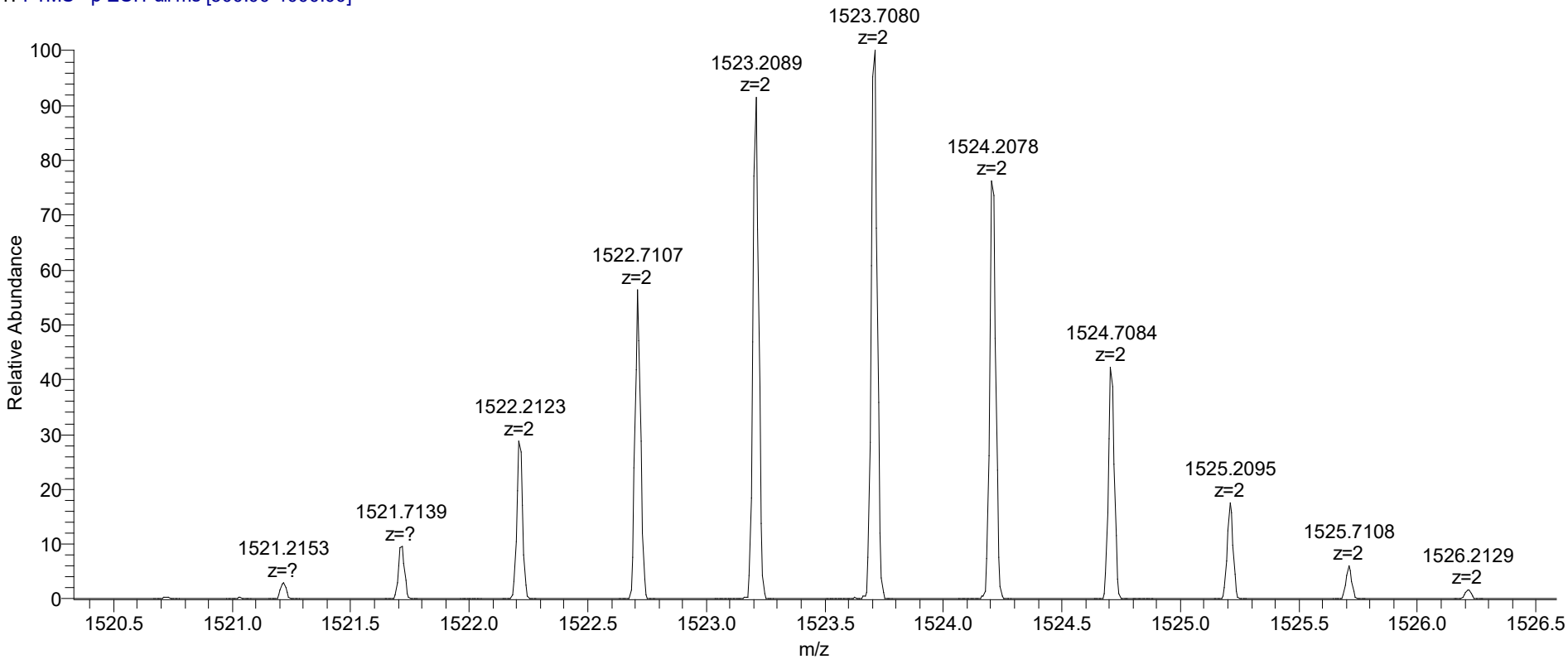
Q Exactive
High-Res Mass Spec

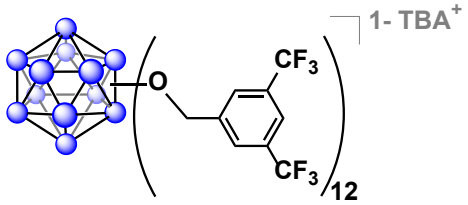




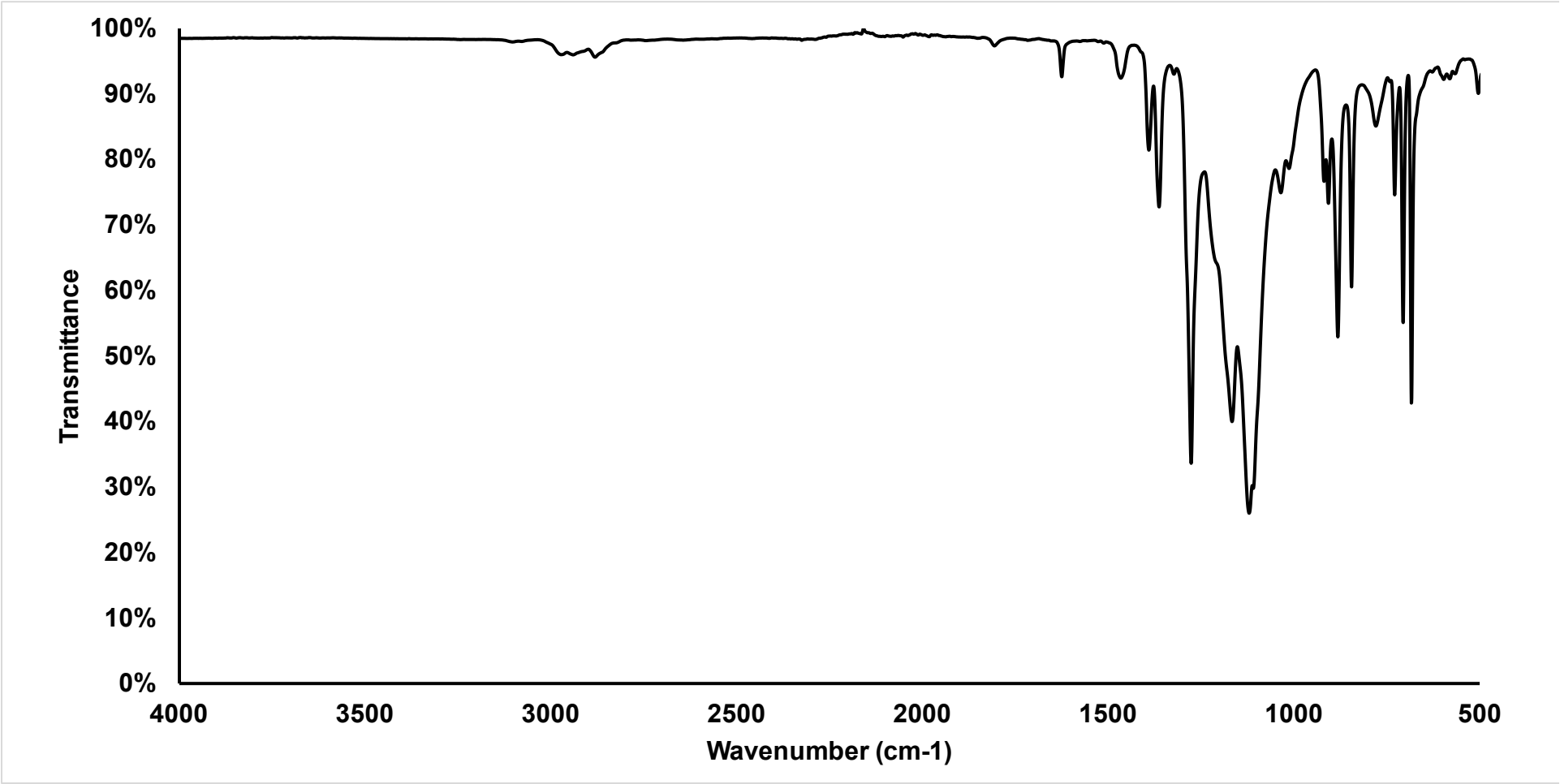
Q Exactive
High-Res Mass Spec

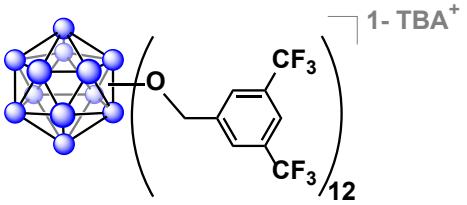
3-5-bis_2 #1 RT: 0.01 AV: 1 NL: 1.75E7
T: FTMS - p ESI Full ms [500.00-4000.00]



