that a completely dried resin of this type has a capacity of 5.4. The percentage swelling[3] was found to be 154% which indicates a divinylbenzene content of about 4.5% in the polymerization mix.

The preparation of the partially neutralized resins varied according to the cation involved. With tetramethyl- and tetrabutylammonium ions the calculated amount of the quaternary hydroxide solution was added to the stirred suspension of swollen resin in water over a period of 30 minutes. At the end of this period the supernatant liquid was neutral to litmus. The resin was washed with a large volume of water, then with acetone, and was dried in vacuum at 50° and then allowed to equilibrate with room air.

With cetyltrimethylammonium ion the resin suspension was stirred with a solution of the calculated amount of the quaternary bromide, and measurement of the acid titer of the supernatant liquid showed that the exchange was almost complete. The resin was washed with alcohol and water until the filtrate gave no test for bromide, and dried and air equilibrated as usual. With the sample prepared from the IR-112 four determinations each of available hydrogen and of total exchange capacity gave a figure of 31.7 ± 0.4% for the % hydrogen ion remaining. Essentially the same figure was found in samples after they had been used for the catalysis of ethyl hexanoate and of methyl phenylacetate.

With methyltribenzylammonium ion a procedure analogous to that used with cetyltrimethylammonium ion led to the replacement of only 50% of the available hydrogen ion. A second treatment of the product thus obtained with fresh quaternary iodide led to a product containing 31.7 ± 0.2% of unneutralized hydrogen ion in the IR-112 case. This value was found to be essentially unchanged after the resin had been used as catalyst with each of the three esters involved in the present study.

In determining the total exchange capacity of the resins which had been partially neutralized with cetyltrimethylammonium and with methyltribenzylammonium ions it was found to be necessary to use six successive treatments of 15 minutes each with 50 ml. of 4 N hydrochloric acid in 50% ethanol, in order to convert the sample completely to the hydrogen form.

Rate Studies.—These followed methods previously described.[3] With the IR-112 resin 1 g. of resin in the hydrogen form and correspondingly larger amounts of the partially neutralized resins were employed with 10 ml. of liquid reaction mixture. With the DVB-1/2 resin only about one-tenth of this amount of resin could be used because of the bulk of the swollen resin.

NEW YORK, N. Y.


Kinetics of Iodine-catalyzed Aromatic Bromination. II. Bromination of Toluene

BY TEIJI TSURUTA, KEN-ICHI SASAKI AND JUNJI FURUKAWA

The rates of the iodine-catalyzed bromination of toluene in carbon tetrachloride were measured in the dark at 25°. The results showed that the rate expression which had previously been derived from the assumption that the rate-determining step is the loss of hydrogen bromide from a 1:1 aromatic–bromine complex still held well in this case. The dependence of the reaction rate v upon the concentration of toluene was examined. This examination has revealed the rate expression $v = k[C_6H_5Me \cdot Br_2][BrI]^3$ to be the most probable expression within the four possible ones. An induction period was observed in the bromination. But the nature of this induction period is still obscured in the present stage.

In the first paper[1] of this series, we suggested that the previous interpretations of data on the rate of iodine-catalyzed aromatic bromination are not satisfactory. A new rate expression for the bromination was derived under the assumption that the rate-determining step is the loss of bromo-anion from a 1:1 aromatic–bromine complex

$$C_6H_6Br_2 + mBrI \xrightarrow{k} C_6H_5Br + mBrI \qquad (1)$$
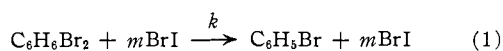
where k is the rate constant of the reaction and m is a constant. The rate v of reaction 1 can be written as

$$v = k[C_6H_6Br_2][BrI]^m \qquad (2)$$

Equation 2 was transformed into equation 3 or 4 by use of the relations between the equilibrium concentrations of the various halogen species and the total bromine and iodine in the reaction mixture

$$v = Muw^m \qquad (3)$$

or

$$\log (v/u) = \log M + m \log w \qquad (4)$$

where

$M = \{2/(1 + K_4\phi)\}^m K_2 k\phi/(1 + K_2\phi)$

$u = \{X^2/(X + Y)\} + \{(L/K_1)X^2Y^2/(X + Y)^3\}$(related to bromine)

$w = \{XY/(X + Y)\} - \{(L/K_1)X^2Y^2/(X + Y)^3\}$ (related to iodine bromide)

(1) T. Tsuruta, K. Sasaki and J. Furukawa, THIS JOURNAL, **74**, 5995 (1952).

