ELECTRONIC SUPPLEMENTARY INFORMATION

for the paper entitled

Efficient Masking of Corrosion and Fission Products such as Ni(II) and Pd(II) in the Presence of the Minor Actinide Am(III) using Hydrophilic Anionic or Cationic *Bis*-Triazines

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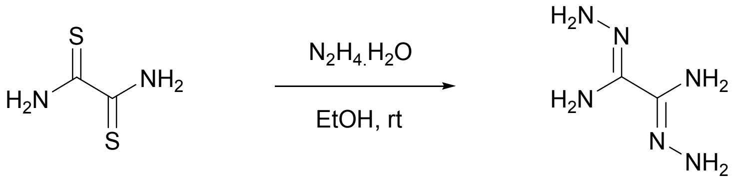
1. Experimental Procedures

General Procedures

Melting points were obtained on a Stuart SMP10 instrument. IR spectra were recorded as Nujol® mulls (N) on a Perkin Elmer RX1 FT-IR instrument. ^{1}H, ^{13}C−{^{1}H} and ^{13}C NMR spectra were recorded using either a Bruker AMX400 or an Avance DFX400 instrument. Chemical shifts are reported in parts per million downfield from tetramethylsilane. Assignments were verified with ^{1}H-^{1}H and ^{1}H-^{13}C COSY experiments as appropriate. Mass spectra were obtained under electrospray conditions on a Thermo Scientific LTQ Orbitrap XL instrument. Elemental microanalyses were carried out by Medac Ltd., Brunel Science Centre, Surrey (UK). Solvent extraction studies were performed at the Karlsruher Institut für Technologie (Germany). All organic reagents were obtained from either Acros or Aldrich, while inorganic reagents were obtained from either BDH or Aldrich and used as received.

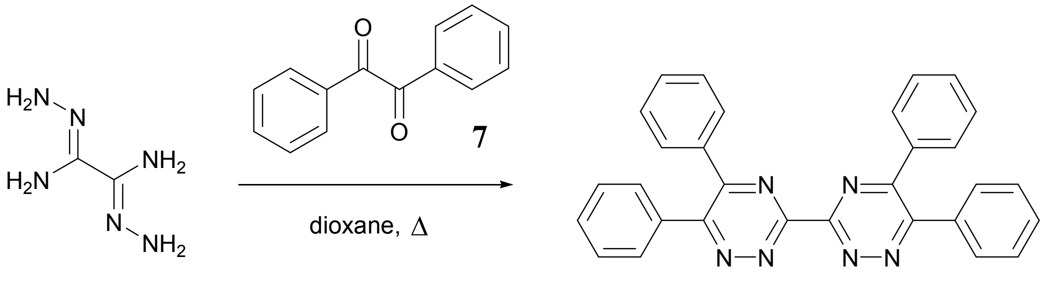
(1*Z*,2*Z*)-Ethanedihydrazonamide 6[1–3]



5 **6**

Dithiooxamide **5** (5.28 g, 43.926 mmol) was suspended in ethanol (50 mL) and water (50 mL) and hydrazine hydrate (50 mL, 64 %) was added slowly dropwise over 10 minutes. The colour changed from deep red to light yellow during the addition. The resulting suspension was stirred at ambient temperature for 2 days. The insoluble solid was then filtered and washed successively with water (100 mL), water/ethanol (1:1, 50 mL) and ethanol (20 mL) and then dried in air to afford the title compound **6** as a cream coloured solid (3.08 g, 61 %). The hydrogen sulfide (caution: stench) in the filtrate was destroyed by the addition of excess iron(II) sulfate forming a black solution. ^{1}H NMR (400.1 MHz, DMSO-d_6) δ: 4.94 (br s, 4H, 2 × NH_2), 5.00 (br s, 4H, 2 × NH_2) ppm; ^{13}C NMR (100.6 MHz, DMSO-d_6) δ: 140.7 (2 × quat) ppm.

5,5',6,6'-Tetraphenyl-3,3'-bi-1,2,4-triazine 8[4–6]

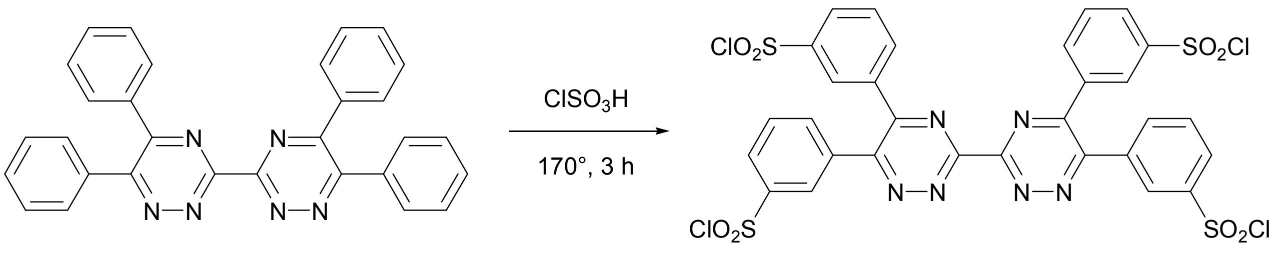


6

8

The starting material **6** (0.50 g, 4.310 mmol) was suspended in dioxane (50 mL) and benzil **7** (1.99 g, 9.482 mmol, 2.2 eq) was added. Triethylamine (4.0 mL) was added and the suspension was heated under reflux for 2 days. The flask was allowed to cool to room temperature and the insoluble solid was filtered and washed with methanol (100 mL) and diethyl ether (50 mL) and allowed to dry in air to afford the title compound **8** as a yellow solid (1.55 g, 77 %). ^{1}H NMR (400.1 MHz, $CDCl_3$, Me_4Si) δ: 7.36–7.51 (m, 12H, 8 × *m*-Ar*H* and 4 × *p*-Ar*H*), 7.71 (d, $J = 6.8$, 4H, 4 × *o*-Ar*H*), 7.76 (d, $J = 7.3$, 4H, 4 × *o*-Ar*H*) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$, Me_4Si) δ: 128.6 (4 × *m*-Ar*C*), 128.7 (4 × *m*-Ar*C*), 129.7 (4 × *o*-Ar*C*), 130.1 (4 × *o*-Ar*C*), 130.1 (2 × *p*-Ar*C*), 131.1 (2 × *p*-Ar*C*), 135.0 (2 × quat), 135.1 (2 × quat), 156.3 (2 × quat), 157.3 (2 × quat), 159.2 (2 × quat) ppm.

3,3’,3’’,3’’’-[3,3’-Bi-1,2,4-triazine-5,5’,6,6’-tetrayl]tetrabenzenesulfonyl chloride 9

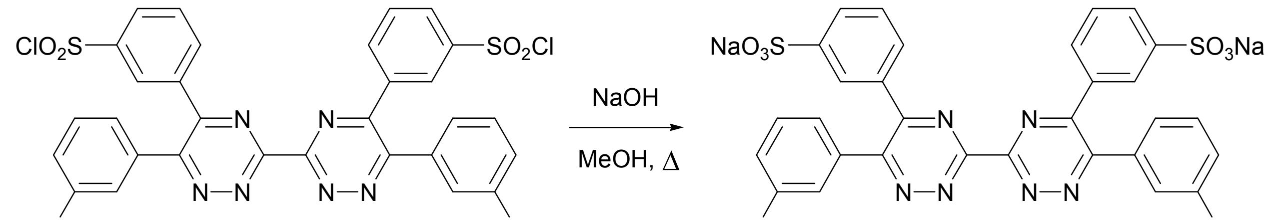


8

9

The starting material **8** (0.61 g, 1.314 mmol) was placed into a 50 mL round bottomed flask and chlorosulfonic acid (10 mL) was added. The solution was heated at 170 °C for 3 hours. The solution was allowed to cool to room temperature and was cautiously poured onto ice (100 mL). The precipitated solid was filtered and washed with water (200 mL) and was allowed to dry in air. The solid was dried 70 °C for 2 hours to afford the title compound **9** as a light brown solid (1.07 g, 95 %). Mp 177–182 °C (from H_2O); Found: C, 41.66; H, 2.05; N, 9.48 %; $C_{30}H_{16}N_6O_8S_4Cl_4$ requires C, 41.97; H, 1.88; N, 9.79 %; IR ν_{max} (N) 3162, 1720, 1644, 1614, 1508, 1477, 1421, 1366, 1311, 1143 (SO_2), 1118 (SO_2), 1027 (SO_2), 993, 908, 846, 801, 733, 686, 674, 607 cm^{-1}; ^{1}H NMR (400.1 MHz, DMSO-*d*$_6$) δ: 7.29–7.33 (m, 6H, 4 × $ArSO_2Cl$ 4-H and 2 × $ArSO_2Cl$ 6-H), 7.63–7.72 (m, 6H, 4 × $ArSO_2Cl$ 5-H and 2 × $ArSO_2Cl$ 6-H), 8.20–8.25 (m, 4H, 4 × $ArSO_2Cl$ 2-H) ppm; ^{13}C NMR (100.6 MHz, DMSO-*d*$_6$) δ: 125.6 (2 × $ArSO_2Cl$ C-6), 126.4 (2 × $ArSO_2Cl$ C-2), 127.0 (2 × $ArSO_2Cl$ C-2), 127.6 (2 × $ArSO_2Cl$ C-5), 127.8 (2 × $ArSO_2Cl$ C-4), 127.9 (2 × $ArSO_2Cl$ C-4), 129.2 (2 × $ArSO_2Cl$ C-5), 130.1 (2 × $ArSO_2Cl$ C-6), 130.1 (2 × quat), 134.6 (2 × quat), 134.7 (2 × quat), 148.3 (2 × quat), 156.1 (2 × quat), 157.0 (2 × quat), 159.1 (2 × quat) ppm; HRMS (CI) *m*/*z* 782.9943 (as tetrasulfonic acid monoanion): calculated for $[C_{30}H_{19}N_6O_{12}S_4]^-$ 782.9938.

