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Boron Aryls as Electron Donors: π-Complexes of Arylboranes with Tetracyanoethylene

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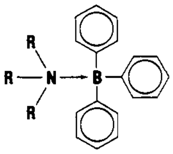
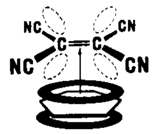
Abstract: Triphenylborane (TPB), tri-*o*-tolyborane (TOTB), and trimesitylborane (TMB) form 1:1 colored complexes with tetracyanoethylene (TCNE), in which the triarylborane donates π-electrons from its phenyl rings to the vacant antibonding orbital of TCNE. The absorbance maxima (λ, nm), molar absorbtivities (ε, L/(mol cm)), and formation constants (*K*, L/mol) for the complexes are TPB–TCNE (385, 219 ± 33, 3.2 ± 0.5), TOTB–TCNE (415, 134 ± 14, 4.5 ± 0.3), and TMB (475, 89 ± 14, 2.3 ± 0.4). The λ and *K* values are similar to those for corresponding benzene–TCNE complexes except for TMB–TCNE, the low value of which is due to steric hindrance. The ε values of triarylborane–TCNE complexes are generally an order of magnitude lower than those for benzene–TCNE complexes. Ionization energies (eV) calculated for triarylboranes from charge-transfer frequencies of the complexes are TPB (9.2), TOTB (8.9), and TMB (8.5). No spectral evidence is found for the formation of colored complexes between TPB and alkylnitriles, cyanoolefins, olefins, iodine, and bromine. ICl reacts irreversibly with TPB and TMB.

Triphenylborane (TPB) was synthesized by Krause[1] in 1922. In the following decade Krause and co-workers synthesized a variety of methyl-substituted triarylboranes and prepared many of their complexes with ammonia, methylamines, pyridine, and piperidine.[2-4] These complexes are stable white solids containing equimolar amounts of the borane and the nitrogenous base. The bonding interaction involves the donation of a pair of nonbonded electrons from the nitrogen to a vacant 2p orbital in the boron. This electron-pair-acceptor behavior, characteristic of trivalent monoboranes, is referred to as a n → v interaction, where n refers to the nonbonded electron pair of the donor molecule NR_3 and v refers to vacant bonding orbital of the acceptor molecule BR_3.

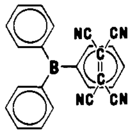


All examples of monoborane complexes cited in recent reviews[5,6] show the borane acting as the electron acceptor and do not consider the possibility that boranes could behave as electron donors.

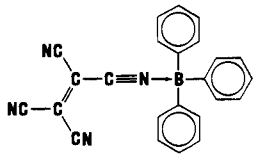
In the course of our study of triarylborane complexes, we observed that mixtures of TPB and the well-known electron acceptor, tetracyanoethylene (TCNE), in dichloromethane had a strong yellow color and an absorbance maximum at 385 nm. This behavior is strikingly similar to that of the benzene–TCNE mixture which has an absorbance maximum at 384 nm[7] attributed to a 1:1 charge-transfer complex. The bonding in this complex involves a bπ → aπ interaction in which π-bonding electrons from benzene are transferred to the vacant π-antibonding orbitals associated with the olefinic linkage in TCNE.[8-10]



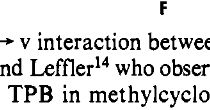
A comparison of the TPB–TCNE and benzene–TCNE cases suggests that TPB might donate electrons to TCNE through its phenyl groups.



However, a TPB–TCNE complex might also be stabilized through a n → v interaction wherein a nonbonded electron-pair on a cyano group of the TCNE is donated to the vacant orbital of TPB.



The trihalides of boron, for example, form several well-characterized complexes with the alkyl cyanides.[11] Structural[12] and spectroscopic[13] studies of acetonitrile–boron trifluoride show that the complex has a linear C-C-N-B skeleton and that the C-N and N-B bond lengths (113 and 163 pm) and vibration frequencies are those expected for a n → v complex.



$$\mathrm{H_3C{-}C{\equiv}N{\rightarrow}\underset{F}{\overset{F}{B}}{-}F}$$

Evidence for a n → v interaction between acetonitrile and TPB is given by Ramsey and Leffler[14] who observed a strong absorbance peak at 287 nm for TPB in methylcyclohexane which they attributed to an intramolecular bπ → v charge-transfer transition between a phenyl group and the boron atom. When TPB was dissolved in acetonitrile, the 287 nm peak disappeared, suggesting

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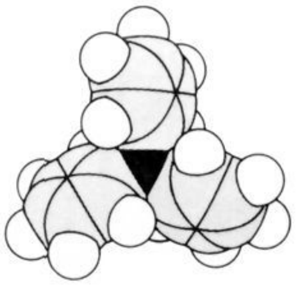


Figure 1. Space-filling model of triphenylborane.

