barrier of 3300 ± 400 cal./mole,[24] which is in excellent agreement with the predicted value mentioned above. The entropy at 298.10°K. has been calculated to be 64.7 ± 0.3 cal./deg. per mole from molecular and spectroscopic data, the experimental value of the potential barrier and the experimental value of the entropy at the boiling point. This value at 298.10°K. is the entropy to be used in thermodynamic calculations.

In these calculations the nuclear spin entropy, $8\ R \ln 2 = 11.015$ cal./deg. per mole, has been neglected. Table IX contains a summary of the calculations.

TABLE IX

CALCULATION OF THE MAGNITUDE OF THE POTENTIAL BARRIER IN PROPANE

	$T = 231.04$°K. $P = 1$ atm. cal./deg. per mole	$T = 298.10$°K. $P = 1$ atm.
$S_{\text{trans.}} = 3/2\ R \ln M + 5/2\ R \ln T - R \ln P - 2.300$	36.02	37.28
$S_{\text{external rotation}} = R/2 \ln I_xI_yI_z \times 10^{120} + 3/2\ R \ln T - R \ln 2 - 6.851$	20.46	21.22
$S_{\text{free internal rotation}} = R/2 \ln I_r^2 \times 10^{80} + R \ln T - R \ln 9 - 3.050$	6.32	6.83
$S_{\text{vibrat.}} = \Sigma_{\nu_1 \text{ to } \nu_{25}} S_{\text{Einstein}}$	1.05	2.22
	63.85 ± 0.25	67.55
Calorimetric entropy	60.45 ± 0.10	
$S_{\text{free}} - S_{\text{hindered}}$	3.40 ± 0.3	2.86
Entropy of propane with a potential barrier of 3300 cal./mole (nuclear spin entropy not included)		64.7 ± 0.3

Summary

The heat capacity of condensed propane has been measured from 15°K. to the boiling point. The melting point was found to be 85.45 ± 0.05°K., the boiling point 231.04 ± 0.05°K., the heat of fusion 842.2 ± 0.8 cal./mole and the heat of vaporization 4487 ± 4 cal./mole.

Vapor pressure measurements have been made on liquid propane and the results have been represented by the equation (liquid propane, 166 to 231°K. (0°C. = 273.10°K.))

$$\log_{10} P_{(\text{Int. cm. Hg})} = -(1325.358/T) + 9.64920 - 0.0118950T + 0.000013420T^2$$

The density of propane gas at 298.10°K. and one atmosphere was found to be 1.8325 ± 0.0007 g./liter.

At one atmosphere at the boiling point the experimental entropy of propane (ideal gas) was evaluated as 60.45 ± 0.10 cal./deg. per mole.

The entropy of propane calculated with the assumption of free internal rotation minus the experimental entropy, $S_{\text{free}} - S_{\text{experimental}}$ (at the boiling point), was found to be 3.40 ± 0.3 cal./deg. per mole, which corresponds to a potential barrier of 3300 ± 400 cal./mole hindering the internal rotation of the methyl groups.

The entropy of propane (ideal gas, with a 3300 cal./mole barrier) at one atmosphere and 298.10°K. was found to be 64.7 ± 0.3 cal./deg. per mole (nuclear spin entropy not included). This is the entropy value to be used in thermodynamic calculations.

BERKELEY, CALIF. RECEIVED MARCH 30, 1938

[24] Refer to Table I of ref. 4.

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, DUKE UNIVERSITY]

Vapor Pressure Studies. I. Evidence for the Validity of Raoult's Law. The Systems Benzene–Diphenyl, Benzene–Benzyl Benzoate, Ethyl Acetate–Benzyl Benzoate

BY H. H. GILMANN[1] AND PAUL GROSS

Attention recently has been drawn[2] to the paucity of direct experimental evidence which is available in support of Raoult's law. In view of its importance for modern solution theory and because of the difficulty of giving a general theoretical foundation to the law, the desirability[3] of adding to this evidence has been emphasized.

In connection with a study of the stability of addition compounds in non-aqueous systems as indicated by partial pressure determinations, partial pressure data were also obtained for certain binary systems consisting of a volatile and a non-volatile component. Some of these cases furnish evidence bearing on the validity of Raoult's law.

The systems in question are benzene–diphenyl, benzene–benzyl benzoate and ethyl acetate–benzyl benzoate. Benzyl benzoate boils at 324°

[1] Part of a thesis of H. H. Gilmann submitted in partial fulfilment of the requirements for the Ph.D. in Chemistry.

