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BORON

ANNUAL SURVEY COVERING THE YEAR 1978

Part II

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CONTENTS

1 REVIEWS AND SUMMARY ACCOUNTS

The use of alkenyl-, alkynyl- and cyanoboates as synthetic intermediates in organic syntheses has been surveyed (134) and industrial uses of boron compounds have been reviewed (130).

Boron, Part II; Annual Survey covering the year 1977 see J. Organometal. Chem., Vol. 163 (1978)59-96.

Other summary accounts deal with transition metal complexes of the borabenzene anion (60) and adducts of the mixed trihalides of boron (59). An impressive compilation of ^{11}B NMR data has been assembled by NÖTH and WRACKMEYER (20).

A detailed presentation of borazine and its derivatives is given in a new volume of the New Supplement Series of the GMELIN Handbook (62); for some relevant remarks see J. Organometal. Chem. 119 (1975) 58. Another volume of the handbook (213) deals with small boron hydrides that contain up to four boron atoms, and boron-halogen species are the topic of a third new volume (214).

2 SELECTED DATA OF GENERAL INTEREST

No simple correlation exists between the boron-11 and carbon-13 chemical shifts of the boron-bonded phenyl carbon atom of phenylborane derivatives, although stereochemical factors appear to influence the absolute values of the carbon-13 chemical shifts (28). Not surprisingly, the ^{13}C NMR signal of the boron-bonded carbon atom of phenylboranes is more readily detected on recording the spectrum of a neat compound rather than a diluted species (157).

Isolated reports are appearing in the literature which are concerned with the laser-induced chemistry of boron compunds. One such report describeds that of diborane(6) which leads to the formation of polyboranes (236); surprisingly, this does not proceed via a chain mechanism and no $B_{20}H_{16}$ was detected among the reaction products. For studies of the laser chemical reaction of BCl_3 with $SiCl_4$, see (61).

A study of the electrochemical behavior of some heterocyclic organoboranes (250) as well as a report on the properties of some tetraphenylborate complexes of transition metals (206) may also be of general interest. For the scope of the reaction of organylboranes with lithium derivatives in order to synthesize lithium organylborates as well as the stability of the latter, see (255).

Dialkylboranes have been used for a stereoselective synthesis of 1,2-dialkylated ethenyl bromides by monohydroboration (131). For the hydroboration of carbonyl compounds employing dimethyl sulfide-borane, see (145).

Boron can be determined cerimetrically as borotartartrate (138).

3 TRIORGANYLBORANES AND RELATED SPECIES

Trisalkenylboranes have been prepared from diolefins via organylaluminum derivatives (194). The hydroboration of 1-halo-1-alkynes with dialkylboranes followed by treatment with t-butyllithium proceeds readily at -78°C to afford stereochemically pure cis-alkenylboranes in excellent yields (90). Highly reactive alkyl-allyl-boranes have been prepared by allylation of R_2BXR' (X = O, S) or $RB(XR')_2$, respectively. using allylic derivatives of aluminum, magnesium or boron in exchange reactions (42). The resultant unsymmetrical triorganylboranes are thermally stable up to 100°C but undergo a permanent allylic rearrangement. Under the influence of ROH, the B-allyl bond is preferentially attacked. Reaction of the alkyl-allyl-boranes with carbonyl compounds occurs at the same site (43). Alkylation of $B(OCH_3)_3$ or $B(SCH_3)_3$ with $t\text{-}C_4H_9Li$ occurs stepwise with the formation of $B(t\text{-}C_4H_9)_3$ as the final product (40). The intermediates $t\text{-}C_4H_9B(SCH_3)_2$ and $(t\text{-}C_4H_9)_2BSCH_3$ can be isolated. Tris-t-butylborane isomerizes at temperatures above 130°C to yield a mixture of butylboranes.

Ferrocenyldibromoborane reacts with 1,1,4,4-tetramethyl-1,4-distanna-2,5-cyclohexadiene to yield 1,4-bis(ferrocenyl)-1,4-dibora-2,5-cyclohexadiene (221); the ferrocenyl moieties can be displaced by controlled methanolysis of the species. Organoboration of dialkenyldimethylstannanes with trialkylboranes has been used for the preparation of 1-stannacyclopentadienes that are dialkylborylated in the 3-position (160). Alkynyltrimethylstannanes react with 1-organyl-1-boracyclopentanes to yield 1-bora-cyclohexylid-2-enes as the only product (166). Treatment of $R_2Sn(C{\equiv}CCH_3)_2$ with trialkylboranes gives 1-bora-4-stannacyclohexadienes or 2,5-bis(dialkylboryl)-1-stannacyclopent-3-enes (47).

For the preparation of 9-deuterio-9-borabicyclo(3.3.1)-nonane, see (204); for that of 9-borabicyclo(3.3.1)nonanylamines, see (201). 9-Borabicyclo(3.3.1)nonane and similar bulky diorganylboranes such as disiamylborane add cleanly to vinyltrimethylsilane with high regiospecifity to give the corresponding β-silylethylboranes (103).

An improved synthesis of monoisopinocampheylborane has been developed (38) and the preparation of a species of high optical purity has been described (202). Diphenylborane, $(C_6H_5)_2BH$, which can be prepared by the reaction of $(C_6H_5)_2BOCH_3$ with $LiAlH_4$, reacts readily with olefins to yield alkyl-diphenylboranes (93).

For a study of the reaction of trialkylboranes with lead tetraacetate or phenyliodoso acetate to give alkyl acetates, see (227). Trimethylcyclopropene reacts with triallylboranes in two ways (122). The major pathway is the addition of the allyl and diallylboryl moieties to the cyclopropene double bond to form a substituted 2-allylcyclopropylborane, The second pathway involves cleavage of the C^2-C^3 single bond of the ring to give substituted 1,5-heptadiene-4-ylborane.

The electrochemical reaction of trialkylboranes with acetone has been described (132). Electrolysis of trialkylboranes in the presence of α,β-unsaturated esters has been developed as a procedure for the preparation of the corresponding alkylated carboxylic acid esters in good yield (225).