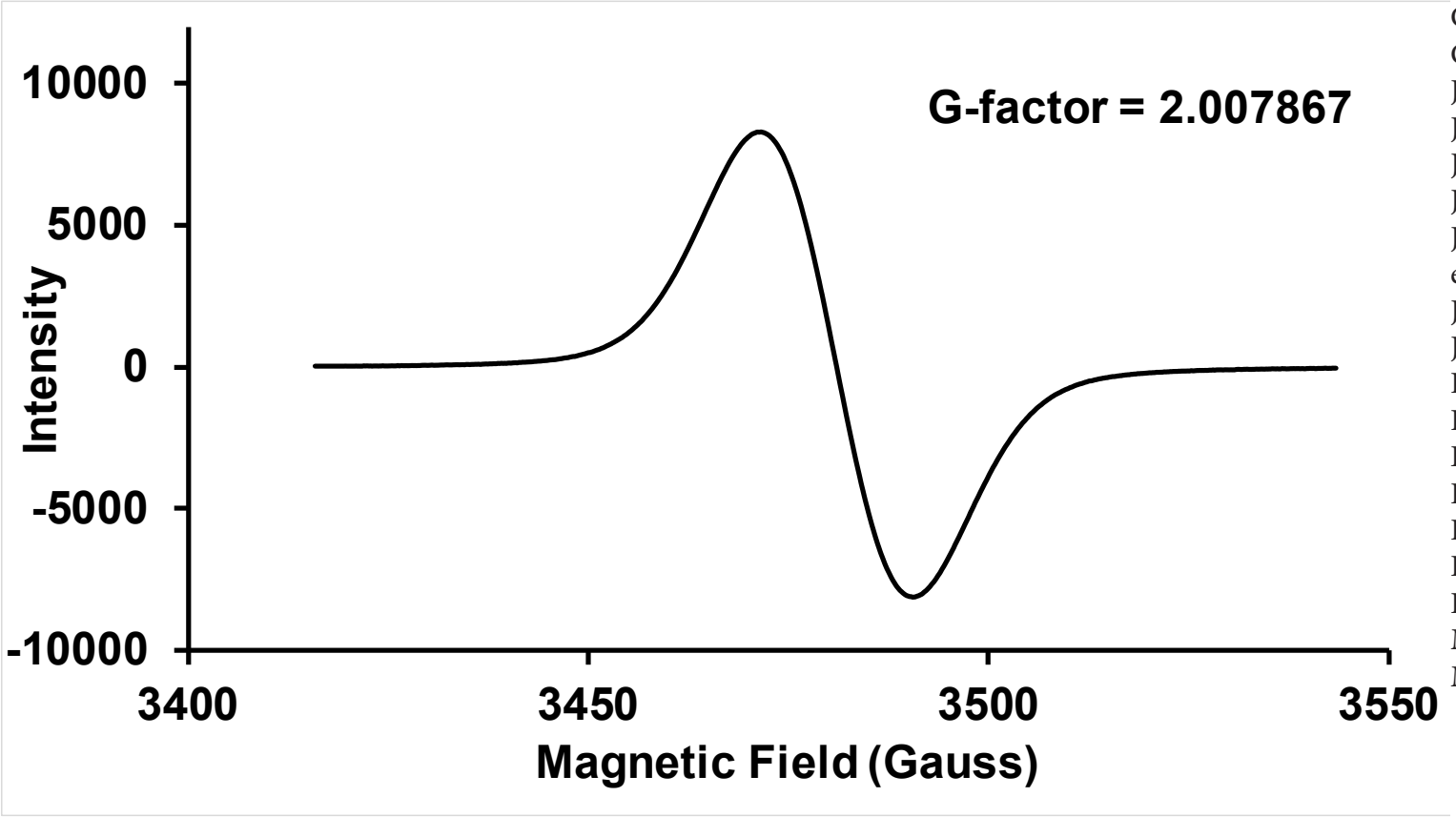

1- TBA+
CF3
O
CF3
12

IR

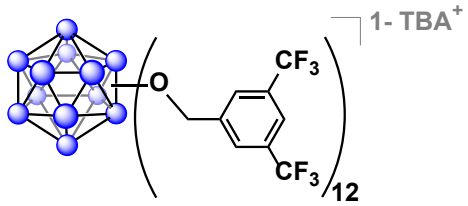

100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
Transmittance
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)



EPR

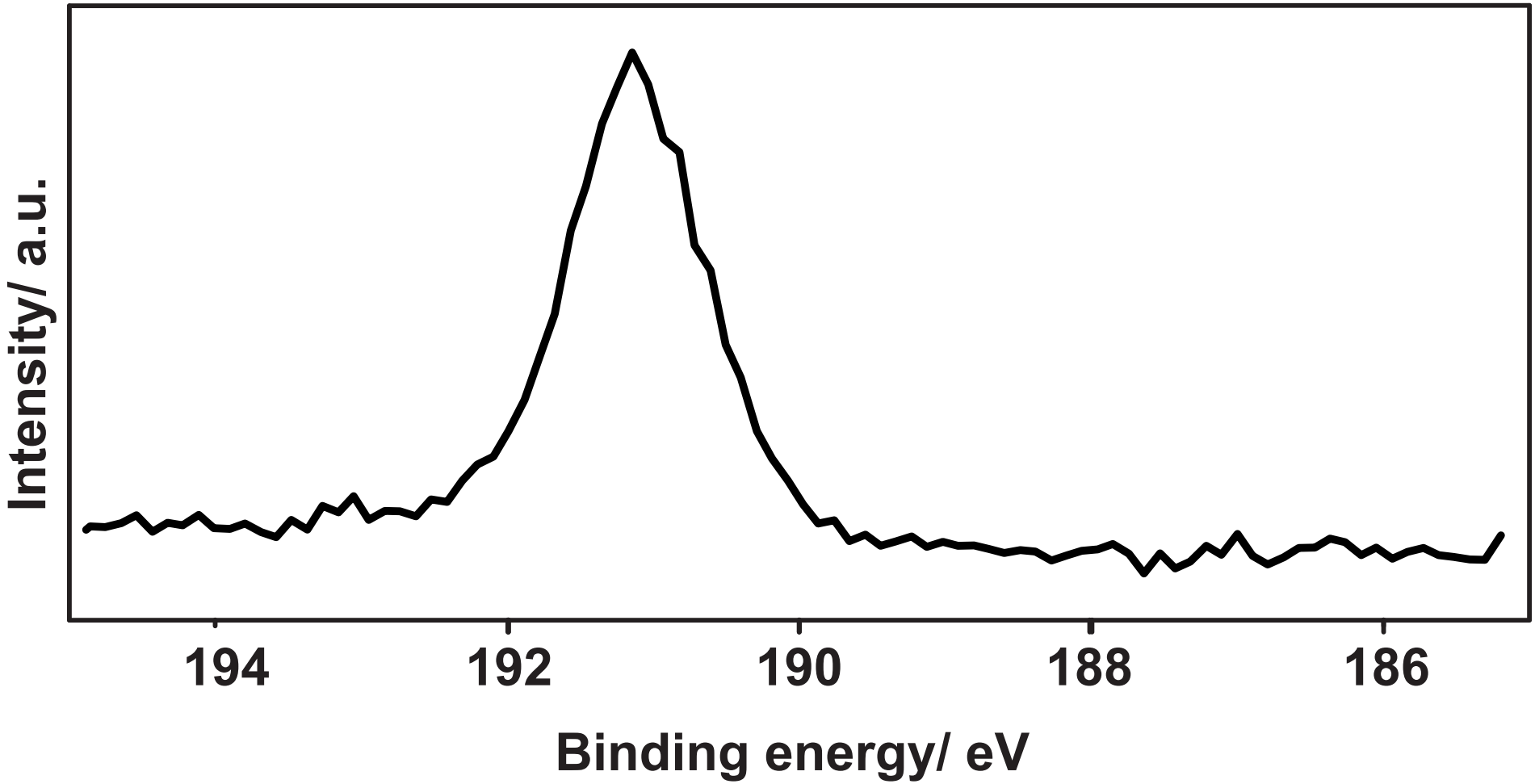


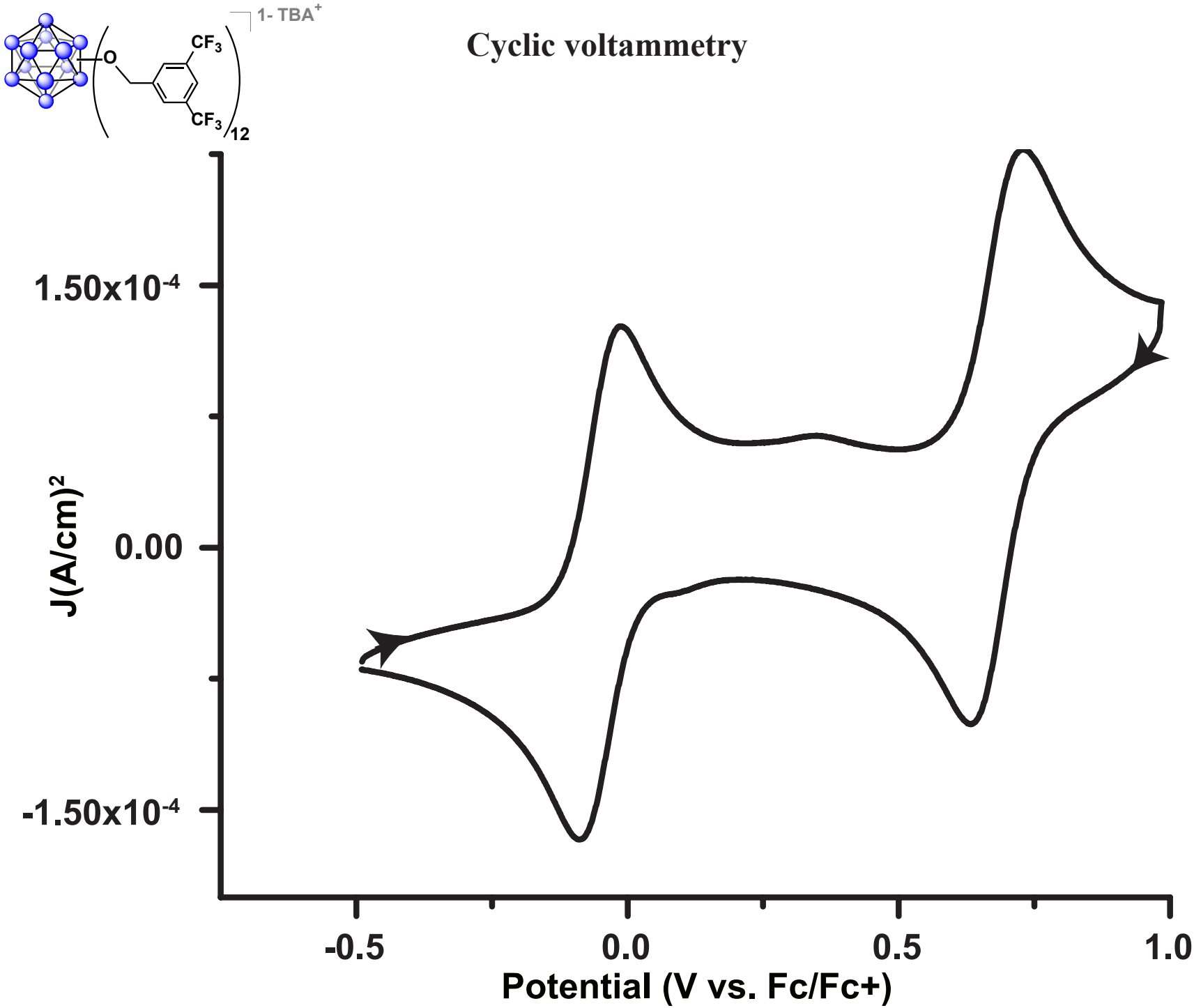
DOS	Format
ANZ	1024
MIN	-8134.161133
MAX	8283.838867
JSS	0
GST	3415.875
GSI	127.47
JUN	G
JON	Bruker BioSpin GmbH
JDA	6/4/2015
JTM	16:02
JRE	c:\programfiles\bruker-emx\syscal\st0103.cal
JEX	field-sweep
JSD	1
HCF	3479.61
HSW	127.47
EMF	0
RCT	20.48
RTC	20.48
RRG	2.83E+03
RMA	4
MF	9.778519
MP	6.38E-01

