BORON

ANNUAL SURVEY COVERING THE YEAR 1977*

PART II

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CONTENTS

1 REVIEWS AND SUMMARY ACCOUNTS

The NOBEL lecture by W. N. LIPSCOMB on the boranes and their derivatives has been published (106, see also 175). Also, a persona retrospective on boron hydride chemistry by A. B. BURG (156) is interesting reading.

Summary accounts on the preparation and application of organo-

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* Boron II, Annual Survey covering the year 1976 see J. Organometal. Chem., 147(1978)p.63-125.

boranes in organic syntheses (19, 54, 101), of cyclic coordination compounds of boron (102), the formation of boron heterocycles via cyclic hydroboration (115), and of the aminoboronation reaction (103) are available. A review of new varieties of sandwich complexes (29) makes reference to various boron-containing species, metal derivative of the borazines have been compiled elsewhere (41), and EISCH (245) describeds rearrangements of unsaturated organoboron compounds. Other surveys are concerned with recent developments in the chemistry of metal complexes of poly(1-pyrazolyl)borates (20), the use of catecholborane (107) and 9-borabicyclo(3.3.1)nonane (228) in organic syntheses, and some practical applications of boron derivatives (104, 105). A short article on boron-11 NMR spectroscopy may be of interest to the novice (140).

Dicarba-closo-dodecaboranes and their numerous derivatives are the subject of two new volumes of the GMELIN Handbuch der Anorganischen Chemie (11, 12) published within the New Supplement Series of the Handbook. (See some relevant remarks in J. Organometal. Chem., 119 (1975) 58.) Two additional volumes of the GMELIN Handbook are concerned with boron-oxygen derivatives. The first one (14) presents borate-polyol complexes in solution, $B(OR)_3$ species as well as derivatives of the B_2O_3 and B_3O_3 ring systems, whereas peroxyboranes, (organyloxy)organylboranes, diboryl oxides, and O-B-N heterocyclic systems are the topics of another volume (119). A fifth volume (16) is devoted to a presentation of selected boron-hydrogen species, particularly (organyl)hydroboranes, amine-trihydroboranes, and partially halogenated polyboranes. Nitrogen donor molecule adducts of (organyl)hydroboranes and trihaloboranes are the primary topic of a sixth volume (18).

2 SELECTED DATA OF GENERAL INTEREST

2.1 BORON-HYDROGEN SPECIES

A molecular beam velocity analysis spectrometry study (5) has provided unambiguous evidence for the generation of BH_3 in the pyrolysis of diborane(6). For a study of closo-boron hydrides containing 13 to 24 boron atoms, see (141); for a survey-type study of the reactions of catecholborane with functional groups, see (297).

The major advantage of the chloroborane-dimethylsulfides, which are being advocated as new hydroboration reagents (39), seems to reside in their chemical stability at room temperature (as compared to the corresponding etherates). Similarly, hydroboration utilizing dimethylsulfide-monobromoborane provides a general synthesis for the preparation of (dialkyl)bromoboranes and their derivatives (206). Dimethylsufide-dibromoborane shows a remarkable reactivity as a hydroborating reagent as compared to related dichloroborane derivatives (207). (The cited dimethylsulfide-haloboranes are readily obtained in a ligand exchange reaction between dimethylsulfide-trihaloboranes and dimethylsulfide-trihydroborane (260).) For new preparations of various borane reagents utilizing dimethylsulfide-borane, see (217); for the monohydroboration of silylated acetylenes, see (27).

The use of sodium (acyloxy)hydroborate (57) and tris(2-methoxyethoxy)hydroborate (81) as reducing agents has been described, and monoisopinocampheylborane has been shown to be a new chiral hydroboration reagent for relatively hindered olefins (274). Hydroboration of enehydrazones of the type$ROOCH{=}CCH_3{-}NCH_3{-}N{=}CHR'$ yields (organyl)dihydroxyboranes which are stabilized by internal N-B coordination (71). For the hydroboration of cyclic allenes, see (173).

For the reactions of tetra-n-butyldiborane(6) with acetals (73), dichloro ethers (143), alkyl α-chloroalkyl ethers (74), and 1,4-dialkoxy-2-butenyl ethers (76) see the indicated references. Triethylamine-thexylborane was found to react with representative olefins with displacement of tetramethylethylene and the formation of the corresponding triethylamine-monoalkylborane (200). For a study on the regioselective hydroboration of methylvinylchlorosilanes, see (197).

The reaction of B_2H_6 with isobutene under the influence of IR-laser irradiation proceeds different than in a thermal reaction (30). Though butylboranes are formed in either case, the laser-enhanced reaction occurs by activation of terminal BH groups. A study of reactions of diborane(6) with organic halides in the presence of metallic Li, Na, K, or Ca indicates the intermediate formation of organylborane species (193). For the reaction of B_2H_6 with some styrene oxides, see (146); for that with epoxides, see (147); and for the boronation of polystyrene, see (165).

The microwave spectrum of $CH_3B_2H_5$ has been studied (138) and the molecular structure of ClB_2H_5 was determined by gaseous electron diffraction (263).

The molecule $H_2P\text{-}BH_2$ is calculated by the ab initio MO-SCF-LCAO method to be most stable in the non-planar form, although a substantial degree of P-B multiple bond character appears to be evident for the linear arrangement (290). In this connection, attention is called to vibrational spectroscopic studies on the well-characterized $[(CH_3)_2P\text{-}BH_2]_3$ (285), derivatives thereof (220), and on $[(CH_3)_2P\text{-}BH_2]_4$ (229).

Finally, a qualitative study of the transition metal-catalyzed deuterium exchange at terminal B-H bonds (21) appears to be of general interest.

2.2 NMR STUDIES

Various coupling constants $^1J(^{11}BX)$ with X = ^1H, ^{13}C, ^{15}N, ^{19}F, ^{29}Si, ^{31}P, ^{77}Se, ^{119}Sn, and ^{207}Pb and chemical shifts $\delta^{11}B$ and δX have been determined for a number of trigonal boron compounds (72); as a rule the sign and order of magnitude of the coupling constants are determined by the FERMI contact term. The intensity of ^{13}C NMR signals of carbon atoms bonded to boron can frequently be increased by recording the spectra at low temperatures (8). Though this technique results in a loss of fine structure, it enables the determination of chemical shift data for NMR signals that are either broad or unobservable at ambient temperature. On the other hand, high temperature recording of spectra seems to be a useful means for resolving multiplet resonance signals that are collapsed in ambient temperature spectra. Boron-11 and ^{13}C chemical shift data on alkinylboranes containing the C≡C-B skeletal unit indicate π-electron back-donation from the CC triple bond into the CB bond (1). This interpretation is supported by ^{14}N NMR data on (alkinyl)aminoboranes. It is worth noting that the diamagnetic anisotropy of the CC triple bond is responsible for an additional high-field shift of $\delta^{11}B$ as compared with data on analogous alkenylboranes.