[2] Guggenheim, *Trans. Faraday Soc.*, **33**, 161 (1937).

[3] Hildebrand, THIS JOURNAL, **59**, 794 (1937).

According to Rechenberg[4] it has a vapor pressure of 4.5 mm. at 156°. By means of a log p *vs.* $1/T$ plot the data given by Rechenberg above 156° were extrapolated to 95°, the highest temperature investigated here. The vapor pressure found thus was 0.3 mm. Since the partial pressure of the benzyl benzoate from the solutions could be no greater than this, its contribution to the total vapor pressure would be negligibly small. The measurement of the total vapor pressure of the systems containing benzyl benzoate would therefore be a direct determination of the partial pressure of the volatile component, benzene or ethyl acetate. A test of Raoult's law with systems of this type has the definite advantage that it involves fewer assumptions than earlier tests such as that of Zawidski with systems involving two volatile components. These necessitated the assumption of the validity of the gas laws and of Dalton's law of partial pressures in order to calculate the partial vapor pressures from the composition of the liquid condensate of the vapor phase which had been determined analytically.

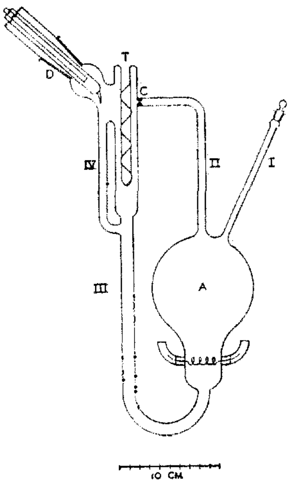

Fig. 1.

The same considerations apply to the system benzene–diphenyl in the range of temperatures between 50 and 80°. Here the partial pressure of the benzene can be placed equal to the total pressure of the solution with but negligible error. At the highest temperatures measured, 90–95°, the vapor pressure of the diphenyl is no longer negligible. However, even in these cases an estimate of the contribution that the partial pressure of its vapor makes to the total indicates that it is of the same magnitude as the experimental error. These cases will be considered in detail later.

Apparatus and Method

The total vapor pressures were determined by an ebulliometric method using a modification of the apparatus of Swietoslawski.[5] The principle of this is based on the earlier apparatus of Cottrell and need not be reviewed here. The precise boiling point elevation measurements of Washburn and Reade[6] and the extensive tests of his own apparatus by Swietoslawski[7] show that this method gives true values for the vapor–solution equilibrium and that it is capable of great precision. Our experience confirms this. The apparatus is shown in Fig. 1. The large bulb A (600 cc.) is filled through the stoppered side-tube I with a solution of known composition to a point just below the end of tube II. The solution is boiled by the platinum heating coil and also, if necessary, by external heating by means of gas micro burners heating the lower portion of A. A stream of mixed vapor and bubbles rises in II and is ejected through the constriction C onto the thermocouple well T. The volatile component is condensed in an efficient inner jacket condenser attached to D so designed as to have a minimum hold-up of returning condensate. The latter is by-passed through tube IV to prevent cooling of the vapor equilibrium chamber surrounding T. The return tube III is brought into the bottom to promote mixing and circulation. This and the heating coil which prevents bumping at low pressures are modifications in Swietoslawski's original design.

A pressure control system, containing a large thermally insulated reservoir of 30-liter capacity to minimize pressure variations, was attached to the upper end of the condenser. Pressures were read with a baromanometer similar in design to that of Germann[8] employing tubes of 13-mm. i. d. and with an electric device for setting the zero position in the arm connected to the pressure system. The scale was ruled on a strip of plate glass on a Geneva Society dividing engine and checked with a cathetometer. Pressures could be read to 0.2–0.3 mm. Atmospheric pressure readings taken at intervals with the baromanometer were compared with those of a "Greene Precision Barometer" which had a 0.75-inch (1.9-cm.) diameter mercury column and were found to be in agreement within the precision of the readings on the baromanometer. All pressures are corrected to sea level at 34° latitude and 0°.

Temperatures were determined by means of a 24-junction copper–constantan thermocouple designed according to the recommendations of White.[9] This was calibrated by the Bureau of Standards at six temperatures, the cold junctions being at zero. The Bureau's certificate furnished a four constant equation to represent the dependence of e. m. f. on temperature. A three constant equation of the type recommended by Adams[10] was found to fit their calibration data somewhat better and was therefore used. The hot junctions of the thermocouple were placed in the glass well T and covered with "Nujol" to about 1 cm. depth to ensure good thermal transfer from the walls of the well. E. m. f.'s were read with a Leeds and Northrup calibrated Type K potentiometer.