For the oxidation of triorganylboranes with excess pyridinium chlorochromate to yield ketones, see (222). Protonolysis of alkenylboranes under neutral conditions by treatment with catalytic amounts of palladium diacetate, see (261). Carbonylation of trialkylboranes using $Li(HAl(OCH_3)_3)$ provides the intermediate $Li(R_2B\text{-}CHR\text{-}OAl(OCH_3)_3)$; acid treatment of this species under mild conditions causes a second alkyl group migration from boron to carbon (174).

Thermal decomposition of 1,1-bis-trimethylstannyl)-2-dialkylborylalkenes leads to allenes in a deorganylborylation reaction (77):

$$2\ ((CH_3)_3Sn)_2C{\equiv}CR(BR_2) \longrightarrow$$

$$BR_3 + ((CH_3)_3Sn)_2C{=}C{=}CR\text{-}C(BR_2)(Sn(CH_3)_3)_2.$$

Activated triethylborane has been used for the quantitative determination of hydroxyl groups in lignites (114). Triethylborane organoborylates the multiple bond of alkynyltrimethylstannanes and the reaction can be utilized for the synthesis of substituted allenes (107). Triethylborane interacts with 2-isopropyl-6-methyl-4-pyrimidone with the elimination of ethane to yield ethyl-bis(2-isopropyl-6-methyl-4-pyrimidyloxy)borane (123); the latter is a new reagent suitable for the ethylborodiylation.

The interaction of trialkylboranes with the dianion of phenoxyacetic acid has been utilized for the synthesis of carboxylic acids (147). Trialkylboranes can be oxidized with the complex $MoO_5 \cdot OP(N(CH_3)_2)_3$ to yield, upon hydrolysis, the corresponding alkanols (165). Trialkylboranes have been utilized to synthesize α-hydroxysilanes by the interaction of trimethyl(dibromomethyl)silane with lithium and trialkylboranes in tetrahydrofuran (158). Triethylalkynylborates, $((C_2H_5)_3BC{=}CC_6H_5)^-$, interact with sulfonyl chlorides to yield compounds of the type $(C_2H_5)_2B\text{-}CC_2H_5{=}CC_6H_5\text{-}SO_2R$ and the structure of the species with R = $CH_2C_6H_4$-4-NO_2 has been studied by X-ray diffraction (258). It was found that one of the S-bonded oxygen atoms coordinates with the boron to produce a heterocyclic moiety. Chloramine-T and some of its analogs react with triorganylboranes to give N-substituted sulfonamides, most likely via aminoborane-type intermediates (17).

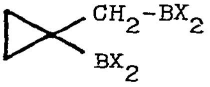
Pyridine-9-borabicyclo(3.3.1)nonane is a very mild but highly selective reducing agent (70). For the hydroboration of 3-butenyl derivatives with 9-borabicyclo(3.3.1)nonane, see (99), and for the use of 9-bromo-9-borabicyclo(3.3.1)-nonane as reagent for ether cleavage, see (100).

The deboronation of α-(phenylthio)alkylboranes by N-chlorosuccinimide has been studied (96); the procedure provides for a convenient synthesis of monothioacetals and acetals.

For a study of the vibrational spectrum of tris(perfluorovinyl)borane, see (54). Also, a study of the vibrational spectra of cis and trans 1,2-dimethyldiborane(6) has been reported (183). The NMR spectra of (cyclopentadienyl)dimethylborane and (pentamethylcyclopentadienyl)dimethylborane indicate fluxionally behavior for the species (109). Ion cyclotron resonance techniques have been used to determine the acid-base properties of 1-methyl-1,4-dihydroborabenzene (126). The resultant data suggest that there is little interaction between the diene π-system and the electron-deficient boron.

4 BORON-HALOGEN SPECIES

The reaction of KBF_4 with $AlBr_3$ to yield BBr_3 has been used for the preparation of the boron-10 labelled compound (173). The interaction of $Si(CH_3)_4$ with trihaloboranes at elevated temperatures is readily directed to yield either CH_3BX_2 or $(CH_3)_2BX$ (X = Br, I) in virtually quantitative yield (5). Boron trichloride interacts with $((CH_3)_3Si)_2CHLi$ to yield the species $(((CH_3)_3Si)_2CH)_2BCl$ (46); the chlorine of the haloborane derivative is readily displaced by groups such as NH_2, $N(CH_3)_2$ or OH. Addition of B_2X_4 (X = F, Cl) to the double bond of methylenecyclopropane yields the diboryl species



which is formed even in the presence of excess B_2Cl_4 (50). 3-Hexyne interacts with BI_3 with the formation of a cis-trans mixture of $(C_2H_5)(I_2B)C{=}CI(C_2H_5)$ (9); the mixture reacts with $(-BI-S-)_3$ to yield the heterocycle $S(-BI-CC_2H_5{=})_2$, the iodine of which is readily displaced by various other substituents.

The BCl_3-catalysed reaction of benzene with $HSiCl_3$ to yield $C_6H_5SiCl_3$ is likely to proceed via phenylchloroborane intermediates (74). For the cationic polymerization of styrene with boron trihalides, see (265).

The vibrational spectra of organyl-iodoboranes RBI_2 and R_2BI with R = CH_3, CD_3, C_2H_5 and C_6H_5 have been studied; a complete assignment of the vibrational modes of the methyl derivatives is proposed (243). A detailed spectroscopic analysis of ethyldifluoroborane has been described (36) and the vibrational spectrum of methyldifluoroborane and isotopically labelled derivatives thereof has been studied (37). Also, the microwave spectrum of phenyldichloroborane has been reported (136) and the species FHBOH has been detected by microwave spectroscopy (133). For base adducts of CH_3BBr_2 and $(CH_3)_2BBr$, see (260).

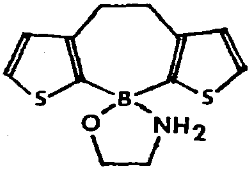
5 CHALCOGEN DERIVATIVES

Alcoholysis of (diethylamino)dialkylboranes has been described as a convenient route to yield (organyloxy)-dialkylboranes (92). The species $HB(OH)_2$ has been detected by microwave spectroscopy (133) and the interaction of trifluoroacetic acid with THF·BH_3 was found to produce $HB(O\text{-}CO\text{-}CF_3)_2$·THF (144); this latter reagent is acid-stable and can be used in hydroboration reactions. Diborane(6) reacts with aryltin or aryllead compounds to yield arylhydroboranes which, after hydrolysis, are isolated as the corresponding (aryl)hydroxyboranes (75). (Phenyl)dihydroxyborane, $C_6H_5B(OH)_2$, has been found to undergo condensation reactions with various silanes to yield several novel heterocyclic systems comprised of B, O, Si, and (sometimes) N as anular atoms (78).

