N_2 in Equation 3 gives $D^2 = 18.3$. We take from the curve the values of N_2 and N_2' given in Table III, and substitute them in the formula,[2] p. 144,

TABLE III

t	N_2	N_2'	v_1	v_2	D^2
150	0.450	0.820	116	66.0	19.6
154	.494	.796	117	66.2	19.2
158	.550	.760	118	66.4	17.5

$RT \ln (N_2'/N_2) = v_2 (\mathfrak{V}_1^2 - \mathfrak{V}_1'^2) D^2$, getting the figures in the last column of the table. Table IV,

TABLE IV

t, °C.	0	25	35	50	150	154	158	161
D^2	32.3	30.5	30.1	28.7	19.6	19.2	17.5	18.3

plotted in Fig. 2, summarizes all the values of D^2 calculated in this paper from the experimental data. The decline toward high temperatures is very similar to that encountered with the systems previously investigated. It will be worth while to re-examine this system as soon as a more reliable figure for the heat of fusion of iodine can be obtained. Meanwhile, it is evident that the methods here illustrated are again successful in correlating the solubility of a substance in its solid and liquid forms, in spite of the difference of 100° between the two sets of data.

In conclusion, it is interesting to compare the heat of solution of iodine in carbon tetrachloride, as measured by Blair and Yost,[12] 5990 ± 60 cal. per mole of iodine in very dilute solutions, about 0.1 mole per cent., at 25°. The figures in Table I correspond to a free energy of solution in excess of the ideal free energy, $\bar{F}_2 - \bar{F}_2^i = 1790$ cal. at 25° when $N_2 = 0.015$. Dividing this by $(1 - N_2)^2$ gives 1845 cal. for an infinitely dilute solution. If these solutions are completely "regular," in the sense of having ideal entropy of solution, this figure should represent the heat absorbed in mixing liquid iodine with carbon tetrachloride, or, added to the heat of fusion, which we have taken to be 3725 cal., we get 5570 cal., which agrees with the 5990 cal. of Blair and Yost as closely as could be expected.

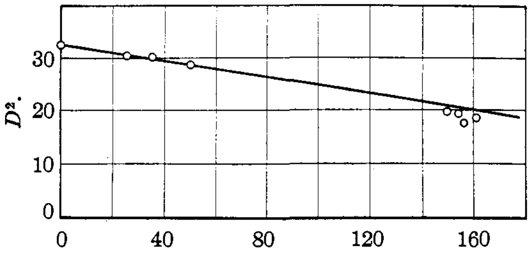

Fig. 2.

Summary

The predicted existence of a two liquid phase system in the case of iodine and carbon tetrachloride has been verified, the critical solution temperature being 160.5° and the corresponding composition, 0.68 mole per cent. of iodine. The correlation between the solubility of iodine in the solid and liquid states has been discussed.

BERKELEY, CALIF. RECEIVED AUGUST 9, 1937

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[12] C. M. Blair and D. M. Yost, THIS JOURNAL, 55, 4489 (1933).

[CONTRIBUTION FROM THE GATES AND CRELLIN LABORATORIES OF CHEMISTRY, CALIFORNIA INSTITUTE OF TECHNOLOGY, No. 616]

The Molecular Structures of Boron Trimethyl, Trifluoride, Trichloride, and Tribromide. The Covalent Radius of Boron

BY HENRI A. LÉVY AND L. O. BROCKWAY

The molecular structures of the compounds of boron are of special interest because the unusual number of electrons occurring in many of them suggests that their structures may be different from those of corresponding compounds of the other non-metallic elements of the first row of the periodic table. Because of the anomalous electronic structures of the stable hydrides due to the lack of enough electrons to form electron-pair bonds throughout the respective molecules, other compounds of boron were chosen in the hope of determining a characteristic single bond covalent radius for comparison with the radii of other elements. Although boron in its normal valence compounds (in which it is coördinated with three univalent atoms or groups) has only six electrons around it in place of the octet found in the normal valence compounds of the following first row elements, the number of electrons in the BX_3 compounds is sufficient for the formation of electron pair bonds. In particular, boron trimethyl was investigated because the methyl derivatives of a number of non-metallic elements have previously been found to contain single covalent bonds.[1] In addition we have investigated boron trifluoride, trichloride, and tribromide.

