MINIMUM BOILING-POINTS AND VAPOR COMPOSITION, II

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The relations existing between the liquid and vapor phase of two-component systems have for some time been a subject of investigation in this laboratory. The present paper deals more especially with the relations existing between vapor-pressure curves and minimum boiling-points, and is a continuation of the work of Pettit.¹ In that paper, it was shown that in every case where it was definitely known that the vapor-pressure curves of two components intersected, there was to be observed at some concentration a minimum boiling-point.

The converse of this Bancroft theory does not hold — that is, that all mixtures with minimum or maximum boiling-points have intersecting vapor-pressure curves since it is known that such are observed with two liquid layer systems. Besides this restriction of limited solubility, or the formation of compounds, the writer was aware of no reason why this converse might not hold — namely, that those liquids which being thoroughly consolute and forming no compounds, present an abnormal type of boiling-point curve, possess intersecting vapor-pressure curves; whereas those which show the normal type of boiling curve have vapor-pressure curves which do not intersect. This was suggested by a recent contribution to the study of maximum and minimum boiling-points.² A remark was there made "Acetone (55.5° – 56.5°) and benzene (79° – 79.5°) in the ratio of 5:1 approximately showed a tendency to gather at 57° – 58° , this fraction constantly increasing throughout the course of repeated dis-

¹ Jour. Phys. Chem. 3, 349 (1899).

² Ryland. Am. Chem. Jour. 22, 384 (1899).

tillations. But the proportions could not be found for which all of the mixture would boil at this point, as in every case there were small portions from 57° to 79° ."

Acetone and benzene are consolute, do not form compounds, and possess vapor-pressure curves which do not intersect. If as is suggested above, there is a temperature at which the composition of liquid and vapor-phases is the same, this will offer a very good case to determine what, if any, other factors intervene to prevent the application of the converse of the Bancroft theory.

The object of the work was, therefore, to examine more carefully the compositions of coexistent vapor and liquid phases of all concentrations of acetone and benzene, and then to ascertain if there were, in the cases of minimum boiling-points found experimentally, any which did not conform to the theory.

Benzene and Acetone

Material. — The benzene used (80° – 81° so-called thiophene free) was repeatedly shaken with small amounts of sulphuric acid, treated with sodium hydroxide and then fractionally distilled. During distillation, the stillhead was maintained at 78.5° . The first and last portions of the distillate were discarded while the remainder was further purified by fractional freezing. The purity was tested by determination of the boiling-point and specific gravity. For the former was found 79.44° at 740 mm — compare with Landolt and Börnstein, 79.34° at 740. The specific gravity $25^{\circ}/25^{\circ}$ was 0.8760. Compare with Herz¹ $22^{\circ}/22^{\circ}$ 0.8759, and Linebarger² $25^{\circ}/25^{\circ}$ 0.87661.

The acetone was allowed to stand for some days over calcium chloride, and then fractionally distilled, the first and last fractions being discarded. The boiling-point of this was 55.76° at 740 mm, and 56.56° at 760, — compare with 55.6° ³ 56.53° at 760 (Landolt and Börnstein). The specific gravity of the acetone was 0.7886 $25^{\circ}/25^{\circ}$ (compare with Perkin),⁴ 0.7867

¹ Ber. chem. Ges. Berlin, **3**, 2671 (1898).

² Am. Chem. Jour. **18**, 429 (1896).

³ Perkin. Jour. Chem. Soc. **45**, 478 (1882).

⁴ Perkin. Ibid. **45**, 448 (1884).

at $25^{\circ}/25^{\circ}$. This would seem to indicate the presence of an impurity, such as water, but after several fractional distillations the refractive indices of distillate and residue showed no difference whatever.

Method. — The method employed was to determine, first of all, the form of the boiling-point curve; then to obtain a method of analysis of mixtures of acetone and benzene, and finally to remove the vapor-phase which is in equilibrium with the liquid of known concentration at a definite temperature and pressure, and analyze it.