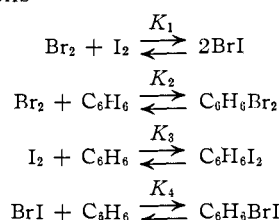ϕ = mole fraction of the free aromatic hydrocarbon
X = total concentration of bromine
Y = total concentration of iodine
$L = \{4(1 + K_2\phi)(1 + K_3\phi)/(1 + K_4\phi)\} - K_1$
$L/K_1 \doteqdot -1$

and K_1, K_2, K_3 and K_4 are the equilibrium constants of the reactions

$$Br_2 + I_2 \underset{\longleftarrow}{\overset{K_1}{\rightleftarrows}} 2BrI$$

$$Br_2 + C_6H_6 \underset{\longleftarrow}{\overset{K_2}{\rightleftarrows}} C_6H_6Br_2$$

$$I_2 + C_6H_6 \underset{\longleftarrow}{\overset{K_3}{\rightleftarrows}} C_6H_6I_2$$

$$BrI + C_6H_6 \underset{\longleftarrow}{\overset{K_4}{\rightleftarrows}} C_6H_6BrI$$

When ϕ is constant, the straight line relationship between $\log (v/u)$ and $\log w$ was expected. This relation was confirmed by recalculating Bruner's data[2] in which ϕ might be considered as a constant because the reactions were all carried out in pure benzene. From the slope of the straight line, it was found that m was equal to 3 in the case of benzene.

This paper presents the results obtained from several series of experiments in which the rates of bromination of toluene in carbon tetrachloride were measured in the dark at 25°. In series I of the experiment, every initial concentration of total bromine was kept at a specific value, while in series

(2) L. Bruner, Z. physik. Chem., **41**, 514 (1902).

II the ratio X/Y was kept constant. Series III was supplemental measurements to series I and II. The dependence of the reaction rate v upon the toluene concentration was examined in series IV. In series V_1 chloroform was used as the solvent. Series VI was undertaken to examine effects of molecular oxygen or benzoyl peroxide.

Experimental

Materials.—Commercial C.P. toluene,[3] carbon tetrachloride,[4] chloroform[5] and iodine[6] were purified in the usual manner. Bromine was prepared from C.P. grade potassium bromide and potassium bichromate.[7]

Procedure.—The reactions were carried out in a dark room. A reaction mixture was placed in a 100-ml. volumetric flask immersed in a thermostat of which the temperature was controlled to $25 \pm 0.5°$. The kinetics were measured by periodic withdrawal of the mixture. Ten milliliters of toluene was used except for series IV. A typical set of measurement is as follows:

Initial concentrations of the reactants were 12.92×10^{-2} M bromine, 2.19×10^{-2} M iodine and 155.65×10^{-2} M toluene in carbon tetrachloride. Reaction mixture was 60 ml. Two milliliters of the sample was pipetted out, added into the excess potassium iodide solution and the total halogen was titrated with $N/50$ thiosulfate. The results are shown in Fig. 5 (curve III-6). As is seen in Fig. 5, since most of the curves change their slopes very slowly, the rate v of the bromination was estimated graphically from the slope of the straight line portion between the time interval about 10 minutes, but the portions corresponding to the induction period (see below) were excluded from the rate estimation.

Results and Discussion

Determination of the Reaction Order m of Iodine Bromide.

Firstly, it has been confirmed that an uncatalyzed bromination does not take place under the experimental conditions.[8] Accordingly, in the series I, II and III, since toluene was used in excess compared with halogen, ϕ or M in equations 3 and 4 is regarded as a constant; therefore, a straight line relationship between log (v/u) and log w is expected. In Fig. 1, it can be seen that the relationship actually exists and the slope of the line is equal to about 3 (probable error, 2.2%) in just the same way as in Bruner's measurements. Therefore, for toluene, equation 3 is transformed into equation 5.

$$v = M u w^3 \qquad (5)$$

Maximum Point of the Reaction Rate.