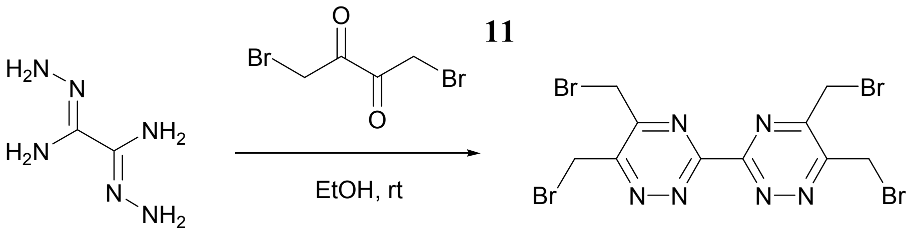
Tetrasodium 3,3',3'',3'''-[3,3'-bi-1,2,4-triazine-5,5',6,6'-tetrayl]tetrabenzenesulfonate 10



9 **10**

The starting material **9** (0.85 g, 0.9906 mmol) was suspended in methanol (40 mL) and a solution of sodium hydroxide 0.24 g, 5.944 mmol, 6 eq) in methanol (20 mL) was added. The flask was heated under reflux for 4 hours. The flask was allowed to cool to room temperature and the insoluble solids were filtered and washed with methanol (10 mL). The excess sodium hydroxide in the filtrate was neutralized by the dropwise addition of HCl (ca. 1 mL, 2 M) until the pH was neutral. The filtrate was diluted with acetone (500 mL) and the precipitated solid was filtered and washed with methanol (10 mL) and acetone (100 mL) and was allowed to dry in air. The solid was dried at 70 °C for 1 hour to afford the title compound **10** as a cream coloured solid (0.57 g, 66 %). Mp above 300 °C (from MeOH); Found: C, 40.89; H, 2.12; N, 9.45; S, 14.54 %; $C_{30}H_{16}N_6O_{12}S_4Na_4$ requires C, 41.29; H, 1.85; N, 9.63; S, 14.69 %; IR ν_{max} (N) 3448, 3076, 1637, 1502, 1475, 1419, 1359, 1340, 1185 (SO_2), 1124 (SO_2), 1039 (SO_2), 996, 910, 874, 850, 803, 734, 688, 615 cm^{-1}; ^{1}H NMR (400.1 MHz, D_2O) δ: 7.52 (t, *J* = 7.8, 2H, 2 × $ArSO_3Na$ 5-H), 7.53 (t, *J* = 7.8, 2H, 2 × $ArSO_3Na$ 5-H), 7.65 (app d, *J* = 7.8, 2H, 2 × $ArSO_3Na$ 4-H), 7.79–7.82 (m, 2H, 2 × $ArSO_3Na$ 4-H), 7.86–7.89 (m, 4H, 4 × $ArSO_3Na$ 6-H), 7.95 (t, *J* = 1.6, 2H, 2 × $ArSO_3Na$ 2-H), 8.00 (t, *J* = 1.6, 2H, 2 × $ArSO_3Na$ 2-H) ppm; ^{13}C NMR (100.6 MHz, D_2O) δ: 126.0 (2 × $ArSO_3Na$ C-2), 126.7 (2 × $ArSO_3Na$ C-2), 127.2 (2 × $ArSO_3Na$ C-6), 127.4 (2 × $ArSO_3Na$ C-6), 128.2 (2 × $ArSO_3Na$ C-5), 129.6 (2 × $ArSO_3Na$ C-5), 132.5 (2 × $ArSO_3Na$ C-4), 132.9 (2 × $ArSO_3Na$ C-4), 134.3 (2 × quat), 134.5 (2 × quat), 143.0 (2 × quat), 143.3 (2 × quat), 158.1 (2 × quat), 158.6 (2 × quat), 158.6 (2 × quat) ppm; HRMS (CI) *m/z* 782.9941 (as tetrasulfonic acid monoanion): calculated for $[C_{30}H_{19}N_6O_{12}S_4]^-$ 782.9938.

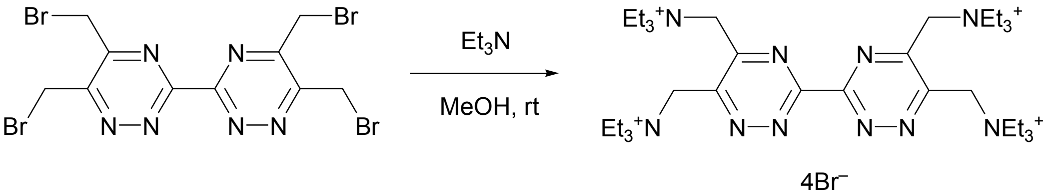
5,5',6,6'-Tetrakis(bromomethyl)-3,3'-bi-1,2,4-triazine 12[7]



6 **12**

1,4-Dibromo-2,3-butanedione **11** (2.20 g, 9.051 mmol, 2.1 eq) was dissolved in ethanol (25 mL) and the solution was cooled to 0 °C. The starting material **6** (0.50 g, 4.310 mmol) was slowly added as a solid in small aliquots over 15 minutes. The suspension was stirred at 0 °C for 1 hour and the flask was allowed to warm to room temperature and stirring was continued for a further 2 hours. The insoluble solid was then filtered and washed with ethanol (100 mL) and diethyl ether (50 mL) and was allowed to dry in air to afford the title compound **12** as a yellow solid (1.59 g, 70 %). ^{1}H NMR (400.1 MHz, $CDCl_3$, Me_4Si) δ: 4.84 (s, 4H, 2 × CH_2Br), 5.06 (s, 4H, 2 × CH_2Br) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$, Me_4Si) δ: 26.2 (2 × CH_2Br), 27.3 (2 × CH_2Br), 157.4 (2 × quat), 157.7 (2 × quat), 160.1 (2 × quat) ppm.

3,3'-Bi-1,2,4-triazine-5,5',6,6'-tetrayltetrakis(*N*,*N*,*N*-triethylmethanaminium) tetrabromide 13



12

13

The starting material **12** (0.50 g, 0.940 mmol) was suspended in methanol (60 mL) and the flask was cooled to 0 °C using an ice bath. Triethylamine (1.31 mL, 9.402 mmol, 10 eq) was added slowly dropwise over 5 minutes and the flask was stirred at 0 °C for 45 minutes. The flask was then allowed to warm to room temperature and stirring was continued for 3 days. The insoluble solids were then filtered and washed with methanol (10 mL) and the filtrate was evaporated. The resulting dark solid was triturated with acetone (100 mL) and then filtered and washed with acetone (100 mL) and allowed to dry in air to afford the title compound **13** as a brown solid (0.57 g, 65 %). Mp 175–180 °C (decomposed); Found: C, 43.56; H, 7.07; N, 15.37; Br, 33.88 %; $C_{34}H_{68}N_{10}Br_4$ requires C, 43.60; H, 7.32; N, 14.95; Br, 34.13 %; IR ν_{max} (N) 3391, 2983, 2946, 2072, 1627, 1517, 1452, 1395, 1325, 1232, 1187, 1156, 1101, 1074, 1007, 793 cm^{-1}; ^{1}H NMR (400.1 MHz, D_2O) δ: 1.18–1.25 (m, 18H, 2 × $N(CH_2CH_3)_3$), 1.26–1.33 (m, 18H, 2 × $N(CH_2CH_3)_3$), 2.14 (s, 4H, 2 × $CH_2N(CH_2CH_3)_3$), 3.26 (s, 4H, 2 × $CH_2N(CH_2CH_3)_3$), 3.52–3.65 (m, 12H, 2 × $N(CH_2CH_3)_3$), 3.71–3.88 (m, 12H, 2 × $N(CH_2CH_3)_3$) ppm; ^{13}C NMR (100.6 MHz, D_2O) δ: 7.1 (2 × $N(CH_2CH_3)_3$), 7.4 (2 × $N(CH_2CH_3)_3$), 30.2 (2 × $\mathit{C}H_2N(CH_2CH_3)_3$), 48.8 (2 × $\mathit{C}H_2N(CH_2CH_3)_3$), 54.2 (2 × $N(\mathit{C}H_2CH_3)_3$), 54.6 (2 × $N(\mathit{C}H_2CH_3)_3$) ppm. Note: Quaternary carbons not visible.

