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Marian Góral, David G. Shaw, Andrzej Mączyński, Barbara Wiśniewska-Gocłowska, and Paweł Oracz

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IUPAC-NIST Solubility Data Series. 96. Amines with Water
Part 3. Non-Aliphatic Amines

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The mutual solubilities and related liquid-liquid equilibria of 36 binary systems of non-aliphatic amines with water are exhaustively and critically reviewed. Reports of experimental determination of solubility that appeared in the primary literature prior to the end of 2010 are compiled. For 13 systems, sufficient data are available to allow critical evaluation. All data are expressed as mass percent and mole fraction as well as the originally reported units. © *2012 American Institute of Physics*. [http://dx.doi.org/10.1063/1.4756039]

CONTENTS

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List of Tables

1. Preface

This work incorporates compilations based on reports in the peer-reviewed scientific literature prior to the end of 2010.[1–32] To facilitate comparison of data, all original results are expressed in terms of mass percent and mole fraction as well as the units reported by the original investigators. For evaluated systems, reported solubility values are characterized as recommended, tentative, doubtful, or not evaluated according to criteria described in Sec. 1.3 of Part 1.[33]

This paper is Part 3 of a three-part compilation and evaluation of the mutual solubilities of water and amines; the first two parts have previously appeared in this journal.[33,34] A guide to this series, which provides a comprehensive overview of the procedures of data compilation and evaluation used in these volumes, is available.[35,36]

2. Solubility Data for Non-Aliphatic Amines with Water

2.1. Aniline + water

Components:	Evaluators:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-1]	A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy
(2) Water; H_2O; [7732-18-5]	of Sciences, Warsaw, Poland, January, 2012.

2.1.1. Experimental solubility studies for aniline (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 1 together with the temperature range.

TABLE 1. Experimental solubility studies for aniline (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Alexejew[1]	281–438	Mutual	Maslovskaya et al.[9]	288–318	Mutual
Campbell[2]	282–370	Mutual	Sazonov et al.[10]	302–319	Mutual
Campbell and Brown[3]	303 and 308	Mutual	Schreinemakers[11]	323–421	Mutual
Grenner et al.[4]	298–333	Mutual	Sidgwick et al.[12]	287–440	Mutual
Griswold et al.[5]	298–323	Mutual	Smith and Drexel[13]	298	(2) in (1)
Hansen et al.[6]	298	(1) in (2)	Smith et al.[14]	298	(2) in (1)
Herz[7]	295	(1) in (2)	Vaubel[15]	298	(1) in (2)
Krupatkin[8]	293–323	(2) in (1)	Vorobieva and Krupatkin[16]	298	Mutual

2.1.2. Upper critical point

The upper critical solution temperature $T_c = 439.0$ K and the corresponding mole fraction of the amine $x_c = 0.16$ were adjusted to the experimental data as described in Ref. 33, Sec. 1.2.

2.1.3. Critical evaluation of the solubility of aniline (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_1 = \ln x_c + a_1(T_c/T - 1) + a_2|(1 - T/T_c)|^{(1/3)} + a_3(1 - T/T_c), \quad (1)$$

where x_c and T_c listed above were used. The coefficients a_1, a_2, and a_3 obtained from regression of the experimental solubilities are $a_1 = 2.40$, $a_2 = -4.003$, and $a_3 = -4.63$.

Following the criteria discussed in Ref. 33, Sec. 1.3, all the data were classified as recommended, tentative, or doubtful. Points below 436.8 K deviating from the reference curve by more than 10% of the calculated mole fraction are considered doubtful. According to the criteria, the other points are tentative or recommended. The three points above 436.8 K at both sides of the critical point deviate from the reference curve by more than 0.5 K and are classified as doubtful.

All the experimental data and reference values are listed in Table 2 and shown in Fig. 1.

2.1.4. Critical evaluation of the solubility of water (2) in aniline (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = \ln (1 - x_c) + b_1(T_c/T - 1) + b_2|(1 - T/T_c)|^{(1/3)} + b_3(1 - T/T_c), \quad (2)$$

where x_c and T_c listed above were used. The coefficients b_1, b_2, and b_3 obtained from regression of the experimental solubilities are $b_1 = 2.08$, $b_2 = -0.573$, and $b_3 = -6.01$.

Following the criteria discussed in Ref. 33, Sec. 1.3, all the data were classified as recommended, tentative, or doubtful. Points below 436.8 K deviating from the reference curve by more than 10% of the calculated mole fraction are considered

doubtful. According to the criteria, the other points are tentative or recommended.

All the experimental data and reference values are listed in Table 3 and shown in Fig. 1.

Components:	Original Measurements:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3] (2) Water; H_2O; [7732-18-5]	[1]W. Alexejew, Ann. Phys. Chem. **28**, 305 (1886).

Variables:	Prepared by:
Temperature: 8–164.5 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	$100\,w_1$	x_1 (compiler)
16	3.11	6.17×10^{-3}
55	3.58	7.13×10^{-3}
77	5.25	0.0106
142	14.11	0.0308
156	21.01	0.0490
164.5	36.87	0.1016

Solubility of water in aniline

t/°C	$100\,w_2$	x_2 (compiler)
8	4.58	0.199
25	4.98	0.213
39	5.43	0.229
68	6.04	0.249
137	15.97	0.496
157.5	25.94	0.644

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. Into a tared glass tube component (1) was introduced and weighed, component (2) was added through a capillary funnel. The tube was sealed, reweighed fastened to the bulb of a Hg thermometer and repeatedly heated and cooled in a water or glycerol bath until the mixture became, respectively, homogenous and turbid.

Source and Purity of Materials:
(1) Not specified.
(2) Not specified.

Estimated Error:
Not specified.

TABLE 2. Experimental values for solubility of aniline (1) in water (2)

T/K	Experimental values x_1 (R = recommended, T = tentative, D = doubtful)	Reference values x_1
281.8	6.8×10^{-3} (T; Ref. 2)	6.77×10^{-3}
287.0	7.20×10^{-3} (T; Ref. 12)	6.90×10^{-3}
288.2	4.1×10^{-3} (D; Ref. 9), 5.5×10^{-3} (D; Ref. 9)	6.94×10^{-3}
289.2	6.17×10^{-3} (D; Ref. 1)	6.96×10^{-3}
290.8	7.26×10^{-3} (T; Ref. 12)	7.01×10^{-3}
295.2	6.85×10^{-3} (R; Ref. 7)	7.16×10^{-3}
295.9	7.30×10^{-3} (R; Ref. 12)	7.18×10^{-3}
298.2	7.0×10^{-3} (R; Ref. 4), 7.17×10^{-3} (R; Ref. 5), 7.30×10^{-3} (R; Ref. 5), 7.31×10^{-3} (R; Ref. 6), 0.0101 (D; Ref. 9), 6.2×10^{-3} (D; Ref. 9), 6.8×10^{-3} (R; Ref. 15), 5.95×10^{-3} (D; Ref. 16)	7.26×10^{-3}
298.6	7.2×10^{-3} (R; Ref. 2)	7.28×10^{-3}
300.2	7.35×10^{-3} (R; Ref. 12)	7.34×10^{-3}
302.2	7.0×10^{-3} (R; Ref. 10)	7.42×10^{-3}
303.2	6.66×10^{-3} (D; Ref. 3)	7.46×10^{-3}
303.8	7.49×10^{-3} (R; Ref. 12)	7.49×10^{-3}
305.8	7.65×10^{-3} (R; Ref. 12)	7.58×10^{-3}
307.9	7.75×10^{-3} (R; Ref. 12)	7.68×10^{-3}
312.2	8.0×10^{-3} (R; Ref. 10)	7.89×10^{-3}
313.0	7.91×10^{-3} (R; Ref. 12)	7.94×10^{-3}
318.2	0.0101 (D; Ref. 9), 0.0118 (D; Ref. 9)	8.23×10^{-3}
318.8	8.0×10^{-3} (R; Ref. 10)	8.27×10^{-3}
321.2	8.2×10^{-3} (R; Ref. 2)	8.42×10^{-3}
323.2	8.41×10^{-3} (R; Ref. 5), 8.83×10^{-3} (R; Ref. 5), 7.0×10^{-3} (D; Ref. 11)	8.55×10^{-3}
323.3	8.39×10^{-3} (R; Ref. 12)	8.56×10^{-3}
325.2	8.62×10^{-3} (R; Ref. 12)	8.69×10^{-3}
327.2	8.72×10^{-3} (R; Ref. 12)	8.83×10^{-3}
328.2	7.13×10^{-3} (D; Ref. 1)	8.90×10^{-3}
331.9	9.14×10^{-3} (R; Ref. 12)	9.19×10^{-3}
333.2	8.0×10^{-3} (D; Ref. 4)	9.30×10^{-3}
334.6	9.35×10^{-3} (R; Ref. 12)	9.42×10^{-3}
336.0	9.47×10^{-3} (R; Ref. 12)	9.54×10^{-3}
339.2	9.76×10^{-3} (R; Ref. 12)	9.83×10^{-3}
339.5	9.9×10^{-3} (R; Ref. 2)	9.86×10^{-3}
341.2	8.0×10^{-3} (D; Ref. 11)	0.0100
341.8	0.0101 (R; Ref. 12)	0.0101
345.5	0.0104 (R; Ref. 12)	0.0105
350.2	0.0106 (R; Ref. 1)	0.0110
350.3	0.0111 (R; Ref. 12)	0.0110
352.9	0.0114 (R; Ref. 12)	0.0113
354.8	0.0117 (R; Ref. 12)	0.0116
359.8	0.0125 (R; Ref. 12)	0.0123
363.1	0.0131 (R; Ref. 12)	0.0128
366.6	0.0137 (R; Ref. 12)	0.0133
368.2	0.0140 (R; Ref. 11)	0.0136
369.9	0.0144 (R; Ref. 2)	0.0139
382.0	0.0165 (R; Ref. 12)	0.0165
398.5	0.0212 (R; Ref. 12)	0.0218
415.2	0.0308 (R; Ref. 1)	0.0314
418.3	0.0341 (R; Ref. 12)	0.0341
421.2	0.0368 (R; Ref. 11)	0.0370
429.2	0.0490 (T; Ref. 1)	0.0493
439.0	0.16 (Estimated upper critical point)	0.16

T/K	Experimental values x_1 (D = doubtful)	Reference values T/K
437.7	0.102 (D; Ref. 1)	438.4
440.2	0.077 (D; Ref. 12)	436.5
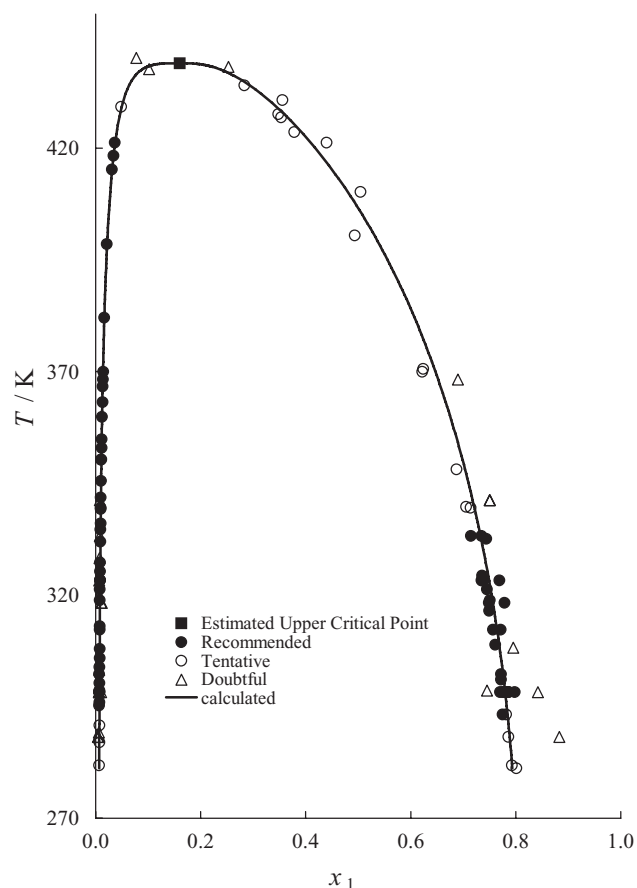
FIG. 1. Mutual solubility data for aniline (1) + water (2).

Experimental Values

Solubility of aniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
8.6	3.4	6.8×10^{-3}
25.4	3.6	7.2×10^{-3}
48.0	4.1	8.2×10^{-3}
66.3	4.9	9.9×10^{-3}
96.7	7.0	0.0144

Solubility of water in aniline

$t/°C$	$100\,w_2$	x_2 (compiler)
8.6	4.8	0.207
25.4	6.2	0.255
48.0	6.2	0.255
66.3	7.2	0.286
96.7	10.5	0.378

TABLE 3. Experimental values for solubility of water (2) in aniline (1)

T/K	Experimental values x_2 (R = recommended, T = tentative, D = doubtful)	Reference values x_2
281.2	0.199 (T; Ref. 1)	0.207
281.8	0.207 (T; Ref. 2)	0.207
288.2	0.117 (D; Ref. 9), 0.214 (T; Ref. 9)	0.212
293.2	0.225 (T; Ref. 8), 0.218 (T; Ref. 12)	0.216
298.2	0.213 (R; Ref. 1), 0.225 (R; Ref. 4),	0.220
	0.219 (R; Ref. 5), 0.221 (R; Ref. 5),	
	0.222 (R; Ref. 5), 0.158 (D; Ref. 9),	
	0.214 (R; Ref. 9), 0.230 (R; Ref. 13),	
	0.202 (R; Ref. 14), 0.214 (R; Ref. 16)	
298.6	0.255 (D; Ref. 2)	0.221
301.0	0.228 (R; Ref. 12)	0.223
302.2	0.228 (R; Ref. 10)	0.224
308.2	0.205 (D; Ref. 3)	0.231
308.8	0.239 (R; Ref. 12)	0.231
312.2	0.229 (R; Ref. 1), 0.243 (R; Ref. 10)	0.236
316.4	0.251 (R; Ref. 12)	0.241
318.2	0.221 (R; Ref. 9), 0.251 (R; Ref. 9)	0.244
318.8	0.249 (R; Ref. 10)	0.244
321.2	0.255 (R; Ref. 2)	0.248
323.2	0.261 (R; Ref. 5), 0.264 (R; Ref. 5),	0.251
	0.265 (R; Ref. 8), 0.231 (R; Ref. 11)	
324.3	0.264 (R; Ref. 12)	0.252
332.5	0.256 (R; Ref. 12)	0.266
333.2	0.265 (R; Ref. 4), 0.285 (R; Ref. 4)	0.267
339.5	0.286 (T; Ref. 2)	0.279
339.7	0.294 (T; Ref. 12)	0.279
341.2	0.25 (D; Ref. 1), 0.25 (D; Ref. 11)	0.282
348.1	0.313 (T; Ref. 12)	0.297
368.2	0.31 (D; Ref. 11)	0.348
369.9	0.378 (T; Ref. 2)	0.353
370.6	0.376 (T; Ref. 12)	0.355
400.4	0.507 (T; Ref. 12)	0.469
410.2	0.496 (T; Ref. 1)	0.520
421.2	0.56 (T; Ref. 11)	0.590
423.6	0.622 (T; Ref. 12)	0.608
426.9	0.647 (T; Ref. 12)	0.635
427.6	0.652 (T; Ref. 12)	0.641
430.7	0.644 (T; Ref. 1)	0.669
434.0	0.717 (T; Ref. 12)	0.706

T/K	Experimental values x_1 (D = doubtful)	Reference values T/K
438.2	0.747 (D; Ref. 12)	436.9

Auxiliary Information

Method/Apparatus/Procedure:
The conductimetric method was used. Samples of 20–30 g of saturated solution of (1) in (2) or of (2) in (1) were titrated with normal alkali. The conductivity apparatus comprised an audio frequency oscillator or tuning fork vibrator, Wagner ground, and variable capacity in parallel with the resistance box. The end point was at least as clear with tuning fork hummer as with the audio oscillator. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Not specified.
(2) Not specified.

Estimated Error:
Solubility: mass fractions w_1 and w_2 = 0.5% (accuracy).

Components:	Original Measurements:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3] (2) Water; H_2O; [7732-18-5]	[3]A. N. Campbell and E. M. Brown, Trans. Faraday Soc. **29**, 835 (1933).
Variables: One temperature: 30 °C	**Prepared by:** B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	$100\,w_1$	x_1 (compiler)
30	3.35	6.66×10^{-3}

Solubility of water in aniline

t/°C	$100\,w_2$	x_2 (compiler)
30	4.75	0.205

Auxiliary Information

Method/Apparatus/Procedure:
The results are part of various ternary solubility measurements. No more details were reported in the paper.

Source and Purity of Materials:
(1) Not specified.
(2) Not specified.

Estimated Error:
Not specified.

