Supporting Information

Design of Multivalent Galactosyl Carborane as a Targeting Specific Agent for Potential Application to Boron Neutron Capture Therapy

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(1) Materials. *N*-((5-Hexynyl) phthalimide (Aldrich), acetonitrile (CH$_3$CN, Merck),
toluene (Merck), sodium borohydride (NaBH$_4$, Acros), iso-propanol (i-PrOH, Merck),
hydrochloric acid (HCl, Acros), acetic acid (HOAc, Acros), dimethyl formaldehyde
(DMF, Merck), triethyl amine (NEt$_3$, Merck), 4-dimethylaminopyridine (DMAP,
Acros), 4-nitrophenyl chloroformate (NPCC, Acros), O-(7-azabenzotriazol-1-yl)-
1,1,3,3-tetramethyluronium hexafluorophosphate (HATU, Biochem), 2-(1H-
Benzotriazole-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate (HBTU,
Anaspec), copper(I) iodide (CuI, Acros), copper(II) sulfate (CuSO$_4$, Merck), sodium
ascorbate (Acros), ethanol (EtOH, Merck), tert-butanol (t-BuOH, Acros),
3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide (MTT, Sigma),
decaborane (B$_{10}$H$_{14}$, CMS), and sodium borocaptate (Na$_2$B$_{12}$H$_{11}$SH or BSH, Katchem
Ltd., Prague, Czech Republic) were used as received. The P2 gel was purchased from
Bio-Rad. Analytical thin-layer chromatography (TLC) and reverse-phase TLC were
performed using pre-coated plates (Silica Gel 60 F$_{254}$ and 60 RP-18F$_{254S}$, respectively,
Merck). Silica gel 60 and C-18 reverse-phase gel (Merck) were used for flash
chromatography. All reactions were carried out in oven-dried glassware (120 °C)
under a nitrogen atmosphere unless indicated otherwise. All solvents were dried and
distilled using standard techniques.

(2) General Measurements. $^1$H-, $^{13}$C- and $^{11}$B-NMR spectra were recorded using a
Bruker AV-400 or a DMX-600 MHz spectrometer. Chemical shifts are expressed in
ppm and were referenced to the residual CDCl$_3$ (7.24 ppm) or CD$_3$OD (3.31 ppm)
resonances, which served as internal standards. The coupling constants, $J$, are
reported in Hz.
(3) Synthesis of chemical compounds.

Pent-4-ynoic acid \{5-(bis\{6-(beta-D-galactopyranosyl)-hexylcarbamoyl\}-methyl\}-amino)-5- \{6-(beta-D-galactopyranosyl)-hexylcarbamoyl\}-pentyl\}-amide

(4) A solution of compound 1 \((N,N\text{-Bis[carboxymethyl]\text{-L-lysine}},^1 \text{145 mg, 0.55 mmol})\) and compound 2 \(2 \text{ (130 mg, 0.66 mmol)}\) in anhydrous DMF and H\(_2\)O (3/1, 8 mL) was added NEt\(_3\) (76.5 \(\mu\text{L, 0.55 mmol})\) at 4°C. The resulting solution was stirred for 16 h under \(\text{N}_2\). After completion of the reaction monitored by TLC, the solvent was evaporated. The crude mixture was purified by column chromatography (CH\(_3\)CN/H\(_2\)O, 1/100–1/15 gradient) on reverse-phase silica gel (98 mg, 52%). The above pure product (100 mg, 0.29 mmol) was dissolved in anhydrous DMF (5 mL) followed by addition of compound 3 \(3 \text{ (6-aminohexyl-beta-D-galactopyranoside,}^3 \text{ 327 mg, 1.17 mmole)}\), HATU (445 mg, 1.17 mmol) and NEt\(_3\) (168 \(\mu\text{L, 1.17 mmol})\) under nitrogen. The reaction was kept stirring at 45 °C for 16 h. After removal of solvent, the resulting residue was subjected to P2 gel filtration (eluent: H\(_2\)O) and then reverse-phase silica gel chromatography (CH\(_3\)CN/H\(_2\)O, 1/8–1/4 gradient) to give 4 \(168 \text{ mg, 51% yield})\). \(^1\text{H NMR (400 MHz, MeOD,} \delta\): 1.46 (14H, m), 1.53 (8H, m), 1.63 (8H, m), 2.29 (1H, m), 2.39 (2H, m), 2.45 (2H, m), 3.04-3.29 (8H, m), 3.29-3.64 (17H, m), 3.74 (6H, m), 3.85 (3H, m), 3.90 (3H, m), 4.22 (3H, d, \(J = 6.24 \text{ Hz})\); \(^13\text{C NMR (150 MHz, MeOD,} \delta\): 14.49, 23.58, 25.39 × 3, 26.45 × 3, 28.84, 28.98 × 3, 29.33 × 3, 34.74, 38.80, 38.84, 38.98, 55.80, 61.10 × 3, 65.87, 68.93 × 3, 69.25, 69.37
× 3, 71.22 × 3, 73.65 × 3, 75.14 × 3, 82.39, 103.55 × 3, 171.38, 172.45, 172.58, 173.23; IR (KBr): 2042 cm⁻¹ (alkyne, C≡C), 1643 cm⁻¹ (C=O); HRMS (ESI) calcd for C₅₁H₉₁N₅O₂₂Na [M + Na]^+ 1148.6053, found 1148.6063.