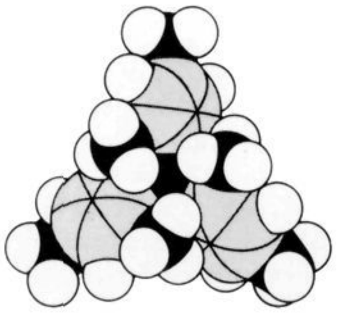
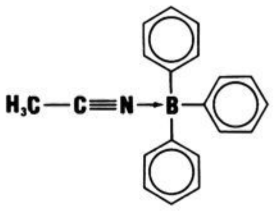


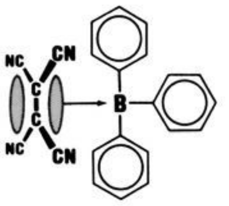
Figure 2. Space-filling model of trimesitylborane.

that the internal bπ → v transition was replaced by an intermolecular n → v interaction between acetonitrile and TPB.



This explanation is supported by the observation that the intramolecular charge-transfer peak for trimesitylborane (TMB) at 331 nm is scarcely affected when its solvent is changed from methylcyclohexane to acetonitrile.[15] These observations can be reconciled by examining diagrams of the space-filling models of TPB (Figure 1) and TMB (Figure 2). The boron atom in TPB is completely exposed, whereas the boron atom in TMB is completely shielded by the six ortho methyl groups attached to the phenyl rings. Therefore a n → v interaction between acetonitrile and TPB should occur easily, whereas the same type of interaction between acetonitrile and TMB should be completely blocked. While the electron-donor ability of simple nitriles is firmly established, there is no report of such behavior for TCNE.

A third possible interaction between TCNE and TPB involves the donation of bπ electrons from the TCNE olefinic bond to the vacant orbital of a TPB molecule.



No examples of stable bπ → v complexes involving an olefin and a monoborane have appeared in the literature, although this kind of interaction may occur as a first step in the hydroboration of olefins.

The TPB–TCNE case is an example of an interaction between two molecules, both of which have the potential of acting as either electron acceptors or electron donors through their various functional components. Triarylboranes, which normally act as electron acceptors because of the electron-deficient boron, might act as electron donors through the π-electrons in the phenyl rings. TCNE, which normally acts as an electron acceptor through the π-antibonding orbitals of its olefinic bond, might act as an electron donor through the nonbonded electron pairs of its four nitrogen atoms, or through the π-bonding electrons of its olefinic linkage. The study of the TPB–TCNE interaction should lead to insights on the interplay of inter- and intramolecular electronic effects and steric factors in these compounds.

Table I. Characteristics of TCNE Complexes with Benzene and Some Derivatives in Dichloromethane[a]

complex	λ_{max}, nm	ϵ_{max}	K, L/mol
benzene–TCNE	384	3570	2.00
toluene–TCNE	406	3330	3.70
mesitylene–TCNE	461	3120	17.3

[a] Reference 5.

Two sets of experiments were performed to determine which of the three modes of interaction gives rise to the colored complex. In the first series TCNE was mixed successively with TPB, tri-*o*-tolylborane (TOTB) and TMB in dichloromethane. The number of methyl groups shielding the boron atom increases from zero to three to six in this series. If complex formation occurs by the bπ → aπ mode, TCNE should form colored complexes with all three boranes; the spectral characteristics and equilibrium expressions will be expected to be similar to those of the complexes of TCNE with benzene, toluene, and mesitylene (Table I). The reaction (eq 1) and equilibrium expression (eq 2) for benzene–TCNE describe the general behavior of these systems.

$$C_6H_6 + TCNE \rightleftharpoons C_6H_6\text{–}TCNE \tag{1}$$

$$K = [C_6H_6\text{–}TCNE]/[C_6H_6][TCNE] \tag{2}$$

If the complexes are stabilized by the n → v or bπ → v modes their formation constants should decrease in the order TPB–TCNE > TOTB–TCNE > TMB–TCNE. In the case of TMB no complex is expected to form because of steric hindrance.

