BORON

ANNUAL SURVEY COVERING THE YEAR 1982
Part II

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CONTENTS

As in the past, this survey reports on organoboron species and related materials containing isolated boron atoms. Carborane and polyborane derivatives as well as hydroboration as a tool in organic chemistry are generally not considered. However, some aspects are included that are not strictly organometallic in nature but nevertheless should be of interest to the reader.

Although the material is grouped into major sections, this guiding principle is not strictly adhered to. Various compounds can be classified differently and chemical relationships occasionally prompted a rearrangement of the discussion sequence.

1 REVIEWS AND SUMMARY ACCOUNTS

A new treatise on organometallic chemistry contains several chapters on relevant aspects of boron compounds (220). Organoboron compounds are the topic of another book (203), and books on the gas chromatography of organometallic compounds (71) and organometallic syntheses (134) include sections on boron chemistry. Also, another volume of the GMELIN handbook describing boron compounds has been published (204).

New reviews deal with haloboranes and their alkyl derivatives (185), the synthesis of alkyl-substituted boron hydrides (192), and the use of organylborates in synthetic reactions (74). Borides and boranes have been surveyed by LIPSCOMB (20), and PELTER has reviewed the carbon-carbon bond formation via boron reagents (231).

2 SELECTED DATA OF GENERAL INTEREST

Primary amines containing ^{15}N are readily synthesized via the reaction of organylboranes with ^{15}N-enriched ammonium hydroxide in the presence of sodium hypochlorite (213).

Boric acid can be extracted with cis-9,10-dihydrostearic acid (25) or with 2-alkyl 1,3-butanediols (233). A new spectrophotometric determination of boron by solvent extraction with hydrobenzoin and crystal violet has been described (174), and a similar method based on pyrocatechol violet has been reported elsewhere (234). For some analytical investigations in the boron-carbon system, see (22). The determination of iron as a macrocyclic complex with nickel oxime and boric acid has been described (186).

Silanol groups of the zeolite HY react with diborane(6) to yield, ultimately, Si-O-BH_2 moieties. At high temperatures, the zeolite lattice is attacked by the BH_2 groups with the formation of Si-O-B=O linkages (68). UV-Visible spectral shifts observed from p-dimesitylboryl-containing dyes in acoholic NaOH solutions are caused by attack of the base on the boron atom to yield the light-sensitive "borate" structure (117).

Oxygen-17 NMR chemical shifts for a variety of organoboron-oxygen heterocycles have been reported (53). The data are discussed in terms of varying B-O π-bonding.

The sorption and desorption of boron in soils has been studied (97), and the effect of excessive doses of boron on soils and plants has been evaluated (230).

3 HYDRIDIC SPECIES

Dimethylsulfide-borane continues to be a fashionable reagent in organic synthesis; see, for example, (178). Interestingly, the reduction rate increases considerably if the dimethyl sulfide is removed from the reaction mixture (168). Pyridine is an effective borane scavenger to avoid overreduction in the conversion of acid chlorides to the corresponding aldehydes employing $NaBH_4$ (227). For the use of thexylborane in organic syntheses, see (209); and for reductions with tetrabutyldiborane(6), see (122). Reactions of arylthallium compounds with diborane(6) have been studied (246); (aryl)hydroxyboranes or phenols are obtained via organoboron intermediates. A detailed study of the hydroboration of alkynes with dialkylboranes has been presented elsewhere (114).

The reaction of THF-BH_3 with phosphorus ylides provides simple access to monoalkylboranes (8); the reaction seems to be particularly advantageous for the preparation of mixed trialkylboranes. Boranes derived from the hydroboration of methylenecyclobutane rearrange on heating in situ at 100 to 160°C to yield open-chain structures (228).

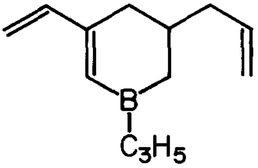
Exchange reactions of triorganylboranes with borane(3) in THF and $(CH_3)_2S$, respectively, have been studied by ^{11}B NMR spectroscopy (190). The molecular structure of $CH_3B_2H_5$ has been determined by microwave spectroscopy (32); and deuterium quadrupole coupling in $OC\text{-}BD_3$ has been studied (218).