As an over-all check on the pressure and temperature readings the boiling temperatures of redistilled water under five different pressures from 352.6 to 751.8 mm. were determined and found to agree within 0.01° with those given in "International Critical Tables" in all but one case which differed by 0.02°. The vapor pressures of benzene from 40 to 80° and those of ethyl acetate from 40

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(4) Rechenberg, "Einfache und fraktionierte Destillation," 2 Aufl., Schimmel & Co., Miltitz, 1923, p. 305.

(5) Swietoslawski, *Bull. soc. chim.*, [4] **71**, 717 (1927).

(6) Washburn and Reade, THIS JOURNAL, **41**, 729 (1919).

(7) Swietoslawski, *J. Chem. Ed.*, **51**, 469 (1928).

(8) Germann, THIS JOURNAL, **36**, 3456 (1914).

(9) White, *ibid.*, **36**, 2292 (1914).

(10) Adams, *J. Wash. Acad. Sci.*, **3**, 469 (1913).

to 90° were determined. Since these compounds were carefully purified and the data in the literature, especially for ethyl acetate, show some disagreement it seems worth while to record our observed values here. Table I gives the values for benzene and Table II those for ethyl acetate.

TABLE I

t	p, mm.
36.73	157.8
40.90	188.5
45.13	223.9
50.19	272.1
55.09	326.7
60.63	389.9
64.89	461.2
69.67	544.7
74.98	646.0
79.78	753.0

TABLE II

t	p, mm.
39.46	183.7
53.35	324.1
67.96	557.1
77.04	758.6
83.01	919.8
87.58	1061.4

Large scale smoothed curves of the values in Tables I and II were drawn and the values for each even ten-degree interval found. These values are listed in the columns headed 100% in Tables V and VI. These were compared with the best values in the literature listed in "International Critical Tables." The benzene values agreed within 1 mm. with those of Young at 40, 50 and 60° and with those of Smith and Menzies at 65, 70 and 80°. The ethyl acetate values agreed within 1 mm. with those of Rechenberg[11] at 40, 60, 70, 80 and 90° but differed by 2.2 mm. at 50°, 3.4 mm. at 60°, and 2.0 mm. at 80° from those in "International Critical Tables." At 60 and 90° the difference from the latter values was less than 1 mm.

TABLE IV

Mole % Benzene 69.93, Diphenyl 30.07

t	55.26	59.63	69.55	79.88	90.26	95.65			
p, mm.	232.2	271.8	381.8	530.6	722.7	841.1			
t	55	60	65	70	75	80	85	90	95
p, mm.	233	274	327	386	453	532	615	712	826

In view of the purity of our substances and the comparisons cited for water, benzene and ethyl acetate we estimate that in the determination of the vapor pressures of pure substances the method will probably not result in errors greater than 0.03° in temperature, and 1.5 mm. in the pressure values. The errors in the case of the solutions will be discussed later.

Materials

Benzene.—A good grade was purified as follows: (a) shaken with concentrated sulfuric acid until no coloration was perceptible, (b) washed with water, then with 25% sodium hydroxide solution and then with water, (c) shaken with mercury, (d) washed with water and then dried over calcium chloride and phosphorus pentoxide, (e) distilled in a large Dufton column and the fraction distilling between 80.15 and 80.17° was then (f) fractionally recrystallized, that with melting point 5.42–5.46° being stored over sodium for use.

Diphenyl.—This was recrystallized twice from absolute alcohol, dried, and then distilled twice under about 7 mm. pressure at 112°. The product so obtained had a melting point of 68.95°.

Ethyl Acetate.—Eastman Kodak Company best grade was distilled and the fraction between 77.00 and 77.18° was dried over phosphorus pentoxide for three weeks. It was then distilled in the Dufton column, the large fraction distilling between 77.14 and 77.16° being used.

Benzyl Benzoate.—This was fractionally distilled under reduced pressure. Attempts at distillation at ordinary pressure as recommended by Berkeley[12] resulted in slight cracking and so were abandoned. The melting point of each fraction was determined and the best fractions redistilled. The best fractions from this redistillation melted from 19.10 to 19.20° and these were used.