The determinations for the boiling-point curve were made at first with the ordinary Beckmann apparatus, but this being found to be too small, there was substituted for it a large test-tube, such as is used for the outer heating jacket of a vapor-density apparatus. This was provided with a long glass return condenser, while through the cork carrying the thermometer was inserted a glass tube provided with a rubber cap and pinch-cock. Through this tube, liquid could be added to the boiling-flask from the pycnometer. The ratio of the space occupied by the vapor to that occupied by the liquid in the boiling tube was approximately 3:1, and its effect is therefore negligible in this work — more so than when only one component is volatile.

In the preliminary experiments, the boiling tube was protected by an asbestos mantle and heated directly by the Bunsen flame. It proved almost impossible to obtain constant results by this method. It was, therefore, discarded for the method employed by Taylor,¹ which requires the use of a water-bath kept at a temperature which maintains regular ebullition and also a constant rate of return from the condenser. In the present work it was found that from four to seven degrees was the difference in the temperature of the bath and the boiling flask. The other precautions recorded by Taylor, such as the depth of immersion of the boiling tube in the bath and the use of large amounts of tetrahedra and garnets were also observed. Diffusion and con-

¹ Taylor. Jour. Phys. Chem. 4, 290 (1900). Compare also with Schreinemakers. Zeit. phys. Chem. 35, 460 (1900).

sequent loss of vapor were prevented by having the upper end of the condenser connected to a small U arm partially filled with mercury (which also furnishes a gauge as to the rapidity with which the water should pass through the condenser and a guide as to the evenness of ebullition); also by cooling the liquid in the boiling tube before more was added from the pycnometer. This is very essential, especially when diluting the benzene solutions with acetone.

TABLE I

I.	II.	III.	IV.	V.
Acetone %	Benzene %	Boiling- point	Boiling-point at 740 mm.	Bath
100.00	0.00	55.30°	55.76°	58°
95.01	4.99	55.72	56.18	60
90.89	9.11	56.00	56.46	61
86.92	13.08	56.40	56.86	61
83.84	16.16	56.65	57.11	61
81.22	18.78	56.90	57.36	61
79.27	20.73	57.05	57.51	61
75.83	24.17	57.40	57.86	62
73.63	26.37	57.65	58.11	63
68.96	31.04	58.25	58.71	63
62.59	37.41	59.00	59.46	64
59.42	40.58	59.45	59.91	64
54.30	45.70	60.20	60.66	62
52.24	47.76	60.50	60.96	62
46.64	53.36	61.80	62.04	66
42.13	57.87	62.60	62.84	67
38.31	61.69	63.30	63.54	68
36.21	63.89	63.70	63.94	68
32.84	67.16	64.40	64.64	70
32.58	67.42	64.50	64.74	68
30.24	69.76	65.15	65.34	68
28.39	71.61	65.50	65.74	70
26.25	73.75	66.10	66.34	71
23.44	76.56	66.90	67.10	72
19.66	80.34	68.50	68.74	75
14.15	85.85	70.30	70.54	78
11.71	88.29	72.10	72.34	81
7.22	92.78	74.20	74.44	81
6.18	93.92	74.70	74.94	81
0.00	100.00	79.20	79.44	82

The boiling-point of a weighed amount of pure acetone was first determined; when it had remained constant for fifteen minutes, the tube was cooled, a weighed amount of benzene added, and the boiling-point observed under similar conditions, the number of drops of condensed liquid returned being constant. A number of measurements were made in this manner. Then the boiling-point of benzene was taken, and to it was added acetone in known amounts. In this way the general form of the boiling-point curve was obtained. The results are shown in Table I. In the first and second columns is given the percentage composition of each component in the liquid. The third gives the boiling-point as calculated, making allowance for scale and air corrections. The first fourteen observations were made under a barometric pressure of 730.6 mm (corrected to 728.3 mm); the remainder at a pressure of 736.4 mm (corrected to 734.4 mm at Ithaca). Correction to a pressure of 740 mm requires the addition of 0.46° approximately to the first fourteen measurements, of 0.24° to the remainder. These corrected values are given in the fourth column, while the last records the temperature of the bath.