In the second series of experiments TPB is mixed successively with five compounds which have some or all of the functional components of TCNE, i.e., acetonitrile [CH_3CN], 2-methylpropionitrile [$(CH_3)_2CHCN$], acrylonitrile [$CH_2{=}CHCN$], fumaronitrile [*trans*-$CNCH{=}CHCN$], and 2,3-dimethyl-2-butene [$(CH_3)_2C{=}C(CH_3)_2$]. The first two compounds are alkyl nitriles, the next two are cyanoolefins, and the last is an olefin which has a carbon skeleton similar to TCNE. Complex formation with any of the first four is possible through the *n* → *v* interaction; complex formation with any of the last three is possible through the bπ → v interaction.

The results of studies of mixtures of TPB and TMB with halogens are also reported.

Experimental Section

Reagents. Tri-*o*-tolyborane was prepared by the method of Wittig and Herwig[16] with modifications suggested by Köster;[17] it was purified by vacuum sublimation and recrystallization from heptane and characterized by its melting point 67–70 °C (lit.[16] mp 67–69 °C) and its ultraviolet and infrared spectrum. Triphenylborane, trimesitylborane, 2-methylpropionitrile, acrylonitrile, and 2,3-dimethyl-2-butene were obtained from the Aldrich Chemical Co. and used without further purification, except for TMB which was recrystallized from ethanol. The melting points of TPB and TMB were 140–144 °C (lit.[17] mp 148 °C) and 192–194 °C (lit.[18] mp 190.5–191.5 °C). Acetonitrile, Burdick and Jackson HPLC Grade, was used without purification. Tetracyanoethylene, Eastman Reagent, was vacuum sublimed; mp 199–200 °C (lit.[19] mp 198–200 °C). Fisher Spectroanalyzed methylene chloride was dried by refluxing and distilling over P_2O_5. Mallincrodt analytical reagent iodine and bromine and spectral grade carbon tetrachloride were used without purification. MCB purified iodine monochloride was recrystallized prior to use.

Stock solutions of triarylboranes in dichloromethane were prepared by transferring solids directly into tared volumetric flasks and diluting to the mark with dichloromethane. Mixtures were prepared by transferring measured amounts of stock solutions into 10-mL glass-stoppered volumetric flasks. All weighings were made with a Mettler PC-180 top-loading balance, and solution transfers were made with a calibrated semiautomatic Selectapette pipet. All operations involving TPB and TOTB were carried out in a drybox flushed with 99.99% nitrogen.

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Table II. Absorbance Data for TPB-TCNE Solutions in CH_2Cl_2 at 20 °C

10^3C_{TPB}, M	10^3C_{TCNE}, M	A (385 nm)
1.77	80.9	0.080
3.54	71.9	0.148
5.31	63.0	0.195
7.00	54.0	0.231

Table III. Absorbance Data for TOTB-TCNE Solutions in CH_2Cl_2 at 20 °C

10^3C_{TOTB}, M	10^3C_{TCNE}, M	A (415 nm)
2.43	81.0	0.092
2.91	68.0	0.092
12.15	45.0	0.295
17.01	27.0	0.260
8.74	9.7	0.051

Table IV. Absorbance Data for TMB-TCNE Solutions in CH_2Cl_2 at 20 °C

10^3C_{TMB}, M	10^3C_{TCNE}, M	A (475 nm)
177.8	8.5	0.222
158.1	17.0	0.409
118.6	33.9	0.656
98.8	42.4	0.713
79.0	50.9	0.707

Spectra were recorded on a Beckmann ACTA-CIII spectrophotometer with 1-cm quartz cells.

Triarylborane–TCNE Solutions. The triarylboranes form colorless solutions in dichloromethane and have sharp absorbance cutoffs between 325 and 375 nm (see Figure 3). When mixed with TCNE, which has a sharp cutoff at 300 nm, they form solutions which are colored yellow, orange, and brick red having symmetrical absorbance peaks centered at 385, 415, and 475 nm, respectively. The colors are attributed to 1:1 complexes on the basis of continuous variations plots which show an absorbance maximum near 50 mol % in each instance.

A series of mixtures of TCNE with TPB, TOTB, and TMB were prepared and analyzed spectroscopically. Data are given in Tables II–IV. Relative concentrations of the various reagents were adjusted to yield absorbance values near 0.1 and above. TCNE was used in excess with TPB and TOTB in order to minimize overlap between the absorbances of the complexes and the uncomplexed reagents. TMB was used in excess with TCNE because the limited solubility of TCNE in dichloromethane (<0.1 M) made it impossible to use TCNE in excess and still obtain absorbance values in the optimum region. The raw absorbance data were analyzed at several wavelengths by Liptay's procedure[20] to yield the corrected absorbance values presented in Tables II–IV.