Several 1,1-bis(trimethylenedioxyboryl)-1-alkenes and 1,1-bis(ethylenedioxyboryl)-1-alkenes have been prepared by the reaction of ketones with the corresponding lithium triborylmethide (161). On treatment of 1,1-bis(trimethylenedioxyboryl)-2-phenylethene with methyllithium and subsequent oxidation phenylacetone is obtained, indicating methyl migration from boron to carbon (164).

Bis(dialkylboryl) oxides, $(R_2B)_2O$, interact with 2-amino- and 2-hydroxypyridines at elevated temperatures with the release of alkane to give species of the type $(C_5H_4N)\text{-}2\text{-}Z\text{-}BR\text{-}O\text{-}BR_2$ (Z = O, NH) in which the BR_2 group is back-coordinated to the pyridyl nitrogen atom (16). N-Methylacethydroxamic acid reacts with bis(diphenylboryl) oxide in 2:1 molar ratio to yield $CH_3\text{-}CO\text{-}NCH_3\text{-}O\text{-}B(C_6H_5)_2$ (124); X-ray crystallographic data show the molecule to exist in a coordinated cyclic structure.

The crystal and molecular structure of 4-methyl-2-phenyl-1,3,5,2-oxadiazaboroline has been studied by X-ray diffraction (190); with the exception of the N-O bond, all bonds were found to have significant π-character. The crystal structure of



has been studied and the B-N bond distance was found to be 1.635 Å (191).

Treatment of 2-(α-bromoalkyl)-1,3,2-dioxaboracycloalkanes with organolithium or Grignard reagents results in essentially quantitative replacement of bromine by an organic moiety (69). Metathetic ring-ring equilibria in 1,3,2-dioxaboracycloalkane systems have been described (57) and the mass spectra of 2-(p-substituted)phenyl-1,3,2-dioxaborolanes have been discussed (170). Mass spectral and ^{1}H NMR data on boroxines, $(\text{-BR-O-})_3$, and trioxadiborolanes, $O(\text{-BR-O-})_2$, give some evidence for aromatic character of these heterocycles. Several new 1,3,5,2-oxadiazaboroles have been prepared (137), and for the enthalpy of formation of 2,8,9-trioxa-5-aza-1-boratricyclo(3.3.3.0)undecane, see (58).

Reaction of α-benzil monoxime with BF_3 yields a 1:1 molar adduct but with BCl_3, BBr_3 or $C_6H_5BCl_2$ one halogen is displaced as hydrogen halide with the formation of the corresponding oxyborane derivatives (179).

Bis(organyloxy)-α-phenylthioalkylboranes, $(RO)_2B\text{-CHR}'(SC_6H_5)$, are readily deprotonated to give boron-containing carbanions of wide synthetic utility (151). The conversion of (aryl)dihydroxyboranes to the cyclic pinacol derivatives facilitates their characterization (216) and catecholic amino acids and catecholamines have been separated by chromatography on immobilized (phenyl)dihydroxyborane (189). Flavanoids can de betermined by spectrophotometry using (2-aminoethyloxy)diphenylborane (185). (3,5-Dichlorophenyl)dihydroxyborane and (3-nitrophenyl)dihydroxyborane have been used to derivatize difunctional species (amines, alcohols) and to study gas chromatographic properties of the resultant heterocycles (259).

The compound $(C_6H_5)_2B$-OCH_2CH_2-NH-CH_2CH_2OH is stabilized by intramolecular N-B coordination; ^1H and ^{13}C NMR spectroscopic studies indicate three temperature-dependent reversible exchange processes to occur in solution, i.e., proton exchange between OH and NH, nitrogen inversion, and ligand exchange (119). Spectroscopic (149) as well as chemical data (150) illustrate that diphenylboryl chelates of 1,5-diphenylcarbazone are derived from the enol form.

A facile and convenient synthesis of organyloxyboranes involves the reaction of methylsulfide-borane with alcohols or phenols (56). Perhydrolysis of tris(alkyloxy)boranes, $B(OR)_3$ with R = CH_3, C_3H_7 or C_4H_9, in the presence of inorganic or organic bases yields non-crystalline peroxopolyborates that contain active oxygen as B-O-O groups (6). Nuclear magnetic resonance studies of $B(OCH_3)_3$ in methanol solution indicate fast methoxyl exchange with a large deuterium solvent isotope effect (192). For forbidden reflections in the X-ray diffraction of boratran, $B(OC_2H_4)_3N$, see (252).

Esterification of adamantyl-containing silanols with boric acid yields tris(1-adamantylsilyloxy)boranes (34). Reaction of $CH_3CN \cdot BCl_3$ with tetraphenylporphine affords the corresponding chloroboryl complex, chromatography of which on alumina results in ligand exchange producing a tetrahydroxydiboron porphyrin (256). For cyclic boron derivatives obtained from the interaction of BF_3 with N-alkylated amino acids, see (248); and for BF derivatives of N-substituted salicyl aldimines, see (200). Bis(difluoroboron dimethylglyoximato)nickel(II) and its 1:1 aniline adduct have been described (199) and tetracoordinated Schiff base complexes of boron have been prepared by reacting boric acid and the Schiff base in acetic anhydride medium (247). Schiff base derivatives derived from 2,4-pentanedione and containing a tetracoordinated (diacetyloxy)boryl moiety have also been described (35). For boron complexes of tridentate Schiff bases, see (184, 203). The dipole moments of boron chelates with tropolene and 1,3-diketone derivatives have been studied (205). For sodium pentaerythritoldiborate, see (263), and for the synthesis of a boron chelate of 1-methyl-4,5,6,7-tetrahydroindigo, see (262). Bis(para-amino salicyl)-borates have been described alsewhere (117, 118).