4 TRIORGANYLBORANES AND SOME RELATED SPECIES

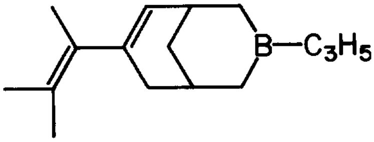
A simple synthesis of borinane has been described (55). The reaction of 1,3-diene addition compounds with magnesium with haloboranes provides a simple route to 3-borolenes; 3-phenylborolene and its adduct with trimethylamine were characterized in detail (63).

(Dialkyl)vinylboranes are readily prepared by hydroboration of alkynes with dialkylboranes (17). Also, a general synthesis of (dialkyl)cis-1-bromo-1-alkenylboranes has been described (162). The stepwise hydridation of $(CH_3)_2S\text{-}BRBr_2$ with $LiAlH_4$ followed by stepwise hydroboration of two different alkenes was found to give access to mixed trialkylboranes, BRR'R" (28).

The interaction of allylboranes with 1-ethoxy-2-trimethylsilyl acetylene occurs with the formation of species of the type $R'_2B\text{-}C(Si(CH_3)_3)\text{=}C(OC_2H_5)\text{-}CRH\text{-}CH\text{=}CH_2$, which - even at room temperature - readily eliminate $R'_2BOC_2H_5$ (50). The condensation of triallylborane with vinyl- and isopropenylacetylene yields, depending on the temperature, the corresponding substituted derivatives of 2-borinene(20 to 80°C)



or 3-borabicyclo(3.3.1)non-6-ene (120 to 140°C) (51):



Rearrangements of 3-borabicyclo(3.3.1)nonanes (31) and thermal reactions of B-alkyl-9-borabicyclo(3.3.1)nonanes (150) have been described. 9-Borabicyclo(3.3.1)nonane (= 9-BBN) and its derivatives continue to be fashionable reagents in organic synthesis. Various stereoselective reductions employing 9-BBN have been described (103, 165) and the kinetics of hydroboration of haloalkenes with 9-BBN (75) were found to parallel those of the parent alkenes; similar observations were made for 1-halo-1-alkynes (76, see 151). Also, hydroborations of alkenes with the 9-BBN dimer in non-coordinating solvents and of 9-BBN adducts with donor molecules were examined in order to elucidate the role of complexing agents (239). Several additional studies employing 9-BBN and derivatives thereof in organic syntheses have been reported (41, 149, 165).

Carbonylation of the THF complex of 1-boraadamantane gives the (1-adamantyl)dihydroxyborane, which can be isolated as the heterocyclic derivative ad-B(-O-CH_2-)$_2$$CH_2$ (6). The patter species is also obtained from 1-bromoadamantane and Cl-B(-O-CH_2-)$_2$$CH_2$ by coupling with metallic sodium. It is very sensitive to hydrolysis, which is accompanied by partial B-C bond cleavage. It reacts with CH_3MgI to yield ad-B$(CH_3)_2$, some reactions of which are described.

The reaction of tris(trimethylsilyl)silyllithium with trigonal boranes has been found to lead to species such as $R_2B\text{-}Si(SiR_3)_3$ (R = CH_3) (13). Methoxyboranes, $R_{3-n}B(OR)_n$, add to the silyl reagent to form the corresponding borates.

(Dialkyl)vinylboranes react with iodine in the presence of a base to form cis-alkenes (18); see also (16). For additional reactions of organylboranes with iodine (100, 182) and related reactions (167, 177, 182) for organic syntheses, see the indicated references. Additional applications of triorganylboranes in organic syntheses include the interaction of trialkylboranes with aromatic sulfonyl azides (101) or with allyl phenyl ether (226) and various other reactions (96, 119, 126, 152, 166, 183). SCHIFF bases react with triorganylboranes and carbon monoxide in the presence of catalytic amounts of cobalt carbonyl to give amides (176). For a preparation of ^{13}C-labelled carboxylic acids via organylboranes + ^{13}CO, see (247).

For studies of the mass spectral fragmentation of trimethylborane, see (233). The vibrational spectrum of (cyclopropyl)dimethylborane has been examined (93) and the N-H stretching region of the infrared spectrum of $(C_3H_7)_2B(CH_2)_3NH_2$ has been studied in detail (198). Carbon-13 NMR data for some phenylethynylboranes have been reported (67).

