Principal Investigator:	Debra A. Feakes, Associate Professor
Title:	Conference: Support of the Ninth Boron in the Americas Workshop
Grant Number:	DE-FG02-04ER63818
Project ID:	0010008
Award Register:	ER63818
Program Manager:	Prem C. Srivastava, Ph. D., Division: SC-73
Grant Amount:	$8,750

FINAL REPORT

The Ninth Boron in the Americas Workshop was held at Texas State University – San Marcos, formerly known as Southwest Texas State University, between May 19th and May 22nd, 2004. The conference was attended by 73 researchers (see attached list of registrants), representing both academic and industrial institutions throughout the United States of America and also institutions in Canada, Mexico, England, India, Japan, and Singapore. A total of 33 oral presentations and 31 poster presentations were given.

The bulk of the support for the conference was provided through registration fees and industrial support (see attached list of industrial and other donors); however, the contributions were not sufficient to provide student travel stipends and student awards. As a result, the singular goal of the proposal which was submitted to, and funded by, the United States Department of Energy was to request funds for student travel stipends and presentation awards for the students. As a result, a significant increase in the number of student participants at the conference, compared to prior years, was realized. A total of 34 undergraduate, graduate, and post-doctoral researchers attended the conference.

Each student participant received a travel stipend, ranging in value from $90 to $400 depending on their distance and expense of travel. The lower end stipends covered the cost of the student registration while the higher end stipends covered the cost of the student registration as well as a portion of the additional travel expenses associated with transportation and hotel accommodations. A list of the students which received stipends, their home institution, and the amount of the stipend received is attached. The travel stipends were dispersed directly to the students, in the form of a check, at the registration desk at the beginning of the meeting. The total amount of the travel stipends was $8,450.

A committee of three outstanding scientists, composed of Dr. Donald S. Matteson (Washington State University), Dr. R. Bruce King (University of Georgia), and Dr. Lawrence Barton (University of Missouri – St. Louis), were selected to evaluate the presentations. Awards were given in the following categories: best undergraduate presentation, best graduate oral presentation, best graduate poster presentation, best post-doctoral researcher oral presentation, and best post-doctoral researcher poster presentation.

The committee reviewed the presentations, both oral and poster, and met prior to the final banquet. At the closing ceremonies, five awards, in the amount of $100.00, were announced. Each student award was processed through the university administrative procedure and sent as a check to the individual students at their permanent address. The total amount for the awards was $500. A portion of the travel awards, $200, was allotted from the conference funds in order to cover the total expense ($8,950 as opposed to the award of $8,750). As a result, there is no remaining balance in the grant account.

The abstract book for the conference has been sent under separate cover to Dr. Srivastava.

LIST OF REGISTRANTS

1. Prof. Lawrence Barton, University of Missouri-St. Louis
2. Dr. Narayan Bhat, The University of Texas-Pan American
3. Prof. Mark Bradley, Widener University
4. Prof. Vladimir Bregadze, A.N.Nesmeyanov Institute of Organoelement Compounds
5. Mr. Robert Butterick III, University of Pennsyvania
6. Mr. Jude Clapper, University of Pennsylvania
7. Prof. Rosalinda Contreras, Centro de Investigaciones y de Estudios Avanzados
8. Mr. Gang Dong, University of Tennessee
9. Mr. Jemmis Eluvathingal, University of Hyderabad
10. Dr. Ludvig Eriksson, University of Colorado, Boulder
11. Miss Adriana Esparza Ruiz, Centro de Investigaciones y de Estudios Avanzados
12. Dr. Debra A. Feakes, Texas State University - San Marcos
13. Prof. Thomas P. Fehlner, University of Notre Dame
14. Mr. Matthew Fete, University of Colorado
15. Mr. Matthew Fischer, Saint Louis University
16. Prof. Angelina Flores-Parra, Centro de Investigacion y de Estudios Avanzados
17. Dr. Sundargopal Ghosh, University of Notre Dame
18. Dr. Ewan Hamilton, The Ohio State University at Lima
19. Dr. Bruce Hodson, Baylor University
20. Prof. Narayan Hosmane, Northern Illinois University
21. Dr. Paul A. Jelliss, Saint Louis University
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24. Prof. George Kabalka, University of Tennessee
25. Prof. Steve Kahl, University of California, San Francisco
26. Prof. R. Bruce King, University of Georgia
27. Ms. Arienne King, McMaster University
28. Dr. Goji Kodama, University of Utah
29. Mr. Upal Kusari, University of Pennsylvania
30. Dr. Magdalena Kvicalova, University of Colorado at Boulder
31. Prof. Clint Lane, Northern Arizona University
32. Mr. Mark Lee, University of California – Los Angeles
33. Dr. Peng Lei, Baylor University
34. Ms. Yuqi Li, University of Pennsylvania
35. Dr. Xiulian Lu, Baylor University
36. Dr. Ling Ma, University of California – Los Angeles
37. Dr. Ramon Macias-Maza, University of Notre Dame
38. Prof. John Maguire, Southern Methodist University
39. Mrs. Maria Liliana Marin, CINVESTAV
40. Dr. Karl Matos, BASF Corporation
41. Prof. Donald Matteson, Washington State University
42. Dr. Tom McGrath, Baylor University
43. Mr. W. Jeff McVey, Texas State University - San Marcos

44. Mrs. Michelle Motley, Texas State University - San Marcos
45. Dr. Suresh Murugesan, Texas State University – San Marcos
46. Mr. Abhijit Naravane, University of Tennessee
47. Mr. Brian Newell, Texas State University - San Marcos
48. Mr. Justin Orlando, Saint Louis Department of Chemistry
49. Prof. Robert Paine, Department of Chemistry
50. Mr. Matt Parrot, McMaster University
51. Prof. Robert Parry, University of Utah
52. Prof. P.V. Ramachandran, Purdue University
53. Dr. Angel Ramos-Organillo, Cinvestav-IPN and University of Colima Mexico
54. Mr. Peter Schreiber, Department of Chemistry & Biochemistry
55. Dr. David Schubert, U.S. Borax Inc.
56. Prof. Don Seo, Arizona State University
57. Professor Sheldon Shore, The Ohio State University
58. Ms. Jacqueline Smits, Texas State University – San Marcos
59. Prof. John Soderquist, University of Puerto Rico
60. Mr. Bola Sogbein, McMaster University
61. Dr. Bernard Spielvogel, BoroScience Canada
62. Prof. Gordon Stone, Baylor University
63. Prof. Takagaki, Aino College Hospital
64. Dr. Ken Sukcharoenphon, Baylor University
65. Prof. Lee Todd, Gosport Scientific
66. Ms. Brandy Underwood, University of Tennessee at Knoxville, Chemistry
67. Dr. John Valliant, McMaster University
68. Dr. Reddy Venumbaka, Texas State University-San Marcos
69. Ms. Mandana Visi, U.S. Borax
70. Dr. Kamesh Vyakaranam, University of Colorado at Boulder
71. Prof. Alan Welch, Heriot-Watt University
72. Dr. Andrew Weller, University of Bath
73. Dr. Ying Huai Zhu, Institute of Chemical and Engineering Sciences Ltd.

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STUDENT STIPENDS

Name	*Home Institution*	*Amount*
Robert Butterick III	University of Pennsylvania	$300.00
Jude Clapper	University of Pennsylvania	$300.00
Gang Dong	University of Tennessee at Knoxville	$300.00
Ludvig Eriksson	University of Colorado at Boulder	$250.00
Adriana Esparza-Ruiz	Centro de Investigaciones y de Estudios Avanzados	$400.00
Matthew G. Fete	University of Colorado at Boulder	$250.00
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Magdalena Kvicalova	University of Colorado at Boulder	$250.00
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Kengkaj Sukcharoenphon	Baylor University	$150.00
Kamesh Vyakaranam	University of Colorado at Boulder	$250.00
Brandy Underwood	University of Tennessee at Knoxville	$300.00
Matthew Fischer	University of Missouri at St. Louis	$250.00
	TOTAL	**$8,450.00**

PRESENTATION AWARDS

Name	*Home Institution*	*Amount*
Matthew G. Fete	University of Colorado at Boulder	$100.00
Ling Ma	University of California at Los Angeles	$100.00
Brian Newell	Texas State University at San Marcos	$100.00
Bola O. Sogbein	McMaster University	$100.00
Kengkaj Sukcharoenphon	Baylor University	$100.00
	TOTAL	**$500.00**
	TOTAL OF BOTH STIPENDS AND AWARDS	**$8,950.00**

Program and Abstracts

Boron in the Americas IX Workshop
May 19-22, 2004
Texas State University – San Marcos
San Marcos, TX

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Acknowledgement of Financial Support

Generous financial support for the workshop and student travel stipends
has been provided by the following companies or groups.
We are extremely grateful for their contributions.

Office of Science (BER), U. S. Department of Energy, Grant No. DE-FG02-04ER63818

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Prof. *emeritus* Robert Williams	Loker Hydrocarbon Research Institute of the University of Southern California

Boron in the Americas IX Local Organizing Committee

Dr. Debra A. Feakes
Mr. Alejandro Martinez
Mr. W. Jeff McVey
Mrs. Georgia Teague
Ms. Valerie Creveling
Mr. John Powell

Past Conferences

Boron USA I	Dallas, Texas – 1988 *Host:* Prof. Narayan Hosmane
Boron USA II	Raleigh-Durham, North Carolina – 1990 *Host:* Dr. Bernard Spielvogel
Boron USA III	Pullman, Washington – 1992 *Host:* Prof. Donald Matteson
Boron USA IV	Syracuse, New York – 1994 *Host:* Prof. James Spencer
Boron USA V (BUSA-MEXV)	Guanajuato, Mexico – 1996 *Host:* Prof. Rosalinda Contreras
Boron USA VI	Athens, Georgia – 1998 *Host:* Prof. R. Bruce King
Boron USA VII	Pittsburgh, Pennsylvania – 2000 *Host:* Dr. Joseph M. Barendt
Boron in the Americas VIII	Furnace Creek, California – 2002 *Host:* Dr. David Schubert

The advisory committee voted to change the name from Boron USA to Boron in the Americas at the conference held in Pittsburgh in June, 2000.

Boron in the Americas IX Program Schedule

Wednesday, May 19, 2004

All Day	Participants are picked up either at Austin or San Antonio
1:00 – 6:00 pm	Registration/Check-In Hampton Inn Conference Room
6:30 – 8:30 pm	Mixer at Aquarena Springs (participants will be picked up at the hotels, beginning at 6:00 pm, buses will run continuously from 6:00 pm until 9:00 pm)

Thursday, May 20, 2004

7:30 am	Participants will be picked up at the hotel and transported to the conference site
8:00 – 8:30 am	Registration and Continental Breakfast Foyer outside of Centennial Hall 157
8:30 – 9:00 am	Opening Remarks
9:00 – 10:00 am	Oral Presentations (O1-O2)
10:00 – 10:30 am	Morning Break
10:30 am – 12:00 pm	Oral Presentations (O3-O5)
12:00 – 1:30 pm	Lunch The Den
1:30 – 3:00 pm	Oral Presentations (O6-O8)
3:00 – 3:30 pm	Afternoon Break
3:30 – 5:00 pm	Oral Presentations (O9-O11)
6:00 – 9:00 pm	Poster Session and Dinner LBJ Student Center Ballroom (Buses will provide transportation to the hotels starting at 7:30 pm and ending at 10:00 pm)

Friday, May 21, 2004

7:30 am	Participants will be picked up at the hotel and transported to the conference site
8:00 – 8:30 am	Continental Breakfast Foyer outside of Centennial Hall 157
8:30 – 10:00 am	Oral Presentations (O12-O14)
10:00 – 10:30 am	Morning Break
10:30 am – 12:00 pm	Oral Presentations (O15-O17)
12:00 – 1:30 pm	Lunch The Den
1:30 – 3:30 pm	Oral Presentations (O18-O21)
3:30 – 10:00 pm	Group Activity, Texas Flagship (buses will pick up participants outside of Centennial Hall)

Saturday, May 22, 2004

7:30 am	Participants will be picked up at the hotel and transported to the conference site
8:00 – 8:30 am	Continental Breakfast Foyer outside of Centennial Hall 157
8:30 – 10:00 am	Oral Presentations (O22-O24)
10:00 – 10:30 am	Morning Break
10:30 am – 12:00 pm	Oral Presentations (O25-O27)
12:00 – 1:30 pm	Lunch The Den
1:30 – 3:00 pm	Oral Presentations (O28-O30)
3:00 – 3:30 pm	Afternoon Break
3:30 – 5:00 pm	Oral Presentations (O31-O33)

6:00 – 9:00 pm	Closing Banquet J.C. Kellum Building, 11th Floor (Buses will take the participants to the hotel and back after the close of the meeting)

Sunday, May 23, 2004

All Day	Participants are taken to Austin or San Antonio for departure

Boron in the Americas IX Oral Program

Thursday Morning, May 20, 2004

8:00 – 8:30 am		*Registration and Continental Breakfast*
8:30 – 9:00 am		*Opening Remarks*

Chairpersons: Dra. Rosalinda Contreras and Dr. David Schubert

9:00 – 9:30 am	O1	**Mark W. Lee** and M. F. Hawthorne "Boron-Rich Oligomeric Phosphate Diesters With Applications in BNCT and Drug Delivery" Abstract on Page 19
9:30 – 10:00 am	O2	**Jude Clapper** and Larry G. Sneddon "Syntheses and Structural Studies of 10-Vertex Siladicarbaboranes and the First Silamonocarbaboranes" Abstract on Page 20
10:00 – 10:30 am		*Morning Break*
10:30 – 11:00 am	O3	Michael Ingleson, Nathan Patmore, and **Andrew Weller** "Catching "Greasy Balls": Transition Metal Chemistry of Highly Methylated Carborane Anions" Abstract on Page 21
11:00 – 11:30 am	O4	**Eluvathingal D. Jemmis** "Hückel, Wade's, and Jemmis Rules" Abstract on Page 22
11:30 am – 12:00 pm	O5	**A. S. King**, T. Brenstru, J. F. Britten, G. Ferguson, A. Capretta, and J. F. Valliant "The Synthesis of Sterically Hindered and Robust Carboranyl-Pnictogen Ligands for Coordination Chemistry and Catalysis" Abstract on Page 23

Thursday Afternoon, May 20, 2004

Chairpersons: Dr. Thomas Fehlner and Dr. Goji Kodama

1:30 – 2:00 pm	O6	**Ewan J. M. Hamilton**, Roman G. Kultyshev, Bin Du, Edward A. Meyers, Shengming Liu, and Sheldon G. Shore "A π-Stacking Interaction in the *n*-$B_{18}H_{22}$-Benzene System" Abstract on Page 24
2:00 – 2:30 pm	O7	**Masao Takagaki**, Narayan S. Hosmane, Takeo Tsuruta, and Yoshori Yamamoto "Preliminary Study on Gadolinium Neutron Capture Therapy" Abstract on Page 25
2:30 – 3:00 pm	O8	**Vladimir I. Bregadze**, Irina P. Beletskaya, Sergey N. Osipov, Pavel V. Petrovskii, Zoya A. Starikova, and Sergey V. Timofeev "New α-Amino Acid Derivatives with Carboranyl Fragments in α- and β-Positions" Abstract on Page 26
3:00 – 3:30 pm		*Afternoon Break*
3:30 – 4:00 pm	O9	**David M. Schubert** "Chemistry of Crystalline Hydrated Zinc Borates" Abstract on Page 27
4:00 – 4:30 pm	O10	**Upal Kusari**, Yuqi Li, Mark G. Bradley, and Larry G. Sneddon "Polyborane Reactions in Ionic Liquids. Facile Syntheses of Functionalized Decaboranes and *ortho*-Carboranes" Abstract on Page 28
4:30 – 5:00 pm	O11	**Dong-Kyun Seo**, Li-Ming Wu, Nora Iancu, and Qiangbin Wang "New Synthetic Methodologies for Bulk/Nanostructured Metal Chalcogenides: Utilization of New Boron Chalcogenide Chemistry" Abstract on Page 29

Friday Morning, May 21, 2004

8:00 – 8:30 am *Continental Breakfast*

Chairpersons: Dr. Clint Lane and Dr. John Soderquist

8:30 – 9:00 am O12 **P. Veeraraghavan Ramachandran**
"Synthesis of Amino Acids via Organoboranes"
Abstract on Page 30

9:00 – 9:30 am O13 **Zhu Yinghuai**, Narayan S. Hosmane, and John A. Maguire
"An Effective Asymmetric Alkylation System of Aldimines with Diethylzinc: Mixed Ionic Liquids of [*N*-Pentylpyridium][1-Menthoxymethyl-*closo*-$CB_{11}H_{11}$] and 1-Butyl-3-methylimidazolium Hexafluorophosphate"
Abstract on Page 31

9:30 – 10:00 am O14 **Ling Ma** and M. F. Hawthorne
"Camouflaged Carborane Amphiphiles: Synthesis and Self-Assembly"
Abstract on Page 32

10:00 – 10:30 am *Morning Break*

10:30 – 11:00 am O15 Eda Canales, Eliud Hernandez, and **John A. Soderquist**
"Asymmetric Allylboration with 10-Substituted 9-BBDs: Homoallylic Amines from *N*-TMS Aryl Aldimines and, for the First Time, Aryl and Alkyl *N*-TMS-Ketimines"
Abstract on Page 33

11:00 – 11:30 am O16 Tomoko Ozawa, Raquel A. Santos, Myoung-Seo Koo, **Stephen B. Kahl**, and Dennis F. Deen
"Convection Enhanced Delivery of a Boronated Porphyrin to Intracerebral Human Glioblastoma Xenografts in Athymic Rats"
Abstract on Page 34

11:30 am – 12:00 pm O17 **G. W. Kabalka** and A. R. Mereddy
"Facile Syntheses of Organic Halides Using Organotrifluoroborates"
Abstract on Page 35

Friday Afternoon, May 21, 2004

Chairpersons: Dr. Lawrence Barton and Dr. George W. Kabalka

1:30 – 2:00 pm	O18	Shaowu Du, Bruce Hodson, Jason Kautz, Xiu Lian Lu, **Thomas McGrath**, and F. Gordon A. Stone "Building Butterflies on a Rhenium-Monocarborane Substrate" Abstract on Page 36
2:00 – 2:30 pm	O19	**Brian Newell** and Debra A. Feakes "An Investigation of Nucleophilic Attack on the [*n*-$B_{20}H_{18}]^{2-}$ Anion" Abstract on Page 37
2:30 – 3:00 pm	O20	**J. F. Valliant**, O. O. Sogbein, P. Merdy, K. A. Stephenson, and A. Green "The Synthesis of Tc(I) and Re(I) Carborane Complexes and the Development of Organometallic Radiopharmaceuticals" Abstract on Page 38
3:00 – 3:30 pm	O21	**Kamesh Vyakaranam**, Stefanie Koebe, Hana Divisová, and Josef Michl "The Possible Role of the Carbonium Ylide $C(+)B_{11}Me_{11}(-)$ as a Reaction Intermediate" Abstract on Page 39

Saturday Morning, May 22, 2004

8:00 – 8:30 am *Continental Breakfast*

Chairpersons: Dr. Bernard Spielvogel and Dr. Andrew Weller

8:30 – 9:00 am O22 **Ludvig Eriksson** and Josef Michl
"The Synthesis of Permethylated 1-Vinyl and 1-Ethynyl CB_{11}^- Anions"
Abstract on Page 40