The monomeric reactive molecule $SBCH_3$ has been detected in the products of a high temperature reaction between CH_3SSCH_3 and crystalline boron (110). Tris(ethylthio)borane readily transfers alkylthio groups from boron to carboxylic acids (64); the reaction pathway is complex and involves the intermediate formation of oxybis(diacylboranes).

A cis-trans mixture of $(C_2H_5)(I_2B)C{=}CI(C_2H_5)$ interacts with $(-BI-S-)_3$ in boiling hexane to yield the heterocycle $S(-BI-CC_2H_5{=})_2$ (9); the boron-bonded iodine is readily displaced by such substituents as Br, Cl, OCH_3, SCH_3, $N(CH_3)_2$ or CH_3 with retention of the SB_2C_2 heterocyclic framework.

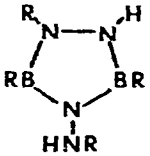
Tris(phenylseleno)borane, $B(SeC_6H_5)_3$, has been found to be a useful reagent for the conversion of aldehydes or ketones into selenoacetals (108).

6 BORON-NITROGEN COMPOUNDS

Efficient syntheses of several tris(diorganylamino)-boranes have been described (24). The stereodynamics of enantiomerization in bis(diisopropylamino)aminoboranes, $((i\text{-}C_3H_7)_2N)_2BNRR'$, has been studied by NMR techniques (152) as has the stability and stereochemistry of some bis(trimethylsilyl)aminoboranes (234). A detailed NMR study on the interaction of tris(diethylamino)borane with PCl_3, $OPCl_3$ and

$SPCl_3$ indicates that the formation of mixed speceis is more favored than is based on a random mechanism (139); individual trends were interpreted on the basis of the Lewis acidity of boron and the effective electronegativity of the phosphorus moieties. The compound $(CF_3)_2C=N-B(N(CH_3)_2)_2$ has been synthesized and its spectroscopic properties were found to be in consonance with those of other compounds of the general structure $(CF_3)_2C=N-BR_2$ (23). The central C=N-B skeleton of these species is virtually linear indicating substantial analogies to allenes due to N-B back-bonding.

Mixed amino-hydrazino-boranes have been prepared by the reaction of 2-chloro- or 2-methylthio-1,3,2-diazaboracycloalkanes with hydrazines (27). The compounds are apparently stabilized by the incorporation of the boron into an anular system; compounds such as $(-CH_2-NCH_3-)_2B-NH-N(CH_3)_2$ were described. The solvolysis of $CH_3B(SCH_3)_2$ with $H_2N-N(CH_3)_2$ proceeds via $CH_3B(SCH_3)(NH-N(CH_3)_2)$ to yield $CH_3B(NH-N(CH_3)_2)_2$ as the ultimate product (8). In contrast, reaction of $CH_3B(SCH_3)_2$ with $CH_3NH-NHCH_3$ leads to the six-membered heterocycle $CH_3B(-NCH_3-NCH_3-)_2BCH_3$ or to the linear molecule $CH_3S-BCH_3-NCH_3-NCH_3-BCH_3-NCH_3-NCH_3-BCH_3-SCH_3$, which decomposes at 160°C with the formation of $CH_3S-BCH_3-NCH_3-NCH_3-BCH_3-SCH_3$. $CH_3B(SCH_3)_2$ and $H_2N-NHCH_3$ interact with the exclusive formation of the following heterocycle:



R = CH_3

1,2-Diorganylhydrazines react with either $RB(SCH_3)_2$ or $(CH_3)_2B_2S_3$ to yield cyclic 1,2,4,5-tetraaza-3,6-diborines (51). The species $RB(-NCH_3-NCH_3-)_2BR$ with R = SCH_3 or $N(CH_3)_2$, which are readily obtained from the reaction of equimolar quantities of BR_3 and N,N'-dimethylhydrazine, are very susceptible to subsequent displacement reactions at the boron sites (120). All species containing the B_2N_4 ring system exhibit a distinct tendency to dimerize.

Diaminoborane, $HB(NH_2)_2$, has been prepared by passing ammonia through molten $H_3N \cdot BH_3$ and the compound was characterized by spectroscopic data (253). Amino-bis(trimethylsilyl)aminoboranes of the type $((CH_3)_3Si)_2N\text{-}BR\text{-}NH_2$ exhibit surprising thermal stability, which event is correlated to the stereochemistry of the species (81). A number of (silylamino)boranes containing bulky t-butyldimethylsilyl groups have been described (82). The colorless liquids are frequently thermally unstable and decompose at or above room temperature with the formation of borazines.

(Diethylamino)dialkylboranes are readily prepared by ligand exchange between trialkylboranes and tris(diethylamino)-borane (92); the reaction is catalyzed by the presence of BH species. The compounds $(C_2H_5)_2B\text{-}N((CH_2)_3\text{-}NH\text{-}B(C_2H_5)_2)_2$ and $HN((CH_2)_3\text{-}NH\text{-}B(C_2H_5)_2)_2$ have been obtained from the reaction of $(CH_3)_2N\text{-}B(C_2H_5)_2$ with $HN((CH_2)_3NH_2)_2$ (26). The reaction of borane(3) with the latter amine yields BH_2B-bridge associated $H_2B\text{-}N((CH_2)_3\text{-}NH\text{-}BH_2)_2$ and cyclic condensation products thereof depending on the reaction conditions and the stoichiometry of the reactants (21). The reaction of $B(N(CH_3)_2)_3$ with the same amine yields 1,8,10,9-triazaboradecalin and the 1-bis(dimethylamino)boryl derivative thereof (21).

(Dimethylamino)diethylborane reacts with either 1-trimethylsilylimidazole or imidazole under formation of (1-imidazolyl)diethylborane (10). The compound exists as an oligomeric species and is chemically quite inert. (Pyridylamino)dialkylboranes interact with the carbonyl group of aldehydes or ketones by 1,2-addition across the multiple bond (13); the resultant species from a coordinated cyclic structure (see also ref. 19).