Radical anions of boranes which are isoelectronic with tertiary alkyl, allyl and benzyl radicals have been characterized by ESR data (113). The radical anion of tetra-t-butyldiborane(4) has been generated from di(t-butyl)haloborane and Na/K alloy and was identified by ESR spectroscopy (59). Similarly, the radical anion of tetra-t-butyltetraborane(4) has been generated from RClB-BClR (R = t-C_4H_9) and Na/K alloy and was characterized by ESR data (65).

5 HALOBORANE DERIVATIVES

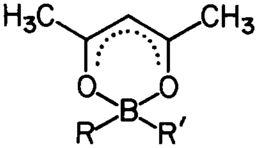
Ab initio and force field calculations on (vinyl)difluoroboranes elaborate on the π-bond contribution (105).

Adducts of the type L-BHRCl (R = $C(CH_3)_2$-$CH(CH_3)_2$) have been prepared by hydroboration of 2,3-dimethylbut-2-ene with L-BH_2Cl (L = THF, $(C_2H_5)_2O$, $(CH_3)_2S$) and their hydroboration properties were examined (27). The adduct of thexylchloroborane with dimethyl sulfide has been found to be a monohydroborating agent of exceptional regioselectivity (163, 164). For additional uses of thexylchloroborane in organic synthesis, see (210, 211).

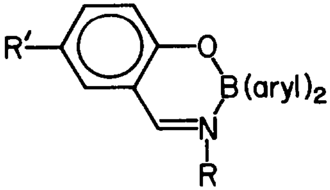
Various reactions of (organyl)haloboranes are reported elsewhere in this present survey. For the rapid incorporation of radio-bromine via the reaction of labelled NaBr with organylboranes, see (244).

6 SELECTED CHELATES

Ligand exchange reactions of boron substituents in chelates of the type



(with R, R' = F, Cl, C_3H_7) have been studied by NMR experiments (69) and the inversion of the configuration at the tetrahedral boron in benzoxazaborones of the type

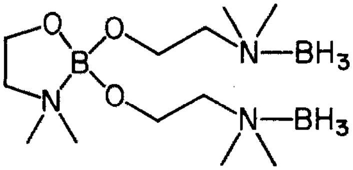


has been characterized quantitaviely by the dynamic NMR method (184). However, it was reported elsewhere that the racemization of dissymmetric diarylboron salicylideneaminato chelates involves B-N bond dissociation to provide for a three-coordinate boron intermediate (196) rather than retaining the four-coordinate environment as suggested earlier (214). This finding (196) is supported by some MNDO calculations (127).

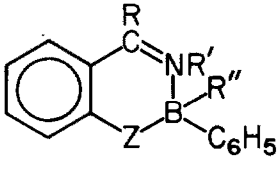
For some structural investigations on boron chelate complexes, see (130).

Boron chelates derived from 2-aminophenol or salicylaldehyde have been prepared (241) and the crystal and molecular structures of the (acetylacetonato)diphenylboron and (tropolonato)diphenylboron chelates have been studied by X-ray diffraction (242). For the crystal and molecular structure of dibutylboryl-2-(N,N'-dicyclohexylamidino)amino thiazolate, see (243).

The nature of products resulting from the reaction of 1,2-aminoalcohols with either THF-BH_3 or $(CH_3)_2S$-BH_3 strongly depends on the experimental conditions. The species



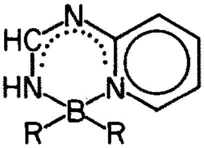
was characterized by ^1H and ^{11}B NMR data as well as by infrared spectroscopy as one of the products; similarly, the BH_3-free compound was identified (216). The stability of fused boroxazolidine-type chelates containing four-coordinate boron has been studied by ^{11}B NMR spectroscopy (189). Also, chelates of the type



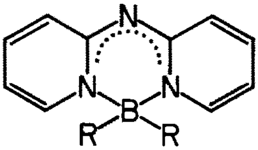
with Z = O or NH have been synthesized and various physical properties of the compounds were reported (61).

Various new (aryloxy)difluoroboron chelates have been described (88, 133). (Aryloxy)difluoroboron chelates are suitable intermediates for regiospecific bromination (212). For some studies on fluoroboron derivatives of azomethines, see (219).