The results are presented graphically by the curve A B C in Figure I. It belongs to the normal type, presenting so far as can be seen no peculiarities which would explain the results obtained by Ryland. It was then found necessary to determine the vapor composition curve.

Analysis of Benzene and Acetone.—It was found that under the laboratory conditions prevailing, analysis by means of specific gravity determinations was the most suitable. Accordingly a number of mixtures of benzene and acetone were made up and the specific gravity $25^{\circ}/25^{\circ}$ was determined. The pycnometers used in this work were of the form recommended by Perkin.¹ The results, which are given in the following table (Table II.) were plotted as a curve and from this curve could be read off at once the composition of any acetone benzene solution whose specific gravity at 25° was known.

¹ Jour. Chem. Soc. 45, 444 (1884).

TABLE II

Acetone %	Benzene %	Sp. Gr. 25°/25°
0.00	100.00	0.8760
9.33	90.67	0.8692
18.03	81.95	0.8589
24.70	75.30	0.8531
39.31	60.69	0.8405
43.28	56.72	0.8371
63.98	36.02	0.8203
74.81	25.19	0.8104
89.51	10.49	0.7972
100.00	0.00	0.7886

Vapor Composition. — For the removal of the vapor-phase, the following scheme was adopted. To the neck of a distilling flask was sealed a side-arm leading directly to a condenser and receiver — a one-piece apparatus. The receiver consisted of a glass bulb to which was attached a stopcock, which was opened when distillation was commenced or when sufficient of the distillate had been collected, and closed during the course of a distillation. Between condenser and receiver was attached a small U-tube which, serving as manometer, indicated perfect or imperfect condensation of the vapor. The neck and side-arm were heated by means of an electric current in order to make sure that the vapor did not change composition in its passage to the condenser. Sufficient liquid was put into the distilling flask to allow 25 cc of the condensed vapor to be removed, while the boiling-point of the liquid varied little more than 0.2°.

Since a knowledge as to the composition of the boiling liquid could be readily determined with a fair degree of accuracy by reference to its boiling temperature, similar conditions were observed in heating the flask as in the determination of the boiling-point curve, the temperature of the bath being in this case, however, from five to ten degrees higher than that of the boiling liquid. The analysis of the condensed vapor was made by the density method by referring to the curve obtained from Table II.

The determination of the total error present in work on

vapor composition is at present in an unsatisfactory state. The error of analysis in the density determinations may be set at ± 1 percent, while the errors due to infrequent readings of the barometer, etc., are negligible in comparison with our lack of knowledge of the true boiling temperature of mixtures of two volatile components and the errors thus introduced. This will be referred to in a subsequent paper.

The results are presented in Table III., which requires little explanation. The composition of the boiling liquid was determined by reference to the boiling curve A B C already determined, while the analysis of the vapor was made by finding on the specific gravity curve the concentration corresponding to the specific gravity found. The vapor composition curve (A D C) is shown in Figure 1.