Scheme S1. Synthesis of the carborane derivative compound 7. Reaction condition: a) B₁₀H₁₄, CH₃CN, toluene, reflux, 4 h, 43%. b) NaBH₄, i-PrOH, rt, 22 h, 76%. c) HCl/HOAc, 95°C, 3 h, 95%

1-(4-Phthalimidobutyl)-1,2-dicarba-closo-dodecaborane (6) A modified procedure from the literature⁴ was employed. A solution of decaborane (1.00 g, 8.18 mmol) in 40 mL of dry toluene and 6.8 mL of dry acetonitrile under nitrogen was heated at reflux for 1 h. A solution of compound 5 (N-(5-Hexynyl) phthalimide, 1.86 g, 8.18 mmol) in 10 mL of dry toluene was then added and solution was heated at reflux for 3 h. The solution was filtered from insoluble material, cooled, and filtered again. Solvent was evaporated at reduced pressure. Flash chromatography of the residue (ethyl acetic acid/hexane 1/20-1/8) gave 1.2 g (43% yield) of the carborane derivative 6 as a white solid. ¹H NMR (400 MHz, CDCl₃, δ): 1.47 (2H, m), 1.63 (2H, quint, J = 7.2 Hz), 2.25 (2H, m), 3.59 (1H, s), 3.63 (2H, t, J = 7.0 Hz), 7.70 (2H, m), 7.81 (2H,
((2-(Hydromethyl)benzoyl)amino)butyl)-o-carborane (S1) To a suspension of compound 6 (556 mg, 1.61 mmole) in i-PrOH (17 mL) and H2O (2.8 mL) was added NaBH4 (349 mg, 8.05 mmole) and the resultant mixture was stirred under nitrogen at room temperature for 22 h. The result solution was evaporated at reduced pressure. The mixture was vigorously extracted with hot water twice (5 mL) and the dried product was recrystallized from ethanol (2.4 mL) and water (1.3 mL) to give product S1 (424 mg, 76% yeild). 1H NMR (400 MHz, CDCl3, δ): 1.57 (4H, m), 2.26 (2H, m), 3.42 (2H, dt, J = 6.4, 12.6 Hz), 3.60 (1H, s), 4.57 (2H, s), 6.57 (1H, s), 7.35 (2H, m), 7.44 (1H, m), 7.51 (1H, m); 13C NMR (100 MHz, CDCl3, δ): 26.38, 28.95, 37.48, 39.22, 61.22, 64.65, 74.86, 127.63, 128.27, 130.82, 131.31, 135.54, 139.71, 170.07; 11B NMR (192 MHz, CDCl3, δ): -3.93 (1B), -7.36 (1B), -10.92 (2B), -13.17 (2B), -13.78 (2B), -14.68 (2B); IR (KBr): 2599(B-H), 2571(B-H), 1619 (C=O) cm-1; HRMS (ESI) calcd for C14H27B10NO2Na [M + Na]+ 374.2870, found 374.2870.