The molecular structure of



has been determined by X-ray diffraction (70). 2,2'-Dipyridylamine, $R_2'NH$, reacts with $RSBR_2$ ($R = C_4H_9$) to initially form the aminoborane $R_2'N-BR_2$ (36). On heating of the material to a temperature above 100°C, the boryl group migrates to form the inner complex



The molecular structure of bis(N-methylimidazole)bis(diphenylborondimethylglyoximato)iron(II) has been determined (124) and some new rhenium betaketoiminato boron chelates have been described (159).

7 BORON-OXYGEN COMPOUNDS

The syntheses and selected properties of some 1,2-oxaborolanes have been described (40). The key step of a facile preparation of (halomethyl)oxyboranes of the type $XH_2CB(OR)_2$ involves a tin-hydride reduction of $Cl_2HCB(OR)_2$ species (64). Phenylthiomethyl derivatives of boron of the type $C_6H_5SCH_2B(OR)_2$ have also been prepared (29). The species can be deprotonated with lithium diisopropylamide to yield $Li(C_6H_5SCHB(OR)_2)$, which are reactive intermediates. For example, they can be alkylated with alkyl halides to yield $C_6H_5SCHR'B(OR)_2$ or can be brominated with elemental bromine. The preparation of the highly sterically hindered species $(R_3Si)_3CB(C_6H_5)(O(CH_2)_4$-

$C(SiR_3)_3$) with R = CH_3 has been described elsewhere (110). The molecular structure of the compound was determined by X-ray diffraction. The deprotonation of cyclic organyloxyboranes of the type $H_2C(B(-O(CH_2)_nO-))_2$ is readily effected with lithium 2,2,6,6-tetramethylpiperidine (26). The resultant diboryl methide salt can be alkylated with alkyl halides. The deprotonation and alkylation process can be repeated to yield species of the type $RR'C(B-O(CH_2)_nO-))_2$.

(Organyloxy)boranes of the type $R_nB(OR')_{3-n}$ with n = 1 or 2 react with organic peroxides, R"OOH, by rapid displacement of OR' by OOR" groups besides oxidative and protolytic cleavage of B-C bonds. The reactions are facilitated by increasing coordinating power of the organyloxyborane and electrophilicity of the proton in the peroxide (237).

The preparations of bicyclic (aryl)oxyboranes derived from N-hydroxy-alphaaminocarboxylic acids (195) and of 2,8,9-trioxa-5-aza-1-borabicyclo(3.3.3)undecane have been reported . For the direct chiral synthesis of (organyl)dioxyboranes via asymmetric hydroboration displacement, see (155); and for 3-buten-1-oxy derivatives of boron, see (9). For the preparation and reactions of (vinyl)oxyborane copolymers, see (91).

(Dialkylamino)alkoxyphenylboranes, $R_2N-B(OR')(C_6H_5)$, have been prepared and their NMR data were discussed in detail (120). The mass spectra of C-trifluoromethyl-substituted boratranes, $B((OCH_2CH_2)_n(OCH(CF_3)CH_2)_{3-n})_3N$, are characterized by the ready elimination of a $OCHCF_2$ group (73).

For a comparative study on the chelation of pentitols and D-mannitol with (phenyl)dihydroxyborane, see (131). On-column derivatization has been employed for the chromatography of (aryl)hydroxyboranes (217). Various oxyborane derivatives are used as stationary phase in chromatography (54, 78, 106, 125, 220).

Tris(trimethylsilyloxy)borane can be prepared directly from hexamethyldisilazane and boric acid by heating a mixture of the reactants to 70 to 120°C (72).

Recent studies (161) shed some doubt on the previously claimed ready symmetrization of $HB(OCH_3)_2$ to yield B_2H_6 and $B(OCH_3)_3$ but tend to establish the former as a fairly stable species. For a study on the hydrolysis of (organyloxy)organylboranes, see (123). The stability of epoxy organyloxoboroxin systems has been investigated elsewhere (79).

Various 1,3,5,2-oxadiazaboroles have been prepared and were characterized by ^1H NMR and mass spectral data (85).