A maximum point of the reaction rate has also been observed in this case. As was shown in the previous paper, the optimum point is to be expressed by equation 6

$$Y = [\{4(1 + K_2\phi)(1 + K_3\phi)/K_1(1 + K_4\phi)^2\}(9/4) + (3/4)]X \qquad (6)$$

where $(1 + K_2\phi)(1 + K_3\phi)/(1 + K_4\phi)^2$ is probably less than unity[1] and K_1 is several hundreds[1] in magnitude, so the first term is negligibly small compared with the second. Therefore, from equation 6, the optimum point can be expressed as equation 7

$$Y = (3/4)X \qquad (7)$$

(3) C. Schwalbe, Chem. Zentr., 76, I, 360 (1905).

(4) W. Schmitz-Dumont, Chem. Ztg., 21, 511 (1897).

(5) Chloroform was purified by shaking with sulfuric acid.

(6) F. P. Treadwell, "Lehrbuch der analytischen Chem.," Vol. II, 5 Auflage, Franz Deuticke, Leipzig und Wien. 1937, p. 551.

(7) A. Scott, J. Chem. Soc., 103, 847 (1913).

(8) Equation 3 or 4 was derived from the assumption that an uncatalyzed bromination is absent.

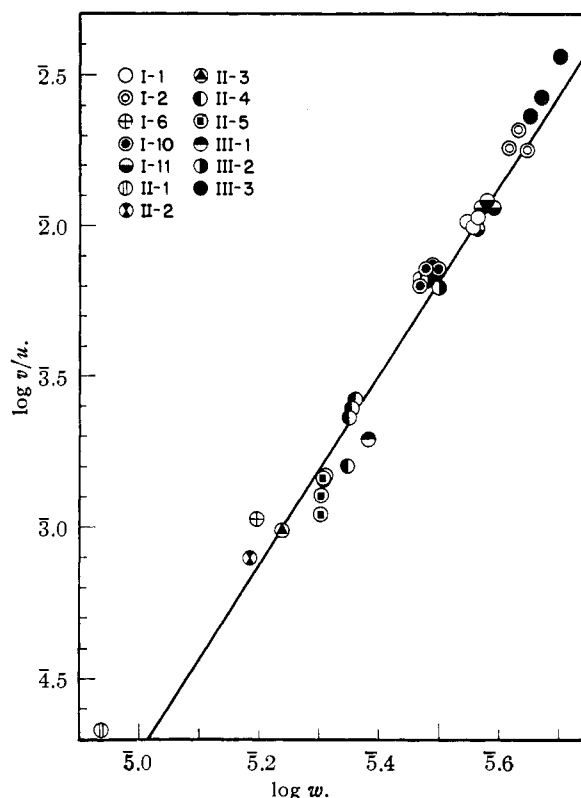Fig. 1.—log v/u vs. log w.

This relation can be confirmed from Table I or Fig. 2.

TABLE I

RELATIONSHIP BETWEEN THE RELATIVE TOTAL CONCENTRATION OF BROMINE TO IODINE AND THE REACTION RATE

No.	Y/X	Reaction rate $v \times 10^5$ (mole/ml. min.)	No.	Y/X	Reaction rate $v \times 10^5$ (mole/ml. min.)
		(i) At $X = 7.33 \times 10^{-5}$ mole/ml.			
III-4	0.300	0.0163	I-12	0.765	0.0833
III-6	.300	.0137	I- 2	0.860	.0666
III-5	.308	.0163	I- 8	1.030	.0483
I-10	.514	.0310	I- 3	1.235	.0205
I-1	.625	.0490	I- 4	1.254	.0097
		(ii) At $X = 8.00 \times 10^{-5}$ mole/ml.			
I-6	0.209	0.0053	I- 1	0.573	0.0666
III-6	.275	.0166	I-12	.700	.1333
III-5	.282	.0185	I- 7	.916	.1112
I-9	.333	.0307	I- 3	1.130	.0278
I-10	.471	.0390	I- 4	1.150	.0120

Dependence of the Reaction Rate upon the Concentration of Toluene.

