BORON

ANNUAL SURVEY COVERING THE YEAR 1980

PART II

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CONTENTS

1 REVIEWS AND BOOKS

Boron cage compounds (110), carboranes including their metal complexes (141), boron subhalides (262), and boron heterocycles as ligands in transition metal chemistry (142) are the topics of recent reviews. A brief accounting of the present status of phosphaneborane chemistry has also been

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Previous survey see J. Organometal. Chem., Vol. 196 (1980) p. 1 - 35.

presented (245) and the NOBEL Lecture by H. C. BROWN has been published (235, 240).

A new book (66) contains a brief survey of boron-nitrogen chemistry; and an exhaustive treatise on the application of boron compounds in biology, medicine and pharmacy has been compiled by KLIEGEL (188).

The 20 volumes of the New Supplement Series of the GMELIN Handbook dealing with boron compounds have been updated to a uniform literature closing date of the end of 1977 (144). However, the literature coverage is somewhat restricted as compared to the original volumes and concentrates on the inorganic aspects; the order of presentation is guided by the GMELIN principle of the last position. A formula index is compiled in the last of the three parts of the volume.

2 SOME DATA OF GENERAL INTEREST

New and interesting boron compounds of general interest include the B_6 ring derivative $(-BN(CH_3)_2-)_6$, which can be isolated from the distillation residue of the dehalogenation of $ClB(N(CH_3)_2)_2$ with Na/K alloy (128). The cited compound, containing the B_6 ring in chair conformation, is the first homocyclic boron species and the first boron(1) compound that is not electron-deficient. An inorganic porphin, B_8S_{16}, has been obtained by fusion of B_2S_3 with S_8 in vacuum (130); the molecule consists of four B_2S_3 rings bridged by S groups into a macrocyclic structure.

A study of ^1H and ^{13}C NMR spectra of phenylboranes of the type $C_6H_5BX_2$ (28) is principally in consonance with the findings of an earlier more exhaustive investigation (29). Previous comparisons of boron-11 chemical shifts of trigonal boranes with carbon-13 chemical shifts of isoelectronic carbenium ions have been re-examined (93). The observed trends allow for a correlation of pi-bonding in boron compounds and pi-delocalization in carbenium ions and also illustrate a reflection of structural parameters in the chemical shift values. Also of general interest is a semiempirical theory of boron chemical shifts utilizing gauge-invariant atomic orbitals (198).

MNDO Calculations have been performed on 28 boron compounds of various types including species such as HBO, $B(CH_3)_3$, $OCBH_3$ and $(CH_3)_2B_2H_4$, and correlations with observed spectra led to estimates of group corrections (247); correlated vibrational frequencies were used to assign or predict the spectra of other species such as vinylborane. An empirical prediction of heats of formation and redistribution reactions of boron compounds using nonbonded interaction energy terms for redistribution equilibria has also been developed (58).

For a new spectrophotometric determination of boron employing 4-(p-sulfophenylazo)-1,8-dihydroxynaphthalene, see (133); for the determination of boron in geological materials, see (242).

Also of general interest may be a report describing the synthesis of boron-containing A and Y zeolites and a study of the effects of boron on their catalytic activity in the dehydration of alcohols (55).

3 HYDRIDIC SPECIES

The transition-metal catalyzed reaction of alkynes with pentaborane(9) proceeds indeed via alkenylated pentaboranes, which can then be converted to small carboranes (210). Diborane reacts with lithium diarylcuprates by transfer of phenyl to boron (216); the resultant mixtures of triarylboranes and monoorganylboranes can be separated. Monoisopinocampheylborane has been used for the hydroboration of phenylated tertiary olefins (155); and 1,4-oxathiane-borane (with the boron being bonded to the sulfur atom!) has been suggested as a new and convenient hydroboration reagent (15).

Boron-11 NMR data show that the hydroboration of 3-methyl-1,3-butadiene proceeds via 3-methyl-1-boracyclopentane as an intermediate (183). Similarly, several intermediates in the hydroboration of 1,5-cyclooctadiene could be identified by ^{11}B NMR measurements (184).

Ab-initio molecular orbital studies examine the interaction through triple bonds in borylpolyynes, $H(C{\equiv}C)_nBH_2$, and aminoborylpolyynes, $H_2N(C{\equiv}C)_nBH_2$ (n = 1 to 4) (181).

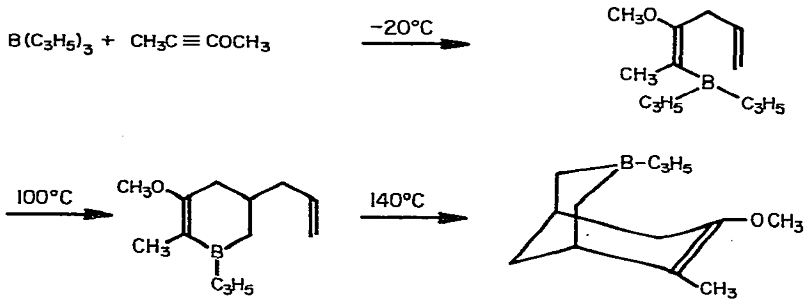
The structure of cis- and trans-1,2-dimethyldiborane(6) has been examined by gas-phase electron diffraction at room temperature (234).

4 TRIORGANYLBORANES

Thexylchloroborane, $(CH_3)_2HC\text{-}C(CH_3)_2\text{-}BHCl$, obtained from thexylborane and HCl in ether, can be employed in a hydroboration/alkylation sequence to yield mixed (thexyl)-diorganylboranes (209). The stepwise hydroboration of alkenes with thexylchloroborane has been developed as a general synthesis of ketones (232).

Originating from triallylborane and 3-methyl-1,2-butadiene, 4,4-dimethyl-1-boraadamantane has been prepared and some of its properties have been determined (123). Another route to the cited compound is described elsewhere (125) and a new method for the synthesis of 1-boraadamantane and its complex compounds has also been developed (124). 3,5-Dimethyl-1-boraadamantane has been obtained from propargyl methyl ether and tris(2-methylallyl)borane (126). The reaction of triallylborane with propargyl tetrahydropyranyl ether has also been studied (127).

Condensation of triallylboranes with 1-alkoxypropynes occurs by addition of the diallylboryl fragment in the beta position to the alkoxy group (122). Triallylborane and 1-methoxypropyne interact in the following fashion:



A new synthesis of 2-alkoxy-1,4-pentadienes is based on the interaction of allyldialkylboranes or alkyldiallylboranes, respectively, with alkoxyacetylenes (267). Reaction of the latter reagent with triallylboranes was utilized for the synthesis of allylacetylenes (268).