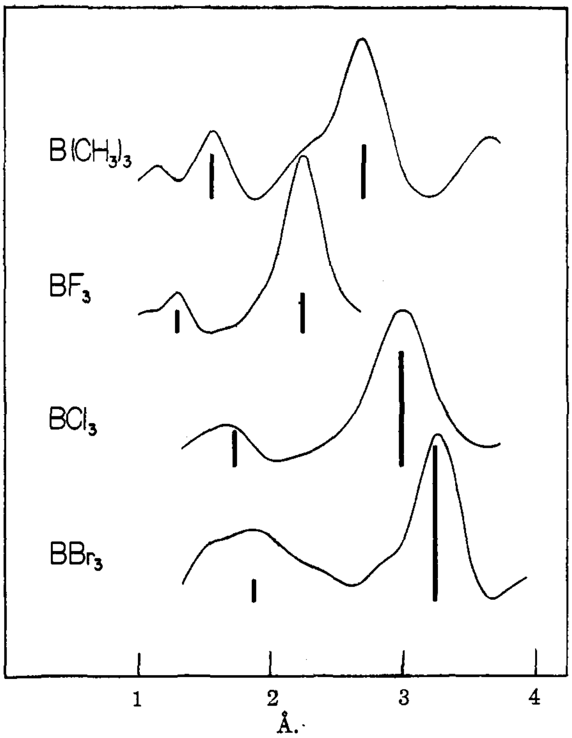

Fig. 1.—Curves showing the observed radial distributions of scattering matter. The vertical lines mark the interatomic distances as determined by comparison of the photographs with theoretical scattering curves.

The vapors of these substances were used in obtaining electron diffraction patterns by the known procedure[2] with an electron wave length of 0.0611 Å. and a camera distance near 10.5 cm. Photographs were obtained showing from six to ten apparent maxima which were measured visually. The corresponding s_0 values (equal to $4\pi (\sin \theta/2)/\lambda$ where θ is the angle of scattering and λ the electron wave length) are tabulated for each compound, and they were combined with the visually estimated intensities (shown under the columns headed *I*) to give the observed radial distributions of scattering matter shown by the curves in Fig. 1. The tables also show the comparison of the *s* with the "calculated s_0" values (taken from the theoretical scattering curves in Figs. 2–5) to give observed molecular dimensions.

Boron Trimethyl.—From a sample of boron trimethyl prepared by Dr. A. Burg of the University of Chicago, photographs were obtained showing a pattern of six well-defined rings which is well represented by the 120° curve in Fig. 2. The characteristic features used in distinguishing among the various molecular models are the following. The third and fourth maxima are about equal in intensity, and are resolved by a distinct minimum. The fifth maximum, slightly more intense than those preceding, is followed by a "shelf," whose intensity is about equal to that of the following (sixth) maximum. The radial distribution function (Fig. 1) shows sharp peaks at 2.69 Å., assigned to the C–C distance, and at 1.57 Å., corresponding to the B–C distance, together with other less reliable humps. Theoretical scattering curves (Fig. 2) were calculated for plane and pyramidal arrangements of tetrahedral methyl groups with C–H = 1.05 Å. about a central boron atom; approximation to free rotation of methyl groups was achieved by averaging configurations differing by methyl group rotations of 60°, a procedure which has been shown in this Laboratory to be satisfactory. A B–C distance of 1.53 Å. and C–B–C angles of 120, 118, 116, and 110° were assumed. That the 110° model is unsatisfactory is evident in that the corresponding curve shows a "shelf" following the third peak instead of the fifth. Inasmuch as the 116° curve shows the fourth peak weaker than the third and the fifth resolved from the hump following it, it is likely that the boron bond angle is larger than 116°. Furthermore, since the sixth peak appears too intense compared to the shelf in the 116 and 118° models, we feel that the photographic evidence favors the 120° model.

TABLE I

BORON TRIMETHYL

Max.	Min.	*I*	s_0	$s_{\text{calcd.}}$ 120°	$s_{\text{calcd.}}/s_0$
1		5	3.072	2.58	(0.840)
	2		4.178	3.79	(.907)
2		10	5.351	5.40	1.009
	3		6.470	6.60	1.020
3		7	7.577	7.70	1.016
	4		8.43	8.70	1.032
4		7	9.51	9.63	1.012
	5		10.98	11.10	1.003
5		8	12.35	12.78	1.035
5a		4	14.14	14.47	1.023
	6		15.70	15.90	1.013
6		4	16.92	17.38	1.027

Mean 1.019

C–C = 2.70 Å.
B–C = 1.56 Å.

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[1] L. O. Brockway and H. O. Jenkins, THIS JOURNAL, **58**, 2036 (1936).

[2] L. O. Brockway, *Rev. Modern Physics*, **8**, 231 (1936).