As was stated above, equation 5 was derived from the assumptions

$$MeC_6H_5Br_2 + mBrI \xrightarrow{k} MeC_6H_4Br + HBr + mBrI \qquad (8)$$

and
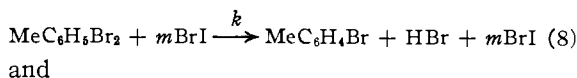
$$v = k[MeC_6H_5Br_2][BrI]^m \qquad (9)$$

Though Bruner's and the present data (series I, II and III) both fit equation 5, it cannot immediately be concluded that the reaction 8 or equation 9 is a unique mechanism, because the both measurements were all carried out with larger excess of
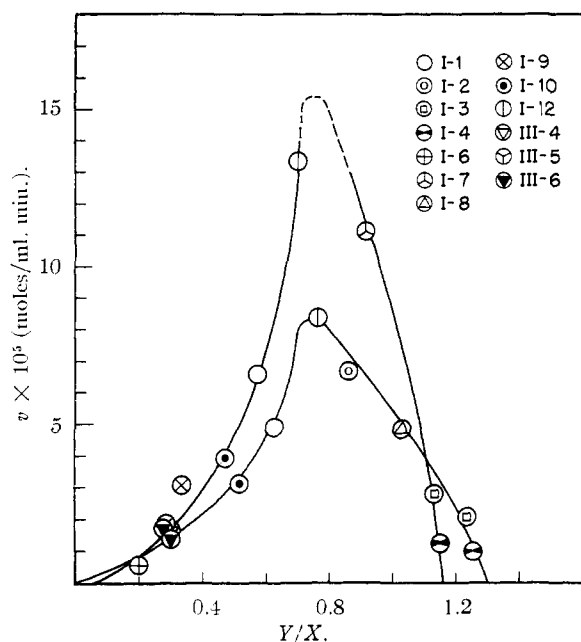
Fig. 2.—Relationship between the mole ratio (Y/X) and the reaction rate (v) at the constant total bromine (X): upper curve, $X = 8.00 \times 10^{-5}$ mole/ml.; lower curve, $X = 7.33 \times 10^{-5}$ mole/ml.

benzene and toluene, thus equation 5 could also be derived from equation 10, 11 or 12 as well as any combinations of these

$$v = k'[\text{Br}_2][\text{BrI}]^m \qquad (10)$$

$$v = k''[\text{Br}_2][\text{MeC}_6\text{H}_5\text{BrI}]^m \qquad (11)$$

or

$$v = k'''[\text{MeC}_6\text{H}_5\text{Br}_2][\text{MeC}_6\text{H}_5\text{BrI}]^m \qquad (12)$$

where m is equal to 3.

Equations 9, 10, 11 and 12 are transformed as

$v = Muw^3$	$(9')$	$v = M'uw^3$	$(10')$
$v = M''uw^3$	$(11')$	$v = M'''uw^3$	$(12')$

where

$$M = K_2 k \left(\frac{2}{1 + K_4\phi}\right)^3 \left(\frac{\phi}{1 + K_2\phi}\right) \qquad (9'')$$

$$M' = k' \left(\frac{2}{1 + K_4\phi}\right)^3 \left(\frac{1}{1 + K_2\phi}\right) \qquad (10'')$$

$$M'' = k'' \left(\frac{2K_4\phi}{1 + K_4\phi}\right)^3 \left(\frac{1}{1 + K_2\phi}\right) \qquad (11'')$$

and

$$M''' = k'''K_2 \left(\frac{2K_4}{1 + K_4\phi}\right)^3 \left(\frac{\phi^4}{1 + K_2\phi}\right) \qquad (12'')$$

If ϕ is a constant, the straight line relationship between v and uw^3 can be explained by any of the four equations. To determine the most probable mechanism within equations 9, 10, 11 and 12, series IV of the experiment was carried out varying the initial concentration of toluene from $3.11 \times 10^{-2} M$ to $312 \times 10^{-2} M$. Some examples of the measurements are illustrated in Fig. 3.