The Lewis acidity of the $(CH_3)_2BN$ group as compared to the proton acidity of the NH group increases in the series $(CH_3)_2B\text{-}NHR$ with R = $N(CH_3)_2$, $Si(CH_3)_3$, $B(CH_3)_2$; this is evidenced in a study on the N-metallation of the cited species by interaction with lithium organyls (193). The N-lithiation of $(CH_3)_2B\text{-}NHCH_3$ by organolithium reagents, LiR, is characterized by a solvent-dependent competition between the formation of $(CH_3)_2B\text{-}NLiCH_3$ and of $Li((CH_3)_2BR\text{-}NHCH_3)$, although the latter compounds decompose slowly with the formation of the desired $(CH_3)_2B\text{-}NLiCH_3$ (39).

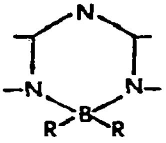
The molecular and crystal structures of the compounds $(4\text{-}Br\text{-}C_6H_4)(CH_3)N\text{-}BCl(C_6H_5)$ and $(2\text{-}CH_3\text{-}4\text{-}Br\text{-}C_6H_3)(CH_3)N\text{-}BCl(C_6H_4\text{-}4\text{-}CH_3)$ have been determined by X-ray diffraction techniques (76). The cis-trans isomerism in a series of phenylated methylaminoboranes has been studied by ^1H NMR spectroscopy and the effects of the substitution at the phenyl sites are discussed (49). Heats of formation of a number of alkyl-substituted aminoboranes have been determined (52). For the use of secondary (anilino)dichloroboranes in a new general process for specific hydroxybenzylation and hydroxyalkylation of secondary anilines, see (128). Carbon-13 NMR data on $(2\text{-}C_5H_4N)NH\text{-}B(C_2H_5)_2$ suggest that at room temperature and below the compound exists in at least two forms; only at higher temperatures does the normal aminoborane form become exclusive (22).

The reactions of bis(trimethylsilyl)acyl amides with haloboranes have been described in detail (156); (diorganyl)-haloboranes were found to yield monomeric iminoboranes which are in equilibrium with monomeric amidoboranes. Monomeric amido-amino-boranes have been obtained by the interaction of N-trimethylsilylamides with (amino)haloboranes, e.g., (3):

$$R\text{-}CO\text{-}NR\text{-}SiR_3 + R''_2N\text{-}BR'Cl \longrightarrow R_3SiCl + R''_2N\text{-}BR'\text{-}NR\text{-}CO\text{-}R.$$

Silylamidines react readily with (diorganyl)haloboranes to yield the corresponding amidinoboranes (211). The reaction of N,N-dialkylamidines with (alkylthio)dialkylboranes yields N'-dialkylboryl-N,N-dialkylamidines (14).

(Vinylamino)dialkylboranes interact with nitriles under formation of dialkylboryl-β-diiminates (15). The principal fragmentation of diorganylborylimidoylamidinates under electron impact involves the loss of boron-bonded hydrocarbon moieties (233), thus illustrating an inherent stability of the chelate ring in the initial species:

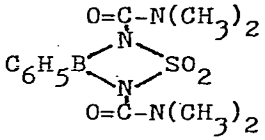


Various representatives of these latter have been synthesized and salts thereof, which are to be viewed as boronium salts, were also obtained and characterized (224).

Boron trihalides have been mentioned to react with N,N-dihalo arylsulfonylamides, $ArSO_2NX_2$, to yield B-trihalo-N-triarylsulfonylborazines, $(\text{-}BX\text{-}NSO_2Ar\text{-})_3$ (33). The borazine $(\text{-}BC_2H_5\text{-}N(2\text{-}C_5H_4N)\text{-})_3$ has been obtained from the thermal decomposition of (2-pyridylamino)diethylborane (22). For the formation of borazines by thermal decomposition of some t-butyldimethylsilyl-substituted aminoboranes, see (82). Also, B-trifluoro-N-trimethoxyborazine has been prepared and its vibrational spectrum has been reported (133). The vibrational spectra of B-trimethylborazine and isotopically labelled derivatives thereof have been studied and the suggested assignments of fundamentals are supported by a normal coordinate treatment (115). For ab initio calculations on the electronic structure and magnetism of borazine, see (169), and for a study of the allowed character of the 1900 Å band of borazine, see (140). Triplet states of borazine and N-trimethylborazine have been determined by trapped electron spectroscopy (239).

Reaction of 2-chloro-pentamethylborazine with silylated acid amides or thioacid amides yields the corresponding (thio)amido-substituted borazines (212). Similarly, trimethylsilylamidines were found to react with 2-chloro-pentamethylborazine to afford the corresponding 2-amidinoborazines (167). 2,4-Dichloroborazines interact with bis(trimethylsilyl)amine to yield, upon pyrolysis of the initial products, macrocyclic polyborazines in which four or five borazine moieties are bridged via NH groups into a macrocyclic system (1). A similar system in which six borazine units are linked by oxygen was obtained by interaction of 2,4-dichloro-tetramethylborazine with N,N-dimethylformamide and dimethylamine (4). Heat-resistant borazine copolymers and adhesives based on these are described elsewhere (264).

Bis(dimethylamino)phenylborane reacts with $SO_2(NCO)_2$ to yield the unusual cyclic species (177):



Efficient syntheses of 2-hydro-1,3,2-diazaboracycloalkanes have been described (25), and the reaction of 1,3-dimethyl-2-chloro-1,3,2-diazaboracyclopentane with $LiC{\equiv}C{-}N(C_2H_5)_2$ was found to yield the corresponding C-borylated inamine (72). Several boron derivatives of pyrimidines and borazaroquinazoline species have been prepared by using the carbanions obtained from tetrakis(trimethylenedioxyboryl)methane and tetrakis(ethylenedioxyboryl)methane, respectively, as reactive intermediates (143). Insertion of isocyanates into an anular B-N bond has again been studied (44, 71). The ^1H and ^{13}C chemical shift differences $\Delta\delta = \delta(NCH_2) - \delta(NCH_3)$ of 1,3-dimethyl-1,3,2-diazaboracycloalkanes are dependent on the ring size (28).