2. Solvent Extraction Studies

General procedures

Two-phase extraction experiments were performed by contacting organic phases containing the extracting agent CyMe$_4$-BTBP **1** and aqueous phases containing several metal ions and the masking agents **10** or **13** in nitric acid.

In a first set of experiments, the aqueous phases were solutions containing the masking agent **10** or **13**, Ni(II), Cd(II), Pd(II), Ag(I) (10^{-4} mol/L each), ^{241}Am(III) + ^{152}Eu(III) (1 kBq/mL each) in HNO_3 (1, 2 or 3 mol/L). Masking agent concentrations were 20 mmol/L. The organic phase was a solution of 10 mmol/L CyMe$_4$-BTBP **1** in 1-octanol. Each 0.5 mL of organic and aqueous phases were contacted (30 min at 2500/min) at 20 °C using an orbital shaker. After centrifugation, the phases were separated and ^{241}Am and ^{152}Eu activities were determined. Inactive metal ion concentrations were determined in aqueous samples using ICP-MS.

In a second set of experiments, higher metal ion concentrations were used and the masking agents **10** or **13** were added the day before running the extractions. Shaking times were varied. Aqueous phases were solutions of Ni(II), Cd(II), Pd(II), Ag(I) (10^{-3} mol/L each) in HNO_3 (2 mol/L) with or without a masking agent. Masking agent concentrations were 20 mmol/L. The organic phase was a solution of 10 mmol/L CyMe$_4$-BTBP **1** + 5 mmol/L TODGA in 1-octanol. Inactive metal ion concentrations were determined in the organic (after stripping into glycolate solution; *A/O* = 10) and aqueous samples using ICP-MS. However, mass balances were low in most cases; hence the distribution ratios were determined from the initial and equilibrium aqueous concentrations.

A third set of experiments was made with the ammonium salt masking agent **13** by varying the concentration of the masking agent. Aqueous phases were solutions of Ni(II), Cd(II), Pd(II), Ag(I) (10^{-3} mol/L each) in HNO_3 (2 mol/L). The organic phase was a solution of 10 mmol/L CyMe$_4$-BTBP

1 + 5 mmol/L TODGA in 1-octanol. Inactive metal ion concentrations were determined in the aqueous samples using ICP-MS, and distribution ratios were determined from initial and equilibrium aqueous concentrations.

Distribution ratios (*D*) between 0.1 and 100 exhibit a maximum error of ± 5 %. The error may be up to ± 20 % for smaller and larger values.

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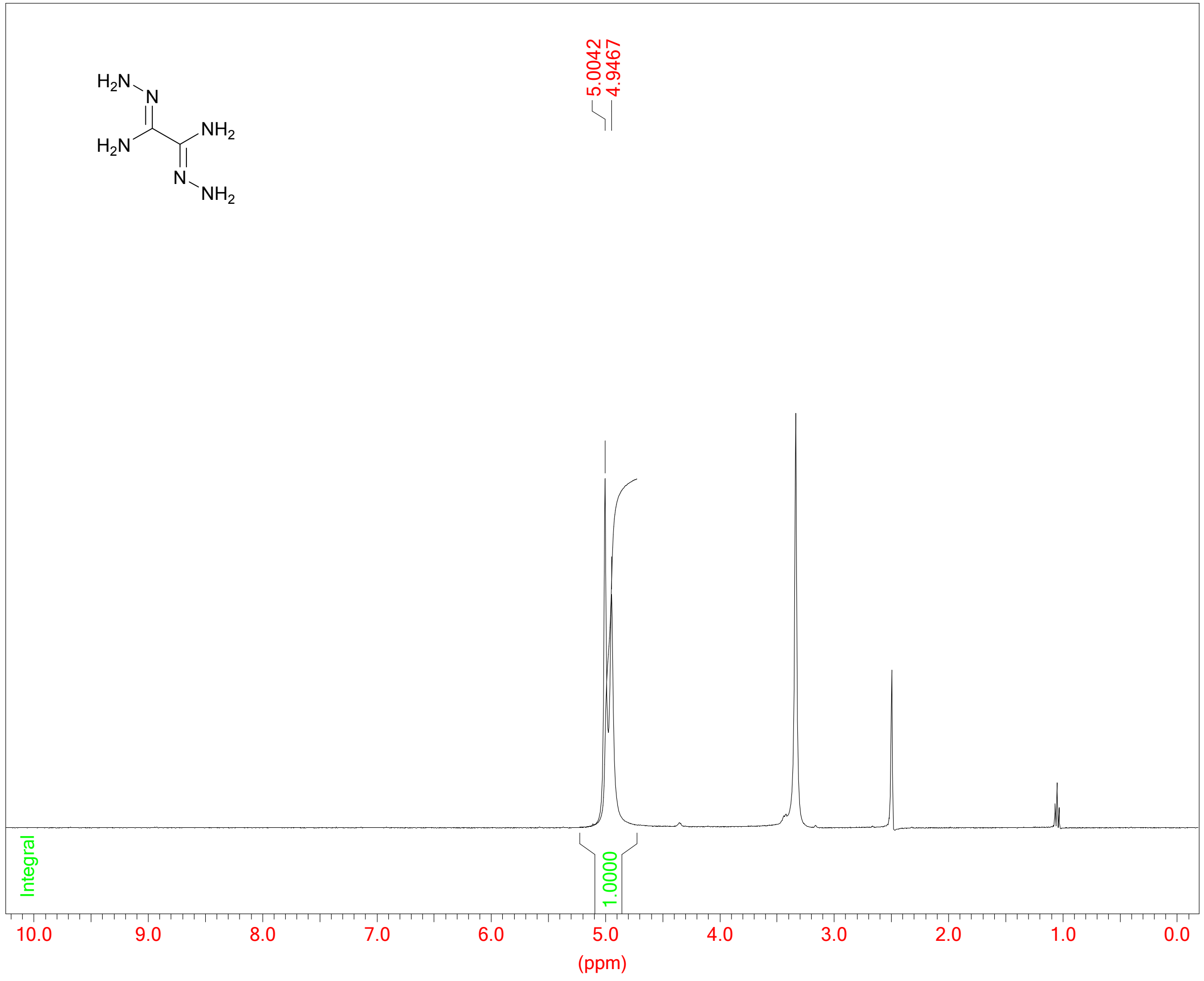

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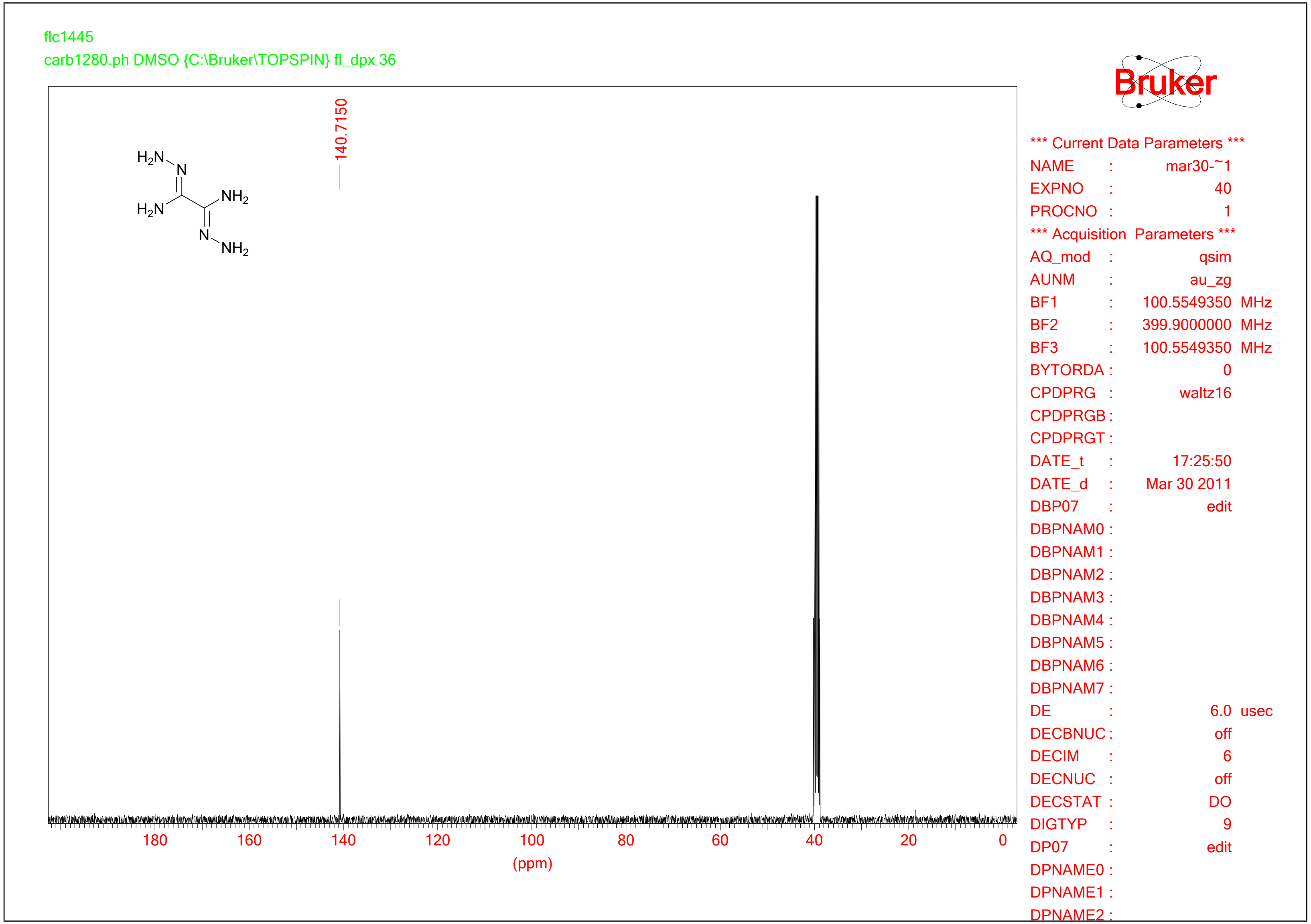