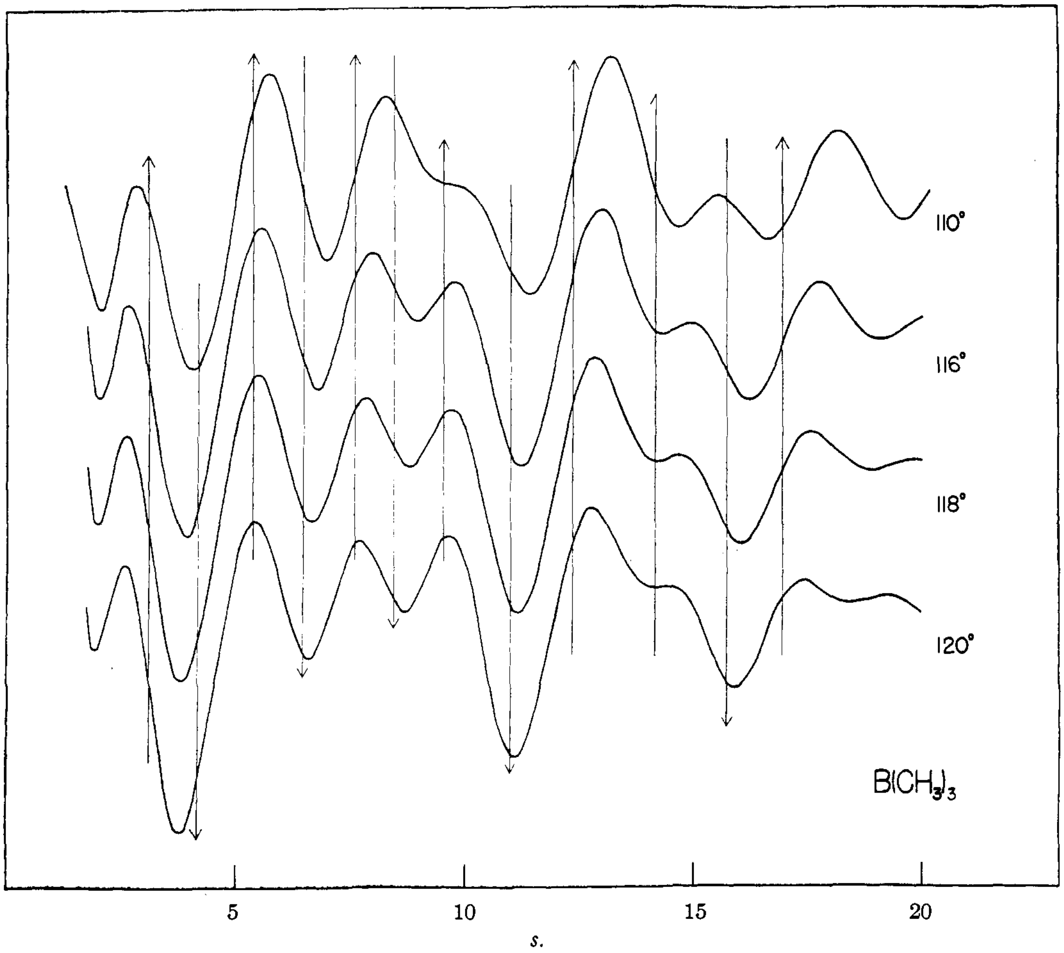

Fig. 2.—Theoretical scattering curves for various models of boron trimethyl. The arrows indicate the positions of maxima and minima observed on the photographs.

The quantitative comparison of Table I and the radial distribution results lead to the final values of C–C = 2.70 ± 0.03 Å., C–B = 1.56 ± 0.02 Å., and < C–B–C = 120° (within 3°).

Boron Trifluoride.—Boron trifluoride was prepared by the method of Balz and Schiemann[3] by the decomposition of phenyldiazonium fluoroborate previously obtained from diazotized aniline and fluoroboric acid. The product was cooled to −78° and subsequently subjected to a vacuum distillation. Photographs show seven strong well-defined and two weak rings, whose spacing is about uniform. The radial distribution curve (Fig. 1) shows a sharp peak at 2.26 Å., corresponding to the F–F distance, together with other less reliable peaks at shorter distances. Theoretical scattering curves (Fig. 3) were calculated for plane and pyramidal configurations of three fluorine atoms around a central boron, assuming a B–F distance of 1.30 Å. and F–B–F angles of 120, 118, 116, and 109°28′. As the angle is decreased the fifth peak becomes too strong compared to the fourth and seventh, while the sixth maximum becomes too well resolved from the fifth and not sufficiently resolved from the seventh. This evidence fixes the angle at a value greater than 116° and favors a planar model.

The quantitative comparison of Table II together with the evidence derived from the radial distribution curves leads to the final values F–F = 2.25 ± 0.03 Å., B–F = 1.30 ± 0.02 Å., and < F–B–F = 120° (within 3°).

In a recent paper Braune and Pinnow[4] reported a planar configuration with a B–F distance of 1.31 Å. Raman[5] and infrared[6] spectral data are consistent with a planar configuration. Applica-

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[3] G. Balz and G. Schiemann, *Ber.*, **60**, 1186 (1927).

[4] H. Braune and P. Pinnow, *Z. physik. Chem.*, **B35**, 239 (1937).

[5] Anderson, Lassettre and Yost, *J. Chem. Phys.*, **4**, 703 (1936).

[6] Bailey, Hale and Thompson, *Proc. Roy. Soc.* (London), **A161**, 107 (1937).

