Supporting Information for: Simple Synthesis, Halogenation and Rearrangement of *closo*-1,6-$C_2B_8H_{10}$

Mario Bakardjiev,[a] Bohumil Štíbr,[a] Josef Holub,[a] Zdeňka Padělková[b] and Aleš Růžička[b]
[a]Institute of Inorganic Chemistry, Academy of Sciences of the Czech Republic, 250 68 Řež, Czech Republic
[b]Department of General and Inorganic Chemistry, Faculty of Chemical Technology, University of Pardubice, Studentská 573, 532 10 Pardubice, Czech Republic

X-ray crystallography. The X-ray data for colorless crystals of **8X-2** (grown by slow sublimation) were obtained at 150K using Oxford Cryostream low-temperature device and a Nonius KappaCCD diffractometer with MoKα radiation (λ = 0.71073 Å), a graphite monochromator, and the ϕ and χ scan mode. Data reductions were performed with DENZO-SMN.[1] The absorption was corrected by integration methods.[2] Structures were solved by direct methods (Sir92)[3] and refined by full matrix least-squares based on F^2 (SHELXL97).[4] Hydrogen atoms could be mostly localized on a difference Fourier map. However, to ensure uniformity of treatment of crystal structures, they were recalculated into idealized positions (riding model) and assigned temperature factors $U_{iso}(H) = 1.2\ U_{eq}$(pivot atom) with 1.1 Å distance for B-H and C-H bonds in the carborane cage. Crystallographic data for **8-X-*closo*-1,6-$C_2B_8H_9$ (8X-2)**: $C_2H_9B_8Br$, M = 199.48, orthorhombic, *Pbca*, *a* = 12.0870(5), *b* = 11.4220(9), *c* = 12.2411(8) Å, β = 90 °, Z = 8, V = 1689.98(19) Å^3, D_c = 1.568 g.cm^{-3}, μ = 4.772 mm^{-1}, T_{min}/T_{max} = 0.241/0.377; $-15 \le h \le 14$, $-12 \le k \le 14$, $-14 \le l \le 15$; 11981 reflections measured (θ_{max} = 27.5 °), 11850 independent (R_{int} = 0.0935), 1458 with $I > 2\sigma(I)$, 100 parameters, *S* = 1.137, *R1*(obs. data) = 0.0366, *wR2*(all data) = 0.0880; max., min. residual electron density = 0.677, -0.862 eÅ^{-3}. Crystallographic data for structural analyses have been deposited with the Cambridge Crystallographic Data Centre, CCDC deposition no. 1033002. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EY, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http:// www.ccdc.cam.ac.uk.