No simple correlation seems to exist between $\delta^{13}C$ of the phenyl carbon atom bonded to boron and $\delta^{11}B$ of phenylborane derivatives (8). Effects of simultaneous ^{13}C-^{1}H and ^{13}C-^{11}B decoupling on the ^{13}C NMR spectra of organoboron species have been studied and the absence of ^{13}C-O-^{11}B coupling was indicated (286). Finally, a new technique has been developed to study the reaction of trialkylboranes with oxygen directly in the

2.3 PHYSIOLOGICAL AND RELATED STUDIES

Complexing of some antibiotica with boric acid has been studied (129) and the pharmacological activity of 10,10-dihydroxy-10,9-boroxaphenanthrene has been evaluated (131). Elsewhere (77), an investigation of the interaction of $C_6H_5B(OH)_2$ with asp. melleus semi-alkaline protease is described.

Several reports deal with the effects of boron deficiency on plant growth (58 to 61, 306). Other studies are concerned with the effect of boron on cell elongation and division in squash roots (172) and on cereal yield and on nitrogen concentration in plant tissue in conjunction with limestone (174).

The distribution of tetraphenylborate ion across the membranes of γ-irradiated SMP has been investigated (56). For the tetraphenylboratometric determination of alkaloids, see (127).

3 TRIORGANYLBORANES AND RELATED SPECIES

Sodium triethyl-trimethylsilylethynylborate and triphenyl-trimethylsilylethynylborate, respectively, when treated with electrophiles form silylated (vinyl)diorganylboranes in which the diorganylboryl and trimethylsilyl groups are normally in cis-fonformation (282). For a convenient preparation of (alkynyl)-dialkylboranes, $R_2BC{\equiv}CR'$, by reaction of $LiC{\equiv}CR'$ with R_2BOCH_3 and subsequent treatment of the thus obtained ate complexes with $(C_2H_5)_2O{\cdot}BF_3$, see (10).

Sequential treatment of an ethynylalkanol acetate with n-butyllithium produces an allenic borane, $R_2B{-}CR{=}C{=}CR'_2$ (83); the latter may be protonated to form either an allene or an acetylene. Additional new triorganylborane species include dialkyl(p-alkylphenyl)boranes (120), boracyclopent-3-enes (24), $CH_3B(C_2H_5)_2$ and $(CH_3)_2BC_2H_5$ (9), and (1-stannocyclopenta-dienyl)dialkylboranes (202). Several new 1-boraadamantane derivatives have been described (238, 241) and were found to possess increased complexing ability as compared to trialkylboranes For a crystal structure study of B-hexamethyl hexaboraadamantane, see (294).

Triorganylboranes interact with monoalkynylstannanes under mild conditions by organoboronation of the triple bond (181). Rearrangement of 1-cyclopropylidenealkylboranes to homopropargylboranes has been described (223), and for the conversion of (alkenyl)dialkylboranes to olefins with $(C_2H_5)_3N \cdot Pd(OAc)_2$ at room temperature, see (291).

Numerous publications deal with the highly fashionable reagent 9-borabicyclo(3.3.1)nonane and derivatives thereof. The utilization of the reagent in organic syntheses (110, 165) includes the interaction of α,β-unsaturated aldehydes and ketones (135), the preparation of new derivatives of the basic skeleton (153, 170, 300), ate complexes (40, 109, 230, 296) and various reactions of such derivatives (25, 111, 130, 137, 205, 298, 299). Also, various complexes of 9-borabicyclo(3.3.1)nonane with tertiary amines have been described (264).

Two skeletal rearrangements of the 7-borabicyclo(2.2.1)heptadiene system have been described briefly; they involve (a) a degenrate suprafacial sigmatropic migration of the 7-substituted boron moiety, and (b) a nondegenerate anionic aryl shift from carbon to boron (195). The hydroboration of bicyclo(3.3.1)nona-2,6-diene yields a polymeric species of the composition $(C_9H_{14}BH)_x$ which contains three types of bicyclic fragments containing exocyclic BH groups (122). Similar results are obtained when tetraethyldiborane(6) is used as a hydroboration reagent. Pyrolysis of the polymeric species yields various 2-boraadamantane derivatives

Effects of temperature and reaction time in the pyrolysis of trimethylborane have been studied by gas chromatography and mass spectrometry (294). Two primary reactions compete in the photolysis of tris(1-naphthyl)borane (96). One is the expected simple boron-carbon bond cleavage but the second involves the formation of a monovalent organoboron species, $C_{10}H_7B:$, which can be viewed as a carbene analog. The intermediate formation of the corresponding methyl derivative, $CH_3B:$, has previously been postulated to occur in the reaction of CH_3BBr_2 with C_8K (121).

Oxidation of a solution of trimethylborane with air yields

$CH_3OB(CH_3)_2$ and $HOB(CH_3)_2$ as the major products (51); $CH_3OOB(CH_3)_2$ is observed as the reactive intermediate which provides for all of the identified final products. The B-C bond of organylboranes such as BR_3, R_2BOR', or $RB(OR')_2$ is smoothly protolyzed by NaOH in ethylene glycol at 135-200°C (123). Also, a new study of the known oxidation of organylboranes with trimethylamine N-oxide has been described (185).

Trialkylboranes react with NCl_3 by homolytic substitution at the boron center to yield alkyl chlorides (198); the fate of the boron moiety has not yet been determined. For a study of reactions of trialkylboranes with acyl carbanion equivalents, see (171). A facile electrochemical alkylation of 1-alkynes with organylboranes has been described (156).

For an experimental study of physical properties of triethylborane, see (55). Various physical data for a wide variety of organoboron species have been calculated by the MNDO method (279). Studies of the vibrational spectra of methyl and deuteriomethyl derivatives of boron in the CH/CD stretching region indicate a C-H bond strength variation with the internal rotation angle (114).