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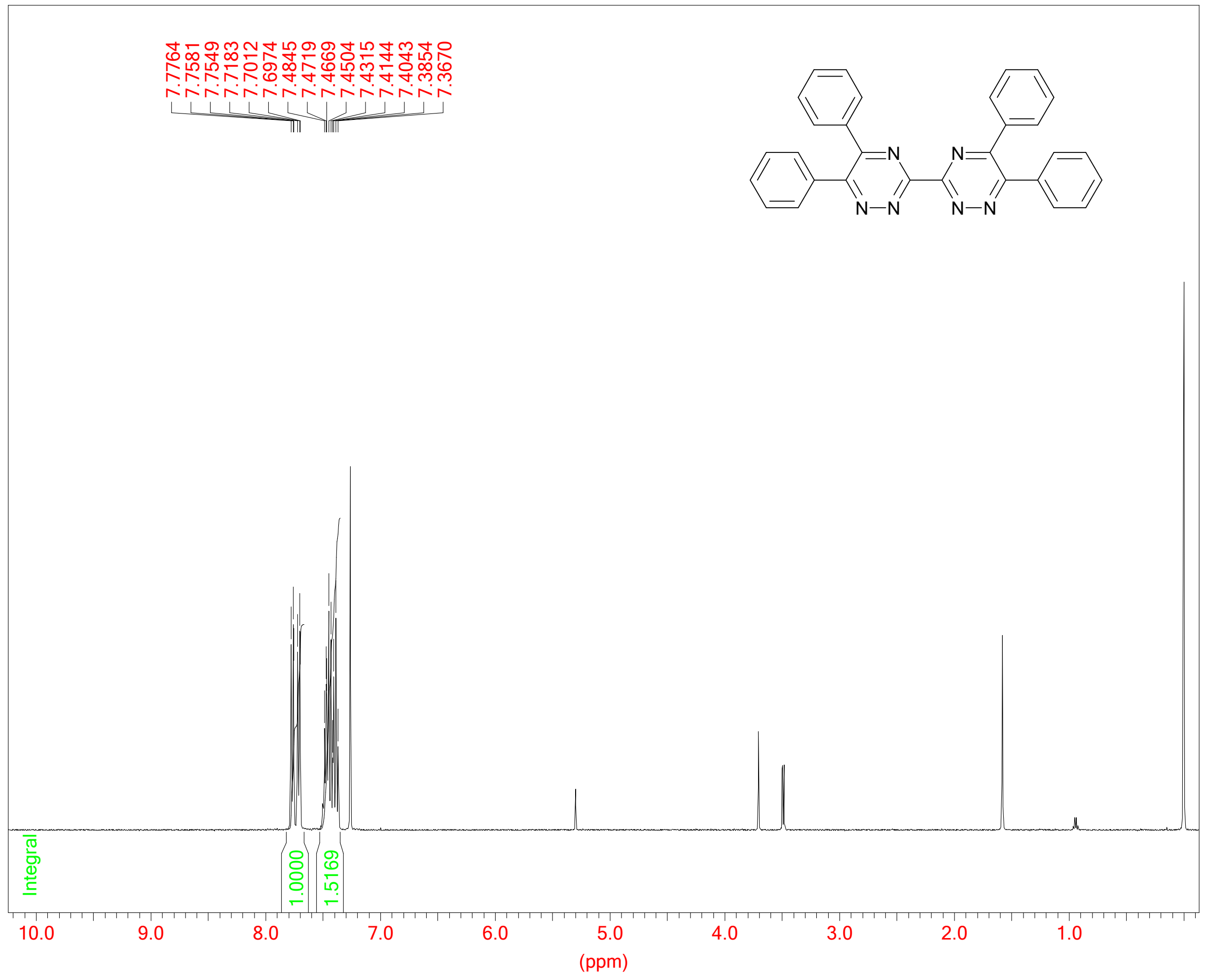

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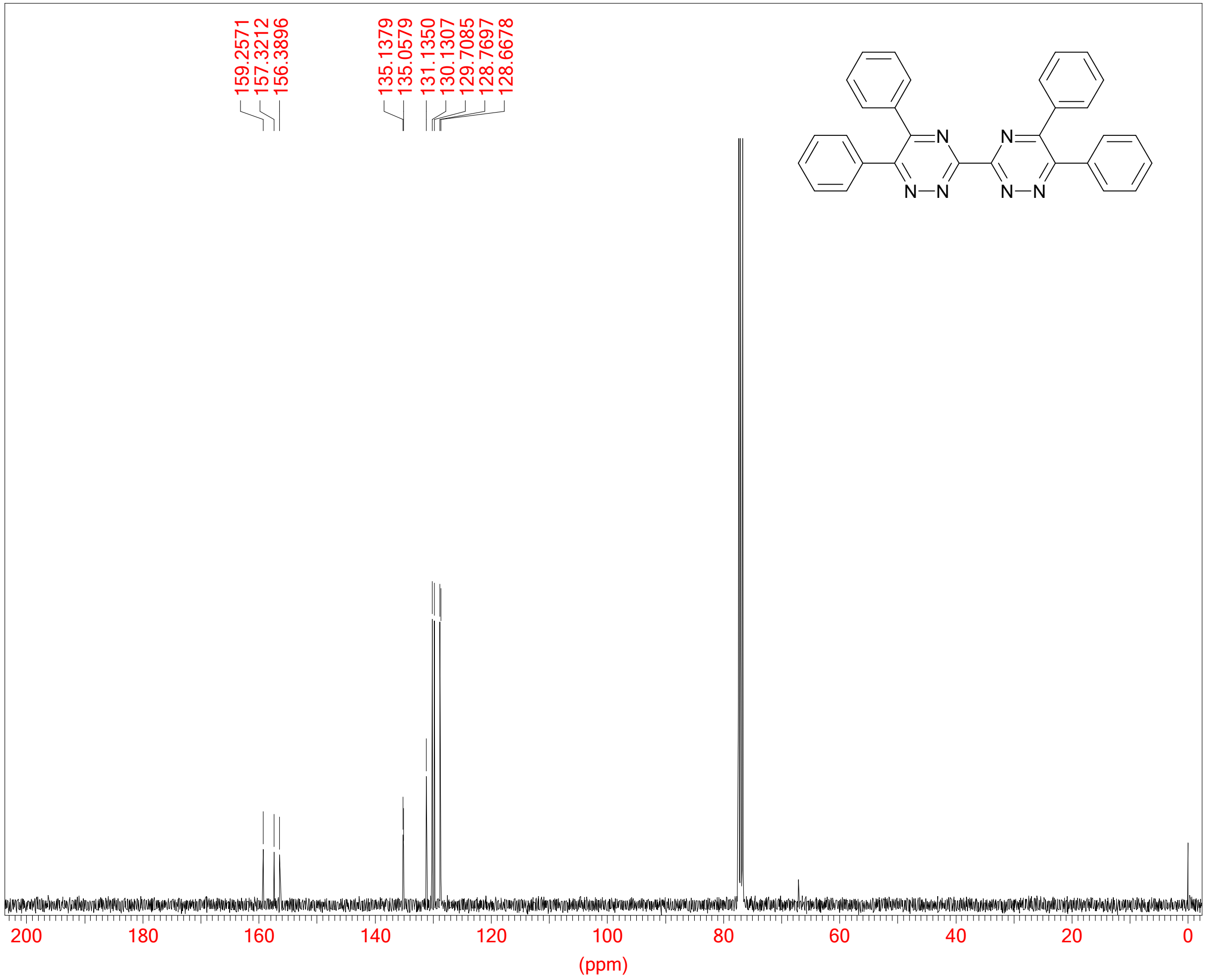