Components:	Original Measurements:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3] (2) Water; H_2O; [7732-18-5]	[4]A. Grenner, M. Klauck, R. Meinhardt, R. Schumann, and J. Schmelzer, J. Chem. Eng. Data **51**, 1009 (2006).
Variables: Temperature: 298.15 and 333.15 K	**Prepared by:** B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

T/K	$100\,w_1$ (compiler)	x_1
298.15	3.5	7×10^{-3}
333.15	4.0	8×10^{-3}

Solubility of water in aniline

T/K	$100\,w_2$ (compiler)	x_2
298.15	5.32	0.225
333.15	6.52	0.265
333.15	7.16	0.285

Auxiliary Information

Method/Apparatus/Procedure:
The photometric turbidity titration method was used. An amine sample of known mass was transferred to a tempered glass vessel. Temperature was held constant with a thermostat within a range of ±0.1 K. Water was added via automatic buret. The mixture was stirred and placed in an ultrasonic wave generator to promote phase mixing. The solubility limit was obtained by measurement of the light transmission using a photometer. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Acros Organic, Belgium; p.a. reagent grade; $n_D^{20} = 1.5862$, $d_4^{20} = 1.02172$.
(2) $n_D^{20} = 1.3336$, $d_4^{20} = 0.99816$.

Estimated Error:
Temperature: ±0.1 K.

Components:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[5]J. Griswold, J.-N. Chew, and M. E. Klecka, Ind. Eng. Chem. **42**, 1246 (1950).

Variables:
Temperature: 25 and 50 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	$100\,w_1$	x_1 (compiler)
25	3.60	7.17×10^{-3}
25	3.66	7.30×10^{-3}
50	4.20	8.41×10^{-3}
50	4.40	8.83×10^{-3}

Solubility of water in aniline

t/°C	$100\,w_2$	x_2 (compiler)
25	5.15	0.219
25	5.21	0.221
25	5.22	0.222
50	6.40	0.261
50	6.50	0.264

Auxiliary Information

Method/Apparatus/Procedure:
The cloud-point method was used. Samples of (1) or (2) were placed in glass-stoppered flasks mounted on a rotating shaft and immersed in a water bath. Compositions were determined by titrating a known sample, alternately adding the major and minor components from microburets until the two-phase point was found accurately. The flask was immersed in the bath after each titration to maintain thermal equilibrium. Finally, one drop of reagent would cause the second phase to appear or disappear. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Dow Chemical Co.; Vacuum distilled discarding first 25%; stored over solid NaOH.
(2) Distilled.

Estimated Error:
Temperature: ±0.05 °C.
Solubility: mass fractions w_1 and $w_2 = 0.1\%$.

Components:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[6]R. S. Hansen, Y. Fu, and F. E. Bartell, J. Phys. Chem. **53**, 769 (1949).

Variables:
One temperature: 25.0 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	$100\,w_1$	x_1 (compiler)
25.0	3.669	7.314×10^{-3}

Auxiliary Information

Method/Apparatus/Procedure:
The interferometer method was used. An excess of (1) was added to (2) in a mercury-sealed flask which was shaken mechanically for 48 h in an air chamber thermostated at (25.0 ± 0.1) °C. The flask was then allowed to stand for 3 h in an air bath, after which a portion of the water-rich phase was removed by means of a hypodermic syringe and compared interferometrically with the most concentrated solution of (2) that could be prepared.

Source and Purity of Materials:
(1) Source not specified; reagent grade; distilled and redistilled; b.p. 183 °C (742 mmHg).
(2) Distilled from alkaline permanganate solution.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.003\%$ (mean of three determinations).

Components:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[7]W. Herz, Ber. Dtsch. Chem. Ges. **31**, 2669 (1898).

Variables:
One temperature: 22 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	ml (1)/100 ml (2)	$100\,w_1$ (compiler)	x_1 (compiler)
22	3.481	3.44	6.85×10^{-3}

Auxiliary Information

Method/Apparatus/Procedure:
The densimetric method was used. The composition of the saturated solution was determined via calibration density measurements carried out on a series of synthetic solutions. The maximum difference between the actual and the synthetic densities was a few in the third decimal place.

Source and Purity of Materials:
(1) Not specified.
(2) $d_4^{22} = 0.9980$.

Estimated Error:
Not specified.

Components:	**Original Measurements:**
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]	[8]I. L. Krupatkin, Zh. Obshch. Khim. **25**, 1871 (1955).
(2) Water; H_2O; [7732-18-5]	

Variables:	**Prepared by:**
Temperature: 20 °C and 50 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of water in aniline

t/°C	$100\,w_2$	x_2 (compiler)
20	5.00	0.225
50	6.15	0.265

Auxiliary Information

Method/Apparatus/Procedure:
The titration method was used. The measurements were carried out in a water thermostat. Component (1) was titrated with component (2) until the mixture became turbid. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Source not specified; distilled; b.p. 184 °C.
(2) Twice distilled.

Estimated Error:
Not specified.

Components:	**Original Measurements:**
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]	[9]N. V. Maslovskaya, R. V. Mertslin, and E. F. Zhuravlev, Zh. Obshch. Khim. **43**, 2339 (1973).
(2) Water; H_2O; [7732-18-5]	

Variables:	**Prepared by:**
Temperature: 15–45 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	$100\,w_1$	x_1 (compiler)
15	2.8	5.5×10^{-3}
15	2.1	4.1×10^{-3}
25	5.0	0.0101
25	3.1	0.0062
45	5.0	0.0101
45	5.8	0.0118

Solubility of water in aniline

t/°C	$100\,w_2$	x_2 (compiler)
15	5.0	0.214
15	2.5	0.117
25	5.0	0.214
25	3.5	0.158
45	5.2	0.221
45	6.1	0.251

Auxiliary Information

Method/Apparatus/Procedure:
The titration method was used. No more details were reported in the paper. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Source not specified; dried over KOH and distilled at 188 °C.
(2) Twice distilled.

Estimated Error:
Temperature: ±0.1 °C.

Components:	**Original Measurements:**
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]	[10]V. P. Sazonov, V. V. Filippov, and N. V. Sazonov, J. Chem. Eng. Data **46**, 959 (2001).
(2) Water; H_2O; [7732-18-5]	

Variables:	**Prepared by:**
Temperature: 29.0–45.6 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	$100\,w_1$ (compiler)	x_1
29.0	3.52	7×10^{-3}
39.0	4.00	8×10^{-3}
45.6	4.00	8×10^{-3}

Solubility of water in aniline

t/°C	$100\,w_2$ (compiler)	x_2
29.0	5.41	0.228
39.0	5.85	0.243
45.6	6.03	0.249

Auxiliary Information

Method/Apparatus/Procedure:
The titration method was used. Measurements were carried out in 15-ml flat-bottomed glass vessels immersed in a 30-l water thermostat with transparent sides. A sealed magnetic stirrer was used for continual agitation of the mixtures. The temperature variations at a specified temperature were ±0.02 °C. The addition of a component was made with a microsyringe, the needle of which was pushed through the hermetic rubber stopper. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Reakchim, Russia; pure reagent grade; purity 99 mass%; dried with KOH and vacuum distilled twice; $n_D^{20} = 1.4263$, $d_4^{20} = 0.7785$.
(2) Twice distilled.

Estimated Error:
Temperature: ±0.02 °C.
Solubility: mole fractions x_1 and x_2 = 1% (relative error).

Components:	Original Measurements:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3] (2) Water; H_2O; [7732-18-5]	[11]A. H. Schreinemakers, Z. Phys. Chem. **29**, 577 (1899).
Variables:	**Prepared by:**
Temperature: 50–148 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	$100\,w_1$	x_1 (compiler)
50	3.5	7.0×10^{-3}
68	4.0	8.0×10^{-3}
95	7.0	0.0140
148	16.5	0.0368

Solubility of water in aniline

t/°C	$100\,w_2$	x_2 (compiler)
50	5.5	0.23
68	6.0	0.25
95	8.0	0.31
148	20	0.56

The upper critical solution temperature was reported to be 167 °C.

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. The temperature of appearance and disappearance of turbidity was observed visually. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Not specified.
(2) Not specified.

Estimated Error:
Not specified.

Components:	Original Measurements:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3] (2) Water; H_2O; [7732-18-5]	[12]N. V. Sidgwick, P. Pickford, and B. H. Wilsdon, J. Chem. Soc., Trans. **99**, 1122 (1911).
Variables:	**Prepared by:**
Temperature: 13.8–167.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	$100\,w_1$	x_1 (compiler)
13.8	3.611	7.20×10^{-3}
17.6	3.640	7.26×10^{-3}
22.7	3.663	7.30×10^{-3}
27.0	3.685	7.35×10^{-3}
30.6	3.752	7.49×10^{-3}
32.6	3.830	7.65×10^{-3}
34.7	3.879	7.75×10^{-3}
39.8	3.956	7.91×10^{-3}
50.1	4.187	8.39×10^{-3}
52.0	4.300	8.62×10^{-3}
54.0	4.347	8.72×10^{-3}
58.7	4.552	9.14×10^{-3}
61.4	4.652	9.35×10^{-3}
62.8	4.709	9.47×10^{-3}
66.0	4.847	9.76×10^{-3}
68.6	4.984	0.01005
72.3	5.166	0.01043
77.1	5.481	0.01110
79.7	5.640	0.01143
81.6	5.754	0.01168
86.6	6.120	0.01246
89.9	6.436	0.01314
93.4	6.690	0.01368
108.8	7.960	0.01646
125.3	10.08	0.02123
145.1	15.43	0.03410
167.0	30.18	0.07718

Solubility of water in aniline

t/°C	$100\,w_2$	x_2 (compiler)
20.0	5.12	0.218
27.8	5.41	0.228
35.6	5.73	0.239
43.2	6.08	0.251
51.1	6.49	0.264
59.3	6.24	0.256
66.5	7.47	0.294
74.9	8.09	0.313
97.4	10.45	0.376
127.2	16.58	0.507
150.4	24.12	0.622
153.7	26.14	0.647
154.4	26.60	0.652
160.8	32.88	0.717
165.0	36.40	0.747

The upper critical solution temperature was reported to be 165 °C.

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. Mixtures of aniline and water in known proportions were heated until they became homogeneous, and then cooled slowly; the temperature of separation being noted. The determinations were repeated several times.

Source and Purity of Materials:
(1) Not specified.
(2) Conductivity water.

Estimated Error:
Not specified.

Components:	Original Measurements:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]	[13]J. C. Smith and R. E. Drexel, Ind. Eng. Chem. **37**, 601 (1945).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
One temperature: 25 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility water in aniline

$t/°C$	$100\,w_2$	x_2 (compiler)
25	5.05	0.230

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. The solubility of (2) in (1) was determined by pipetting a known amount of (1) into a small glass-stoppered Erlenmeyer flask to give a total volume of about 30 ml. Component (2) was then added until a slight turbidity remained after thorough shaking. A small excess of (2) was allowed to separate, and the organic phase was carefully sampled with a pipet. The samples were analyzed for (2), using the Karl Fischer method. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Source not specified; twice distilled under vacuum and dried over NaOH; $n_D^{25} = 1.5840$, $d_4^{25} = 1.0156$.
(2) Not specified.

Estimated Error:
Not specified.

Components:	Original Measurements:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]	[14]J. C. Smith, N. J. Foecking, and W. P. Barber, Ind. Eng. Chem. **41**, 2289 (1949).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
One temperature: 25 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of water in aniline

$t/°C$	$100\,w_2$	x_2 (compiler)
25	3.62	0.202

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. The solubility of (2) in (1) was determined by the Karl Fischer method. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Source not specified; distilled under vacuum and dried over NaOH; $n_D^{25} = 1.5840$, $d_4^{25} = 1.0173$.
(2) Distilled.

Estimated Error:
Not specified.

Components:	Original Measurements:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]	[15]W. Vaubel, J. Prakt. Chem. **160**, 72 (1895).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
One temperature: 20 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
20	3.4	6.8×10^{-3}

Auxiliary Information

Method/Apparatus/Procedure:
Three methods were used. The solubility of (1) in (2) was determined by back-weighing the excess of undissolved (1), evaporating of (2) and back-weighing of excess of (1), and titration (2) with normal acid solutions. No more details were reported in the paper.

Source and Purity of Materials:
(1) Not specified.
(2) Not specified.

Estimated Error:
Not specified.

Components:	Original Measurements:
(1) Aniline (Aminobenzene, Anyvim, Benzenamine, Phenylamine); C_6H_7N; [62-53-3]	[16]L. D. Vorobieva and I. L. Krupatkin, Fazovye Ravnovesiya **8**, 26 (1974).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
One temperature: 25 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of aniline in water

t/°C	$100\,w_1$	x_1 (compiler)
25	3.00	5.95×10^{-3}

Solubility of water in aniline

t/°C	$100\,w_2$	x_2 (compiler)
25	5.00	0.214

Auxiliary Information

Method/Apparatus/Procedure:
The titration method was used. The measurements were carried out in a water thermostat. The data were reported together with the ternary system aniline—water—sodium thiocyanate. No more details were reported in the paper.

Source and Purity of Materials:
(1) Source not specified; distilled; b.p. 184.2 °C, $n_D^{25} = 1.5862$, $d_4^{25} = 1.0224$.
(2) Twice distilled.

Estimated Error:
Temperature: ±0.1 °C.

2.2. 3-Methylpyridine + water

Components:	**Original Measurements:**
(1) 3-Methylpyridine (3-Picoline, β-Picoline); C_6H_7N; [108-99-6] (2) Water; H_2O; [7732-18-5]	[17]O. Flaschner, J. Chem. Soc., Trans. **95**, 668 (1909).

Variables:	**Prepared by:**
Temperature: 49.4–152.5 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 3-methylpyridine in water

t/°C	$100\,w_1$	x_1 (compiler)
49.4[a]	26.4	0.0649
54.5	16.4	0.0366
61.0	12.7	0.0274
125.7	12.7	0.0274
140.0	16.4	0.0366
152.5[a]	26.4	0.0649

Solubility of water in 3-methylpyridine

t/°C	$100\,w_2$	x_2 (compiler)
51.4[a]	64.5	0.9038
53.7	57.7	0.8758
63.3	47.7	0.8250
83.5	40.3	0.7773
133.3	40.3	0.7773
146.5	47.7	0.8250
151.0	57.7	0.8758
152.0	64.5	0.9038

[a]Critical opalescence.

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. The operation was carried out in Jena-glass tubes. No more details were reported in the paper.

Source and Purity of Materials:
(1) Schuchardt; crystallized as zinc salt; isolated and distilled; b.p. 145.08 °C.
(2) Not specified.

Estimated Error:
Not specified.

Components:	**Original Measurements:**
(1) 3-Methylpyridine (3-Picoline, β-Picoline); C_6H_7N; [108-99-6] (2) Water; H_2O; [7732-18-5]	[18]G. Schneider, Z. Phys. Chem. (Frankfurt) **37**, 333 (1963).

Variables:	**Prepared by:**
Temperature: 20.95–139.70 °C Pressure: 140.1−353.1 MPa	B. Wisniewska-Goclowska

Experimental Values

Solubility of 3-methylpyridine in water

p/MPa	t/°C	$100\,w_1$	x_1 (compiler)
175.6	65.50	21.0	0.0489
155.1	75.00	21.0	0.0489
152.1	82.35	21.0	0.0489
163.1	91.80	21.0	0.0489
181.1	98.65	21.0	0.0489
219.1	109.15	21.0	0.0489
348.1	20.95	27.1	0.0671
309.1	31.10	27.1	0.0671
262.1	41.35	27.1	0.0671
221.1	50.40	27.1	0.0671
255.1	119.85	27.1	0.0671
353.1	139.70	27.1	0.0671
166.6	65.20	27.5	0.0684
149.6	73.70	27.5	0.0684
140.6	82.30	27.5	0.0684
148.1	90.70	27.5	0.0684
174.6	101.20	27.5	0.0684
150.6	72.90	36.7	0.1008
140.1	82.70	36.7	0.1008
146.1	89.30	36.7	0.1008
155.1	94.00	36.7	0.1008
224.6	62.40	46.3	0.1429
185.6	73.70	46.3	0.1429
176.6	82.05	46.3	0.1429
177.6	89.45	46.3	0.1429
186.6	96.70	46.3	0.1429
197.6	101.50	46.3	0.1429

Auxiliary Information

Method/Apparatus/Procedure:
The cloud-point method was used. The turbidity of a known composition sample was observed visually in the thermostated high-pressure steel chamber. Pressure was measured with Bourdon-manometers of different ranges.

Source and Purity of Materials:
(1) Gesellschaft für Teerverwertung mbH Duisburg-Meiderech; distilled at normal pressure; $n_D^{20} = 1.5066 \pm 0.0001$.
(2) Twice distilled.

Estimated Error:
Temperature: $<\pm 0.1$ °C.
Solubility: $<\pm 0.2$ mol %.
Pressure: $<\pm 0.5$ MPa at $p < 160$ MPa,
$<\pm 1.0$ MPa at 160 MPa $< p < 250$ MPa,
$<\pm 5.0$ MPa at $p > 250$ MPa.

2.3. 2,3-Dimethylpyridine + water

Components:
(1) 2,3-Dimethylpyridine (2,3-Lutidine); C_7H_9N; [583-61-9]
(2) Water; H_2O; [7732-18-5]

Evaluators:
A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland, January, 2012.

2.3.1. Experimental solubility studies for 2,3-dimethylpyridine (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 4 together with the temperature range.

2.3.2. Critical points

The lower critical point was determined from experimental data by the procedure described in Ref. 33, Sec. 1.2. The upper critical point was reported by Cox,[19]

The lower critical point is: $T_L = 289.2$ K and $x_L = 0.048$,
The upper critical point is: $T_U = 465.8$ K and $x_U = 0.095$,

where x_L and x_U are the critical mole fractions of 2,3-dimethylpyridine.