(Dialkylamino)dialkinylboranes react with $(CH_3)_2SnH_2$ under formation of 1-stanna-4-bora-2,5-cyclohexadienes (82). Subsequent transamination or alcoholysis yield access to additional derivatives of the SnC_4B heterocycle, of which the alkoxy derivatives can be B-alkylated with aluminum alkyls or $t\text{-}C_4H_9Li$. The reaction of $B(i\text{-}C_3H_7)_3$ with the tin derivative $(CH_3)_3SnC\equiv C\text{-}C\equiv CSn(CH_3)_3$ proceeds via an enyne intermediate which could be identified by NMR spectrocopy.

Alpha-silyl-alpha-borylmethane derivatives of the general structure $R_3Si\text{-}CHR'\text{-}BR'_2$ are formed on interaction of $Li(R_3SiCHX)$ with trialkylboranes (193); these can then be oxidized to alpha-hydroxyorganylsilanes.

Pentaphenylborole, $C_6H_5B(\text{-}CC_6H_5\text{=}CC_6H_5\text{-})_2$, has been synthesized in pure state (217). Reduction of the species with metallic potassium in tetrahydrofuran produces the dianion. Reduction of $FB(\text{-}CCH_3\text{=}CCH_3\text{-})_2BF$ with metallic potassium proceeds in two steps (109). Initially, acquisition of one electron yields the unstable radical anion; then F^- is removed by the potassium to yield the dehalogenated radical anion of a 1,4-diborabenzene, $(B(-CCH_3=CCH_3-)_2B)^{\dot{-}}$.

New studies employing 9-borabicyclo(3.3.1)nonanes include a ^{11}B and ^{13}C NMR examination of B-substituted derivatives and their pyridine complexes (24); some interesting photoelectron spectroscopic studies (90); a ^{13}C NMR investigation of the interaction of 9-borabicyclo(3.3.1)-nonane with amines of increasing steric requirements (212); the reaction of B-alkyl derivatives with vinyllithium in the presence of iodine to yield trisubstituted ethylenes (271); the formation of ate complexes from lithium methoxyallene and B-alkyl-9-borabicyclo(3.3.1)nonanes and their reaction with acetic acid to give 1-alkyl-1-methoxycyclopropanes (35); the generation of alkenylcopper species from alkenyldialkylboranes derived from 9-borabicyclo(3.3.1)nonane and dicyclohexylborane, which undergo stereospecific cross-coupling with allylic halides (22); kinetic studies on the reaction of 9-borabicyclo(3.3.1)nonane with representative alkenes (190); and the employment of 9-alkyl-9-borabicyclo-(3.3.1)nonanes in homologation of organylboranes via carbonylation/reduction (194). Lithium alkyls react with 1-boraadamantane in THF to yield lithium 1-alkyl-1-bora-adamantanates (246). The latter can be converted to 7-methylene-3-alkyl-3-borabicyclo(3.3.1)nonanes by treatment with acetyl chloride.

The first representatives of stable tetraalkyldiboranes(4) have been described (1,2). In both cases the stabilization is achieved by attaching t-butyl groups to the boron and the following species were described: $B_2(t\text{-}C_4H_9)_3CH_3$ (1), $B_2(t\text{-}C_4H_9)_2(CH_2\text{-}t\text{-}C_4H_9)_2$ (2), $B_2(t\text{-}C_4H_9)_3(CH_2\text{-}t\text{-}C_4H_9)$ (2).

The application of organylboranes in organic synthesis continues to receive wide attention. For example, reaction of trialkylboranes with 1,2-dimethoxyethenyllithium provides a convenient route to symmetric 1,1-dialkylethenes (149); reaction of ICl with trialkylboranes is a mild but effective conversion of alkenes into alkyl iodides (211); and the cyanidation of organylboranes with labelled KCN permits ready introduction of ^{13}C or ^{14}C into organic molecules (16). The palladium-catalyzed cross-coupling reaction of 1-alkenylboranes with allylic or benzylic bromides gives access to 1,4-alkadienes and allylbenzenes (150), and the reaction of trialkylboranes with 1-alkyne and lead(IV) acetate is a new and regio- and stereoselective one-pot synthesis of enol acetates (148). (1-Halo-1-alkenyl)dialkylboranes react with lead(IV) acetate to yield 1-halo-1,2-dialkylethylenes(171), and alkenylboranes react with palladium acetate to give olefinic derivatives in stereoselective manner (172). The methylcopper-induced cross-coupling of alkenylboranes with organic halides provides a convenient method for the synthesis of 1,4-dienes and monoolefins (89). The stereochemistry of the molybdenum peroxide oxidation of organylboranes to alcohols has also been studied (231).

The reaction of organylboranes with acetals and formals has been studied (42), and the stereospecific alkylation of phenylacetylene by electrochemical reactions of organylboranes has been described (146).

Lithium enolates derived from ketones do not interact with triorganylboranes; however, based on ^{1}H and ^{11}B NMR evidence, the corresponding potassium enolates do react with relatively unhindered triorganylboranes (33). Carbanions stabilized by an adjacent boron atom have been prepared in good yield by proton abstraction from dimesitylboranes of the type R_2B-CNR'R" with R = mesityl and R', R" = H, CH_3, C_2H_5 (32).

The base-induced migration of an alkyl group from boron to carbon in a gamma-acetylvinylborane occurs predominantly in an anti manner with respect to the leaving group (23). Triorganylboranes, BR_3, interact with organic CNO or CNN moieties in a 1,3-addition of the R and BR_2 units, whereas NNO groups are reduced to the NN moiety (135). The addition of methylborylene, CH_3B:, to acetylenes has been employed for the preparation of 1,4-diboracyclohexa-2,5-dienes (225), and insertion of CH_3B: into C-H bonds in benzenechromium tricarbonyl and in ferrocene has been observed (224).

For some studies on the dynamic stereochemistry of 9-mesityl-9,10-dihydro-9-boraanthracenes, see (153). The degradation of tripropylborane with t-butyl hydroperoxide proceeds via a radical mechanism (116). Hypochlorites do not oxidize triorganylboranes (26).

A ^{11}B and ^{13}C NMR study of (dimesityl)alkylboranes has shown that the mesityl carbon atoms are insensitive to the character of the alkyl group (31). Sequential displacement of the methyl hydrogens of (dimesityl)methylborane gives rise to an alpha-effect but the chemical shift of the alpha-carbon atom in (dimesityl)benzylborane is insensitive to replacement of H by CH_3 (31).

The mass spectra of the inner complexes of (3-aminopropyl)dialkylboranes and some of their derivatives have been examined (121).