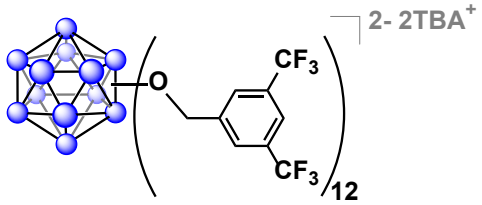


B 1s XPS

BRUKER

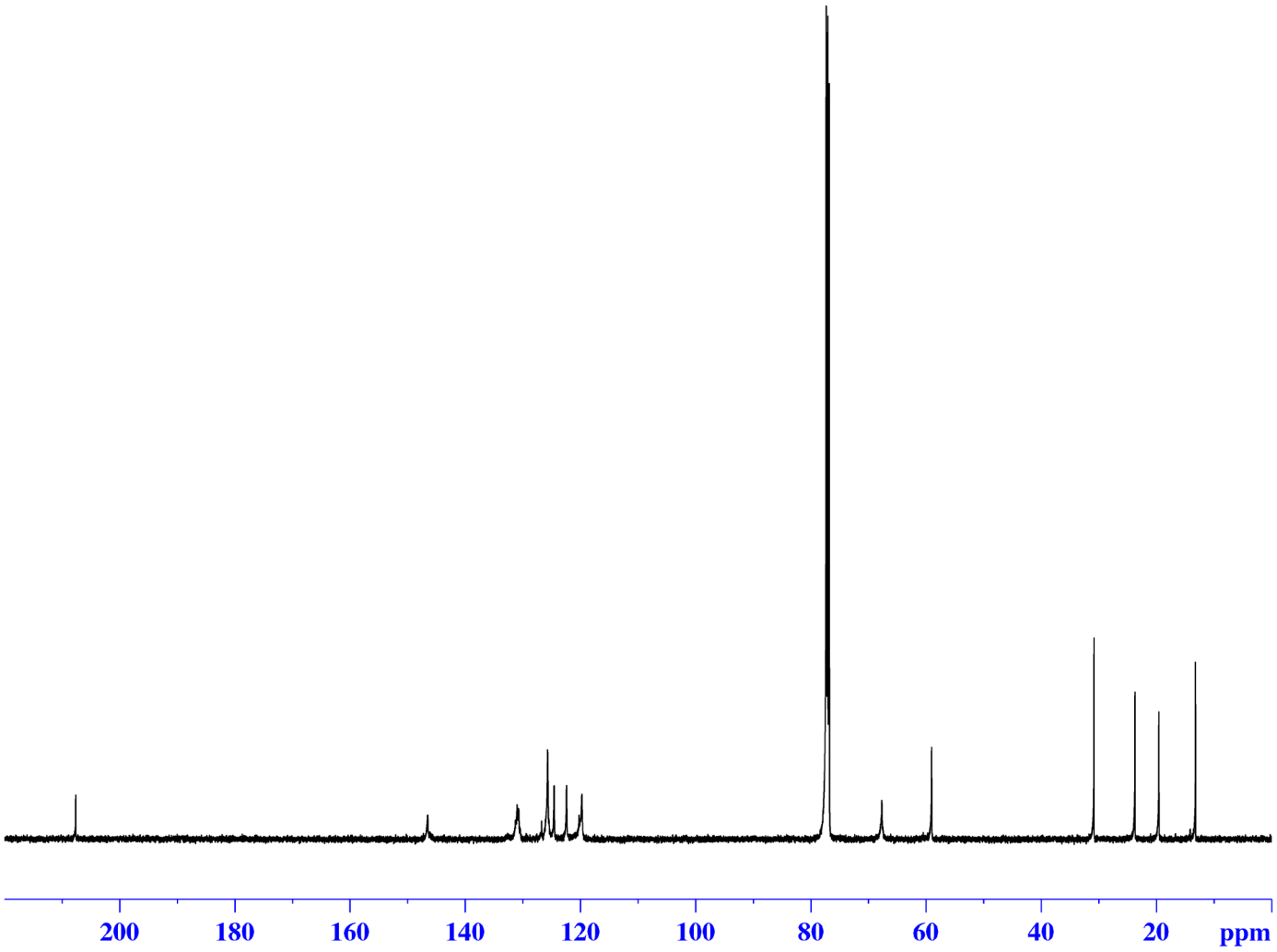



1- TBA+
CF3
O
CF3
12
Cyclic voltammetry
1.50x10-4
0.00
-1.50x10-4
J(A/cm)2
-0.5
0.0
0.5
1.0
Potential (V vs. Fc/Fc+)



^{13}C NMR

207.606
146.498
130.946
130.716
126.705
125.673
124.542
122.376
120.212
119.704
67.636
59.003
30.801
23.683
19.526
13.174



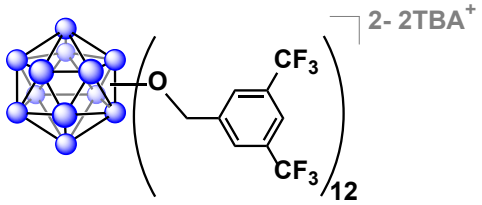
Current Data Parameters
NAME B12(O-3,5-bisTFMBn)12
EXPNO 700
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150829
Time 22.21
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

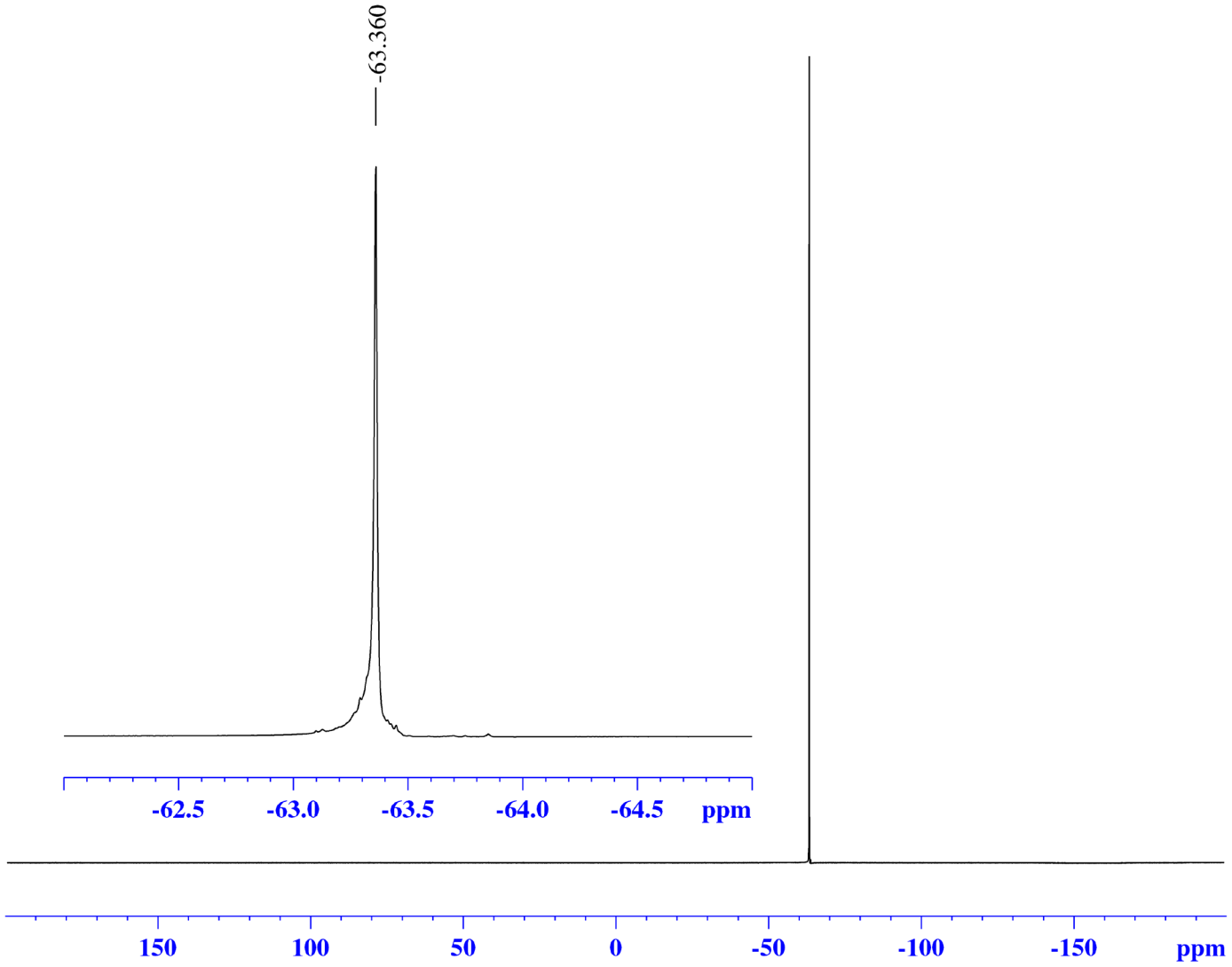
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{19}F NMR