2.3.3. Critical evaluation of the solubility of 2,3-dimethylpyridine (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

TABLE 4. Experimental solubility studies for 2,3-dimethylpyridine (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Cox[19]	290 and 466	(1) in (2)	Stephenson[20]	289–363	Mutual

TABLE 5. Experimental values for solubility of 2,3-dimethylpyridine (1) in water (2)

T/K	Experimental values x_1	Smoothed values T/K
289.2	0.0336 (Ref. 20)	289.5

T/K	Experimental values x_1	Smoothed values x_1
289.2	0.0480 (Estimated lower critical point)	0.0386
291.2	0.0227 (Ref. 20)	0.0231
293.2	0.0191 (Ref. 20)	0.0191
298.2	0.0156 (Ref. 20)	0.0144
303.2	0.0123 (Ref. 20)	0.0120
308.2	0.0106 (Ref. 20)	0.0106
313.2	9.5×10^{-3} (Ref. 20)	9.6×10^{-3}
323.2	8.4×10^{-3} (Ref. 20)	8.4×10^{-3}
333.2	7.6×10^{-3} (Ref. 20)	7.8×10^{-3}
343.2	7.5×10^{-3} (Ref. 20)	7.5×10^{-3}
353.2	7.6×10^{-3} (Ref. 20)	7.5×10^{-3}
363.2	7.6×10^{-3} (Ref. 20)	7.6×10^{-3}

T/K	Experimental values x_1	Smoothed values T/K
465.8[a]	0.095 (Ref. 19)	465.9

[a]Upper critical solution temperature.

$$\ln x_1 = z_L \ln x_L + z_U \ln x_U + c_1 \, z_L z_U / T + c_2 |z_L z_U|^{1/3} + c_3 z_L z_U, \tag{3}$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{4}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients c_1, c_2, and c_3 obtained from regression of the experimental solubilities are $c_1 = -1277.82$, $c_2 = -3.263$, and $c_3 = 3.16$.

The solubility of (1) in (2) is reported by Stephenson[20] only and therefore the data are not classified. All the experimental data and smoothed values are listed in Table 5 and shown in Fig. 2.

2.3.4. Critical evaluation of the solubility of water (2) in 2,3-dimethylpyridine (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = z_L \ln (1 - x_L) + z_U \ln (1 - x_U) + d_1 \, z_L z_U / T + d_2 |z_L z_U|^{1/3} + d_3 \, z_L z_U, \tag{5}$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{6}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients d_1, d_2, and d_3 obtained from regression of the experimental solubilities are $d_1 = 131.77$, $d_2 = -0.139$, and $d_3 = -1.23$.

With the exception of one point of Cox[19] at 289.7 K, the other part of the solubility curve was measured by Stephenson[20] only. Therefore, the data were not classified. All the experimental data and the smoothed values are listed in Table 6 and shown in Fig. 2.

FIG. 2. Mutual solubility data for 2,3-dimethylpyridine (1) + water (2).

Experimental Values

Solubility of water in 2,3-dimethylpyridine

$t/°C$	$100 w_2$	x_2 (compiler)
16.5	74	0.944
192.6	61.5	0.9047

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. Solution temperatures above room temperature were determined with the aid of an electrically heated metal block; a sealed tube containing a known mixture of (1) and (2) was fixed in a cavity in the middle of the block and could be observed through the windows. The liquid layers were mixed by rotating the block. For solution temperatures below room temperature, the tube containing the mixture was fixed in a bucket which was then lowered into an unsilvered Dewar vessel filled with methanol and shaken manually. The temperature of the bath was lowered by addition of solid carbon dioxide and then allowed to rise at a rate of 0.1 °C per min until the cloud point was reached.

Source and Purity of Materials:
(1) Kodak Ltd.; purified by a combination of fractional distillation and fractional freezing; contained 0.2% of 2,4-dimethylpyridine and 0.04% of water.
(2) Not specified.

Estimated Error:
Temperature: ±0.2 at 16.5 °C and ±0.4 at 192.6 °C.
Solubility: mass fraction $w_1 = ±1\%$ (standard deviation).

Components:	**Original Measurements:**
(1) 2,3-Dimethylpyridine (2,3-Lutidine); C_7H_9N; [583-61-9]	[19]J. D. Cox, J. Chem. Soc. **1954**, 3183.
(2) Water; H_2O; [7732-18-5]	
Variables:	**Prepared by:**
Temperature: 16.5 and 192.6 °C	B. Wisniewska-Goclowska

TABLE 6. Experimental values for solubility of water (2) in 2,3-dimethylpyridine (1)

T/K	Experimental values x_2	Smoothed values T/K
289.2	0.925 (Ref. 20)	290.0

T/K	Experimental values x_2	Smoothed values x_2
289.7	0.944 (Ref. 19)	0.932
291.2	0.919 (Ref. 20)	0.915
293.2	0.893 (Ref. 20)	0.899
298.2	0.870 (Ref. 20)	0.869
303.2	0.841 (Ref. 20)	0.845
308.2	0.820 (Ref. 20)	0.823
313.2	0.802 (Ref. 20)	0.804
323.2	0.773 (Ref. 20)	0.771
333.2	0.752 (Ref. 20)	0.744
343.2	0.716 (Ref. 20)	0.722
353.2	0.708 (Ref. 20)	0.705
363.2	0.690 (Ref. 20)	0.693

Components:	**Original Measurements:**
(1) 2,3-Dimethylpyridine (2,3-Lutidine); C_7H_9N; [583-61-9]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).
(2) Water; H_2O; [7732-18-5]	
Variables:	**Prepared by:**
Temperature: 16.0−90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,3-dimethylpyridine in water

$t/°C$	$100 w_1$	x_1 (compiler)
16.0	17.15	0.03364
18.0	12.15	0.02273
20.0	10.40	0.01915
25.0	8.60	0.01558
30.0	6.91	0.01233
35.0	5.99	0.01060
40.0	5.37	9.45×10^{-3}
50.0	4.82	8.44×10^{-3}
60.0	4.37	7.63×10^{-3}
70.0	4.30	7.50×10^{-3}
80.0	4.33	7.55×10^{-3}
90.0	4.35	7.59×10^{-3}

Solubility of water in 2,3-dimethylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
16.0	67.6	0.925
18.0	65.6	0.919
20.0	58.5	0.893
25.0	52.9	0.870
30.0	47.0	0.841
35.0	43.4	0.820
40.0	40.5	0.802
50.0	36.4	0.773
60.0	33.8	0.752
70.0	29.8	0.716
80.0	29.0	0.708
90.0	27.2	0.690

The lower critical solution temperature was reported to be 16 °C.

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.20\%$ and mass fraction $w_2 = 1.0\%$ (standard deviation).

2.4. 2,4-Dimethylpyridine + water

Components:	**Evaluators:**
(1) 2,4-Dimethylpyridine (2,4-Lutidine); C_7H_9N; [108-47-4]	A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of
(2) Water; H_2O; [7732-18-5]	Sciences, Warsaw, Poland, January, 2012.

2.4.1. Experimental solubility studies for 2,4-dimethylpyridine (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 7 together with the temperature range.

2.4.2. Critical points

The Lower Critical Point was determined from experimental data by the procedure described in Ref. 33, Sec. 1.2; the Upper Critical Point was reported by Andon and Cox,[21]

TABLE 7. Experimental solubility studies for 2,4-dimethylpyridine (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Andon and Cox[21]	297 and 462	(1) in (2)	Stephenson[20]	297–363	Mutual
Jones and Speakman[22]	296–342	Mutual			

The lower critical point is: $T_L = 296.1$ K and $x_L = 0.059$,

The upper critical point is: $T_U = 461.9$ K and $x_U = 0.090$,

where x_L and x_U are the critical mole fractions of the amine.

2.4.3. Critical evaluation of the solubility of 2,4-dimethylpyridine (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_1 = z_L \ln x_L + z_U \ln x_U + c_1\, z_L z_U /T + c_2 |z_L z_U|^{1/3} + c_3\, z_L z_U, \tag{7}$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{8}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients c_1, c_2, and c_3 obtained from regression of the experimental solubilities are $c_1 = -3060.70$, $c_2 = -3.671$, and $c_3 = 8.54$.

For this system, there are no data reported by three independent sources. Thus, following the criteria discussed in Ref. 33, Sec. 1.3, the data are classified as tentative or doubtful. All the experimental data and smoothed values are listed in Table 8 and shown in Fig. 3.

2.4.4. Critical evaluation of the solubility of water (2) in 2,4-dimethylpyridine (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = z_L \ln(1 - x_L) + z_U \ln(1 - x_U) + d_1\, z_L z_U /T + d_2 |z_L z_U|^{1/3} + d_3\, z_L z_U, \tag{9}$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{10}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients d_1, d_2, and d_3 obtained from regression of the experimental solubilities are $d_1 = 361.97$, $d_2 = -0.215$, and $d_3 = -1.52$.

For this system, there are no data reported by three independent sources. Thus, following the criteria discussed in Ref. 33, Sec. 1.3, the data are classified as tentative or doubtful. All the experimental data and smoothed values are listed in Table 9 and shown in Fig. 3.

TABLE 8. Experimental values for solubility of 2,4-dimethylpyridine (1) in water (2)

T/K	Experimental values x_1 (T = tentative)	Smoothed values T/K
296.2	0.0415 (T; Ref. 22)	296.3

T/K	Experimental values x_1 (T = tentative, D = doubtful)	Smoothed values x_1
296.1	0.059 (Estimated lower critical point)	0.0590
296.6	0.0544 (D; Ref. 21)	0.0352
297.2	0.0298 (T; Ref. 20)	0.0297
299.2	0.0220 (T; Ref. 20)	0.0220
301.2	0.0187 (T; Ref. 20)	0.0183
303.2	0.0159 (T; Ref. 20)	0.0160
308.2	0.0123 (T; Ref. 20), 0.0125 (T; Ref. 22)	0.0124
313.2	0.0103 (T; Ref. 20)	0.0105
318.2	9.1×10^{-3} (T; Ref. 20)	9.23×10^{-3}
323.2	8.5×10^{-3} (T; Ref. 20)	8.41×10^{-3}
327.5	8.66×10^{-3} (T; Ref. 22)	7.92×10^{-3}
333.2	7.5×10^{-3} (T; Ref. 20)	7.48×10^{-3}
343.2	7.2×10^{-3} (T; Ref. 20)	7.12×10^{-3}
353.2	7.2×10^{-3} (T; Ref. 20)	7.10×10^{-3}
363.2	7.2×10^{-3} (T; Ref. 20)	7.33×10^{-3}

T/K	Experimental values x_1	Smoothed values T/K
461.9[a]	0.0899 (Ref. 21)	461.9

[a]Upper critical solution temperature.

TABLE 9. Experimental values for solubility of water (2) in 2,4-dimethylpyridine (1)

T/K	Experimental values x_2 (T = tentative)	Smoothed values T/K
296.2	0.927 (T; Ref. 22)	296.2

T/K	Experimental values x_2 (T = tentative, D = doubtful)	Smoothed values x_2
297.2	0.903 (T; Ref. 20)	0.903
299.2	0.881 (T; Ref. 20)	0.884
300.4	0.880 (T; Ref. 22)	0.876
301.2	0.871 (T; Ref. 20)	0.872
303.2	0.861 (T; Ref. 20)	0.861
308.2	0.837 (T; Ref. 20)	0.841
312.2	0.830 (T; Ref. 22)	0.826
313.2	0.833 (T; Ref. 20)	0.823
318.2	0.806 (T; Ref. 20)	0.808
323.2	0.799 (T; Ref. 20)	0.794
326.2	0.781 (T; Ref. 22)	0.786
333.2	0.771 (T; Ref. 20)	0.769
341.7	0.727 (D; Ref. 22)	0.751
343.2	0.746 (T; Ref. 20)	0.748
353.2	0.729 (T; Ref. 20)	0.730
363.2	0.718 (T; Ref. 20)	0.717

Components:	Original Measurements:
(1) 2,4-Dimethylpyridine (2,4-Lutidine); C_7H_9N; [108-47-4] (2) Water; H_2O; [7732-18-5]	[21]R. J. L. Andon and J. D. Cox, J. Chem. Soc. **1952** 4601.

Variables:	Prepared by:
Temperature: 23.4 °C and 188.7 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,4-dimethylpyridine in water

t/°C	$100\,w_1$	x_1 (compiler)
23.4 ± 0.2[a]	25.5	0.0544
188.7 ± 0.3[b]	37.0	0.0899

[a]Lower critical solution temperature.
[b]Upper critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
The titration synthetic method was used. Both components were weighed into thick-wall Pyrex tubes which were then sealed. Phase transition temperatures were determined with mixtures in a high-capacity oil bath the temperature of which, measured by means of a set of laboratory standard thermometers graduated in fifths of a degree, could be raised or lowered at any rate down to 0.1 °C per min. For work above 200 °C, an electrically heated metal block was used.

Source and Purity of Materials:
(1) Source not specified; purity 99.65 mol %; the residual impurities were related alkylpyridines and water, hydrocarbons were absent.
(2) Not specified.

Estimated Error:
Temperature: see above.

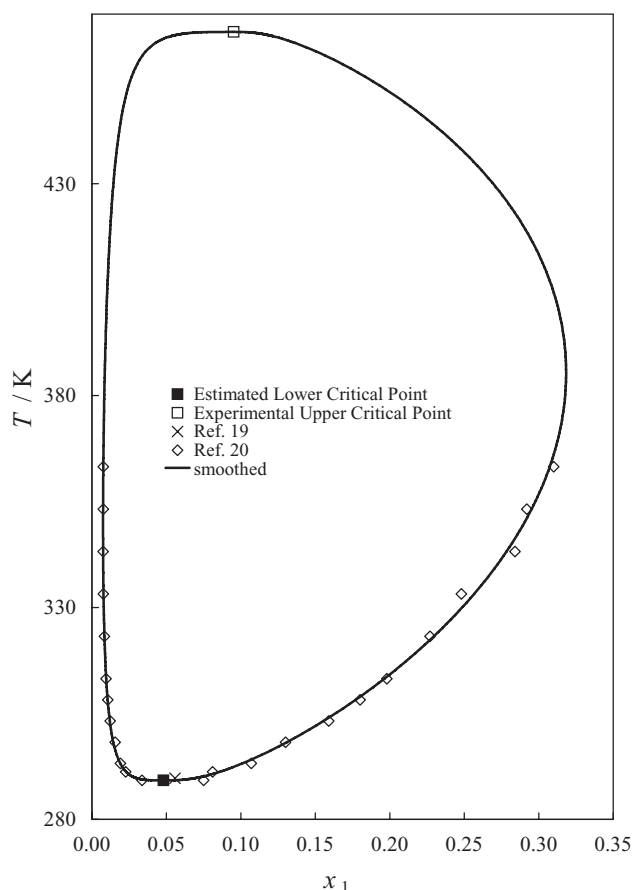
FIG. 3. Mutual solubility data for 2,4-dimethylpyridine (1) + water (2).

Components:
(1) 2,4-Dimethylpyridine (2,4-Lutidine); C_7H_9N; [108-47-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[22]W. J. Jones and J. B. Speakman, J. Am. Chem. Soc. **43**, 1867 (1921).

Variables:
Temperature: 23.0–150 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,4-dimethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
23.0	20.47	0.04147
35.0	7.00	0.01250
54.3	4.96	8.66×10^{-3}

Solubility of water in 2,4-dimethylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
23.0	67.96	0.9265
27.2	55.11	0.8795
39.0	45.14	0.8303
53.0	37.50	0.7811
68.5	30.92	0.7269
>150	23.14	0.6416
>150	15.99	0.5309

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. No more details were reported in the paper.

Source and Purity of Materials:
(1) Source not specified; redistilled before use from solid sodium hydroxide.
(2) Conductivity water.

Estimated Error:
Not specified.

Components:
(1) 2,4-Dimethylpyridine (2,4-Lutidine); C_7H_9N; [108-47-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).

Variables:
Temperature: 24.0−90.0 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,4-dimethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
24.0	15.43	0.02977
26.0	11.78	0.02196
28.0	10.20	0.01874
30.0	8.76	0.01589
35.0	6.89	0.01229
40.0	5.84	0.01032
45.0	5.16	9.07×10^{-3}
50.0	4.83	8.46×10^{-3}
60.0	4.28	7.46×10^{-3}
70.0	4.14	7.21×10^{-3}
80.0	4.11	7.16×10^{-3}
90.0	4.15	7.23×10^{-3}

Solubility of water in 2,4-dimethylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
24.0	61.0	0.903
26.0	55.5	0.881
28.0	53.1	0.871
30.0	51.1	0.861
35.0	46.3	0.837
40.0	45.6	0.833
45.0	41.2	0.806
50.0	40.0	0.799
60.0	36.2	0.771
70.0	33.1	0.746
80.0	31.1	0.729
90.0	30.0	0.718

The lower critical solution temperature was reported to be 24 °C.

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.20\%$ and mass fraction $w_2 = 1.3\%$ (standard deviation).

2.5. 2,5-Dimethylpyridine + water

Components:
(1) 2,5-Dimethylpyridine (2,5-Lutidine); C_7H_9N; [589-93-5]
(2) Water; H_2O; [7732-18-5]

Evaluators:
A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland, January, 2012.

2.5.1. Experimental solubility studies for 2,5-dimethylpyridine (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 10 together with the temperature range.