Data

TABLE III

Mole % Benzene 69.93, Diphenyl 30.07

t	48.50	58.65	58.76	68.33	68.49	77.98	78.09	86.51	92.66
p, mm.	179.3	261.6	264.3	364.6	367.0	497.3	499.0	641.3	762.6
t	50	55	60	65	70	75	80	85	90
p, mm.	192	232	275	327	386	453	530	614	709

Samples of the actual data obtained are given in Tables III and IV. These values of the pressure at different temperatures for each solution of fixed composition were plotted and a smooth curve drawn through the points. From this the pressures at 10° intervals were found and these are tabulated in Tables V, VI and VII under the corresponding compositions in mole per cent. In the row immediately below each value of the mole per cent. (N) is tabulated the value of the quantity 100 Δ defined by the equation

$$\Delta = (p/p_0) - N$$

Here p is the observed pressure at the mole per cent. in question and p_0 is the vapor pressure of the pure volatile component at the temperature concerned, as listed in the row marked 100 mole per cent. Δ is the difference between the mole fraction computed from Raoult's law and the actual composition of the solution and is a measure of the deviations from the law. These deviations are averaged for constant temperature in the last row at the bottom and for constant composition in the last column at the right of each table.

Before discussing the significance of the data a closer evaluation of the possible errors of the

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(11) Rechenberg, Ref. 4, p. 190.

(12) Berkeley, *J. Chem. Soc.*, **109**, 520 (1916).

TABLE V

SYSTEM BENZENE–DIPHENYL, PARTIAL PRESSURE OF BENZENE IN MM.

Mole % C_6H_6	t....50	60	70	80	90	95	Av. 100 Δ
69.93	192	274	386	531	710	826	
100Δ	1.18	0.32	0.25	0.21	−0.39	0.01	0.39
78.58	215	309	435	593	802	923	
100 Δ	1.05	0.65	0.51	−0.25	−0.03	−0.43	.49
84.79	228	333	472	645	855	1007	
100 Δ	−0.35	0.59	1.03	0.41	−1.05	0.48	.65
88.96	241	352	492	672	913	1052	
100 Δ	0.30	1.30	0.50	0.19	0.46	0.12	.48
93.00	249	363	511	704	.....	.....	
100 Δ	−0.78	0.08	−0.09	0			.24
100.00	270	390	550	757	1021[a]	1181[a]	
Av. 100 Δ	0.73	0.59	0.48	0.21	0.48	0.26	

[a] Calculated from the equation given in "I. C. T.," Vol. III, p. 221.

TABLE VI

SYSTEM BENZENE–BENZYL BENZOATE, PARTIAL PRESSURE OF C_6H_6 IN MM.

Mole % C_6H_6	t....50	60	70	80	90	95	Av. 100 Δ
50.14	132	191	269	364	493	583	
100 Δ	−1.25	−1.17	−1.23	−2.06	−1.85	−0.77	1.39
61.90	169	240	337	465	627	730	
100 Δ	0.69	−0.36	−0.63	−0.47	−0.49	−0.09	0.46
73.57	199	291	405	558	745	864	
100 Δ	0.13	1.04	0.07	0.14	−0.60	−0.41	.40
80.22	216	317	443	608	822	945	
100 Δ	−0.22	1.06	0.32	0.10	0.29	−0.20	.37
91.22	248	358	503	688	932	1074	
100 Δ	0.63	0.58	0.24	−0.34	0.06	−0.28	.36
100.00	270	390	550	757	1021	1181	
Av. 100 Δ	0.38	0.84	0.50	0.62	0.66	0.35	

TABLE VII

SYSTEM ETHYL ACETATE–BENZYL BENZOATE, PARTIAL PRESSURE OF ETHYL ACETATE IN MM.

Mole % ethyl acetate	t....50	60	70	80	90	95	Av. 100 Δ
50.27	154	224	324	451	608	705	
100 Δ	3.95	3.19	4.00	3.81	3.35	3.47	3.63
67.50	199	295	424	589	798	932	
100 Δ	2.57	2.91	3.52	3.12	2.87	3.54	3.09
79.81	228	341	488	681	929	1079	
100 Δ	0.47	1.57	1.93	1.84	2.09	2.43	1.72
89.43	261	376	543	751	1025	.....	
100 Δ	2.47	0.31	1.53	0.62	0.96		1.18
100.00	284	419	597	834	1134	1312[a]	
Av. 100 Δ	2.36	1.99	2.75	2.10	2.57	3.15	