(Aminobutyl)-o-carborane Hydrochloride (7) A solution of the compound S1 (1.00
was heated on a water bath for 3 h. After removal of the solvent by high vacuum, the residue was stirred with dichloromethane (15 mL) for 2 h. The finely crystalline hydrochloride was collected (583 mg, 95% yield). $^1$H NMR (400 MHz, MeOD, $\delta$): 1.60 (4H, m), 2.33 (2H, dd, J = 6.8, 8.9 Hz), 2.90 (2H, dd, J = 6.7, 7.2 Hz), 4.59 (1H, s); $^{13}$C NMR (100 MHz, MeOD, $\delta$): 26.01, 26.50, 36.75, 38.84, 62.42, 75.43; $^{11}$B NMR (192 MHz, MeOD, $\delta$): -4.43 (1B), -7.61 (1B), -11.23 (2B), -13.28 (2B), -13.59 (2B), -14.70 (2B); IR (KBr): 2588 (B-H) cm$^{-1}$; HRMS (ESI) calcd for C$_6$H$_{22}$B$_{10}$N [M]$^+$ 218.2683, found 218.2686.

Scheme S2. Synthesis of linker compound 8.

**Carbonic acid 6-azido-hexyl ester 4-nitro-phenyl ester (8)** 6-Azido-hexan-1-ol, S2 (1.00 g, 6.98 mmol) was added to a solution of DMAP (2.13 g, 17.46 mmol) in pyridine, and followed by the addition of 4-nitrophenyl chloroformate (3.52 g, 17.46 mmol) in ice bath. The yellow mixture reaction was stirred for 16 h at room temperature. After completion of the reaction as monitored by TLC, the reaction was evaporated. Then, the residue was added EA and the organic layer was washed with water and brine solution. The organic layer was dried (MgSO$_4$) and concentrated. The product was purified by column chromatography (EA/Hexane, 1:20-1:3) on silica gel to give compound 8 (1.85 g, 86%). $^1$H NMR (400 MHz, MeOD, $\delta$): 1.42 (4H, m), 1.61 (2H, m), 1.76 (2H, m), 3.26 (2H, t, J = 6.8 Hz), 4.27 (2H, t, J = 6.6 Hz), 7.35 (2H, d, J = 9.2 Hz), 8.26 (2H, d, J = 9.2 Hz); $^{13}$C NMR (100 MHz, MeOD, $\delta$): 25.27, 26.30, 28.37, 28.69, 51.28, 69.35, 115.61, 121.61, 125.30, 126.21, 152.55; HRMS
(FAB) calcd for C\textsubscript{13}H\textsubscript{17}N\textsubscript{4}O\textsubscript{5} [M + H]\textsuperscript{+} 309.1199, found 309.1194.

\[\text{[5-{Bis-[(4-o-carborane-butylcarbamoyl)-methyl]-amino}-5-(4-o-carborane-butyl carbamoyl)-pentyl]- carbamic acid 6-azido-hexyl ester (9)}\]

A solution of compound 1 (\(N,N\)-Bis[carboxymethyl]-L-lysine\textsuperscript{,1} 300 mg, 1.15 mmol) and compound 8 (424 mg, 1.37 mmol) in anhydrous DMF and H\textsubscript{2}O (3/1) 6 mL was added NEt\textsubscript{3} (399 \(\mu\text{L}, 2.88 \text{ mmol}) and stirred at room temperature for 16 h under N\textsubscript{2}. After completion of the reaction monitored by TLC, the solvent was evaporated. The crude mixture was purified by column chromatography (MeOH/DCM, 3/100–1/1 gradient) on silica gel (437 mg, 88.5%). The product (135 mg, 0.31 mmol) was further dissolved in anhydrous DMF (3 mL) and followed by addition of compound 7 (268 mg, 1.25 mmole), HBTU (474 mg, 1.25 mmol) and NEt\textsubscript{3} (173 \(\mu\text{L}, 1.25 \text{ mmol}) under nitrogen. The reaction was kept stirring at 45 °C for 16 h. After solvent removal, the residue was added EA and the organic layer was washed with water and brine solution. The organic layer was dried (MgSO\textsubscript{4}) and concentrated. The product was subjected silica gel chromatography (MeOH/DCM, 1/100–1/10 gradient) to give 9 (170 mg, 53% yield). \(^1\text{H} \text{NMR} (400 \text{ MHz, CDCl}_3, \delta): 1.38 (4H, m), 1.47 (12H, m), 1.59 (6H, m), 1.82 (4H, m), 2.20 (6H, m), 3.10-3.40 (13H, m), 3.63 (4H, m), 3.70 (1H, m), 4.00 (2H, m), 4.87 (1H, b), 7.22 (1H, b), 7.51 (2H, b); \(^{13}\text{C} \text{NMR} (100 \text{ MHz, CDCl}_3, \delta): 23.58, 25.46, 26.47, 27.16, 28.70, 28.81, 29.11, 37.43, 38.38, 38.57, 38.68, 40.26, 51.28, 56.15, 61.33, 64.87, 74.97, 157.17, 171.48, 172.28; \(^{11}\text{B} \text{NMR} (192 \text{ MHz, CDCl}_3, \delta):
-4.01 (3B), -10.96 (3B), -13.20 (6B), -12.00 ~ -18.00 (18B); IR (KBr): 2588 (B-H), 2096 (N-N), 1645 (C=O) cm⁻¹; HRMS (ESI) calcd for C₃₅H₈₆B₃₀N₈O₅Na [M + Na]⁺ 1051.9410, found 1051.9420.