TABLE 10. Experimental solubility studies for 2,5-dimethylpyridine (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Andon and Cox[21]	286 and 480	(1) in (2)	Stephenson[20]	286–363	Mutual

2.5.2. Critical points

The Lower Critical Point was determined from experimental data by the procedure described in Ref. 33, Sec. 1.2; the Upper Critical Point was reported by Andon and Cox,[21]

The lower critical point is: $T_L = 286.2$ K and $x_L = 0.056$,

The upper critical point is: $T_U = 480.1$ K and $x_U = 0.093$,

where x_L and x_U are the critical mole fractions of the amine.

2.5.3. Critical evaluation of the solubility of 2,5-dimethylpyridine (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_1 = z_L \ln x_L + z_U \ln x_U + c_1\, z_L z_U/T \\ + c_2\, |z_L z_U|^{1/3} + c_3\, z_L z_U, \tag{11}$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{12}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients $c_1, c_2,$ and c_3 obtained from regression of the experimental solubilities are $c_1 = -2100.08$, $c_2 = -3.905$, and $c_3 = 5.85$.

At the lower critical region, a point at $T = 286.2$ K reported by Stephenson[20] agrees with a single point of Andon and Cox[21] given in Sec. 2.5.4. These two points are classified as tentative. The other points of Stephenson[20] are not supported by any other source and are not classified. All the experimental data and smoothed values are listed in Table 11 and shown in Fig. 4.

2.5.4. Critical evaluation of the solubility of water (2) in 2,5-dimethylpyridine (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = z_L \ln(1 - x_L) + z_U \ln(1 - x_U) + d_1\, z_L z_U/T \\ + d_2\, |z_L z_U|^{1/3} + d_3\, z_L z_U, \tag{13}$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{14}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients $d_1, d_2,$ and d_3 obtained from regression of the experimental solubilities are $d_1 = -1547.76$, $d_2 = -0.178$, and $d_3 = 3.03$.

TABLE 11. Experimental values for solubility of 2,4-dimethylpyridine (1) in water (2)

T/K	Experimental values x_1 (T = tentative)	Smoothed values T/K
286.2	0.0381 (T; Ref. 20)	286.4

T/K	Experimental values x_1	Smoothed values x_1
286.2	0.0560 (Estimated lower critical point)	0.0560
287.2	0.0294 (Ref. 20)	0.0288
289.2	0.0208 (Ref. 20)	0.0211
291.2	0.0177 (Ref. 20)	0.0175
293.2	0.0148 (Ref. 20)	0.0153
298.2	0.0117 (Ref. 20)	0.0119
303.2	0.0102 (Ref. 20)	0.0100
313.2	8.5×10^{-3} (Ref. 20)	7.9×10^{-3}
323.2	6.4×10^{-3} (Ref. 20)	6.9×10^{-3}
333.2	6.1×10^{-3} (Ref. 20)	6.3×10^{-3}
343.2	6.4×10^{-3} (Ref. 20)	6.1×10^{-3}
353.2	6.0×10^{-3} (Ref. 20)	6.1×10^{-3}
363.2	6.2×10^{-3} (Ref. 20)	6.2×10^{-3}

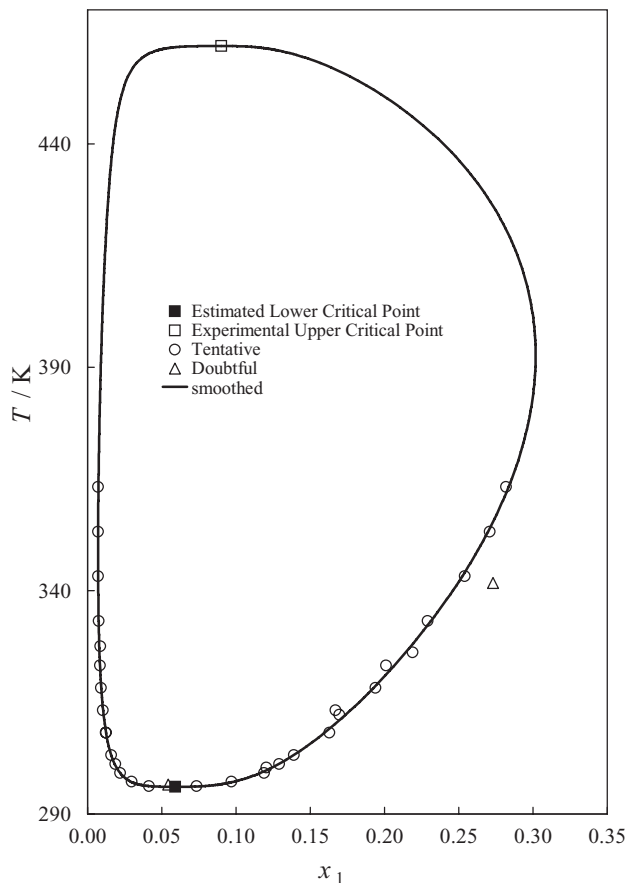| T/K	Experimental values x_1	Smoothed values T/K
480.1[a]	0.093 (Ref. 21)	480.1

[a]Upper critical solution temperature.
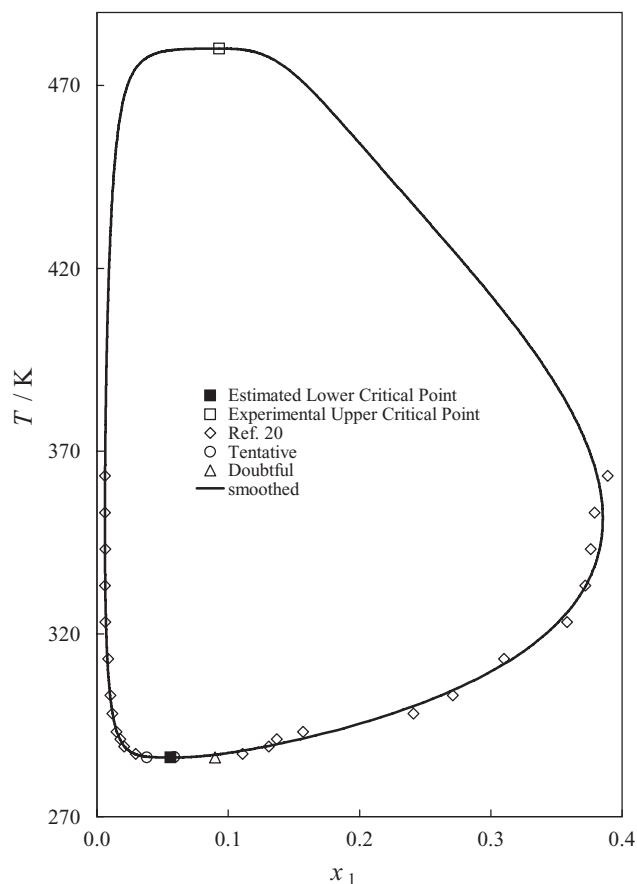
FIG. 4. Mutual solubility data for 2,5-dimethylpyridine (1) + water (2).

TABLE 12. Experimental values for solubility of water (2) in 2,5-dimethylpyridine (1)

T/K	Experimental values x_2 (T = tentative, D = doubtful)	Smoothed values T/K
286.2	0.910 (D; Ref. 20)	287.0
286.3	0.941 (T; Ref. 21)	286.3

T/K	Experimental values x_2	Smoothed values x_2
287.2	0.889 (Ref. 20)	0.905
289.2	0.869 (Ref. 20)	0.872
291.2	0.863 (Ref. 20)	0.846
293.2	0.843 (Ref. 20)	0.823
298.2	0.759 (Ref. 20)	0.776
303.2	0.729 (Ref. 20)	0.738
313.2	0.690 (Ref. 20)	0.684
323.2	0.642 (Ref. 20)	0.649
333.2	0.628 (Ref. 20)	0.628
343.2	0.624 (Ref. 20)	0.617
353.2	0.621 (Ref. 20)	0.615
363.2	0.611 (Ref. 20)	0.619

At the lower critical region, the single point of Andon and Cox[21] at 286.2 K can be compared with data of Stephenson[20] reported for both liquid phases. The points in this region are classified as tentative or doubtful (see Tables 11 and 12). The rest of data are not classified. All the experimental data and smoothed values are listed in Table 12 and shown in Fig. 4.

Components:	Original Measurements:
(1) 2,5-Dimethylpyridine (2,5-Lutidine); C_7H_9N; [589-93-5] (2) Water; H_2O; [7732-18-5]	[21]R. J. L. Andon and J. D. Cox, J. Chem. Soc. **1952**, 4601.

Variables:	Prepared by:
Temperature: 13.1 °C and 206.9 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,5-dimethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
13.1 ± 0.2[a]	27	0.059
206.9 ± 0.5[b]	38	0.093

[a]Lower critical solution temperature.
[b]Upper critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
The titration synthetic method was used. Both components were weighed into thick-wall Pyrex tubes which were then sealed. Phase-transition temperatures were determined in a high-capacity oil bath the temperature of which, measured by means of a set of laboratory standard thermometers graduated in fifths of a degree, could be raised or lowered at any rate down to 0.1 °C per min. For work above 200 °C, an electrically heated metal block was used.

Source and Purity of Materials:
(1) Source not specified; purity 99.85 mol %; the residual impurities were related alkylpyridines and water, hydrocarbons were absent.
(2) Not specified.

Estimated Error:
Temperature: see above.

Components:	Original Measurements:
(1) 2,5-Dimethylpyridine (2,5-Lutidine); C_7H_9N; [589-93-5] (2) Water; H_2O; [7732-18-5]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).

Variables:	Prepared by:
Temperature: 13.0−90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,5-dimethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
13.0	19.05	0.03807
14.0	15.26	0.02939
16.0	11.21	0.02079
18.0	9.68	0.01771
20.0	8.22	0.01484
25.0	6.57	0.01169
30.0	5.79	0.01023
40.0	4.85	8.50×10^{-3}
50.0	3.71	6.44×10^{-3}
60.0	3.53	6.12×10^{-3}
70.0	3.67	6.37×10^{-3}
80.0	3.47	6.01×10^{-3}
90.0	3.55	6.15×10^{-3}

Solubility of water in 2,5-dimethylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
13.0	62.90	0.910
14.0	57.50	0.889
16.0	52.80	0.869
18.0	51.40	0.863
20.0	47.40	0.843
25.0	34.60	0.759
30.0	31.20	0.729
40.0	27.20	0.690
50.0	23.20	0.642
60.0	22.10	0.628
70.0	21.80	0.624
80.0	21.60	0.621
90.0	20.90	0.611

The lower critical solution temperature was reported to be 13 °C.

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 96 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.12\%$ and mass fraction $w_2 = 0.8\%$ (standard deviation).

TABLE 13. Experimental solubility studies for 2,6-dimethylpyridine (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Andon and Cox[21]	307 and 504	(1) in (2)	Krupatkin and Beleva[24]	310	(1) in (2)
Cox and Herington[23]	307–333	Mutual	Loven and Rice[25]	307	Mutual
Flaschner[17]	319–438	Mutual	Stephenson[20]	307–363	Mutual

2.6. 2,6-Dimethylpyridine + water

Components:	Evaluators:
(1) 2,6-Dimethylpyridine (2,6-Lutidine); C_7H_9N; [108-48-5] (2) Water; H_2O; [7732-18-5]	A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland, January, 2012.

2.6.1. Experimental solubility studies for 2,6-dimethylpyridine (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 13 together with the temperature range.

2.6.2. Critical points

The Lower Critical Point was determined from experimental data by the procedure described in Ref. 33, Sec. 1.2; the Upper Critical Point was reported by Andon and Cox,[21]

The lower critical point is: $T_L = 307.2$ K and $x_L = 0.067$,

The upper critical point is: $T_U = 503.9$ K and $x_U = 0.105$,

where x_L and x_U are the critical mole fractions of the amine.

2.6.3. Critical evaluation of the solubility of 2,6-dimethylpyridine (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_1 = z_L \ln x_L + z_U \ln x_U + c_1 \, z_L z_U / T + c_2 \, |z_L z_U|^{1/3} + c_3 \, z_L z_U, \quad (15)$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \quad (16)$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients c_1, c_2, and c_3 obtained from regression of the experimental solubilities are $c_1 = 537.70$, $c_2 = -4.656$, and $c_3 = -0.47$.

The data of Flaschner[17] for both liquid phases differ greatly from other sources and are classified as doubtful. The remaining data outside the critical region were measured by only two

TABLE 14. Experimental values for solubility of 2,6-dimethylpyridine (1) in water (2)

T/K	Experimental values x_1 (R = recommended)	Reference values T/K
307.1	0.0654 (R; Ref. 25), 0.0634 (R; Ref. 25), 0.0643 (R; Ref. 25)	307.2
307.1	0.0572 (R; Ref. 25)	307.2
307.1	0.0591 (R; Ref. 25), 0.0611 (R; Ref. 25), 0.0621 (R; Ref. 25)	307.2
307.1	0.0540 (R; Ref. 25)	307.2
307.1	0.0501 (R; Ref. 25)	307.2
307.2	0.0476 (R; Ref. 20)	307.3
307.2	0.067 (R; Ref. 21)	307.3
307.2	0.0467 (R; Ref. 25)	307.3
307.2	0.0665 (R; Ref. 23)	307.2
307.2	0.0645 (R; Ref. 23), 0.0645 (R; Ref. 23)	307.2
307.2	0.0603 (R; Ref. 23)	307.2
307.3	0.0541 (R; Ref. 23)	307.3
307.3	0.049 (R; Ref. 23), 0.047 (R; Ref. 23)	307.3

T/K	Experimental values x_1 (T = tentative, D = doubtful)	Reference values x_1
307.1	0.067 (Estimated lower critical point)	0.067
307.4	0.044 (T; Ref. 23)	0.0417
307.5	0.0424 (T; Ref. 23)	0.0396
307.6	0.041 (T; Ref. 23)	0.0382
308.2	0.0264 (D; Ref. 20)	0.0308
309.2	0.0241 (T; Ref. 20)	0.0252
310.2	0.0214 (T; Ref. 20), 0.025 (D; Ref. 23)	0.0219
310.3	0.0600 (D; Ref. 24)	0.0217
311.2	0.0181 (T; Ref. 20)	0.0197
312.2	0.0170 (T; Ref. 20)	0.0181
313.2	0.0166 (T; Ref. 20), 0.020 (D; Ref. 23)	0.0168
314.2	0.0148 (T; Ref. 20)	0.0157
316.2	0.0135 (T; Ref. 20)	0.0140
318.2	0.0127 (T; Ref. 20)	0.0128
318.5	0.0591 (D; Ref. 17)	0.0127
321.3	0.0358 (D; Ref. 17)	0.0114
323.2	9.7×10^{-3} (T; Ref. 20), 0.012 (D; Ref. 23)	0.0107
330.9	0.0226 (D; Ref. 17)	8.84×10^{-3}
333.2	7.7×10^{-3} (T; Ref. 20), 0.010 (D; Ref. 23)	8.45×10^{-3}
343.2	6.8×10^{-3} (T; Ref. 20)	7.25×10^{-3}
347.7	0.0173 (D; Ref. 17)	6.88×10^{-3}
353.2	6.4×10^{-3} (T; Ref. 20)	6.52×10^{-3}
363.2	6.2×10^{-3} (T; Ref. 20)	6.05×10^{-3}
378.2	0.0173 (D; Ref. 17)	5.65×10^{-3}
405.9	0.0226 (D; Ref. 17)	5.54×10^{-3}
426.7	0.0358 (D; Ref. 17)	5.93×10^{-3}
437.2	0.0591 (D; Ref. 17)	6.32×10^{-3}

T/K	Experimental values x_1	Smoothed value T/K
503.9[a]	0.105 (Ref. 21)	503.9

[a]Upper critical solution temperature.

independent sources. Thus, following the criteria discussed in Ref. 33, Sec. 1.3, these data are classified as tentative or doubtful. In the critical region, four independent data sources are reported. The points deviating from the reference curve by more than 0.5 K are classified as doubtful. The other points in the critical region are recommended.

All the experimental data and reference values are listed in Table 14 and shown in Fig. 5.
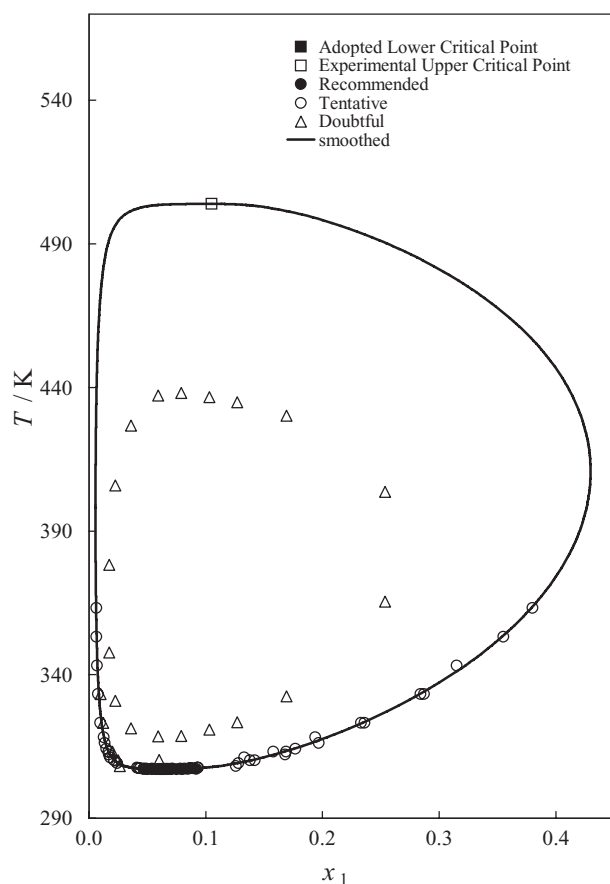
FIG. 5. Mutual solubility data for 2,6-dimethylpyridine (1) + water (2).