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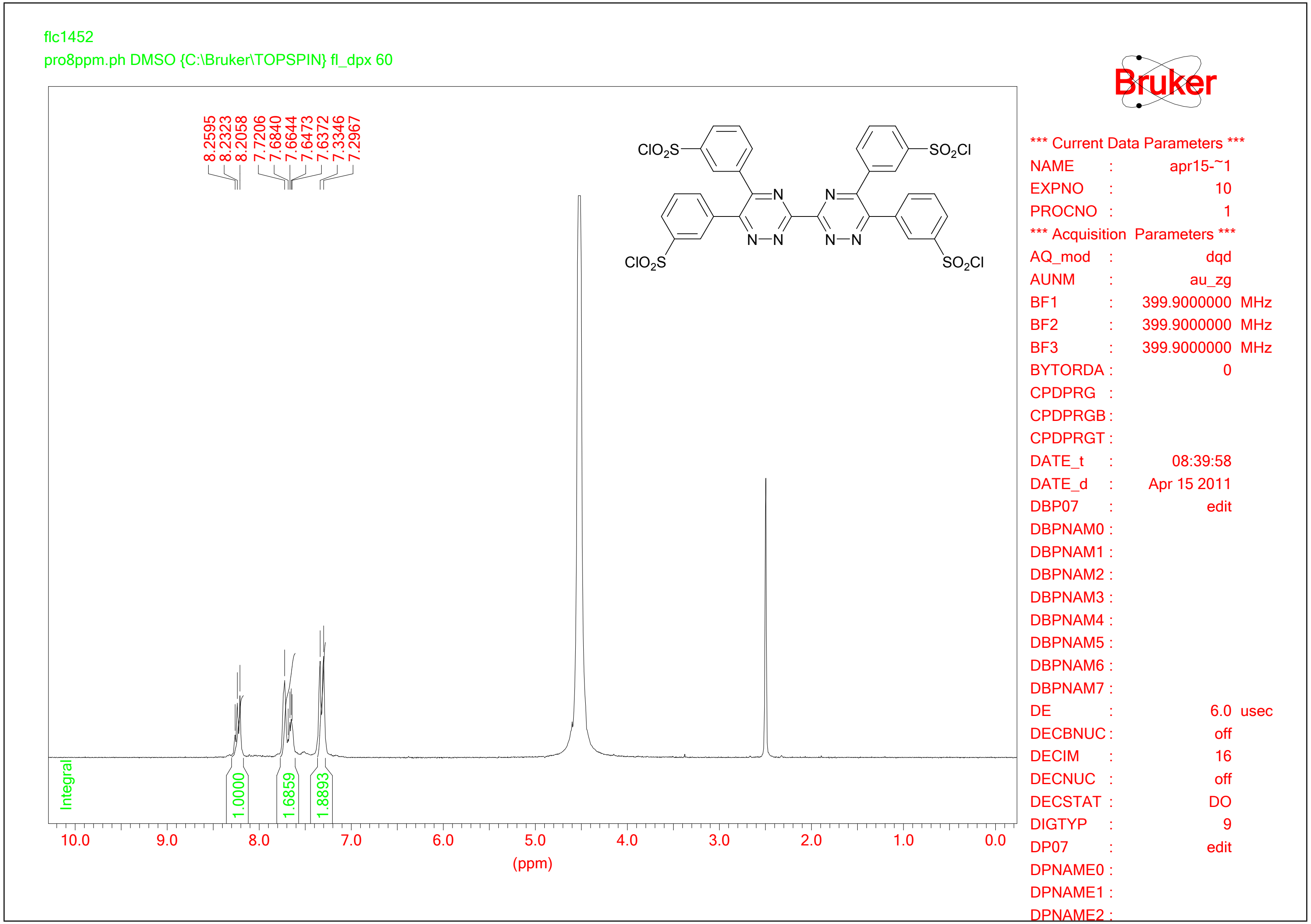

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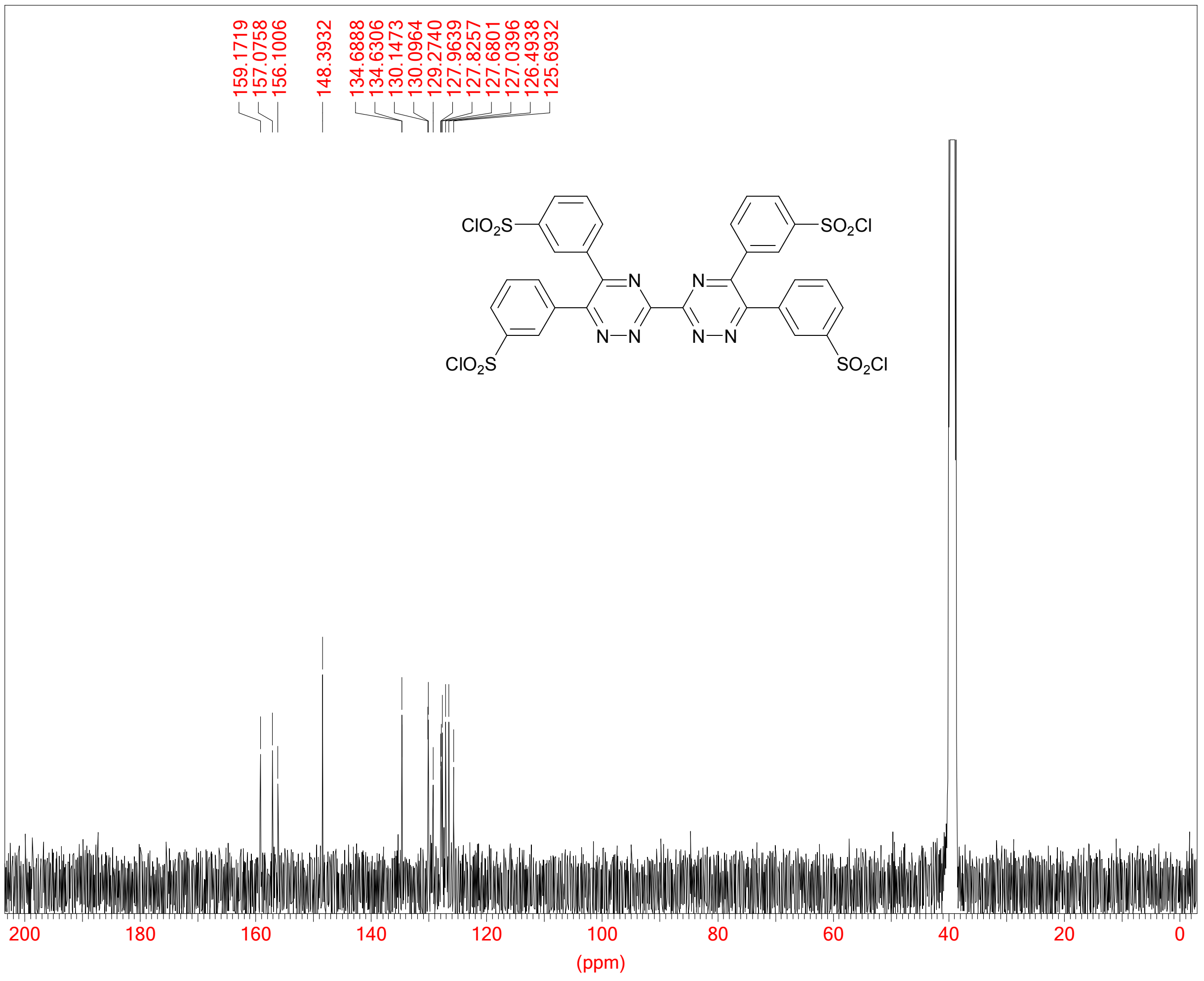