Figure 3 shows that the reaction rates are markedly affected by variations in ϕ at the low concentration of toluene, but are little affected at the high concentration. This result cannot be interpreted by any equations besides $9'$. This will be understood from Fig. 3 if the curves IV-3

(initial volume of toluene, 5 ml. ($\phi = 0.0755$), $Y = 3.5 \times 10^{-2} M$) and IV-4 (initial volume of toluene, 15 ml. ($\phi = 0.2265$), $Y = 3.5 \times 10^{-2} M$) are compared. The rates v at the point of $(X + Y) = 11.00 \times 10^{-2}M$ are estimated from the two curves to be 0.021×10^{-5} mole/ml. min. and 0.026×10^{-5} mole/ml. min., respectively.
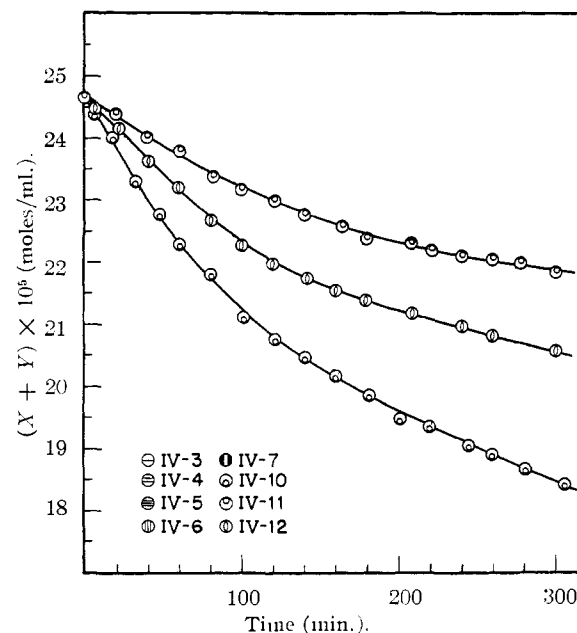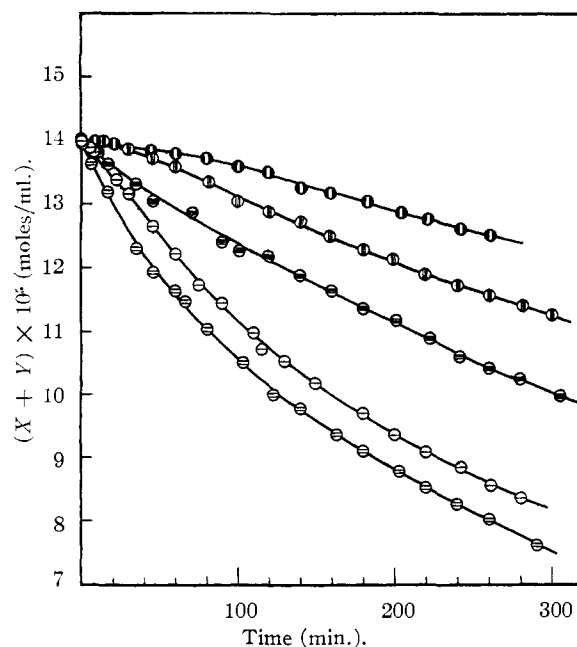

Fig. 3.—The dependence of the reaction rate upon the concentration of toluene. The initial volume of toluene: IV-3, 5 ml.; IV-4, 15 ml.; IV-5, 2 ml.; IV-6, 1 ml.; IV-7, 0.5 ml.; IV-10, 0.5 ml.; IV-11, 0.2 ml.; IV-12, 0.3 ml.

Inserting the two sets of the values for v, ϕ and K_2 ($= 1.44$[9]) into equation $9''$, the value 2.02 was obtained for K_4.

(9) R. M. Keefer and L. J. Andrews, THIS JOURNAL, 72, 4677 (1950).

Though any data on the equilibrium constant K_4 for the complex formation between toluene and iodine monobromide have not yet been presented, the value 2.02 seems to be a quite reasonable magnitude compared with $K_2 = 1.44$. On the other hand, from equation 11″, K_4 was evaluated as about 60. It is evident that 60 is too large a value for K_4. Furthermore, equation 12″ demands a negative value (about -46) of K_4. Equation 10″ can easily be excluded, because it suggests that the rate v should decrease with increasing ϕ value, K_2 or K_4 being positive.