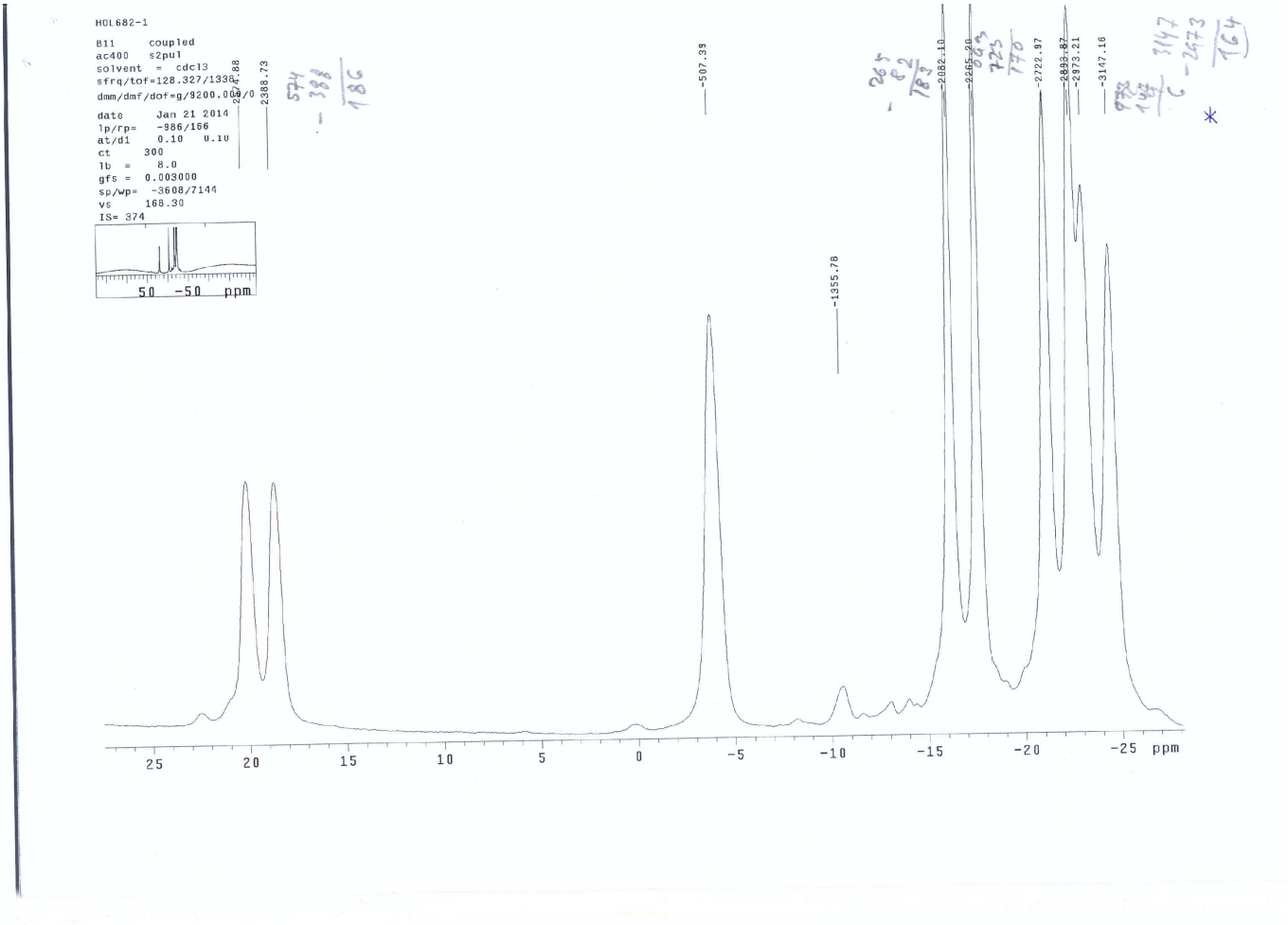
References

(1) Otwinowski, Z.; Minor, W. *Meth. Enzym*. **1997**, *276*, 307-326.
(2) Coppens, P. In *Crystallographic Computing,* Ahmed, F. R.; Hall, S. R.; Huber, C. P., Eds.; Munksgaard, Copenhagen, **1970**, pp. 255 – 270.
(3) Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A. *J. Appl. Crystallogr.* **1993**, *26*, 343-350.
(4) Sheldrick, G. M. SHELXL-97, Program for the Refinement of Crystal Structures, University of Göttingen, Göttingen (Germany), **1997**.