TABLE III

Boiling-points uncorrected	Bath	Barometer corrected	Mean boiling-points corr. to 740 mm.	Composition of boiling liquid		Specific gravity of distillate 25°/25°	Composition of distillate	
				Acetone %	Benzene %		Acetone %	Benzene %
56.9° to 57.4°	67°	739.9	57.37°	81.0	19.0	0.7988	88.5	11.5
57.4 to 57.4	62	734.0	57.62	78.0	22.0	0.7997	87.0	13.0
57.4 to 57.6	64	734.0	57.72	77.0	23.0	0.8013	85.5	14.5
58.0 to 58.2	67	739.9	58.32	72.5	27.5	0.8029	83.2	16.8
58.2 to 58.4	64	734.0	58.52	70.5	29.5	0.8053	80.8	19.2
59.8 to 59.9	63	731.8	60.17	58.5	41.5	0.8132	71.0	29.0
61.1 to 61.3	65	731.8	61.51	49.0	51.0	0.8188	64.8	35.2
62.9 to 63.7	67	731.8	63.62	38.0	62.0	0.8258	57.0	43.0
64.8 to 65.5	78	739.9	65.37	30.5	69.5	0.8311	50.8	49.2
64.5 to 65.9	69	731.8	65.52	30.0	70.0	0.8322	49.8	50.2
66.1 to 66.4	70	738.7	66.29	27.0	73.0	0.8358	45.7	54.3
67.1 to 67.5	71	738.7	67.35	23.0	77.0	0.8377	43.0	57.0
67.1 to 67.8	78	739.9	67.67	22.0	78.0	0.8394	41.4	58.6
69.0 to 70.0	77	738.7	69.55	17.5	82.5	0.8440	36.0	64.0
70.0 to 71.1	75	739.9	70.77	14.5	85.5	0.8472	32.8	67.2
72.0 to 73.0	79	738.7	72.55	10.5	89.5	0.8546	24.0	76.0
75.1 to 76.3	83	738.7	75.75	5.5	94.5	0.8650	12.0	88.0
77.0 to 78.0	87	738.7	77.55	3.0	97.0	0.8704	5.7	94.3

It will be noticed that the points as determined experimentally lie very well on smooth curves and notwithstanding the errors undoubtedly present show the general direction of the curve quite well. That the two curves approach very close to each other at the lower end explains the observation of Ryland. The 'gathering' at 57° - 58° may be illustrated by examination of the relative positions of the curves. Suppose that one takes 100 grams of a mixture of B. P. 57° at 740 mm. It contains 84

Fig. 1

percent acetone. Distil until it reaches B. P. of 58° . It then has a composition of 75 percent acetone. From the vapor-curve it is apparent that the mean composition of the vapor given off is 87.5 percent. If x represents the total weight of vapor given off, then

$$\left(84 - \frac{87.5}{100}x\right) : \left(16 - \frac{12.5}{100}x\right) = 75 : 25.$$

Therefore x is 72 and the residue is 28 grams. Such a calculation between limits may, of course, be carried out at any part of the curve, and shows that the more closely the curves approach each other, the greater is the tendency to gather.

II. Intersecting Vapor-Pressure Curves

When it was found that acetone and benzene mixtures showed no peculiarities, it was decided that all the known cases of minimum boiling-points should be examined, the work of Ryland having shown the existence of a great number. Accordingly, the vapor-pressure curves for all the common organic liquids were plotted, the major part of the data being obtained from Landolt und Börnstein's Tabellen. (See list at end of this paper.) In the diagram (Fig. 2) the pressure scale is changed at 1400 mm to read in atmospheres in order that the pressure curves may be followed to the critical temperatures and pressures.

In Table IV. have been collected all the cases which are known to have intersecting vapor-pressure curves, and which in conformity with the theory have maximum or minimum boiling points. Most of the intersections may be observed in Fig. 2. The numbers in this and in the subsequent tables denote the references given in Table XI. It will be seen that there are twenty cases which support the theory.

In Table V., there have been gathered the cases where, although the vapor-pressure curves intersect, the boiling-point curve shows no abnormality. These five cases must be classed as exceptions to the theory if the data given be accepted as correct. Preliminary experiments show that some of the measurements need to be repeated before they may with certainty be classed as exceptions. If these cases be studied at the temperature where the vapor-pressures of the two substances are the same, and it be found that all concentrations show the same vapor-pressure, a new and unexpected type will have been discovered. If, however, a maximum or minimum be found at this, but not at some other pressure, the examination of the continuous change will be of interest and value. These cases deserve a careful examination.

The other tables present data which do not bear directly on the Bancroft theory. Non-intersection of the vapor-pressure curves does not necessarily indicate that the boiling curve will be normal. Such may (VI.) or may not (V., VII., and VIII.)