4 BORON-HALOGEN SPECIES

A facile high yield synthesis of CH_3BBr_2 rpoceeds according to (128):

$$N[Si(CH_3)_3]_3 + 3\ BBr_3 \longrightarrow 3\ CH_3BBr_2 + N[Si(CH_3)_2Br]3$$

(Dialkyl)bromoboranes are readily obtained in a hydroboration procedure utilizing dimethylsulfide-monobromoborane (206). 1,1,-3,3-Tetramethyl-1,3-disilacyclobutane interacts thermally with trihaloboranes, BX_3 (X = F, Cl, Br), to yield the ring cleavage product $X(CH_3)_2Si\text{-}CH_2\text{-}Si(CH_3)_2\text{-}CH_2\text{-}BX_2$ in almost quantitative yield (26). Thermal rearrangement for the species (except where

X = F) is observed at 180°C and is likely to proceed by an intramolecular mechanism. Ferrocene reacts with BX_3 (X = Br, I) to give (ferrocenyl)dihaloboranes and ferrocenium tetrahaloborates. The latter are also obtained from ferrocene, BX_3, and HX but are thermally labile and readily convert to (ferrocenyl)dihaloboranes (194). For a comparative study on (ferrocenyl)dihaloboranes, see (211).

(Pentamethylcyclopentadienyl)dichloroborane has a fluxional structure exhibiting sigmatropic rearrangement whereas the trimethyl amine adduct of the compound is a static molecule (33). Interaction of (pentamethylcyclopentadienyl)trimethylgermane with boron triiodide leads to the formation of (pentamethylcyclopentadienyl)-diiodoborane (301); the latter species readily converts to a monocarbon carborane.

The mechanism of the haloboration of n-hex-1-yne with BCl_3 or BBr_3, respectively, to yield (haloorganyl)haloboranes has been elaborated upon (192). For the reaction of (dialkenyl)chloroboranes with three molar equivalents of $CuCH_3$ to yield dienes, see (277).

The yield of interconversion of BCl_3 and $B(CH_3)_3$ under CO_2-laser irradiation to yield CH_3BCl_2 is dependent upon the laser frequency (148). In another study of laser-specific reactions involving boron compounds the trimerization of tetrachloroethylene sensitized by BCl_3 has been investigated (155).

Vibrational spectra and structures of $CF_2{=}CF{-}BF_2$ and $CF_2{=}CF{-}BCl_2$ have been studied and substituent effects of boron-substituted phenylboranes, $C_6H_5BX_2$ (including X = F, Cl), on the UV, proton NMR, and photoelectron spectrum of benzene have been compared with results of a modified CNDO/2 calculation (248).

Chemical shifts $\delta^{35}Cl$ of chloroboranes of the type $Cl_{3-n}BX_n$ (X = organyl, NR_2, OR, SR; n = 0, 1, 2) can be interpreted in terms of a B-Cl bond order greater than one (48); the absolute δ values are directly influenced by the additional boron substituents.

For the microwave spectrum, structure, and quadrupole coupling constants of $ClBF_2$, see (167); and for the molecular structure and barrier to internal rotation for B_2F_4, see (233). Also, the electronic structure of FBO has been calculated (154) and the vibrational spectrum of Cl_2BSH and that of Cl_2BSD has been recorded and the barrier of the species to internal rotation was evaluated (139).

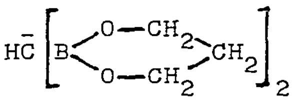
5 CHALCOGEN DERIVATIVES

Reaction of dichloromethyllithium with $B(OCH_3)_3$ yields $Cl_2HC\text{-}B(OH)_2$ (in impure form); the species is readily converted to corresponding linear and cyclic organyloxyboranes (187). The new (organyl)dihydroxyborane $C_6H_5\text{-}CO\text{-}NH\text{-}CH_2\text{-}B(OH)_2$ represents an example of an amino acid substrate where the carboxyl carbon atom has been replaced by boron (275); kinetic inhibition studies with α-chymotrypsin show the species to be a very powerful inhibitor. An allene-diene rearrangement has been observed in a study on the hydrochlorination of (3,3-dimethylallenyl)dihydroxyborane (237). The protolysis and a prototropic rearrangement of (cyclopentadienyl)dihydroxyborane and organyloxy derivatives thereof have been described briefly (6). The condensation of $C_6H_5B(OH)_2$ with pentane 1,5-diols yields oligomeric boron heterocycles rather than 1,3,2-dioxaboracyclooctanes (182).

The photochemical reaction of trialkylboranes in alcohol appears to be a convenient synthesis of 2-alkyl-1,3,2-dioxaborolanes (209). As shown in an X-ray diffraction study, the condensation product of D-mannitol with $C_6H_5B(OH)_2$ contains three five-membered heterocyclic moieties with the 1,3,2-dioxaborolane arrangement (125). Light-induced bromination has been used for the preparation of (α-bromoalkyl)organyloxyboranes (231).

Convenient methods have been developed for the synthesis of 1,2-oxaborinanes and 1,2-oxaborepanes based on the alkaline hydrolysis of the corresponding δ- or ε-bromoalkyldihydroxyboranes or their anhydrides (240); a detailed ^{11}B NMR analysis of the complexing ability og 1,2-oxaboracyclanes with nitrogen bases has been described (242). Additional heterocycles that have been reported include 2,1,3-boroxazines (227) and 1-aza-3,4-dioxa-2,5-diborolidines (97). For studies on the O-diethylborylation of carbohydrates leading to heterocyclic structures, see (302).

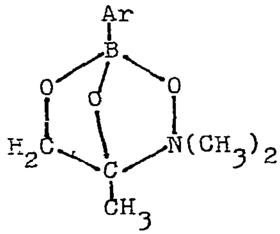
Deprotonation of gem-bis(trimethylenedioxyboryl)methane to yield the diboryl carbanion



has been described briefly (215). The species reacts with alkyl halides to yield the neutral which can again be deprotonated to achieve substitution of both methane hydrogens by alkyl groups.

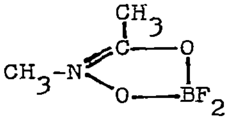
The mass spectra of bis(trimethylsilyloxy)organylboranes contain two rearrangement ions involving the loss of CH_3BO units (288); the mechanism postulated for the process is supported by deuterium-labelling experiments and metastable transitions. For NMR studies on (furanyl)dihydroxyboranes, see (272); for the protodeboronation and pK values of (furanyl)hydroxyboranes, see (161). A study of pK_a values indicates the existence of a ring-chain equilibrium for $(HO)_2B\text{-}C_6H_4\text{-}2\text{-}CH(CH_2C_6H_5)(2'\text{-}C_3N_2H_5)$ (271).