[a] Interpolated from a log p *vs.* $1/T$ plot of the values given in "I. C. T.," Vol. III, p. 219.

method as a whole in the case of the solutions is desirable. Further errors in addition to those previously evaluated as inherent in the pressure and temperature measurements, are of two sorts. The first involves the assumption that the contribution to the observed pressure of the partial pressure of the benzyl benzoate or diphenyl is negligible. This is clearly the case for benzyl benzoate whose vapor pressure is below 0.5 mm. even at 95°. The case of diphenyl requires further consideration. The only vapor pressure data at low temperatures for diphenyl available in the literature are those quoted by Montillon, Rohrbach and Badger[13] for temperatures above 69.2°. From a smoothed curve through these values the following vapor pressures were found: 0.56 mm. at 70°, 1.3 mm. at 80°, 2.5 mm. at 90° and 3.3 mm. at 95°. These figures measure the es-

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(13) Quoted in inches of mercury in *Ind. Eng. Chem.*, **23**, 764 (1931), as data from the Swann Chemical Company.

caping tendency of the pure diphenyl. Since the maximum concentration in the solutions is only 30 mole % diphenyl its escaping tendency will be materially lower in all cases. To attempt to estimate the actual partial pressures would beg the question at hand so that we will use the above figures to estimate the error due to the volatility of the diphenyl, realizing that they represent an extreme and improbable upper limit. At 50, 60, 70, and 80° this greatest possible contribution to the total pressure would be of the same order or less than the other errors in the method already discussed. At 90 and 95° the total pressure could be 0.4% greater than the partial pressure of benzene for the 30 mole per cent. diphenyl solution. These values could result in errors in the calculated mole per cent. of benzene from 0.2 to 0.3% at 90 and 95°. These estimates apply only to the highest diphenyl concentration at these temperatures. For all lower concentrations at these temperatures these greatest possible errors due to this cause would be materially smaller. Again it must be emphasized that these figures are based on the improbable premise that the partial pressure of diphenyl is the same from its 30 mole per cent. solution in benzene as its vapor pressure as a pure liquid.

The second type of errors are those due to the possible errors in the composition of the solution phase. The errors in weighing the components of a given solution were much less than one per mille and due precautions were taken that no serious loss of the volatile component occurred on transfer to the ebulliometer. This was weighed at the beginning and end of each run and only inappreciable losses in weight were found, indicating that the condenser was effectively stopping loss of the volatile component. Any possible error due to the segregation of a small, more or less constant fraction of the volatile component as it drained down from the condenser was minimized by the large volume of solution employed. The fact that reasonable differences in the rate of heating did not affect the boiling temperatures also indicated that any error due to such fractional condensation was small.

An over-all check on the reliability of the method was obtained by making two sets of duplicate runs with solutions of identical composition in which different pressures and temperatures were employed in each case. A sample of one pair of these runs is given in Tables III and IV together with the values of the pressures found independently from the smoothed curves for each run, for each 5° interval. The average discrepancy in the pressure readings for these two sets of duplicate runs was 1.0 mm. with one difference as great as 3 mm. These estimates indicate that the average error in terms of the pressures for these systems would be less than 2 mm. and that occasional errors of 3 mm. might occur. The presence of these errors could affect the values of p/p_0 by 1% or less on the average at the low pressures and by approximately 0.5 to 0.7% at the higher pressures. This might result in values of 100 Δ of these orders independent of any real deviation from Raoult's law. Furthermore, a very few values of 100 Δ of twice this amount might be expected. Examination of Tables V and VI indicates that the values of 100 Δ are on the whole less than these error limits would indicate. Therefore these two systems obey Raoult's law within the limits of error over the temperature and concentration ranges measured.

The deviations in the system ethyl acetate–benzyl benzoate as shown in Table VII are from two to three times greater than this experimental error and are all positive indicating a real, though small, positive deviation from Raoult's law in this system.

Discussion

Two assumptions are necessary for the derivation of Raoult's law on simple statistical grounds:[14] first, that the two molecular species have closely similar molecular fields and second, that they have the same size. In the system benzene–diphenyl the first of these conditions is fulfilled but not the second. Since benzyl benzoate contains the carbonyl and ether groups, both of which are polar and should have a different field than that of benzene, it might appear that neither of the conditions are fulfilled in the benzene–benzyl benzoate system. It is probable, however, that the major portion of the contacts with neighboring benzene molecules will be made by the phenyl rings and that these will shield the ester group and will to a large extent determine the character of the external molecular field of the molecule. Its field should thus be similar to that of the benzene.