Tri-galactosyl-linker-tri-carborane (10) Compound 9 (50mg, 48.85 μmol) and compound 4 (55 mg, 48.83 μmol) were dissolved in EtOH/H₂O/t-BuOH (3/2/5) 6mL. Sodium ascrobate (1 mg, 5.05 μmol), CuI (1mg, 5.25 μmol), NEt₃ (50 μl, 0.36 mmol) and CuSO₄ were added. The reaction mixture was stirred at room temperature for 65 h and progress was monitored by TLC. Upon completion of reaction, the solvent was removed under reduced pressure and the crude product was purified by using P2 gel filtration (eluent, H₂O) to give compound 10 (102 mg, 98% yield). ¹H NMR (600 MHz, MeOD, δ): 1.25-1.45 (30H, m), 1.45-1.53 (14H, m), 1.53-1.76 (8H, m), 1.86-2.00 (4H, m), 2.26-2.38 (8H, m), 2.50-2.60 (2H), 3.00-3.25 (21H, m), 3.37-3.60 (18H, m), 3.62 (1H, s), 3.74 (6H, m), 3.84 (3H, m), 3.89 (3H, m), 4.01 (2H, m), 4.21 (3H, d, J = 7.44 Hz), 4.37 (1H, m), 4.53 (3H, s); ¹³C NMR (150 MHz, MeOD, δ):24.85, 24.97,26.40, 26.56, 26.72, 26.78, 27.16, 27.48, 27.83, 29.74, 29.84, 30.37, 30.52, 30.72, 31.15, 38.36, 39.50, 39.63, 40.17, 40.33, 41.47, 51.29, 52.36, 52.41, 57.07, 62.49, 63.68, 65.63, 67.07, 67.14, 70.31, 70.67, 72.59, 75.05, 76.57, 77.16, 104.99, 159.17, 173.79, 173.92, 174.45, 174.59, 174.70; ¹¹B NMR (192 MHz, CDCl₃, δ): -4.49 (3B), -7.63 (3B), -11.28 (6B), -13.20 (12B), -14.71 (6B); IR (KBr): 2584
(B-H), 1647 (C=O) cm$^{-1}$; HRMS (MALDI) calcd for C$_{86}$H$_{177}$B$_{30}$N$_{13}$O$_{27}$Na [M + Na]$^+$ 2172.7162, found 2172.3516.

(4) The $^{11}$B-NMR, mass data, HPLC and IR of boron compounds

Figure S1. The $^{11}$B NMR spectra of all carborane derivatives give the similar B atom peaks.
Figure S2. The mass spectrometry identified carborane derivatives compound 10. Due to the presence of two natural isotopes of boron (\(^{10}\text{B}, 19.6\%\) and \(^{11}\text{B}, 80.4\%\)), the spectrum shows characteristic isotope patterns.
Figure S3. The HPLC spectrum of compound 10. Column: Vydac C18-column (Cat#218TP54, 4.6mm I.D. *250 mm); Eluent: H$_2$O (0.1%TFA )/ Acetonitrile.

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Column: Vydac C18-column (Cat#218TP54, 4.6mm I.D. *250 mm)
Eluent: H$_2$O (with 0.1%TFA ) / Acetonitrile

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Figure S3. The HPLC spectrum of compound 10. Column: Vydac C18-column (Cat#218TP54, 4.6mm I.D. *250 mm ); Eluent: H$_2$O (0.1%TFA )/ Acetonitrile.
(5) Cell culture conditions

HepG2 and Hela cells were cultured in 10 cm$^2$-dish with Dulbecco's Modified Eagle's Medium (DMEM, Gibco), 10% heat-inactivated fetal bovine serum (FBS, Gibco), 100 U/mL penicillin (Gibco), 100 µg/mL streptomycin (Gibco), and 0.25 µg/mL Fungizone (BioSOURCE). Cells were incubated at 37 °C in an ambient air/5% CO$_2$ atmosphere and subcultured every 3 days.

