



# Synthesis and Analysis of Isotopically Pure *closo*-1, 2-Dicarbadoodecaborane

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Kaileen Blanchard  
Aashani Tillekaratne  
Advisor: Prof. M. Trenary  
University of Illinois-Chicago  
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# Overview

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- Research boron compounds for hydrogen storage uses
  - High H wt %
  - May keep boron cage structure when dehydrogenated
- Dehydrogenation and re-hydrogenation of boron clusters
  - Synthesis of sodium borohydride
  - Calculated spectrum of decaborane
  - Experimental IR spectrum for decaborane



# Synthesis of Sodium Borohydride

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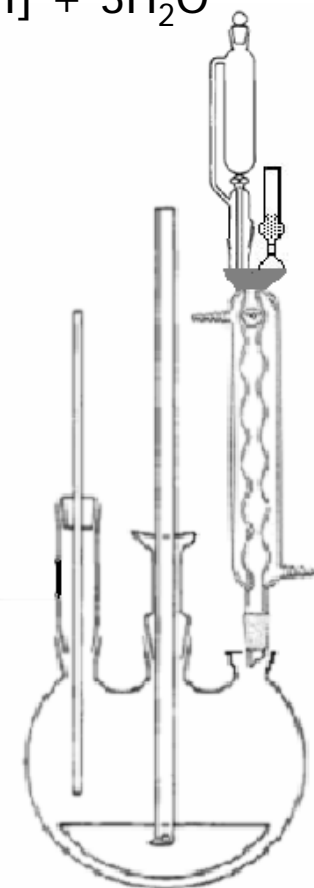
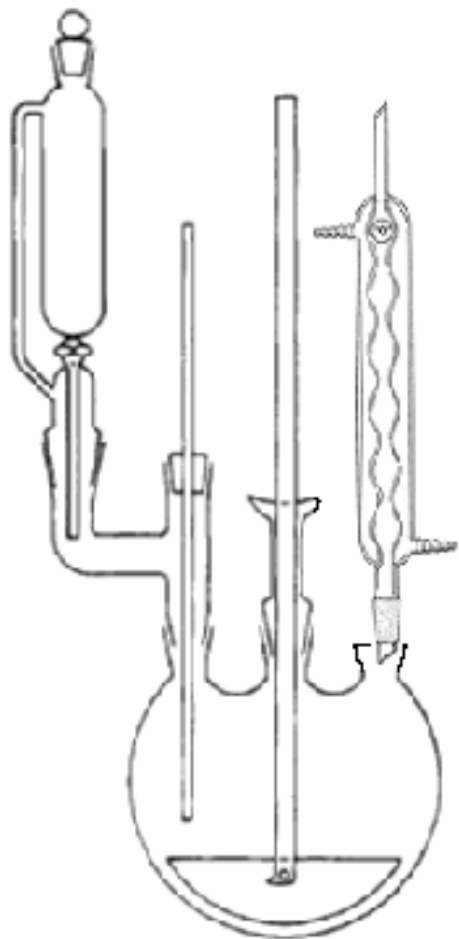
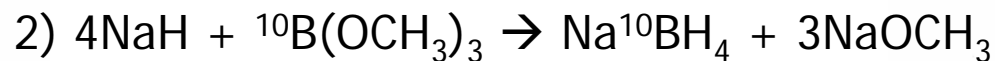
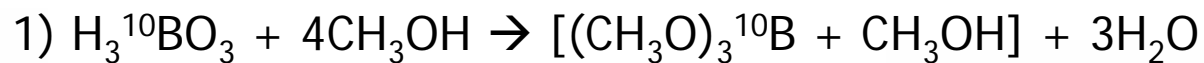
- Successes

- Completed synthesis of sodium borohydride
- Calculated IR spectrum for decaborane molecule
- Obtained experimental IR and mass spectra for decaborane molecule