N-Oxidation of $(HOCH_2)_2CCH_3\text{-}N(CH_3)_2$ yields the ligand $(HOCH_2)_2CCH_3\text{-}N(O)(CH_3)_2$, which reacts with aryldihydroxyboranes to form betaines (52):

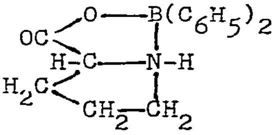


For photo-conductive charge-transfer boron diketonate compounds, see (221).

Crystals of (N-methylacethydroxamato)difluoroborane



are monoclinic (250); the structure features a planar five-membered BO_2CN ring with an O-B distance of 1.496 and 1.497 Å, respectively. The crystal structure of L-prolinato-diphenylborane has been determined by X-ray diffraction (249). The species consists of discrete molecules of the type



which are linked by NH···O hydrogen bridging. For a new study on the crystal and molecular structure of $C_6H_5B(OH)_2$, see (112).

Association reactions of $C_6H_5B(OH)_2$ (and of boric acid) with OH^- ion have been studied by a temperature-jump rate method (224). For the deuterio deprotonation of some borazarothienopyridines, see (100), and for the kinetics and mechanism of hydrolysis of 2-phenyl-1,3,2-benzodiazaborole, see (136).

The controlled reaction of $THF \cdot BH_3$ with carboxylic acids gives access to acyloxyboranes, $H_{3-n}B(OCOR)_n$ with n = 1, 2, 3 (274). Cinnamyl derivatives of the types $B(OR)_3$, $[(RO)_2B]_2O$, and $(-BOR-O-)_3$ with $R = CH_2CH{=}CHC_6H_5$ have been prepared from B_2O_3 and cinnamyl alcohol (90); infrared and ^1H NMR spectral data of the species are presented. Boron derivatives of the SCHIFF bases derived from the condensation of 1-phenyl-1,3-butanedione with hydroxy alkylamines or arylamines have been obtained from the interaction of $B(OC_2H_5)_3$ with the SCHIFF bases (91). $B(OSO_2CF_3)_3$ in excess trifluoromethanesulfonic acid is comparable in acidity to $SbF_5\cdot3SO_3$ in HSO_3F, the latter being the most acidic system known until now (268).

Careful interaction of $B(OCH_3)_3$ in CH_3OH with $NaOCH_3$yields crystalline $Na[B(OCH_3)_4]\cdot CH_3OH\cdot1.5H_2O$ (188) and perhydrolysis of the species has been utilized for the preparation of alkali and alkaline earth metal peroxoborates (180). For an electron spin resonance study on $B(OCH_3)_3$ that indicates the existence of a novel σ-radical with a one-electron bond, see (208). Solvent properties of the $B(OCH_3)_3/CH_3OH$ azeotrope have been studied (78 to 80) and glycol-borate complexes were investigated in aqueous medium (53). For the kinetics of the trimethoxyboroxine-induced thermal polymerization of phenylglycidyl ether, see (42); for the kinetics of the reaction of boric acid with salicylic acid, see (210). Polarographic studies of the interaction of bori acid with 1-hydroxy- and 1,2-dihydroxy-9,10-anthraquinones and anthrahydroquinones have also been reported (226).

The heterocycle 3,5-dimethyl-1,2,4-trithiodiborolane, $S(-BCH_3-S-)_2$, is readily cleaved by primary amines (44). Subsequently, either intramolecular cyclocondensation occurs with the formation of 1,2,4-dithiaazaborolidines, $RN(-BCH_3-S-)_2$, or else borazines, $(-BCH_3-NR-)_3$, as well as 1,3,5-thiadiazatriborines, $S(-BCH_3-NR-)_2BCH_3$, are obtained in an intermolecular process. The distribution of products is governed by steric and electronic effects of the amine reagent. For the hydroboration of alkenes and alkynes with 1,3,2-dithiaborolane, see (232).

The He(I) photoelectron spectra of some thioboranes of the type $XC_6H_4SB(\underline{n}-C_3H_7)_2$ with X = 4-Cl, 3-Cl, 4-Br, H, $4-CH_3$, $3-CH_3$, $4-OCH_3$ have been recorded and were assigned (196); the interaction between the phenyl ring of the species and the SBR_2 group is briefly discussed.

X-Ray diffraction studies on the species B_2S_3 reveal that it consists of B_2S_2 and B_3S_3 rings that are linked by S bridges to form almost planar infinite high polymer layers (31). For the crystal structure of bis(diethylamino)dithiaboratane, a compound containing a four-membered ring system of alternating boron and sulfur atoms, see (116).

Heating metathioboric acid, $(-BSH-S-)_3$, with elemental sulfur yields the species $H_2S\cdot xBS_2$, whereas diiodotrithiadiborolane, $S(-BI-S-)_2$, reacts with H_2S under formation of $H_2B_2S_5$ (254); the latter species decomposes and polymerizes spontaneously in the solid state to yield $H_2S\cdot xBS_2$.

6 BORON-NITROGEN COMPOUNDS

Various syntheses of aminoboranes containing relatively unusual N-substituents have been reported. For example, N-alkyl-ω-cyanoalkylamino(trimethyl)silanes have been reacted with bis-(dimethylamino)chloroborane and (halo)diorganylboranes, respectively, to yield N-alkyl-N-ω-cyanoalkylaminoboranes, e.g., $(NCCH_2)(CH_3)N-BR_2$ (85); the reaction of N-substituted thioamides with bromoboranes was found to yield thioamidoboranes, $(R'CS)(R)N-BR''_2$, which are monomeric in the gas phase (86); and new (phosinoamino)boranes of the type $(R_2P)(R')N-BR''_2$ have been prepared by reaction of silylaminophosphines with haloboranes (47). (2-Pyrimidylamino)dialkylboranes have been prepared by condensation of 2-aminopyrimidine with trialkylboranes (68), and B and/or Si halogenated (silylamino)boranes are readily obtained from Si-halogenated disilazanes and haloboranes (50), as is illustrated by the following equation:

$$[(CH_3)_2Si]_2NCH_3 + R_2BX \longrightarrow (CH_3)_2SiX_2 + X(CH_3)_2Si-NCH_3-BR_2$$

Such species can also be obtained from the reaction of $[-Si(CH_3)_2-NCH_3-]_3$ with haloboranes by degradation of the Si-N heterocycle.