A boron analog of uracil has supposedly been obtained from the reaction of biuret with BH_3 generated in situ (155). In aqueous medium $NaBH_4$ was found to react with biuret to form a complex anion. Aminopyridines react with trimethylamine-iodoborane under iodide ion displacement and formation of aminopyridine-trimethylamine-dihydroboronium(1+) iodides (11). In the case of 2-aminomethylpyridine the process is accompanied by simultaneous displacement of trimethylamine and leads to a salt containing a cyclic cation.

Haloboranes, R_2BX, react with N-trimethylsilyl-triphenylphosphinimine to yield the corresponding N-borylated species, $(C_6H_5)_3P{=}N{-}BR_2$ (116). The nature of R determines the structure of the latter species; if R is an organic group, the products are monomeric whereas in the case of R = halogen associated - most likely dimeric - species are obtained.

Pyrazaboles (= dimeric 1-pyrazolylboranes) containing boron-bonded hydrocarbon groups exhibit the loss of hydrocarbon moieties as the major fragmentation process under electron impact (2). Carbon-13 NMR studies demonstrate the existence of conformational isomers when substituents at the pyrazabole framework are sufficiently bulky to cause steric interaction.

7 ACID-BASE ADDUCTS

In the thermal degradation of $H_3N \cdot BH_3$, vigorous decomposition begins at about 120°C (148); at 200°C the composition of the product approaches the stoichiometry

BNH. The basicity of $P(OR)_n(NR_2)_{3-n}$ species with respect to BH_3 has been studied by NMR spectroscopy (129), and a vibrational analysis has been performed on silylphosphine-borane (182).

Base displacement reactions have been studied between trimethylamine-fluoroboranes and phosphines as well as amines (195); fluorine bonded to boron was found to increase the hardness of the acid as compared to boron-bonded hydrogen. Extensive D/H exchange occurs when $(CH_3)_2ND \cdot BH_3$ is reacted with chlorine to achieve B-halogenation (84); the process appears to involve loss of deuterium chloride from a molecule activated as a result of halogenation. Very little isotope exchange is observed if bromine is used as halogenating agent and no exchange at all occurs in the case of iodine.

A general synthesis of amine-cyanoboranes by interaction of sodium cyanotrihydroborate with amine hydrochlorides has been described (232).

The microwave spectrum of $(CH_3)_3N \cdot BH_2F$ has been studied (141) as has the microwave spectrum of $(CH_3)_3N \cdot B(CH_3)_3$ including isotopically labelled derivatives thereof; molecular parameters are presented.

For the synthesis and X-ray crystal structure characterization of ammine-cyanoborane, see (242). Qualitative ^{11}B NMR studies of the acid methanolysis of $Na(CH_3)_2N \cdot 2BH_3$, see (229).

The barrier of internal rotation about the B-N bond for $(CH_3)_3N \cdot BF_3$ and $(CH_3)_3N \cdot BCl_3$ was estimated to be 6.2 and 13.7 kcal/mole, respectively (178); these estimates are based on the experimentally determined mean amplitudes of vibration for the rotation-dependent distances. For additional data on the rotational behavior of the trimethylamine adducts of BF_3 and BCl_3, see also (198).

N,N,N'N'-Tetramethylethylene diamine (TMED) reacts with $(C_2H_5)_2O\cdot BF_3$ to form the expected adduct $TMED\cdot 2BF_3$ (101). Similarly, monoalkylboranes from 1:2 molar adducts with the base but also 1:1 molar species, both of which are air stable. However, the monoalkylborane is readily displaced from these complexes by BF_3 to provide for easy generation of RBH_2 species. Complexes of BF_3 with phosphates, arsonates and phosphinates have been described elsewhere (135). For a vibrational spectroscopic study of the adducts of BF_3 with one or two equivalents of methanol, see (142).

Addition of one equivalent of elemental halogen to a solution of sodium cyanotrihydroborate yields oligomeric cyanoborane (85); the monomer can be obtained by coordination with a Lewis base such as pyridine. Excess chlorine, however, leads to B-halogenation and Cl_2BCN can be isolated as pyridine adduct.

The compounds $(CH_3)_3P\cdot BH_nX_{3-n}$ (X = Cl, Br, I; n = 0, 1, 2, 3) have been obtained by halogenation of trimethylphosphine-borane and by direct coordination of trimethylphosphine with boron triiodide (65). The ^1H NMR chemical shifts of the methyl groups evidence an increasing degree of deshielding as the size or the number of boron-bonded halogen is increased. For a NQR spectroscopic study of $Br_3P\cdot BBr_3$ and $I_3P\cdot BBr_3$, see (226). The reaction of $((CH_3)_4N)B_3H_8$ with $(C_6H_5)_2PCl$ leads unexpectedly to the formation of the linear $BH_2Cl-P(C_6H_5)_2-BH_2-P(C_6H_5)_2Cl$ species, the structure of which was confirmed by NMR spectroscopy and single-crystal X-ray analysis (111). $(t\text{-}C_4H_9)_3P\cdot BH_3$ has been prepared and was found to be thermally quite stable (219). The ring of $(-P(CF_3)_2-BH_2-)_3$ is easily cleaved by excess of trimethylamine to give $(CH_3)_3-BH_2-P(CF_3)_2$ and the boronium salt $((CH_3)_3N)_2BH_2)(H_2B(P(CF_3)_2)_2)$ (196); a similar and even faster reaction occurs with trimethylphosphine. For the preparation and characterization of $B_2H_4\cdot 2P(CH_3)_3$, see (231).

Trialkylboranes form 1:1 molar complexes with ketimines in an exothermic reaction (32). On heating in vacuum, ready dissociation into the initial components occurs. Urea, methylurea and thiourea form 1:1 molar complexes with triarylboranes (209). In the 1:1 molar complexes of triarylboranes with guanidine the boron is bonded to the imino nitrogen (210).

8 IONIC COMPOUNDS

Several amine-phosphine- and diphosphine-dihydroboronium salts have been synthesized and were characterized (86). An X-ray diffraction study of the boronium salt $((pz)_3BC_2H_5)(PF_6)$ (pz = 1-pyrazolyl) shows the structure of the species to be consistent with that postulated on the basis of NMR data (257).