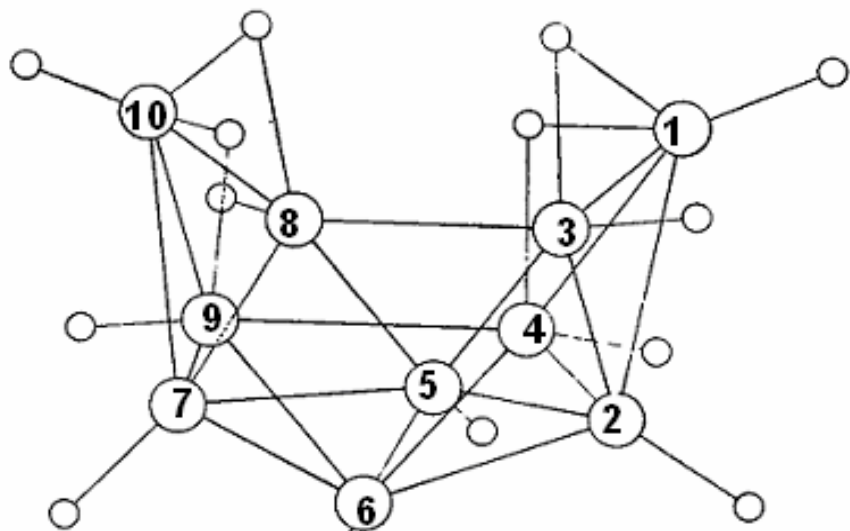
- Challenges

- Time limit
- Modify procedure to adapt for high pressure system
  - Should ideally be closed system
  - Methyl borate reacts with water to form toxic and flammable gases.
  - Nitrogen balloon

# Synthesis Path



# Calculated IR Spectrum of Decaborane



- Successful analysis of Decaborane in many different isotopical combinations
  - Naturally abundant boron, 2 B-10, and 4 B-11 in different positions
  - Spectra with four B-10 atoms split results of two parent spectra (1&10 spectrum and 4&9 spectrum → 1, 10, 4, & 9 spectrum)
  - All B-10 spectrum higher frequencies than all B-11 spectrum

# Frequencies and Intensities of Calculated IR Spectrum for Decaborane

	Frequency	Intensity		Frequency	Intensity
51	1620.082	166.5482	51	1617.199	163.6955
52	1688.922	26.7224	52	1686.873	26.506
53	1963.22	0.0006	53	1957.345	0.0006
54	1980.619	0.0475	54	1975.066	0.0219
55	2010.55	23.7791	55	2003.864	23.0512
56	2028.702	28.5894	56	2022.572	27.9628
57	2668.894	21.9604	57	2657.517	22.7129
58	2672.837	30.7701	58	2661.189	31.1307
59	2688.775	30.29	59	2676.882	32.983
60	2690.45	75.2196	60	2678.568	75.4788
61	2695.703	28.1957	61	2683.988	29.3509
62	2696.948	0.0005	62	2685.242	0.0005
63	2701.295	38.4201	63	2689.459	34.8367
64	2702.036	212.46	64	2690.237	207.8269
65	2710.149	159.5785	65	2697.928	152.3123
66	2714.375	6.6564	66	2701.96	7.9993

All B-10

All B-11



# Frequencies and Intensities of Calculated IR Spectrum for Decaborane

## All B-11

	Frequency	Intensity
1	1617.199	163.6955
2	2690.237	207.8269
3	2697.928	152.3123

## All B-10

	Frequency	Intensity
1	1620.082	166.5482
2	2702.036	212.46
3	2710.149	159.5785

## B-10 positions 1 & 10

	Frequency	Intensity
1	1618.438	165.9285
2	2690.243	207.682
3	2708.896	125.6003

## B-10 positions 1, 10, 4, & 9

	Frequency	Intensity
1	1619.132	166.0974
2	2691.306	119.3179
3	2701.055	101.4835
4	2709.367	135.1202