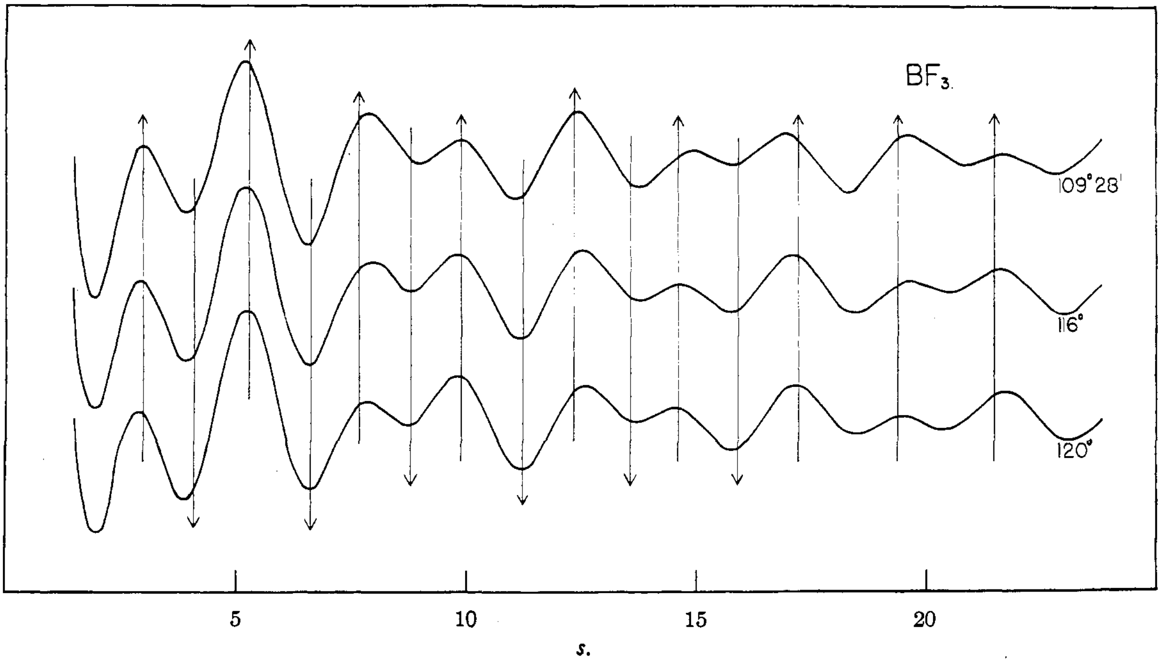

Fig. 3.—Theoretical electron scattering curves for boron trifluoride.

tion of Badger's rule to the force constant from infrared data gives 1.33 Å., for the B–F distance.

TABLE II

BORON TRIFLUORIDE

Max.	Min.	I	s_0	$s_{\text{calcd.}}$ 120°	$s_{\text{calcd.}}/s_0$
1		8	3.259	3.16	(0.969)
	2		4.446	4.21	(.946)
2		12	5.748	5.69	.990
	3		7.232	7.21	.997
3		7	8.403	8.58	1.021
	4		9.610	9.49	0.988
4		8	10.81	10.71	.991
	5		12.23	12.20	.997
5		5	13.45	13.71	1.020
	6		14.82	14.94	1.009
6		2	15.96	15.87	0.995
	7		17.37	17.21	.991
7		4	18.59	18.70	1.007
8		1	21.13	21.28	1.007
9		1	23.45	23.70	1.011
					Mean 1.002
					F–F 2.25 Å.
					B–F 1.30 Å.

Boron Trichloride.—Photographs of boron trichloride obtained with a sample of Kahlbaum's C. P. preparation showed nine well-defined uniformly spaced rings. The radial distribution curve (Fig. 1) shows a tall, sharp peak at 2.99 Å., assigned to the Cl–Cl distance, with a small, less reliable peak at a shorter distance. Theoretical scattering curves (Fig. 4) were calculated for plane and pyramidal arrangements of chlorine atoms around a central boron, assuming a Cl–Cl distance of 3.00 Å. and a Cl–B–Cl angle of 120, 118, 116, 110, and 90°. As the angle is decreased, the fourth maximum drops below the third and fifth, contrary to the appearance of the photographs, where the third and fourth are equal, with the fifth weaker. In addition, the resolution of the sixth and seventh peaks becomes less distinct than that of the fifth and sixth in the smaller angle models, whereas in the photographs the reverse is true. These features make it improbable that the angle is less than 116°, and favor the planar configuration. The quantitative comparison shown in Table III together with the radial distribution result leads to the final values Cl–Cl = 2.99 ± 0.03 Å., B–Cl = 1.73 ± 0.02 Å., < Cl–B–Cl = 120° (within 3°). A previous electron diffraction investigation[6a] of this compound by Wierl[7] resulted in the value 3.03 ± 0.05 Å. for the Cl–Cl distance, with the configuration undetermined. The zero value obtained in the measurement of the dipole moment in benzene solution[8] supports the planar structure, as do also Raman spectral data.[5] From the latter, with the

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[6a] The electron-diffraction value 1.76 ± 0.02 Å. for the B–Cl distance in BCl$_3$ has been reported, since our work was submitted for publication, by Gregg, Hampson, Jenkins, Jones and Sutton, *Trans. Faraday Soc.*, **33**, 852 (1937).