To confirm the validity of equation 9′, it is transformed into equation 13.

$$2\left[\frac{\phi uw^3}{(1 + K_2\phi)v}\right]^{1/3} = \frac{1}{2(kK_2)^{1/3}}(1 + K_4\phi) \quad (13)$$

A linearity between $(\phi uw^3/(1 + K_2\phi)v]^{1/3}$ and ϕ is expected from equation 13. Calculating with the experimental data[10] the linearity which is illustrated in Fig. 4, has been confirmed.
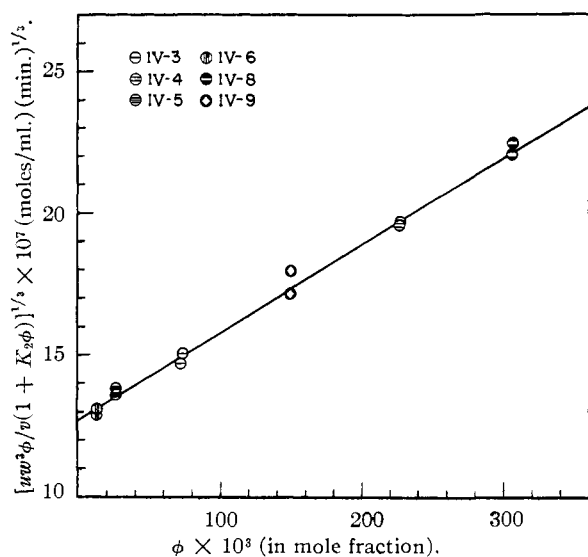

Fig. 4.—$[uw^3\phi/v (1 + K_2\phi)]^{1/6}$ vs. ϕ. The initial volume of toluene: IV-6, 1 ml.; IV-5, 2 ml.: IV-3, 5 ml.; IV-9, 10 ml.; IV-4, 15 ml.; IV-8, 20 ml.

(10) In our calculation, the mole fraction of the total unreacted toluene was regarded as that of the free toluene ϕ. It is evident that this approximation is quite reasonable where toluene was used in larger excess compared with halogens. The same approximation is still true even when the toluene concentration becomes very small. The complex concentration [MeC₆H₅Br₂] can be calculated from the equilibrium relation[11]

$$Br_2 + MeC_6H_5 \underset{}{\overset{K_2}{\rightleftarrows}} MeC_6H_5Br_2$$
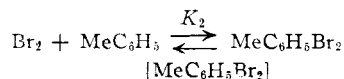
$$K_2 = \frac{[MeC_6H_5Br_2]}{([Br_2]_t - [MeC_6H_5Br_2])((MeC_6H_5)_t - (MeC_6H_5Br_2))}$$

where [MeC₆H₅Br₂] is the molar concentration of the complex, [Br₂]ₜ − [MeC₆H₅Br₂] the molar concentration of the free bromine, (MeC₆-H₅)ₜ − (MeC₆H₅Br₂) the mole fraction of free toluene, (MeC₆H₅Br₂) in this term being expressed in terms of mole fraction.

Inserting the following sets of known values into the equation, the concentration of the free toluene was found to be larger than 97% of the total, [MeC₆H₅]ₜ, at every case: $K_2 = 1.44$,[9] $Br_2 = 20 \times 10^{-2} M$, total moles of the solution $= 10 M$ and [MeC₆H₅]ₜ $= 1.33 \times 10^{-2} M$, $4.62 \times 10^{-2} M$, 15.34 $\times 10^{-2} M$, 30.19 $\times 10^{-2} M$ and 78.00 $\times 10^{-2} M$.

Even if the complexes with the other halogen species were also taken into account, at least 95% of the total toluene probably remains free under the present experimental conditions.

(11) H. A. Benesi and J. H. Hildebrand, This Journal, **71**, 2703 (1949).

If the mole fraction ϕ is very small, equation 13 can be simplified as

$$v/uw^3 = (2^3kK_2)\phi \quad (14)$$

The relation has also been proved to be in good accord with the experiments (Table II). Equation 14 means a linear dependence of the rate upon the toluene concentration in a small region of the latter. This is in agreement with Robertson's result.[12]

TABLE II

No.	$\frac{v/uw^3 \times 10^6}{\left(\frac{ml.}{moles}\right)^3\left(\frac{l.}{min.}\right)}$	Mole fraction $\phi \times 10^3$	$8kK_2 = v/uw^3\phi \times 10^3$ $\left(\frac{ml.}{moles}\right)^3\left(\frac{l.}{min.}\right)$
IV-10	22.53	4.38	5.14
IV-10	19.45	3.60	5.40
IV-10	16.06	2.95	5.44
IV-10	14.21	2.45	5.80
IV-10	14.02	2.24	6.26
IV-12	16.00	2.39	6.69
IV-12	13.96	2.06	6.78
IV-12	9.48	1.59	5.94
IV-12	6.65	1.26	5.27
IV-11	9.32	1.69	5.52
IV-11	8.10	1.26	6.41

Mean   5.88 ± 0.18

These results will make it possible to assume that equation 9 is the most probable rate law within the four equations.