5 (ORGANYL)HALOBORANES

Alkylbenzenes react with dihaloboranes (generated in situ) to yield a mixture of m- and p-alkylphenyldihaloboranes with the elimination of hydrogen (258).

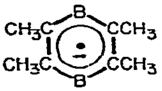
Hydroboration of alkenes with dimethyl sulfide-dihaloboranes gives ready access to (alkyl)dihaloboranes; some unusual trends and directive effects in the reactions have been studied (19). Similarly, hydroboration of alkynes with dimethyl sulfide-dibromoborane presents a convenient preparation of (alkenyl)dibromoboranes (20); the reagent is highly selective and is sensitive to both steric and electronic effects. For the hydroboration of some cyclic dienes employing monochloroborane, see (60). Thexylchloroborane hydroborates alkenes with excellent stereoselectivity to afford thexylalkylchloroboranes (248).

The diborane(4) derivatives $B_2Cl_2(t\text{-}C_4H_9)_2$ as well as $B_2Br_2(t\text{-}C_4H_9)_2$ have been described (1). The B-B bond of diborane(4) derivatives such as B_2Cl_4 or $B_2(N(CH_3)_2)_4$ and also those of B_4Cl_4 are cleaved on treatment with chloramines (106); oxidation products are BCl_3 or the respective aminoborane and diborylamine.

Acetoxydifluoroborane has been utilized for the acylation of cyclobutene (185). The 1:1 molar adduct of BF_3 with acetic acid reacts with azines containing active hydrogen, H_2Az, to from crystalline FBAz species (330).

For some comments on the symmetry group of methyldifluoroborane, see (174). For measurements of the adiabatic electron affinities of various trihaloboranes (including unsymmetrical species), see (69); the resultant values are used to obtain B-N bond energies for the adducts of the haloboranes with trimethylamine. The formation of $HBCl_2$ has been observed when a BCl_3/H_2 misture is irradiated with a pulsed CO_2 laser (11). For correlation effects of the pair boron cyanide - boron isocyanide, see (78). For the vibrational spectra of X_2BNCS (X = Cl, Br, I) and Cl_2BNCO, see (75); and for a detailed spectroscopic study on (cyclopropyl)difluoroborane, see (241).

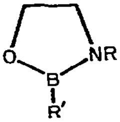
The photoelectron spectrum of $FB(-CCH_3{=}CCH_3-)_2BF$ has been compared with that of its organic analog, duroquinone, and the one-electron reduction behavior of the two species has been studied (168). The radical anion of the boron compound is short-lived. In the presence of metallic potassium, a secondary radical anion is formed which was identified as



6 BORON-OXYGEN COMPOUNDS AND BORIC ACID COMPLEXES

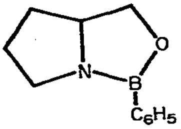
Allylbisoxyboranes have been obtained by reacting allyltrialkyltin with 2-chloro-1,3,2-dioxaborolane (40). Interaction of trans-2-chlorobuta-1,3-dienylmercury chloride with BCl_3 and subsequent methanolysis yields the compound trans-$CH_2{=}CH{=}CCl{-}CH{-}B(OCH_3)_2$ (67). The same material can be obtained by interaction of BCl_3 with vinylacetylene and subsequent methanolysis; however, in this latter case a mixture of cis and trans isomer is obtained. Trichloroborane also adds to isopropenylacetylene to yield the species $CH_2{=}CCH_3{-}CCl{=}CH{-}B(OCH_3)_2$ as methanolysis product. Deprotonation of $(CH_3)_3SiCH_2B(-O-C(CH_3)_2-)_2$ yields an anion which reacts with carbonyl compounds, R_2CO, to form alkenebisoxyboranes, $R_2C{=}CH{-}B(-O-C(CH_3)_2-)_2$, or with alkyl halides to form alpha-trimethylsilylalkylbisoxyboranes (80).

Various 1,3,2-azaoxaboracyclopentanes



have been obtained by the interaction of disilylated ethanolamines, $R_3Si{-}NR{-}CH_2{-}CH_2{-}O{-}SiR_3$, with haloboranes (86).

Characteristic physical data of the various species including those N-methylated derivatives where the boron substituent is H, F, Cl or Br are reported. Closeley related is the species

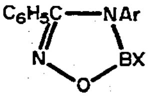


which was obtained from the reaction of bis(dimethylamino)-phenylborane with L-2-pyrrolidinylmethanol (131); analogous condensations occur on reaction of the cited borane with 2-amino-3-hydroxypyridine and 2-(o-aminophenyl)benzimidazole.

Bis(divinylboryl) oxide, $((CH_2=CH)_2B)_2O$, has been prepared by controlled hydrolysis of (divinyl)chloroborane (249). The NMR spectra of the species are interpreted in terms of mesomeric interactions between the vacant pπ orbital of boron and free electron pairs on oxygen as well as the pi-electron system of the vinyl moieties.

p-Dihydroxyborylphenylalanine has been resolved and was found to be a useful agent in the synthesis of peptides (237); it was incorporated into simple di- and tripeptides. Reaction of alkanolamines with tris(ethoxy)borane in 3:1 molar ratio yields the corresponding tris(aminoalkyloxy)-boranes as distillable liquids (261). For the employment of dioxyborylethyl moieties, $O_2BC_2H_5$, as protective groups in carbohydrate syntheses, see (269, 270). The pH dependency of complexation of organyldihydroxyboranes with diols has been studied using $C_6H_5B(OH)_2$ as model compound (266).

3-Aryl-4-phenyl-1,3,5,2-oxadiazaboroles



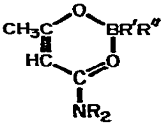
with X = OR, C_6H_5, $OSiR_3$, $OSnR_3$ have been synthesized from

N-arylbenzamidoximes in a condensation reaction (161). The diborane(4) derivatives $B_2(t\text{-}C_4H_9)_2(OCH_3)_2$ and $B_2(t\text{-}C_4H_9)_3(OCH_3)$ have been described and were used for the synthesis of tetraalkyldiboranes(4) (1,2).

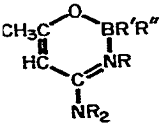
The homologation of carbonyl compounds to aldehydes via lithium bis(ethylenedioxyboryl) methide has now been described in detail (27). Similarly, interaction of boronic esters with (trimethylsilyl)chloromethyl lithium proceeds via an ate complex which rearranges with the displacement of chloride ion to form the homologous alpha-trimethylsilylboronic esters in high yield (30). Cyclic ethylenedioxyboryl species of the type $RB(\text{-}O\text{-}CH_2\text{-})_2$ react rapidly with $LiCHCl_2$ at low temperatures to form an intermediate which rearranges near room temperature with the formation of $R\text{-}CHCl\text{-}B(\text{-}O\text{-}CH_2\text{-})_2$ (255). This homologation process appears to be of general utility for the preparation of alpha-chloro organylborane species. Direct chiral syntheses employing boron derivatives of pinanediol have been described (254); and for metatheses of 2-ethyl-1,3,2-dioxyboracycloalkanes of medium ring size, see (252).