[7] R. Wierl, *Ann. Physik*, **8**, 521 (1931).

[8] H. Ulich and W. Nespital, *Z. Elektrochem.*, **37**, 559 (1931).

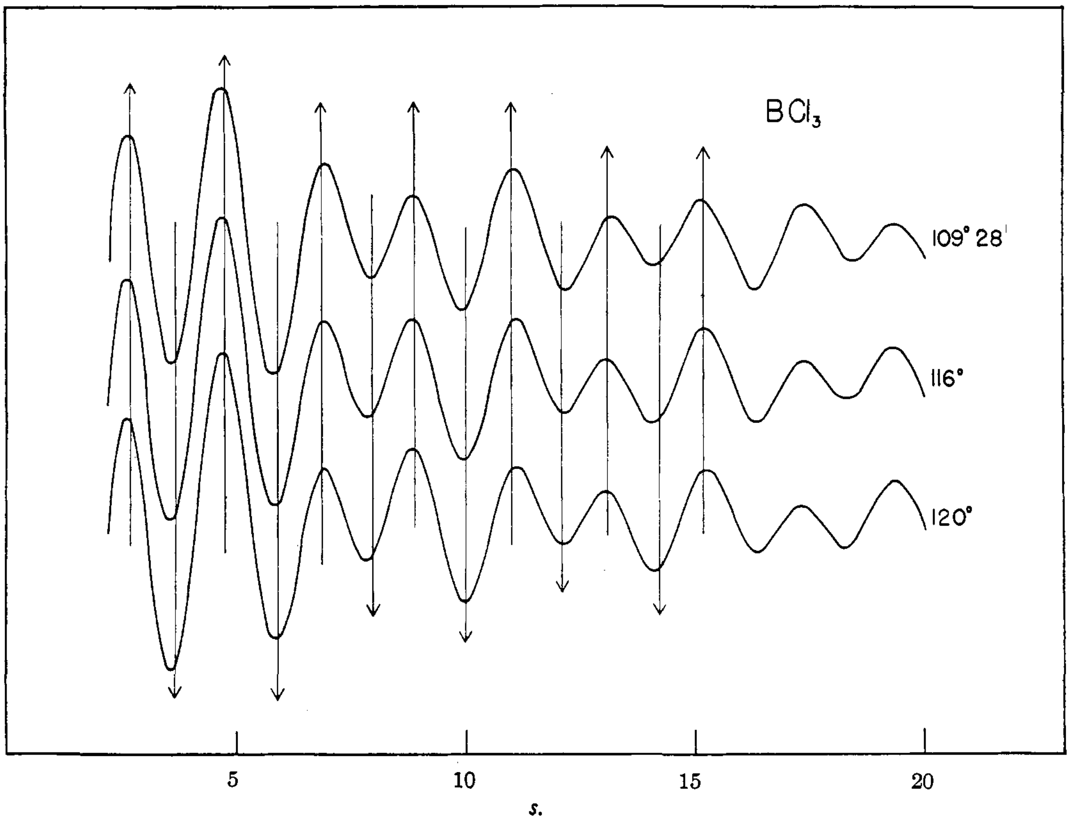

Fig. 4.—Theoretical electron scattering curves for boron trichloride.

aid of Badger's rule, the B–Cl distance is estimated to be 1.72 Å.

TABLE III

BORON TRICHLORIDE

Max.	Min.	I	s_0	$s_{\text{calcd.}}$ 120°	$s_{\text{calcd.}}/s_0$
1		14	2.678	2.59	(0.968)
	2		3.645	3.53	(.968)
2		22	4.733	4.67	.987
	3		5.906	5.82	.985
3		11	6.837	6.88	1.006
	4		7.949	7.78	0.979
4		13	8.877	8.80	.991
	5		9.987	9.93	.994
5		6	10.99	11.09	1.009
	6		12.08	12.09	1.001
6		2	13.08	12.99	0.993
	7		14.25	14.07	1.988
7		3	15.19	15.21	1.001

Mean 0.994

Cl–Cl 2.98 Å.
B–Cl 1.72 Å.