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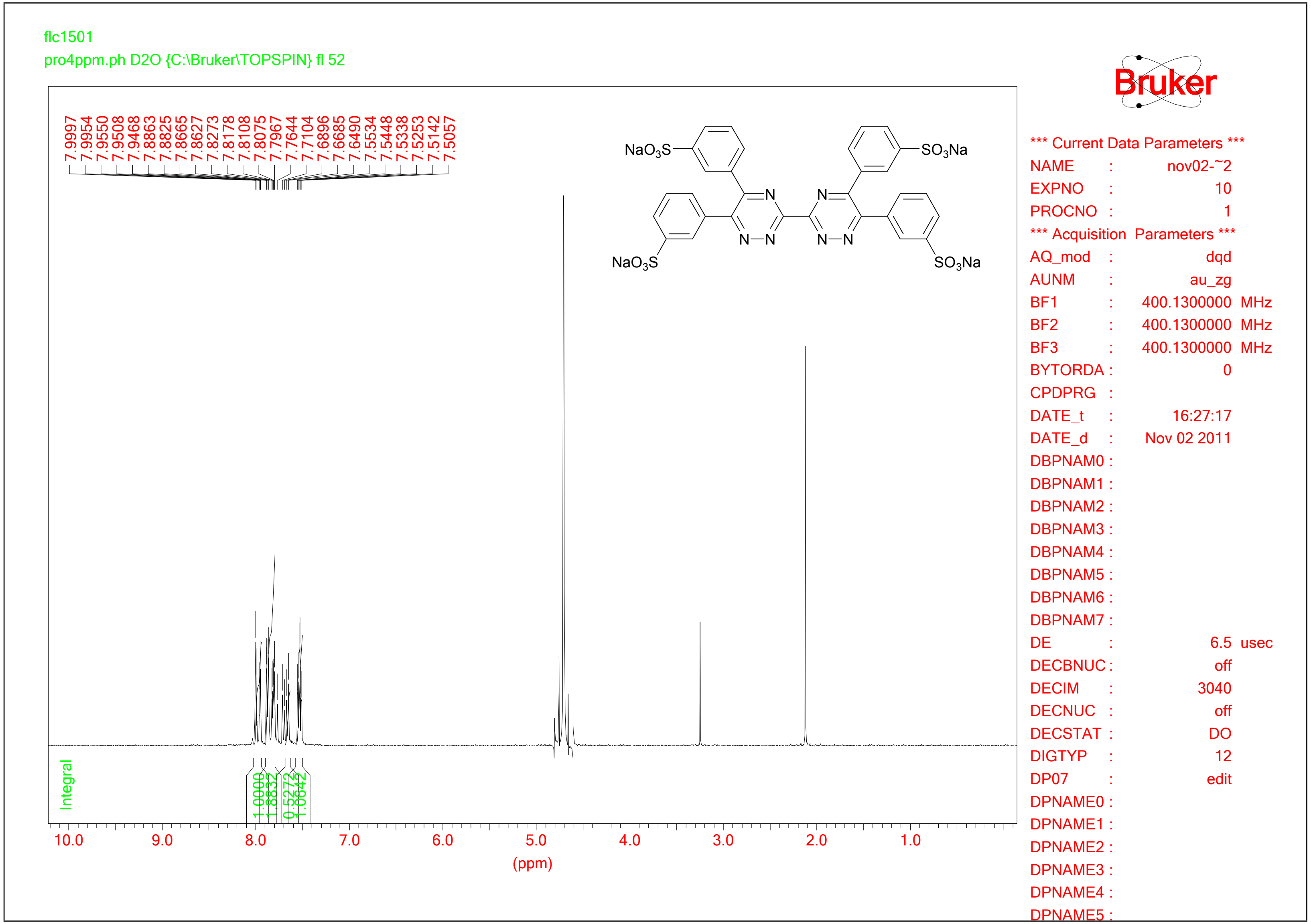

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flc1501
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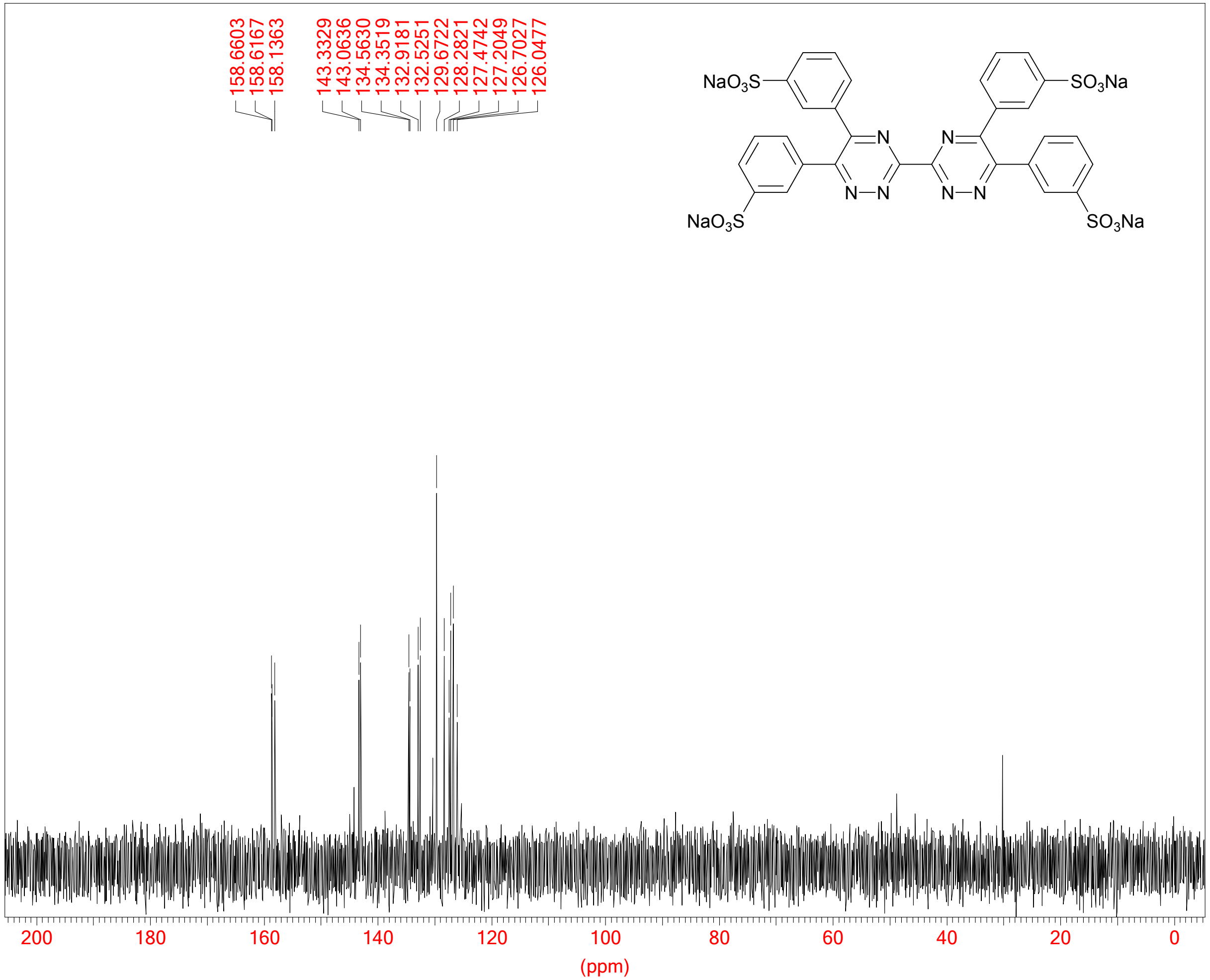
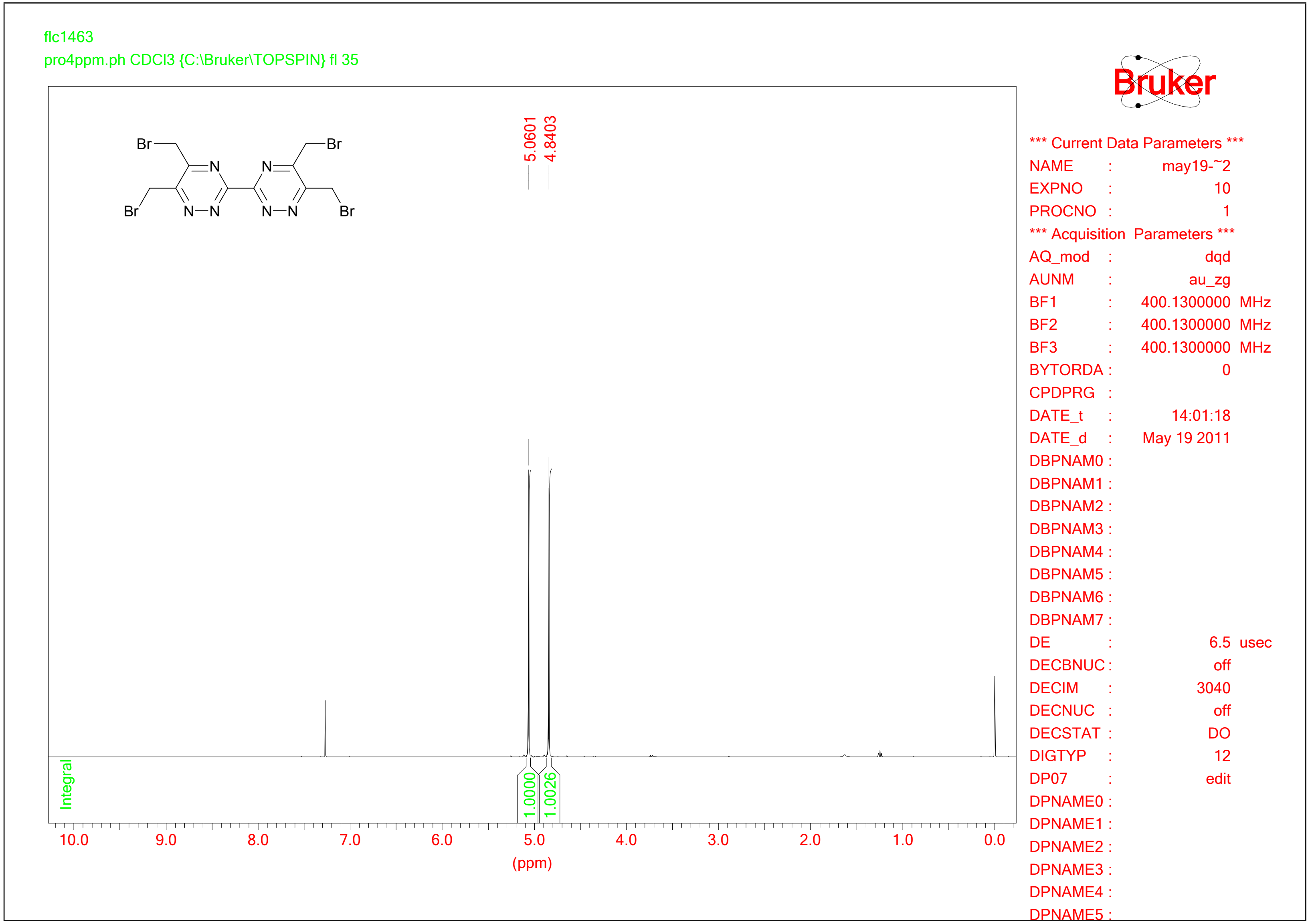
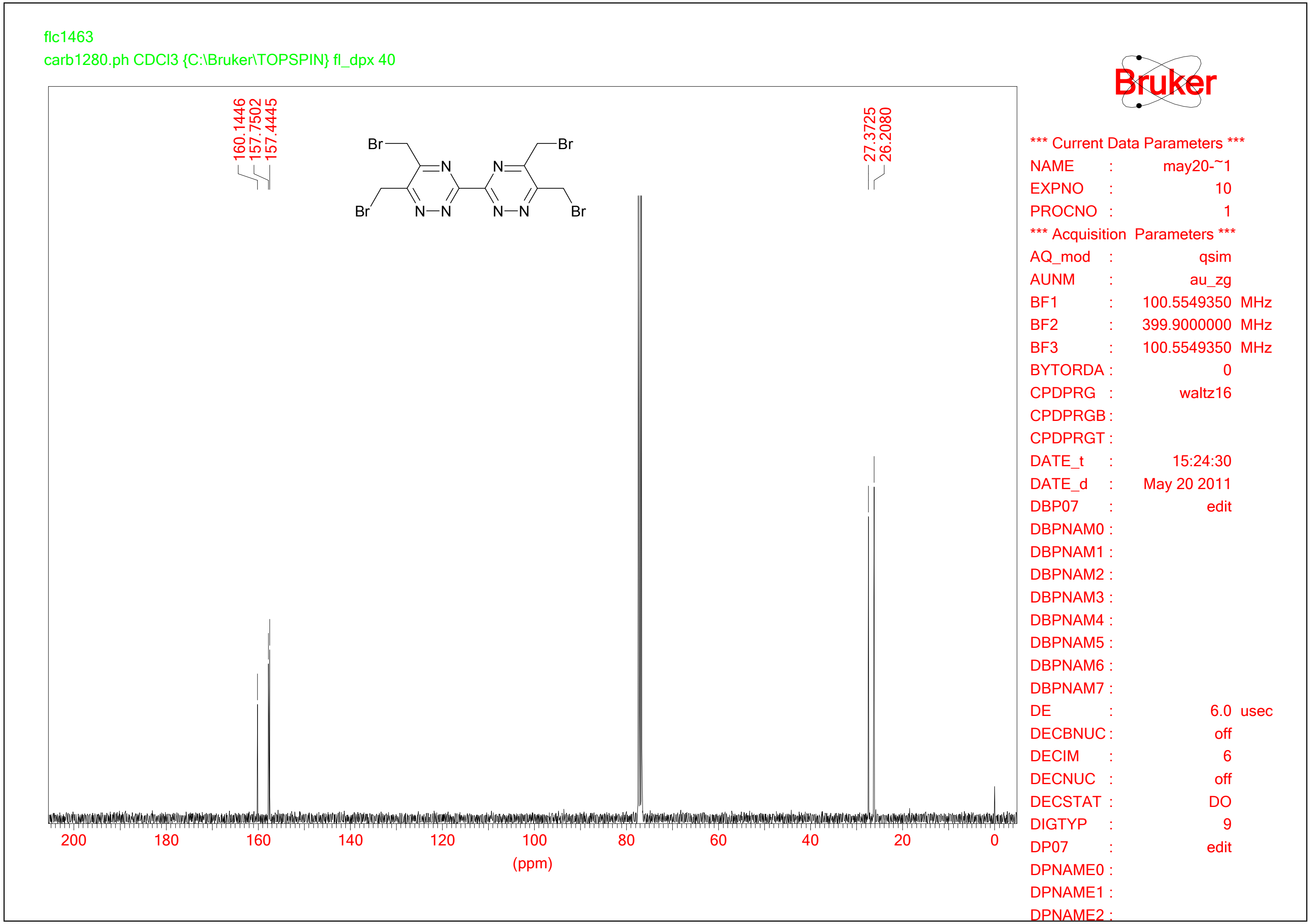