The ^{17}O chemical shifts of boron-oxygen compounds containing trigonal boron can be interpreted in terms of B-O(pp)pi-interaction (6). A decrease in the pi-bond order is indicated by the ^{17}O data in the series $(R_2B)_2O$ — $(\text{-}BR\text{-}O\text{-})_3$ — R_2BOR — $RB(OR)_2$ — $B(OR)_3$ ($R = CH_3$, C_2H_5). For a brief note on the exchange of O-bonded $B(C_2H_5)_2$ groups in polyhydroxyalkanes, see (156). For the utilization of oxyborylated species in the synthesis of pure carbohydrates, see (158 to 160). The interaction of carbohydrate phenylboronates with methanol (227) and acetone (228) has been studied; for the interaction of $(\text{-}BC_6H_5\text{-}O\text{-})_3$ with acyclic vicinal pentaols, see (229). Various B-alkenyloxyboroxines have been obtained from the reaction of tris(alkenyloxy)boranes with boric acid at 100 to 120°C (233); the species partially decompose in a reverse reaction on heating under reduced pressure. O-Borylated hydroxylamines, $C_6H_5CHR\text{-}NCH_3\text{-}OBR'_2$, are reduced by triorganylboranes to yield $C_6H_5CHR\text{-}NCH_3\text{-}BR''_2$ and $R''OBR'_2$ (135).

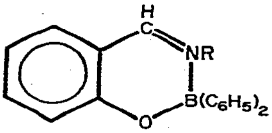
(Diorganyl)haloboranes react with alpha-trimethylsilyl-N,N-diorganyl acetamides in a condensation/elimination reaction to yield heterocyclic species of the following type (163):



However, if R = C_6H_5 and R" = NR_2, the linear species containing trigonal boron is formed. N,N-Diorganyl acetamides react with N-trimethylsilylated monoaminoboranes to yield heterocycles of the type (163):

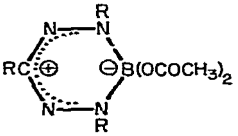


The influence of the amine substituent R in a series of substituted diphenylboron chelates derived from salicylaldehyde azomethines



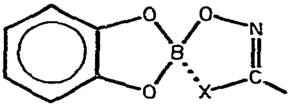
has been studied by spectroscopic means (195). Also, the electronic structures and conformations of some diphenylboron derivatives of aromatic and heterocyclic hydroxy and mercapto azomethine species have been studied by electronic absorption and emission spectroscopy and dipole moments (219). The data indicate four-coordination of boron by intramolecular complex formation. For some studies on dioximatonickel diphenylboron chelates, see (196).

Triphenylformazane, RHN-N=CR-N=NR (R = C_6H_5), reacts with diboron tetraacetate to yield the species

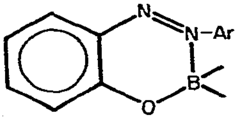


besides tetraphenyltetrazolium borate (164); other triarylformazanes behave similarly. For the molecular structure of diphenylboron-N-methyl-2-oxy-1-naphthaldiminate, see (111).

Reaction of 2-alkoxy-1,3,2-benzodioxaborole with monofunctional bidentate oximes such as furfuralaldoxime yields chelated structures of the general type (203):



Similarly, bidentate chelate reagents containing labile protons (OH, NH_2) in ortho-position to the azo group react with diphenylboryl oxide (or trihaloboranes) to yield chelates of the type (72):



Reaction of tris(isopropyloxy)borane, $B(OR)_3$, with N-hydroxyalkyl-benzylideneamines (= sbH) give $(RO)_2Bsb$, $ROB(sb)_2$, or $B(sb)_3$, respectively, depending on the sotichiometry of the reagents (65).

The mass spectral fragmentation of dioxazaboracycloalkanes and (aminoalkoxy)diphenylboranes indicates the existence of intramolecular donor-acceptor interaction between N and B when steric hindrance is absent (119). The mass spectral fragmentation of various boron chelates of aromatic azo compounds has also been studied (71).

Boron complexes of bifunctional tridentate SCHIFF bases (175) and of SCHIFF bases derived from o-hydroxyacetophenone and amines (176) have been described. For the synthesis and characterization of oxybis(diacetoxyborane) derivatives of bifunctional tridentate aldimines, see (173).

Oxybis(diacetoxyborane), $((AcO)_2B)_2O$, reacts with dibasic tridentate SCHIFF bases in 1:1 and 1:2 molar ratio to yield $LB\text{-}O\text{-}B(OAc)_2$ and $(LB)_2O$ (where LH_2 = SCHIFF base (207).

The electric conductivity of aqueous and methanol solutions of boric acid complexes with salicylic acids have been studied (243). For the reactions of boric acid with oligo alcohols, see (244).

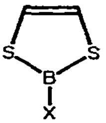
Complex formation equilibria in aqueous solution of boric acid and glycol have been studied (96) as were the interactions of boric acid and polar organic compounds (97). Boric acid can be extracted with 2-methyl-2,4-pentanediol (98), and the extraction with 2,6-dimethyl-4,6-octanediol can be applied in the analytical chemistry of boron (99).

The hydrolysis of $B(OCH_3)_3$ in organic solvents and in the presence of metal alkoxides appears to proceed in stepwise fashion (73); the slowest step is likely to involve the equilibrium $(ROB(OH)_3)^- + ROB(OH)_2 \rightleftharpoons (RO(HO)_2BOB(OR)(OH))^- + H_2O$. Association constants for the mannitol-borate and glucose-borate complexes have been determined by potentiometric titration and some infrared spectral data on such species have been reported (206). The syntheses and derivatographic as well as infrared spectroscopic studies on borate complexes of diols and polyalcohols have been reported (197) and the extraction of boric acid with 2,2-dipropyl- and 1,3-propanediol have been investigated (199). For complexes formed by boric acid with citric acid and metal ions, see (56). An X-ray crystallographic study of alkali metal borodicitrates has been reported (100).

The crystal structure of rubidium bis(p-aminosalicylato)-borate (200) and its hydrate (201) have been studied and complexes of boron with salicylic acid are described elsewhere (202). For the thermal decomposition of disalicylborates, see (206).