The O-diethylborylation in the synthesis of saccharides has been studied (207). Diethylboryl groups are readily displaced by trimethylsilyl moieties on reaction with the trimethylsilyl enolate of pentane-2,4-dione (208). (Methoxy)-dialkylboranes (179) and (gamma-alkoxyallyl)oxyboranes (14, 104, 225) have also been used in organic syntheses. Boroxazines have been employed in a regiospecific synthesis of 1,3-diketones (99), cyclic (vinyl)oxyboranes in an aldol condensation (83), and (alkenyl)dimethoxyboranes in additions to aldehydes (89). Aromatic esters have been prepared by a direct condensation employing tris(aryloxy)boranes (109).

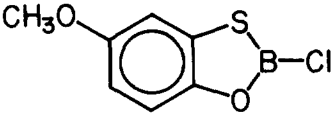
Bis(5-aminosalicyl)borate dihydrate has been described (132). Several bis(oxalato)borates and bis(malonato)borates have been prepared and were characterized by infrared and ^{11}B NMR data (138). The crystal structures of alkali metal bis-(malonato)borates (24) and of rubidium bis(citrato)borate (23) have been studied by X-ray diffraction. Also, the crystal structure of the complex $(CH_3COO)_2B(acac)$ has been determined (4).

Interaction of boric acid with 2,2'-imino bisethanol in aqueous medium yields a substituted ammonium salt containing the cyclic trimetaborate anion (37). The interaction of boric acid with 2,2',2"-nitrilo triethanol also yields an ammonium salt containing a boroxin ring in the anion (200). Similar reactions of boric acid with diethylene triamine (191) and dibutylamine (201) have been described.

8 SULFUR-CONTAINING DERIVATIVES

Trimethylsilylthioboranes, $(CH_3)_3Si-S-BRR'$, have been prepared and were found to be thermally quite stable. In contrast, the corresponding $(CH_3)_3Ge-S-BRR'$ species undergo rearrangement reactions on attempted distillation (94).

2-Mercapto-4-methoxyphenol reacts with boron trichloride to yield the heterocycle



The boron-bonded chlorine of the compound is readily dispaced by various organic groups via standard procedures (181).

Exchange reactions of annular boron in sulfur-containg ring systems, e.g., $(-BCH_3-S-S-BCH_3-)S + 2\ BBr_3 \rightarrow 2\ CH_3BBr_2 + (-BBr-S-S-BBr-)S$, as well as analogous reactions involving $(-BCH_3-S-S-BCH_3-)NCH_3$ and $(-BCH_3-NCH_3-NCH_3-BCH_3-)S$, respectively, all occur by an endocyclic mechanism (129). The latter is also effective in corresponding reactions of the 1,3,2-dithioborolane system. This was elucidated by employing boron-10-labelled boron tribromide. Similar studies on boron exchange between triaza- and thiazadiborolidines are described elsewhere (52). One intermediate from such an exchange reaction between (-BBr-S-S-BBr-)S and $(-NCH_3-CH_2-CH_2-NCH_3-)BCH_3$ is the 1:1 molar adduct of the 1,3,2-trimethyldiazaborolidine and the new heterocycle 4,6-dibromo-1,2,3,5,4,6-tetrathiodiborinane (144). The species

(-BBr-S-S-BBr-)S reacts with alkynes, RC≡CR', to give the heterocycle (S-CR=CR'-S-)BBr; boron-bonded bromine of the latter compound is readily displaced, e.g., by reaction with amines or organyllithium. Similarly, $(-BCH_3-S-S-BCH_3-)S$ was found to react with isocyanates or carbodiimides to yield heterocycles of the type $(-S-CS-NR-S-)BCH_3$ (12). Reaction of (-BBr-S-S-BBr-)S with certain sulfinylamines yields (-BBr-S-S-BBr-)NR species. However, the S-S bond of the starting material may also be cleaved by sulfinylamines to yield $(-BCH_3-NR-S-BCH_3-)S$. Various by-products are obtained in these reactions; they include boroxins and heterocyclic dioxazatriboranes as well as oxadiazatriboranes (30).

Ab initio calculations of structures and properties (222) and studies on geometries and stabilities (221) of sulfidoboranes, R-B=S, have been reported.

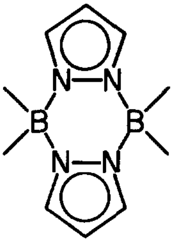
9 BORON-NITROGEN CHEMISTRY

Synthesis and properties of a series of (dialkylamino)-chlorophenylboranes (58), $R_2N-BCl(C_6H_5)$, as well as (alkylamino)- and (dialkylamino)piperidinoboranes (121) have been described. The rotational barrier about the B-N bond in (dialkylamino)fluorophenylboranes has been determined by variable-temperature ^{13}C NMR experiments (118).