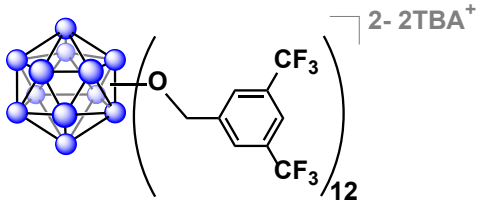


Current Data Parameters
NAME B12(O-3,5-bisTFMBn)12
EXPNO 121
PROCNO 1

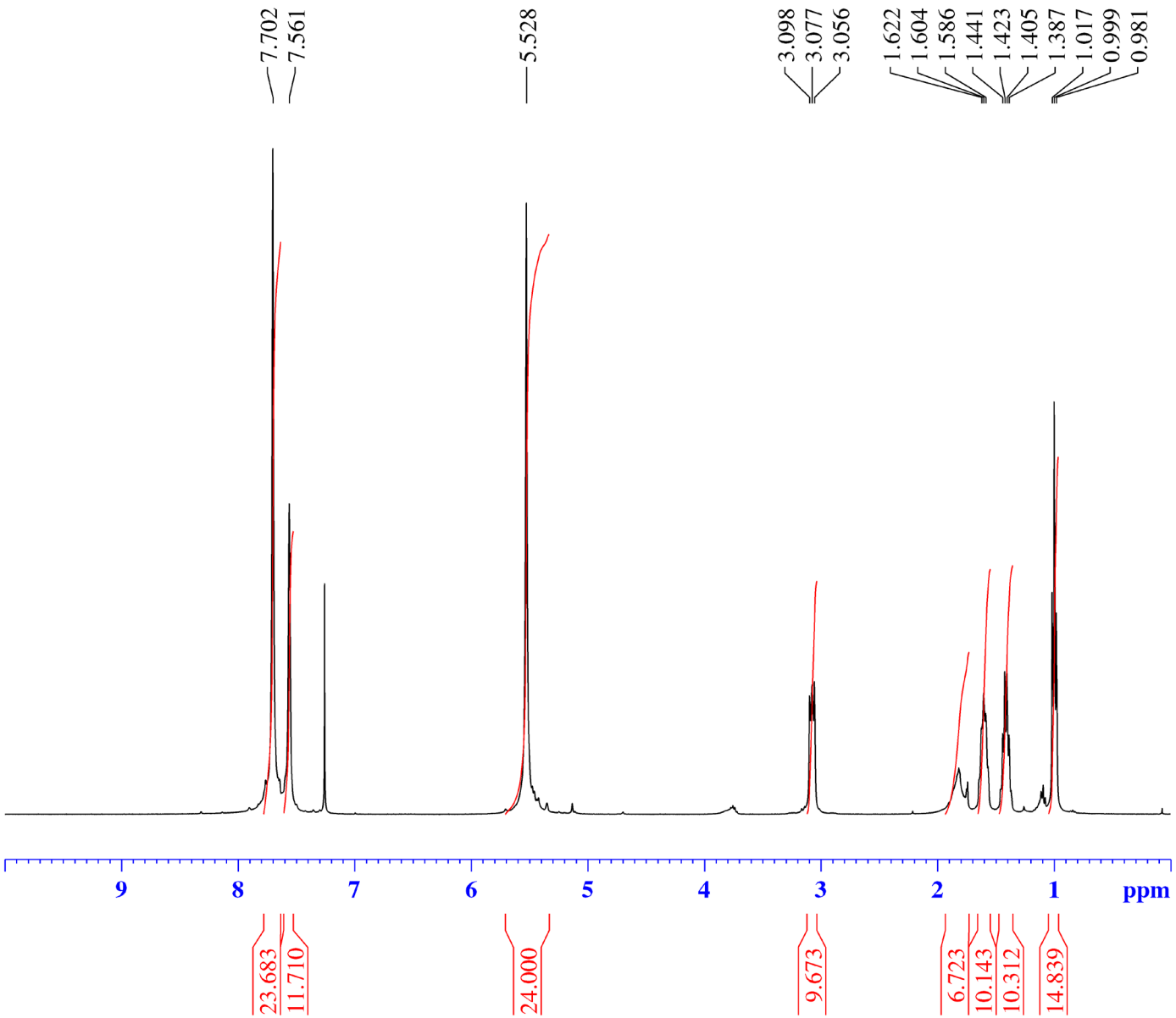
F2 - Acquisition Parameters
Date_ 20150824
Time 17.26
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgflqn30
TD 262144
SOLVENT CDCl3
NS 32
DS 0
SWH 150000.000 Hz
FIDRES 0.572205 Hz
AQ 0.8738133 sec
RG 189.85
DW 3.333 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 376.4983660 MHz
NUC1 19F
P1 14.50 usec
PLW1 17.00000000 W

F2 - Processing parameters
SI 262144
SF 376.4983660 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



^{1}H NMR

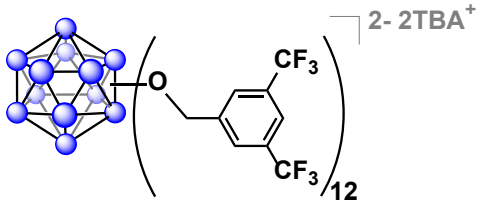


Current Data Parameters
NAME B12(O-3,5-bisTFMBn)12
EXPNO 122
PROCNO 1

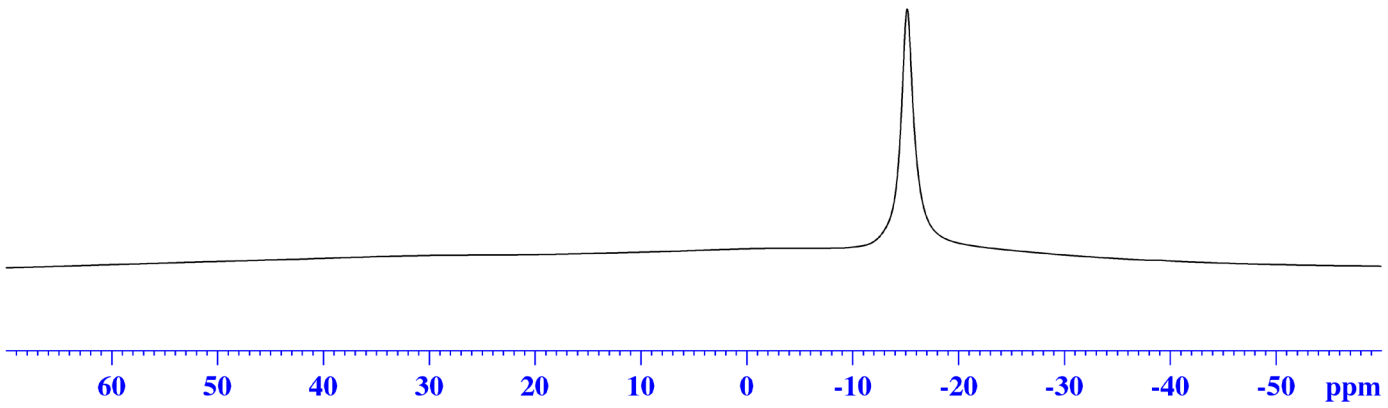
F2 - Acquisition Parameters
Date_ 20150824
Time 17.32
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 64
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300184 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



^{11}B {^{1}H} NMR



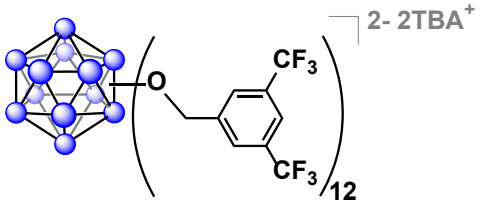
Current Data Parameters
NAME B12(O-3,5-bisTFMBn)12
EXPNO 120
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150824
Time 17.23
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.2 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

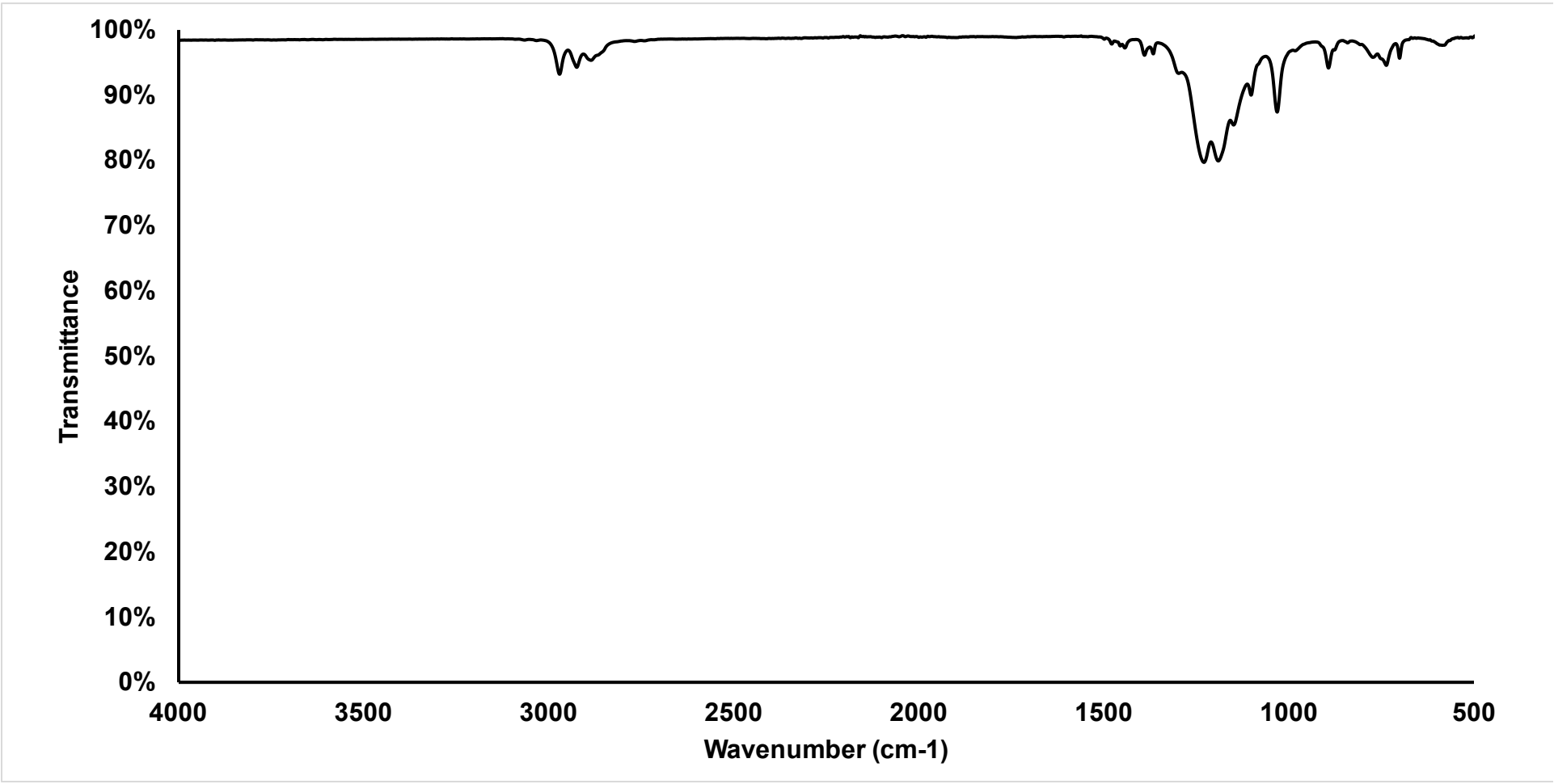
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