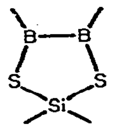
7 BORON-SULFUR DERIVATIVES

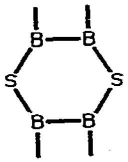
Various boron-substituted 1,3,2-dithiaboroles

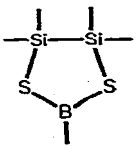


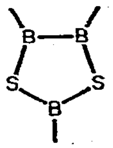
have been obtained by several different routes (182, see also 167). This new class of heterocyclic system can be considered as a 5 π-electron system, as is evidenced by NMR spectroscopic data.

The new ring systems









have also been obtained and several derivatives were characterized in detail (7).

Reaction of N-sulfinylamines with (methylthio)organylboranes proceeds quantitatively according to: $RN{=}S{=}O + 3\ R'_2BSCH_3 \longrightarrow RN(BR'_2){-}SSCH_3 + CH_3SSCH_3 + (R'_2B)_2O$; the process involves an insertion of the N=S bond into the B-S sequence and a complex redox reaction (85). The course of the reaction depends on steric factors as well as the reactivity of neighboring groups; this is illustrated by additional studies involving N,N'-disulfinyl diamines and N-sulfinyl sulfonamides.

Systems of the type $(RS)_nB(N(CH_3)_2)_{3-n}$ with R = CH_3 or C_2H_5 exist in equilibrium mixtures in which the presence of the unsymmetrical compounds with n = 1 or 2 can be demonstrated by NMR techniques (87); however, the pure species cannot be isolated. Carbon-13 chemical shift data have been reported for $C_6H_5B(SC_2H_5)_2$ and $(C_6H_5)_2BSC_2H_5$ (87).

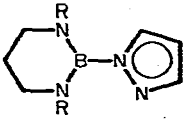
The crystal and molecular structures of the permethylated heterocycles containing the annular arrangements -NBSSB- and -SBNNB-, respectively, have been determined by X-ray diffraction (250); the annular atoms in both systems are essentially coplanar.

8 BORON-NITROGEN COMPOUNDS

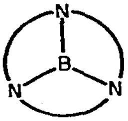
Trichloroborane chlorinates di- and trisilylamines containing at least one Si-H bond (52); however, depending on the reaction conditions, the silylaminoboranes $(R_3Si)(R_2SiCl)N{-}BHCl$, $(R_2SiCl)_2N{-}BH_2$ and $(R_2SiCl)(R)N{-}BHCl$ can also be obtained and were characterized (R = CH_3). **(N-Lithiomethylamino)dimethylborane**, $Li(CH_3)N{-}B(CH_3)_2$, has been employed to prepare a variety of N-functional (amino)-dimethylboranes of the type $X(CH_3)N{-}B(CH_3)_2$ with X =

BCH_3-$N(CH_3)_2$, ½ $Si(CH_3)_2$, $Si(CH_3)_2$-NCH_3-$Si(CH_3)_2Br$, $P(CH_3)_2$, $As(CH_3)_2$ (8), $HgCH_3$ and ½ Hg (9).

The first monomeric pyrazol-1-ylborane containing trigonal boron has been prepared (112); it appears that due to electronic effects, 1,3-dimethyl-2-(pyrazol-1'-yl)-1,3,2-diazaboracyclohexane does not form a dimeric pyrazabole structure.



Vibrational spectroscopic and NMR data support the existence of a linear C=N-B skeleton in monomeric iminoboranes of the type R_2C=N-BR'_2 (187). However, this allene-like arrangement of the central moiety of the compounds does not seem to enhance the N-B bond strength. On the other hand, the rigid C=N-BN_2 unit in $(CF_3)_2C$=N-$B(N(CH_3)_2)_2$ makes this compound colored and theoretical and experimental data on the electronic structure of the species are in good agreement. The effect of angle strain on hybridization has been studied on the four tricyclic trisaminoboranes of the general structure



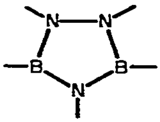
involving ethylene and/or trimethylene bridges (152). With increasing number of ethylene versus trimethylene bridges, a trigonal-planar array of the central BN_3 moiety is increasingly distortetd but a semblence of coplanarity is maintained by rehybridization of the nitrogen atoms into pyramidal geometry. In the case of all bridges being ethylene groups, the molecule exists as a dimer involving four-coordinate boron. The bisethylene-bridged species exists normally also as the dimer but the monomer prevails in the gas phase at high temperatures.

The vibrational spectra of several bis(dimethylamino)-haloboranes have been recorded and the proposed assignments of the fundamentals are substantiated by a normal coordinate treatment (162).

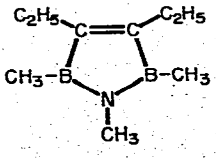
A new diborane(4) derivative is the species $B_2(t\text{-}C_4H_9)_2$-$(N(CH_3)_2)_2$ (1).

(Amino)haloboranes are intermediates in a one-step synthesis of 2-aminobenzhydrols from anilines and benzaldehydes with the aid of (dichloro)phenylborane and triethylamine (38).

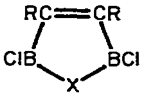
The reaction of bis(dimethylamino)phenylborane with 2-amino-3-hydroxypyridine, L-2-pyrrolidinylmethanol and 2-(o-aminophenyl)benzimidazole, respectively, proceeds with the elimination of dimethylamine to yield heterocyclic organoboron-nitrogen derivatives in all cases (131). Techniques have been developed for the preparation of derivatives of triazadiborolidines

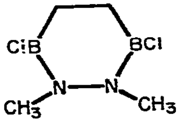


in which one boron atom is replaced by Si, P or As (91); NMR spectroscopic data of these novel ring systems are reported. The crystal and molecular structures of two 1,2,4,3,5-triazadiborolidines have been studied by X-ray diffraction (250). The 1,2,5-azadiborolene

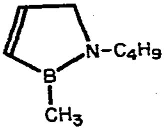


is obtained from the reaction of the corresponding S-containing organoboron heterocycle with $(R_3Sn)_2NR$ ($R = CH_3$) (142). The compound, L, reacts with transition metal carbonyls to form complexes such as $LFe(CO)_3$, $(C_5H_5)CoL$ and L_2Ni; the triple-decker complex LCoLCoL was also prepared. Reacting $X(SiR_3)_2$ ($R = CH_3$, X = S, NR, NR-NR) with Cl_2B-CR'=CR'-BCl_2 (R' = H, CH_3) and Cl_2B-CH_2-CH_2-BCl_2, respectively, yields the heterocyclic species (167):



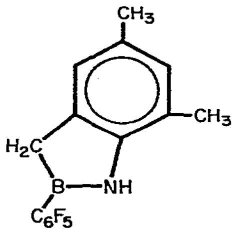


Most of the former can be B-alkylated by reaction with tetramethyltin. The dilithio salt LiCH=CH-CH_2-N(t-C_4H_9)Li interacts with CH_3BBr_2 to form the following heterocycle (218):



The latter species reacts with $Fe(CO)_5$ to give the dimer $(LFe(CO)_2)_2$. The Fe-Fe bond of this complex can be oxidized with I_2.