9:00 – 9:30 am O23 **Ling Ma** and M. F. Hawthorne
"Closomers of High Boron Content: Synthesis, Characterization, and Potential Application as Unimolecular Nanoparticle Delivery Vehicles for BNCT"
Abstract on Page 41

9:30 – 10:00 am O24 **Ramón Macías**, Thomas P. Fehlner, and Alicia M. Beatty
"Chemistry of [1-Cp*-1-H-*arachno*-1-IrB_4H_9]: Reactions with Lewis Bases, Bromine, and [η^6-(1,2-$(CH_3)_2$-C_6H_4)$Mo(CO)_3$]"
Abstract on Page 42

10:00 – 10:30 am *Morning Break*

10:30 – 11:00 am O25 **Matthew G. Fete** and Josef Michl
"1-H-2,3,4,5,6-$F_5(CF_3)_6CB_{11}$(-) and 1-H-$(CF_3)_{11}CB_{11}$(-): New Trifluoromethylated Carborane Anions"
Abstract on Page 43

11:00 – 11:30 am O26 Ruaraidh McIntosh, David Ellis, Georgina M. Rosair, and **Alan J. Welch**
"Unanticipated Expansion of a 12-Vertex Molybdacarborane"
Abstract on Page 44

11:30 am – 12:00 pm O27 Matthew Fischer, **Paul A. Jellis**, Justin L. Mason, Jamie M. Nazzoli, Justin H. Orlando, and Lisa M. Phifer
"Electrochemical and Spectroscopic Study of Ruthena- and Rhenacarborane Complexes"
Abstract on Page 45

Saturday Afternoon, May 22, 2004

Chairpersons: Dr. Stephen Kahl and Dr. Thomas McGrath

1:30 – 2:00 pm	O28	**R. Bruce King** "Cyclopentadienylmetal Vertices in Polyhedral Boranes: Geometry and Chemical Bonding" Abstract on Page 46
2:00 – 2:30 pm	O29	Rhodri L. Thomas, Mitsuhiro Hata, Nigam P. Rath, Grainne Biddlecombe, and **Lawrence Barton** "Formation Group 4 Transition Metal Derivatives of Small Boranes and Novel Borane Coupling Reactions" Abstract on Page 47
2:30 – 3:00 pm	O30	**Donald S. Matteson** "Conversion of Alkylboronic Esters Alkyldichloroboranes" Abstract on Page 48
3:00 – 3:30 pm		*Afternoon Break*
3:30 – 4:00 pm	O31	**Sundargopal Ghosh**, Bruce C. Noll, and Thomas P. Fehlner "Metal-Carbonyl Promoted Direct Synthesis of Novel Decametallaborane System: *Exo-Nido*-(η^5-$C_5Me_5Ru)_2B_{10}H_{16}$ and *Nido*-(η^5-$C_5Me_5Ru)_2B_{10}H_{16}$" Abstract on Page 49
4:00 – 4:30 pm	O32	**Angel Ramos-Organillo**, Angelina Flores-Parra, and Rosalinda Contreras "NMR Study and In Solid State of Coordinating Behaviour of 2-Substituted Benzimidazole Compounds" Abstract on Page 50

Boron in the Americas IX Poster Program

Thursday Evening, May 20, 2004
LBJ Student Center Ballroom, 6:00 – 9:00 pm

P1 **Peng Lei**, Thomas D. McGrath, and F. Gordon A. Stone
"Synthesis and Structure of the Novel and Zero-Valent Di-Molybdenum-Carborane Trianion [1,3,6-{$Mo(CO)_3$}-3,6-(μ-H)$_2$-1,1,1-$(CO_3)_2$-Ph-*closo*-1,2-$MoCB_9H_7]^{3-}$ and its Reactivity by Substitution and Oxidation"
Abstract on Page 52

P2 Matthew Fischer, Paul A. Jelliss, Justin L. Mason, **Justin H. Orlando**, and Keith Wampler
"Preliminary Investigations Into the Photophysics and Electrochemistry of [3,3-$(CO)_2$-3-NO-*closo*-3,1,2-$ReC_2B_9H_{11}$]
Abstract on Page 53

P3 **Adriana Esparza-Ruiz**, Julio Hernández-Díaz, Fabiola Téllez, Angelina Flores-Parra, and Rosalinda Contreras
"New Boron Heterocycles Derived from Benzimidazole, Benzotriazole, and Benzothiazole"
Abstract on Page 54

P4 **Magdalena Kvícalová**, Brook McConnell, Hana Divisová, and Josef Michl
"Preparation of C-Functionalized Derivatives of $CB_{11}H_{12}^-$"
Abstract on Page 55

P5 M. -L. Yao, **A. Naravane**, and G. W. Kabalka
"Synthesis of 1-Amino-3-[(Dihydroxyboryl)Ethyl]-Cyclobutanecarboxylic Acid as a Potential Boron Neutron Capture Therapy Agent"
Abstract on Page 56

P6 Nicolas Merle, Christopher G. Frost, Michael C. Willis, and **Andrew S. Weller**
"Transition Metal Complexes of Chelating Phosphine Boranes"
Abstract on Page 57

P7 **Kanth V. B. Josyula**, Peng Gao, and Chris Hewitt
"Dichloroborane-Dioxane: An Exceptional Reagent for the Preparation of Alkenyl- and Alkylboronic Acids"
Abstract on Page 58

P8 **Kengkaj Sukcharoenphon**, Thomas D. McGrath, and F. Gordon A. Stone
"Synthesis and Derivatization of Charge-Compensated Triruthenamonocarborane Complexes Containing Thioethers $[Ru_3(CO)_8(\mu^3{:}\eta^5\text{-}7\text{-}L\text{-}CB_{10}H_{10}]$ (L = SMe_2, SC_4H_8)"
Abstract on Page 59

P9 **Matt Fischer**, Paul A. Jellis, and Lisa Phifer
"Synthesis of Bimetallic Metallocarboranes with Chromophoric Chelating Ligands and the Study of their Spectroscopic Properties"
Abstract on Page 60

P10 **María Liliana Marín** and Rosalinda Contreras
"Boron-Phosphorus and Boron-Nitrogen Adducts BX_3ER_3 (E = P, N; R = Cl, F or H) Derived from *o*-Methoxyphenylphosphines and *o*-Methoxyphenylamine"
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Abstracts of

ORAL

Presentations

BORON-RICH OLIGOMERIC PHOSPHATE DIESTERS WITH APPLICATIONS IN BNCT AND DRUG DELIVERY

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Los Angeles, California, 90095

Boron-rich oligomeric phosphate diesters (OPDs) are potential agents for use in boron neutron capture therapy. Furthermore, recent in vitro studies have suggested that OPDs might find application in the delivery of drugs whose site of action lies within the cell nucleus. Here we describe the large scale, solution phase synthesis of OPDs as well as the results of in vitro tumor uptake studies.

SYNTHESES AND STRUCTURAL STUDIES OF 10-VERTEX SILADICARBABORANES AND THE FIRST SILAMONOCARBABORANES

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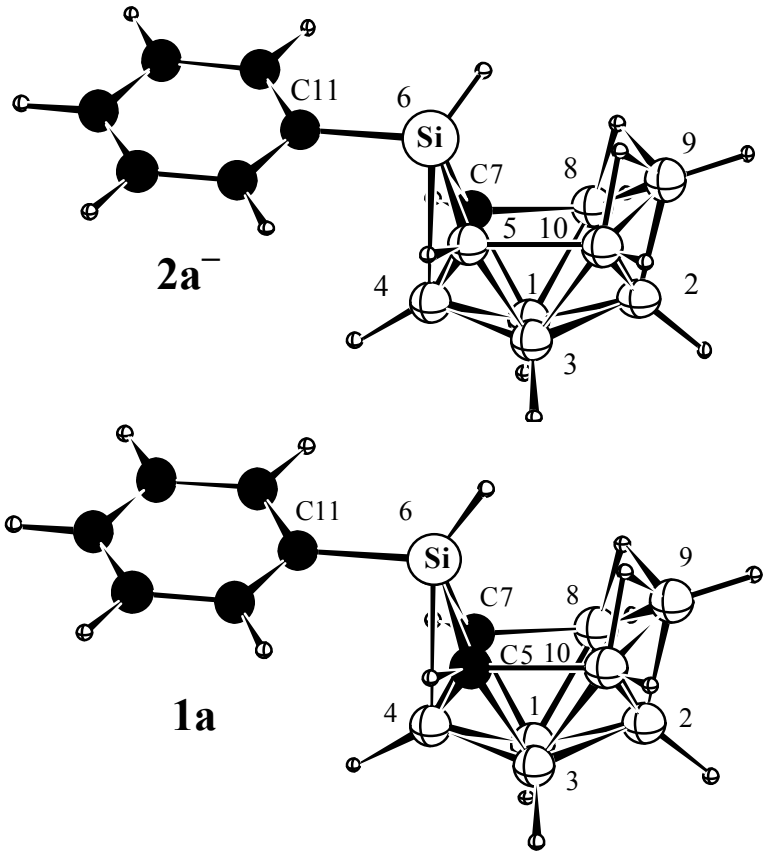
The first 10-vertex siladicarbaboranes, 6-R-*arachno*-6,5,7-$SiC_2B_7H_{12}$ (R = Ph, **1a** or Me, **1b**) and silamonocarbaboranes, PSH^+(6-R-*arachno*-6,7-$SiCB_8H_{12}^-$) (R = Ph, **2a**$^-$ or Me, **2b**$^-$) have been synthesized via the proton sponge initiated in situ dehydrohalogenation reactions of *arachno*-4,6-$C_2B_7H_{13}$ and *arachno*-4-CB_8H_{14} carbaboranes with $RSiHCl_2$ (R = Ph or Me). DFT/GIAO computations have confirmed that **1** and **2** have *arachno* cage frameworks based on an icosahedron missing two adjacent vertices. In both compounds the silicon and carbon atoms are located in adjacent positions on the open six-membered face.



DFT optimized geometries for *exo*-6-Ph-*arachno*-5,6,7-$SiC_2B_7H_{12}$ (**1a)** and *exo*-6-Ph-*arachno*-6,7-$PCB_8H_{12}^-$ (**2a**$^-$**)** at the B3LYP/6-311G* level.

HÜCKEL, WADE'S AND JEMMIS RULES

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Electron Counting Rules have played a central role in the development of modern chemistry. The first benefits of Quantum Mechanics in Organic Chemistry came in the form of Hückel 4n+2 □ electron Rule applicable to cyclic planar conjugated systems. The Hückel Rule provided a structural link between benzenoid aromatics and graphite. Pyramidal carbocations and polyhedral boranes provided the initial connection between 2- and 3- Dimensional Aromatcity. Based on many observations in polyhedral borane chemistry, Wade established the n+1 skeletal electron pair rule for polyhedral boranes. Unlike benzenoid aromatics where the condensation is usually by edge-sharing, there are several ways of condensing polyhedral boranes. These include edge-sharing, triangular face sharing, four atom sharing, and single atom sharing. A new electron counting rule,[1,2] that is applicable for any type of condensation of polyhedral boranes, will be presented. According to this, condensed structures with m+n+o skeletal electron pairs are more stable (m = # of polyhedra, n = # of vertices, and o = # of single-atom bridges). Using the closo-, nido-, and arachno- descriptions familiar in the chemistry of boranes, this electron counting rule can be applied to metallocenes, metallaboranes and any of their combinations.[3] When restricted to 2-dimensional edge-sharing, the mno Rule reduces to the Hückel 4n+2 Rule. A connection between polyhedral boranes and elemental boron, analogous to the benzene–graphite relation, evolves out of this rule.[1b] Application of these ideas[3,3] to boranes, metallaboranes, elemental boron and boron-rich solids will be presented. This also provides a relation between boron and fullerenes.[4]

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THE SYNTHESIS OF STERICALLY HINDERED AND ROBUST CARBORANYL-PNICTOGEN LIGANDS FOR COORDINATION CHEMISTRY AND CATALYSIS

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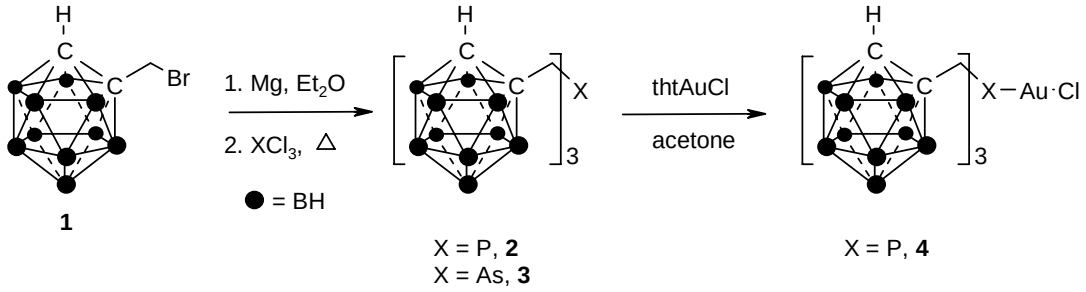
Electron-rich hindered pnictogen ligands such as tri(t-butyl)phosphine and tri(o-tolyl)phosphine are routinely used to isolate unique coordination complexes and as co-ligands in palladium-catalyzed cross-coupling reactions. The effectiveness of such ligands is impaired by their rapid oxidation upon exposure to air and moisture.

Using carboranes (dicarba-*closo*-dodecaboranes), we have prepared and characterized a series of tertiary pnictogen ligands which are both sterically-hindered and resistant to oxidation in air. The synthesis of these ligands, as well as their use in coordination chemistry and palladium-catalyzed cross-coupling reactions, will be presented.



1

X = P, **2**
X = As, **3**

X = P, **4**

PRELIMINARY STUDY ON GADOLINIUM NEUTRON CAPTURE THERAPY

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Since 1990 boron neutron capture therapy (BNCT) for malignant brain tumors has been investigated at Kyoto University Brain Tumor Group. However boron targeting technique is still premature and the investigation of the more efficacious boron carriers has been addressed in this area. ^{157}Gd is an fascinating atom for this therapy, since it has a large thermal neutron cross section of 255,000 barn which is 65 times as that of ^{10}B and releases Auger electrons, internal conversion electrons, γ rays and X rays by a single thermal neutron capture reaction sharing among them the total kinetic energy of 7.7 MeV that is more than 2 times as that of ^{10}B(n, α)^{7}Li reaction. In the boron-based NCT, high LET particles of α and its recoil ^{7}Li particle release 3.3 MeV only within their total trajectory of less than 14μm. Such limited energy transfer within short trajectory in tissue can save serious radiation injury onto the normal brain surrounding the tumor. On the other hand, however, dose distribution in the tumor is sharply dependent on the microdistribution of ^{10}B in tumor that revealed to be heterogeneous carried non-uniform dose distribution in the tumor even aside from a variety of proliferation condition of the tumor cells. Very important point of this therapy is selective destroy of the tumor cells invading peri-tumoral parenchyma shown as abnormal high density area in the T_2-weighted MRI image since 80-90% recurrence occur in this area even after multimodal treatment of the tumor. Dose distribution of Gd-based NCT is more uniform than that of boron-based NCT and might be suitable for pathological heterogeneity of malignant tumors and even on the tumor cells invading into this abnormal high density area in the T_2-weighted MRI image.
In this preliminary study, GdNCT effect on experimental brain tumors was investigated using megulmine gadopentate (GdDTPA) and the theoretical dose distribution is presented. And clinical applicability of a Gd-B complex was assessed on malignant brain tumors.

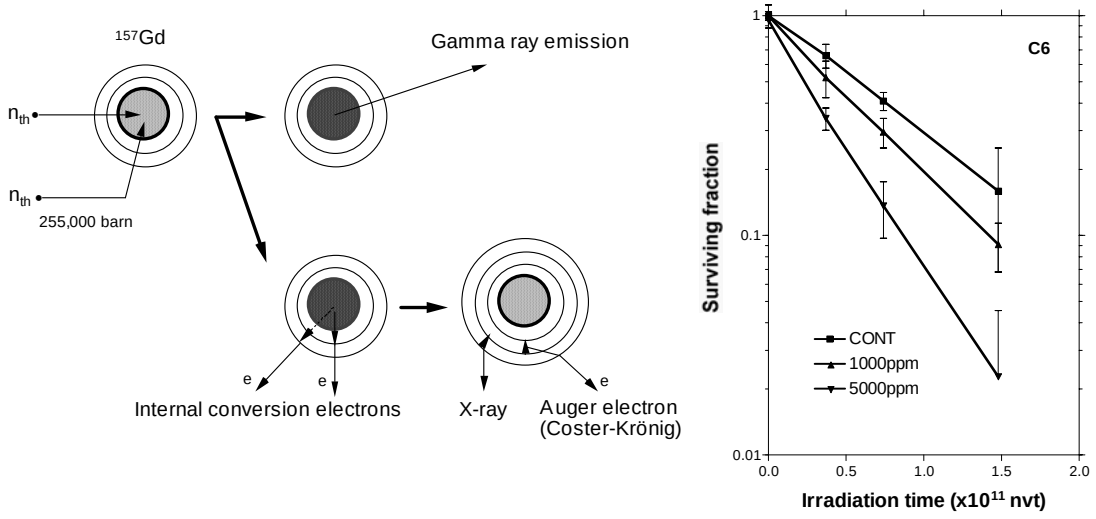

Figure *left*: Thermal neutron capture reaction of ^{157}Gd(n, γ)^{158}Gd, *right*: Surviving fraction of C6 tumor cell line on ^{157}Gd(n, γ)^{158}Gd reaction at the concentration of 1000 and 5000ppm ^{n}Gd. The surviving fraction are almost equivalent to that of 20ppm ^{10}B-BSH.

NEW α-AMINO ACID DERIVATIVES WITH CARBORANYL FRAGMENTS IN α- AND β-POSITIONS

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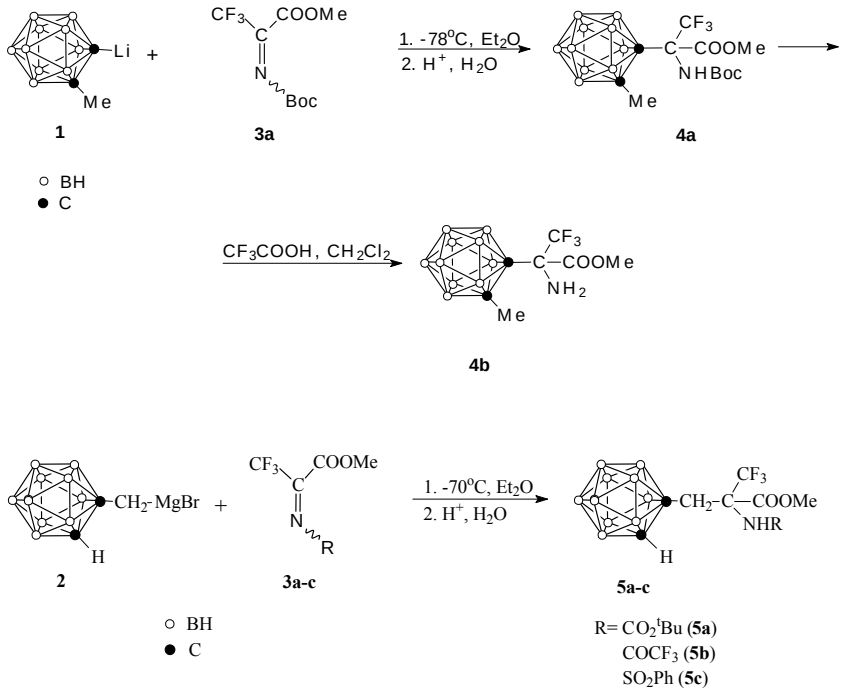
A simple and effective method of synthesis of the derivatives of α-carboranyl-α-trifluoromethyl-α-aminoacid (**4**) and β-carboranyl-α-trifluoromethyl-α-aminoacid (**5**) by interaction of correspondingly methyl-o-carboranyllithiym (**1**) with *tert*-butoxycarbonylimine methyltrifluoropyruvate (**3a**) and *o*-carboranylmethylmagnesium bromide (**2**) with imines $(CF_3)(CO_2Me)C{=}NR$ [R=$CO_2{}^tBu$ (**3a**), $COCF_3$ (**3b**), SO_2Ph (**3c**)] is presented.