TABLE 15. Experimental values for solubility of water (2) in 2,6-dimethylpyridine (1)

T/K	Experimental values x_2 (R = recommended)	Reference values T/K
307.1	0.920 (R; Ref. 25)	307.2
307.1	0.925 (R; Ref. 25)	307.2
307.1	0.929 (R; Ref. 25)	307.2
307.1	0.932 (R; Ref. 25)	307.2
307.2	0.908 (R; Ref. 20)	307.5
307.2	0.915 (R; Ref. 25)	307.3
307.3	0.918 (R; Ref. 23)	307.3
307.3	0.922 (R; Ref. 23)	307.3
307.3	0.928 (R; Ref. 23)	307.2
307.4	0.914 (R; Ref. 23)	307.3

T/K	Experimental values x_2 (T = tentative, D = doubtful)	Reference values x_2
307.4	0.913 (T; Ref. 23), 0.911 (T; Ref. 23)	0.908
307.6	0.907 (T; Ref. 23)	0.903
308.2	0.874 (T; Ref. 20)	0.889
309.2	0.872 (T; Ref. 20)	0.873
310.2	0.862 (T; Ref. 20), 0.858 (T; Ref. 23)	0.861
311.2	0.867 (T; Ref. 20)	0.851
312.2	0.832 (T; Ref. 20)	0.842
313.2	0.842 (T; Ref. 20), 0.831 (T; Ref. 23)	0.833
314.2	0.823 (T; Ref. 20)	0.825
316.2	0.803 (T; Ref. 20)	0.810
318.2	0.806 (T; Ref. 20)	0.796
318.6	0.921 (D; Ref. 17)	0.794
320.9	0.897 (D; Ref. 17)	0.780
323.2	0.767 (T; Ref. 20), 0.764 (T; Ref. 23)	0.766
323.4	0.873 (D; Ref. 17)	0.765
332.5	0.831 (D; Ref. 17)	0.720
333.2	0.716 (T; Ref. 20), 0.713 (T; Ref. 23)	0.717
343.2	0.685 (T; Ref. 20)	0.678
353.2	0.645 (T; Ref. 20)	0.646
363.2	0.620 (T; Ref. 20)	0.621
365.4	0.746 (D; Ref. 17)	0.616
403.7	0.746 (D; Ref. 17)	0.571
430.2	0.831 (D; Ref. 17)	0.579
434.8	0.873 (D; Ref. 17)	0.583
436.6	0.897 (D; Ref. 17)	0.585
438.1	0.921 (D; Ref. 17)	0.587

2.6.4. Critical evaluation of the solubility of water (2) in 2,6-dimethylpyridine (1)

The solubility of (2) in (1), with the exception of outlying data of Flaschner,[17] was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = z_L \ln(1 - x_L) + z_U \ln(1 - x_U) + d_1 \, z_L z_U / T + d_2 \, |z_L z_U|^{1/3} + d_3 \, z_L z_U, \tag{17}$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{18}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients d_1, d_2, and d_3 obtained from regression of the experimental solubilities are $d_1 = 123.90$, $d_2 = -0.254$, and $d_3 = -1.55$.

The solubility outside the critical region was investigated by only two independent sources. Thus, following the criteria discussed in Ref. 33, Sec. 1.3, these data are classified as tentative or doubtful.

All the experimental data and reference values are listed in Table 15 and shown in Fig. 5.

Experimental Values

Solubility of 2,6-dimethylpyridine in water

t/°C	$100\,w_1$	x_1 (compiler)
34.0 ± 0.2[a]	30	0.67
230.7 ± 1[b]	41	0.105

[a]Lower critical solution temperature.
[b]Upper critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
The titration synthetic method was used. Both components were weighed into thick-wall Pyrex tubes which were then sealed. Phase-transition temperatures were determined in a high-capacity oil bath the temperature of which, measured by means of a set of laboratory standard thermometers graduated in fifths of a degree, could be raised or lowered at any rate down to 0.1 °C per min. For work above 200 °C, an electrically heated metal block was used.

Source and Purity of Materials:
(1) Source not specified; purity 99.8 mol %; the residual impurities were related alkylpyridines and water, hydrocarbons were absent.
(2) Not specified.

Estimated Error:
Temperature: see above.

Components:	Original Measurements:
(1) 2,6-Dimethylpyridine (2,6-Lutidine); C_7H_9N; [108-48-5] (2) Water; H_2O; [7732-18-5]	[23]J. D. Cox and E. F. G. Herington, Trans. Faraday Soc. **52**, 926 (1956).

Variables:	Prepared by:
Temperature: 34.6–60.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,6-dimethylpyridine in water

t/°C	$100\,w_1$	x_1
34.06[a,c]	29.1[b]	0.0645
34.075	27.63	0.0603
34.105	25.38	0.0541
34.16[a]	23.5[b]	0.049
34.19	22.69	0.0470
34.26[a]	21.5[b]	0.044
34.40[a]	20.3[b]	0.041
34.34	20.84	0.0424
37.0[a]	13.2[b]	0.025
40.0[a]	10.8[b]	0.020
50.0[a]	6.7[b]	0.012
60.0[a]	5.7[b]	0.010

Solubility of water in 2,6-dimethylpyridine

t/°C	$100\,w_2$	x_2
34.06[a,c]	70.9[b]	0.9355
34.06	70.25	0.9335
34.095	68.51	0.9283
34.12	66.41	0.9216
34.16[a]	65.3[b]	0.918
34.23	64.13	0.9140
34.26[a]	63.8[b]	0.913
34.28	63.22	0.9109
34.40[a]	62.1[b]	0.907
34.41	61.98	0.9065
37.0[a]	50.4[b]	0.858
40.0[a]	55.3[b]	0.831
50.0[a]	35.2[b]	0.764
60.0[a]	29.5[b]	0.713

[a]Author's graphically interpolated data.
[b]Calculated by compiler.
[c]Lower critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
The cloud-point method[21] was used. A temperature bath could be raised at the rate of 0.005 °C/min and cloud points could be thereby be reproduced to within ±0.01 °C or better. The thermometer error was determined as (0.00 ± 0.02) °C.

Source and Purity of Materials:
(1) Source not specified; purity by freezing-point technique (99.89 ± 0.06) mol %; virtually all of the impurity was shown by infrared spectroscopy to be water.
(2) Not specified.

Estimated Error:
Temperature: ±0.02 °C.

Components:	Original Measurements:
(1) 2,6-Dimethylpyridine (2,6-Lutidine); C_7H_9N; [108-48-5] (2) Water; H_2O; [7732-18-5]	[17]O. Flaschner, J. Chem. Soc. **95**, 668 (1909).

Variables:	Prepared by:
Temperature: 45.4–164.9 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,6-dimethylpyridine in water

t/°C	$100\,w_1$	x_1 (compiler)
45.4[a]	33.8	0.0791
45.3	27.2	0.0591
48.1	18.1	0.0358
57.7	12.1	0.0226
74.5	9.5	0.0173
105.0	9.5	0.0173
132.7	12.1	0.0226
153.5	18.1	0.0358
164.0	27.2	0.0591
164.9[a]	33.8	0.0791

Solubility of water in 2,6-dimethylpyridine

t/°C	$100\,w_2$	x_2 (compiler)
47.7	59.4	0.8969
50.2	53.6	0.8730
59.3	45.2	0.8307
92.2	33.1	0.7464
130.5	33.1	0.7464
157.0	45.2	0.8307
161.6	53.6	0.8730
163.4[a]	59.4	0.8969

[a]Critical opalescence.

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. The operation was carried out in Jena-glass tubes. No more details were reported in the paper.

Source and Purity of Materials:
(1) Source not specified; crystallized as zinc salt; isolated, and distilled; b.p. 143.2 °C.
(2) Not specified.

Estimated Error:
Not specified.

Components:	Original Measurements:
(1) 2,6-Dimethylpyridine (2,6-Lutidine); C_7H_9N; [108-48-5]	[24]I. L. Krupatkin and N. N. Beleva, Zh. Fiz. Khim. **54**, 1701 (1980).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
One temperature: 37.1 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,6-dimethylpyridine in water

$t/°C$	$100 w_1$	x_1 (compiler)
37.1[a]	27.5	0.0600

[a]Lower critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
The titration method was used. No more details were reported in the paper. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Source not specified; purified; b.p. 142 °C, $d_4^{20} = 0.920$.
(2) Distilled.

Estimated Error:
Temperature: ±0.05 °C.

Components:	Original Measurements:
(1) 2,6-Dimethylpyridine (2,6-Lutidine); C_7H_9N; [108-48-5]	[25]A. W. Loven and O. K. Rice, Trans. Faraday Soc. **59**, 2723 (1963).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
Temperature: 33.927–34.053 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,6-dimethylpyridine in water

$t/°C$	$100 w_1$ (compiler)	x_1
33.927	28.25	0.06209
33.929	27.89	0.06106
33.931	27.20	0.05913

33.935	26.50	0.05718
33.947	25.35	0.05403
33.979	23.86	0.05007
34.039	22.57	0.04674

Solubility of water in 2,6-dimethylpyridine

$t/°C$	$100 w_2$ (compiler)	x_2
33.927	70.99	0.9357
33.928	71.30	0.9366
33.929	70.61	0.9346
33.930	69.82	0.9322
33.934	68.90	0.9295
33.952	67.44	0.9249
33.984	65.98	0.9202
34.053	64.47	0.9152

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. Mixtures prepared by volume were placed in the thermostat a few thousandths of a degree below the expected separation temperatures and thoroughly stirred with the glass-covered magnetic stirrer. As the temperature of separation of a solution in the critical region was attained, the solution suddenly became very foggy. The temperature of phase separation was measured with the resistance thermometer and resistance bridge.

Source and Purity of Materials:
(1) Eastman Kodak Co.; vacuum distilled once and then purified by preparative gas chromatography.
(2) Distilled from a mixture of $KMnO_4$ and H_3PO_4 in a stream of N_2.

Estimated Error:
Temperature: ±0.001 °C (temperature differences).

Components:	Original Measurements:
(1) 2,6-Dimethylpyridine (2,6-Lutidine); C_7H_9N; [108-48-5]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
Temperature: 34.0−90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,6-dimethylpyridine in water

$t/°C$	$100 w_1$	x_1 (compiler)
34.0	22.9	0.0476
35.0	13.9	0.0264
36.0	12.8	0.0241
37.0	11.5	0.0214
38.0	9.9	0.0181
39.0	9.3	0.0170
40.0	9.1	0.0166
41.0	8.2	0.0148
43.0	7.5	0.0135
45.0	7.1	0.0127
50.0	5.5	9.7×10^{-3}
60.0	4.4	7.7×10^{-3}
70.0	3.9	6.8×10^{-3}
80.0	3.7	6.4×10^{-3}
90.0	3.6	6.2×10^{-3}

Solubility of water in 2,6-dimethylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
34.0	62.3	0.908
35.0	53.9	0.874
36.0	53.4	0.872
37.0	51.3	0.862
38.0	52.4	0.867
39.0	45.5	0.832
40.0	47.3	0.842
41.0	43.8	0.823
43.0	40.7	0.803
45.0	41.1	0.806
50.0	35.6	0.767
60.0	29.8	0.716
70.0	26.8	0.685
80.0	23.4	0.645
90.0	21.5	0.620

The lower critical solution temperature was reported to be 34 °C.

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99+ mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.2\%$ and mass fraction $w_2 = 0.9\%$ (standard deviation).

2.7. 3,4-Dimethylpyridine + water

Components:	Evaluators:
(1) 3,4-Dimethylpyridine (3,4-Lutidine); C_7H_9N; [583-58-4]	A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of
(2) Water; H_2O; [7732-18-5]	Sciences, Warsaw, Poland, January, 2012.

2.7.1. Experimental solubility studies for 3,4-dimethylpyridine (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 16 together with the temperature range.

TABLE 16. Experimental solubility studies for 3,4-dimethylpyridine (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Cox[19]	270 and 436	(1) in (2)	Stephenson[20]	273–363	Mutual

2.7.2. Critical points

The Lower Critical Point was determined from experimental data by the procedure described in Ref. 33, Sec. 1.2; the Upper Critical Point was reported by Cox,[19]

The lower critical point is: $T_L = 269.6$ K and $x_L = 0.048$,

The upper critical point is: $T_U = 435.7$ K and $x_U = 0.086$,

where x_L and x_U are the critical mole fractions of the amine.

2.7.3. Critical evaluation of the solubility of 3,4-dimethylpyridine (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_1 = z_L \ln x_L + z_U \ln x_U + c_1\, z_L z_U/T + c_2\, |z_L z_U|^{1/3} + c_3\, z_L z_U, \qquad (19)$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \qquad (20)$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients $c_1, c_2,$ and c_3 obtained from regression of the experimental solubilities are $c_1 = 121.70$, $c_2 = -4.038$, and $c_3 = 1.03$.

The solubility of (1) in (2) reported by Stephenson[20] and the Upper Critical Point reported by Cox[19] are not classified. All the experimental data and smoothed values are listed in Table 17 and shown in Fig. 6.

TABLE 17. Experimental values for solubility of 3,4-dimethylpyridine (1) in water (2)

T/K	Experimental values x_1	Smoothed values x_1
269.6	0.0480 (Estimated lower critical point)	0.0480
273.2	0.0151 (Ref. 20)	0.0165
283.2	0.0110 (Ref. 20)	0.0102
293.2	8.8×10^{-3} (Ref. 20)	8.4×10^{-3}
303.2	7.4×10^{-3} (Ref. 20)	7.6×10^{-3}
313.2	7.0×10^{-3} (Ref. 20)	7.1×10^{-3}
323.2	6.7×10^{-3} (Ref. 20)	6.9×10^{-3}
333.2	6.8×10^{-3} (Ref. 20)	6.9×10^{-3}
343.2	6.9×10^{-3} (Ref. 20)	6.9×10^{-3}
353.2	7.2×10^{-3} (Ref. 20)	7.1×10^{-3}
363.2	7.6×10^{-3} (Ref. 20)	7.4×10^{-3}

T/K	Experimental values x_1	Smoothed values T/K
435.7[a]	0.0860 (Ref. 19)	435.7

[a]Upper critical solution temperature.
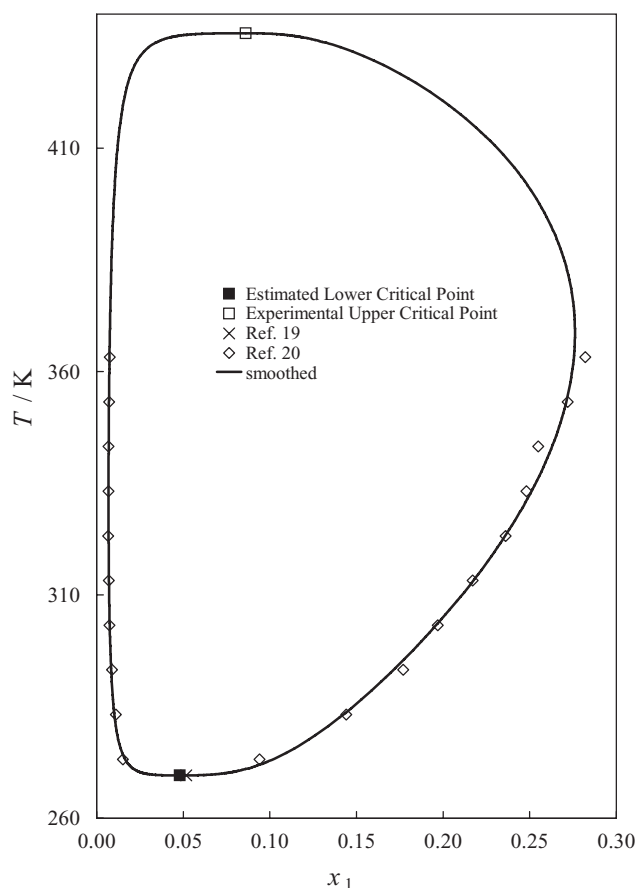
FIG. 6. Mutual solubility data for 3,4-dimethylpyridine (1) + water (2).

TABLE 18. Experimental values for solubility of water (2) in 3,4-dimethylpyridine (1)

T/K	Experimental values x_2	Smoothed values T/K
269.6	0.948 (Ref. 19)	269.7

T/K	Experimental values x_2	Smoothed values x_2
273.2	0.906 (Ref. 20)	0.898
283.2	0.856 (Ref. 20)	0.858
293.2	0.823 (Ref. 20)	0.829
303.2	0.803 (Ref. 20)	0.804
313.2	0.783 (Ref. 20)	0.783
323.2	0.764 (Ref. 20)	0.764
333.2	0.752 (Ref. 20)	0.749
343.2	0.745 (Ref. 20)	0.737
353.2	0.728 (Ref. 20)	0.729
363.2	0.718 (Ref. 20)	0.725

Experimental Values

Solubility of 3,4-dimethylpyridine in water

t/°C	$100\,w_1$	x_1 (compiler)
−3.6[a]	24.5	0.0517
162.5[b]	36	0.086

[a]Lower critical solution temperature.
[b]Upper critical solution temperature.