Some N-silylated bisaminoboranes have been prepared and their stereochemistry was studied by NMR spectroscopy (205). Reactions of (organylsilylamino)boranes (48) and (organylsilylamino)(alkylamino)boranes (49) with various hydrides have been examined in detail.

Aminoboranes of the type $Cl(R')B-N(t-C_4H_9)(SiR_3)$ ($R = CH_3$) react at 530°C in a hot tube by elimination of $ClSiR_3$. The resultant monomeric iminoborane $R'B=N(t-C_4H_9)$ can be stored at -80°C. The species trimerize on heating to yield the corresponding borazines. Reaction of the monomers with BR_3 yields diborylamines whereas with phenyl azide tetrazaborolines are obtained (82). Monomeric carbiminoboranes, $RN{=}CR'{-}BR^2R^3$ with R^2 and/or R^3 amino substituents are readily obtained by treatment of isonitriles with lithium alkyl and subsequent treatment with (amino)haloboranes. Carbiminosilanes react with (amino)haloboranes under formation of silyleneaminoboranes (57).

Two 1,3-dimethyl-2-(methylpyrazol-1'-yl)-1,3,2-diazaboracycloalkanes have been prepared (206). The interaction of such monomeric pyrazol-1-ylboranes containing trigonal boron with pyrazoles yields 1:1 molar adducts containing four-coordinate boron. Reaction of the monomers with monoaminoboranes leads to pyrazaboles of the general structure

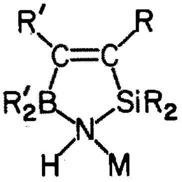


Depending on the reaction conditions, the interaction of tris(dimethylamino)borane with pyrazole (= Hpz) yields the adduct $R_2HN{-}Bpz_3$ or various pyrazaboles (38). The reaction proceeds via the short-lived monomers. Similarly, the reaction of bis(dimethylamino)phenylborane with pyrazole was examined.

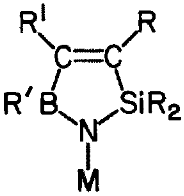
N-Lithio-N,N',N'-triorganylureas and thioureas react with $BrB(CH_3)_2$ to yield the corresponding dimethylboryl derivatives. A similar reaction of the non-lithiated species yields substantial amounts of cyclic by-products (187).

The molecular structure of $HB(NH_2)_2$ has been determined from microwave spectral data (137). Also, the crystal structure of $B(N(CH_3)_2)_3$ has been determined (199).

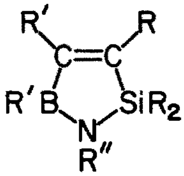
Novel five-membered C_2BNSi heterocycles have been described (7). It was found that the reaction of R^1_2B-CR^1=CR-SiR_3 with MNH_2 (M = Na, K; R = CH_3; R^1 = C_2H_5) initially yields the salt M(NH_2-BR^1_2-CR^1=CR-SiR_3) that eliminates methane to form the heterocycle



The latter, in turn, eliminates ethane to form



which is readily converted to



Theoretical models for interpreting the photoelectron spectrum of borazine, $(\text{-BH-NH-})_3$, have been compared (84). Ligand exchange reactions of boron substituents of borazine were studied by NMR experiments (69). Based on UV spectroscopic data, hexamethylborazine forms a 1:1 molar complex with elemental iodine by n-donor interaction (90). Hexamethylborazine also forms a 1:1 molar complex with $GaCl_3$. The crystal structure of the solid material has been determined by X-ray diffraction and the ring was found to be non-planar. In toluene solution, the species was found to be fluxional at room temperature but seems to become static at lower temperatures

(62). Based on variable-temperature ^{13}C NMR data, two conformers of $(-BC_2H_5-NC_2H_5-)_3Cr(CO)_3$ exist in CD_2Cl_2 solution (112). Thermal decomposition of hexachloroborazine in silica tubes provides for thin protective films (21).

10 COMPLEXES OF BORANES WITH DONOR MOLECULES

The vibrational spectrum of F_2HP-BH_3 (197) and that of CH_3NC-BH_3 (42) have been examined and the molecular structure of $(CH_3)_3P-BI_3$ has been determined by gas-phase electron diffraction (173). The O-B bond in the 1:1 molar adduct of o-nitrophenol with boron trichloride seems to have multiple bond character (172).