A screening study on the reduction of organic compounds with the hydroxytrihydroborate ion $(HOBH_3)^-$, has been published (68). For isotope exchange and hydrolysis reactions of the cyanotrihydroborate ion in DMSO/water mixtures, see (251). Lithium and potassium dihydrocyano-1-pyrrolylborates as well as sodium and potassium cyanohydrodi-1-pyrrolylborates have been synthesized from the respective 1-pyrrolylborane and metal cyanide (163); hydrolysis studies on the species are reported. Sodium tetrakis(1-imidazolyl)borate has been advanced as a novel gravimetric reagent for hydrogen ion (168). The species $(B(NSOF_2)_4)^-$ is a novel anion which has been synthesized with the tetraphenylphosphonium cation (106).

A Grignard reagent, RMgBr, where R is a bulky group, will react with $THF \cdot BH_3$ to give the salt $Mg(HOBF_3)_2 \cdot 4THF$ (153); derivatives of the type $Mg(HOBF_3)_2 \cdot 2L$, $Mg(HOBF_3)_2 \cdot L$ and $MG(HOBF_3)(OH) \cdot THF$ ($L = N(CH_3)_3$, $N(C_2H_5)_3$, C_5H_5N) are also described. The species $NH_4(BF_4) \cdot (CH_2)_6N_4$ is formed on reaction of paraformaldehyde with $H_3N \cdot BF_3$ and ammonium acetate in ethanol solution (207). Copper(I) alkyltrifluoroborate has been introduced as a new alkylating agent (102). The ions $(Cl_3BNCO)^-$ and $(Cl_3BN_3)^-$ have been prepared and their infrared spectra were recorded (218). Boron trichloride reacts with salts of trinitromethane, $(R_4N)(C(NO_2)_3)$ ($R = CH_3$, C_2H_5) to form thermally unstable complexes containing the anion $(Cl_3B\text{-}C(NO_2)_3)^-$; at 20°C decomposition occurs with the formation of R_4N^+, BCl_4^- and N_2O_4 (31).

Rates, stoichiometry and products of the interaction of lithium and sodium hydrides with trialkylboranes of increasing steric requirements to form alkali metal trialkylhydroborates have been studied (125). Lithium trialkylhydroborate reacts with metal carbonyl acyls to give reactive anionic metal formyl complexes (18). Potassium hydride in THF reacts readily with trialkylboranes - even some highly hindered species - to yield the corresponding trialkylhydroborates (94). Spectroscopic studies suggest that in mixtures of $K(HB(C_2H_5)_3)$ and triethylborane in THF at 25°C a hydrogen-bridged diborate ion of the composition $(HB_2(C_2H_5)_6)^-$ is formed (104). Lithium triethylhydroborate is a convenient agent for the reduction of p-toluenesulfonates of primary and secondary alcohols to the corresponding alkanes (98).

Treatment of such dialkylboranes as 9-borabicyclo(3.3.1)-nonane, dicyclohexylborane or disiamylborane with organolithium reagents does not produce the expected lithium trialkylhydroborate (91); rather, a mixture of $Li(R_2BR'_2)$ and $Li(H_2BR_2)$ is obtained. Trialkylboranes react with α-methoxylvinyllithium at low temperature to give the corresponding lithium alkenyltrialkylborate (95). At room temperature and alkyl group migration occurs to produce a new alkenyldialkylmethoxyborate. gem-Dichloroallyllithium reacts with triphenylborane to afford $Li((C_6H_5)_3BCH_2CH=CCl_2)$ as the sole product (238). Michael reactions of trialkylalkenylborates have been described in which alkyl group migration from boron to carbon occurs (245). Treatment of lithium dicyclohexyl-trans-1-alkenyl-1-alkynylborates with either $(C_2H_5)_2O \cdot BF_3$ or $(n\text{-}C_4H_9)_3SnCl$ results in the preferential migration of the alkenyl group from boron to the adjacent alkynyl carbon atom (97).

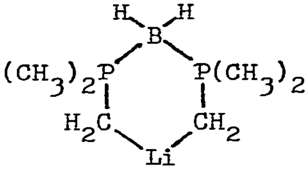
An X-ray diffraction study on $(N_2H_5)(B(C_2H)_4) \cdot N_2H_4$ has been described (63). Elsewhere (175), regio-controlled head-to-tail coupling of allylic borate species with allylic halides is reported.

9 METAL DERIVATIVES

The first diborane(6) derivative in which a Fe atom is a bridging site was obtained by the reaction of $K_2(Fe(CO)_4)$ with $THF \cdot BH_3$ (121); the authors favor the existence of a B-Fe-B three-center bond in the resultant $K(\mu\text{-}Fe(CO)_2B_2H_5)$. Interaction of $Ga(CH_3)_3$ with B_2H_6 as well as the reaction of $Ga(CH_3)_2Cl$ with $LiBH_4$ leads to the formation of $(Ga(CH_3)_2)BH_4$ (112); experimental evidence suggests the presence of two hydrogen bridges between Ga and B. Infrared spectral data suggest the existence of a single Cu-H bridge in the complex $(C_6H_5)_2CH_3PCuBH_4$ (67). The BH_4 group in $((C_6H_5)_3P)_2Cu(BH_4)$ is sufficiently deactivated to reduce carboxylic acid chlorides only to the aldehyde stage (146). For studies on the temperature-dependent NMR behavior of $((CH_3O)_3P)_2CuBH_4$, see (228); for photoelectron spectra of metal tetrahydroborates, see (244).

Various phosphinoborane complexes with transition metal carbonyl moieties, e.g., $(OC)_5M(R_2P\text{-}B(NR'_2)_2)$, have been described (208). Stable complexes of the anion

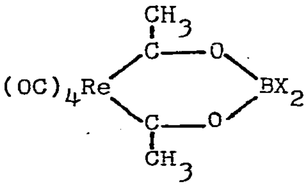
$(H_2B(PR_2-CH_2)_2)^-$ have been synthesized and characterized (105); by reacting $(((CH_3)_3P)_2BH_2)Br$ with two equivalents of lithiumalkyls in THF the lithium complex of an ylide anion



is obtained in which the Li is readily displaced by further reaction with metal halides to give a monocyclic Al complex or bicyclic structures with Zn, Cd, Ni. X-Ray data on the nickel complex suggest a chair conformation for the six-membered rings. For various transition metal complexes of arsapolyboranes, see (197).