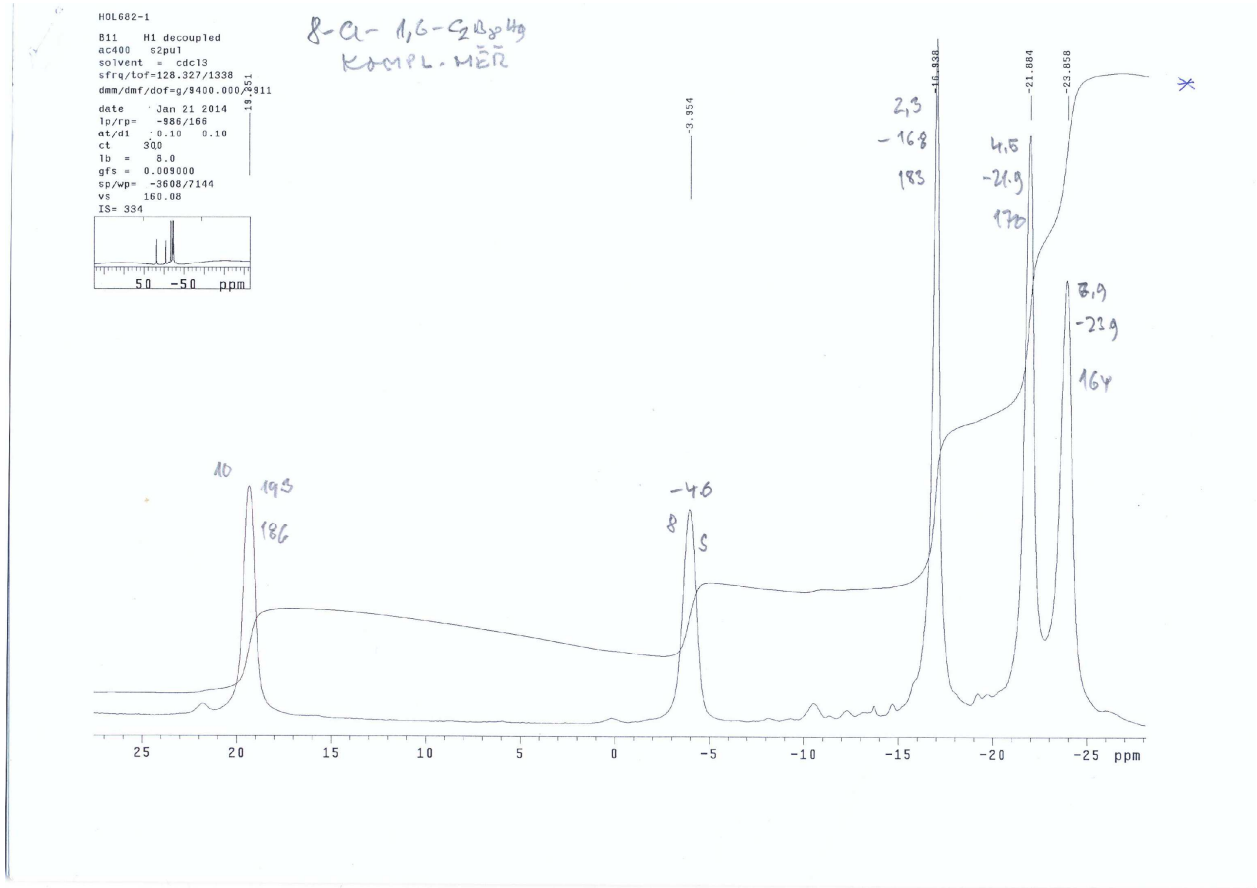
Selection of authentic NMR measurements (exemplified by those on 8-Cl - 1,6-$C_2B_8H_9$ and 2-Cl-1,10-$C_2B_8H_9$- derivatives)

(the spectra for the corresponding Br and I derivatives are similar; 200% enlargement is recommended for better reading of the ppm values)