(6) MTT assay of compound 10 or BSH treated HepG2 and Hela Cells

HepG2 cells (1.0 × 10$^4$) and Hela cells (5.0 × 10$^4$) were respectively seeded in 100 µL DMEM containing 10% FBS in the wells of a 96-well plate and allowed to attach overnight. Because HepG2 and Hela are different in cell size, this two different cell lines were grown to fill up the dish with different cell number. Compound 10 or BSH with series different boron concentration was added to each well, and then the cells were incubated at 37 °C for 6 or 72 h. Cells were washed with PBS, and then 10
μL of 12 mM MTT in PBS and 90 μL DMEM were added to each well. After incubation for another 4 h, 75 μL of medium was removed and 100 μL of DMSO was added to each well. Each mixture was pipetted to ensure complete dispersion of DMSO and then incubated at 37 °C for 10 min. The cell cytotoxicity was determined by measuring the absorption of the cell lysates at 570 nm using a SpectraMax M2e microplate reader. Each optical absorption at 570 nm was related to cell viability by comparison with a control experiment which was carried out under the same conditions except that compound 10 or BSH was not added.

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Figure S5. The MTT assay results of compound 10 treated HepG2 and Hela cells. The cells were culture with series concentrations of compound 10 for 6 h at 37 °C, and then MTT assay was applied to measure cell viability.
Figure S6. The cells were culture with series concentrations of compound 10 for 6 h or 72 h, and then MTT assay was applied to measure cell viability.

Figure S7. The MTT assay was used to determine cell viability of HepG2 cells treated with 25 ppm and 50 ppm, respectively, of BSH for 6 h.

(7) Soft-agar colony formation assay of compound 10 treated HepG2 cells
Figure S8. The colony formation was applied to prove that the compound 10 does not influence cell proliferation. The tumor mass colonies were formed in the melting-temperature agarose (Sigma). The images were captured randomly by TE-2000 inverted microscope equipped with Nikon D50 digital camera (Nikon). The size of tumor was all measured in diameter, and the statistic was normalized with the control group.

(8) Intracellular boron uptake measured by ICP-MS

For boron uptake measurements, HepG2 cells were seeded in the wells of a 6-well plate. When the cells were grown to fill up the dish, the cell number was counted (about 1-2 × 10⁶ cells/well). HepG2 cells were cultured in medium supplemented with boron containing compound 10 (in boron concentration: 12.5, 25, 50 ppm) or BSH (in boron concentration: 50 ppm), respectively. After a 6 h treatment, the medium was removed and the cells were washed with PBS, and then trypsinized to collect cell residues in eppendorf. The cell residues were further digested with 300 μL of 60% HClO₄/30%H₂O₂ (1:2) solution, and then decomposed for 3 h at 80 °C. After cool to room temperature, 700 μL of dd-H₂O was added to the each eppendorf. The boron concentration was determined by ICP-MS (inductively coupled plasma-mass spectrometer, Perkin Elmer, SCIEX ELAN 5000). Finally, the cellular uptakes of boron were calculated based on the ICP-MS data and the number of the
HepG2 cells present in the sample. Three repetitions of each experiment were carried out, and the data are indicated in Figure 1.