Boron Tribromide.—A sample of boron tribromide redistilled from mercury gave photographs of fair quality with a pattern of ten rings more closely spaced but otherwise similar to that of boron trichloride. The radial distribution curve (Fig 1.) shows a tall, sharp peak at 3.27 Å., assigned to the Br–Br distance. Theoretical

TABLE IV

BORON TRIBROMIDE

Max.	Min.	I	s_0	$s_{\text{calcd.}}$ 120°	$s_{\text{calcd.}}/s_0$
1		3	2.567	2.45	(0.954)
	2		3.400	3.29	(.968)
2		12	4.383	4.29	.979
	3		5.447	5.30	.973
3		10	6.296	6.21	.986
	4		7.335	7.18	.979
4		10	8.175	8.11	.992
	5		9.182	9.11	.992
5		8	9.999	10.11	1.011
	6		11.11	11.06	0.995
6		6	11.92	11.96	1.004
	7		12.91	12.91	1.000
7		6	13.80	13.92	1.009
	8		14.86	14.91	1.003
8		2	15.78	15.86	1.005
	9		16.89	16.74	0.991
9		1	17.97	17.75	.988
	10		18.94	18.72	.988
10		$^1/_2$	19.96	19.70	.987

Mean 0.993

Br–Br 3.24 Å.
B–Br 1.87 Å.

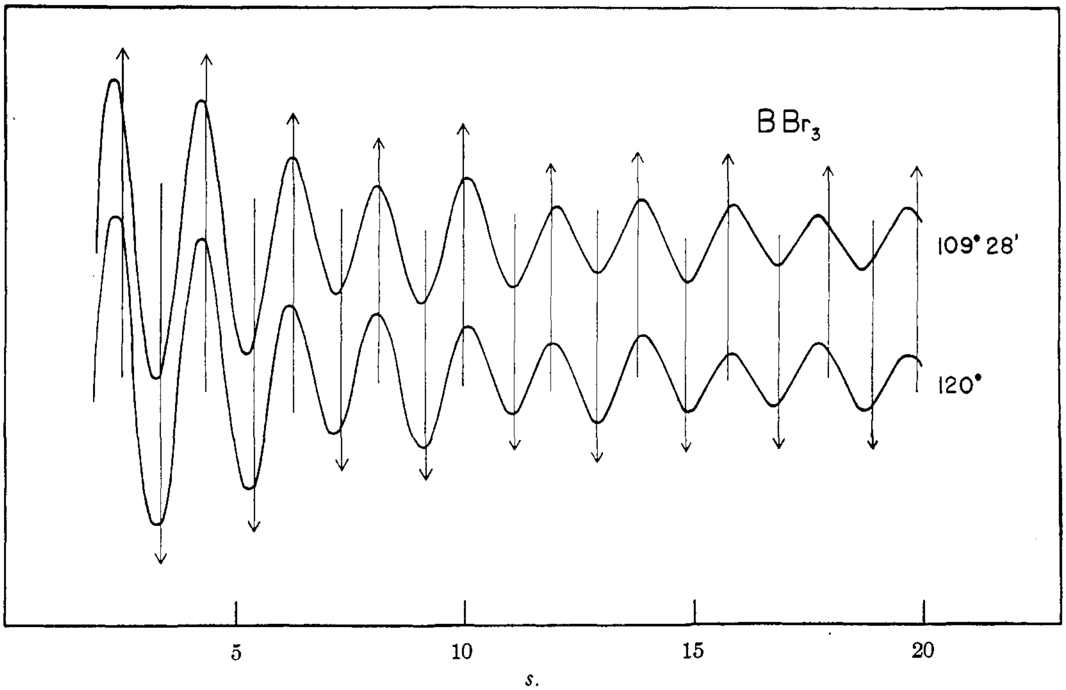

Fig. 5.—Theoretical electron scattering curves for boron tribromide.

scattering curves (Fig. 5) were calculated for models having a Br–Br distance of 3.27 Å. and a Br–B–Br angle of 120, 115, and 109°28′. Because of the large scattering power of bromine in comparison to boron, the two former curves are practically identical; in the latter, however, the fourth maximum falls below the third and fifth, whereas in the photographs the third and fourth appear equal with the fifth weaker. The angle is therefore larger than tetrahedral and is quite probably 120° as in the three compounds previously discussed. The final quantitative results are Br–Br = 3.25 ± 0.03 Å., B–Br = 1.87 ± 0.02 Å., < Br–B–Br = 120° (within 6°).

Raman spectral data[5] are consistent with the planar configuration; with the aid of Badger's rule they lead to a B–Br distance of 1.85 Å.

Discussion

The results of the electron diffraction investigation of these compounds are collected in Table V.