The structures of esters of β-carboranyl-α-trifluoromethyl-α-aminoacids with different protected amino groups were determined by ^1H, ^{19}F NMR and single crystal X-ray analysis.

Acknowledgement
We thank the Russian Foundation for Basic Research (grant 02-03-32680) for financial support.

CHEMISTRY OF CRYSTALLINE HYDRATED ZINC BORATES

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Boron-oxygen compounds have by far the greatest industrial importance of any class of boron compounds. Within this class, metal borates find numerous technological uses. Although alkali and alkaline earth metal borates dominate in terms of tonnage, other synthetic metal borates are also utilized extensively, particularly as additives for polymers and advanced construction products. A good example is found in the zinc borates. Represented by a dozen unique crystalline species, zinc borates are structurally diverse and illustrate many chemical principles general to the industrially important metal borates. These compounds contain BO_3 and BO_4 groups that may occur as isolated oxoanions or linked together by oxygen-sharing to form rings, chains, sheets and networks. So-called hydrated borates, which account for most mineral and synthetic borates used by industry, contain B-OH or OH^- groups, and optionally interstitial H_2O. Hydrogen-bonding also plays a pronounced role in the structures of these materials. Aside from several anhydrous zinc borates, good evidence exists for at least eight unique crystalline hydrated zinc borates. Despite the extensive industrial applications for some of these, only half have been structurally characterized and most of these only in the past few years. This paper summarizes recent developments in the field of zinc borate chemistry and highlights some of the mysteries that still remain.

NEW SYNTHETIC METHODOLOGIES FOR BULK/NANOSTRUCTURED METAL CHALCOGENIDES: UTILIZATION OF NEW BORON CHALCOGENIDE CHEMISTRY

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Boron chalcogenides are volatile and corrosive compounds which have been utilized rather sparingly. We have recently found that they can serve as a convenient source of chalcogens for preparations of various metal chalcogenides.[1] In solid/gas reactions at the temperatures from 350 to 800 °C, boron chalcogenides are prepared *in situ* and react within 24 hours with metal source compounds to provide pure products of targeted chalcogenides such as TiS_2, TiS_3, VS_4, FeS_2, NiS_2, NbS_3, MoS_2, RuS_2, WS_2, Y_2S_3 and RS_2 (R = Ce, Nd, Sm, Eu, Tb and Er).[2] The co-products such as boron oxide or halides can be removed easily and can be recycled if necessary. Preparation of ternary chalcogenides is also viable through the same reaction procedure. In addition, the relatively low temperature condition prevents a significant growth of particles during the reactions, so that the new preparative method allows a simple one-step conversion of metal-oxide nanoparticles into the corresponding chalcogenide nanoparticles.[3] We also have developed a new size-controllable synthetic route for the II-VI quantum dots, which utilizes the good solubility of the chalcogenides in amines.[4] Advantages of the new method will be discussed.

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4. Iancu, N.; Seo, D.-K. *Inorg. Chem.* submitted for publication.

SYNTHESIS OF AMINO ACIDS VIA ORGANOBORANES

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Synthesis of unnatural amino acids is an area of prime importance. During the past decades, our group has developed novel organoborane-mediated methods for the stereoselective synthesis of several classes of organic molecules. Currently, we have been involved in the synthesis of natural and unnatural amino acids, including fluorinated amino acids via organoboranes. The most recent developments from our laboratories will be discussed.

AN EFFECTIVE ASYMMETRIC ALKYLATION SYSTEM OF ALDIMINES WITH DIETHYLZINC: MIXED IONIC LIQUIDS OF [*N*-PENTYLPYRIDIUM][1-MENTHOXYMETHYL-*CLOSO*-$CB_{11}H_{11}$] AND 1-BUTYL-3-METHYLIMIDAZOLIUM HEXAFLUOROPHOSPHATE

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Ionic liquids have drawn considerable attention in recent years as promising organic reaction media. [1] However, there are only a few reports regarding the preparation and use of chiral anion based ionic liquids. [2] We have been interested in the use of ionic liquids, [3] and have synthesized the first chiral carborane anion based on the ionic liquid [*N*-pentylpyridium][1-menthoxymethyl-*closo*-$CB_{11}H_{11}$], which has been characterized by NMR, IR and elemental analysis.

Enantioselective alkylation of imines is one of the significant challenges in asymmetric synthesis. Therefore, the enantioselective additions of diethylzinc (Et_2Zn) to aldimines were examined in the ionic liquid, [N-pentylpyridium][1-menthoxymethyl-$CB_{11}H_{11}$] with the commercially available ionic liquid, 1-butyl-3-methylimidazolium hexafluorophosphate, [BMIM][PF6], as a co-solvent. Secondary amines have been obtained from this new method in yields of up to 95%

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CAMOUFLAGED CARBORANE AMPHIPHILES: SYNTHESIS AND SELF-ASSEMBLY

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Abstract

A series of amphiphilic ammonium salts containing B-polymethylated (camouflaged) carborane groups have been designed and synthesized. Combined hydrophobic and hydrophilic properties of these single-chained amphiphiles allow their spontaneous self-asembly to form rods under a variety of conditions, such as concentration, carborane cage structure, chain-length, counterions, solvents, methods of preparation, the number of attached chains, and the ionic charge. The rods have been studied by TEM, optical microscopy, XRD, TG/DTA and FTIR. These polymethylated carborane derivatives are predisposed to self-assemble, and the hydrophobic interaction played an important role. This work demonstrated the first use of B-polymethylated carborane-containing structures in the self-assembly of supramolecular structures.

ASYMMETRIC ALLYLBORATION WITH 10-SUBSTITUTED 9-BBDS: HOMOALLYLIC AMINES FROM *N*-TMS ARYL ALDIMINES AND, FOR THE FIRST TIME, ARYL AND ALKYL *N*-TMS KETIMINES

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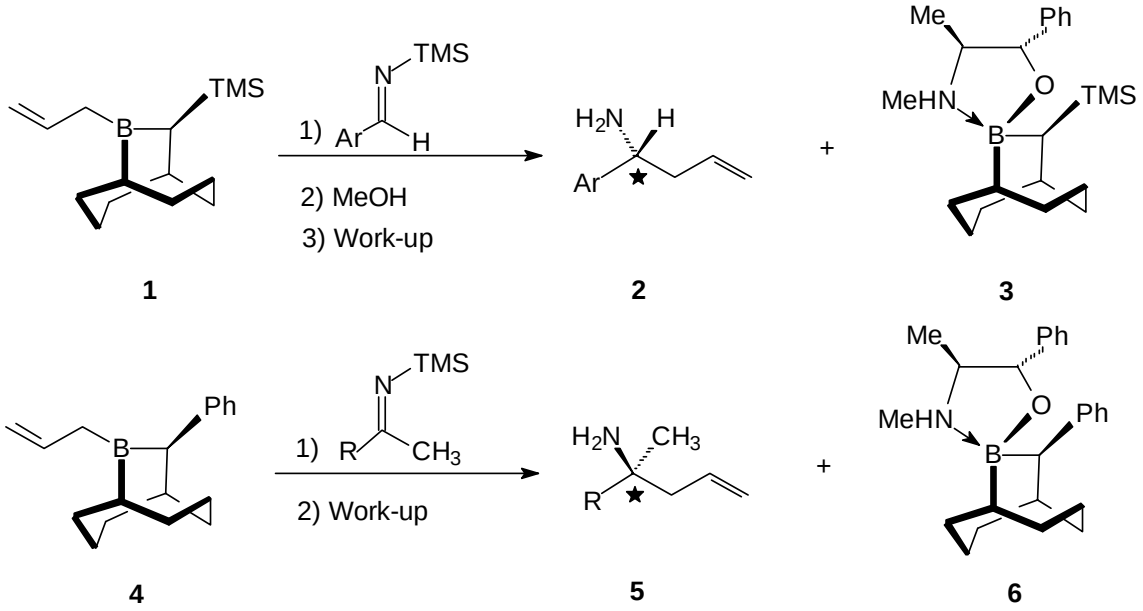
The asymmetric allylboration of *N*- TMS aryl aldimines with *B*-allyl-10-TMS-9-borabicyclo[3.3.2]decane **1** gives 2°-alkyl homoallylic amines **2** in good optical purities (76-89% ee) and chemical yields (60-90%), comparable to those obtained with $AllB(Ipc)_2$. Analogous to this latter process, the *N*-TMS substitution prevents the allylation from occurring even at room temperature. However, the addition of 1 equiv of MeOH generates the free imine (*N*-H) which undergoes allylation with **1** at -78 °C in 9-12 h. The new procedures also employ a convenient non-oxidative work-up which permits the efficient recovery (50-70%) of the chiral boryl moiety as **3** which is recycled directly back to **1** (98%) with allylmagnesium bromide. The previously unknown asymmetric allylboration of *N*-TMS ketimines was examined employing optically pure *B*-allyl-10-Ph-9-borabicyclo[3.3.2]decane **4** which reacts smoothly with *N*-TMS ketimines to produce 3°-alkyl homoallylic amines **5** in high optical purity (R = Ar, *t*-Bu, 89-98% ee) and good yields (50-75%). As expected, simple aliphatic ketimine derivatives exhibit lower selectivities (*i.e.* R = *n*-Pr, 19% ee). This new methodology can be applied to aromatic and aliphatic ketimines providing a general protocol to **5** as well as to the efficient recovery of **6** which is directly converted back to **4** with allylmagnesium bromide**.** The possible origins of the enantioselectivities observed in these processes will be presented and discussed.



CONVECTION ENHANCED DELIVERY OF A BORONATED PORPHYRIN TO INTRACEREBRAL HUMAN GLIOBLASTOMA XENOGRAFTS IN ATHYMIC RATS

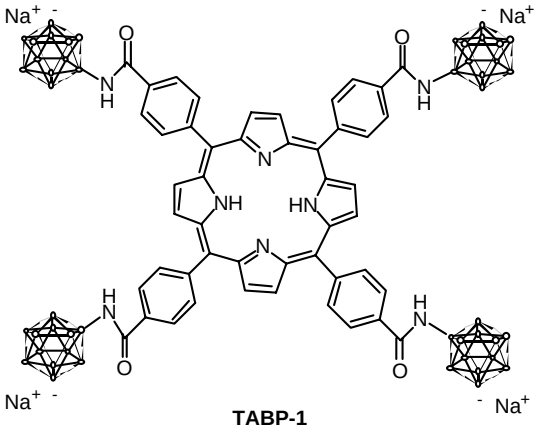
Tomoko Ozawa[1], Raquel A. Santos[1], Myoung-Seo Koo[3], Stephen B. Kahl[3] and Dennis F. Deen.[1,2] *Departments* of Neurological *Surgery[1], Radiation Oncology[2] and Pharmaceutical Chemistry[3], University of California, San Francisco, California, USA*

Purpose: Boron neutron capture therapy (BNCT) is an adjuvant therapy that has the potential to control local tumor growth. A selective delivery of sufficient amounts of boron to individual tumor cells, compared with surrounding normal tissues, is the key for successful BNCT, and new delivery agents are under current investigation. Recently, we have designed and synthesized a new highly water-soluble boronated compound, TABP-1, as a possible BNCT agent. Previously we injected the maximum tolerated dose (15 mg/kg) of TABP-1 systemically via the tail vein into athymic rats bearing intracerebral human glioblastoma U-87 MG xenografts. Our data indicated that TABP-1 injected intravenously accumulated in brain tumors compared to normal brain; however the level of boron in the tumor was less than generally considered optimal for therapy ($\geq$ 30 micrograms/g tissue). In the present study, we investigated whether convection enhanced delivery (CED) could improve the boron distribution.

Methods: TABP-1 was administered directly into intracerebral U-87 MG xenografts using an Alzet osmotic minipump attached to a brain infusion cannula. Doses of TABP-1 ranging from 2.5×10^{-4} mg to 1.0 mg dissolved in 200 microliters of PBS were infused locally over 24 hours. Animals were sacrificed 1 day after completion of the infusion and tissue boron levels measured by inductively coupled plasma atomic emission spectrometry.

Results: Doses ≥0.25 mg produced average tumor boron concentrations significantly greater than those obtained systemically at the MTD. For example, CED administration of 0.5 mg of the compound resulted in a tumor boron level of 97.4 micrograms/g tumor, but a serum level of only 0.4 micrograms/g (tumor-to-serum ratio ≈ 238:1). This mode of local delivery also resulted in relatively high tumor to normal brain ratios of ~5:1 for surrounding brain and ~29:1 for contralateral brain tissue at the 0.5 mg dose. Even at the highest dose (1.0 mg), serum boron levels equaled control levels.

Conclusions: Using CED we may be able to achieve therapeutic BNCT efficacy with minimal systemic toxicity or radiation-induced damage to normal tissue with TABP-1.
Supported by NIH grant CA-82478 and DOE grant DE-AC02-76SF00098.



TABP-1

FACILE SYNTHESES OF ORGANIC HALIDES USING ORGANOTRIFLUOROBORATES

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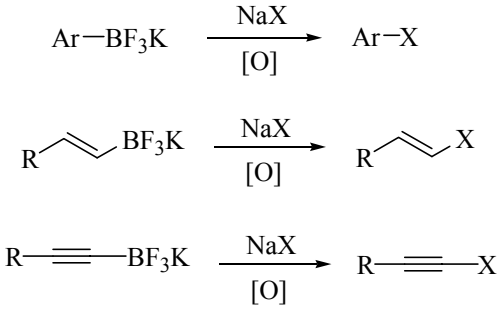
Organic halides are widely used as synthetic intermediates in medicinal and pharmaceutical research. Organometallic reagents are convenient precursors to a wide variety of organic halides but their use is somewhat restricted due to the high reactivity and toxic properties of many of the reagents. The use of organoboranes as precursors to organic halides has been of continuous interest in our laboratory for almost 30 years and we have utilized boron-halogen exchange for preparing a wide variety of radiopharmaceuticals.[1,2] Initially, we developed methods to halogenate trialkylboranes but found that boronic acids and esters were more convenient to handle and could be prepared containing a wide variety of functional groups (an important consideration in pharmaceutical research). [3] However, the use of boronic acids as organohalogen precursors to pharmaceuticals has not kept pace with that of other reagents presumably due to their propensity to form boroxines that are unstable to both air and water.

Recently, potassium aryl- and vinyltrifluoroborates have proven to be versatile intermediates in organic synthesis because of their remarkable chemical stability. They are crystalline solids that are stable to both air and water, and they are readily prepared from the corresponding boronic acids by addition of KHF_2. In addition, trifluoroborate salts have proven to be as versatile as boronic acids in organic synthesis. We wish to describe the use of organotrifluoroborates as precursors to a wide variety of structurally diverse organic iodides and bromides and the application of this new methodology to the rapid and high yield synthesis of high specific activity, iodine-123 labeled aryl, vinyl and alkynyl iodides. [4]



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BUILDING BUTTERFLIES ON A RHENIUM-MONOCARBORANE SUBSTRATE

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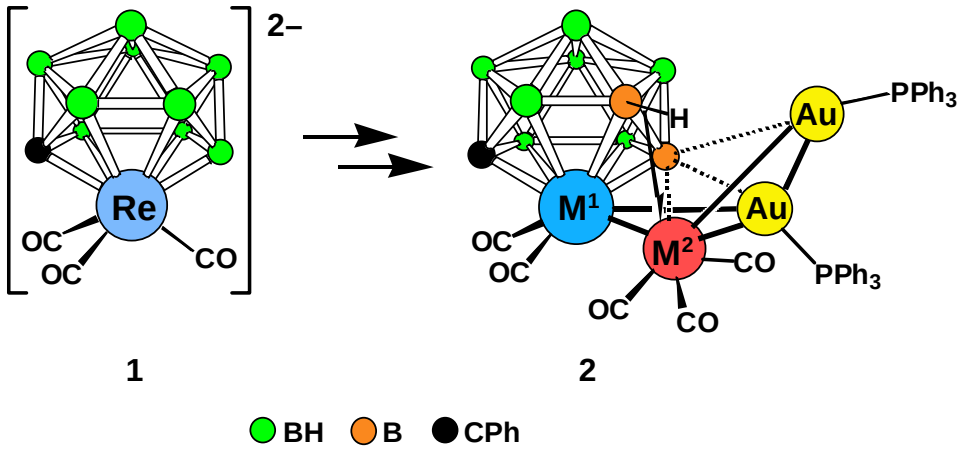
A range of polymetallic species can be prepared by reaction of the rhenium-monocarborane dianion **1** with transition-metal cations.[1] By stepwise addition of the fragments $\{M(CO)_2\}$ (M = Rh or Ir[2]) and then $\{Au(PPh_3)\}$ to **1**, carborane-supported $\{ReMAu_2\}$ butterflies **2** have been assembled. The surprising structures of compounds **2** and related species will be presented and their mechanism of formation discussed.



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AN INVESTIGATION OF NUCLEOPHILIC ATTACK ON THE $[n\text{-}B_{20}H_{18}]^{2-}$ ANION

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The reaction of small nucleophiles (Nu), such as $[OH]^-$ and $[OCH_3]^-$, with the $[n\text{-}B_{20}H_{18}]^{2-}$ anion produces a kinetic isomer, $[B_{20}H_{17}Nu]^{4-}$, characterized by an apical-equatorial connection between the polyhedral borane cages and the substituent location on the equatorial belt adjacent to the boron-boron intercage connection.[1] Acid catalyzed rearrangement of the kinetic isomer yields the thermodynamic isomer, characterized by an apical-apical connection between the polyhedral borane cages.[1] In contrast, the reaction of $[SC(O)OC(CH_3)_3]^-$ and $[n\text{-}B_{20}H_{18}]^{2-}$ produced, directly and in good yield, the $[B_{20}H_{17}SC(O)OC(CH_3)_3]^{4-}$ anion characterized by an apical-apical connection between the polyhedral borane cages and the substituent location on the equatorial belt adjacent to the terminal boron apex.[2] Therefore, the investigation of the reactivity of a variety of sterically hindered nucleophiles and the $[n\text{-}B_{20}H_{18}]^{2-}$ anion was conducted. The solvent utilized in the reactions was tetrahydropyran. Products of the reactions were characterized by ^1H, ^{13}C, and ^{11}B NMR spectroscopy. In each case, spectroscopic evidence indicated that the oxygen atom of the tetrahydropyran solvent was coordinated to the equatorial belt adjacent to the boron-boron intercage connection, an intermediate analogous to that proposed for tetrahydrofuran in some reactions.[3] As a result, two investigations are currently underway. In the first set of reactions, the coordination of different solvents with the $[n\text{-}B_{20}H_{18}]^{2-}$ is being conducted. In the second set of reactions, the investigation of the reaction of sterically hindered nucleophiles with the $[n\text{-}B_{20}H_{18}]^{2-}$ anion in tetrahydrofuran is being conducted. The results of the investigations, to date, will be presented.