Vanadium(II) and chromium(II) complexes with the ligands $(HBpz_3)^-$ and $(Bpz_4)^-$ (pz = 1-pyrazolyl) have been described and are assigned an octahedral geometry on the basis of their electronic spectra (83). The crystal structures of $Cr(H_2Bpz_2)_2$ and $K(V(H_2Bpz_2)_3 \cdot C_2H_5OH$ (83) as well as that of $(HBpz_3)PCH_3(C_2F_4)$ have been determined by X-ray diffraction (241).

The reaction of $(OC)_4Re(CH_3CO)(CH_3COH)$ with boron trihalides yields complexes of the type (237)



The species $(Cu(en)C_6H_5B(C_6H_5)_3)$ has been isolated from the reaction of CuCOCl with en (ethylene diamine); the X-ray structure analysis shows the presence of three-coordinate Cu(I) interacting with the tetraphenylborate anion (246). Preparation, properties and structural studies on the species

$(C_5H_5)Ni(CH_3C(-BC_2H_5-CC_2H_5-)_2)$ have been described (217). Crystal and molecular structures of two molybdenum complexes containing the tetraphenylborate ion as a π-bonding ligand have been resolved (230).

Reduction of $Co(C_5H_5BR)_2$ ($R = CH_3$, C_6H_5) with sodium amalgam gives $Na(Co(C_5H_5BR)_2)$ (220). Thermal decomposition of the anions in the presence of suitable ligands L gives complexes of the type $CoL_2(C_5H_5BR)$. The complexes $Fe(C_5H_5)(C_5H_5BR)$ with $R = CH_3$ or C_6H_5 are obtained on interaction of $Co(C_5H_5BR)_2$ with $(Fe(CO)_2(C_5H_5))_2$ (41). Oxidation of the complex results in ring contraction - boron elimination which is also observed on attempted Friedel-Crafts acetylation. This latter reaction, when applied to $Fe(C_5H_5BCH_3)_2$, yields $(Fe(C_6H_5CH_3)(C_5H_5CH_3))^+$ either solely or in mixture with the 2-acetyl derivative of $Fe(C_5H_5BCH_3)_2$, depending on the reaction conditions. The $Fe(C_5H_5BR)_2$ species are reversibly reduced in cyclic voltammetry processes in THF to yield the monoanion (176); the corresponding chromium complexes form either anions or cations whereas vanadium complexes yield anions exclusively. Thallium borinates, $Tl(C_5H_5BR)$, have been prepared from alkali metal borinates and TlCl in acetonitrile (45).

1-Phenyl-4,5-dihydroborepin (L) reacts with suitably substituted carbonyls of Cr, Mo or W to yield stable complexes of the type $LM(CO)_4$ (162); large upfield ^{11}B NMR shifts in these complexes with respect to the free ligand indicate that the boron atom participates in the metal-to-ligand bonding. Transition metal hexacarbonyls, $M(CO)_6$ with M = Cr or Mo, react with $S(-BCH_3-CC_2H_5=)_2$ (L) to yield complexes of the types $LM(CO)_4$ and $L_2M(CO)_2$, respectively (7). Based on ^{11}B NMR data the ligand L is pentahapto bound to the central metal atom. Simultaneous reaction of the cited thiadiborolene ligand L with $Mn_2(CO)_{10}$ and $((C_5H_5)Fe(CO)_2)_2$ gives the green tripledecker complex L-Fe-L-Mn(CO)$_3$ (127). Reaction of the latter with $C_6H_6/AlCl_3$ gives the salt $((C_6H_6)Fe\text{-}L\text{-}Mn(CO)_3)(AlCl_4)$, which can be pyrolyzed to provide for a small yield of a trinuclear tetradecker complex, $(OC)_3Mn\text{-}L\text{-}Fe\text{-}L\text{-}Mn(CO)_3$. Benzo-1,2,3,6-diazadiborines (L) react with $M(CO)_6$ (M = Cr, Mo, W) to yield the corresponding complexes $LM(CO)_3$ with the release of 3 CO (48). NMR Spectroscopic data suggest the benzo ring to be the donor site to the central metal atom. Also, several tricarbonylchromium complexes of various benzodiazaboroles and phenylboranes have been prepared (73); NMR data illustrate that the metal again is exclusively bonded to the aromatic rings.

Addition of the tetraphenylborate ion as precipitating counter anion to cationic four-coordinate rhodium(I) complexes with nitrogen donor ligands of the type $(NBD)RhL_2^+$ results in a competition between coordination by the tetraphenylborate group (via π-interaction of an aromatic ring) and the nitrogen donor ligands (159).

10 PHYSIOLOGICAL ASPECTS

Aplasmomycin, isolated from streptomyces griseus SS-20 (29), is a borate complex that exhibits activity toward gram-negative bacteria (29, 30); it joins boromycin as the second naturally occurring and boron-containing antibiotic. A biologically active borate derivative of amphotericin B that is soluble in saline solution has been described (80) and the serin-borate complex was found to inhibit a transition state of γ-glutamyl-trans-peptidase (186). Some new potentially antibacterial borazarothienopyridines have been synthesized (240).

Cyclic boronate derivatives of aldosterone have been studied by gas-liquid chromatography and mass spectrometry (181) and the interaction of (alkyl)hydroxyboranes with cholin esterase has been investigated (180). Sugar-borate complexation has been used for the separation of nucleosides on a cation exchange resin (12) and (phenyl)dihydroxyborane has been used as a ligand for the biospecific chromatography of serine proteinase (154). (Phenyl)dihydroxyborane was also found to inhibit sporulation of bacillus subtilis (188); for a study of the binding of (m-nitrophenyl)dihydroxyborane to the active site of subtilisin BfN, see (88).

A study on the photo control of boron metabolism in sea grasses has been published (53) and the effects of the methods of boron application on leaf tissue concentration and control of brown-heart in rutabaja have been investigated (89). For a study on tissue cultures under boron deficiency, see (187).

Membrane electrodes with tetraphenylborate for some basic drugs and their practical use have been described (55).

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