2.7.4. Critical evaluation of the solubility of water (2) in 3,4-dimethylpyridine (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = z_L \ln(1 - x_L) + z_U \ln(1 - x_U) + d_1\, z_L z_U/T + d_2\, |z_L z_U|^{1/3} + d_3\, z_L z_U, \qquad (21)$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \qquad (22)$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients d_1, d_2, and d_3 obtained from regression of the experimental solubilities are $d_1 = 325.67$, $d_2 = -0.189$, and $d_3 = -1.43$.

The data of Cox[19] and Stephenson[20] were measured over different ranges of temperature and, therefore, the data are not classified. All the experimental data and smoothed values are listed in Table 18 and shown in Fig. 6.

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. Solution temperatures above room temperature were determined with the aid of an electrically heated metal block; a sealed tube containing a known mixture of (1) and (2) was fixed in a cavity in the middle of the block and could be observed through the windows. The liquid layers were mixed by rotating the block. For solution temperatures below room temperature, the tube containing the mixture was fixed in a bucket which was then lowered into an unsilvered Dewar vessel filled with methanol and shaken manually. The temperature of the bath was lowered by addition of solid carbon dioxide and then allowed to rise at a rate of 0.1 °C per min until the cloud point was reached.

Source and Purity of Materials:
(1) Prepared by synthesis; distilled; b.p. 111 °C (98 mmHg); appeared from the infrared spectrum to be high purity.
(2) Not specified.

Estimated Error:
Temperature: ±0.3 °C.
Solubility: mass fraction $w_1 = \pm 1\%$ (standard deviation).

Components:
(1) 3,4-Dimethylpyridine (3,4-Lutidine); C_7H_9N; [583-58-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[19]J. D. Cox, J. Chem. Soc. **1954**, 3183.

Variables:
Temperature: (−3.6) and 162.5 °C

Prepared by:
B. Wisniewska-Goclowska

Components:
(1) 3,4-Dimethylpyridine (3,4-Lutidine); C_7H_9N; [583-58-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).

Variables:
Temperature: 0−90.0 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 3,4-dimethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	8.36	0.0151
10.0	6.18	0.0110
20.0	5.04	8.8×10^{-3}
30.0	4.25	7.4×10^{-3}
40.0	4.02	7.0×10^{-3}
50.0	3.84	6.7×10^{-3}
60.0	3.91	6.8×10^{-3}
70.0	3.94	6.9×10^{-3}
80.0	4.11	7.2×10^{-3}
90.0	4.38	7.6×10^{-3}

Solubility of water in 3,4-dimethylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	61.8	0.906
10.0	50.0	0.856
20.0	43.8	0.823
30.0	40.7	0.803
40.0	37.8	0.783
50.0	35.2	0.764
60.0	33.8	0.752
70.0	32.9	0.745
80.0	31.0	0.728
90.0	30.0	0.718

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99.3 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.08\%$ and mass fraction $w_2 = 0.1\%$ (standard deviation).

2.8. 3,5-Dimethylpyridine + water

Components:	**Evaluators:**
(1) 3,5-Dimethylpyridine (3,5-Lutidine); C_7H_9N; [591-22-0]	A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of
(2) Water; H_2O; [7732-18-5]	Sciences, Warsaw, Poland, January, 2012.

TABLE 19. Experimental solubility studies for 3,5-dimethylpyridine (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Cox[19]	261 and 465	(1) in (2)	Stephenson[20]	273–363	Mutual

2.8.1. Experimental solubility studies for 3,5-dimethylpyridine (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 19 together with the temperature range.

2.8.2. Critical points

The Lower Critical Point was determined from experimental data by the procedure described in Ref. 33, Sec. 1.2; the Upper Critical Point was reported by Cox,[19]

The lower critical point is: $T_L = 265.0$ K and $x_L = 0.028$,

The upper critical point is: $T_U = 465.2$ K and $x_U = 0.090$,

where x_L and x_U are the critical mole fractions of the amine.

2.8.3. Critical evaluation of the solubility of 3,5-dimethylpyridine (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_1 = z_L \ln x_L + z_U \ln x_U + c_1\,z_L z_U/T + c_2\,|z_L z_U|^{1/3} + c_3\,z_L z_U, \qquad (23)$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \qquad (24)$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients c_1, c_2, and c_3 obtained from regression of the experimental solubilities are $c_1 = 910.80$, $c_2 = -4.086$, and $c_3 = -2.20$.

The solubility of (1) in (2) reported by Stephenson[20] and the Upper Critical Point reported by Cox[19] are not classified. All the experimental data and smoothed values are listed in Table 20 and shown in Fig. 7.

2.8.4. Critical evaluation of the solubility of water (2) in 3,5-dimethylpyridine (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = z_L \ln(1 - x_L) + z_U \ln(1 - x_U) + d_1\,z_L z_U/T + d_2\,|z_L z_U|^{1/3} + d_3\,z_L z_U, \qquad (25)$$

TABLE 20. Experimental values for solubility of 3,5-dimethylpyridine (1) in water (2)

T/K	Experimental values x_1	Smoothed values x_1
265	0.028 (Estimated lower critical point)	0.028
273.2	7.3×10^{-3} (Ref. 20)	7.7×10^{-3}
283.2	6.1×10^{-3} (Ref. 20)	5.7×10^{-3}
293.2	5.0×10^{-3} (Ref. 20)	4.9×10^{-3}
303.2	4.3×10^{-3} (Ref. 20)	4.4×10^{-3}
313.2	4.1×10^{-3} (Ref. 20)	4.2×10^{-3}
323.2	3.9×10^{-3} (Ref. 20)	4.0×10^{-3}
333.2	3.9×10^{-3} (Ref. 20)	3.9×10^{-3}
343.2	4.1×10^{-3} (Ref. 20)	3.9×10^{-3}
353.2	3.9×10^{-3} (Ref. 20)	4.0×10^{-3}
363.2	4.1×10^{-3} (Ref. 20)	4.1×10^{-3}

T/K	Experimental values x_1	Smoothed values T/K
465.2[a]	0.090 (Ref. 19)	465.2

[a]Upper critical solution temperature.

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \quad (26)$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients d_1, d_2, and d_3 obtained from regression of the experimental solubilities are $d_1 = -167.53$, $d_2 = -0.132$, and $d_3 = -0.59$.
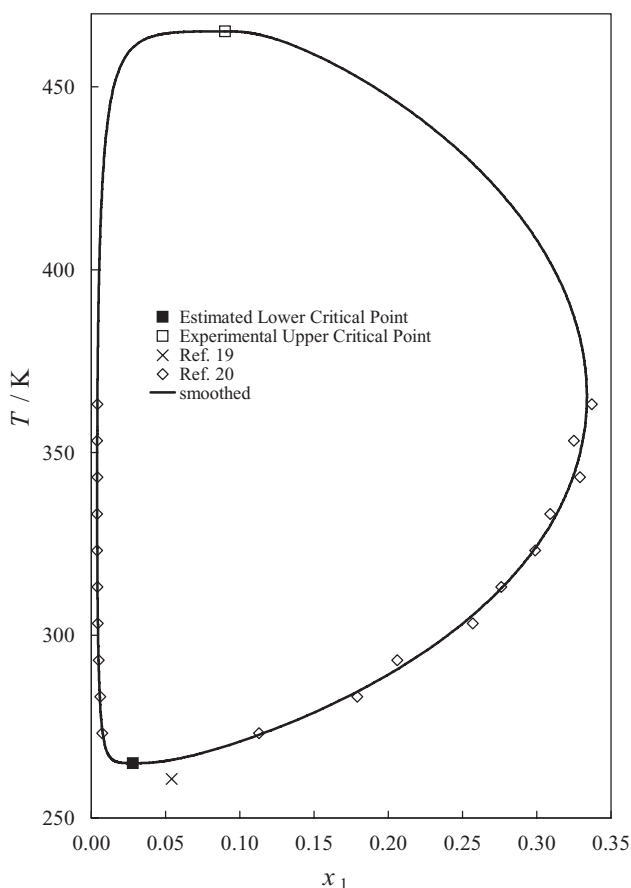
TABLE 21. Experimental values for solubility of water (2) in 3,5-dimethylpyridine (1)

T/K	Experimental values x_2	Smoothed values T/K
260.7	0.946 (Ref. 19)	265.9

T/K	Experimental values x_2	Smoothed values x_2
273.2	0.887 (Ref. 20)	0.884
283.2	0.821 (Ref. 20)	0.828
293.2	0.794 (Ref. 20)	0.784
303.2	0.743 (Ref. 20)	0.750
313.2	0.724 (Ref. 20)	0.723
323.2	0.701 (Ref. 20)	0.703
333.2	0.691 (Ref. 20)	0.687
343.2	0.671 (Ref. 20)	0.676
353.2	0.675 (Ref. 20)	0.669
363.2	0.663 (Ref. 20)	0.666

The solubility of (2) in (1) reported by Stephenson[20] and the Lower Critical Point reported by Cox[19] are not classified. All the experimental data and smoothed values are listed in Table 21 and shown in Fig. 7.

Components:	Original Measurements:
(1) 3,5-Dimethylpyridine (3,5-Lutidine); C$_7$H$_9$N; [591-22-0]	[19]J. D. Cox, J. Chem. Soc. **1954**, 3183.
(2) Water; H$_2$O; [7732-18-5]	

Variables:	Prepared by:
Temperature: -12.5 °C and 192.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 3,5-dimethylpyridine in water

t/°C	$100\,w_1$	x_1 (compiler)
-12.5[a]	25.5	0.0544
192.0[b]	37	0.090

[a]Lower critical solution temperature.
[b]Upper critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. Solution temperatures above room temperature were determined with the aid of an electrically heated metal block; a sealed tube containing a known mixture of (1) and (2) was fixed in a cavity in the middle of the block and could be observed through the windows. The liquid layers were mixed by rotating the block. For solution temperatures below room temperature, the tube containing the specimen was fixed in a bucket which was then lowered into an unsilvered Dewar vessel filled with methanol and shaken manually. The temperature of the bath was lowered by addition of solid carbon dioxide and then allowed to rise at a rate of 0.1 °C per min until the cloud point was reached.

Source and Purity of Materials:
(1) Source not specified; purified by fractional distillation and fractional freezing; b.p. range 172.2−172.8 °C (762 mmHg); purity by freezing point technique 99.2 mol %.
(2) Not specified.

Estimated Error:
Temperature: ± 0.4 °C.
Solubility: mass fraction $w_1 = \pm 1\%$ (standard deviation).

FIG. 7. Mutual solubility data for 3,5-dimethylpyridine (1) + water (2).

Components:
(1) 3,5-Dimethylpyridine
(3,5-Lutidine); C_7H_9N; [591-22-0]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).

Variables:
Temperature: 0−90.0 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 3,5-dimethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	4.2	7.3×10^{-3}
10.0	3.5	6.1×10^{-3}
20.0	2.9	5.0×10^{-3}
30.0	2.5	4.3×10^{-3}
40.0	2.4	4.1×10^{-3}
50.0	2.3	3.9×10^{-3}
60.0	2.3	3.9×10^{-3}
70.0	2.4	4.1×10^{-3}
80.0	2.3	3.9×10^{-3}
90.0	2.4	4.1×10^{-3}

Solubility of water in 3,5-dimethylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	56.8	0.887
10.0	43.5	0.821
20.0	39.3	0.794
30.0	32.7	0.743
40.0	30.6	0.724
50.0	28.3	0.701
60.0	27.3	0.691
70.0	25.5	0.671
80.0	25.9	0.675
90.0	24.9	0.663

The lower critical solution temperature was reported to be −8 °C.

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99+ mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.08\%$ and mass fraction $w_2 = 0.1\%$ (standard deviation).

TABLE 22. Experimental solubility studies for 2-ethylpyridine (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Cox[19]	268 and 505	(1) in (2)	Stephenson[20]	273–363	Mutual

2.9. 2-Ethylpyridine + water

Components:
(1) 2-Ethylpyridine;
C_7H_9N; [100-71-0]
(2) Water; H_2O; [7732-18-5]

Evaluators:
A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland, January, 2012.

2.9.1. Experimental solubility studies for 2-ethylpyridine (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 22 together with the temperature range.

2.9.2. Critical points

The Lower Critical Point was determined from experimental data by the procedure described in Ref. 33, Sec. 1.2; the Upper Critical Point was reported by Cox,[19]

The lower critical point is: $T_L = 268.2$ K and $x_L = 0.070$,

The upper critical point is: $T_U = 504.6$ K and $x_U = 0.109$,

where x_L and x_U are the critical mole fractions of the amine.

2.9.3. Critical evaluation of the solubility of 2-ethylpyridine (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_1 = z_L \ln x_L + z_U \ln x_U + c_1\, z_L z_U / T + c_2\, |z_L z_U|^{1/3} + c_3\, z_L z_U, \qquad (27)$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \qquad (28)$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients c_1, c_2, and c_3 obtained from regression of the experimental solubilities are $c_1 = -1479.04$, $c_2 = -4.871$, and $c_3 = 4.34$.

The solubility of (1) in (2) reported by Stephenson[20] and the Upper Critical Point reported by Cox[19] are not classified. All the experimental data and smoothed values are listed in Table 23 and shown in Fig. 8.

TABLE 23. Experimental values for solubility of 2-ethylpyridine (1) in water (2)

T/K	Experimental values x_1	Smoothed values x_1
268.2	0.07 (Estimated critical point)	0.07
273.2	0.0179 (Ref. 20)	0.0182
283.2	0.0101 (Ref. 20)	0.0102
293.2	7.59×10^{-3} (Ref. 20)	7.47×10^{-3}
303.2	6.24×10^{-3} (Ref. 20)	6.07×10^{-3}
313.2	5.48×10^{-3} (Ref. 20)	5.27×10^{-3}
323.2	4.57×10^{-3} (Ref. 20)	4.79×10^{-3}
333.2	4.40×10^{-3} (Ref. 20)	4.50×10^{-3}
343.2	4.26×10^{-3} (Ref. 20)	4.35×10^{-3}
353.2	4.31×10^{-3} (Ref. 20)	4.29×10^{-3}
363.2	4.43×10^{-3} (Ref. 20)	4.31×10^{-3}

T/K	Experimental values x_1	Smoothed values T/K
504.6[a]	0.891 (Ref. 19)	504.6

[a]Upper critical solution temperature.

2.9.4. Critical evaluation of the solubility of water (2) in 2-ethylpyridine (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = z_L \ln (1 - x_L) + z_U \ln (1 - x_U) + d_1\, z_L z_U / T + d_2\, |z_L z_U|^{1/3} + d_3\, z_L z_U, \qquad (29)$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \qquad (30)$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients d_1, d_2, and d_3 obtained from regression of the experimental solubilities are $d_1 = -427.50$, $d_2 = -0.279$, and $d_3 = -0.73$.

The data of Cox[19] at 268 K and the data of Stephenson[20] at 273–363 K were reported over different ranges of temperature and, therefore, are not classified. All the experimental data and smoothed values are listed in Table 24 and shown in Fig. 8.

Components:	Original Measurements:
(1) 2-Ethylpyridine; C_7H_9N; [100-71-0]	[19]J. D. Cox, J. Chem. Soc. **1954**, 3183.
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
Temperature: -5 °C and 231.4 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2-ethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
-5[a]	34	0.080
231.4[b]	42	0.109

[a]Lower critical solution temperature.
[b]Upper critical solution temperature.
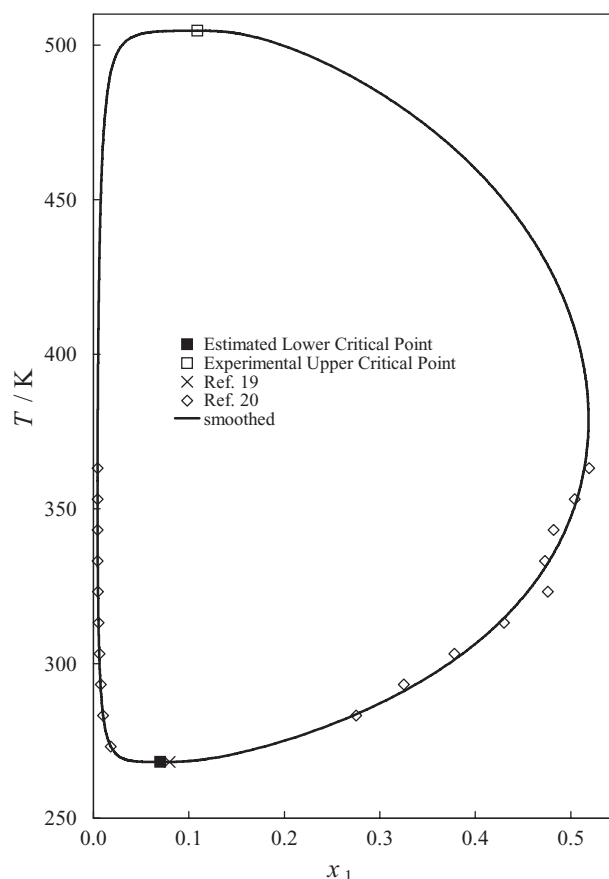
FIG. 8. Mutual solubility data for 2-ethylpyridine (1) + water (2).