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THE SYNTHESIS OF Tc(I) AND Re(I) CARBORANE COMPLEXES AND THE DEVELOPMENT OF ORGANOMETALLIC RADIOPHARMACEUTICALS

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Organometallic complexes of radiometals like ^{99m}Tc are attractive synthons for preparing receptor-targeted radiopharmaceuticals because of their small size and resistance to catabolism. A major obstacle to their use involves finding methods for preparing organometallic-radiometal complexes in aqueous media where the concentration of the radionuclide is typically on the order of 10^{-8} to 10^{-9}M.

Carboranes, unlike the majority of traditional organometallic ligands, are compatible with forming metal complexes in aqueous media. Unfortunately, traditional method for preparing metallocarboranes involves the use of highly basic reaction conditions, which precludes using the clusters to prepare bioconjugates.

We have recently developed a new and highly efficient methodology for preparing metallocarborane complexes of Re and Tc in water under mild reaction conditions. The approach involves reacting *nido*-carborane ligands with $[M(CO)_3(OH_2)_3]^+$ (M= Re, Tc) in the presence of aqueous potassium fluoride. The use of KF as a base afforded the desired metallocarboranes in excellent yields while avoiding the formation of Re-clusters, which are byproducts commonly observed when analogous reactions are carried out in the presence of strong aqueous bases. The KF mediated reactions also work at the tracer level and we have recently reported the first examples of ^{99m}Tc-labeled carborane complexes which was prepared in high radiochemical yield and purity. These organometallic complexes are stable to cysteine and histidine challenges for more than 24 hours making them viable synthons for designing radiopharmaceuticals.

The presentation will cover topics pertaining to the synthesis of ^{99m}Tc and Re labeled carborane complexes. Related advances in the bioconjugation chemistry of carboranes and metallocarboranes will also be described.

THE POSSIBLE ROLE OF THE CARBONIUM YLIDE C(+)$B_{11}ME_{11}$(-) AS A REACTION INTERMEDIATE

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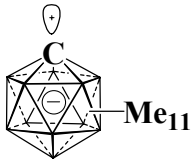
We report synthetic and kinetic evidence for a reactive intermediate that reacts as if it were the carbonium ylide C(+)$B_{11}Me_{11}$(-), which can also be visualized as a carbenoid. This species is produced under various solvolytic conditions, e. g. when 1-[T-Br-$(CH_2)_n$]$CB_{11}H_{11}$(-) (n = 2, 5, or 6) anions are methylated in sulfolane solution with methyl triflate in the presence of CaH_2.



THE SYNTHESIS OF PERMETHYLATED 1-VINYL AND 1-ETHYNYL CB_{11}^- ANIONS

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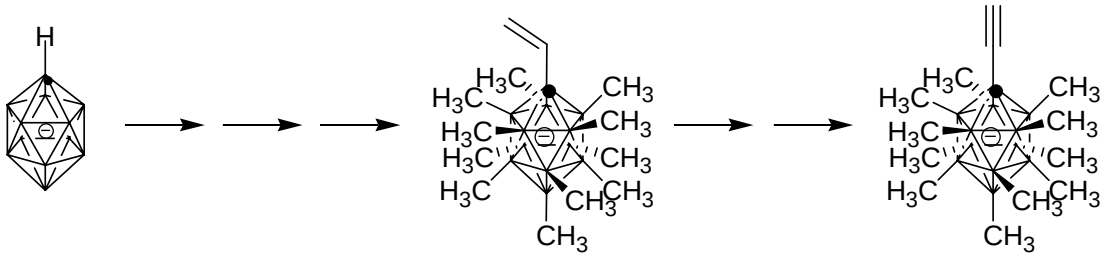
Boulder CO 80309-0215

The synthesis of the 1-vinyl- and 1-ethynyl-$CB_{11}(CH_3)_{11}^-$ anions will be described. The synthetic route involves alkylation on the carborane carbon atom and methylation of the cage boron atoms, followed by Hofmann elimination to afford the vinyl moiety. Addition of bromine followed by dehydrohalogenation yields the ethynyl derivative.



CLOSOMERS OF HIGH BORON CONTENT: SYNTHESIS, CHARACTERIZATION AND POTENTIAL APPLICATION AS UNIMOLECULAR NANOPARTICLE DELIVERY VEHICLES FOR BNCT

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Abstract

Three novel ether- and ester-linked closomers with exciting potential as BNCT drugs have been designed and synthesized. Our Newly developed degradation method employing NaCN as the deboronation reagent was utilized to obtain sodium salts of the *nido*-ether linked closomer polyanions. The water solubility of these sodium salts suggests that the *nido* species may be used as unimolecular nanoparticle delivery vehicles for BNCT or as components of unilamellar liposomes.

CHEMISTRY OF [1-Cp*-1-H-*arachno*-1-IrB_4H_9]: REACTIONS WITH LEWIS BASES, BROMINE, AND [η^6-(1,2-$(CH_3)_2$-C_6H_4)$Mo(CO)_3$]

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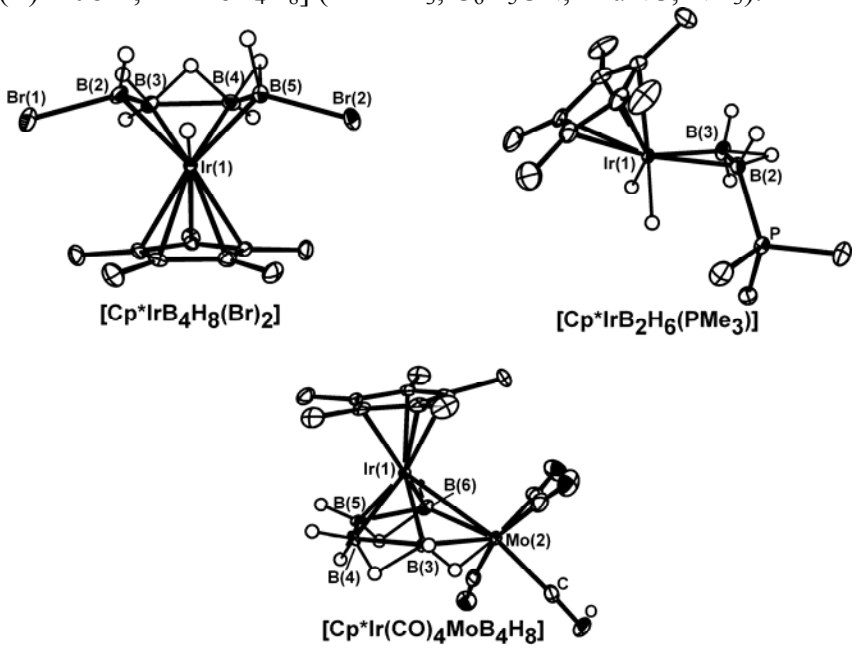
The treatment of [Cp*IrB_4H_{10}] (**1**) with Lewis bases afforded new substituted iridatetra- and iridatriboranes, [Cp*IrB_3H_7L] [L = PMe_3, PMe_2Ph, $PMePh_2$, PPh_3, py, NEt_3] plus BH_3L as coproduct. With PPh_3, py and NEt_3, the formation of the iridapentaborane, [1-Cp*-*nido*-1-IrB_4H_8] was also observed. The substituted iridatetraboranes were to found decompose in solution and in the solid state, affording new iridatriboranes [Cp*IrB_2H_6L]. The decomposition pathways depend on the nature of the Lewis bases. Two routes involving the apparent disproportionation of iridaborane-base adducts were found to compete with the loss of a {BH} unit. A reaction cycle driven by Lewis acid/base chemistry of BH_3 was constructed. In contrast to reaction with nucleophiles, treatment of **1** with bromine and N-bromosuccinimide led to the formation of the dibromo- and monobromoderivatives, [1-Cp*-1-H-2,5-$(Br)_2$-*arachno*-1-IrB_4H_7] and[1-Cp*-1-H-2-Br-*arachno*-1-IrB_4H_8].[1,2]

On the other hand, the reaction of **1** with [η^6-(1,2-$(CH_3)_2$-C_6H_4)$Mo(CO)_3$] in THF gave the metallahexaborane [1-Cp*-2,2,2-$(CO)_3$-2-(THF)-*nido*-1,2-$IrMoB_4H_8$]. This compound is unstable, leading to the formation of the tetracarbonyl analogue [1-Cp*-2,2,2,2-$(CO)_4$-*nido*-1,2-$IrMoB_4H_8$]. The THF-derivative reacts with different nucleophiles such as PPh_3, C_6H_5CN, ^{n}BuNC, and NH_3 to give new molybdenairidaboranes of general formula [1-Cp*-2,2,2-$(CO)_3$-2-(L)-*nido*-1,2-$IrMoB_4H_8$] (L= PPh_3, C_6H_5CN, ^{n}BuNC, NH_3).



1-H-2,3,4,5,6-$F_5(CF_3)_6CB_{11}$(-) AND 1-H-$(CF_3)_{11}CB_{11}$(-): NEW TRIFLUOROMETHYLATED CARBORANE ANIONS

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Methylation[1] of the 1-triisopropylsilyl derivative of the $CB_{11}H_{12}$(-) anion followed by removal of the triisopropyl silyl group yields the hexamethylated anion 7,8,9,10,11,12-$(CH_3)_6CB_{11}H_6$(-), while methylation of the parent anion, $CB_{11}H_{12}$(-), affords the anion 1-H-$CB_{11}Me_{11}$(-). We now report the direct fluorination of these anions with elemental fluorine to yield 1-H-2,3,4,5,6-$F_5(CF_3)_6CB_{11}$(-) (**1**) and 1-H-$(CF_3)_{11}CB_{11}$(-) (**2**), respectively. Like the previously reported anion $(CF_3)_{12}CB_{11}$(-) (**3**),[2] they are resistant to oxidation and stable in highly acidic media. Unlike the salts of **2** and **3**, those of **1** show no signs of explosive behavior. Anions **1** and **2** have an acidic proton on the carbon that is easily removed with a base to allow their incorporation into other structures.

1. King, B. T.; Janoušek, Z.; Grüner, B.; Trammel, M.; Noll, B. C.; Michl, J., *J. Am. Chem. Soc.* **1996**, *118*, 3313.
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UNANTICIPATED EXPANSION OF A 12-VERTEX MOLYBDACARBORANE

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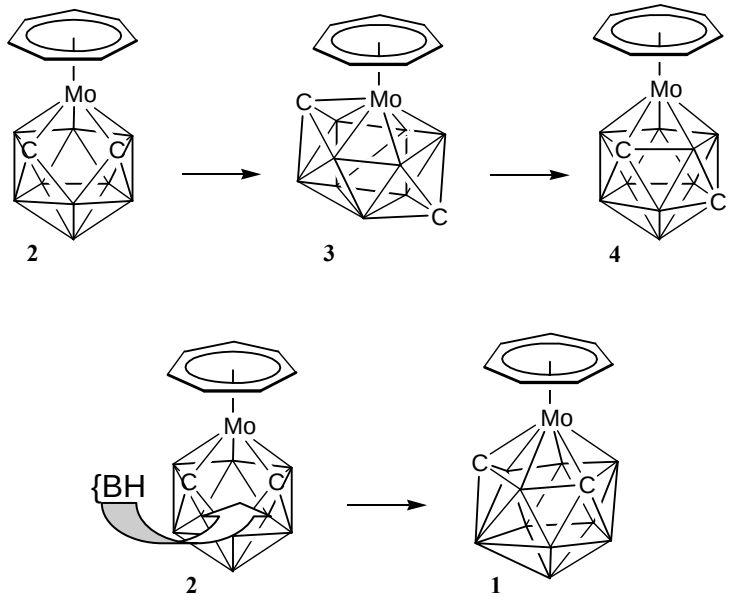
Sodium reduction of 1,2-Ph_2-1,2-*closo*-$C_2B_{10}H_{10}$ followed by addition of $\{Mo(\eta\text{-}C_7H_7)\}^+$ yields the paramagnetic 13-vertex molybdacarborane 1,6-Ph_2-4-(η-C_7H_7)-4,1,6-*closo*-$MoC_2B_{10}H_{10}$ (**1**). Unexpectedly, compound **1** is also afforded when $\{Mo(\eta\text{-}C_7H_7)\}^+$ is added to [7,8-Ph_2-7,8-*nido*-$C_2B_9H_{10}]^-$ previously treated with NaH. When, however, [7,8-Ph_2-7,8-*nido*-$C_2B_9H_{10}]^-$ is deprotonated with BuLi then treated with $\{Mo(\eta\text{-}C_7H_7)\}^+$ three molybdacarboranes are isolated; the pseudocloso species 1,2-Ph_2-3-(η-C_7H_7)-3,1,2-*pseudocloso*-$MoC_2B_9H_9$ (**2**), the non-icosahedral "1,12-Ph_2-2-(η-C_7H_7)-2,1,12-*closo*-$MoC_2B_9H_9$" (**3**), and the isomerised 1,8-Ph_2-2-(η-C_7H_7)-*closo*-2,1,8-$MoC_2B_9H_9$ (**4**).

Compounds **2** and **3** are believed to be intermediates on the isomerisation pathway from the notional, transient, species [1,2-Ph_2-3-(η-C_7H_7)-*closo*-3,1,2-$MoC_2B_9H_9]^‡$ to compound **4**. In the absence of a source of "{BH}", **2** → **3** → **4**. If, however, "{BH}" is available, via NaH degradation of (molybda)carborane, we believe that the 12-vertex pseudocloso compound **2** alternatively is converted into 13-vertex **1** by net {BH} addition.

This reaction represents a new route to 13-vertex metallacarboranes. We are currently exploring its generality.



Molybdacarboranes; C = CPh

Support from the EPSRC and the Carnegie Trust is gratefully acknowledged.

ELECTROCHEMICAL AND SPECTROSCOPIC STUDY OF RUTHENA- AND RHENACARBORANE COMPLEXES

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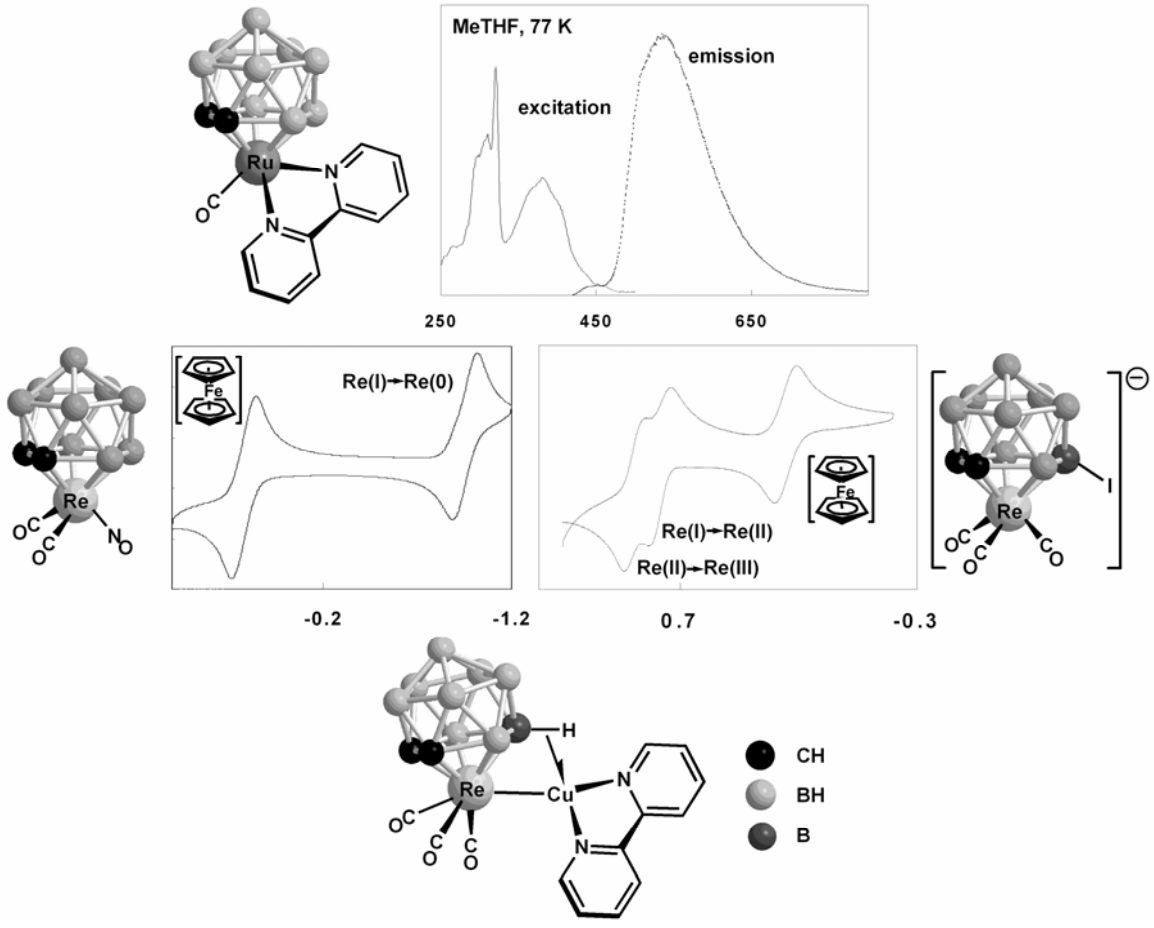
The combination of the metallacarborane architecture with electronically active polypyridyls is a relatively unexplored arena. We are particularly interested in the impact of the carborane cage on the chromophoric and electrochemical properties of the metal-polypyridyl system and have investigated the synthesis and characterization of polypyridyl complexes incorporating ruthenium and rhenium. We can also reveal somewhat unexpected redox chemistry of the anionic complexes [3,3,3-$(CO)_3$-*closo*-3,1,2-$ReC_2B_9H_{11}]^-$ and its neutral nitrosyl derivative [3,3-$(CO)_2$-3-NO-*closo*-3,1,2-$ReC_2B_9H_{11}$], leading to the synthesis and isolation of iodinated rhenacarboranes. In further work we have synthesized bimetallic metallacarborane-polypyridyl systems, where the carborane cage and polypyridyl ligand are linked by a metal-metal bond coupled with B–H→M bridge, such as the Re-Cu complex [3,3,3-$(CO)_3$-μ-3,8-{Cu(2,2'-bipy)}-*closo*-3,1,2-$ReC_2B_9H_{11}$] shown below. The results from luminescence and electrochemistry measurements will be presented, in addition to data from IR, UV-vis, and NMR analysis.