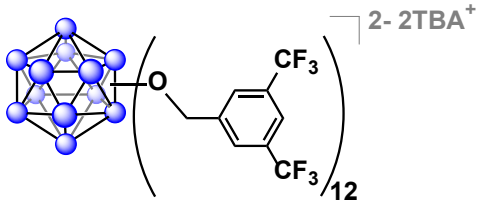
======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40


2- 2TBA+
O
CF3
CF3
12

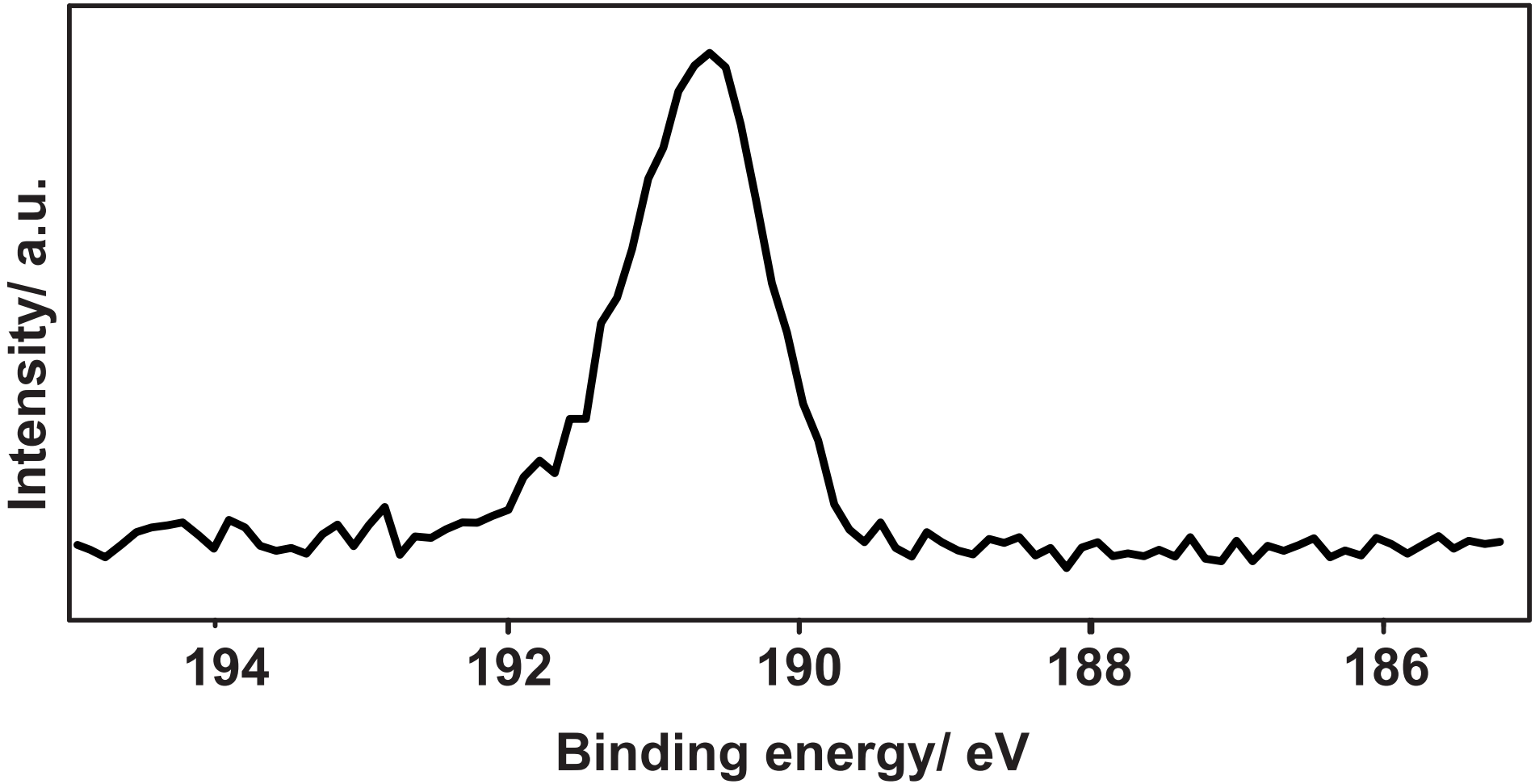
IR

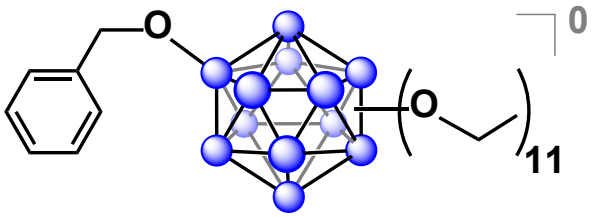

100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
Transmittance
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)



B 1s XPS

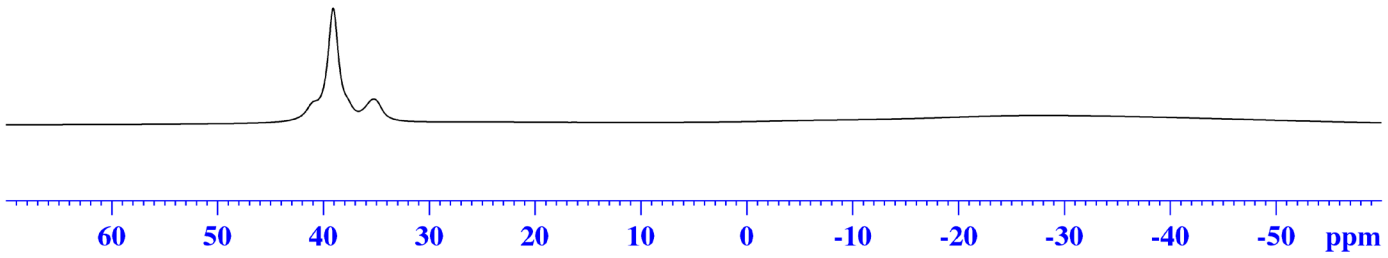
BRUKER





^{11}B {^{1}H} NMR

39.064
35.228



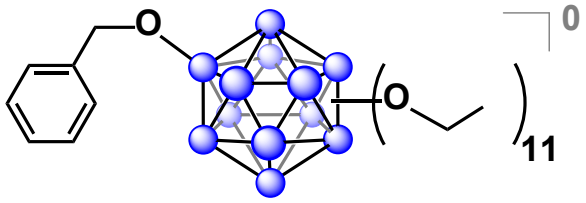
Current Data Parameters
NAME B12(O-Bn)(O-Et)11
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150517
Time 15.01
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgdc.js
TD 5096
SOLVENT CDCl3
NS 1024
DS 0
SWH 51020.406 Hz
FIDRES 10.011854 Hz
AQ 0.0499408 sec
RG 189.85
DW 9.800 usec
DE 6.50 usec
TE 299.1 K
D1 0.00000400 sec
D11 0.03000000 sec
TD0 1