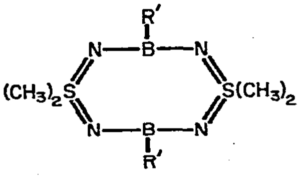
Thermolysis of gaseous RClB-NR'-$Si(CH_3)_3$ species at 500 to 600°C yields $(\text{-BR-NR'-})_n$ with n = 1, 2 or 3 depending on the nature of R and R' (5). In the case of R = C_6F_5 and R' = t-C_4H_9, the elusive monomer as well as the cyclic dimer was obtained. In solution the monomer is relatively stable and the B-N stretching mode of the species is assigned at 2045 cm^{-1}; the compound reacts as a typical dienophile. The steric requirements in the case of R = C_6F_5 and R' = mesityl lead to the formation of the following species:



The role of iodine in the photocyclization of (anilino)-dimesitylboranes has been studied and was found to be concentration-dependent (215).

The unsymmetrically substituted borazine $R_3B_3N_3R_2(SiR_3)$ with R = CH_3 has been characterized (8). A distinct coupling of the B-N and C-C stretching modes has been deduced from the infrared spectra of B-perdeuterophenylborazines (10). For the interpretation of the spectra of substituted borazines by UREY-BRADLEY force fields, see (151). Mass spectra of phenylated borazines exhibit strong peaks for doubly charged ions if the rings are coplanar with the B_3N_3 skeleton (10). Permethylated silaborazines have been obtained, e.g., from $(CH_3)_2Si(NCH_3Li)_2$ and CH_3BrB-NCH_3-$Si(CH_3)_2Br$, and may, in turn, be converted to the corresponding phosphaborazines (92).

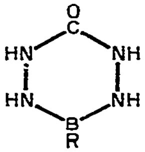
Reaction of $(CH_3)_2S(=NSiR_3)_2$ (R = CH_3) with $C_6H_5BCl_2$ yields the heterocycle



the crystal structure of which was determined (105). Reaction of $C_6H_5BCl_2$ with $O_2S(NR$-$SiR_3)_2$ (R = CH_3) gives a polymer of the composition $O_2S(NR)_2BC_6H_5$.

N-Borylated carbonamide derivatives and related species have received considerable attention. For example, N-trimethylsilylated ureas of the type RR'N-C(O)-NR"-Si$(CH_3)_3$ react with (diorganyl)haloboranes to give the N-borylated species with the elimination of trimethylhalosilane (186). An analogous reaction is observed with the corresponding thioureas (63). However, in some instances boryl group migration is observed to yield isothioureidoboranes, RR'N-C(=NR)-NR"-BR'", which may feature intramolecular N-B coordinate bonding.

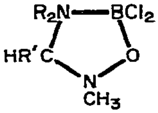
Condensation of carbohydrazide with borane(3) or bis(dimethylamino)phenylborane, respectively, leads to a BN_4C ring system (68):



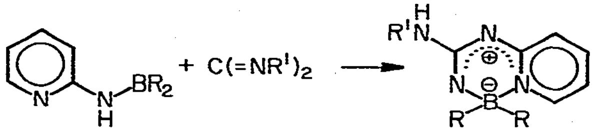
A series of linear N,N'-bis(boryl)hydrazines of the type $CH_3XB-NCH_3-NCH_3-BXCH_3$ (X = F, Cl, Br, SCH_3, $N(CH_3)_2$, CH_3) has been prepared and spectroscopic data on the species are reported (62).

A wide variety of sulfonamidoboranes, $RSO_2-NR^1-BR^2R^3$, has been synthesized and some characteristic spectroscopic data on the species are reported (113).

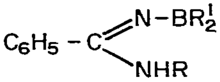
The 1,3-addition of (dimethylamino)dichloroborane to the aldimine oxide $C_6H_5CH=N(CH_3)O$ leads to an equilibrium of the reactants with the following heterocycle (135):



2-Pyridylaminoboranes react with carbodiimides in an exothermic process according to (263):

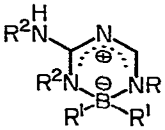


The resulting species are readily protonated with ethereal HCl. Reaction of trialkylboranes with N-alkylbenzamidines yields iminodialkylboranes of the type



$C_6H_5-C(=N-BR'_2)NHR$

which tend to dimerize (264). Reaction of these species with carbodiimides, $C(=NR^2)_2$, produces heterocyclic compounds:



9 LEWIS ACID-BASE ADDUCTS AND RELATED MATERIALS

Borane adducts of a variety of heterocyclic nitrogen bases such as chinoline and aminopyridines have been obtained by interaction of the base with dimethyl sulfide-borane (53); the anular nitrogen atom was always found to serve as the donor site.

Primary and secondary amine-boranes are mild, efficient and stereoselective reducing agents for aldehydes and ketones in protic or aprotic solvents (36); the reactivity patterns are different from those of diborane(6) or sodium tetradyroborate. A remarkable stereoselectivity has also been observed in the reduction of benzaldehyde and acetophenone with a variety of amine-boranes (37).

BF_3 but not B_2H_6 reacts with acyclic and cyclic (crown) polyethers to form LEWIS acid-base adducts (137). On the basis of thermochemical data it was shown that the acceptor powers decrease in the series BX_3 - BR_3 - R_2BOR - $RB(OR)_2$ - $B(OR)_3$ (118).

Triethylenediamine (TED) precipitates BF_3 from diethyl ether as $TED(BF_3)_2$ and as $TED-BF_3$ from tetrahydrofuran, whereas tetramethylethylenediamine (TMED) and TED precipitates BH_3 from either solvent as the 1:2 molar adducts (49).

2-Halogenated pyridines form 1:1 molar adducts with BH_3 and BBr_3, except in the case of the reaction between BBr_3 and 2-cyanopyridine (204); in this latter case, an insertion reaction yields bromo-(2-pyridyl)methylideneamino-dibromoborane, C_5H_4N-2-(CBr=N-BBr_2). Dimethylsulfide-trihaloboranes are reportedly convenient reagents for the dealkylation of aryl ethers (191).

Triarylboranes form 1:1 molar complexes with hydrazines (117); a correlation exists between the chemical stability and the nature and position of substituents on the aromatic nucleus of the triarylboranes. 1,3-Diphenylguanidine reacts with triarylboranes to give 1:1 molar complexes in which the boron is bonded to the imino group (165). And N,N-dimethyl acetamide forms a 1:1 molar complex with (dimethyl)bromoborane (163).