(Dimethylamino)bromoboranes, $(CH_3)_2N\text{-}BRBr$, react with $Hg(CH_2COOCH_3)_2$ to yield either $(CH_3)_2N\text{-}BR(CH_2COOCH_3)$ ($R = C_6H_5$) or $(CH_3)_2N\text{-}BR\text{-}C(OCH_3){=}CH_2$ ($R = N(CH_3)_2$) or mixtures of both types of species ($R = CH_3$, $\underline{i}\text{-}C_3H_7$) (251); the vinyl derivatives rearrange irreversibly to the 2-oxoethylboranes which, in turn, generally undergo thermal decomposition with the formation of CH_2CO and $(CH_3)_2N\text{-}BR(OCH_3)$. (Diorganyl)bromoboranes (or tetra-alkyldiboranes) react with N-trimethylsilylamides under formation of amidoboranes, $(RCO)R'N\text{-}BR''_2$ (190). Similarly, N-trimethylsilylamides react with bis(amino)haloboranes to yield monomeric amido-bisaminoboranes (108). Such species are also obtained from the interaction of N-substituted acyl amides with bis(amino)-haloboranes in the presence of triethylamine (108); amido-bis-(alkyloxy)boranes can be prepared in similar fashion. The reaction of N-substituted haloacetamides generally leads to the formation of amidoboranes (84); in some instances, carbiminoxyboranes and bis(amido)boranes are also obtained.

The mixed amino-hydrazinoboranes $(\text{-}CH_2NCH_3\text{-})_2B\text{-}NH\text{-}N(CH_3)_2$, $[(\text{-}CH_2\text{-}NCH_3\text{-})_2B]_2N\text{-}N(CH_3)_2$, and $(\text{-}CH_2\text{-}NCH_3\text{-})_2B\text{-}NH\text{-}NH\text{-}B(\text{-}NCH_3\text{-}CH_2\text{-})_2$ exhibit relatively high thermal and chemical stability, which featu appears to be due to the incorporation of the boron into the heterocyclic system (2).

Reaction of (dimethylamino)organylhaloboranes with AgCN yields $(CH_3)_2N\text{-}BR(CN)$ (95); thermal treatment of the latter ($R = C_6H_5$) leads to partial symmetrization, the species with $R = CH_3$ readily oligomerizes. (Dimethylamino)silylphosphinoboranes such as $[(CH_3)_2N]_2B\text{-}P[Si(CH_3)_3]_2$, the corresponding diphosphino species as well as a cyclic derivative have been described (64).

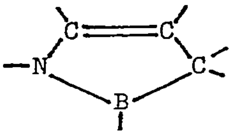
Rotational barriers about the $(CH_3)_2N\text{-}B$ bond of various bis(amino)boranes have been determined by variable ^1H NMR studies (4). The resultant barriers range between 10 to 20 kcal/mol and are discussed primarily in terms of steric interaction between the dimethylamino and the second amino group. The ^{79}Br NQR spectra of (diorganylamino)dibromoboranes have been recorded and a correlatior was found to exist between the ^{79}Br NQR frequency and the length of the N-bonded hydrocarbon chain (246). For additional NQR studies on some species containing N-B bonds, see (222). X-Ray diffraction data on $(Cl_2B\text{-}NH_2)_3$ indicate that the molecules show a nearly ideal chair conformation with a B-N distance of 1.575 Å (189). The two pyrazole rings of 2,6-dibromo-4,4,8,8-tetraethylpyrazabole are coplanar (126); the B-N distances of the species (about 1.7 Å) are somewhat longer than those found in other (1-pyrazolyl)boranes and each boron has one long and one short B-C(ethyl) bond with 1.75 and 1.48 Å, respectively.

Bis(diphenylboryl)amine has been prepared by the ready symmetrization of $(CH_3)_3Si\text{-}NH\text{-}B(C_6H_5)_2$ and of $(CH_3)_2B\text{-}NH\text{-}B(C_6H_5)_2$ (49). In contrast, the species $(CH_3)_3Si\text{-}NCH_3\text{-}B(C_6H_5)_2$ is stable toward rearrangement whereas the diborylamine $(CH_3)_2B\text{-}NCH_3\text{-}B(C_6H_5)_2$ rearranges near 50°C to yield $C_6H_5B(CH_3)_2$ and $(\text{-}BC_6H_5\text{-}NCH_3\text{-})_3$. The thermal stability of diborylamines is significantly increased by N-substitution with bulky groups such as $Si(CH_3)_3$ (252); this feature permits the isolation of $(CH_3)_3SiN(BBrCH_3)_2$ from the reaction of hexamethyldisilazane with CH_3BBr_2. The boron-bonded bromine of the diborylamine can then be replaced by other groups such as OCH_3 or SCH_3 while maintaining the diborylamine moiety. Stepwise cleavage of the Sn-N bond of $N(SnR_3)_3$ (R = normally CH_3) by (diorganyl)bromoboranes ultimately yields tris(diorganylboryl)-amines (43). Based on NMR data, there is an extremely low π-charge density at the boron atoms of the species with $\delta^{11}B$ ranging from approximately -61 to -68 ppm (low field).

N-Chloro-2-propane imine reacts with trimethylborane to yield dimeric (isopropylidenimino)dimethylborane (93); the dimeric structure involves a B_2N_2 ring system as is substantiated by vibrational spectroscopic data. Monomeric (ketimino)diorganylboranes are readily obtained by interaction of ketimines with (organylthio)-diorganylboranes (235, 236). Imine-enamine tautomers have been observed and dimerization is facilitated by aromatic boron substituents (235), particularly if the latter contain methyl groups in the ortho-position (236). Diimino- and trisiminoboranes can similarly be prepared from imines and (organylthio)boranes (234) and the interaction of aminoboranes or alkylarylketiminoboranes with alkylarylketimine hydrochlorides was found to yield cationic complexes with imines as ligands about boron (239). The reaction of alkylarylketiminoboranes with nitriles yields boron β-diiminates, the structures of which were elucidated by spectroscopic methods (243). The species hydrolize in acidic medium with the formation of either boron β-diketonates or β-diketones, whereas interaction with HCl or HBF_4 yields boronium salts with β-diimines as ligands (243).