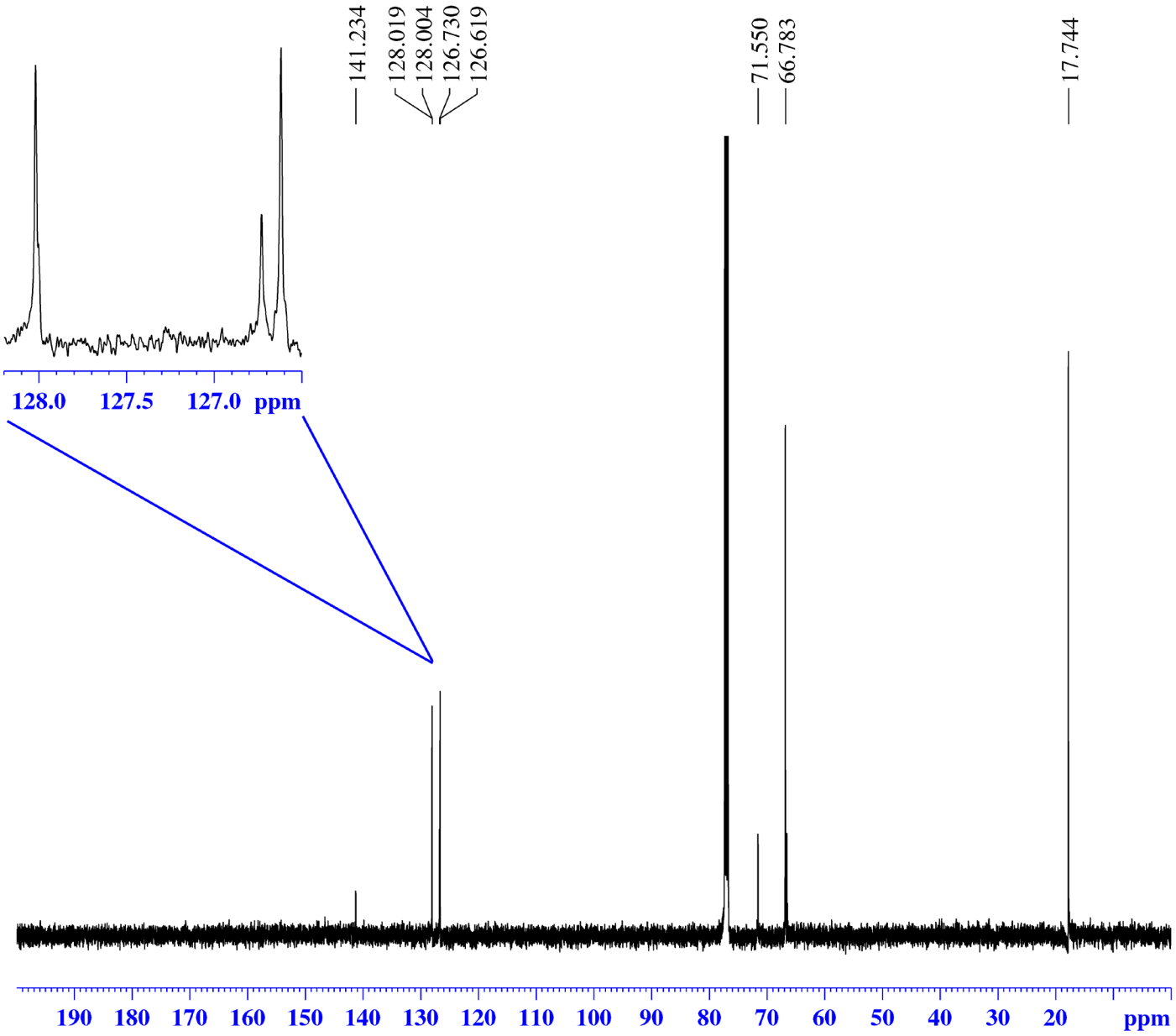
======== CHANNEL f1 ========
SFO1 128.3776052 MHz
NUC1 11B
P1 10.00 usec
PLW1 52.00000000 W

======== CHANNEL f2 ========
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 50.00 Hz
GB 0
PC 1.40



^{13}C NMR



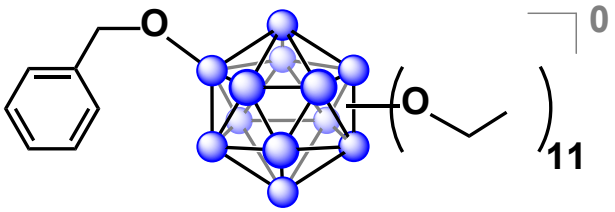
Current Data Parameters
NAME B12(O-Bn)(O-Et)11
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150517
Time 16.29
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577892 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



^{1}H NMR in C6D6

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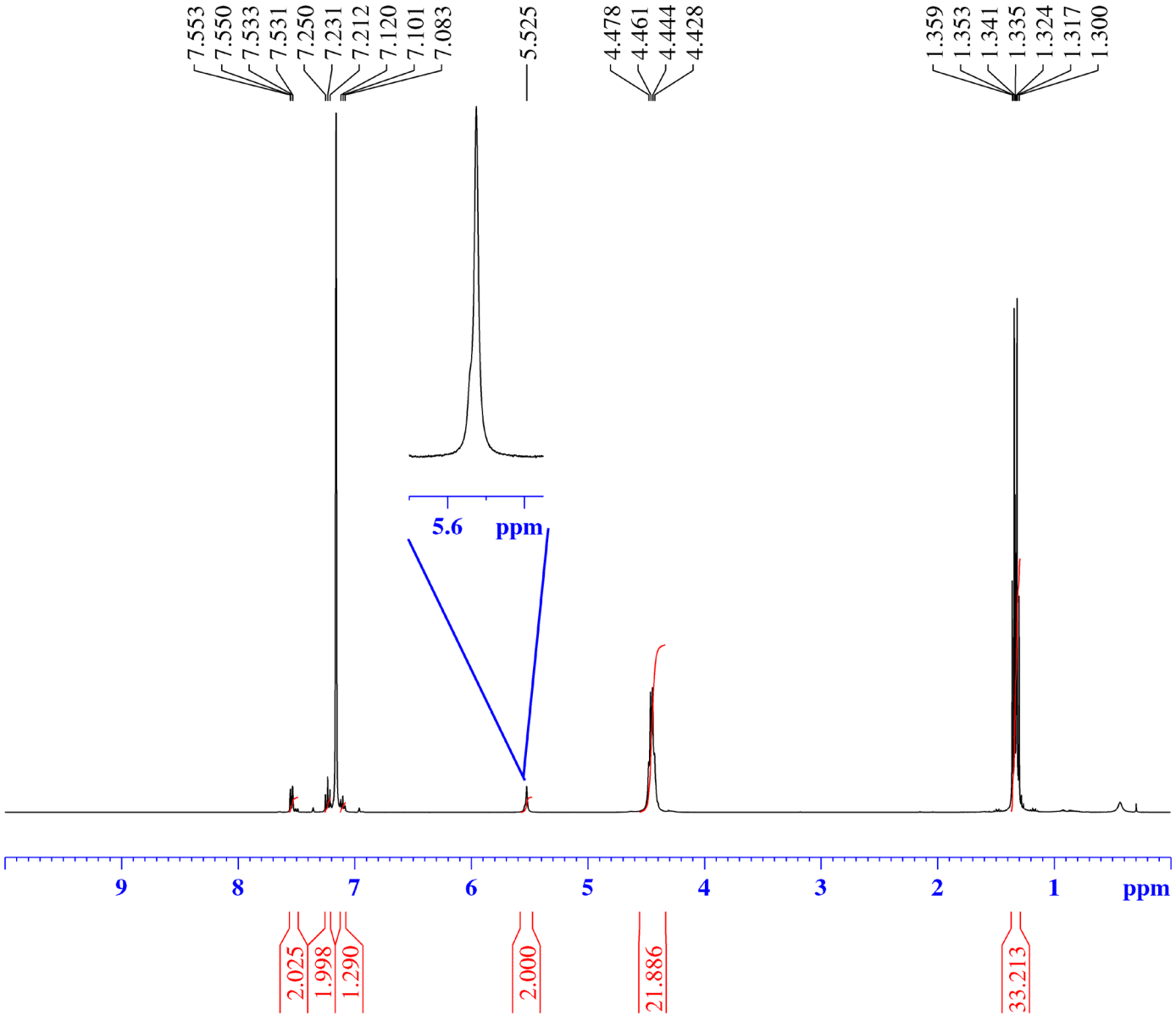
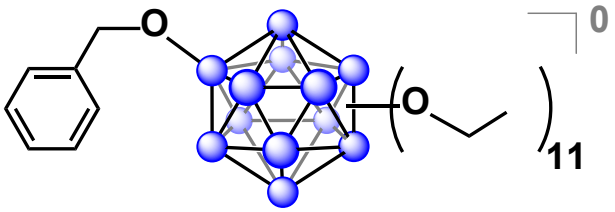


Current Data Parameters
NAME Jan14-2016
EXPNO 100
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160114
Time 14.59
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT C6D6
NS 32
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1299967 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



^{1}H NMR

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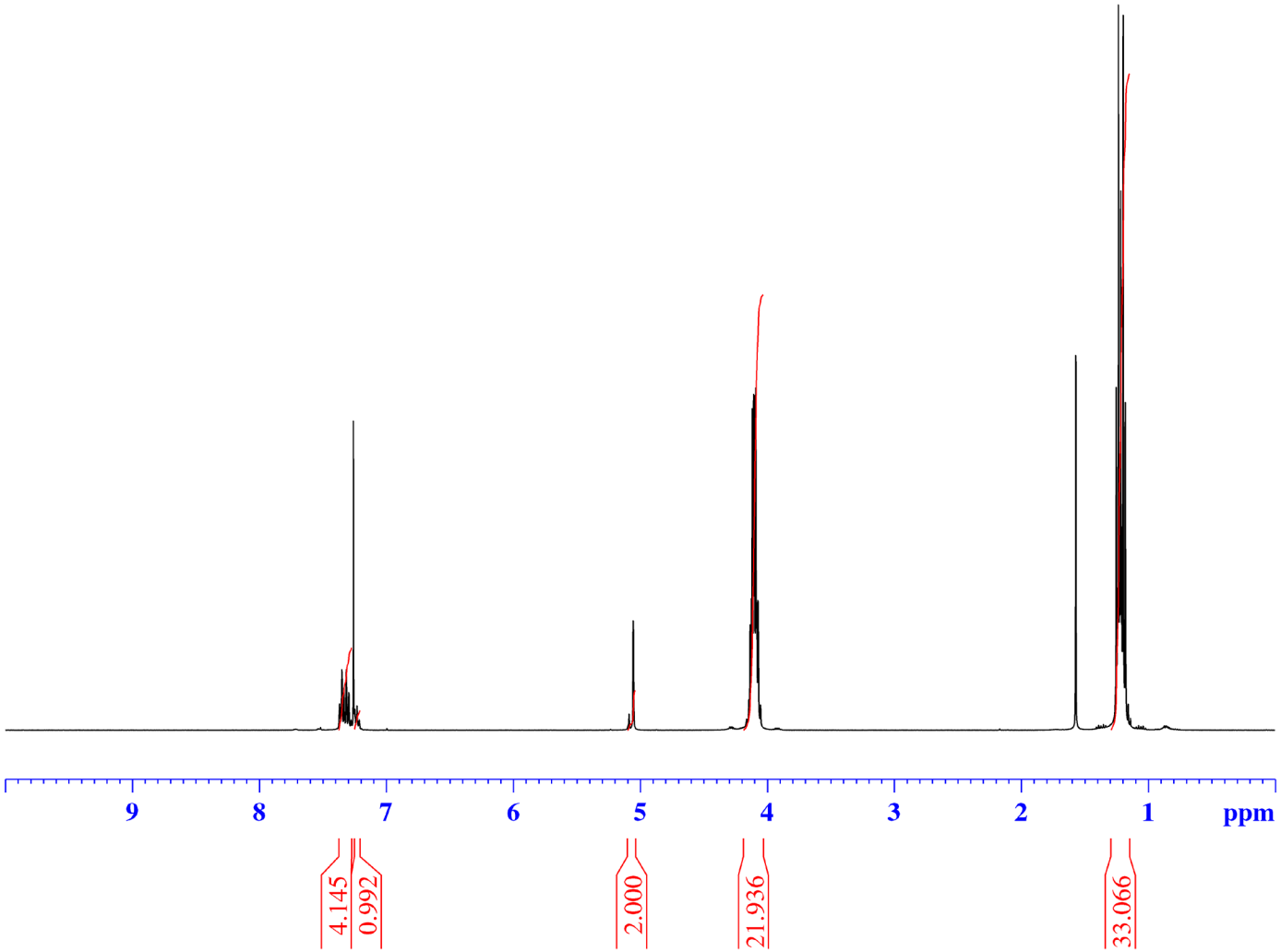
7.367 7.350 7.332 7.327 7.314 7.295 7.277 7.248 7.230 7.213
5.088 5.055 4.143 4.135 4.125 4.117 4.108 4.100 4.090 4.082 4.073 4.052
1.251 1.241 1.234 1.224 1.215 1.207 1.197 1.180