Fig. 2

TABLE IV
Vapor-pressure curves intersect
Mixture has a maximum or minimum boiling-point.

Substances	References
Carbon bisulphide and methyl alcohol	1 (limit. solubility)
“ “ “ ethyl alcohol	1, 4, 20, 21
“ “ “ acetone	1, 18
“ “ “ chloroform	28 (cf. 1, reports a separation)
“ “ “ methyl acetate	1
Acetone and methyl alcohol	5, 6 (cf. 1, reports a separation)
“ “ chloroform (max)	1, 5, 7, 18, 19
Chloroform and methyl alcohol	1, 5, 6
“ “ ethyl alcohol	1, 5, 7
“ “ methyl acetate (max)	1
Methyl alcohol and carbon tetrachloride	8, 9
“ “ “ benzene	1, 9
“ “ “ methyl acetate	1, 10
“ “ “ ethyl acetate	1
Ethyl alcohol and benzene	1, 7, 9, 23
“ “ “ carbon tetrachloride	5
“ “ “ ethyl acetate	1
Propyl alcohol and benzene	1
“ “ “ water	1, 3, 5
Carbon tetrachloride and ethyl acetate	18, 19

TABLE V
Vapor-pressure curves intersect
Mixture has intermediate boiling-point

Substances	References
Acetone and ethyl alcohol	5, 7
Propyl alcohol and ethyl acetate	1
Benzene and carbon tetrachloride	9, 18, 19, 23
Benzene and ethyl acetate	1
Benzene and isobutyl alcohol	1

TABLE VI
Vapor-pressure curves do not intersect
Mixture has intermediate boiling-point

Substances	References
Ether and methyl alcohol	1, 9
" " ethyl alcohol	6
" " acetone	1, 5
" " benzene	9
" " chloroform	1, 9, 28
Carbon bisulphide and acetic acid	1
" " " benzene	9, 12, 13
" " " carbon tetrachloride	4, 21
Acetone and benzene	9, 26
" " ethyl acetate	1
" " water	5, 13, 14
Chloroform and benzene	1, 9, 24
" " carbon tetrachloride	5
" " ethyl acetate	1
Methyl alcohol and water	3, 5, 15
" " " ethyl alcohol	9
Ethyl alcohol and propyl alcohol	1
" " " water	3, 5 (cf. 34) 35
Benzene and <i>n</i> -hexane	16
" " acetic acid	1, 18
Propyl alcohol and isobutyl alcohol	1
Water and acetic acid	3

TABLE VII
Vapor-pressure curves do not intersect
Mixture has minimum boiling-point

Substances	References
Carbon bisulphide and ether	1, 20, 25, 28
Carbon bisulphide and ethyl acetate	1, 4
Acetone and methyl acetate	1
Acetone and carbon tetrachloride	5, 8

TABLE VIII

Vapor-pressure curves probably do not intersect

Mixture has maximum boiling-point

Substances	References
Water and hydrofluoric acid	36
" " hydrochloric acid	37, 38
" " hydrobromic acid	36
" " hydriodic acid	36
" " sulphuric acid	36
" " chloric acid	40
" " nitric acid	36, 37, 38, 39

TABLE IX

No data for vapor-pressures

Mixture has a minimum or a maximum boiling-point

Substances	References
Carbon bisulphide and isopropyl alcohol	I
" " ethyl bromide	I
" " methylal	18
Acetone and ethyl iodide	I
Methyl alcohol and methyl cyanide	22
" " ethyl bromide	I
" " ethyl iodide	I, 12
" " isobutyl iodide	I
Ethyl alcohol and toluene	23
" " ethyl bromide	I
" " ethyl iodide	I
" " isobutyl iodide	I
" " methyl cyanide	22
Propyl alcohol and ethyl iodide	I
" " isobutyl iodide	I
" " toluene	I
Isopropyl alcohol and ethyl iodide	I
" " ethyl acetate	I
" " isobutyl iodide	I
" " water	I, 30
Isobutyl alcohol and ethylene bromide	I
" " isobutyl iodide	I
" " toluene	I