TABLE V

Substance	X–X distance, Å.	B–X distance, Å.
$B(CH_3)_3$	2.70 ± 0.03	1.56 ± 0.02
BF_3	2.25 ± .03	1.30 ± .02
BCl_3	2.99 ± .03	1.73 ± .02
BBr_3	3.25 ± .03	1.87 ± .02

The observed B–C distance of 1.56 Å. in boron trimethyl combined with the carbon single bond covalent radius,[9] 0.77 Å., leads to a value for the radius of boron of 0.79 Å. This value is to be compared with 0.89 Å., obtained by extrapolation of the radii of oxygen, nitrogen and carbon. The large discrepancy between the two, amounting to 0.10 Å., raises the question of what is represented by the extrapolated value. As has been pointed out to us by Professor Pauling, the radius values used in the extrapolation correspond to atoms having complete octets of electrons; and the value 0.89 Å. for boron presumably represents the single bond radius when the boron atom has associated with it four pairs of electrons. In boron trimethyl, on the other hand, the boron atom has only three electron pairs occupying a set of three equivalent planar orbitals probably arising from hybridization of sp^2. The larger relative contribution of the s orbital in bonds of this type as compared with the tetrahedral sp^3 bonds observed in oxygen, nitrogen and carbon would lead to a smaller bond radius. Accordingly, the discrepancy observed may well be due to a fundamental difference in bond type, the radius of 0.79 Å. being associated with boron surrounded by six electrons occupying hybridized sp^2 orbitals and that of 0.89 Å. associated with boron surrounded by a complete octet of electrons in sp^3 orbitals.

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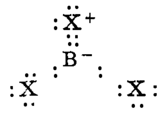
(9) L. Pauling and L. O. Brockway, THIS JOURNAL, **59**, 1223 (1937).

TABLE VI

Substance	Bond distance, Å.	Boron single bond radius, Å.	Remarks
$B(CH_3)_3$	B–C = 1.56 ± 0.02	0.79	sp^2 bond type
Extrapolated from O, N, C radii		.89	sp^3 bond type
BCl_3	B–Cl = 1.73 ± .02	.89	Graphite type resonance assumed
BBr_3	B–Br = 1.87 ± .02	.89	Graphite type resonance assumed
BF_3	B–F = 1.30 ± .02	(.77)	Graphite type resonance assumed
$B_3N_3H_6$	B–N = 1.44 ± .02	.89	Benzene type resonance assumed
BN	B–N = 1.45 ± .01	.87	Graphite type resonance assumed
$BH_3N(CH_3)_3$	B–N = 1.62 ± .15	.92	
B_2H_6	B–B = 1.86 ± .04	.93	
B_5H_9	B–B = 1.76 ± .02	.88	
CaB_6	B–B = 1.716 ± .004	.86	
BH_3CO	B–C = 1.57 ± .03	.80	sp^3 state for carbon
		.88	Resonance with sp^2 state

If we accept the value 0.89 Å. for the tetrahedral radius, a satisfactory explanation of both bond distances and molecular configurations in boron trichloride and boron tribromide is possible. Resonance among the three Lewis structures of the type



would give each B–X bond one-third double and two-thirds single bond character, sp^3 quantization being necessary to provide for the four bond orbitals. Such resonance would constrain the atoms to a coplanar configuration with 120° bond angles, as in graphite and carbonate ion. While some contribution might be made by the structure having three single bonds on boron, the loss of the energy of the extra bond would make this structure relatively less important; and a small contribution would have no effect on the bond distance because the sp^2 bonds in this structure would have a length only slightly greater than that of the bonds in the proposed resonating molecule. Applying the relation between bond distance and double bond character,[9,10] we find that the distances 1.73 and 1.87 Å. observed in the chloride and bromide, respectively, both lead to a value of 0.89 Å. for the single bond tetrahedral radius of boron.

The extremely short bond distance in boron trifluoride under the foregoing assumptions corresponds to a boron radius of 0.77 Å. It is very probable that the structure of the fluoride requires another explanation since abnormally short bond distances are observed in other fluorides as well, *e. g.*, silicon tetrafluoride and phosphorus trifluoride.[11]

Other boron compounds which have been investigated are shown together with the above results in Table VI. In $B_3N_3H_6$[12] the molecule undoubtedly has the electronic structure of benzene and the observed B–H distance of 1.44 ± 0.02 Å. corresponds to a single bond boron radius of 0.89 Å. The boron nitride crystal has been found[12a] to have a graphite-like structure with B–N = 1.45 ± 0.01 Å., corresponding to a single bond boron radius of 0.87 Å. The single bond B–N distance in $BH_3N(CH_3)_3$[13] is combined with the nitrogen radius 0.70 Å. to give a boron radius of 0.92 Å. The boron–boron distances observed in B_2H_6[14] and B_5H_9[15] are treated as single bonds to give radii of 0.93 and 0.88 Å., respectively. It should be noted that in the latter substance one of the five boron atoms forms only three bonds while the others each form four, and the average boron radius observed is perhaps 0.03 or 0.04 Å. smaller than it would be if all of the atoms were of the tetrahedral type. In CaB_6[16] each boron forms five bonds with an observed boron–boron separation of 1.716 ± 0.004 Å.