CYCLOPENTADIENYLMETAL VERTICES IN POLYHEDRAL BORANES: GEOMETRY AND CHEMICAL BONDING

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Most cyclopentadienylmetallaboranes containing the vertex units CpM using three internal orbitals and donating one to three skeletal electrons (i.e., M is a transition metal from Group 8, 9, or 10; Cp = typically η^5-C_5H_5 or η^5-Me_5C_5) have structures closely related to binary boranes or borane anions. Smaller clusters of this type, such as metallaborane analogues of *arachno*-B_4H_{10} (e.g., $(CpIr)_2B_2H_8$), *nido*-B_5H_9 (e.g., $(CpRh)_2B_3H_7$ and $(CpRu)_2B_3H_9$), *arachno*-B_5H_{11} (e.g., $CpIrB_4H_{10}$), $B_6H_6^{2-}$ (e.g., $(CpCo)_4B_2H_4$), *nido*-B_6H_{10} (e.g., $CpIrB_5H_9$ and $(CpRu)_2B_4H_{10}$), and *arachno*-B_6H_{12} (e.g., $(CpIr)_2B_4H_{10}$), have the same skeletal electron counts as those of the corresponding boranes. However, such clusters with eight or more vertices, such as metallaborane analogues of $B_8H_8^{2-}$ (e.g., $(CpCo)_4B_4H_4$), *arachno*-B_8H_{14} (e.g., $(CpRu)_2B_6H_{12}$), and *nido*-$B_{10}H_{14}$ (e.g., $(CpRu)_2B_8H_{12}$), have two skeletal electrons less than the corresponding metal-free boranes analogous to the skeletal electron counts of *isocloso* boranes relative to those of metal-free deltahedral boranes. Some metallaboranes have structures not analogous to metal-free boranes but instead analogous to metal carbonyl clusters such as 3-capped square pyramidal $(CpRu)_2B_4H_8$ and $(CpRu)_3B_3H_8$ analogous to $H_2Os_6(CO)_{16}$ and capped octahedral $(CpRh)_3B_4H_4$ analogous to $Os_7(CO)_{21}$. In the metallaborane structures closely related to metal-free boranes the favored degrees of BH and CpM vertices appear to be 5 and 6, respectively.

Cyclopentadienylmetal vertices of earlier transition metals (Groups 6 and 7) can only donate positive numbers of skeletal electrons if they provide four rather than the usual three internal orbitals. The polyhedra for the metallaboranes $(CpM)_2B_nH_{n+4}$ (M = Cr, Mo, W; n = 4, 5) are derived from (n+1)-gonal bipyramids by removal of an equatorial vertex. The deltahedra for the larger metallaboranes $(CpW)_2B_7H_9$, $(CpRe)_2B_nH_n$ ($7 \leq n \leq 10$), and $(CpW)_3B_8H_9$ are derived from the corresponding $B_nH_n^{2-}$ deltahedra by sufficient diamond-square-diamond processes to provide vertices of degrees ≥ 6 for each of the CpM vertices.

FORMATION GROUP 4 TRANSITION METAL DERIVATIVES OF SMALL BORANES AND NOVEL BORANE COUPLING REACTIONS.

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This paper describes our attempts to prepare early transition metals derivatives of small borane clusters. In the case of pentaborane(9) we were able to effect either cage coupling or the formation of metallaboranes, depending on the conditions used. Reaction between $Li[B_5H_8]$ and Cp_2TiCl_2 afforded no observable products but reaction with $CpTiCl_3$ resulted in the formation of $B_{10}H_{14}$ in good yields In reactions with $CpZrCl_2$, pale orange solid, μ-$(Cp_2ClZr)B_5H_8$ (**1**), which exists as a B_5H_9 cage with a Cp_2ClZr moiety replacing a bridging H atom is formed in 70 % yield. **1** exhibits some interesting variable temperature NMR spectral behavior. Passage of a CH_2Cl_2 solution of **1** through silica gel affords **2**, $[(Cp_2Zr)_2B_5H_8][B_{11}H_{14}]$, a yellow air-stable crystalline solid, in 14 % yield. The cation in **2**, $[(Cp_2Zr)_2B_5H_8]^+$, consists of a distorted *spiro*[2.2]pentane-like B_5 moiety comprising two B_3 triangles sharing a naked boron vertex. The two triangles are twisted 72.8° with respect to each other and the two $[Cp_2Zr]$ groups bond in a trihapto-arrangement to the two opposite B-B-B edges. Each exterior B-Zr edge is H-bridged and the B atoms possess terminal hydrogens. The structure of the cation in **2** conforms to the Polyhedral Skeletal Electron Pair Theory by assuming that the unique boron atom is an intra-cage one. Reactions of Cp_2HfCl_2 with $Li[B_5H_8]$ lead to the formation of the analogue of **2**, $[(Cp_2Hf)_2B_5H_8][B_{11}H_{14}]$ (**3**). The precursor to **3**, i.e., the Hf analogue of **1**, is not observed. When B_5H_9 is treated with Cp_2ZrCl_2/*n*-BuLi in the presence of triphenylphosphine, *arachno*-$B_9H_{11}(PPh_3)_2$ (**4**) is formed in relatively high yield, apparently from oxidative coupling of B_5H_9. The novel structure of **4** will be discussed as will attempts to couple the $[B_3H_8]^-$ anion analogously to the reported coupling of "B_3H_7" by Brellochs and Binder.[1]

1. B. Brellochs and H. Binder, *Angew. Chem*. (1988) 100, 270.

CONVERSION OF ALKYLBORONIC ESTERS TO ALKYLDICHLOROBORANES

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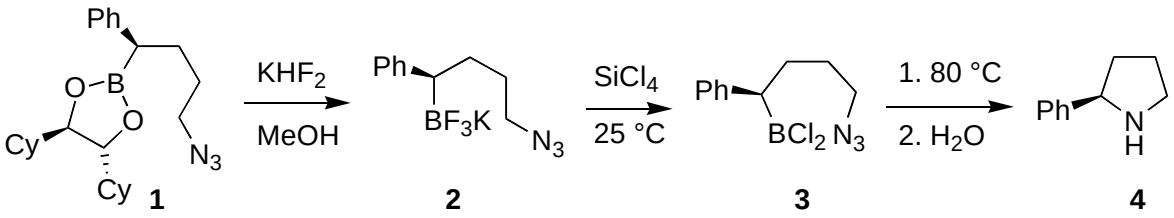
The utility of boronic esters in asymmetric synthesis is well established,[1] but some of the useful transformations of trialkylboranes fail because of the low reactivity of the boronic ester group. Vedejs' conversion of arylboronic acids via trifluoroborate salts to aryldifluoroboranes[2] led us to a useful conversion of asymmetric azido boronic esters such as **1** to the corresponding enantiopure secondary amine (**4**).[3] The reaction of the boron function with azides proceeded much better when the trifluoroborate salt was treated with $SiCl_4$ than with Me_3SiCl.



1 **2** **3** **4**

We have now found that the reason for this result is that $SiCl_4$ rapidly converts the trifluoroborate salt (**2**) to alkyldichloroborane (**3**) in the presence of acetonitrile or THF. Reaction of $RBCl_2$ with RN_3 requires heating, and $RBCl_2$ reacts faster and more efficiently than $RBCl_2$.

It may be surprising to chemists conditioned to think of fluoride as bonding extremely strongly to silicon, but the conversion of BF_3 and $SiCl_4$ to BCl_3 and SiF_4 is endothermic in the gas phase, $\Delta H°_{298}$ = +6.2 k*J*-mol^{-1} per bond; $\Delta G°_{298}$ = +5.4 k*J*-mol^{-1} per bond.[5] From our recent ^{11}B NMR data, it appears that one of the deciding factors in the reaction of RBF_2 with $SiCl_4$ is that acetonitrile or THF complexes more strongly with $RBCl_2$ than with RBF_2, and a second important factor is the rapid escape of gaseous SiF_4 from the reaction mixture.

The new chemistry provides solvated asymmetric $RBCl_2$ under mild conditions. Additional properties and potential applications of $RBCl_2$ and RBF_2 will be discussed.

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METAL-CARBONYL PROMOTED DIRECT SYNTHESIS OF NOVEL DECAMETALLABORANE SYSTEM: *EXO-NIDO*-(η^5-$C_5Me_5Ru)_2B_{10}H_{16}$ AND *NIDO*-(η^5-$C_5Me_5Ru)_2B_{10}H_{16}$

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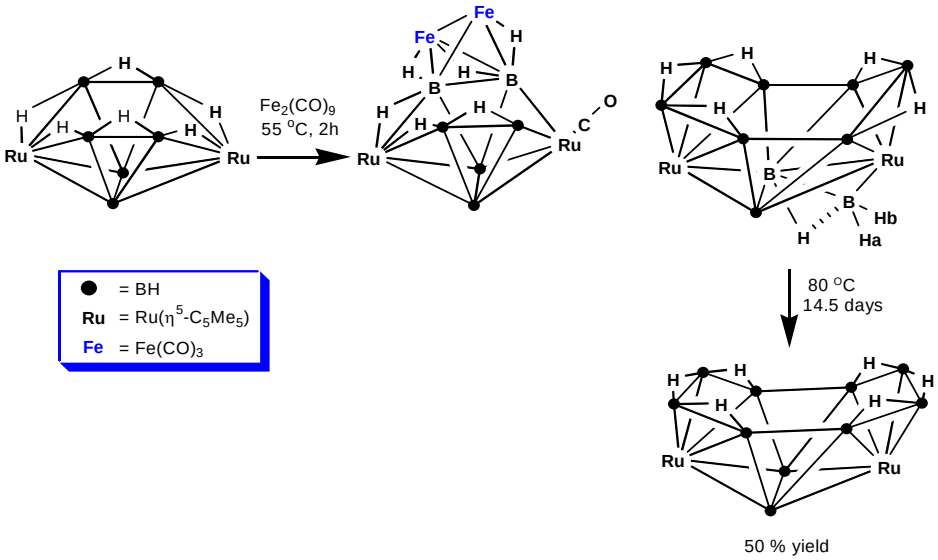
After the discovery of metal promoted fusion of boron containing clusters in 1974, Grimes and co-workers have extensively studied this class of reaction[1] and have synthesized some dimetallacarborane derivatives of $B_{10}H_{12}$ and $B_{12}H_{16;}$ however, no neutral *nido*-dimetallaborane derivatives of $B_{12}H_{16}$ are known. Recently, we have described *arachno*-$(Cp^*Ru)_2B_nH_{12}$[2] (n = 6 and 8) with unusual shape and electron count. We report here two new metallaboranes, derivatives of $B_{12}H_{16}$, which exhibit structural dynamics of a type novel to metallaborane chemistry. Both *exo*-polyhedral metal and borane fragments are involved in metallaborane formation. Mild pyrolysis of *arachno*-$(Cp^*Ru)_2B_6H_{12}$ -with 3 equivalent of $Fe_2(CO)_9$ results in two metallaborane compounds in moderate yield. One of the metallaboranes has been characterized as a metal carbonyl adduct of *arachno*-$(Cp^*Ru)_2B_6H_{12}$ as 5,8-$Fe_2(CO)_6$-(η5-C_5Me_5RuCO)(η5-$C_5Me_5Ru)B_6H_{10}$, and the second light yellow metallaborane has been characterized as *exo-BH*$_2$*-nido*-(η^5-$C_5Me_5Ru)_2B_9H_{14}$. Pyrolysis of *exo-BH*$_2$*-nido*-(η^5-$C_5Me_5Ru)_2B_9H_{14}$ for a prolonged period of time results in colorless symmetrical *nido*-(η^5-$C_5Me_5Ru)_2B_{10}H_{16}$. The X-ray structure analysis and NMR experiments of these two isomeric metallaboranes demonstrates irreversible insertion of the *exo*-polyhedral BH_2 unit into the main framework geometry with a half-life of 14.5 days.



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NMR STUDY AND IN SOLID STATE OF COORDINATING BEHAVIOUR OF 2-SUSTITUTED BENZIMIDAZOLE COMPOUNDS

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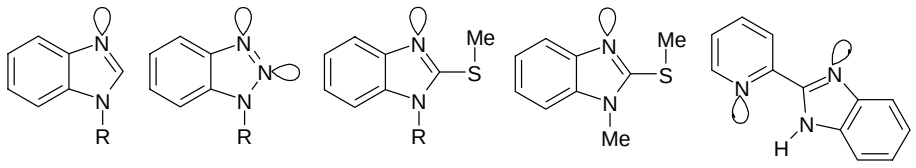
We are studying the chemistry of 2-sustituted benzimidazolic and triazolic compounds. Scheme 1 shows molecules with nitrogen atoms that can coordinate Lewis acids, they have also acidic protons that can be replaced by main group atoms. In this report, we will show the coordinating behaviour of free **1**-**5** and silylated **7**-**8** benzimidazolic compounds with BH_3THF and BF_3OEt_2.



Scheme 1.

As we expect, [1-4] molecules **1**-**4**, **6** and **8** gave N→BX_3 (X = H, F) compounds; whereas compound **7** gave a tetrameric compound similar to that observed by Weiss. [5] (figure 1a), and the reaction of **5** with one or two equivalents of BH_3THF gave a tetracyclic compound with BH_2 and BH_3 groups (figure 1b).

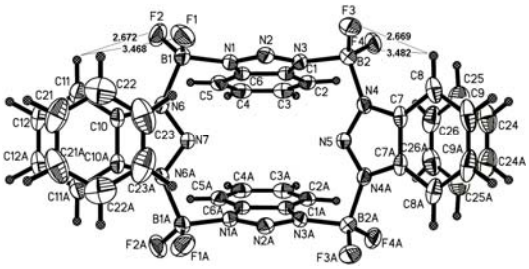


Figure 1a.

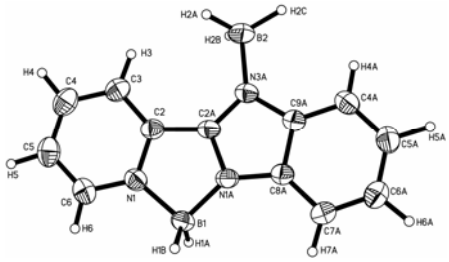


Figure 1b.

In compounds **3**, **4** and **8**, the preferred conformation of S-Me group was established by NMR, specially coupling constants $^{n}J(^{13}C\text{-}^{19}F)$, these results and the analyses of the X-ray structures will be discussed.

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Abstracts of

POSTER

Presentations

SYNTHESIS AND STRUCTURE OF THE NOVEL AND ZERO-VALENT DI-MOLYBDENUM-CARBORANE TRIANION [1,3,6-{MO(CO)$_3$}-3,6-(μ-H)$_2$-1,1,1-(CO)$_3$2-PH-*CLOSO*-1,2-MOCB$_9$H$_7$]$^{3-}$ AND ITS REACTIVITY BY SUBSTITUTION AND OXIDATION

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Treatment of [NEt$_4$][6-Ph-*nido*-6-CB$_9$H$_{11}$] in tetrahydrofuran (THF) with BuLi (2 equiv) followed by 2 equivalents of [Mo(NCMe)$_3$(CO)$_3$] gives the title trianion isolated by addition of [NEt$_4$]I as the salt [NEt$_4$]$_3$[1,3,6-{Mo(CO)$_3$}-3,6-(μ-H)$_2$-1,1,1-(CO)$_3$-2-Ph-*closo*-1,2-MoCB$_9$H$_7$]. In the anion (Figure 1), one {Mo(CO)$_3$} group is bonded exo-polyhedrally to a {*closo*-1,2-MoCB$_9$} cage system by a dative Mo→Mo bond supported by three-center two-electron B—H ⌐ Mo linkages. Salts of the new trianion react readily with several cationic transition metal-ligand fragments with substitution of the exo-polyhedrally bonded, zero-valent {Mo(CO)$_3$} group by a cationic fragment. Species prepared include [NEt$_4$][1,3-{Pt(dppe)}-3-μ-H-1,1,1-(CO)$_3$-2-Ph-*closo*-1,2-MoCB$_9$H$_8$] (dppe = Ph$_2$PCH$_2$CH$_2$PPh$_2$) and [NEt$_4$]$_2$[1,3,6-{Mn(CO)$_3$}-3,6-(μ-H)$_2$-1,1,1-(CO)$_3$-2-Ph-*closo*-1,2-MoCB$_9$H$_7$]. Reaction of the trianion with excess CNBu^t and Ag[PF$_6$] (4 equiv) in MeCN gives the zwitterionic monomeric Mo^{II} complex, [1,1,1,1,3-(CNBu^t)$_5$-2-Ph-*closo*-1,2-MoCB$_9$H$_8$]. The title trianion reacts with HBF$_4$·OEt$_2$ (2 equiv) in the presence of low pressure CO and [NEt$_4$]I with oxidation of the metal center and formation of an unusual cage expansion species, [NEt$_4$][2,2,2-(CO)$_3$-2-I-1-Ph-8-OH-*closo*-2,1,8-MoC$_2$B$_9$H$_9$]. The structures of all these species were confirmed by X-ray diffraction experiments.

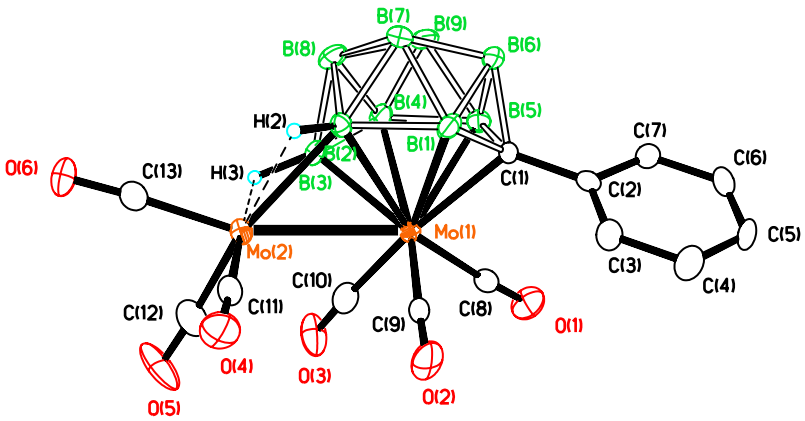

Figure 1. Molecular structure of the trianion [1,3,6-{Mo(CO)$_3$}-3,6-(μ-H)$_2$-1,1,1-(CO)$_3$-2-Ph-*closo*-1,2-MoCB$_9$H$_7$]$^{3-}$ with thermal ellipsoids drawn at 45% probability.