TABLE 24. Experimental values for solubility of water (2) in 2-ethylpyridine (1)

T/K	Experimental values x_2	Smoothed values T/K
268.2	0.920 (Ref. 19)	268.3

T/K	Experimental values x_2	Smoothed values x_2
273.2	0.816 (Ref. 20)	0.821
283.2	0.725 (Ref. 20)	0.728
293.2	0.675 (Ref. 20)	0.663
303.2	0.622 (Ref. 20)	0.614
313.2	0.570 (Ref. 20)	0.575
323.2	0.524 (Ref. 20)	0.546
333.2	0.527 (Ref. 20)	0.523
343.2	0.518 (Ref. 20)	0.506
353.2	0.496 (Ref. 20)	0.494
363.2	0.481 (Ref. 20)	0.486

Auxiliary Information

Method/Apparatus/Procedure:

The synthetic method was used. Solution temperatures above room temperature were determined with the aid of an electrically heated metal block; the sealed tube containing a known mixture of (1) and (2) was fixed in a cavity in the middle of the block and could be observed through the windows. The liquid layers were mixed by rotating the block. For solution temperatures below room temperature, the tube containing the mixture was fixed in a bucket which was then lowered into an unsilvered Dewar vessel filled with methanol and shaken manually. The temperature of the bath was lowered by addition of solid carbon dioxide and then allowed to rise at a rate of 0.1 °C per min until the cloud point was reached.

Source and Purity of Materials:

(1) Prepared by synthesis; distilled; b.p. range 144.5–144.8 °C (700 mmHg); no impurity apart from water by infrared spectroscopy.
(2) Not specified.

Estimated Error:

Temperature: ± 0.3 at -5 °C and ± 1 at 231.4 °C.
Solubility: mass fraction $w_1 = \pm 1\%$ (standard deviation).

Components:	Original Measurements:
(1) 2-Ethylpyridine; C_7H_9N; [100-71-0]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
Temperature: 0−90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2-ethylpyridine in water

t/°C	$100\,w_1$	x_1 (compiler)
0.0	9.80	0.01794
10.0	5.72	0.01010
20.0	4.35	7.59×10^{-3}
30.0	3.60	6.24×10^{-3}
40.0	3.17	5.48×10^{-3}
50.0	2.66	4.57×10^{-3}
60.0	2.56	4.40×10^{-3}
70.0	2.48	4.26×10^{-3}
80.0	2.51	4.31×10^{-3}
90.0	2.58	4.43×10^{-3}

Solubility of water in 2-ethylpyridine

t/°C	$100\,w_2$	x_2 (compiler)
0.0	42.8	0.816
10.0	30.7	0.725
20.0	25.9	0.675
30.0	21.7	0.622
40.0	18.2	0.570
50.0	15.6	0.524
60.0	15.8	0.527
70.0	15.3	0.518
80.0	14.2	0.496
90.0	13.5	0.481

TABLE 25. Experimental solubility studies for 3-ethylpyridine (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Cox[19]	469	(1) in (2)	Stephenson[20]	273–363	Mutual

Auxiliary Information

Method/Apparatus/Procedure:

The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:

(1) Aldrich Chemical Co. or TCI America; purity 99.9 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:

Temperature: ± 0.1 °C.
Solubility: mass fraction $w_1 = 0.04\%$ and mass fraction $w_2 = 0.3\%$ (standard deviation).

2.10. 3-Ethylpyridine + water

Components:	Evaluators:
(1) 3-Ethylpyridine; C_7H_9N; [536-78-7]	A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland, January, 2012.
(2) Water; H_2O; [7732-18-5]	

2.10.1. Experimental solubility studies for 3-ethylpyridine (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 25 together with the temperature range.

2.10.2. Critical point

The Lower Critical Point was determined from experimental data by the procedure described in Ref. 33, Sec. 1.2; the Upper Critical Point was reported by Cox,[19]

The lower critical point is: $T_L = 261.2$ K and $x_L = 0.068$,

The upper critical point is: $T_U = 468.8$ K and $x_U = 0.090$,

where x_L and x_U are the critical mole fractions of the amine.

TABLE 26. Experimental values for solubility of 3-ethylpyridine (1) in water (2)

T/K	Experimental values x_1	Smoothed values x_1
273.2	7.08×10^{-3} (Ref. 20)	7.21×10^{-3}
283.2	5.48×10^{-3} (Ref. 20)	5.40×10^{-3}
293.2	4.72×10^{-3} (Ref. 20)	4.68×10^{-3}
303.2	4.26×10^{-3} (Ref. 20)	4.33×10^{-3}
313.2	4.21×10^{-3} (Ref. 20)	4.16×10^{-3}
323.2	4.19×10^{-3} (Ref. 20)	4.08×10^{-3}
333.2	3.91×10^{-3} (Ref. 20)	4.04×10^{-3}
343.2	4.01×10^{-3} (Ref. 20)	4.04×10^{-3}
353.2	3.96×10^{-3} (Ref. 20)	4.06×10^{-3}
363.2	4.22×10^{-3} (Ref. 20)	4.08×10^{-3}
T/K	Experimental values x_1	Smoothed values T/K
468.8^a	0.0899 (Ref. 19)	468.8

^{a}Upper critical solution temperature.

2.10.3. Critical evaluation of the solubility of 3-ethylpyridine (1) in water (2)

The solubility of (1) in (2) reported by Stephenson[20] was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_1 = z_L \ln x_L + z_U \ln x_U + c_1 \, z_L z_U / T \\ + c_2 \, |z_L z_U|^{1/3} + c_3 \, z_L z_U, \tag{31}$$

where z_L, z_U are temperature-dependent fractions,

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{32}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients c_1, c_2, and c_3 obtained from regression of the experimental solubilities are $c_1 = 277.84$, $c_2 = -6.756$, and $c_3 = 4.46$.

The solubility of (1) in (2) reported by Stephenson[20] and the Upper Critical Point reported by Cox[19] are not classified. All the experimental data and smoothed values are listed in Table 26 and shown in Fig. 9.

2.10.4. Critical evaluation of the solubility of water (2) in 3-ethylpyridine (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = z_L \ln (1 - x_L) + z_U \ln (1 - x_U) + d_1 \, z_L z_U / T \\ + d_2 \, |z_L z_U|^{1/3} + d_3 \, z_L z_U, \tag{33}$$

where z_L, z_U are temperature-dependent fractions,

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{34}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients d_1, d_2, and d_3 obtained from regression of the experimental solubilities are $d_1 = 352.33$, $d_2 = -0.712$, and $d_3 = -1.29$.

The solubility of (2) in (1) below the Upper Critical Temperature was reported by Stephenson[20] only and is not
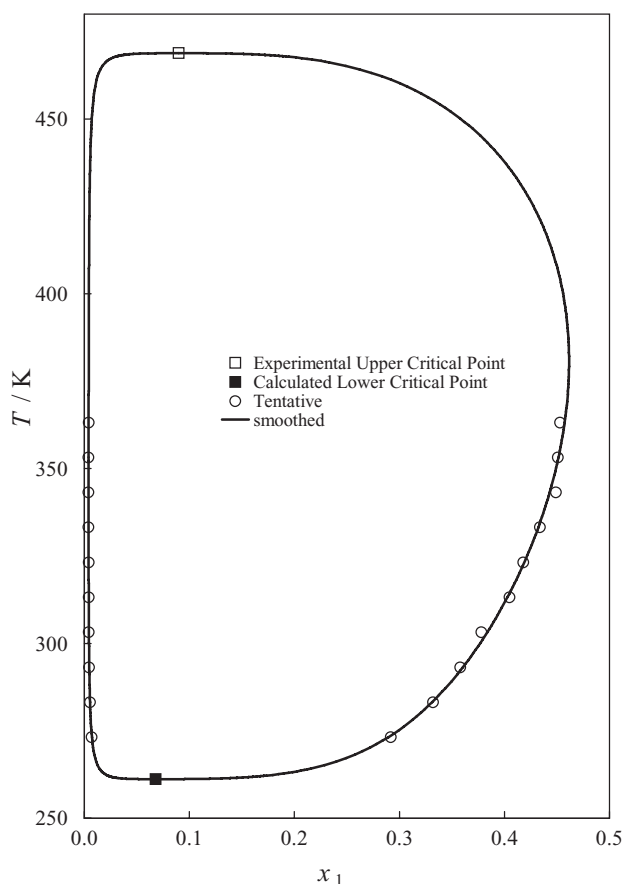
FIG. 9. Mutual solubility data for 3-ethylpyridine (1) + water (2).

classified. All the experimental data and smoothed values are listed in Table 27 and shown in Fig. 9.

Components:	Original Measurements:
(1) 3-Ethylpyridine;	[19]J. D. Cox, J. Chem. Soc. **1954**, 3183.
C$_7$H$_9$N; [536-78-7]	
(2) Water; H$_2$O; [7732-18-5]	
Variables:	Prepared by:
Temperature: -35 °C and 195.6 °C	B. Wisniewska-Goclowska

TABLE 27. Experimental values for solubility of water (2) in 3-ethylpyridine (1)

T/K	Experimental values x_2	Smoothed values x_2
273.2	0.708 (Ref. 20)	0.711
283.2	0.668 (Ref. 20)	0.669
293.2	0.642 (Ref. 20)	0.640
303.2	0.622 (Ref. 20)	0.616
313.2	0.595 (Ref. 20)	0.597
323.2	0.582 (Ref. 20)	0.581
333.2	0.566 (Ref. 20)	0.568
343.2	0.551 (Ref. 20)	0.557
353.2	0.549 (Ref. 20)	0.549
363.2	0.547 (Ref. 20)	0.543

Experimental Values

Solubility of 3-ethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
195.6[a]	37	0.090

[a]Upper critical solution temperature.

The lower critical solution temperature was reported to be −35 °C.

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. Solution temperatures above room temperature were determined with the aid of an electrically heated metal block; the sealed tube containing a known mixture of (1) and (2) was fixed in a cavity in the middle of the block and could be observed through the windows. The liquid layers were mixed by rotating the block. For solution temperatures below room temperature, the tube containing the mixture was fixed in a bucket which was then lowered into an unsilvered Dewar vessel filled with methanol and shaken manually. The temperature of the bath was lowered by addition of solid carbon dioxide and then allowed to rise at a rate of 0.1 °C per min until the cloud point was reached.

Source and Purity of Materials:
(1) Prepared by synthesis; distilled; b.p. 166 °C.
(2) Not specified.

Estimated Error:
Temperature: ±3 at −35 °C and ±0.5 at 195.6 °C.
Solubility: mass fraction $w_1 = ±1\%$ (standard deviation).

Components:	**Original Measurements:**
(1) 3-Ethylpyridine; C_7H_9N; [536-78-7]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).
(2) Water; H_2O; [7732-18-5]	

Variables:	**Prepared by:**
Temperature: 0−90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 3-ethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	4.07	7.08×10^{-3}
10.0	3.17	5.48×10^{-3}
20.0	2.74	4.72×10^{-3}
30.0	2.48	4.26×10^{-3}
40.0	2.45	4.21×10^{-3}
50.0	2.44	4.19×10^{-3}
60.0	2.28	3.91×10^{-3}
70.0	2.34	4.01×10^{-3}
80.0	2.31	3.96×10^{-3}
90.0	2.46	4.22×10^{-3}

Solubility of water in 3-ethylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	29.0	0.708
10.0	25.3	0.668
20.0	23.2	0.642
30.0	21.7	0.622
40.0	19.8	0.595
50.0	19.0	0.582
60.0	18.0	0.566
70.0	17.1	0.551
80.0	17.0	0.549
90.0	16.9	0.547

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 97 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.06\%$ and mass fraction $w_2 = 0.2\%$ (standard deviation).

2.11. 4-Ethylpyridine + water

Components:	**Evaluators:**
(1) 4-Ethylpyridine; C_7H_9N; [536-75-4]	A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland, January, 2012.
(2) Water; H_2O; [7732-18-5]	

2.11.1. Experimental solubility studies for 4-ethylpyridine (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 28 together with the temperature range.

2.11.2. Critical points

The Lower Critical Point was determined from experimental data by the procedure described in Ref. 33, Sec. 1.2; the Upper Critical Point was reported by Cox,[19]

TABLE 28. Experimental solubility studies for 4-ethylpyridine (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Cox[19]	254 and 455	(1) in (2)	Stephenson[20]	273–363	Mutual

TABLE 29. Experimental values for solubility of 4-ethylpyridine (1) in water (2)

T/K	Experimental values x_1	Smoothed values T/K
254.2	0.0614 (Ref. 19)	254.2

T/K	Experimental values x_1	Smoothed values x_1
254.2	0.066 (Estimated lower critical point)	0.066
273.2	8.68×10^{-3} (Ref. 20)	8.65×10^{-3}
283.2	6.99×10^{-3} (Ref. 20)	6.91×10^{-3}
293.2	5.78×10^{-3} (Ref. 20)	5.97×10^{-3}
303.2	5.56×10^{-3} (Ref. 20)	5.40×10^{-3}
313.2	4.98×10^{-3} (Ref. 20)	5.06×10^{-3}
323.2	4.87×10^{-3} (Ref. 20)	4.87×10^{-3}
333.2	4.72×10^{-3} (Ref. 20)	4.79×10^{-3}
343.2	4.98×10^{-3} (Ref. 20)	4.79×10^{-3}
353.2	4.86×10^{-3} (Ref. 20)	4.87×10^{-3}
363.2	4.96×10^{-3} (Ref. 20)	5.03×10^{-3}

T/K	Experimental values x_1	Smoothed values T/K
455.0[a]	0.0881 (Ref. 19)	455.0

[a]Upper critical solution temperature.

The lower critical point is: $T_L = 254.2$ K and $x_L = 0.061$,
The upper critical point is: $T_U = 455.0$ K and $x_U = 0.088$,

where x_L and x_U are the critical mole fractions of the amine.

2.11.3. Critical evaluation of the solubility of 4-ethylpyridine (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_1 = z_L \ln x_L + z_U \ln x_U + c_1\, z_L z_U / T \\ + c_2\, |z_L z_U|^{1/3} + c_3\, z_L z_U, \tag{35}$$

where z_L, z_U are temperature-dependent fractions

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{36}$$

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients c_1, c_2, and c_3 obtained from regression of the experimental solubilities are $c_1 = -432.39$, $c_2 = -4.583$, and $c_3 = 1.94$.

The data of Cox[19] at 254 K and the data of Stephenson[20] at 273–363 K were reported over different ranges of temperature and, therefore, are not classified. All the experimental data and smoothed values are listed in Table 29 and shown in Fig. 10.

2.11.4. Critical evaluation of the solubility of water (2) in 4-ethylpyridine (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2,

$$\ln x_2 = z_L \ln (1 - x_L) + z_U \ln (1 - x_U) + d_1\, z_L z_U / T \\ + d_2\, |z_L z_U|^{1/3} + d_3\, z_L z_U, \tag{37}$$

where z_L, z_U are temperature-dependent fractions,

$$z_L = (T_U - T)/(T_U - T_L); \quad z_U = 1 - z_L. \tag{38}$$
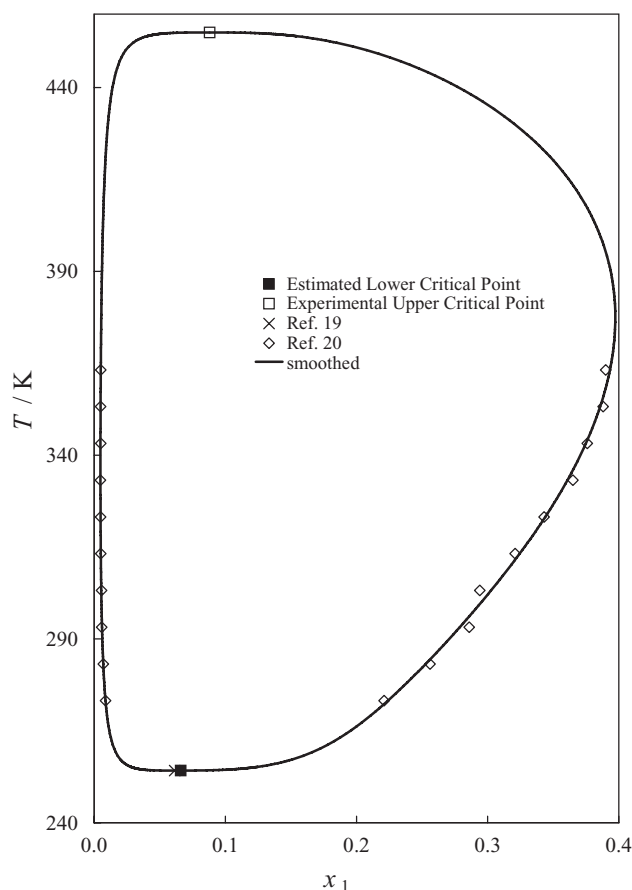
FIG. 10. Mutual solubility data for 4-ethylpyridine (1) + water (2).

Values of the critical parameters x_L, x_U, T_L, T_U are listed above. The coefficients d_1, d_2, and d_3 obtained from regression of the experimental solubilities are $d_1 = 644.23$, $d_2 = -0.414$, and $d_3 = -2.42$.

The solubility of (2) in (1) below the Upper Critical Temperature is reported by Stephenson[20] only, and therefore it is not classified. All the experimental data and smoothed values are listed in Table 30 and shown in Fig. 10.