Tertiary aminecomplexes of trinitratoborane of the type L-$B(ONO_2)_3$ have been synthesized as stable, crystalline compounds (120).

The vibrational spectrum of acetonitrile-borane has been recorded and was analyzed (57). Also, the vibrational spectra of $(CH_3O)_3P-BH_3$ and deuterated derivatives thereof have been examined (84). Elsewhere, the vibrational spectra of $(CH_3)_3As-BX_3$ (X = Cl, Br, I) are reported and assignments to the fundamentals are suggested (205). The molecular structures of trimethylamine complexes of BCl_3 and BBr_3

have been determined by gas-phase electron diffraction (134).

Ammonia-carboxyborane, $H_3N\text{-}BH_2COOH$, the boron analog of glycine, has been prepared and X-ray diffraction data evidence a dimeric structure (208). Some phosphanealkyleneboranes, $R_3\overset{+}{P}CHR'\overset{-}{B}H_3$, have been obtained by reaction of ylids with THF-borane (83). The crystal and molecular structure of $(CH_3)_3\overset{+}{P}CH_2\overset{-}{B}H_3$ has been determined (108). Species of the type $(C_6H_5)_{3-n}(2\text{-}C_5H_4N)_nP\cdot BH_3$ with n = 1 to 3 have been prepared by reaction of the respective phosphine with $THF\cdot BH_3$; based on spectroscopic data the BH_3 group is bonded to the P atom rather than the pyridyl nitrogen (259). Bisborane adducts can be formed by certain bicyclic aminophosphanes in which the P atom is in a bridgehead position and bonded to one N and two O atoms (253). Crystal structure data confirm that the borane is bonded to P and N, evidencing an unusually strong basic character of the latter atom.

10 BORONIUM(1+) AND BORATE(1-) SPECIES

Bonzoate ester/BF_3/BCl_3 solutions in $CDCl_3$ or CH_2Cl_2 give low-temperature fluorine-19 NMR spectra which indicate the existence of $(ester)_2BF_2^+$ cations (4); for the cation $(hmpa)_2BF_2^+$, see (147).

Divalent boron cations of the type L_3BH^{2+} (where L is a pyridine and anions are Br^-, I^- or PF_6^-) have been obtained by nucleophilic displacement on $(CH_3)_3N\text{-}BHBr_2$ or pyridine-dihaloboranes (70). The displacement is controlled by favorable steric factors in and the basicity of the attacking amine and also by the nature of the displaced halogen (where iodine derivatives react faster than the bromine derivatives).

The reaction of trialkylboranes with t-butyllithium is a general and convenient method for the preparation of lithium trialkylhydroborates (41). The role of lithium trialkylhydroborate as a reducing agent in organic syntheses has been illustrated by a series of representative examples (18). The reagent has also been found to exhibit exceptional ability for the hydrodehalogenation of alkyl halides (25), and it effects a mild desulfurization of thiols (in the presence of transition metal chlorides) to yield hydrocarbons (129).

Lithium 1-alkynyltrialkylborates interact with methyl vinyl ketone in the presence of $TiCl_4$ in a MICHAEL-type reaction (44). Copper(I) 1-alkenyltrimethylborates couple with allylic bromides or 2-propinyl bromide (170).

The reaction of vinyltrialkylborates with iodine has been developed for a new synthesis of 1,1-dialkylethenes (74); and sodium methoxyalkenyldialkylborates react rapidly with $CuBr \cdot (CH_3)_2S$ via alkenylcopper intermediates to afford symmetrical conjugated dienes (21).

The coupling constant $^{11}B-^{13}C$ and several others in lithium tetramethylborate have been determined in 1,2-dimethoxyethane solution (192). Examination of the ^{13}C NMR spectra of trialkylallylborates indicates clearly the relative importance of sigma-pi conjugation between the CC double bond and the CB bond (143).

The crystal structure of the monoetherate of tetrameric sodium trimethylhydroborate consists of alternating Na and H atoms at the corners of a very distorted cube (236). For the vibrational spectra of some volatile metal tetrahydroborates, see (251).

Tetrabutylammonium tetrabutylborate has been employed as a reference electrolyte for the evaluation of single-ion conductances in nonaqueous solvents (17).

The reaction of $(CH_3)_3SnCF_3$ with BF_3 in 3:1 molar ratio yields, besides $F_3BCF_3^-$, the anion $F_2B(CF_3)_2^-$, which was isolated as its potassium and cesium salt, respectively (12). The crystal structure of $Cs(F_2B(CF_3)_2)$ has been determined by X-ray diffraction and the vibrational spectrum of the species has also been studied (114). The related anions $H_3BSiF_3^-$ and $H_2B(SiF_3)_2^-$ have also been reported (81). Reaction of $LiSi(CH_3)_3$ with various boranes leads to the formation of trimethylsilylborate anions such as $R'_3BSiR_3^-$, $R'_2B(SiR_3)_2^-$ and $B(SiR_3)_4^-$ (14).

Several ring-substituted potassium tetraphenylborates have been prepared (95) and the reagent properties of the tetrakis(3,5-dichlorophenyl)borate ion have been evaluated (94). Sodium tetraphenylborate effects the direct phenylation of tellurium species (223). In conjunction with vibrational spectroscopic studies, the crystal structure of ammonium tetraphenylborate (as determined by X-ray diffraction) gives evidence of hydrogen bonding (136). The crystal structure of acetylcholine tetraphenylborate has been determined by X-ray diffraction (101); the molecular geometry of acetylcholine in this salt differs from those observed in the bromide and iodide but agrees with that of the chloride. For ion pairing in solutions of sodium tetraphenylborate, see (239).

The first boronitroxides, $M^+(RN(O\cdot)BH_3)^-$, have been detected during the reaction of nitroso compounds with alkali metal tetrahydroborates (154); the reaction is likely to proceed via the radical anion $BH_3\cdot^-$.

Electrolysis of sodium cyanotrihydroborate in acetonitrile at an iron electrode yields $(CH_3CN)_4Fe(BH_3CN)_2$; but at Mo or V electrodes, the salt is oxidized to $Na(H_3BCNBH_2CN)$ (213).

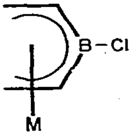
Fluorine-19 chemical shifts in some tetrafluoroborates have been determined (79). A molecular orbital study on XBH_3^-

species (X = Li, BeH, BH_2, CH_3, NH_2, OH and F) has been reported elsewhere (61).