PRELMINARY INVESTIGATIONS INTO THE PHOTOPHYSICS AND ELECTROCHEMISTRY OF [3,3-$(CO)_2$-3-NO-*closo*-3,1,2-$ReC_2B_9H_{11}$]

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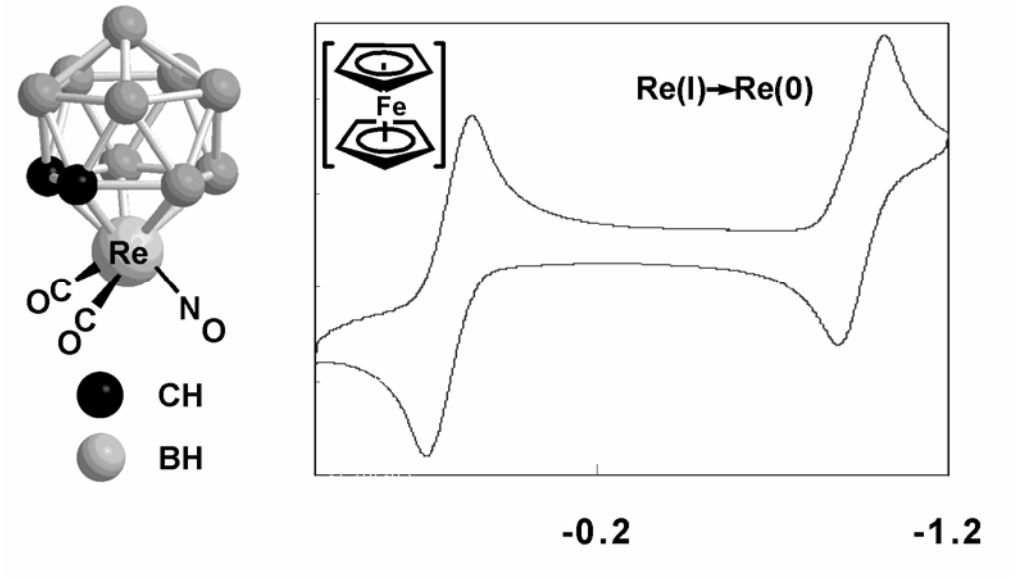
Dept. of Chemistry, Saint Louis University, 3501 Laclede Ave., St. Louis MO 63103

The nitrosyl rhenacarborane complex, [3,3-$(CO)_2$-3-NO-*closo*-3,1,2-$ReC_2B_9H_{11}$], has been reported to undergo carbonyl substitution reactions with stronger σ-donors, such as phosphines and isonitriles. The parent complex, however, has lately displayed some interesting photophysical and electrochemical characteristics. UV-vis spectroscopic measurements in photolyzed methyl-THF glass at –90 ºC had revealed unexpected dynamic behavior of the nitrosyl ligand. More recently, cyclic voltammetry has yielded a reversible one-electron reduction, in contrast to the precursor complex anion, [3,3,3-$(CO)_3$-*closo*-3,1,2-$ReC_2B_9H_{11}$]$^-$, which undergoes an irreversible one-electron oxidation. This redox process is apparently tied to a rapid, reversible chemical reaction, the nature of which is being investigated and will be discussed. Furthermore, spectroscopic measurements of MeTHF glass at 77 K have shown long-lived (290 μs) emission of blue (450 nm) light. This is again in contrast to the complex, [3,3,3-$(CO)_3$-*closo*-3,1,2-$ReC_2B_9H_{11}$]$^-$, which is completely non-emissive.



NEW BORON HETEROCYCLES DERIVED FROM BENZIMIDAZOLE, BENZOTRIAZOLE AND BENZOTHIAZOLE

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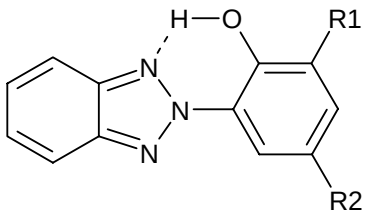
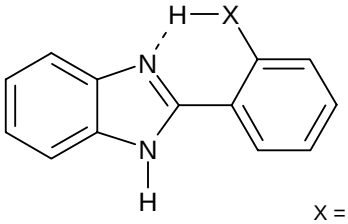
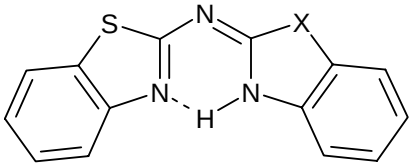
Our research group has been interested in studying aromatic heterocycles of biological interest. Several main group compounds, derived from benzimidazole and benzothiazole, have been reported[1-3]. These polyfuntional molecules bear nitrogen, oxygen and sulphur atoms as basic sites for coordination and acidic protons, which can be substituted by Lewis acids.

We have prepared new boron heterocycles from BH_3, $B(NMe_2)_3$ and BPh_2OH. In these compounds the boron atoms form an electronically delocalized six membered ring. All compounds were characterized by NMR studies and some of them by x-ray diffraction studies.



R1 = 1,1,3,3-tetramethylbutyl, R2 = H
R1, R2 = 1-methyl-1-phenylethyl





X = NH, O, S

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SYNTHESIS OF 1-AMINO-3-[(DIHYDROXYBORYL)ETHYL]-CYCLOBUTANECARBOXYLIC ACID AS A POTENTIAL BORON NEUTRON CAPTURE THERAPY AGENT

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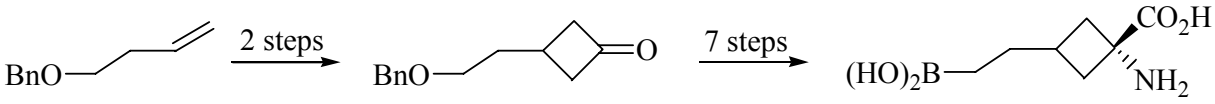
Boron neutron capture therapy (BNCT) is a cancer treatment in which a compound containing boron-10 is selectively delivered to a tumor tissue prior to its irradiation by neutrons.[1] It is believed that amino acids are preferentially taken up by growing tumor cells. Positron emission tomography (PET) investigations carried out at The University of Tennessee using carbon-11 labeled 1-aminocyclobutanecarboxylic acid (ACBC) demonstrated that this amino acid localizes in tumors more avidly than 4-dihydroxyborylphenylalanine (BPA).[2]

We have focused our efforts on the synthesis of boronated ACBC derivatives for a number of years. Carborane[3] and phenylboronic acid[4] ACBC derivatives were prepared previously. Two 3-butylboronic and 3-methylboronic acid ACBC derivatives were also synthesized.[5] As part of a structure-activity relationship (SAR) study, we synthesized a novel 3-ethylboronic acid substituted ACBC in 9 steps from but-3-enyloxymethylbenzene. The new agent is currently being evaluated as a BNCT agent.



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DICHLOROBORANE-DIOXANE: AN EXCEPTIONAL REAGENT FOR THE PREPARATION OF ALKENYL- AND ALKYLBORONIC ACIDS

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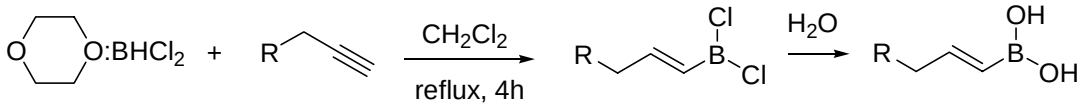
Milwaukee, WI 53233, USA

Terminal alkynes and alkenes were conveniently hydroborated to the corresponding terminal alkenyl- and alkyldichloroboranes using dichloroborane-dioxane in dichloromethane. These dichloroboranes were hydrolyzed by water to the corresponding alkenyl- and alkylboronic acids in moderate to good yields



SYNTHESIS AND DERIVATIZATION OF CHARGE-COMPENSATED TRIRUTHENAMONOCARBORANE COMPLEXES CONTAINING THIOETHERS $[RU_3(CO)_8(\mu_3{:}\eta^5\text{-}7\text{-}L\text{-}CB_{10}H_{10})]$ (L = SME_2, SC_4H_8).

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In extending the studies on triruthenium cluster complexes coordinating to an 11-vertex carborane cage, we have prepared the title compounds (L = SMe_2 (**1**), L = SC_4H_8 (**2**)) by reaction of $[Ru_3(CO)_{12}]$ with *nido*-7-L-$CB_{10}H_{12}$ in refluxing toluene to further explore their potentially interesting derivative chemistry. Reactions of **1** with PR_3 (R = Me, Cy, Ph) afford initial substitution products $[Ru_3(PR_3)(CO)_7(\mu_3{:}\eta^5\text{-}7\text{-}SMe_2\text{-}CB_{10}H_{10})]$ that subsequently yield $[Ru_3(PR_3)(CO)_7(\mu\text{-}H)(\mu_3\text{-}\sigma{:}\eta^5\text{-}7\text{-}SMe_2\text{-}CB_{10}H_9)]$, where a B-H bond adds across the triruthenium cluster. Reactions of **1** and **2** with diphosphines result in 4 different product types dependent on the diphosphine used. Demethylation of $[Ru_3(\mu\text{-}dppm)(CO)_6(\mu_3{:}\eta^5\text{-}7\text{-}SMe_2\text{-}CB_{10}H_{10})]$ (**3**) with excess $[N^nBu_4]F$ in refluxing THF yields $[N^nBu_4][Ru_3(\mu\text{-}dppm)(CO)_6(\mu_3{:}\eta^5\text{-}7\text{-}SMe\text{-}CB_{10}H_{10})]$ (**4**) which can be further derivatized into the neutral complex **5** (Figure 1) by addition of $[AuCl(PPh_3)]$ in the presence of $Tl[PF_6]$.

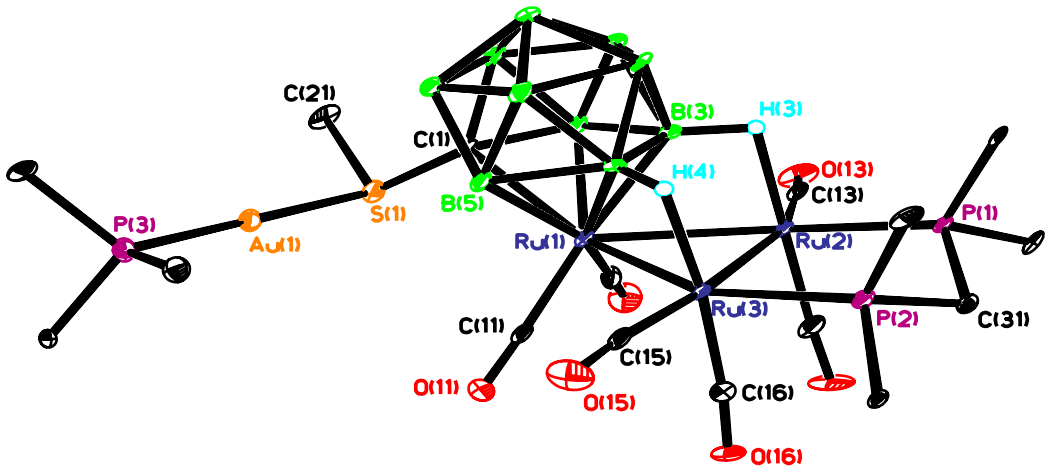

Figure 1. Molecular structure of $[Ru_3(\mu\text{-}dppm)(CO)_6(\mu_3{:}\eta^5\text{-}7\text{-}PPh_3AuSMe\text{-}CB_{10}H_{10})]$ (**5**). Note: Phenyl rings have been omitted for clarity.

SYNTHESIS OF BIMETALLIC METALLOCARBORANES WITH CHROMOPHORIC CHELATING LIGANDS AND THE STUDY OF THEIR SPECTROSCOPIC PROPERTIES.

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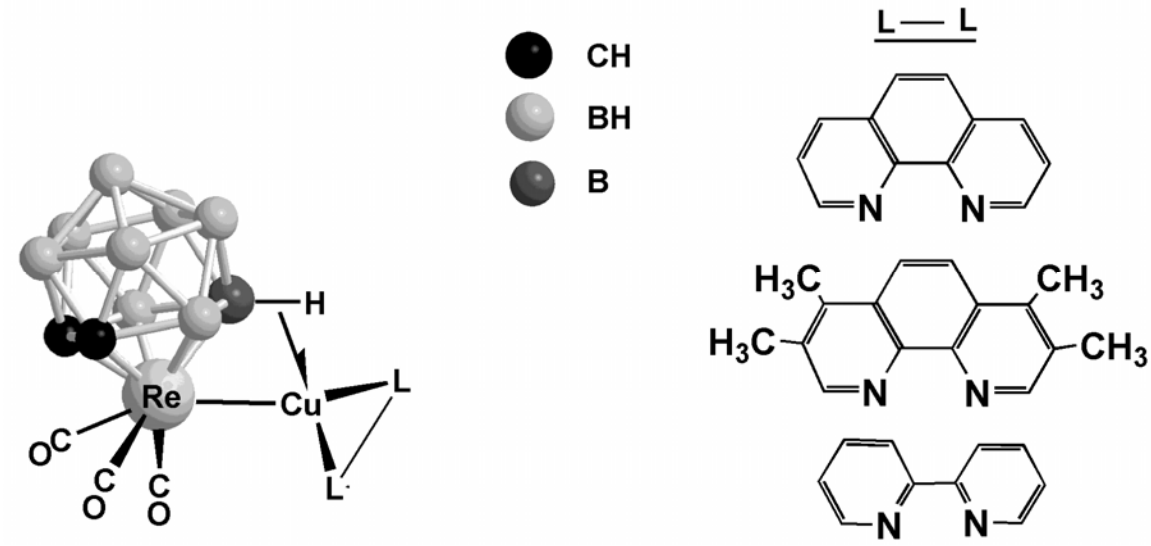
The synthesis of the neutral complex [3,3,3-$(CO)_3$-μ-3,8-{Cu(2,2'-bipyridyl)}-*closo*-3,1,2-$ReC_2B_9H_{11}$] has been accomplished by a similiar synthetic pathway established by Stone *et al*. The carborane cage and bipyridyl ligand are linked via a Re-Cu bond coupled with a B-H→Cu bridge. This complex has been structurally characterized by IR, NMR, UV-VIS, and single crystal X-ray crystallography. In a similar fashion other related complexes have been synthesized, including [3,3,3-$(CO)_3$-μ-3,8-{Cu(1,10-phenanthroline)}-*closo*-3,1,2-$ReC_2B_9H_{11}$], [3,3,3-$(CO)_3$-μ-3,8-{Cu(3,4,7,8-tetramethyl-1,10-phenanthroline)}-*closo*-3,1,2-$ReC_2B_9H_{11}$], and [3,3,3-$(CO)_3$-μ-3,8-{Pt(2,2'-bipyridyl)}-*closo*-3,1,2-$ReC_2B_9H_{11}]^+[BF_4]^-$. These compounds have been studied using low temperature fluorescence spectroscopy and potentiometry in order to elucidate their optoelectronic properties.



BORON-PHOSPHORUS AND BORON-NITROGEN ADDUCTS $BX_3 \cdot E(R)_3$, (E=P, N; X=CL, F OR H) DERIVED FROM O-METHOXYPHENYLPHOSPHINES AND O-METHOXYPHENYLAMINE

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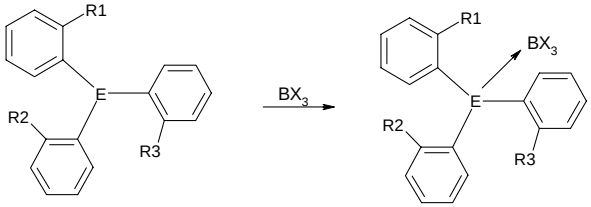
o-Methoxyphenylphosphines are an important group of multidentate ligands which forms coordination compounds with metals due the presence of oxygen and phosphorus atoms [1, 2]. The chemistry of boron-phosphorus adducts has been less studied than boron-nitrogen adducs [3]. In our research group, we are interested in understand the nature of bonds like $P \rightarrow BX_3$, $N \rightarrow BX_3$ and to evaluate weak interactions, steric effects and the changes in the electronic distribution using ^{1}H, ^{13}C, ^{31}P, ^{11}B, ^{19}F and NMR data [4–7].

We have isolated the 1**:**1 Lewis acid-base adducts $BX_3 \cdot E(R)_3$, (E = P, N; X = Cl, F or H) derived from o-methoxyphenylphosphines and tri-(o-methoxyphenyl)amine. They were characterized by multinuclear NMR spectroscopy, infrared, mass spectrometry and elemental analysis.



1 E = P
R1 = -OCH_3, R2 and R3 = H
2 E = P
R1 and R2 = -OCH_3, R3 = H
3 E = P and **4** = N
R1, R2 and R3 = - OCH_3

X = Cl, F or H

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POLYBORANE REACTIONS IN IONIC LIQUIDS. A NEW EFFICIENT ROUTE TO *o*-CARBORANES

Yuqi Li,[1,*] Mark G. Bradley,[2] Patrick J. Carroll[1] and Larry G. Sneddon[1]

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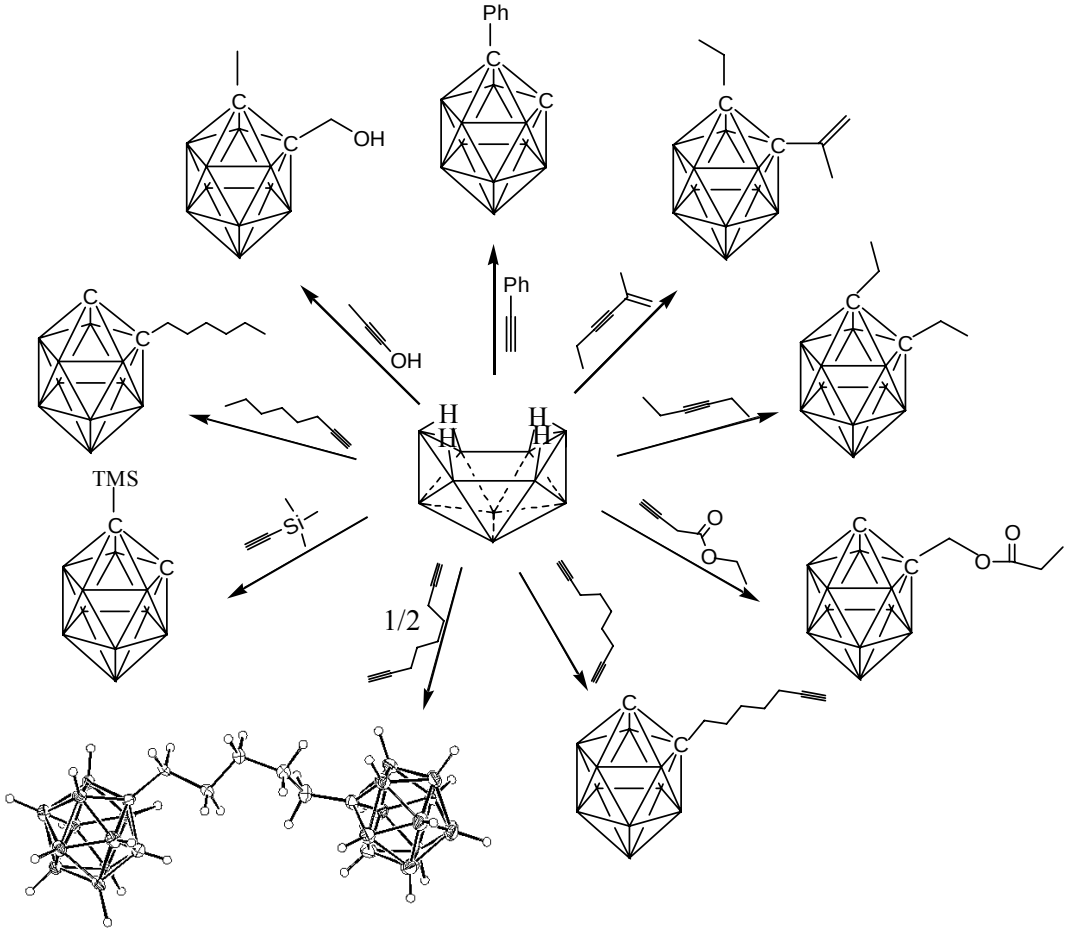
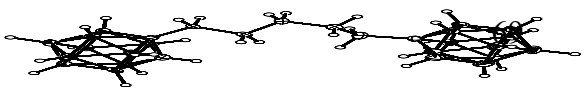
[2] Widener University, Chester, PA 19013-5792

Ionic liquids (IL) are attractive substitutes for conventional solvents for organic and inorganic synthesis. We have now found that decaborane alkyne insertions occur in ionic liquids with a wide range of alkynes to produce excellent yields of 1,2-$R_2C_2B_{10}H_{10}$ products without the need of the base catalyst used in conventional reactions. Reactions are carried out under biphasic conditions with reaction times typically ranging from only a few minutes to one hour. The ionic liquid route provides a new, one-step, high yield route to functionalized *ortho*-carboranes.