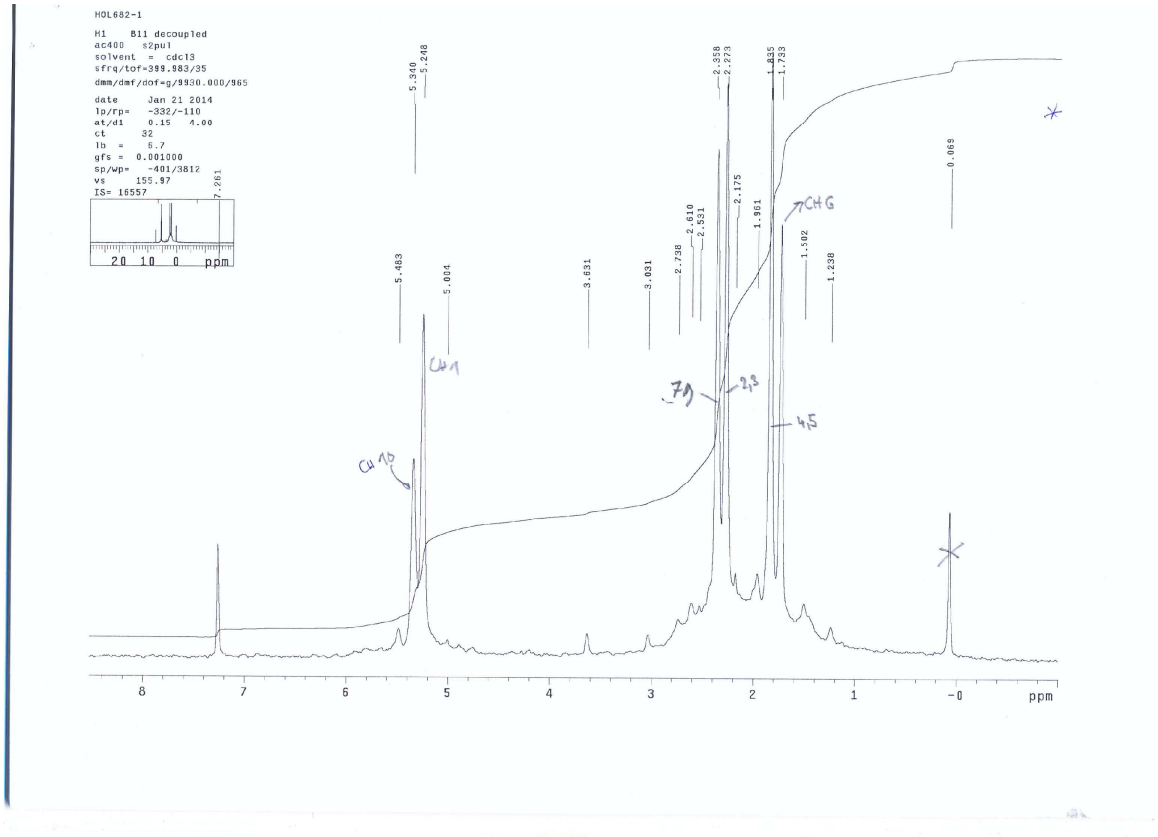
8-Cl-1,6-$C_2B_8H_9$, ^{11}B-NMR