(9) Thermal neutron irradiation of HepG2 cells

An adequate number of HepG2 cells in 1 mL of complete Dulbecco’s modified Eagle medium (DMEM) were seeded in the wells of a 12-well plate and incubated at 37 °C in a CO₂ incubator for 20 h. HepG2 cells were then incubated in 50 μg B/mL (boron concentration at 50 ppm) of compound 10 or BSH containing media for 6 h. Cells untreated with boron drug were used as the controls. The cell culture plates were removed from the incubator and transported to Tsing Hua Open Pool Reactor (THOR), inserted into a phantom (20x20x10 cm³) and irradiated with an thermal neutron flux of 1.1 × 10⁹ n/cm² s in an ambient temperature (25 ± 3 °C) at various time intervals to obtain different thermal neutron fluencies. The control cells were also moved to THOR, but not irradiated. Following irradiation, cells were cultured in a boron-free medium for seven to ten days to allow for colony formation. Colonies were fixed with methanol and acetic acid solution, followed by staining with crystal violet. Next, the reduction in survival in compound 10 or BSH treated culture after neutron irradiation was investigated. Cell morphology was observed by microscopy. Plating efficiency is defined as the number of colonies observed, as divided by the number of cells plated. Meanwhile, surviving fraction refers to the number of colonies counted, as divided by the number of colonies plated with a correction for plating efficiency.
Figure S9. Distribution and morphological changes of compound 10 or BSH treated HepG2 cells after neutron irradiation. HepG2 cells were treated with 50 μg B/mL of compound 10 or BSH containing media for 6 h. (a, d) The non-irradiated cells, (b, e) cells were irradiated with thermal neutron fluency of 2.05x10^{11} n/cm^2, and (c, f) thermal neutron fluency of 4.9x10^{11} n/cm^2. (bar, 200 μm)

References

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**Electronic Supplementary Material (ESI) for Chemical Communications**

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**Chemical Structure**

![Chemical Structure](image)

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**NMR Spectra**

![NMR Spectra](image)
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Time            14.31
INSTRUM        spect
PROBHD         5 mm DUL  13C-1
PULPROG        zg30
TD             32768
SOLVENT        CDCl3
NS             70
DS              0
SWH            6410.256 Hz
FIDRES         0.195625 Hz
AQ             2.5559540 sec
RG              4
DW            78.000 usec
DE             6.00 usec
TE             300.0 K
D1            2.00000000 sec
TD0            1

======== CHANNEL f1 ========
NUC1            1H
P1             10.00 usec
PL1            -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI             16384
SF          400.1500167 MHz
WDM         EM
SSB            0
LB             0.00 Hz
GB             0
PC             1.00
Current Data Parameters
NAME LCH-319
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date 20081226
Time 14.30
INSTRUM spect
PROBHD 5 mm DGL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 25
DS 0
SWS 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
BG 4
DW 22.000000 us
DE 6.000000 us
TE 300.0 K
D1 2.0000000000 us
d11 0.000000000000 us
DELTA 1.89999998 sec
TDO 1

====== CHANNEL f1 ======
NUC1 13C
PL1 1.00 dB
SFO1 100.628866 MHz

====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 us
PL2 -2.00 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.151601 MHz

F2 - Processing parameters
SI 32768
SF 100.6178069 MHz
WDW EM
SSB 0
LD 3.00 Hz
GB 0
PC 1.00
Current Data Parameters

NAME LCH-B1-two
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100316
Time_ 11.01
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 52768
SOLVENT CDCl3
NS 200
DS 0
SWH 41322.313 Hz
FIBRES 1.261057 Hz
AQ 0.3965428 sec
RG 4096
DW 12.100 usec
dE 6.50 usec
tE 296.7 K
D1 1.00000000 sec
d11 0.30000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

 ======== CHANNEL f1 ========
NUC1 11B
P1 13.00 usec
PL1 0.00 dB
SF01 192.2150880 MHz

 ======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCP02 92.00 usec
PL2 120.00 dB
PL1 11.30 dB
PL13 14.00 dB
SF02 599.1029955 MHz

F2 - Processing parameters
SI 32768
SF 192.2153251 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
Current Data Parameters
NAME            LCH-320
EXPO            1
PROCNO          1

F2 - Acquisition Parameters
Date_          20090105
Time             14.08
INSTRUM         spect
PROBHD         5 mm DUL 13C-1
PULPROG        zg30
TD              32768
SOLVENT        CDCl3
NS              40
DS              0
SWH            6410.256 Hz
FIDRES         0.195625 Hz
AQ            2.5559540 sec
RG              4
DW             78.000 usec
DE              6.00 usec
TE             300.0 K
D1            2.00000000 sec
TD0            1

======== CHANNEL f1 ========
NUC1            1H
P1             10.00 usec
P21          -2.40 d
FIDQ        400.1528010 MHz

F2 - Processing parameters
SI            16384
SF         400.1500167 MHz
WDW         EM
SSB            0
LB            0.00 Hz
GB            0
PC             1.00

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Current Data Parameters
NAME            LCH-320
EXPNO            2
PROCNO            1