The boron–carbon distance 1.57 ± 0.03 Å. observed in BH_3CO[13] combined with the tetrahedral radius for carbon gives a boron radius of 0.80 Å. On the other hand, the carbon–oxygen distance is observed to be near that in carbon monoxide, and accordingly the carbon–oxygen bond presumably involves resonance between double- and triple-bonded structures. In the structure having a double bond between carbon and oxygen the carbon atom is holding only three electron pairs, and in accordance with the suggestion made

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(10) L. Pauling, L. O. Brockway and J. Y. Beach, THIS JOURNAL, **57**, 2705 (1935).

(11) L. O. Brockway and F. T. Wall, *ibid.*, **56**, 2373 (1934).

(12) S. H. Bauer, paper submitted to THIS JOURNAL.

(12a) O. Hassel, *Norsk Geol. Tidsskrift*, **9**, 266 (1926).

(13) S. H. Bauer, THIS JOURNAL, **59**, 1804 (1937).

(14) S. H. Bauer, *ibid.*, **59**, 1096 (1937).

(15) S. H. Bauer and L. Pauling, *ibid.*, **58**, 2403 (1936).

(16) L. Pauling and S. Weinbaum, *Z. Krist.*, **87**, 181 (1934).

above in the case of boron trimethyl the carbon radius in this structure would be about 0.10 Å. less than the tetrahedral radius. In resonance with the triple-bonded structure the carbon–oxygen distance would be unaffected, but the single bond distance between tetrahedral boron and carbon atoms would be decreased by about 0.08 Å. With this allowance the observed boron–carbon distance gives a boron radius of 0.88 Å.

The values for the boron radius listed in Table VI lie within 0.04 Å. of 0.89 Å. with two exceptions. In the first, boron trimethyl, the bonds are of a different type from those in the other compounds; and in the second, boron trifluoride, the anomalies observed in other fluorides make it difficult to interpret the observed distance with any assurance. The general agreement among the other data lends support to the value first obtained by extrapolation from the other first row elements. In the three compounds in which boron–boron distances have been observed there exists an insufficient number of electrons to form electron-pair bonds in every bond position. While this deficiency undoubtedly affects the character of the bonds, it is not evident that the bond distances are materially affected since the average of the three observed radii is 0.89 Å.

We are indebted to Professor Anton Burg of the University of Chicago for the sample of boron trimethyl, to Dr. S. H. Bauer for data on triborine triamine, borine trimethylammine, and borine carbonyl, and to Professor Linus Pauling for consultation and advice.

Summary

The electron diffraction investigation of the molecular structures of boron trimethyl, trifluoride, trichloride, and tribromide shows that these molecules are planar with the distances B–C = 1.56 ± 0.02 Å., B–F = 1.30 ± 0.02 Å., B–Cl = 1.73 ± 0.02 Å., and B–Br = 1.87 ± 0.02 Å., respectively.

These data together with those from six other boron compounds listed in Table VI support a value of 0.89 Å. for the single bond radius of boron surrounded by an octet of electrons and of 0.79 Å. for boron with only six electrons.

PASADENA, CALIF. RECEIVED JULY 31, 1937

[CONTRIBUTION FROM THE LABORATORY OF PHYSICAL CHEMISTRY, UNIVERSITY OF WISCONSIN]

The Photo-oxidation of Acetone Vapor

BY PAUL FUGASSI[1]

It was observed that when acetone vapor, containing some oxygen, is exposed to ultraviolet light (3130 Å.), a sharp change from blue fluorescence to green fluorescence occurs when all the oxygen is consumed.[2] This test was made the means of analyzing for traces of oxygen.[3] The work has been extended to include a measurement of the quantum efficiency of the photo-oxidation.

A small rectangular cell of quartz was equipped with a side chamber holding several thin-walled bulbs ranging from 0.4 to 1.0 cc. in volume. The bulbs had been sealed previously to a vacuum line and filled with air at varying pressures.

The air bulbs and the magnetic hammer were placed in position and the cell was filled partially with purified acetone and evacuated till only a few drops of liquid remained. The sealed cell was then placed in a thermostat at 25°, and the vapor was radiated with a beam of light of 3130 Å. from a capillary mercury arc lamp and a large monochromator.[4]

Usually an exposure for one to two minutes was sufficient to change the fluorescence of the vapor from blue to green, showing that most of the oxygen had been pumped off.

The bulbs of oxygen were then broken one at a time and the period of radiation necessary to change the color from blue to green was measured for each bulb. The data are listed in Table I.

The energy values employed in the calculation of ϕ were measured with an integrating thermopile which had been calibrated against a standard lamp from the U. S. Bureau of Standards. The transmission of the reaction cell alone was determined in each experiment by taking light in-

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[1] Present address: Department of Chemistry, Carnegie Institute of Technology.

[2] Damon and Daniels, THIS JOURNAL, **55**, 2363 (1933).

[3] Damon, *Ind. Eng. Chem., Anal. Ed.*, **7**, 133 (1935).

[4] Heidt and Daniels, THIS JOURNAL, **54**, 2382 (1932).