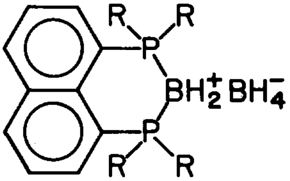
Amine-cyano(pyrrol-1-yl)borane complexes of the type $L-BH(NC_4H_4)CN$ containing a chiral boron atom have been described (188). The reaction of $Cl(CH_3)_2P-BH_3$ with the lithium salts of acetamide , N-methyl acetamide or N-methyl formamide produces the amides substituted at the nitrogen with the $P(CH_3)_2(BH_3)$ moiety, i.e., phosphine-borane analogs of trimethylsilyl amides (116). Also, a new method for the preparation of 2-aminopyridine-borane and its analogs has been described (98). Triarylboranes form 1:1 molar complexes with antipyrine in which the exocyclic oxygen atom serves as donor site (238).

Morpholine-borane reacts with sodium hypochlorite in 1:4 molar ratio whereby three OCl^- species are utilized for hydride oxidation and a fourth is consumed in the chlorination of morpholine (43). An unsymmetrical cleavage of μ-dimethylaminodiborane(6) has been observed on reaction with o-phenylenediamine (5).

11 BORON CATIONS

Details for the syntheses and extensive characterizations of dicoordinate amidoboron cations have now been presented in two independent publications (156, 157). Bromide ion abstraction from bisaminoboranes of the type (tmp)(R_2N)BBr or monoaminoboranes (tmp)BR'Br (tmp = 2,2,6,6-tetramethylpiperidino; R' = CH_3, C_6H_5) with $AlBr_3$ in CH_2Cl_2 produces the cation $(tmp{=}B{=}NR_2)^+$ or $(tmp{=}B\text{-}R')^+$, respectively (156). Similarly, $((i\text{-}C_3H_7)_2N)_2BCl$ reacts with $AlCl_3$ to yield $((i\text{-}C_3H_7)_2N{=}B{=}N(i\text{-}C_3H_7)_2)^+$ (157). NMR and IR data on the various species as well as an X-ray diffraction study on one representative (156) confirm the existence of the cations contaiing two-coordinate boron.

Reaction of $C_{10}H_6\text{-}1,8\text{-}(PR_2)_2$ (R = CH_3) with THF-BH_3 yields the corresponding bis(borane) adduct with P-B coordination besides the following boronium(1+) salt (146):



12 ORGANYLBORATES(1-) AND RELATED SPECIES

A detailed preparation of sodium cyanotrihydroborate has been described (135). Ab initio molecular orbital calculations have been made to study the binding prefrence of CN^- for BX_3 (X = H, F, Cl, CH_3); electronegative substituents were shown to favor binding through nitrogen (148).

Molecular geometries have been calculated for organylborates in order to determine the energies for sequential 1,2-shifts from boron to carbon (15). The conversion $(C_5H_5)_2Zr(CH_3)_2 \longrightarrow (C_5H_5)_2Zr(BH_4)_2$ proceeds via intermediates containing coordinated $(H_3BCH_3)^-$ and $H_2B(CH_3)_2^-$ moieties. This observation suggests that insertion of BH_3 into the Zr-CH_3 bond occurs (153). Lithium methyltrihydroborate is a stereoselective agent for the reduction of cyclic ketones (224).

Methoxyboranes, $R_{3-n}B(OR)_n$ (R = CH_3), add to tris(trimethyl)silyllithium to form the corresponding borates (13). Similarly, the reaction of $LiSiR_3$ with $B(OR)_3$ yields the trimethylsilylborates $Li(B(SiR_3)_4)$ (145).

The synthesis and ligand properties of the dihydrobis-(5-nitroindazolyl)borate anion have been reported (102), and the hydrolysis of hydro(pyrrol-1-yl)borate ions has been examined in detail (107). The first poly(pyrazol-1-yl)borate ion containing two different pyrazole moieties has been obtained by the reaction of 3,5-dimethylpyrazole-borane with sodium hydride and subsequent treatment of the $Na(H_3BN_2C_3H(CH_3)_2)$ with pyrazole. Several complexes of the obtained new ligand $(H_2(H_3C_3N_2)BN_2C_3H(CH_3)_2)^-$ were described (236).