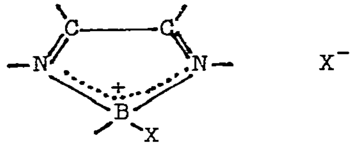
1,2-Bis(alkylamino)disilanes react with dichloroboranes to form a five-membered BN_2Si_2 heterocycle (62). Various 1,2,4-triaza-3,5-diborolidines containing the NB_2N_2 ring system have also been described (46). The species 3,5-difluoro-1,2,4-trimethyl-1,2,4-triaza-3,5-diborolidine has been obtained by fluorination with SbF_3 or BF_3 of the corresponding B-methylthio or B-dimethylamino derivative, respectively (45); the compound exists as an unusual dimer, the formation of which involves fluorine migration. Thermolysis of sterically hindered borylamino compounds of the type $X(NCR_3-BR'_2)_2$ with X = PCH_3, $P(S)CH_3$, $Si(CH_3)_2$, etc., and R = CH_3, leads to the exclusive formation of four-membered heterocycles involving the XN_2B arrangement as annular unit (143).

(Enamino)dialkylboranes complex with t-C_4H_9CN at 20°C (17); on heating of the adducts, a rearrangement occurs with the formation of 1,2-azaborolidines containing the following skeletal unit:



For the heats of conbustion of some 1,2-dialkyl-1,2,azaborolidines, see (178).

Monomeric 1,3-dimethyl-2-cyano-1,3,2-diazaborolidine has been described (95) and 1,3,2-diazaborolium salts of the type

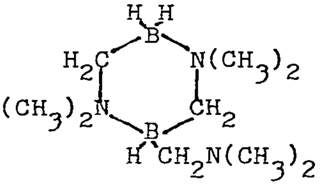


have been obtained from the reaction of α,β-diimines with (organyl) dihaloboranes (70); in some instances such salts could be reduced to yield 1,3,2-diazaborolines. For a detailed study on the transmission of substitutent effects through 1,3,2-diazaborolidines by NMR techniques, see (216). Interaction of $C_6H_5B(-NC_2H_5-CH_2-)_2CH_2$ with C_6H_5NCO yields an eight-membered heterocycle in an aminoboronation process; the species exhibits a clearly resolved ^1H NMR spectrum which is interpreted by utilizing spectral data on some related compounds (269, 270).

Reaction of $(CH_3)_3N \cdot BH_3$ with aminopyridines results only in the formation of 1:1 molar adducts of the latter with BH_3, in which the pyridyl nitrogen serves as the donor site (89). However, 2-aminomethylpyridine reacts with trimethylamine-borane under analogous conditions to yield $2\text{-}(H_2B\text{-}NH\text{-}CH_2)C_5H_4N \cdot BH_3$ (89). (Pentamethylcyclopentadienyl)-bis(dimethylamino)borane has been prepared and was found to have a static structure at 40°C (33); at higher temperature, however, a fluctional structure is observed for the species. (2-Pyridylamino)dialkylboranes react with formamide in an aminoboronation process (13). Similarly, (2-pyridylamino)diphenylborane reacts with isocyanates and isothiocyanates in a 1,2-addition of the pyridylamino and diphenylboryl moieties of the aminoborane across the CN bond of the organic species (88). In all these aminoboronation products the boron is in four-coordinate environment due to π-bonding with the pyridyl nitrogen; a coordinated cyclic structure results.

Photolysis of a mixture of 2-aminopyridine and (diphenyl)-chloroborane in the presence of iodine yields 10-phenyl-8,9-diaza-10-boraphenanthrene (92). 2-Trifluoroacetylamino benzonitrile reacts with haloboranes to yield derivatives of 1,3,2-diazaboranaphthalene (84). The reaction of N-2,4,6-trichlorophenyl-trifluoroacetamide (and similar N-acylated species) leads, however, to the formation of derivatives of the 2,6,9-trioxa-4,8-diaza-1,5-diborabicyclo(3.3.1)nonadiene (87). Monomeric crystalline borazaropyridines containing B-H units do not show reducing power nor are they active as hydroborating reagents (142). This behavior supports the assumption of heteroaromatic character for such compounds. For the synthesis and some NMR studies of 4,5-borazarofuro(2.3-c)pyridines, see (82); some electronic properties of 2-methyl-2,1-borazaronaphthalene have been evaluated (159).

A series of compounds featuring a common structural sequence of four-coordinate BCN moieties has been described (247); this includes linear as well as cyclic neutral compounds and the difunctionally substituted hydroborate ion $[(CH_3)_2N-CH_2-BH_2-CH_2-N-C_2H_4-BH_2-CH_2N(CH_3)_2]^-$. The species



has been isolated as a product from the reaction of bis(dimethylamine)dihydroboronium chloride with n-butyllithium in the presence of glyme (113); the exocyclic nitrogen reacts as a typical tertiary amine but with methylene chloride the $CH_2N(CH_3)_2$ group is replaced by $CHCl_2$.

On the basis of kinetic studies (273), steric factors are most important for the rate of ring ligand displacement in the reaction of $(-BR-NR'-)_3Cr(CO)_3$ with tertiary phosphites. Also, steric effects in the synthesis of _ortho_ and _para_ isomers of unsymmetrically substituted borazines have been commented upon, utilizing results of the chlorination of 1-methylborazine with various halogenating agents as the experimental basis (258). A study of the infrared and NMR spectra of B-monosubstituted borazines reaches the conclusion that the N-bonded hydrogen atoms of the species do not lend themselves to hydrogen bonding (259). Preliminary data of a X-ray diffraction study on B-trichloro-N-triphenylborazine suggest that the ring and anular-bonded atoms are all coplanar (212); however, steric interaction of the phenyl groups results in their being tilted out of the central plane by 77 to 87°. Mercury-photosensitization of alkylborazine/hydrogen mixtures leads to products identified as dimers of an intermediate radical formed by hydrogen abstraction from the alkyl group (7); hexamethylborazine reacts with hydrogen atoms by H/CH_3 exchange at the boron sites. B-Tri-_n_-butylborazine forms heat-resistent copolymers with N-monosubstituted unsaturated carboxylic acid amides (75).

7 ACID-BASE ADDUCTS

The microwave spectrum of $OC\cdot BH_3$ has been recorded and structure, quadrupole coupling constants, and dipole moment of the species were determined (168). Mass spectral data on the interaction of $OC\cdot BH_3$ with O and N atoms indicate that the molecule is destroyed by O atoms via two initiation steps, one in which the O abstracts H from the boron, another one wherein the O attaches to boron directly by ejecting CO (284). Also, a

MO study of the bonding in $OC \cdot BH_3$ has been reported (162).