Induction Period of the Bromination.—An induction period was observed in the bromination. Series V and VI of the experiment showed that the period became longer, the lower the concentrations of the reactants, or the less polar the solvent. Thus the induction period was very prominent when the reaction was carried out in carbon tetra-
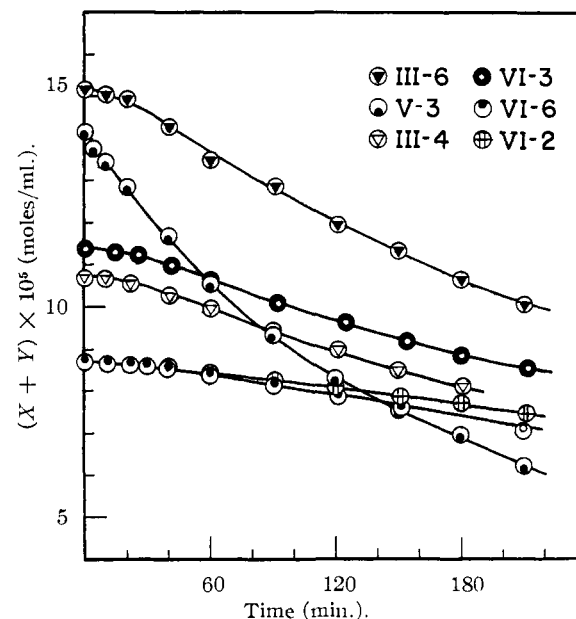

Fig. 5.—Induction period of the bromination (solvent): III-6 (CCl₄); V-3 (CHCl₃); III-4 (CCl₄); VI-3 (CCl₄ saturated with O₂); VI-6 (CCl₄); VI-2 (CCl₁ containing 5.6 mg. of Bz₂O₂).

(12) P. W. Robertson, J. E. Allan, K. N. Haldane and M. G. Simmers, J. Chem. Soc., 933 (1949).

chloride with the reactants in low concentration. Some examples of the induction period can be seen in Fig. 5.

The addition of benzoyl peroxide or molecular oxygen seems to have little effect on the induction period (Fig. 5). From the above experiment, it might be considered that the induction period was not produced from a radical reaction. It may be possible to interpret the induction period in terms of the time intervals required for the establishment of equilibria between toluene and halogen and their 1:1 complexes. But this is very unlikely because the complex formation seems to proceed very rapidly.[13]

Since retarding action of "unknown impurities"

(13) According to Benesi and Hildebrand,[11] "all solutions were freshly prepared directly before making absorption measurements." Their reproducible data of the spectra indicate the complex is formed very rapidly and the equilibrium is little shifted during the measurement.

is also not impossible, we do not present any discussions on the nature of this induction period.

From the kinetic studies on the bromination of benzene without solvent and toluene in carbon tetrachloride, it was revealed that the rate of the reaction was proportional to $[RHBr_2][BrI]^3$ (RH = benzene or toluene) in either case. However, this does not mean that the above relation is always true in other cases.[14] More detailed studies on various effects upon the reaction rate or the induction period may afford a more distinct picture as to the reaction mechanism.

Acknowledgment.—The authors wish to express their sincere thanks to Professor Ryohei Oda for his suggestive discussions, and to Mr. Shiro Ohshima for assistance in the kinetic measurement.

(14) In the bromination of toluene in chloroform, m seems to be smaller than 3.

TAKATSUKI, OSAKA-FU. JAPAN

The Infrared Spectra of Stable Pentaborane and Deuterated Pentaborane

BY HENRY J. HROSTOWSKI AND GEORGE C. PIMENTEL

Deuterated pentaborane was prepared by catalytic exchange between B_5H_9 and deuterium at room temperature. The infrared absorption spectra of gaseous B_5H_9 and B_5D_9 were obtained. Consideration of boron isotopic band structure and frequency shift on deuteration permits identification of the vibrational modes of several fundamentals. An assignment of the fundamental frequencies is given and the calculated entropy of gaseous B_5H_9 at 296°K. is compared to the experimental value. Characteristic vibrational frequencies of boranes are discussed.