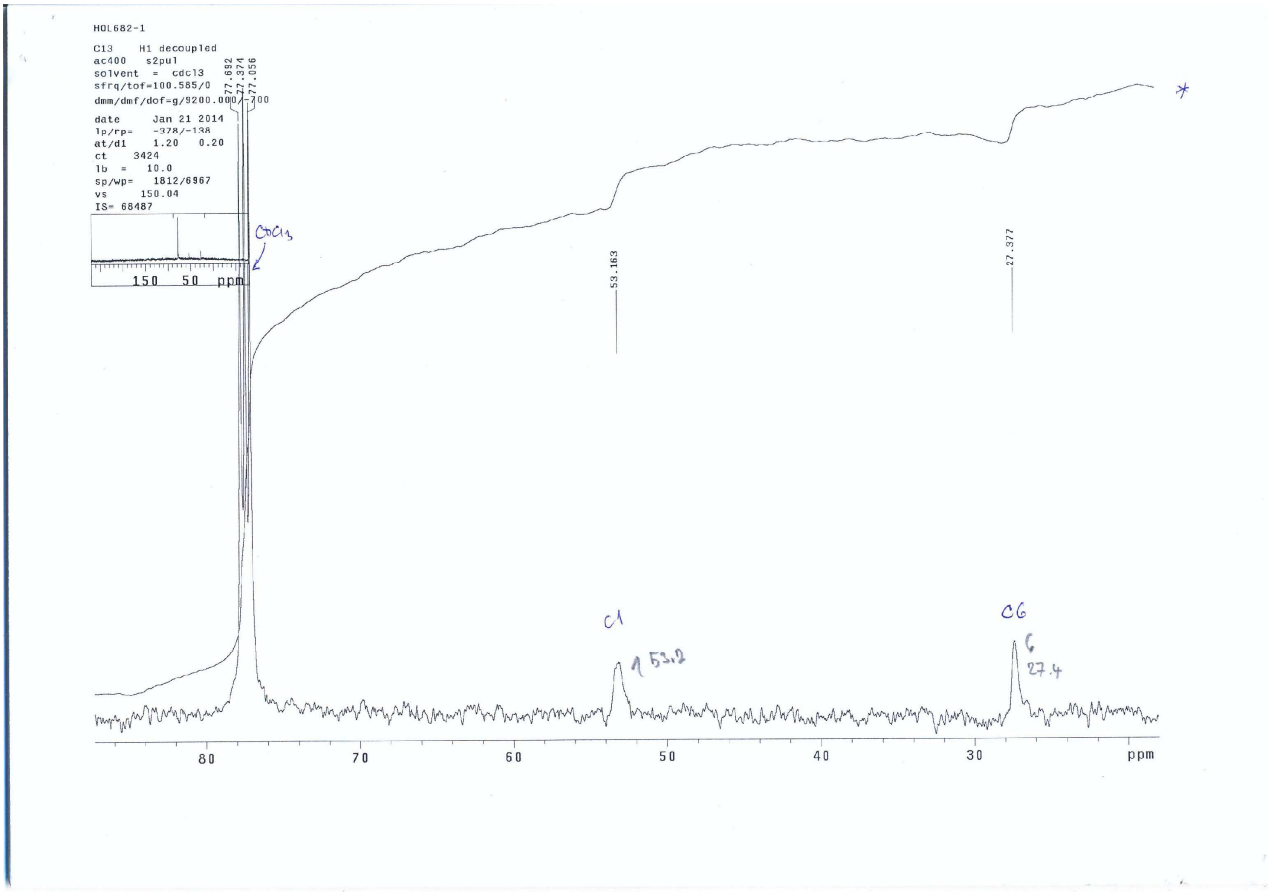
8-Cl-1,6-$C_2B_8H_9$, $^{11}B\{^1H\}$-NMR