New synthetic approaches to $H_3N \cdot BH_3$ and its deuterated derivatives have been described (184). For a MO study of the bonding in the molecule, see (162); and for a new study of the vibrational spectrum and force field of $(CH_3)_3N \cdot BH_3$, see (179). The microwave spectrum and structure of methyl isocyanide-borane have been studied (217) and the molecular structure of the adduct $N(-CH_2CH_2O-)_3P \cdot BH_3$ has been determined by X-ray diffraction (264) and was found to be in consonance with NMR data. Borane(3) complexes of (methoxy)trifluoromethylphosphines are described elsewhere (265) and methylamino-bis(difluorophosphine) (= L) complexes of the composition $L \cdot BH_3$, $L \cdot (BH_3)_2$, and $L \cdot B_3H_7$ have been characterized by spectroscopic techniques (261).

A NP_2B_2 ring system is the backbone of the new species metyhlamino-bis(difluorophosphine)-diborane(4), which is obtained from treatment of bis(trifluorophosphine)-diborane(4) with methylamino-bis(difluorophosphine) at -23°C (214). For the vibrational spectrum of tetramethyldiphosphine-bisborane, see (160); for that of trimethylarsine-borane, see (149); and for some comments on phosphine-monohaloborane species, particularly ^{11}B NMR data, see (213).

The acid-catalyzed hydrolysis of $(CH_3)_3N \cdot BH_2N_3$ has been studied (262) and trimethylamine-isocyanoborane, the first known tetrahedral boron isocyanide, has been described elsewhere (163). Reactions of the latter with Lewis acids such as BH_3 or BCl_3 result in the formation of the corresponding adducts (23); boronium cations are easily formed by the boryl isocyanide displacement of the iodide from several amine-iodoboranes.

Tris(o-alkyloxyphenyl)boranes form 1:1 molar complexes with 1-(2-aminoethyl)piperazine, in which the primary amino group serves as the donor site (244). However, in the presence of a 300% excess of the borane, 2:1 molar adducts are formed in which both nitrogen atoms of the LEWIS base engage in coordination.

Infrared data on solid mixtures of BF_3 and NO suggest the formation of addition compounds at low temperatures (3); it is not known whether O or N serve as donor sites in such species. The molecular structures of the complexes of dimethylamine and dimethyl ether with BF_3 have been studied (157) and on the basis of a microwave spectral study of $(CH_3)_3N \cdot BF_3$ the B-N distance of the species was determined to be 1.636 Å (263). A total of 19 isotopic species of various adducts of the type $(CH_3)_3N \cdot BX_3$ (X = F, Cl, Br, I) have been studied by solid state infrared spectroscopy (289) and electron diffraction studies on gaseous $(CH_3)_3N \cdot BF_3$ and $(CH_3)_3N \cdot BCl_3$ show staggered structures for the species with a B-N distance of 1.664 and 1.659 Å, respectively (218). The vibrational spectra of $(CH_3)_3P \cdot BX_3$ (X = Cl, Br, I) have been recorded and assignments to the fundamentals are supported by a normal coordinate analysis of the species (255). Reactions of BH_3 with a ligand containing both an As and a P donor site have been studied (292); the ligand $(C_6H_5)_2PCH_2CH_2As(C_6H_5)_2$ readily displaces AsR_3 from $R_3As \cdot BH_3$ (R = CH_3, C_2H_5, C_6H_5) due to the superior basicity of P as compared to As. Some thermodynamic data on the complex formations are reported.

Pyridine froms 1:1 molar complexes with triallylboranes but tetraethyl ethylenediamine forms 1:1 and 1:2 adducts (15); bipyridyl (= L) reacts with triallylborane with the formation of the boronium salt $[R_2BL_2]BR_4$. For a series of adducts of the type <u>trans</u>-$HPtL_2NC \cdot BR_3$ (L = triethylphosphine), see (22); the species readily convert to the thermodynamically more stable <u>trans</u>-$HPtL_2CN \cdot BR_3$ isomers.

8 BORATE SALTS AND RELATED SPECIES

Ab initio calculations on $[H_3BNC]^-$ and $[H_3BCN]^-$ indicate substantial differences in their electronic distributions (38). For the reduction of indoles emplying Na$[H_3BCN]$, see (281). The unusual complex $[Na\{H_3N\cdot BH_2(CN)\}_6]I$ has been prepared in liquid NH_3 and the structure of the species has been studied by single-crystal X-ray analysis (295).

LiH reacts with trimethylborane or triethylborane in ether solvents to form Li$[HBR_3]$ as monoetherates (253). The species can be decomposed by thermal treatment and the thus obtained "activated" LiH reacts with various trialkylboranes in the absence of solvent to form lithium trialkylhydroborates. In THF or glyme solvents, LiH reacts even with two moles of $B(CH_3)_3$ to form Li$[(CH_3)_3BH\text{-}B(CH_3)_3]$ (276). For the reduction of alkyl methanesulfonate esters with lithium triethylhydroborate, see (169).

Data accumulated in studies of the fluorine resonance spectrum of an equimolar solution of BF_3 and BrF_3 in SO_2FCl are best interpreted by a complex of the type $[BrF_2][BF_4]$ (181). Boron trichloride reacts with AgCN or CuCN to yield the respective metal tetracyanoborate (63); both salts appear to be chemically extremely stable. Alkali metal tetrachloroborates react with SO_3 in liquid SO_2 with the formation of mixed chlorosulfatoborates (152). The enthalpies of formation of K$[B(ClO_4)_4]$ and $B(ClO_4)_3$ have been determined (34).

The structure of K$[CF_3BF_3]$ has been determined by X-ray diffraction and the vibrational spectrum of the salt was re-examined (256). The formation of $[R_3BF]^-$ and $[R_2BF_2]^-$ species in reactions of SF_5^- or SF_6^- with neutral boranes, BR_3 with R = CH_3, C_2H_5, <u>i</u>-C_3H_7, or F, has been studied in trapped-in cyclotron resonance techniques (257). Sodium triphenylcyanoborate has been advocated as a selective reagent for separating ^{137}Cs in environmental samples (166). For the crystal structure of tributylammonium tetraphenylborate monohydrate, see (305).