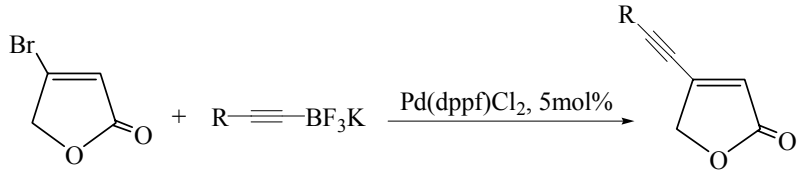
SYNTHESES OF 4-(1-ALKYNYL)-2(5H)-FURANONES AND COUMARINS VIA THE PALLADIUM-CATALYZED CROSS-COUPLING REACTIONS OF POTASSIUM ALKYNYLTRIFLUOROBORATES

G. Dong,* B. Venkataiah, and G. W. Kabalka

Department of Chemistry and Radiology, The University of Tennessee, Knoxville, TN 37996

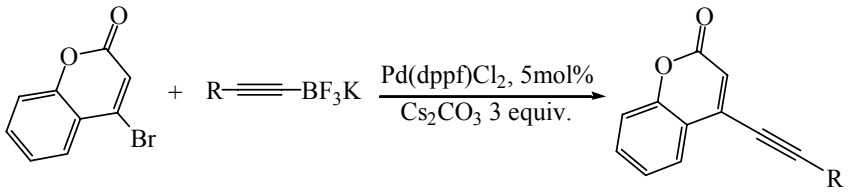
Recently, 4-(1-alkynyl)-substituted 2(5H)-furanones were found to exhibit potent cytotoxicity.[1] However, the methods for synthesizing of 4-alkynyl 2(5H)-furanones are limited.[2] Due to their ready availability and chemical stability, potassium organotrifluoroborates have attracted increasing attention in organic chemistry.[3] In connection with our ongoing studies of the cross-coupling reactions of potassium organotrifluoroborates,[4] we developed an efficient synthesis of 4-alkynyl substituted 2(5H)-furanones using potassium alkynyltrifluoroborates in the presence of $Pd(dppf)Cl_2$. The reactions proceed smoothly in the absence of base and provide excellent yields of the desired products. (Figure 1).

Figure 1.



The new method was applied to the synthesis of 4-alkynylcoumarins. Interestingly, these reactions require base. The reactions of 4-bromocoumarin with various potassium alkynyl-trifluoroborates were studied and the yields are excellent (Figure 2).

Figure 2.



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BORATES AS SOLID STATE MATERIALS PRECURSORS

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Dept. of Chemistry, University of New Mexico, Albuquerque, NM
US Borax, Inc., Valencia, CA

Solid state compounds containing boron play many important roles in modern materials science and new approaches for the synthesis of solid state boron materials are sought. Boric acid, for example is used in modest quantities to prepare the industrial ceramic boron nitride. Recently, our group has developed aerosol methods for the preparation of hexagonal BN having a unique spherical particle morphology. This presentation will summarize our recent efforts to obtain these materials from $(MeO)_3B$ and from aqueous solutions containing a variety of ammonium and quanidinium borate salts.

RUTHENIUM CARBENE CATALYZED CROSS METATHESIS (CM) AND RING CLOSING METATHESIS (RCM) OF DECABORANE AND O-CARBORANE BASED OLEFINES

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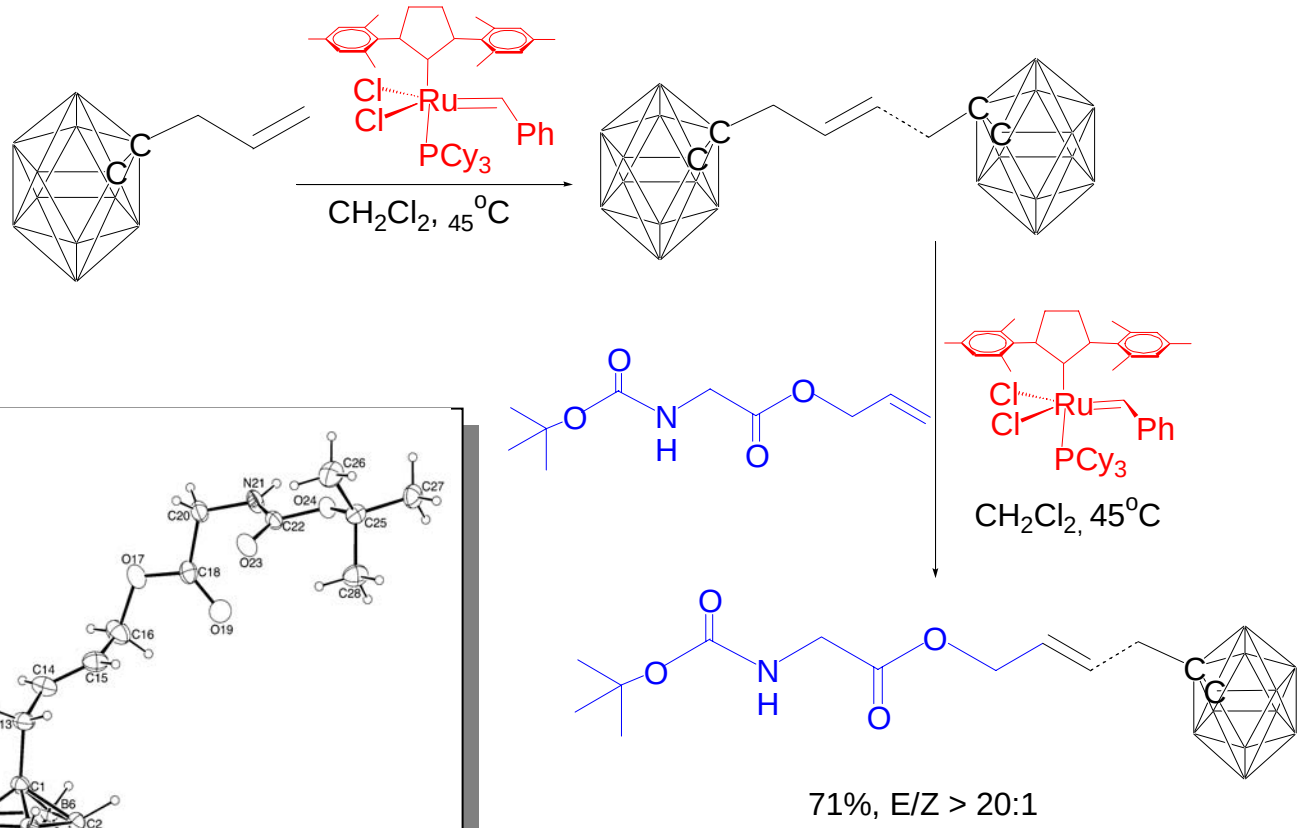
Alkenylborane metathesis reactions catalyzed by Grubbs-type ruthenium carbene catalysts provide efficient routes for the synthesis of organopolyboranes. Both 6-hexenyldecaborane and allyl-*o*-carborane undergo catalyzed homemetathesis to form olefin-bridged polyboranes. Cross metathesis (CM) with a variety of olefins provide routes to functionalized polyboranes, including potential BNCT drugs. Dialkenylpolyboranes have also been found to readily undergo catalyzed ring closing metathesis (RCM).



BORON OXIDE FILLED POLYCARBONATE FOR FIRE RESISTANT APPLICATIONS

Suresh Murugesan, Chano Morin and Sreenuvasalu R. Venumbaka*

Institute of Environmental and Industrial Sciences
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San Marcos, TX 78666

Abstract

Flammability of the materials is an important property, which limits the usage of organic polymers in many Industrial applications. In order to improve the flame resistance of these types of materials many different types of additives are added to them of which most of them are halogenated compounds. In this study, boron oxide is *in-situ* generated from an alkoxide in 4X4 plaques of the polycarbonate using sol-gel procedure. Then these composites were tested for fire resistant property by measuring peak heat release rate (PHRR), weight loss, time to ignition. Also mechanical properties of these composites were studied. Preliminary results reveal that composites fire resistant property was improved 30-50% depending on the amount of the filler. The toughness of the composites has increased compared to the unfilled PC plaques.

THE SYNTHESIS, BIOCONJUGATION AND PRELIMINARY BIODISTRIBUTION STUDIES OF Tc-METALLOCARBORANES

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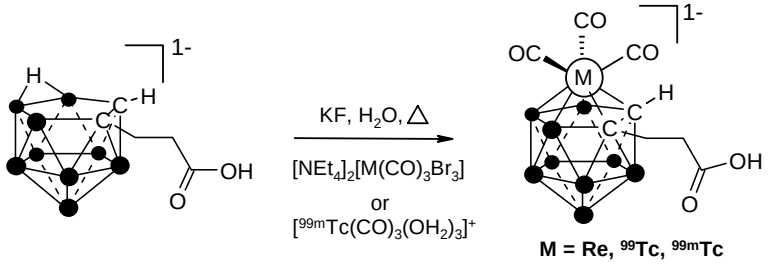
[2]*Center for Radiopharmaceutical Science of the ETH Zürich, Paul Scherer Institute Villigen and the University Hospital Zürich, CH-5232 Villigen, Switzerland*

We recently described a new method for the synthesis of Tc and Re metallocarborane complexes in water.[1] The synthetic methodology involves reacting *nido*-carborane ligands with $[M(CO)_3(OH_2)_3]^+$ (M = Tc, Re) in the presence of fluoride. The resulting carborane complexes have a number of features that make them attractive synthons for designing targeted radiopharmaceuticals. These include inertness, compact size and ease of functionalization.

We have recently begun developing a new series of carborane ligands and carborane-biomolecule conjugates that are designed to deliver Tc-to specific receptor sites. These include carbohydrate, alkylamino, pyridinium and peptide-carborane derivatives. The synthesis and characterization of these metallocarborane derivatives will be discussed and along with some preliminary biodistribution data.



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REACTION OF $HCB_{11}Me_{11}^{\square}$ AND $CB_{11}Me_{12}^{\square}$ RADICALS WITH DISILANES

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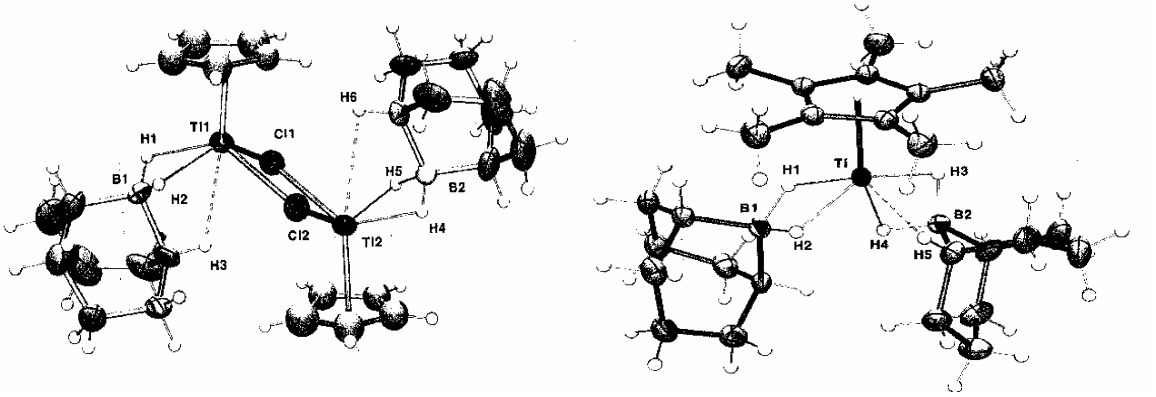
Solutions of the carboranyl radicals $HCB_{11}Me_{11}^{\square}$ and $CB_{11}Me_{12}^{\square}$ in pentane react with Si_2Me_6 at room temperature or with Si_2-*t*-Bu_6 even at -100 °C to form $SiMe_4$ or MeSi-*t*-Bu_3 and boron containing products which are stable below -60 °C but can apparently be trapped even at higher temperatures. With nucleophiles such as benzene, bromobenzene, phenylacetylene, and *n*-butyllithium, these materials yield 12-substituted methylated CB_{11}^{-} anion products and thus behave as if their structure were that of the boronium ylides $HCB_{11}Me_{10}$ or $CB_{11}Me_{11,}$ with no ligand in position 12.

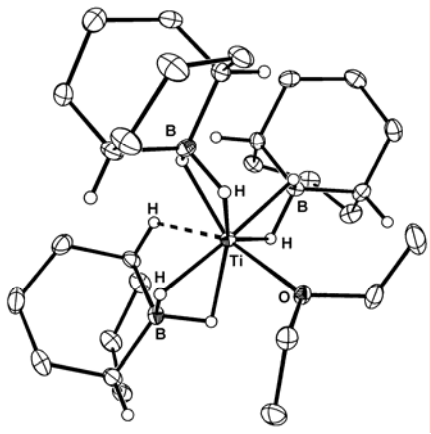
METALLA-ORGANOHYDROBORATES AS POTENTIAL PRECURSORS FOR THE CATALYSIS OF OLEFIN POLYMERIZATION

Jungsu Park, Errun Ding, Xeunian Chen, Fabrice Lacroix, Fiu-Chen Liu, Shengming Liu, Xianlong Ge, William Starnes, Jr. Sheldon Shore*

Department of Chemistry, The Ohio State University, Columbus, 43210

Several titanium and lanthanide-organohydroborates have been prepared, some of these are under investigation as precursors for catalysis of olefin polymerization. Some examples of complexes prepared are shown below and examples of polymerization reactions will be presented.





BORATE DERIVATIVES OF DIAMINES

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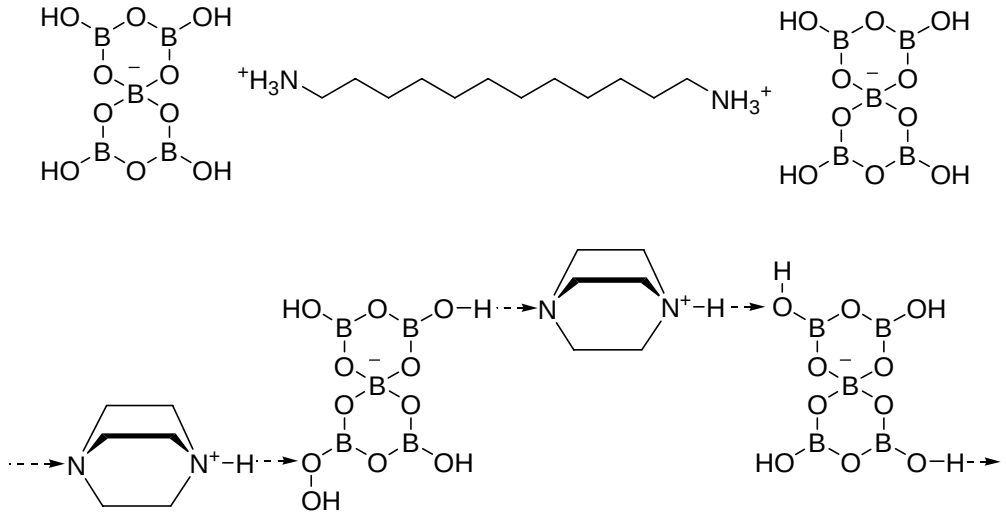
(2) McCullough Crystallography Laboratory, University of California, Los Angeles, USA 90095-1569

Boron-oxygen compounds have vastly greater technological importance than any other class of boron compounds. Aside from boric acid and boric oxide, the majority of these compounds contains interstitial metal cations that serve as Lewis acid acceptors for oxygen of anionic borate structural units. Borate compounds containing non-metal cations have been studied to a far lesser extent. In contrast to metals, non-metal cations generally do not coordinate oxygen and may act as hydrogen bond donors to, or acceptors from, borate structural units. Syntheses and structural characterizations of crystalline borates containing non-metal cations derived from various diamines are described. In some cases, non-metal cations may direct the formation of unusual borate structures, as in imidazolium nonaborate, $[C_3N_2H_5]^+_3\,[B_9O_{12}(OH)_6]^{3-}$. In other cases, borate anions form extended H-bonded networks that direct cation conformation, as in a series of novel pentaborates containing $[H_3N(CH_2)_nNH_3]^{2+}$ cations (n = 5-12) that alternatively arrange linearly along channels or fill pockets with disordered alkane moieties. The 1,12-diaminododocane bis(pentaborate), ignoring the important anion-anion and cation-anion H-bonds, is illustrated below. Also pictured is the pentaborate derivative of the $[N(CH_2CH_2)_3NH]^+$ cation, which crystallizes in the asymmetric space group Cc and exhibits highly directional cation-anion as well as anion-cation H-bonding. The anion-anion H-bonding interactions are not included in this picture.



RADIOLABELED ANABASEINE DERIVATIVES FOR USE IN THE EARLY DETECTION OF LUNG CANCER

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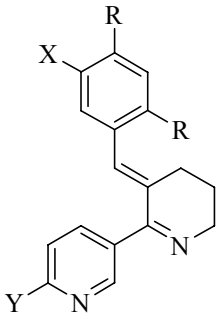
Departments of Chemistry and Radiology, The University of Tennessee
Knoxville, TN 37996

The use of single photon emission tomography (SPECT) and positron emission tomography (PET) in Nuclear Medicine has resulted in the need for a variety of physiologically active compounds labeled with short-lived radioisotopes. Researchers at he University of Tennessee have found that small cell lung carcinoma express an α_7-bungarotoxin (α_7-BTX) sensitive neuronal nicotinic acetylcholine receptor comprised of α_7 subunits (α_7-nAChR) which control cell growth.[1] Anabaseine and derivatives such as dimethoxybenzylideneanabaseine have been demonstrated to have a high affinity for this receptor.[2] A radiolabeled derivative of anabaseine could thus be valuable for the early detection of lung cancer using PET and SPECT.

Iodine-125 with a half-life of 60 days was used in the initial synthesis of a radiolabeled anabaseine derivative. The chemistry is readily transferable to iodine-123 which is the isotope of choice for SPECT as well as iodine-124, a positron emitting radionuclide.



	R	X	Y
a	CH_3	^{123}I	H
b	CH_3	H	^{123}I
c	OCH_3	^{123}I	H
d	OCH_3	H	^{123}I

References:

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A TRIBUTE TO THE LIFE OF ROY M. ADAMS

Bernard F. Spielvogel* and David Badger#

*BoroScience Canada, 101 Research Drive, Dartmouth, Nova Scotia, B2Y 4T6, Canada
Department of Chemistry, Geneva College, Beaver Falls, PA, 15010

Professor Adams was an outstanding dedicated undergraduate educator, spending 39 years as a full time faculty member at Geneva College. Although retiring from Geneva in 1985, he continued to teach there until his death on March 26, 2003 at the age of 83. In addition to his academic duties, Dr Adams served as a consultant to nearby Callery Chemical Company and was intimately involved in the high energy boron fuels effort. He also consulted with Battelle Memorial Institute and the Midwest Research Institute. For more than 35 years, he dedicated his spare time to developing standards for chemical nomenclature and is now recognised as being largely responsible for the 1961 IUPAC rules on boron nomenclature. He co-authored “Boron, Metallo-Boron Compounds and Boranes”, published in 1964 and which even to this day serves as one the most useful books on boron science. Professor Adam`s contributions to boron chemistry will be presented.