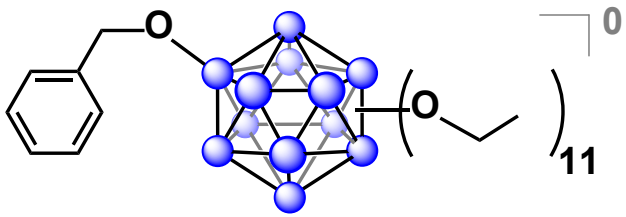


Current Data Parameters
NAME Jan13-2016
EXPNO 142
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160113
Time 20.50
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 32
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

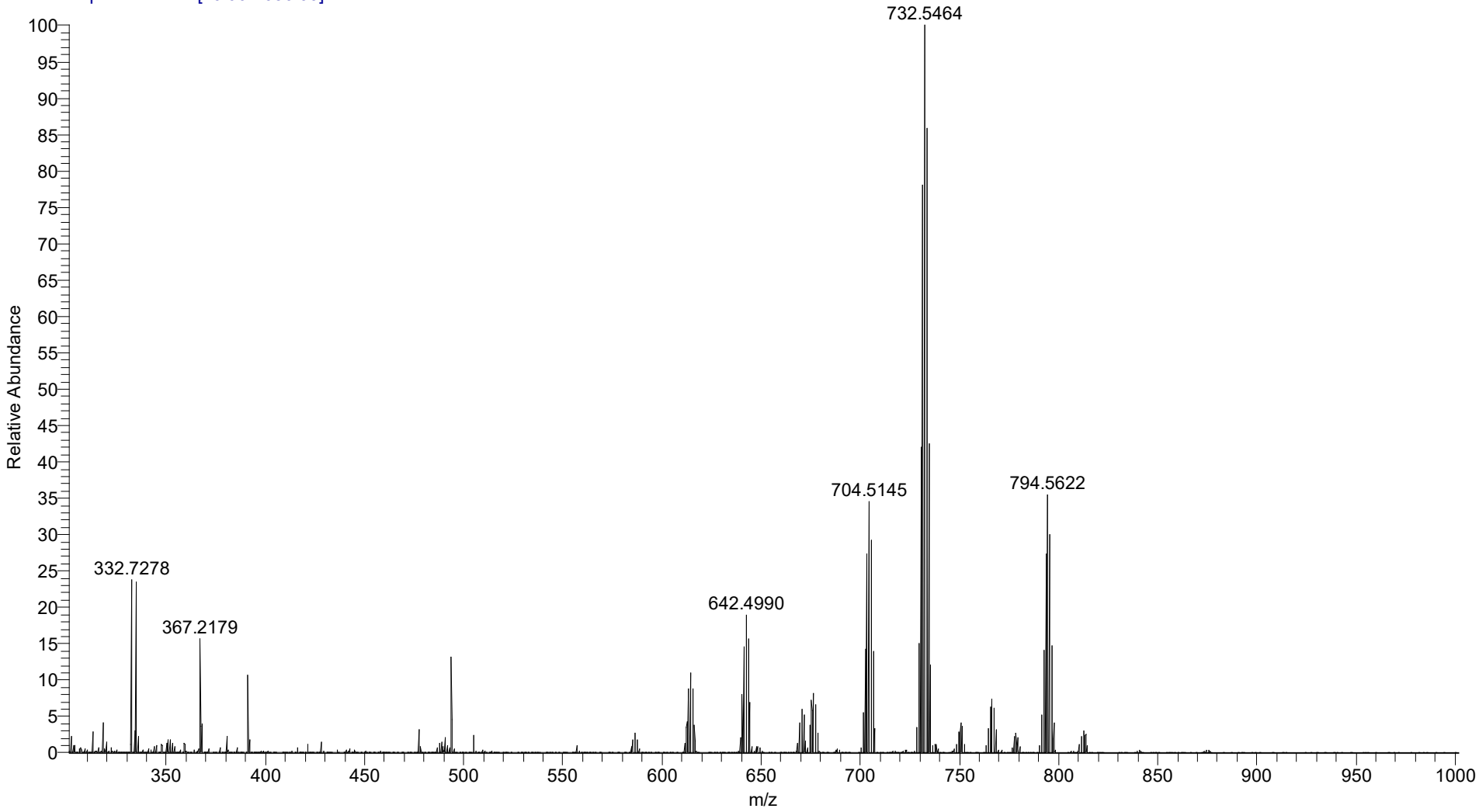
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

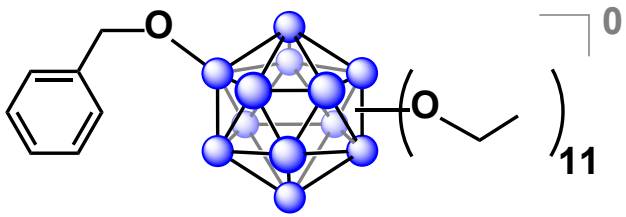
F2 - Processing parameters
SI 65536
SF 400.1300184 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



DART
High-Res Mass Spec

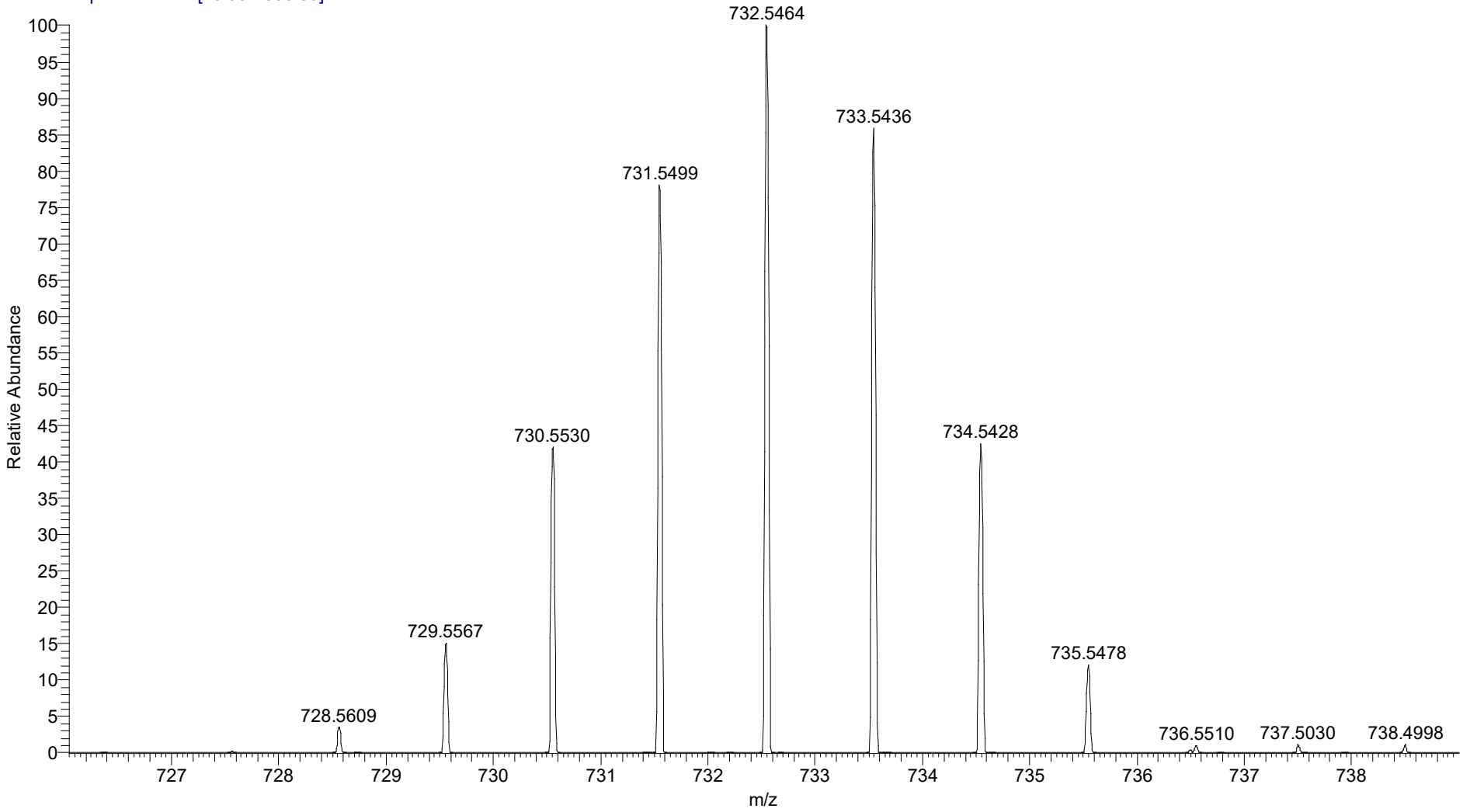
AW-Bn1Et11 #3-52 RT: 0.03-0.52 AV: 50 NL: 2.50E5
T: FTMS + p NSI Full ms [70.00-1050.00]