TABLE IX—(*Continued.*)

Substance	References
Amyl alcohol and ethylene bromide	I
“ “ “ isobutyl iodide	I
“ “ “ metaxylene	I
“ “ “ paraxylene	I
“ “ “ orthoxylene	I
Allyl alcohol and toluene	I
“ “ “ water	2
Water and formic acid	3, 15
“ “ butyric acid	I, 2, 3
“ “ trimethylcarbinol	31
“ “ pyridine	18, 33
Acetic acid and toluene	I, 18
“ “ “ pyridine	27
“ “ “ metaxylene	I
Butyric acid and brombenzene	I
Propionic acid and pyridine	27
Formic acid and pyridine	27
Benzene and isopropyl alcohol	I
“ “ allyl alcohol	I
“ “ ethyl iodide	I

TABLE X

No data for vapor-pressures
Mixture has intermediate boiling-point

Substances	References
Acetone and ethyl bromide	I
Chloroform and ethyl iodide	I
“ “ ethyl bromide	I
“ “ isopropyl alcohol	I
Toluene and methyl alcohol	I
“ “ ethyl alcohol	I
“ “ isopropyl alcohol	I
“ “ amyl alcohol	I
“ “ benzene	17
“ “ chlorbenzene	18
“ “ brombenzene	18
“ “ carbon tetrachloride	23, 24
“ “ isobutyl iodide	I
“ “ chloroform	9, 24

TABLE X—(*Continued.*)

Substances	References
Ethyl iodide and carbon bisulphide	I
“ “ “ carbon tetrachloride	18
“ “ “ isobutyl alcohol	I
Isobutyl alcohol and metaxylene	I
Amyl alcohol and brombenzene	I
Ethylene bromide and metaxylene	I
“ “ “ propylene bromide	18
Water and glycerine	35
“ “ propionic acid	3
Benzene and ethylene chloride	18, 19

be the case. Tables IX. and X. present a number of cases where the measurements for the vapor-pressure curves are lacking. Such may be subjected to the test when these data are found. Other cases which should be examined are the five pairs of liquids which Kahlbaum¹ found possessing intersecting vapor-pressure curves, also the nine pairs discovered by Wöringer.² It is also interesting to note that of the 126 known cases examined in regard to the form of the boiling-point curve, 74 show maxima or minima boiling-points, while 52 present the normal form.

Cornell University,
February, 1901.

¹ Kahlbaum. *Zeit. phys. Chem.* **26**, 605 (1898).

² *Zeit. phys. Chem.* **34**, 257 (1900).

TABLE XI
List of references for boiling points

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37. Roscoe and Dittmar. *Ibid.* **112**, 327 (1859).
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39. Mitscherlich. *Pogg. Ann.* **18**, 152 (1832).
40. Roscoe. *Lieb. Ann.* **121**, 353 (1861).

TABLE XII
References for data for vapor-pressure curves

Substances	References
Ether, Regnault	Landolt und Börnstein Tabellen
Carbon bisulphide, Regnault	" " " "
Acetone, Regnault	" " " "
Chloroform, Regnault	" " " "
Methyl alcohol, Regnault	" " " "
Carbon tetrachloride, Young	Phil. Mag. (5) 34 , 512 (1892)
Benzene, Regnault, Young	Landolt und Börnstein Tabellen
Ethyl alcohol, Ramsay and Young	" " " "
Water, Regnault	" " " "
Propyl alcohol, Schmidt	" " " "
Isobutyl alcohol, Schmidt	" " " "
Acetic acid, Ramsay and Young	" " " "
Ethyl bromide, Regnault	" " " "
Ethylene bromide, Regnault	" " " "
Ethyl acetate, Young and Thomas	Jour. Chem. Soc. 63 , 1259 (1893)
Methylacetate, Young and Thomas	" " " "