BORONATED ANALOGUES OF COX II INHIBITORS AS POTENTIAL BNCT AGENTS

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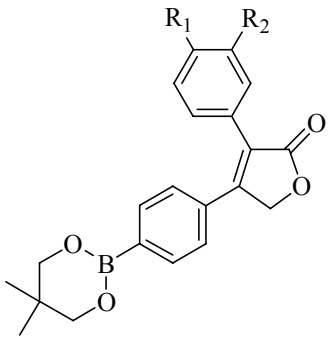
Departments of Chemistry and Radiology, The University of Tennessee
Knoxville, TN, 37996

Boron neutron capture therapy (BNCT) is a cancer treatment in which boron-10 nuclei are irradiated with thermal neutrons to generate high linear energy transfer particles. Many classes of compounds have been developed as potential BNCT agents; these include boronated carbohydrates, porphyrins, amino acids, and other biomolecules.[1] Cyclooxygenase (COX) enzyme inhibitors have been of interest as potential BNCT agents in our laboratory for a number of years because of their affinity for inflammatory processes.

3,4-Diarylfuranones are potent COX-2 inhibitors.[2,3] For this reason we are interested in the structure activity relationship (SAR) and chemistry of these compounds. We have synthesized a variety of functionally substituted diarylfuranone derivatives. The boronic esters of the diarylfuranones are readily prepared by Suzuki-Miyaura chemistry using *bis*(neopentylglycolato)diboron and a palladium catalyst. The synthesis of these diarylfuranone boronic esters will be described.



where R_1, R_2 = H, Br, Cl, Me

References:

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A FACIE REACTION OF α-BROMO-(*Z*)-1-ALKENYLBORONATE ESTERS WITH TRIMETHYLGERMYLLITHIUM. NEW ACCESS TO ALKYL TRIMETHYLGERMYL KETONES

Narayan G. Bhat* and Vanessa L. Perez

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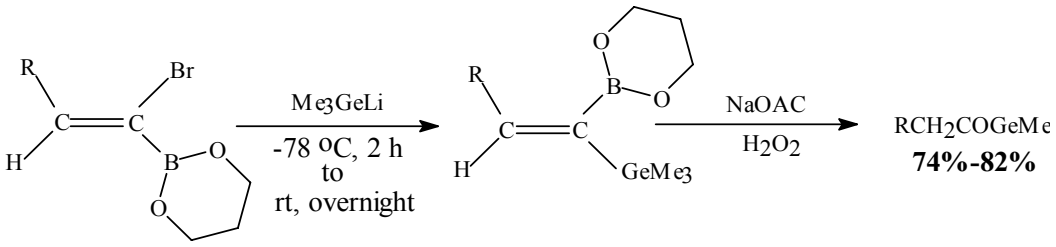
A convenient, novel synthesis of (*E*)-*gem*-dimetalloakenes containing boron and germanium from the Z-1-bromo-1-alkenylboronate esters is developed. α-Bromo-(Z)-1-alkenylboronate esters readily prepared from the literature procedures smoothly undergo a reaction with freshly generated trimethylgermyllithium in hexamethylphosphoramide (HMPA) at -78 ^{o}C to provide the corresponding "ate" complexes. These "ate" complexes undergo intramolecular nucleophilic substitution reactions to provide the corresponding (*E*)-1-alkenylboronate esters containing trimethylgermyl moiety. These intermediates are oxidized with hydrogen peroxide and sodium acetate to provide the corresponding alkyl trimethylgermyl ketones in good isolated yields (74%-82%) and they are characterized by the spectral data (PMR and CMR).



PREPARATION OF CARBORANE-CONTAINING CHOLESTEROL DERIVATIVES FOR POTENTIAL APPLICATION IN BNCT

Jacqueline Smits,* Colby C. Tate, and Debra A. Feakes

Department of Chemistry and Biochemistry, Texas State University – San Marcos, San Marcos, TX 78666

Therapeutically useful concentrations of boron can be delivered to the interior of tumor cells using unilamellar liposomes of a specific size and composition, thus providing a potential tumor specific delivery vehicle for application in boron neutron capture therapy. A wide variety of boron-containing compounds can be incorporated into the liposomes. Hydrophilic boron-containing compounds can be encapsulated in the aqueous core of the liposomes while lipophilic boron-containing compounds can be embedded in the lipophilic bilayer. Although the majority of compounds investigated to date have been water-soluble derivatives of polyhedral borane anions, the use of lipophilic compounds provides several advantages. Lipophilic compounds are incorporated to a much higher degree than the hydrophilic compounds and provide a means of enhancing the delivered dose without increasing the osmotic stress on the bilayer. The standard lipid bilayer utilized in prior experiments has been composed of a 1:1 molar ratio of cholesterol to phospholipid (typically distearoylphosphatidylcholine, DSPC). The incorporation of carborane-containing derivatives of cholesterol should enhance the delivered dose of boron while minimizing the deleterious effects to the bilayer stability. A series of carborane-containing derivatives have been reported by our group; however, the original synthesis required many steps and resulted in relatively low overall yields of the carborane derivative. More recently, we have investigated an alternative synthetic methodology, the optimization of which will be presented in this poster.

SYNTHESES AND PROPERTIES OF HALF-SANDWICH METALLATRICARBADECABORANYL COMPLEXES

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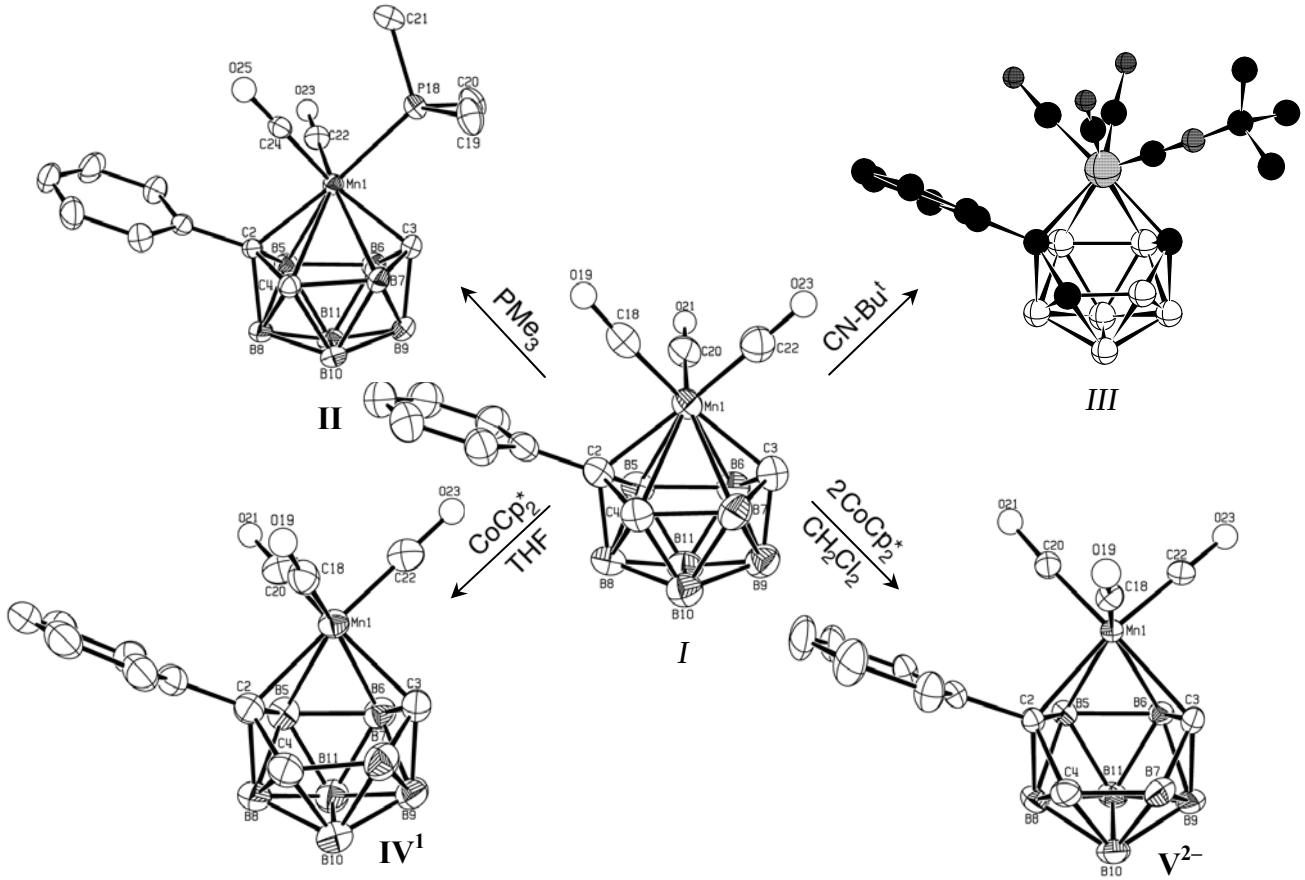
The tricarbadecaboranyl ligand 6-Ph-*nido*-5,6,9-$C_3B_7H_9^-$ has been used to synthesize a variety of half-sandwich metal complexes. Reaction of 1,1,1-$(CO)_3$-2-Ph-*closo*-1,2,3,4-$MnC_3B_7H_9$ (**I**) with a strong lewis base (e.g. PMe_3) yields the monosubstituted dicarbonyl complex 1,1-$(CO)_2$-1-PMe_3-2-Ph-*closo*-1,2,3,4-$MnC_3B_7H_9$ (**II**), while reaction of **I** with a weaker lewis base (e.g. $CNBu^t$) yields the ring-slipped, monosubstituted tricarbonyl complex 1,1,1-$(CO)_3$-1-$CNBu^t$-2-Ph-*nido*-1,2,3,4-$MnC_3B_7H_9$ (**III**). Depending on reaction stoichiometry and solvent, the reaction of $CoCp^*_2$ with **I** yields either mono- ($\mathbf{IV^{1-}}$) or di-anionic ($\mathbf{V^{2-}}$) metallatricarbadecaboranyl complexes. The syntheses and properties of these metal complexes will be presented.

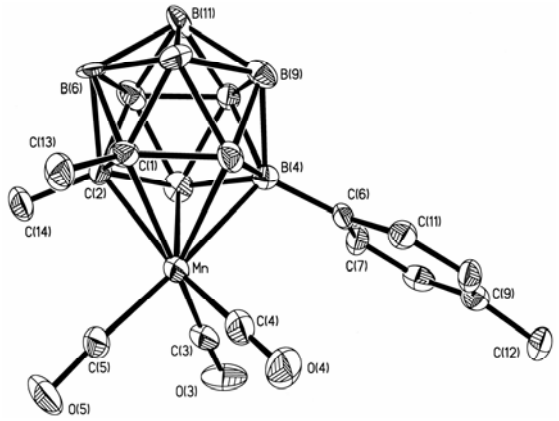


REACTIVITY OF [PPN][3,3,3-(CO)$_3$-1,2-Me$_2$-*closo*-3,1,2-MnC$_2$B$_9$H$_9$] AND ITS DERIVATIVES WITH ELECTROPHILES

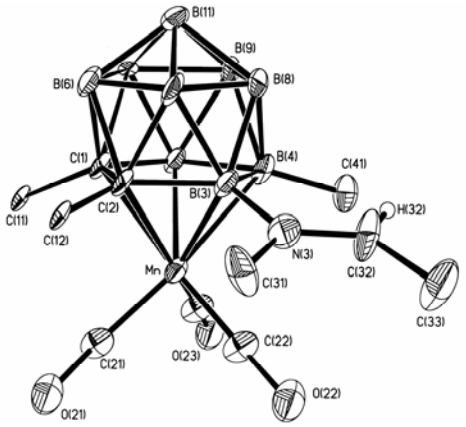
Mitsuhiro Hata, Jason A. Kautz, Xiu Lian Lu,* Thomas D. McGrath, and F. Gordon A. Stone

Department of Chemistry and Biochemistry, Baylor University, Waco, Texas 76798-7348

Although Cs[3,3,3-(CO)$_3$-*closo*-3,1,2-MnC$_2$B$_9$H$_{11}$] was firstly reported by Hawthorne and co-workers many years ago,[1,2] its chemistry has scarcely been studied. In our group, it has proved possible to abstract hydride from a carborane boron vertex by reaction with certain electrophiles. Studies have shown that reaction of [PPN][3,3,3-(CO)$_3$-1,2-Me$_2$-*closo*-3,1,2-MnC$_2$B$_9$H$_9$] (**1**) with I$_2$ afforded [PPN][8-I-3,3,3-(CO)$_3$-1,2-Me$_2$-*closo*-3,1,2-MnC$_2$B$_9$H$_8$] (**2**), of which the latter further reacted with Grignard reagents RMgBr in the presence of catalytic [PdCl$_2$(PPh$_3$)$_2$] to give [PPN][8-R-3,3,3-(CO)$_3$-1,2-Me$_2$-*closo*-3,1,2-MnC$_2$B$_9$H$_8$] (R = Me, **3**; R = *p*-C$_6$H$_4$Me, **4**). Treatment of **3** with I$_2$ afforded the further substituted complex [PPN][4-I-3,3,3-(CO)$_3$-1,2,8-Me$_3$-*closo*-3,1,2-MnC$_2$B$_9$H$_7$] (**5**), whereas **3** with MeOTf in CH$_2$Cl$_2$–MeCN gave neutral zwitterionic [4-{(*Z*)-N(Me)=C(H)Me}-3,3,3-(CO)$_3$-1,2,8-Me$_3$-*closo*-3,1,2-MnC$_2$B$_9$H$_7$] (**6**). The structures of **4** and **6** were determined by X-ray diffraction analysis (shown below).



Molecular structure of the anion of **4**



Molecular structure of **6**

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SYNTHESIS OF A POLYALKENE CARRYING MULTIPLE 2, 2, 2-TRIFLUOROETHYLATED ICOSAHEDRAL CARBORATE ANIONS

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We find that the carbenoid derived from $CF_3CH_2N_2$ and a metal catalyst inserts CF_3CH_2 groups into BH bonds of the $CB_{11}H_{12}(-)$ anion. This reaction is the starting point for the synthesis of a partially fluorinated ionomer reminiscent of Nafion, but carrying the very bulky $CB_{11}H_n(CH_2CF_3)_{11-n}(-)$ substituents instead of sulfonate anions. Proton conductivity of the new polymer as a function of temperature is under investigation

EVALUATION OF THE BINDING OF POLYHEDRAL BORANE ANIONS WITH ALBUMINS AND SIMPLE AMINO ACIDS

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Unilamellar liposomes of a specific size and composition have been shown to deliver therapeutic quantities of boron to tumors in biodistribution experiments and, as a result, have potential applications in boron neutron capture therapy. A wide variety of boron-containing compounds can be incorporated into the liposomes. Water-soluble boron-containing compounds can be encapsulated within the aqueous core of the liposomes while water-insoluble boron-containing compounds can be incorporated into the lipophilic bilayer of the liposomes. Although both classifications of compounds have been utilized, the mechanism of uptake and retention of the water-soluble boron-containing compounds has been investigated to the greatest extent. Essentially any water-soluble boron-containing compound can be encapsulated into the core of the liposomes; however, only compounds which possess suitable reactivity are retained within the tumor for any significant period of time.

The $[B_{20}H_{17}SH]^{4-}$ anion has been evaluated and exhibits significant tumor uptake and retention in *in vivo* biodistribution experiments. Several mechanisms of retention can be proposed. In the first proposed mechanism, the thiol substituent has the potential to form disulfide bonds with intracellular protein moieties. In the second proposed mechanism, the anion has the potential to oxidize *in vivo* to the more reactive $[B_{20}H_{17}SH]^{2-}$ anion, capable of both disulfide bond formation and attack by nucleophilic protein substituents. The reaction of the $[B_{20}H_{17}SH]^{4-}$ with albumins has been investigated and the products of the reactions analyzed by HPLC, CZE, and MALDI mass spectrometry. The results indicate that a strong interaction occurs between the anion and the albumins; however, the nature of the binding, in terms of covalent or electrostatic interaction, is not clear. In order to evaluate the potential for covalent binding, the reaction of the $[B_{20}H_{17}SH]^{4-}$ anion with simple amino acids and metabolites has been investigated. The results of these investigations, to date, will be presented.

CARBORANE CONTAINING DENDRIMERS FOR DRUG DELIVERY

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Dendrimers are highly branched, three-dimensional polymers that can be prepared through step-by-step reactions diverging from a central core. Their monodispersity, three-dimensional structure, high molecular weight, and increased functionality significantly improve chemical and physical properties (solubility, viscosity) when compared to linear polymers, or small molecules. In addition, the stepwise synthesis of dendrimers allow for significant control over structure, functionality, and overall molecular size. Consequently, dendrimers are considered to be a leading candidate as building blocks for the construction of nanoscale objects, molecular devices and advanced drug delivery systems, amongst numerous other applications.

Implementation of a dendrimer as a drug delivery system will be the focus of this presentation. It is believed that potent hydrophilic or hydrophobic drugs can be incorporated into the dendrimer structure in such a way that they are masked, or hidden, by the dendrimer periphery, preventing them from being degraded or absorbed at inappropriate times. The ability to tailor the peripheral functional groups of the dendrimer then provides methods for incorporating targeting agents that will send the "packaged" drug molecules to appropriate destinations. Specifically, the heart of this project is to insert carborane clusters into the branches of a water-soluble polyester dendrimer scaffold (Figure 1). The ability to make carborane clusters soluble in an aqueous environment is of interest because of their use in arthritis treatment by way of Boron Neutron Capture Synovectomy (BNCS).

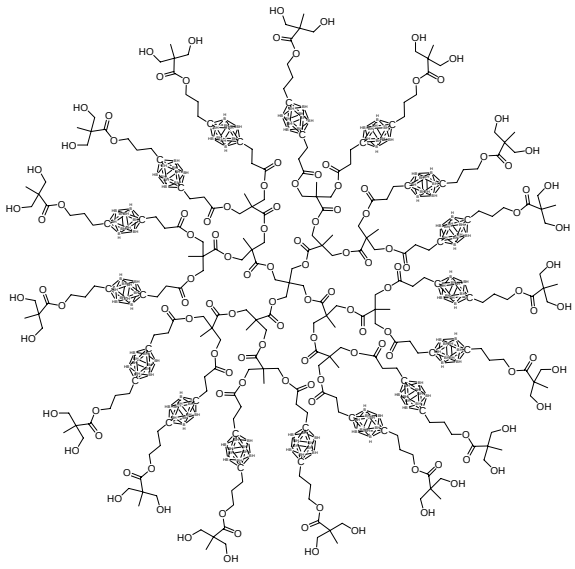

Figure 1. Structure of a carborane-containing aliphatic poly(ester) dendrimer.

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