The crystal structures of $H(F_3BOH)$ and $H(F_3BOCH_3)$ have been studied by X-ray diffraction (10), and the molecular structure of piperidinium tetramethoxyborate has been determined (45). The vibrational spectrum of $K(F_3BCH_3)$ and isotopically labelled derivatives thereof as well as the crystal structure of the species have been examined (193).

Carbon-13 NMR data have been reported for lithium tetrakis-(phenylethynyl)borate, $Li(B(C{=}CC_6H_5)_4)$ (67). The molecular structure of bis(4-methylpyridine)hydrogen(I) tetraphenylborate has been determined (44). Also, boron 1s binding energies for alkali metal tetraphenylborates have been determined (46) and studies on the solvation of the tetraphenylborate ion have been reported (128). The heats of dilution of aqueous solutions of sodium tetraphenylborate at 25°C have been determined (87) and conductance and viscosity measurements on tetrabutylammonium tetraphenylborate in non-aqueous solvents at 25°C have been performed (232). Heats of solution of sodium tetraphenylborate in water and water/t-butanol have also been determined (245).

Lithium trialkylalkynylborates underdo unique MICHAEL reactions involving migration of an alkyl group from boron to carbon (60); the resultant vinylboranes are of great synthetic potential.

13 METAL DERIVATIVES

A new ruthenium hydride complex containing a bidentate BH_4 group has been reported (154). Similarly, the BH_4 group serves as a bidentate ligand in a terpyridine-cobalt complex (3), but the BH_4^- ion is attached in monodentate fashion in a copper(I) complex (171).

Some new ruthenium and rhodium complexes of divinylboranes have been described (229); the species $(OC)_3Ru(\eta^5\text{-}(C_2H_3)_2BCl)$ and $((CH_3)_5C_5)Rh(\eta^5\text{-}(C_2H_3)_2BX)$ ($X = CH_3$, OCH_3, C_6H_5) were characterized and some chemical reactions were reported.

The reaction of $K((OC)_5CrCN)$ with haloboranes, R_2BX, yields the corresponding isocyanoborane complexes $(OC)_5CrCNBR_2$; the compounds were extensively characterized and some reactions have been studied (115).

Several transition metal complexes derived from the heterocyclic 4-π-electron donor ($-BR'-CR'=CR-SiR_2-NR-$) ($R = CH_3$; $R' = C_2H_5$) have been described (143) and unusual iron complexes of ($-BR'-CR=CR-CH_2-SiR_2-NR-$) have been briefly reported (47). The heterocycle ($-BR-CH=CH-CH_2-NSiR_3-$) ($R = CH_3$) has been converted to the anion (142); the latter reacts with $FeBr_2$ or $CoBr_2$, respectively, to yield the corresponding sandwich complexes. Detailed data on such species and an extension to include Ti and V complexes have been described elsewhere (235). There are two general methods for the formation of the cited complexes: (a) Reaction of the heterocycle with metal vapor; and (b) reaction of the heterocycle anion with metal bromide. X-Ray structural data on representative species have been presented.

New reports on the syntheses of pyrazol-1-ylborate complexes and/or reactions or structures thereof deal with the following metals: Zr (158), Mo (2, 19, 34, 35, 86, 180, 240), W (2, 34, 86), Co (39, 169, 236), Ni (236), Rh (139, 140, 141), Pt (111), Zn (236), Cu (136, 169), and Yb (170).

14 BIOLOGICAL ASPECTS

A report on in-vitro studies on the preparation of boron-labelled antibodies to carcino-embryonic antigens for boron-10 neutron capture therapy has appeared (92). Some decachloro-o-carborane derivatives that are suitable for the preparation of boron-labelled biological macromolecules have been described elsewhere (215).

A theoretical study of the structure and properties of the boron analog of glycine has been reported (95) and the actual preparation of boron analogs of glycine has been described (56).

The fractionation of membrane proteins on (dihydroxy)phenylborane-agarose has been studied (80) and amine-boranes have been used as reducing agents for the reductive alkylation of proteins (77).

The inhibition of lipo protein lipose by (dihydroxy)phenylborane (81) and the antivirial activity of boron chelates derived from 2-aminopyridine have been studied (108).

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