The present measurements taken in conjunction with the solubility determinations of War-

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[14] Hildebrand, THIS JOURNAL, **59**, 795 (1937).

ner, Scheib and Svirbely[15] and the freezing point determinations of Washburn and Reade[16] indicate that the system benzene–diphenyl approximates fairly closely to the ideal over a large range of temperature. Furthermore, determinations of the lowering of the freezing point of benzene by benzyl benzoate made by Kendall and Booge[17] indicate that the system benzene–benzyl benzoate remains approximately ideal at considerably lower temperatures than those of the present measurements.

The system ethyl acetate–benzyl benzoate shows small but definite positive deviations from Raoult's law which persist substantially unchanged over a considerable temperature range. Two factors appear to be of importance in this case. In the first place contacts will be largely between the alkyl groups of the ethyl acetate and the phenyl groups of the benzyl benzoate. It is probable that the fields of these would be dissimilar as the solubility[14] of diphenyl in heptane deviates greatly from the ideal. Second, the shielding of the polar ester group in ethyl acetate is less complete than in benzyl benzoate. It is probable that this group accounts for an appreciable portion of the attractive forces between ethyl acetate molecules themselves. When these are surrounded in part by benzyl benzoate molecules it would seem probable that the separation between the polar ester groups in the two molecules of unlike size would be greater and their mutual attractions smaller. As a consequence the ethyl acetate molecules would show a higher escaping tendency.

If this analysis of the situation in these systems is correct it would indicate that differences in the volume of the components of a system are of secondary importance as determining its deviation from Raoult's law in comparison with differences in the effective molecular fields. The effective fields will be those of the component atoms or groups in the molecules which are most frequently in contact.

Summary

The total vapor pressures of the systems benzene–diphenyl, benzene–benzyl benzoate and ethyl acetate–benzyl benzoate have been determined from 50 to 95° over limited ranges of concentration. The bearing of these measurements on the validity of Raoult's law as applied to these systems is discussed. The benzene–diphenyl and benzene–benzyl benzoate systems follow Raoult's law within the limits of the experimental error. The ethyl acetate–benzyl benzoate system shows deviations several times larger than the experimental error, indicating a slight, but real, positive deviation from Raoult's law.

DURHAM, N. C. RECEIVED MARCH 31, 1938

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(15) Warner, Scheib and Svirbely, *J. Chem. Phys.*, **2**, 591 (1934).

(16) Washburn and Reade, *Proc. Nat. Acad. Sci.*, **1**, 191 (1915).

(17) Kendall and Booge, THIS JOURNAL, **38**, 1712 (1916).

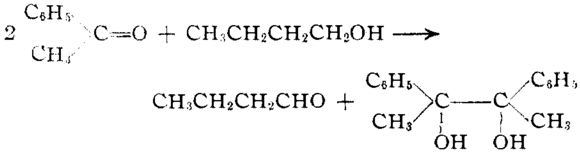
[CONTRIBUTION FROM THE DANIEL SIEFF RESEARCH INSTITUTE]

Photochemical Interaction between Ketones and Alcohols

BY CH. WEIZMANN, ERNST BERGMANN AND YEHUDA HIRSHBERG

Ciamician and Silber[1] were the first ones to observe that ketones and (primary or secondary) alcohols, under the influence of light, may undergo a process of mutual oxidation and reduction. But although the amount of available experimental material is not small,[2] the elementary photochemical processes, apparently, have not yet been elucidated. In connection with other experiments which will be referred to later on, we have started an investigation of the questions concerned, primarily of the reaction products in some typical cases

Acetophenone and butanol on irradiation with the mercury arc give the pinacol of the former substance, butanol being dehydrogenated to form butyraldehyde according to the scheme



Fifty per cent. of the theoretical amount of the aldehyde could be traced by titration according

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(1) (a) Ciamician and Silber, *Ber.*, **33**, 2911 (1900); (b) **34**, 1537 (1901); (c) **36**, 1575 (1903); (d) **43**, 945 (1910); (e) **44**, 1280 (1911); (f) **45**, 1540 (1912); (g) **47**, 1806 (1914); (h) **48**, 190 (1915).

(2) (a) Baeyer and Co., German Patent 297,993 (1921); (b) Cohen, *Rec. trav. chim.*, **39**, 243 (1921); (c) Paternò, Chieffi and Perret, *Gazz. chim. ital.*, **44**, I, 151 (1914).