## B-10 positions 4 & 9

	Frequency	Intensity
1	1617.893	163.8553
2	2696.562	119.3658
3	2700.363	141.7553



# Infrared Spectrum for Decaborane

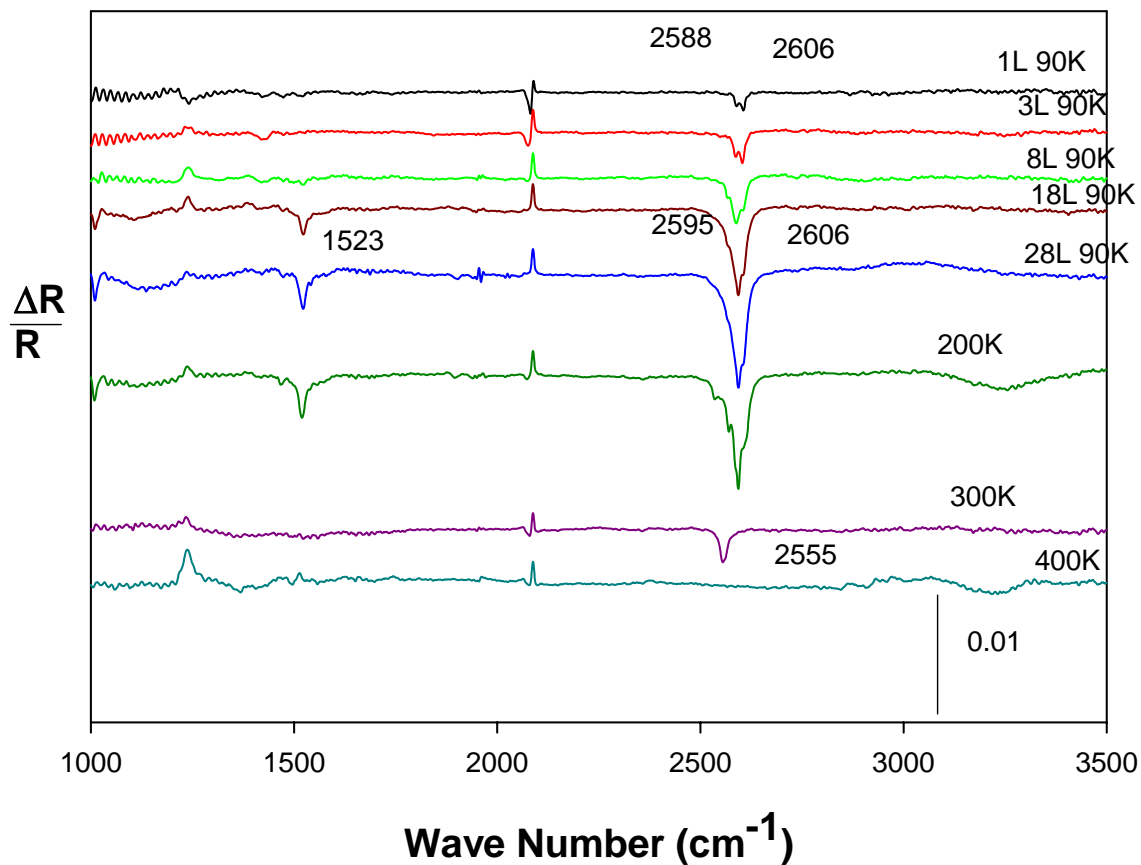
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- Analyzed naturally abundant decaborane using Temperature Programmed Desorption (TPD) and Reflective Absorption Infrared Spectroscopy (RAIRS)
- Obtained mass and infrared spectrum.