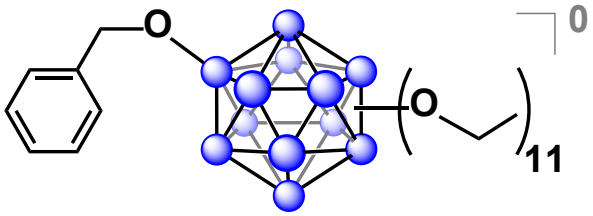




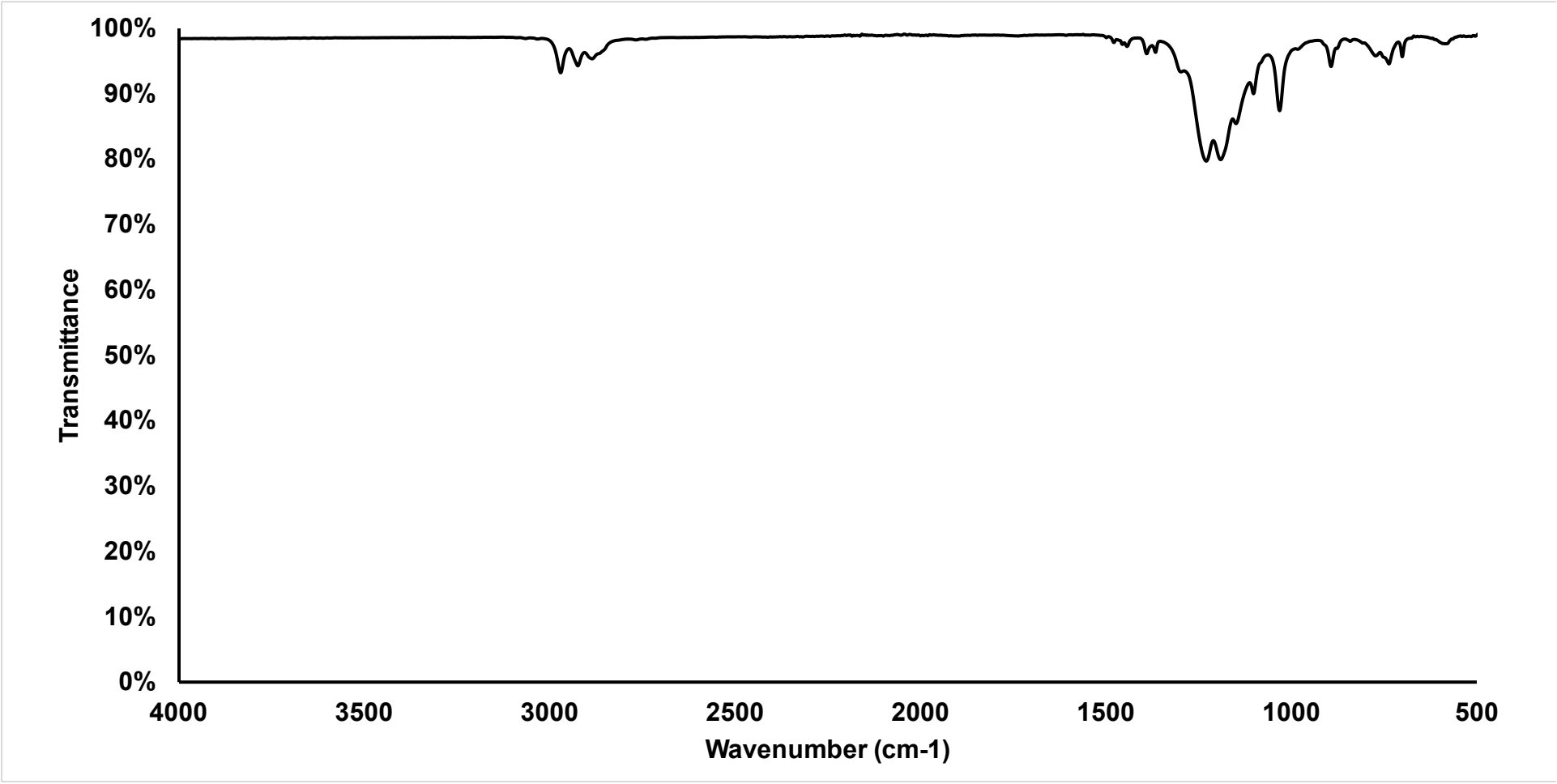
DART
High-Res Mass Spec

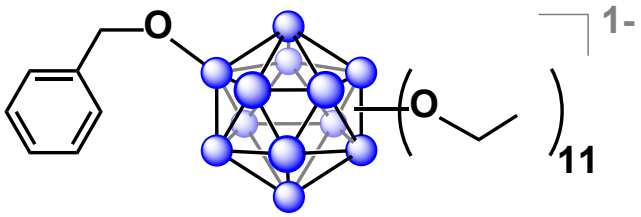
AW-Bn1Et11 #3-52 RT: 0.03-0.52 AV: 50 NL: 2.50E5
T: FTMS + p NSI Full ms [70.00-1050.00]



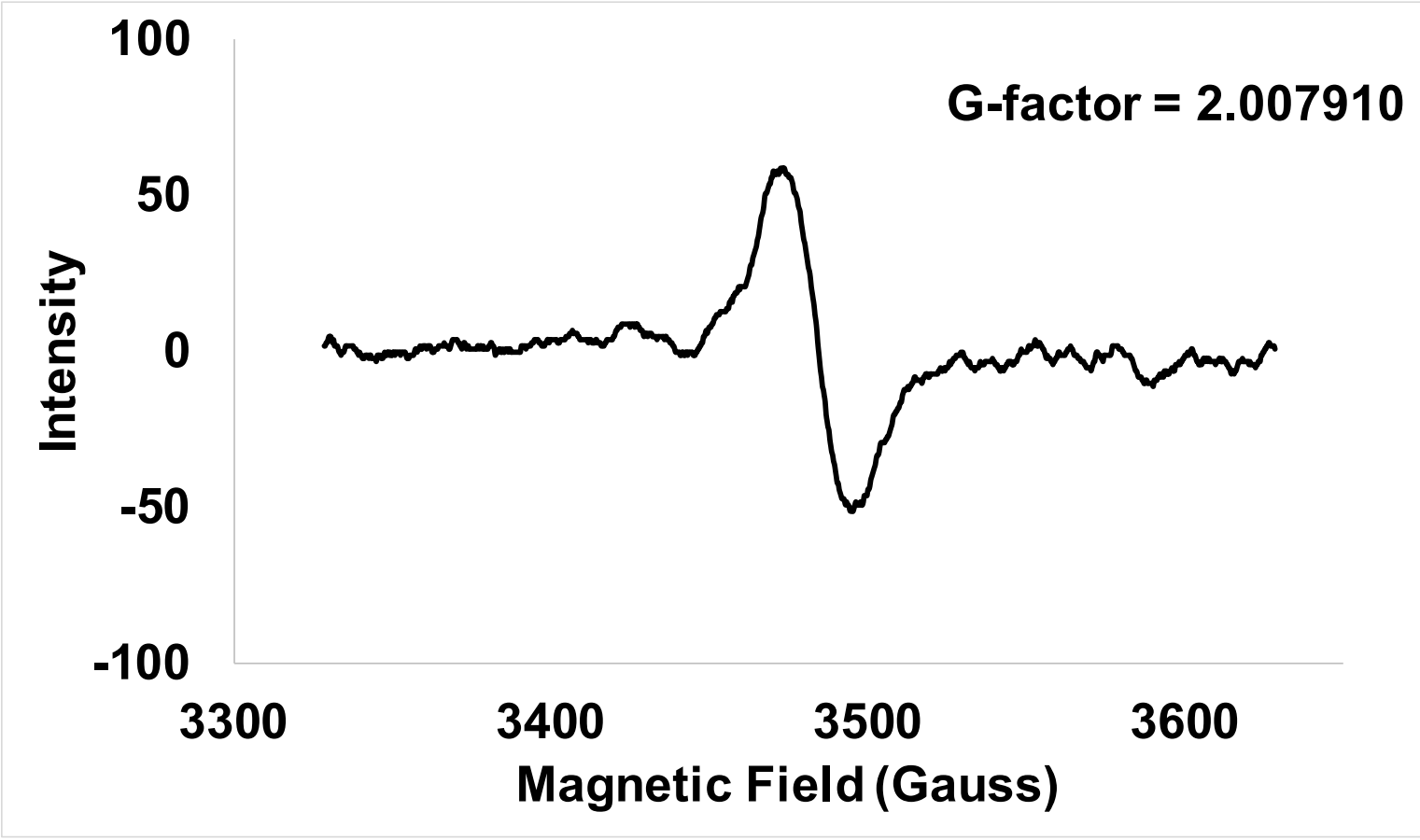

O
O
11
0

IR

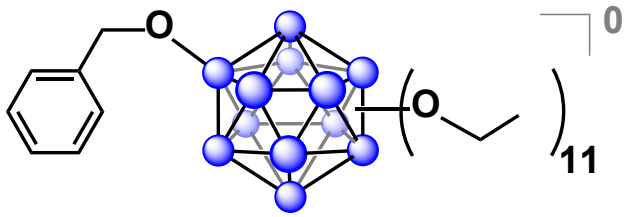

100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
Transmittance
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)



EPR



DOS	Format
ANZ	1024
MIN	-51.404297
MAX	58.595703
JSS	0
GST	3328.55
GSI	300
JUN	G
JON	Bruker BioSpin GmbH
JDA	11/2/2015
JTM	20:39
JRE	c:\programfiles\bruker-emx\syscal\st0103.cal
JEX	field-sweep
JSD	1
HCF	3478.55
HSW	300
EMF	0
RCT	20.48
RTC	327.68
RRG	8.93E+03
RMA	4
MF	9.77575
MP	2.54E+00
MPD	19



Cyclic voltammetry