F2 - Acquisition Parameters
Date_            20090105
Time              14.12
INSTRUM           spect
PROBHD           5 mm DUL 13C-1
FUPROG            zpg30
TD            65326
SOLVENT          CDCl3
NS              1325
DS              0
SWH        22727.273 Hz
FDRES       0.346791 Hz
AQ           1.4018420 sec
NG              4
DW          22,000 usec
DE            6.00 usec
TE        300.0 K
D1        2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999998 sec
TD0           1

======== CHANNEL f1 ========
NUC1                13C
CPDPRG1         waltz16
PL1                10.30 usec
PL12              -2.40 dB
PL13              15.70 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
NUC2               1H
PCPD2            90.00 usec
PL2               -2.00 dB
PL12            16.70 dB
SFO2       400.1516010 MHz

F2 - Processing parameters
SI              32768
SF        100.6178038 MHz
WDW              EMSSB
LB                 0
GB                 0
PC              1.00

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Current Data Parameters
NAME  LCH-B2
PROCNO  1
PROCNO  1

F2 - Acquisition Parameters
Date_  20100121
Time_  10.04
INSTRUM  spect
PROBHD  5 mm QNP 1H/1
PULPROP  zgpg
TD  52768
SOLVENT  CDCl3
NS  500
DS  0
SIN  41322.313 Hz
FIBRES  1.261057 Hz
AQ  0.3965428 sec
RG  4096
DW  12.100 usec
DE  6.50 usec
TE  298.6 K
D1  1.00000000 sec
d1l  0.03000000 sec
DELTA  0.89999998 sec
MCWST  0.00000000 sec
MCWRK  0.01500000 sec

====== CHANNEL f1 ======
NUC1  11B
P1  13.00 usec
PL1  0.00 dB
SF1  192.2150880 MHz

====== CHANNEL f2 ======
CPDPDG2  waltz16
NUC2  1H
PCPD2  92.00 usec
PL2  120.00 dB
PL12  11.30 dB
PL13  14.00 dB
SF2  599.1029955 MHz

F2 - Processing parameters
S1  32768
SF  192.2154033 MHz
WDN  EM
SSB  0
LB  3.00 Hz
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Current Data Parameters
NAME LCH-B3
EXNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100121
Time_ 10.24
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULFROG zgpg
TD 32768
SOLVENT MeOD
NE 200
DS 0
SWH 41322.313 Hz
FIBRES 1.261057 Hz
AQ 0.3965428 sec
RG 4096
DW 12.100 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 11B
P1 13.00 usec
PL1 0.00 dB
SFO1 192.2150880 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 10.00 dB
PL13 14.00 dB
SFO2 599.1029955 MHz

F2 - Processing parameters
SI 32768
SF 192.2154033 MHz
WDN EN
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
Current Data Parameters

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F2 - Acquisition Parameters

| Date_    | 20100719            |
| Time     | 15:03               |
| INSTRUM  | spect               |
| PROBHD   | 5 mm DUL 13C-1      |
| PULPROG  | zgpg30TD            |
| SOLVENT  | CDCl3               |
| NS       | 118                 |
| DS       | 0                   |
| SWH      | 22727.273 Hz        |
| AQ       | 1.4418420 sec       |
| RG       | 2050                |
| DW       | 22.000 usec         |
| TD       | 2.00000000 sec      |
| DELTA    | 1.89999998 sec      |

F2 - Processing parameters

| SI       | 32768               |
| SF       | 100.6178001 MHz     |
| WDW      | EM                  |
| SSB      | 0                   |
| LB       | 3.00 Hz             |
| GB       | 0                   |
| PC       | 1.00                |

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Current Data Parameters
NAME          LCH-495-2
EXPNO            2
PROCNO              1

F2 - Acquisition Parameters
Date_          20100622
Time              16.07
INSTRUM           spect
PROBHD   5 mm DUL 13C-1PUL
PROG          zgpg30
TD             65.516
SOLVENT           CDCl3
NS                  500
SS                   6
DM                   22727.27 Hz
FIDRES         0.346791 Hz
AQ                  1.441849 sec
AQ                  90.5
IM                22,000 usec
DE                  6.00 usec
TE              300.0 K
SI                  11.2000000 sec
DI               0.0300000 sec
DELTA          1.8999999 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
PL1                1.00 dB
SFO1        100.6178 MHz