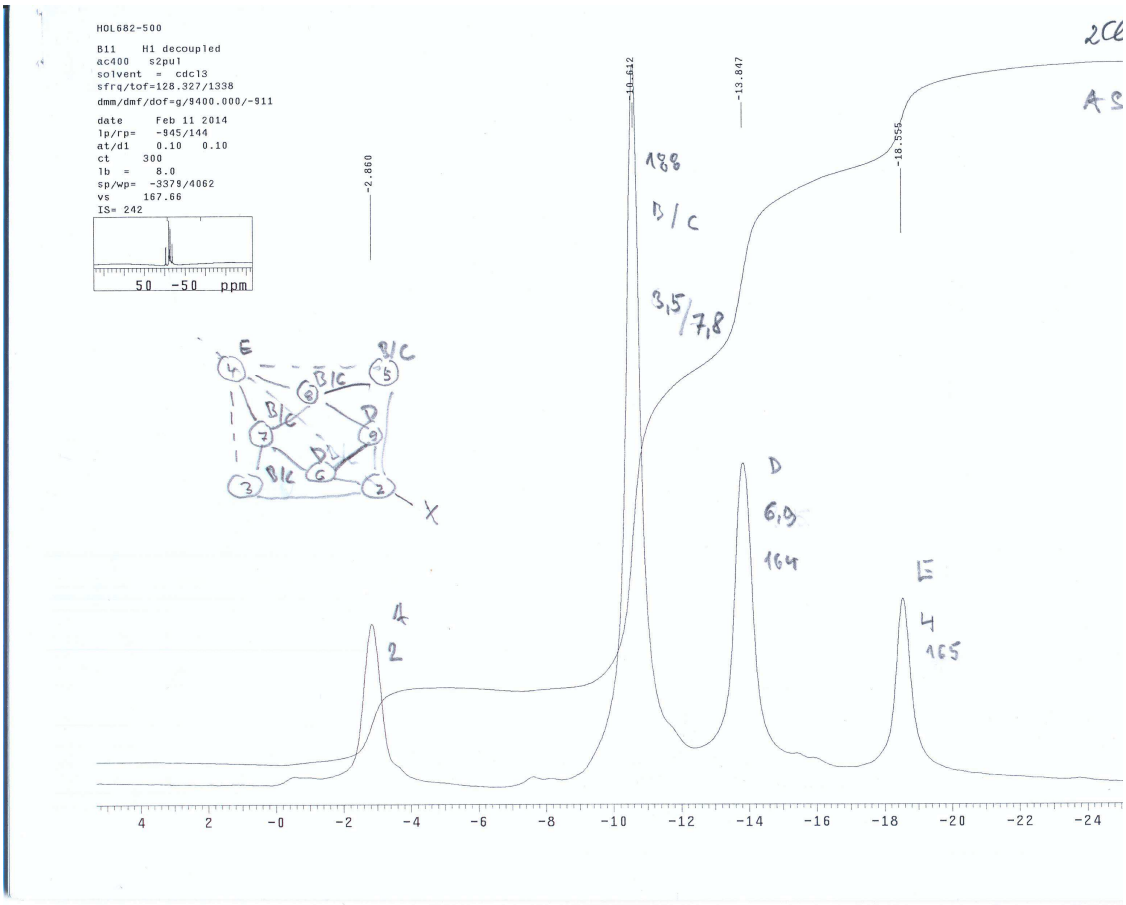
8-Cl-1,6-$C_2B_8H_9$, ^1H-{^{11}B}-NMR



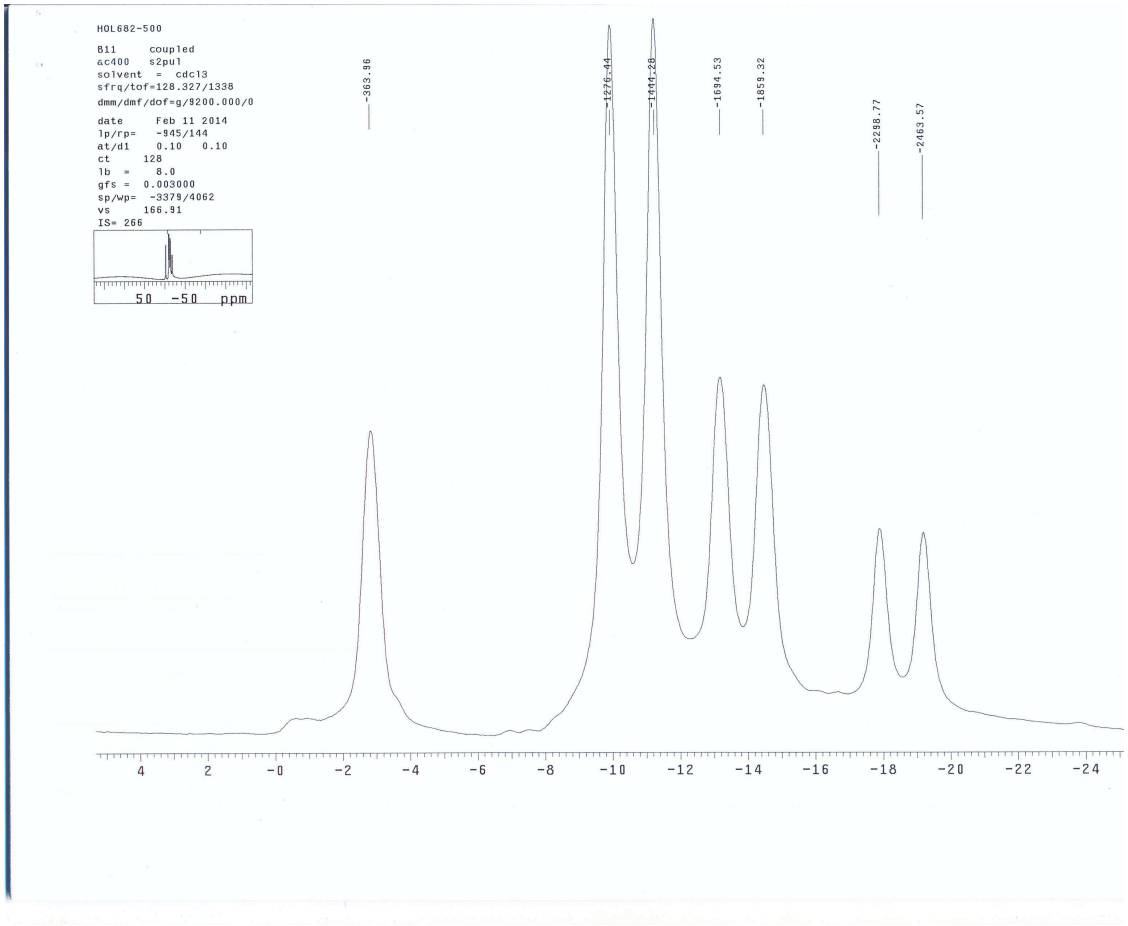