*** Current Data Parameters ***
NAME : nov03-~2
EXPNO : 10
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : qsim
AUNM : au_zg
BF1 : 100.5549350 MHz
BF2 : 399.9000000 MHz
BF3 : 100.5549350 MHz
BYTORDA : 0
CPDPRG : waltz16
CPDPRGB :
CPDPRGT :
DATE_t : 13:45:43
DATE_d : Nov 03 2011
DBP07 : edit
DBPNAM0 :
DBPNAM1 :
DBPNAM2 :
DBPNAM3 :
DBPNAM4 :
DBPNAM5 :
DBPNAM6 :
DBPNAM7 :
DE : 6.0 usec
DECBNUC : off
DECIM : 6
DECNUC : off
DECSTAT : DO
DIGTYP : 9
DP07 : edit
DPNAME0 :
DPNAME1 :
DPNAME2 :



flc1463
pro4ppm.ph CDCl3 {C:\Bruker\TOPSPIN} fl 35

*** Current Data Parameters ***
NAME : may19-~2
EXPNO : 10
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
AUNM : au_zg
BF1 : 400.1300000 MHz
BF2 : 400.1300000 MHz
BF3 : 400.1300000 MHz
BYTORDA : 0
CPDPRG :
DATE_t : 14:01:18
DATE_d : May 19 2011
DBPNAM0 :
DBPNAM1 :
DBPNAM2 :
DBPNAM3 :
DBPNAM4 :
DBPNAM5 :
DBPNAM6 :
DBPNAM7 :
DE : 6.5 usec
DECBNUC : off
DECIM : 3040
DECNUC : off
DECSTAT : DO
DIGTYP : 12
DP07 : edit
DPNAME0 :
DPNAME1 :
DPNAME2 :
DPNAME3 :
DPNAME4 :
DPNAME5 :