The preparation of trialkylvinylborates has been desribed and reactions of the species with oxiranes and with iodine have been studied (177). For the reaction of trialkylvinylborates with aldehydes, see (176, 203, 204). The reaction of alkynyllithium reagents with (methoxy)diorganylboranes yields species of the type $Li[R_2B(OCH_3)(C\equiv CR'')]$ (10); these salts react with $(C_2H_5)_2O \cdot BF_3$ to form (alkynyl)dialkylboranes, $R_2BC\equiv CR'$, in essentially quantitative yield. For the reaction of lithium ethoxycarbonyl- or aroylacetylamide with trialkylboranes and subsequent treatment of the thus obtained borate species with elemental iodine, see (225). Trialkylboranes react with ethoxyethynyllithium to yield lithium ethoxyethynyltrialkylborates, $Li[R_3^-BC\equiv COC_2H_5]$ (124).

Bis(trans-1-hexenyl)chloroborane was converted to bis(trans-1-hexenyl)methylborane by reaction with $LiCH_3$ and was subsequently treated with two molar equivalents of $CuCH_3$ (150); the experimental results, i.e., formation of a dodecadiene species, are interpreted in terms of an intermediate formation of 1-hexenylcopper(I) and conversion of the boron moiety to trimethylborane. For the coupling reaction of copper(I) methyltrialkylborates with aroyl chlorides, see (280). Copper(I) methyltrialkylborates react with ethyl propiolate to form addition products which are readily hydrolized to the α,β-unsaturated esters in good yield (151); for the reaction of copper(I) methyltrialkylborates with ethyl β-bromo acrylates, see (99); and for the reaction of the same reagents with allylic halides, see (304).

The tetraphenylborate ion promotes the hydrogenation of certain planar rhodium(I) cations (134). The conductimetric behavior of sodium tetraphenylborate in H_2O/CH_3OH mixtures at 25°C has been studied (133).

9 METAL DERIVATIVES

The structure of gaseous $(C_5H_5)_2TiBH_4$ has been studied by electron diffraction (118); the cyclopentadienyl rings of the species appear to take an orinetation of relatively high symmetry with regard to each other and the Ti-B axis with a distance of 2.31 Å between the latter two atoms.

Cyanotriphenylborate complxes such as $[(CH_3)_5C\ RhL]$-$[NCB(C_6H_5)_3]$ have been synthesized and were characterized by spectroscopic and X-ray structural techniques (199); the borate group is N-bonded to the central metal atom. For the preparation and properties of di-μ-cyanotrihydroborate-bis(2,2',2"-triaminotriethylamine)dinickel(II) tetraphenylborate, see (28). Reaction of manganese pentacarbonyl bromide with trimethylamine-isocyanoborane results in displacement of one CO group by the amine-borane (303).

Acetonitrile is readily displaced from $(CH_3CN)_3M(CO)_3$ (M = Cr, Mo, W) by $(-BC_6H_5-S-)_3$ in dioxane solution to yield complexes of the type $(-BC_6H_5-S-)_3M(CO)_3 \cdot 1.5C_4H_8O_2$, in which the metal is coordinated to the B_3S_3 ring (201). Utilizing a 1,3,2-diazaborolene, L, in an analogous reaction, the air-stable complex $LCr(CO)_3$ was obtained in dioxane at 85°C (32). Similarly, 3,4-diethyl-1,2,5-thiadiborolenes react with $Fe_2(CO)_9$ to give complexes of the type $S(-BX-CC_2H_5=)_2Fe(CO)_3$ (191). A triple-decker sandwich complex in which three thiadiborolene rings are complexed with two Co atoms has been obtained by reacting the ligand with $Co_2(CO)_8$ in toluene (35). Substitution of THF by (methylthio)-diorganylboranes in $(OC)_5Cr \cdot THF$ yields the rather unstable complexe $(OC)_5Cr \cdot CH_3SBR_2$ with R = CH_3 or C_6H_5 (186). Similarly, the borane C_4H_2S-2-CH_3-4-$B(C_2H_5)_2$ interacts with $(OC)_5Cr \cdot THF$ by displacement of THF by the borane; however, the resultant complex is quite stable due to interaction of the boryl group of the borane ligand with CO ligands (186). The thermal reaction of the species $(=CC_2H_5-BC_2H_5-)CHCH_3$ (= L) with nickelocene yields the sandwich complex $(C_5H_5)NiL$ as an orange-red air-sensitive material (144).

Paramagnetic complexes of the type $Cr(C_5H_5BR)$ with R = CH_3 or C_6H_5 have been prepared by the reaction of potassium borinate, K $[C_5H_5BR]$, with $CrCl_3$ or $CrCl_2 \cdot THF$ (66); the complexes seem to belong to the 16-electron species involving a sandwich structure. Similarly, 1-phenylboracyclodienes, $X(-CH=CH-)_2BC_6H_5$ with X = $(CH_2)_2$, $Si(CH_3)_2$, or $C(CH_3)_2$, obtained from the corresponding stannocyclodienes by reaction with $C_6H_5BCl_2$, readily function as pentahapto ligands in complexes of iron and cobalt (65). Borinato complexes of the types $LMn(CO)_3$, $LRe(CO)_3$, and FeL_2, where L is the 4-methyl-1-phenylborinato ligand, are obtained by thermal reaction of 4,4-dimethyl-1-phenyl-1-bora-2,5-cyclohexdiene with $Mn_2(CO)_{10}$ or $Re_2(CO)_{10}$, respectively, or by thermolysis of $LFe(CO)_3$ (67). The reaction of 1-phenyl-4,5-dihydro-1H-borepin (= L) with $LiC(CH_3)_3$ and $[Rh(1,5\text{-}C_8H_{12})Cl]_2$ yields $LRh(1,5\text{-}C_8H_{12})$ (69); ^{11}B NMR data suggest the existence of a B-Rh bond in the species. Pentaphnelyborole, L = $C_4B(C_6H_5)_5$, reacts with $Fe_2(CO)_9$ to give $LFe(CO)_3$ and with $Ni(CO)_4$ to yield $LNi(CO)_2$ (36); additio borole sandwich complexes of the nido type are reported.

Crystal structures of the two newly prepared complexes $(C_3H_5)Mo(CO)(pz_3BC_6H_5)$ and $(C_7H_7)Mo(CO)_2(pz_3BC_6H_5)$ (pz = 1-pyrazolyl) have been determined (37) and the infrared spectra in the 650 to 1150 cm^{-1} range of several transition metal poly(1-pyrazolyl)borates have been recorded (284).

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