8-Cl-1,6-$C_2B_8H_9$, ^{13}C-{^1H}-NMR



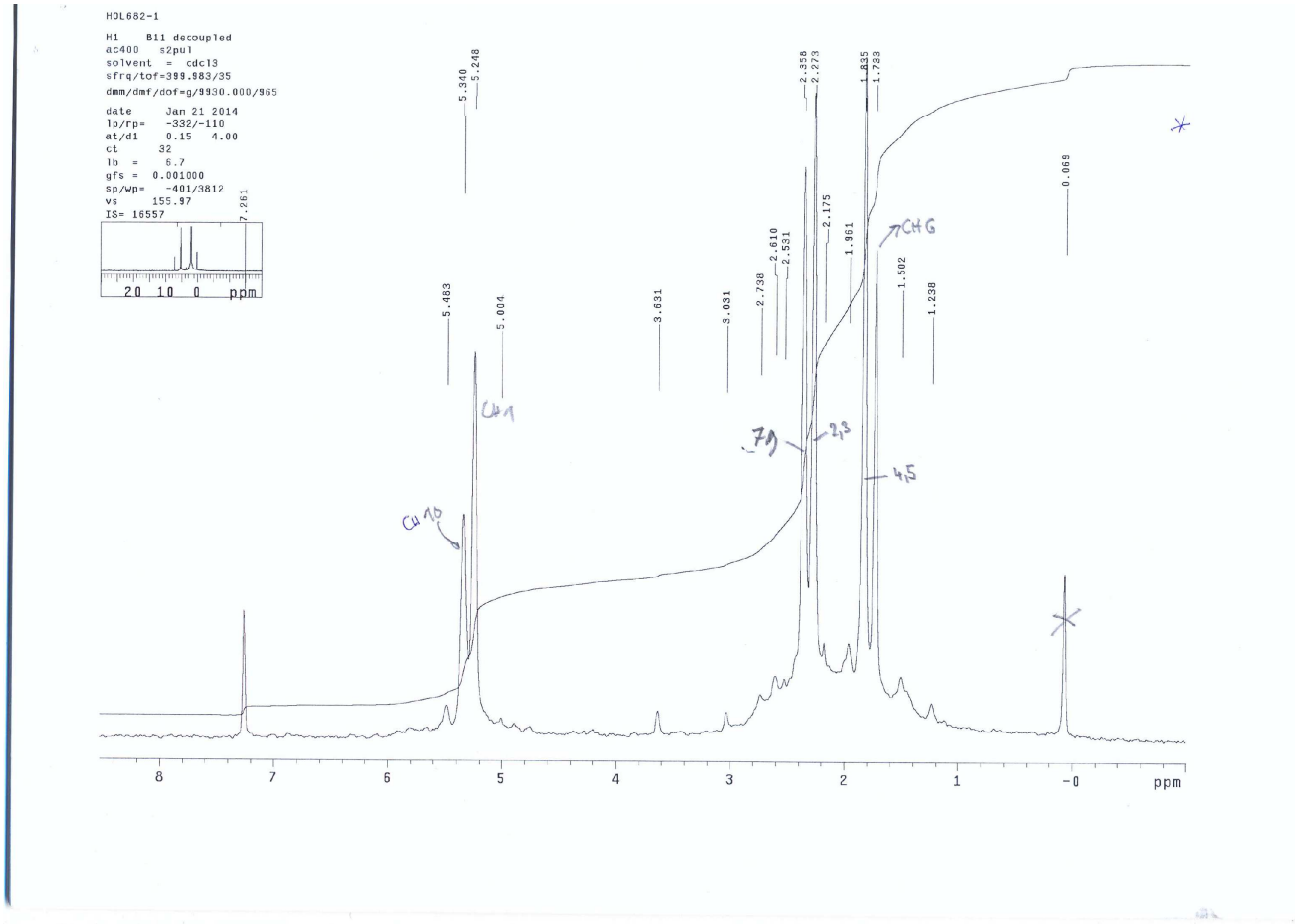
2-Cl-1,10-$C_2B_8H_9$, $^{11}B\{^1H\}$-NMR