======== CHANNEL f2 ========
CP1PRG2  waltz16
PL2                 90.00 usec
DI 40.00 dB
PL12              10.70 dB
P113               10.70 dB
SFO2        400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF          100.6178028 MHz
WDW                  EM
SSB                   0
PC                  1.00

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Current Data Parameters
NAME: JCB-B6  
PROCNO: 1

F2 - Acquisition Parameters
Date: 20101021  
Time: 11.35

INSTRUM: spectPROBHD  5 mm QNP  1H/1PUL
PROGP: zgpg

SOLVENT: CDCl3 NS  500
DS: 0

SWH: 41322.313 Hz
FIDRES: 1.261057 Hz
AQ: 0.3965428 sec
RG: 1024

TE: 298.4 K
DE: 6.50 usec

D1: 1.000000 sec
D11: 0.030000 sec
DELTA: 0.899999 sec

MCREST: 0.000000 sec
MCWRK: 0.015000 sec

======== CHANNEL f1 ========
NUC1: 1H
PL1: 0.00 dB
SFO1: 192.2150880 MHz

======== CHANNEL f2 ========
CPDPRG2: waltz16
NUC2: 1H
PL12: 92.00 usec
SFO2: 595.1029555 MHz

F2 - Processing parameters
SI: 32768
SF: 192.2154033 MHz

LB: 1.00 Hz
PC: 1.00

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Current Data Parameters
NAME     LCH-552-1000926
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20110926
Time              14.56
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD               32768
SOLVENT            MeOD
NS          40
DS                0
SWH            6410.256 Hz
FIDRES         0.195625 Hz
AQ                    2.5559540 sec
RG                    4
DW            78.000 usec
DE                6.00 usec
TE            300.0 K
DI                2.00000000 sec
TD0                    1

-------- CHANNEL f1 --------
NUC1                 1
PH1                  10.00 usec
PL1                 -2.40 dB
SF01               400.1528010 MHz

F2 - Processing parameters
SI                16384
SF                  400.150060 MHz
WW                EM
SSB                0
LB                0.00 Hz
GB                0
PC             1.00
Current Data Parameters
NAME     LCH-552-1-1-2 (600)EXPNO                 2PROCNO                1
F2 - Acquisition Parameters
Date_          20101203Time               7.20INSTRUM           spectPROBHD   5 mm QNPI/1PULPROG            zgpgTD                32768SOLVENT           CDCl3NS                13350DS                 0SWR                  45045.047 HzFIDRES           1.374666 HzAQ            0.3637748 secRG                 2048DW               11.100 usecD1           3.50000000 secD11         0.03000000 secDELTA        3.40000010 secMCREST   0 secMCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13CPL1      4.80 usecPL11          0 dBSFO1      150.6352381 MHz

 allergies

======== CHANNEL f2 ========
CPDPRG2         waltz16NUC2                 1HPCPD2             92.00 usecPL2              120.00 dBPL12               9.00 dBPL13               14.00 dB

F2 - Processing parameters
SI                32768SP           150.6184622 MHzWDW                  EMSSB      0LB                 3.00 HzGB       0PC                 1.00
Current Data Parameters
NAME             LCH-B7
EXENO                1
PROCNO              1

F2 - Acquisition Parameters
Date_          20100121
Time               9:27
INSTRUM           spect
PDBHD     5 mm QNP 1H/1
PULPROG            zgpgTD
TD             32768
SOLVENT            MeOD
NS                  500
DS                0
SWH           4,332,313 Hz
FIDRES        1,261,057 Hz
AQ              0.3965428 secRG
DG             4096
DW          12,100 usec
DE              6.50 usec
DS            296.4 K
D1             1.00000000 sec
D11            0.03000000 sec
DELTA          0.09999998 sec
MCREST        0.00000000 sec
MCWRK        0.01000000 sec

-------- CHANNEL f1 --------
NUC1                11BP1                13.00 usec
PL1              0.00 dB
SFO1        192.2150880 MHz

-------- CHANNEL f2 --------
CPDPRG2         waltz16
NUC2                 1H
PCD2               92.00 usec
PL2              120.00 dB
PL22             11.30 dB
PL23              14.00 dB
SFO2        599.1029955 MHz

F2 - Processing parameters
SI              32768
SF        192,215,003 MHz
WDW           EM
SMB              0
LB             1.00 Hz
GB              0
PC             1.00