Interactions of TPB with Nitriles, Cyanoolefins, and Olefins. One-tenth molar solutions of the following reagents in dichloromethane were prepared: acetonitrile, 2-methylpropionitrile, acrylonitrile, fumaronitrile, and 2,3-dimethyl-2-butene. These were mixed successively in various amounts with 0.1 M TPB. The mixtures were examined spectrophotometrically from 250–700 nm. In no instance was any color observed, nor were any absorbances observed that could be attributed to a complex.

Interactions of Triarylboranes and Halogens. TPB was mixed successively with iodine and bromine in dichloromethane solutions at 20 °C. Concentrations of the reagents varied from 2×10^{-6} to 0.1 M. There was no evidence of complex formation from the spectra of the solutions in the region from 250 to 700 nm. TPB–iodine solutions were unaltered after standing at room temperature for several days. TPB–bromine solutions were stable in the dark but decolorized in the light; bromine appears to react with dichloromethane in light. TPB reacted rapidly and irreversibly with ICl in carbon tetrachloride. TMB reacted slowly with ICl over a period of several hours to yield iodine as one of the products. No evidence of complex formation was found in the spectra from 250 to 700 nm in either case.

Results and Discussion

Data from Tables II–IV are fit to the Scott equation (eq 3)[21]

$$C_XC_Y/A = (1/K\epsilon) + (1/\epsilon)C_X \qquad (3)$$

by least-squares procedures which yield values of ϵ from the slope, and values of K from the intercept. C_X and C_Y are molarities of reactants X and Y where $C_X > C_Y$, A is the absorbance of the complex X-Y. Calculated values of K and ϵ with uncertainties tabulated at 50% confidence limits are shown in Table V.

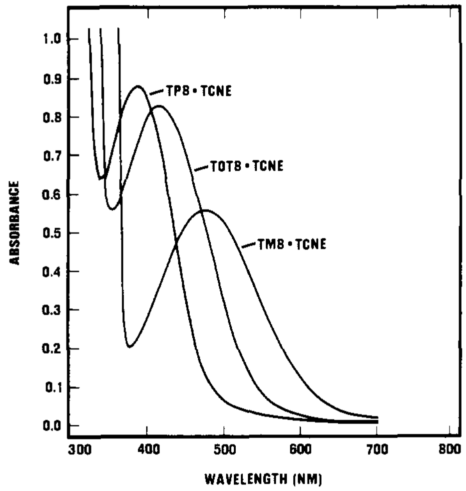

Figure 3. Absorbance curves of triarylborane–TCNE complexes.

Table V. Characteristics of Triarylborane-TCNE Complexes in Dichloromethane (20 °C)

complex	λ_{max}, nm	ϵ_{max}	K, L/mol
TPB–TCNE	385	219 ± 33	3.2 ± 0.5
TOTB–TCNE	415	135 ± 14	4.7 ± 0.5
TMB–TCNE	475	89 ± 14	2.4 ± 0.4

The excellent fit of the data to the Scott equation (eq 3) confirms the formation of 1:1 complexes in equilibrium with the reactants (eq 4).

$$\text{TPB} + \text{TCNE} \rightleftharpoons \text{TPB–TCNE} \qquad (4)$$

The similarity of λ_{max} and K values of benzene–TCNE complexes (Table I) and corresponding values of triarylborane–TCNE complexes (Table V) strongly supports the hypothesis that the latter are bπ → aπ complexes in which the triarylborane is the electron donor. The studies of mixtures of TPB with alkylnitriles, cyanoolefins and olefins show no evidence for the formation of colored complexes. Hence π → v or n → v interactions are ruled out as a significant factor in the formation of colored complexes. The preceding evidence taken together with the evidence for 1:1 complexes indicates that π-electron donation from the phenyl ring of the triarylborane to the TCNE molecule is the primary cause of complex formation between them, as in the third structural diagram.

It is well established that for a series of methylbenzene–TCNE complexes the values of λ_{max} and K increase as the number of methyl groups increases (Table I). These trends are attributed to the decrease in ionization energy of the benzene donor molecule through the electron-releasing effect of the methyl groups. That most values of λ_{max} and K for the triarylborane–TCNE complexes (Table V) are similar to those of the corresponding methylbenzene–TCNE complexes implies that the phenyl rings in the former are not significantly perturbed by their bonding to boron. This finding agrees with the conclusion reached by Armstrong and Perkins[22] whose molecular orbital calculations show that phenyl ring bond orders in TPB are almost identical with those in benzene and that the π-electron transfer to boron is very small. In other words, the triarylboranes should interact with TCNE in much the same way as their benzene analogues.