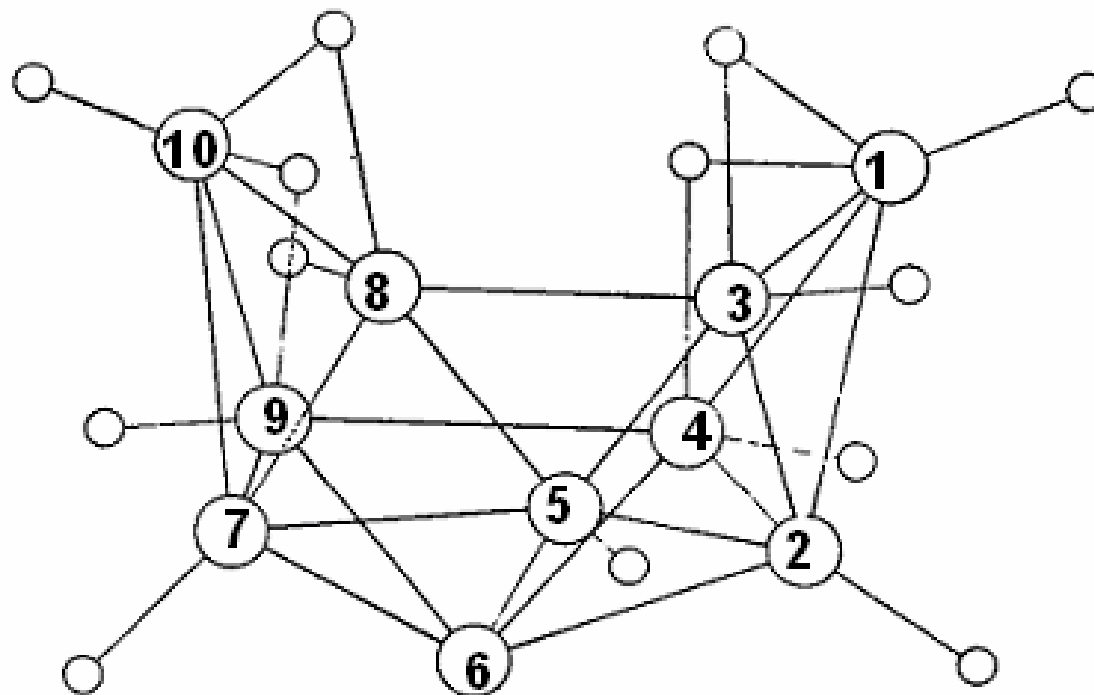


# Naturally Abundant Decaborane Spectrum

Decaborane

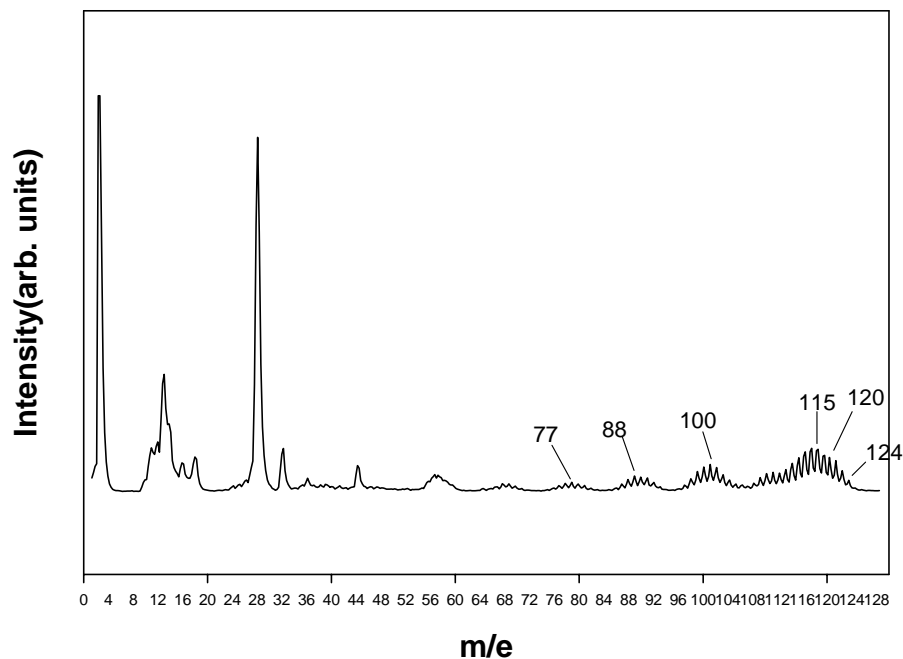


# Chemical Structure of Decaborane



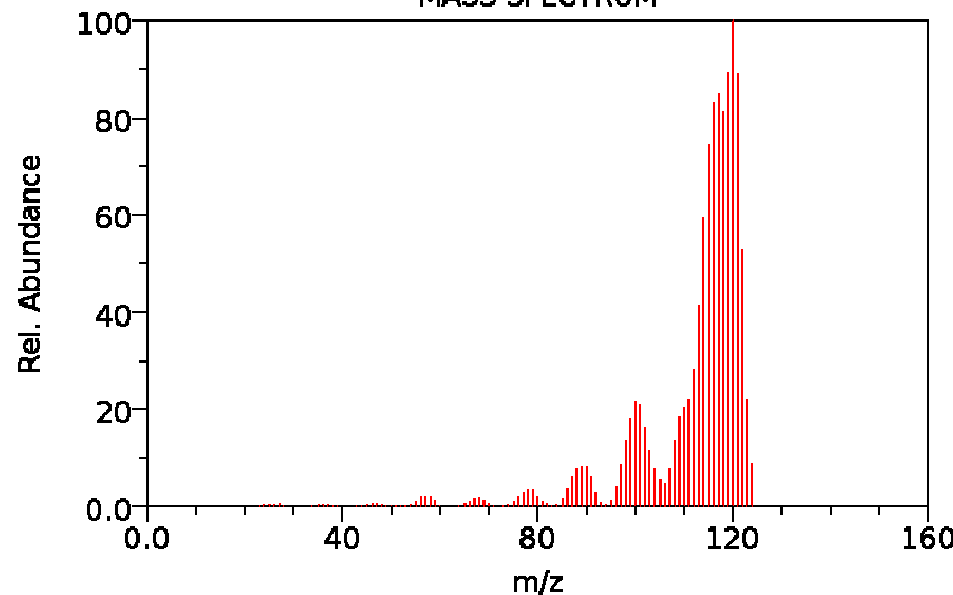
# Mass Spectrum for Decaborane

$B_{10}H_{14}$ -Mass Spectrum



Experimental Spectrum

Decaborane(14)  
MASS SPECTRUM



NIST Chemistry WebBook (<http://webbook.nist.gov/chemistry>)

Reference Spectrum



# References/Acknowledgments

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- 8) NSF CTS-0533499 & 0434201 GOALI: Atomic-scale Investigation of High Dielectric Constant Thin Films Using In Situ and Other Techniques, (Director C.G. Takoudis)