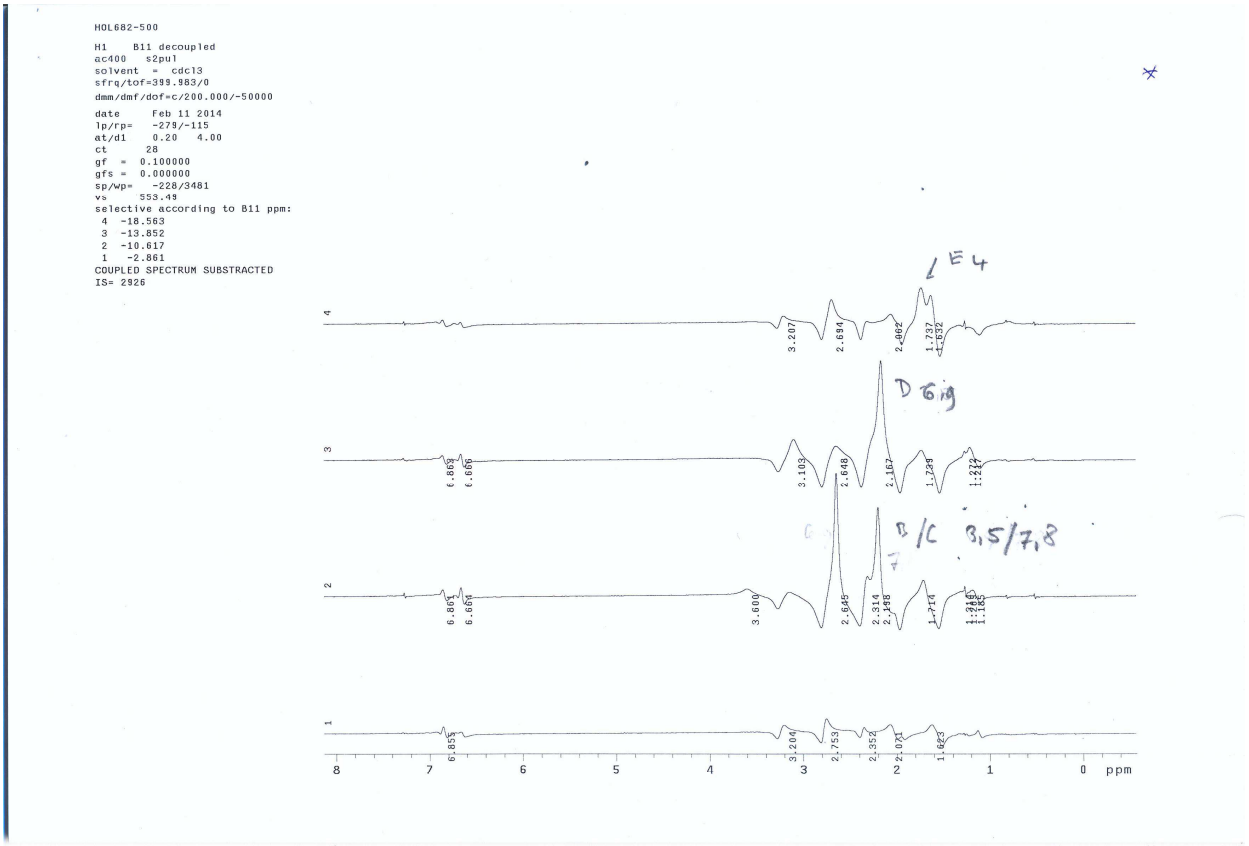
2-Cl-1,10-$C_2B_8H_9$, ^{11}B-NMR



2-Cl-1,10-$C_2B_8H_9$, ^1H-{^{11}B}-NMR



2-Cl-1,10-$C_2B_8H_9$, ^1H-{^{11}B(selective)}-NMR



2-Cl-1,10-$C_2B_8H_9$, ^{13}C-{^1H}-NMR