It follows that the empirical equation (eq 5) of Voigt and Reid[23]

$$h\nu_{CT} = 0.83I_D - 4.42 \qquad (5)$$

may be used to calculate the ionization energies of the triaryl-

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boranes. Equation 5 shows the relationship between the charge-transfer transition frequency of a D–TCNE complex (where D is a benzene derivative) and the ionization energy of the donor (I_D). Substituting the frequencies at maximum absorbance for the triarylborane–TCNE complexes (Table V) into eq 5, we obtain the values 9.2, 8.9, and 8.5 eV for TPB, TOTB, and TMB. The corresponding experimental values for benzene, toluene, and mesitylene are 9.24, 8.82, and 8.93 eV.[24]

The triarylborane–TCNE complexes are similar to corresponding benzene–TCNE complexes in all but two respects. First of all, the molar absorptivities of the former are an order of magnitude smaller. Secondly, the *K* value for TMB–TCNE (2.4) is much smaller than that of mesitylene–TCNE (17.3). This anomaly may be due to interference from the six ortho methyl groups which partially cover the six faces of phenyl rings of TMB. Examination of space-filling models of TMB (Figure 2) and TCNE show that effective overlap of the $b\pi$ and $a\pi$ orbitals is blocked by these methyl groups. Steric effects have been used to account for the large difference in *K* values between the TCNE complexes of hexamethylbenzene (16.9) and hexaethylbenzene (0.32);[7] they may also account for the differences between TMB–TCNE and mesitylene–TCNE.

The existence of many electron donor–acceptor complexes between aromatic hydrocarbons and iodine, bromine, and iodine monochloride has been established and their characteristics have been exhaustively studied, documented and reviewed.[25] It is therefore remarkable that no spectral evidence for triarylborane–halogen complexes has been observed. Either the triarylborane–halogen complexes do not form, of if they do, the charge-transfer bands of these complexes are covered by the absorbance bands of the triarylboranes themselves.

Acknowledgment. We are grateful for financial support from a Northern Michigan University Faculty Research Grant and to Lynn Podehl for valuable technical assistance.

Registry No. TPB·TCNE, 84961-35-3; TOTB·TCNE, 84961-36-4; TMB·TCNE, 84961-37-5; TPB, 960-71-4; TMB, 7297-95-2; ICl, 7790-99-0.

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Conformational, Bond-Order, and Substituent Dependencies of Orthobenzylic Coupling Constants

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Abstract: Because they are a potential source of valuable structural information, experimental and theoretical results are reported for the bond-order, conformational, and substituent dependencies of the long range H–H coupling constants over four bonds (orthobenzylic coupling, J_{ob}) between protons on an sp^3-hybridized α-carbon atom and the ortho protons of aromatic systems. A large number of methyl aromatic systems were synthesized and their coupling constants were accurately measured. The best correlations in this series of compounds were with the square of the mobile π-bond order and with the mutual atom–atom polarizability $\pi_{pp'}$. In addition, several series of compounds with defined geometries and a range of dihedral angles were synthesized and the ^{1}H NMR parameters were measured. The best agreement of the data is with a semiempirical equation, which relates the orthobenzylic coupling constant to $\pi_{pp'}$ and to dihedral angles according to the equation $J_{ob}(\phi,\pi_{pp'}) = 6.90\ \pi_{pp'} \sin^2 \phi - 0.32 \cos^2 \phi$ Hz. Substituent effects on J_{ob} are not usually important; exceptions are found in the 1-substituted acenaphthene derivatives, in which J_{ob} becomes more positive with increasing electronegativity, and in ring-substituted toluenes, having both a nitro and an oxygen group which exert significant bond-fixing effects.

Introduction

The nuclear spin–spin coupling between a proton attached to an sp^3-hybridized α-carbon atom of a side chain and the aromatic protons of a benzene ring is a special case of the more general phenomenon of benzylic coupling.[2-5] In fact, it has been noted[4] that benzylic coupling is a rather arbitrary subdivision of the larger class of coupling constants between protons attached to any benzylic atom and ring protons and includes coupling through heteroatoms.[6-8]

Of the various types of benzylic couplings the most widely investigated[2-13] has been orthobenzylic (J_{ob}) which refers to coupling between protons on the α-carbon atom and an ortho proton of the toluene moiety **1**. Coupling between the benzylic protons and meta and para hydrogens are called meta- and parabenzylic, respectively. The decreased magnitudes of experimental orthobenzylic coupling constants (for toluene $J_{ob} = -0.75$

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[1] (a) University of Arizona. (b) University of Sydney.

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