The infrared and Raman spectra of stable pentaborane, B_5H_9, were first investigated by Taylor, et al.[1] Recently it has been shown that the pentaborane molecule has a C_{4v} symmetry involving a boron skeleton in the form of a tetragonal pyramid.[2,3] One hydrogen atom is bound at each pyramidal apex, accounting for five of the hydrogens. Each of the four remaining hydrogen atoms is bound to the base of the pyramid in a position equidistant from the two nearest boron atoms, thus forming four of the unusual hydrogen "bridge" bonds characteristic of the boranes whose structures are known, B_2H_6 and $B_{10}H_{14}$. Since this structure has been established, it is possible to interpret these spectra in more detail. For this purpose completely deuterated pentaborane was prepared and the infrared spectra of B_5H_9 and B_5D_9 were examined.

Experimental

Deuterated pentaborane was prepared by catalytic exchange between B_5H_9[4] and deuterium, using a chromia–alumina dehydrogenation catalyst (Harshaw No. CR-183). Before each exchange the catalyst was heated under vacuum at 450° for 24 hours. For each exchange, the gaseous mix-

(1) W. J. Taylor, C. W. Beckett, J. Y. Tung, R. B. Holden and H. L. Johnston, *Phys. Rev.*, **74**, 234 (1950).

(2) (a) K. Hedberg, M. E. Jones and V. Schomaker, THIS JOURNAL, **73**, 3538 (1951); (b) W. J. Dulmage and W. N. Lipscomb, *ibid.*, **73**, 3539 (1951).

(3) H. J. Hrostowski, R. J. Myers and G. C. Pimentel, *J. Chem. Phys.*, **20**, 518 (1952).

(4) The B_5H_9 used in these studies was kindly supplied by Dr. A. E. Newkirk of the General Electric Company.

ture of deuterium and pentaborane in a mole ratio of at least 5-to-1 was connected to a receiving trap by a tube containing 5 g. of catalyst. With the catalyst at room temperature the receiving trap was cooled to 77°K. causing the pentaborane to diffuse through the catalyst chamber. The extent of deuteration was observed by periodic comparison of the relative absorptions in the infrared spectral regions where the B–H and B–D stretch motions are expected to occur (2600 and 1900 cm.$^{-1}$, respectively). Using deuterium of 99.5% purity and six exchanges, pentaborane was obtained in which more than 98.5% of the original hydrogen had been replaced by deuterium.[5]

Infrared spectra were obtained using a Perkin–Elmer model 21 spectrophotometer with NaCl prism for the spectral region 2100–650 cm.$^{-1}$, and a Perkin–Elmer model 12C with LiF and KBr prisms for the regions 4000–2100 and 650–400 cm.$^{-1}$, respectively. The spectra were studied using a 10.5-cm. cell with KBr windows sealed on with glyptal. Silicone lubricant was used in the stopcock. Spectral slit widths are shown on the spectra. Frequency accuracy is about ±2 cm.$^{-1}$ between 700 and 1500 cm.$^{-1}$ and decreases to ±5 cm.$^{-1}$ at 2500 cm.$^{-1}$ and at 400 cm.$^{-1}$. The purity of the B_5H_9 was periodically checked by measurement of the vapor pressure at 0°. The measured values always agreed within experimental error (±2 mm.) with the accepted vapor pressure (65.3 mm.).[6] Since the most probable decomposition reactions produce either H_2 or B_2H_6, the results indicate little decomposition. Additional indication of

(5) No exchange was obtained in several preliminary experiments. (a) Deuterium and pentaborane show no exchange after 24 hours in the gas phase at 100°. (b) Increase of surface area using either glass wool or copper foil did not cause exchange in a deuterium–pentaborane mixture with a mole ratio of 5-to-1, a contact time of 15 hours, and the temperature at 100°. (c) Use of a molybdena–alumina catalyst at room temperature resulted in decomposition of pentaborane with production of other boranes including B_2H_6.

(6) H. L. Johnston, E. C. Kerr, J. T. Clarke and N. C. Hallett, Technical Report No. 6, ONR Project No. NR 058061, July 8, 1949.