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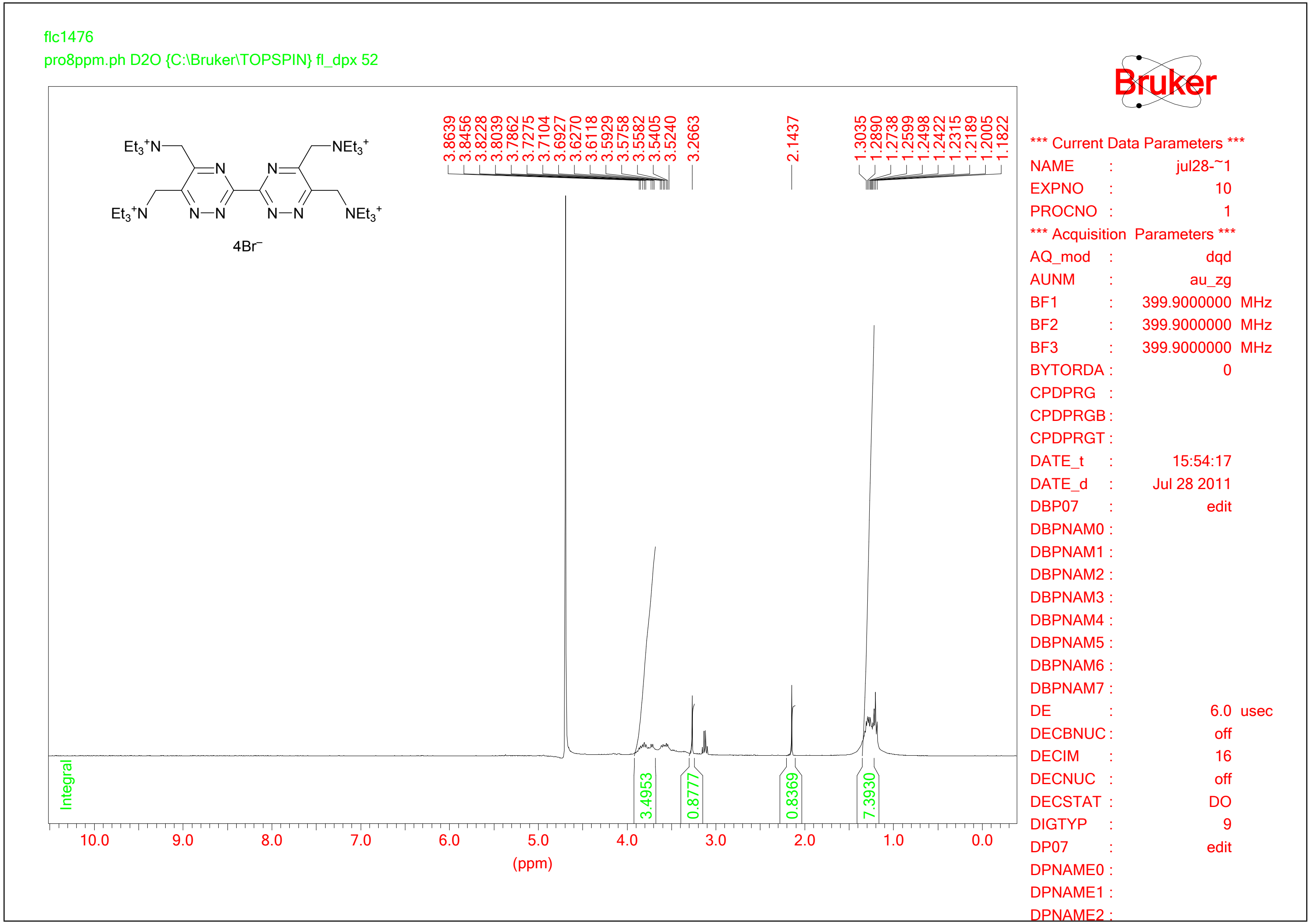
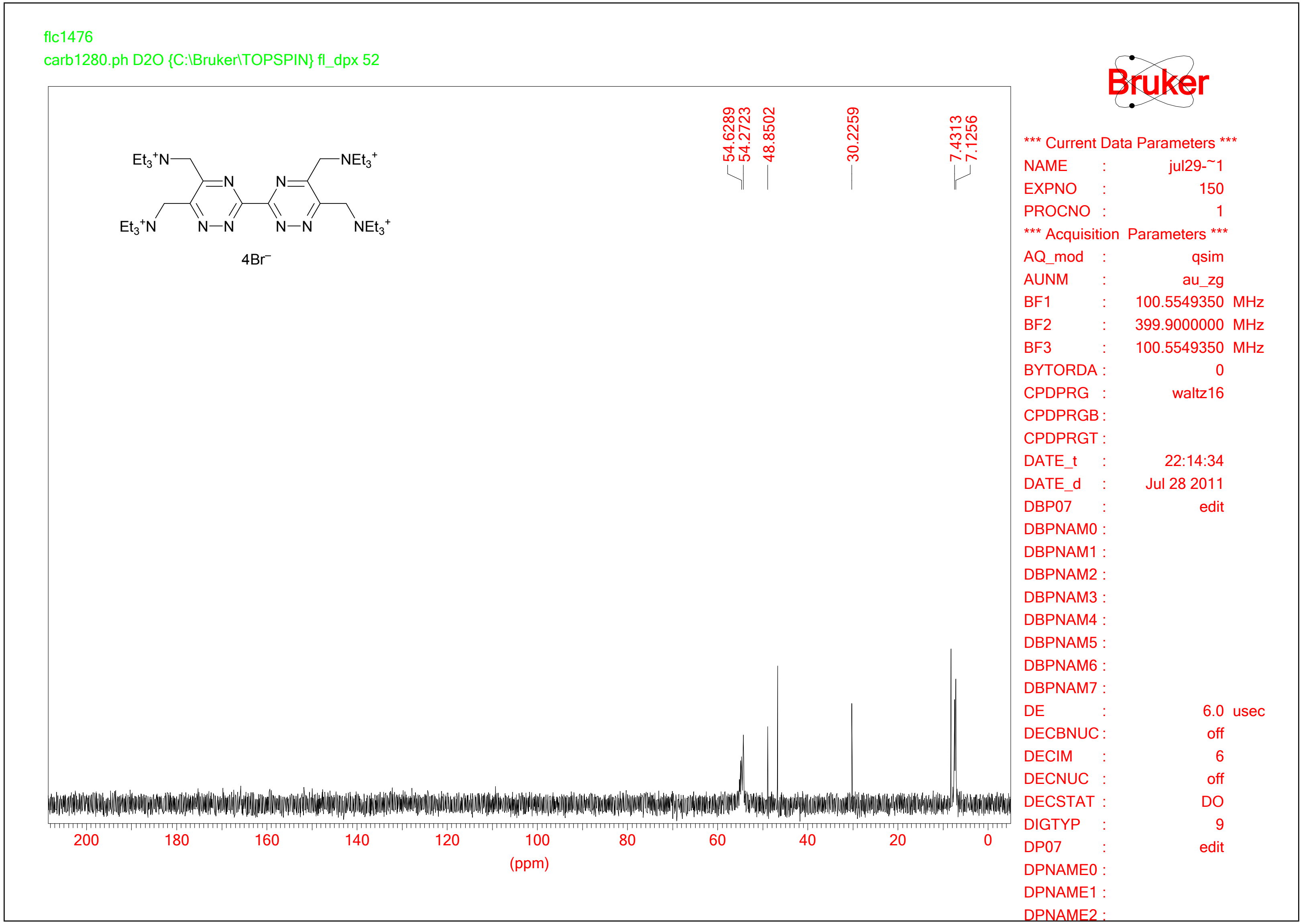
flc1463
carb1280.ph CDCl3 {C:\Bruker\TOPSPIN} fl_dpx 40

*** Current Data Parameters ***
NAME       :        may20-~1
EXPNO      :              10
PROCNO     :               1
*** Acquisition  Parameters ***
AQ_mod     :            qsim
AUNM       :           au_zg
BF1        :     100.5549350  MHz
BF2        :     399.9000000  MHz
BF3        :     100.5549350  MHz
BYTORDA :                  0
CPDPRG     :         waltz16
CPDPRGB :
CPDPRGT :
DATE_t     :        15:24:30
DATE_d     :     May 20 2011
DBP07      :            edit
DBPNAM0 :
DBPNAM1 :
DBPNAM2 :
DBPNAM3 :
DBPNAM4 :
DBPNAM5 :
DBPNAM6 :
DBPNAM7 :
DE         :             6.0  usec
DECBNUC :                off
DECIM      :               6
DECNUC     :             off
DECSTAT    :              DO
DIGTYP     :               9
DP07       :            edit
DPNAME0 :
DPNAME1 :
DPNAME2 :
```



flc1476
pro8ppm.ph D2O {C:\Bruker\TOPSPIN} fl_dpx 52

*** Current Data Parameters ***
NAME : jul28-~1
EXPNO : 10
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
AUNM : au_zg
BF1 : 399.9000000 MHz
BF2 : 399.9000000 MHz
BF3 : 399.9000000 MHz
BYTORDA : 0
CPDPRG :
CPDPRGB :
CPDPRGT :
DATE_t : 15:54:17
DATE_d : Jul 28 2011
DBP07 : edit
DBPNAM0 :
DBPNAM1 :
DBPNAM2 :
DBPNAM3 :
DBPNAM4 :
DBPNAM5 :
DBPNAM6 :
DBPNAM7 :
DE : 6.0 usec
DECBNUC : off
DECIM : 16
DECNUC : off
DECSTAT : DO
DIGTYP : 9
DP07 : edit
DPNAME0 :
DPNAME1 :
DPNAME2 :



flc1476
carb1280.ph D2O {C:\Bruker\TOPSPIN} fl_dpx 52

*** Current Data Parameters ***
NAME : jul29-~1
EXPNO : 150
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : qsim
AUNM : au_zg
BF1 : 100.5549350 MHz
BF2 : 399.9000000 MHz
BF3 : 100.5549350 MHz
BYTORDA : 0
CPDPRG : waltz16
CPDPRGB :
CPDPRGT :
DATE_t : 22:14:34
DATE_d : Jul 28 2011
DBP07 : edit
DBPNAM0 :
DBPNAM1 :
DBPNAM2 :
DBPNAM3 :
DBPNAM4 :
DBPNAM5 :
DBPNAM6 :
DBPNAM7 :
DE : 6.0 usec
DECBNUC : off
DECIM : 6
DECNUC : off
DECSTAT : DO
DIGTYP : 9
DP07 : edit
DPNAME0 :
DPNAME1 :
DPNAME2 :

4. References

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