Dihydroxyboranes, $RB(OH)_2$ with R = CH_3, C_6H_5, C_6H_4-3-NO_2 or OH, form 1:1 molar complexes with bidentate chelating agents such as oxalic acid, salicylic acid, mannitol or catechol according to: $RB(OH)_2 + H_2L \rightleftharpoons RB(OH)L^- + H_3O^+$ (46). The stability constants of such complexes were determined and were found to increase as the ligand and boron acid become more acidic. Thermal treatment of a mixture of biuret with boric acid leads to the formation of bis(biureto)boric acid, $H(B(-NHCONHCONH-)_2)$, the K, Cs, Tl and Ag salt of which are described (3).

11 METAL DERIVATIVES

(Chloro)divinylborane can be generated in situ from BCl_3 and $(CH_3)_2Sn(CH=CH_2)_2$ (39); the species reacts under photochemical conditions with $Fe(CO)_5$ or $C_5H_5Co(CO)_2$ by displacement of two CO groups to form complexes of the type

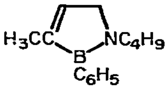


with M = $Fe(CO)_3$ or $Co(C_5H_5)$, respectively. The boron-bonded halogen can be replaced to give additional vinylborane complexes of the same type.

The complex $(CH_3B(-CH=CH-)_2BCH_3)Co(C_5H_5)$ has been synthesized and its structure was determined by X-ray diffraction (115). Complexes of the divinylborane type ML_2 with M = Pd, Pt, Ni and L = $X(-CH=CH-)_2BC_6H_5$ (X = $C(CH_3)_2$, $Si(CH_3)_2$, $(CH_2)_2$) have been prepared (221); based on NMR spectral data, the compounds have a sandwich-type bis(η^5-divinylborane)metal structure with C_2 molecular symmetry.

Reaction of $Co(C_5H_5BCH_3)_2$ with $Mn_2(CO)_{10}$ yields the species $(CO)_3Mn(C_5H_5BCH_3)$ in a ligand transfer reaction (214); the latter compound can be acylated to (2-acetyl-1-methylborinato)tricarbonylmanganese. A new synthesis of tetradecker sandwich complexes of the generalized type C_5H_5-Fe-SB_2C_2-M-SB_2C_2-Fe-C_5H_5 with M = Fe or Co originates from the species $((C_5H_5)Fe(C_2B_2S)_2)_2Zn$, which gives the sandwich anion $((C_5H_5)Fe(C_2B_2S))^-$ (157); the latter reacts with $FeCl_2$ or $CoCl_2$, respectively, to give the cited complex. X-Ray diffraction data for the Fe species are given.

The azaboroline (= L)



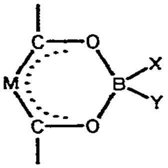
reacts with $Fe(CO)_5$ under formation of $(LFe(CO)_2)_2$ (107). The Fe-Fe linkage of the compound can be cleaved with iodine and the process results in the formation of $LFe(CO)_2I$; whereas reduction with metallic potassium gives $K(LFe(CO)_2)$, whose reaction with $(CH_3)_3SiCl$ results in the formation of $LFe(CO)_2Si(CH_3)_3$.

The reaction of lithium triethylhydroborate with $(C_5H_5Cr(NO)_2)_2$ yields, among other products, the species $(C_5H_5)_2Cr_2(NO)_3(C_2H_5B(C_2H_5)_2)$, in which one of the original bridging NO groups is displaced by a bridging triethylborane moiety; this result is confirmed by a X-ray structure determination of the species (257).

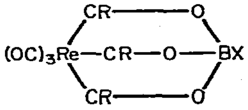
Various studies are concerned with polypyrazol-1-ylborate complexes of transition metals; these include derivatives of iron(II) (47), iridium (179), palladium(II) (104), rhodium (179), tungsten (220), titanium (260), and also thallium(III) (103).

Trihaloboranes form adducts with $(C_5H_5)Fe(CO)_2(COCH_3)$

in which the boron is bonded to the acetyl oxygen (88). This feature is not surprising, since various metal-β-diketonate complexes of boron of the type



with M, for example, $Re(CO)_4$, have been obtained by the reaction of the diketonates with trigonal haloboranes (34, 138). Similarly, triacetyltricarbonylrhenate dianion reacts with trihaloboranes to form neutral compounds where X = F, Cl, Br, I and R = CH_3 (178):



If X = Br, the halogen is readily displaced by alkoxy groups (178); and the crystal structure of the species with X = Cl has been determined by X-ray diffraction (139).

Bis(difluoroborondimethylglyoximato)nickel(II) forms a 1:1 adduct with 4,4'-dipyridine, the crystal structure of which was determined by X-ray diffraction (180). Zwitterionic tetraphenylborate – transition metal complexes have been described as stable homogeneous catalysts (43).

In $(\mu\text{-Br})(CO)_6(B_3H_8)Mn_2$, H-B-H bridges link the two Mn atoms (177). Several tris(methyldiphosphine) complexes of Ag(I) and Cu(I) containing BH_4 or $H_3BCO_2C_2H_5$ units have been prepared (51); the species involve a singly hydrogen-boron bridged structure. For the synthesis and structure of $H(BH_4)(P(CH_3)_3)_4Mo^{II}$, see (77). The crystal structure of $(CO)_2(CH_3)Fe(P(CH_3)_3)_2(CNB(C_6H_5)_3)$ has been investigated by

X-ray diffraction (166). Electrochemical and metathetical preparations of isomers of bis(cyanotrihydroborato)tetrakis(trialkylphosphite)iron(II) complexes have been reported (76).

12 PHYSIOLOGICAL AND RELATED ASPECTS

A voluminous book presents up-to-date references on the application of boron compounds in biology, pharmacy and medicine (188); and toxic action of boron on plants has been summarized elsewhere (132).

The biosynthesis of the boron-containing analog of aplasmomycin and NMR studies on the species have been described (265). Several borylated uracils and analogs thereof have been prepared, some of which were found to exhibit biological activity (145). The antiflammatory activity of amine-cyanoboranes, amine-carboxyboranes and related compounds has been described (189). Some (organyl)hydroxyboranes have been found to inhibit porcine pancreatic lipase (102), another one has been found to bind reversibly to cell walls (169).

Stable morphological changes have been observed after long-term treatment of Riccia fluitans with (dihydroxy)phenylborane (13). Another study (59) reports on the effect ofboron on the incorporation of glucose from UDP-glucose into cotton fibers grown in vitro (59). (Phenyl)hydroxyboranes have been reported as a new group of peptidyl transferase inhibitors (238).

Finally, it may be noted that boron mobility in the soil in relation to doses and forms of nitrogen fertilizers and the effect of boron on plant productivity have been studied (256).

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