Experimental Values

Solubility of 4-ethylpyridine in water

$t/°C$	$100\, w_1$	x_1 (compiler)
-19[a]	28	0.0614
181.8[b]	36.5	0.0881

[a]Lower critical solution temperature.
[b]Upper critical solution temperature.

TABLE 30. Experimental values for solubility of water (2) in 4-ethylpyridine (1)

T/K	Experimental values x_2	Smoothed values x_2
273.2	0.779 (Ref. 20)	0.776
283.2	0.744 (Ref. 20)	0.747
293.2	0.714 (Ref. 20)	0.721
303.2	0.706 (Ref. 20)	0.698
313.2	0.679 (Ref. 20)	0.676
323.2	0.657 (Ref. 20)	0.656
333.2	0.635 (Ref. 20)	0.639
343.2	0.624 (Ref. 20)	0.625
353.2	0.612 (Ref. 20)	0.614
363.2	0.610 (Ref. 20)	0.607

Solubility of water in 4-ethylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	37.2	0.779
10.0	32.8	0.744
20.0	29.6	0.714
30.0	28.8	0.706
40.0	26.2	0.679
50.0	24.4	0.657
60.0	22.6	0.635
70.0	21.8	0.624
80.0	21.0	0.612
90.0	20.8	0.610

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99+ mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.06\%$ and mass fraction $w_2 = 1.2\%$ (standard deviation).

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. Solution temperatures above room temperature were determined with the aid of an electrically heated metal block; the sealed tube containing a known mixture of (1) and (2) was fixed in a cavity in the middle of the block and could be observed through the windows. The liquid layers were mixed by rotating the block. For solution temperatures below room temperature, the tube containing the mixture was fixed in a bucket which was then lowered into an unsilvered Dewar vessel filled with methanol and shaken manually. The temperature of the bath was lowered by addition of solid carbon dioxide and then allowed to rise at a rate of 0.1 °C per min until the cloud point was reached.

Source and Purity of Materials:
(1) Prepared by synthesis; distilled; b.p. range 165–165.5 °C; purity better than 99% by infrared spectroscopy.
(2) Not specified.

Estimated Error:
Temperature: ±0.4 at −19 °C and ±0.3 at 181.8 °C.
Solubility: mass fraction $w_1 = ±1\%$ (standard deviation).

2.12. N,N-Dimethylaniline + water

Components:	Original Measurements:
(1) N,N-Dimethylaniline (N,N-Dimethylphenylamine, N,N-Dimethylbenzenamine); $C_8H_{11}N$; [121-69-7] (2) Water; H_2O; [7732-18-5]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:	Prepared by:
Temperature: 0.0–90.0 °C	B. Wisniewska-Goclowska

Components:	Original Measurements:
(1) 4-Ethylpyridine; C_7H_9N; [536-75-4] (2) Water; H_2O; [7732-18-5]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).

Variables:	Prepared by:
Temperature: 0−90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 4-ethylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	4.95	8.68×10^{-3}
10.0	4.02	6.99×10^{-3}
20.0	3.34	5.78×10^{-3}
30.0	3.22	5.56×10^{-3}
40.0	2.89	4.98×10^{-3}
50.0	2.83	4.87×10^{-3}
60.0	2.74	4.72×10^{-3}
70.0	2.89	4.98×10^{-3}
80.0	2.82	4.86×10^{-3}
90.0	2.88	4.96×10^{-3}

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Experimental Values

Solubility of *N*,*N*-dimethylaniline in water

</div>

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	0.154	2.29×10^{-4}
10.0	0.148	2.20×10^{-4}
20.0	0.154	2.29×10^{-4}
30.0	0.168	2.50×10^{-4}
50.0	0.165	2.45×10^{-4}
60.0	0.156	2.32×10^{-4}
70.0	0.142	2.11×10^{-4}
80.0	0.173	2.57×10^{-4}
90.0	0.181	2.69×10^{-4}

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Solubility of water in *N*,*N*-dimethylaniline

</div>

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	0.061	4.1×10^{-3}
10.0	0.081	5.4×10^{-3}
20.0	0.112	7.5×10^{-3}
30.0	0.136	9.1×10^{-3}
40.0	0.145	9.7×10^{-3}
50.0	0.191	0.0127
60.0	0.227	0.0151
70.0	0.214	0.0142
80.0	0.205	0.0136
90.0	0.273	0.0181

<div align="center">

Experimental Values

Solubility of 2,3-dimethylaniline in water

</div>

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	0.90	1.34×10^{-3}
10.0	0.70	1.04×10^{-3}
20.0	0.64	9.5×10^{-4}
30.0	0.50	7.4×10^{-4}
40.0	0.51	7.6×10^{-4}
50.0	0.56	8.3×10^{-4}
60.0	0.70	1.04×10^{-3}
70.0	0.81	1.21×10^{-3}
80.0	0.73	1.09×10^{-3}
90.0	0.81	1.21×10^{-3}

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Solubility of water in 2,3-dimethylaniline

</div>

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	2.25	0.135
10.0	2.32	0.138
20.0	2.23	0.133
30.0	2.64	0.155
40.0	2.78	0.162
50.0	2.19	0.131
60.0	2.17	0.130
70.0	2.50	0.148
80.0	2.42	0.143
90.0	1.74	0.107

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Auxiliary Information

</div>

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99.7 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.002\%$ and mass fraction $w_2 = 0.005\%$ (standard deviation).

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Auxiliary Information

</div>

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99.5 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.02\%$ and mass fraction $w_2 = 0.08\%$ (standard deviation).

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2.13. 2,3-Dimethylaniline + water

</div>

Components:	Original Measurements:
(1) 2,3-Dimethylaniline (1-Amino-2,3-dimethylbenzene, 1,2-Dimethyl-3-aminobenzene, 2,3-Xylidine); C$_8$H$_{11}$N; [87-59-2] (2) Water; H$_2$O; [7732-18-5]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:	Prepared by:
Temperature: 0.0–90.0 °C	B. Wisniewska-Goclowska

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2.14. 2,4-Dimethylaniline + water

</div>

Components:	Original Measurements:
(1) 2,4-Dimethylaniline (1-Amino-2,4-dimethylbenzene, 1,3-Dimethyl-4-aminobenzene, 2,4-xylidine); C$_8$H$_{11}$N; [95-68-1] (2) Water; H$_2$O; [7732-18-5]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:	Prepared by:
Temperature: 0.0–90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,4-dimethylaniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	0.77	1.15×10^{-3}
10.0	0.61	9.1×10^{-4}
20.0	0.59	8.8×10^{-4}
30.0	0.52	7.7×10^{-4}
40.0	0.53	7.9×10^{-4}
50.0	0.53	7.9×10^{-4}
60.0	0.60	8.9×10^{-4}
70.0	0.59	8.8×10^{-4}
80.0	0.64	9.5×10^{-4}
90.0	0.82	1.22×10^{-3}

Solubility of water in 2,4-dimethylaniline

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	3.86	0.213
10.0	2.96	0.171
20.0	2.60	0.153
30.0	2.53	0.149
40.0	2.50	0.148
50.0	3.19	0.182
60.0	2.55	0.150
70.0	2.60	0.153
80.0	2.59	0.152
90.0	2.98	0.172

Experimental Values

Solubility of 2,5-dimethylaniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	0.68	1.01×10^{-3}
10.0	0.48	7.1×10^{-4}
20.0	0.50	7.4×10^{-4}
30.0	0.45	6.7×10^{-4}
40.0	0.42	6.2×10^{-4}
50.0	0.48	7.1×10^{-4}
60.0	0.51	7.6×10^{-4}
70.0	0.59	8.8×10^{-4}
80.0	0.69	1.03×10^{-3}
90.0	0.69	1.03×10^{-3}

Solubility of water in 2,5-dimethylaniline

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	2.84	0.165
10.0	1.57	0.097
20.0	1.88	0.115
30.0	2.13	0.128
40.0	1.74	0.107
50.0	1.85	0.113
60.0	1.83	0.112
70.0	1.94	0.118
80.0	1.81	0.111

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 98.5 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.02\%$ and mass fraction $w_2 = 0.06\%$ (standard deviation).

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 97 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 0.05\%$ (standard deviation).

2.16. 2,6-Dimethylaniline + water

Components:	Original Measurements:
(1) 2,6-Dimethylaniline (1-Amino-2,6-dimethylbenzene, 1,3-Dimethyl-2-aminobenzene, 2,6-Xylidine); $C_8H_{11}N$; [87-62-7] (2) Water; H_2O; [7732-18-5]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:	Prepared by:
Temperature: 0.0–90.0 °C	B. Wisniewska-Goclowska

2.15. 2,5-Dimethylaniline + water

Components:	Original Measurements:
(1) 2,5-Dimethylaniline (1-Amino-2,5-dimethylbenzene, 1,4-Dimethyl-2-aminobenzene, 2,5-Xylidine); $C_8H_{11}N$; [95-78-3] (2) Water; H_2O; [7732-18-5]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:	Prepared by:
Temperature: 0.0–90.0 °C	B. Wisniewska-Goclowska

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Experimental Values

Solubility of 2,6-dimethylaniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	0.68	1.01×10^{-3}
10.0	0.60	8.9×10^{-4}
20.0	0.47	7.0×10^{-4}
30.0	0.45	6.7×10^{-4}
40.0	0.44	6.5×10^{-4}
50.0	0.48	7.1×10^{-4}
60.0	0.51	7.6×10^{-4}
70.0	0.55	8.2×10^{-4}
80.0	0.63	9.4×10^{-4}
90.0	0.80	1.19×10^{-3}

Solubility of water in 2,6-dimethylaniline

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	1.01	0.065
10.0	0.88	0.057
20.0	0.97	0.062
30.0	1.18	0.075
40.0	1.10	0.070
50.0	1.07	0.068
60.0	1.14	0.072
70.0	1.42	0.089
80.0	1.55	0.096
90.0	1.52	0.094

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.03\%$ and mass fraction $w_2 = 0.07\%$ (standard deviation).

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Experimental Values

Solubility of 3,5-dimethylaniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	0.48	7.1×10^{-4}
10.0	0.47	7.0×10^{-4}
20.0	0.35	5.2×10^{-4}
30.0	0.36	5.3×10^{-4}
40.0	0.53	7.9×10^{-4}
50.0	0.41	6.1×10^{-4}
60.0	0.46	6.8×10^{-4}
70.0	0.55	8.2×10^{-4}
80.0	0.56	8.3×10^{-4}
90.0	0.62	9.2×10^{-4}

Solubility of water in 3,5-dimethylaniline

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	2.08	0.125
10.0	2.18	0.131
20.0	1.72	0.106
30.0	2.27	0.136
40.0	2.52	0.149
50.0	2.09	0.126
60.0	2.15	0.129
70.0	1.87	0.114
80.0	2.38	0.141
90.0	2.07	0.125

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 98.5 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 0.1\%$ (standard deviation).

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2.17. 3,5-Dimethylaniline + water

Components:	**Original Measurements:**
(1) 3,5-Dimethylaniline (1-Amino-3,5-dimethylbenzene, 1,3-Dimethyl-5-aminobenzene, 3,5-Xylidine); $C_8H_{11}N$; [108-69-0]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).
(2) Water; H_2O; [7732-18-5]	
Variables:	**Prepared by:**
Temperature: 0.0–90.0 °C	B. Wisniewska-Goclowska

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2.18. *N*-Ethylaniline + water

Components:	**Original Measurements:**
(1) *N*-Ethylaniline (*N*-Ethylaminobenzene, Ethylphenylamine); $C_8H_{11}N$; [103-69-5]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).
(2) Water; H_2O; [7732-18-5]	
Variables:	**Prepared by:**
Temperature: 0.0–90.0 °C	B. Wisniewska-Goclowska

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Experimental Values

Solubility of N-ethylaniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	0.371	5.53×10^{-4}
10.0	0.292	4.35×10^{-4}
20.0	0.285	4.24×10^{-4}
30.0	0.263	3.91×10^{-4}
40.0	0.255	3.79×10^{-4}
50.0	0.261	3.88×10^{-4}
60.0	0.268	3.99×10^{-4}
70.0	0.281	4.18×10^{-4}
80.0	0.305	4.54×10^{-4}
90.0	0.334	4.98×10^{-4}

Solubility of water in N-ethylaniline

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	0.327	0.0216
10.0	0.374	0.0246
20.0	0.420	0.0276
30.0	0.425	0.0279
40.0	0.412	0.0271
50.0	0.550	0.0359
60.0	0.540	0.0352
70.0	0.593	0.0386
80.0	0.564	0.0367
90.0	0.588	0.0383

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 98 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.001\%$ and mass fraction $w_2 = 0.013\%$ (standard deviation).

Experimental Values

Solubility of 2-ethylaniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
10.0	0.72	1.07×10^{-3}
20.0	0.75	1.12×10^{-3}
30.0	0.70	1.04×10^{-3}
40.0	0.65	9.7×10^{-4}
50.0	0.66	9.8×10^{-4}
60.0	0.74	1.10×10^{-3}
70.0	0.76	1.13×10^{-3}
80.0	0.84	1.25×10^{-3}
90.0	0.87	1.30×10^{-3}

Solubility of water in 2-ethylaniline

$t/°C$	$100\,w_2$	x_2 (compiler)
20.0	1.30	0.0814
30.0	1.88	0.1142
40.0	1.81	0.1103
50.0	1.61	0.0991
60.0	1.63	0.1003
70.0	1.59	0.0980
80.0	1.56	0.0963
90.0	1.48	0.0918

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99+ mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 0.08\%$ (standard deviation).

2.20. 4-Ethylaniline + water

Components:	Original Measurements:
(1) 4-Ethylaniline (p-Ethylaniline, 4-Aminoethylbenzene, 4-Ethylbenzenamine); $C_8H_{11}N$; [589-16-2] (2) Water; H_2O; [7732-18-5]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:	Prepared by:
Temperature: 20.0–90.0 °C	B. Wisniewska-Goclowska

2.19. 2-Ethylaniline + water

Components:	Original Measurements:
(1) 2-Ethylaniline (2-Aminoethylbenzene, o-Ethylaniline, 2-Ethylphenylamine); $C_8H_{11}N$; [578-54-1] (2) Water; H_2O; [7732-18-5]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:	Prepared by:
Temperature: 10.0–90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 4-ethylaniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
20.0	0.51	7.6×10^{-4}
30.0	0.44	6.5×10^{-4}
40.0	0.44	6.5×10^{-4}
50.0	0.46	6.8×10^{-4}
60.0	0.48	7.1×10^{-4}
70.0	0.55	8.2×10^{-4}
80.0	0.59	8.8×10^{-4}
90.0	0.72	1.07×10^{-3}

Solubility of water in 4-ethylaniline

$t/°C$	$100\,w_2$	x_2 (compiler)
20.0	3.58	0.200
30.0	3.60	0.201
40.0	3.13	0.179
50.0	3.48	0.196
60.0	3.09	0.177
70.0	4.84	0.255
80.0	3.49	0.196
90.0	2.87	0.166

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99.5 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 0.06\%$ (standard deviation).

2.21. 3-Ethyl-4-methylpyridine + water

Components:	**Original Measurements:**
(1) 3-Ethyl-4-methylpyridine (4-Methyl-3-ethylpyridine, 3-Ethyl-4-picoline, β-Collidine); $C_8H_{11}N$; [529-21-5] (2) Water; H_2O; [7732-18-5]	[27]V. Rothmund, Z. Phys. Chem. **26**, 433 (1898).
Variables:	**Prepared by:**
Temperature: 6.42–190 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 3-ethyl-4-methylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
6.42 ± 0.13	21.18	0.03842
6.95 ± 0.10	13.76	0.02318
8.97 ± 0.13	8.58	0.01376
11.07 ± 0.13	6.71	0.01058
15.10 ± 0.10	4.78	7.41×10^{-3}
17.10 ± 0.15	4.18	6.45×10^{-3}
28.07 ± 0.23	2.73	4.16×10^{-3}
36.32 ± 0.18	2.16	3.27×10^{-3}
54.25 ± 0.30	1.75	2.64×10^{-3}
108.0 ± 0.3	1.75	2.64×10^{-3}
136.5 ± 1.1	2.16	3.27×10^{-3}
153.8 ± 1.1	2.73	4.16×10^{-3}
190 ± 8	4.18	6.45×10^{-3}

Solubility of water in 3-ethyl-4-methylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
7.22 ± 0.13	66.84	0.9313
12.68 ± 0.08	54.03	0.8877
21.15 ± 0.10	44.18	0.8418
27.30 ± 0.20	39.04	0.8116
37.27 ± 0.13	31.88	0.7589
49.10 ± 0.20	25.03	0.6918
76.85 ± 0.35	14.78	0.5384
83.75 ± 0.75	12.71	0.4947
95.50 ± 1.10	12.40	0.4877
162.7 ± 0.5	12.40	0.4877
164 ± 2	12.71	0.4947
173 ± 8	14.78	0.5384

Auxiliary Information

Method/Apparatus/Procedure:
The synthetic method was used. The measurements were carried out in thick-walled glass capillaries. The temperatures of appearance and disappearance of turbidity were observed and the mean value was adopted.

Source and Purity of Materials:
(1) Not specified.
(2) Not specified.

Estimated Error:
Temperature: see above.

2.22. 5-Ethyl-2-methylpyridine + water

Components:	**Original Measurements:**
(1) 5-Ethyl-2-methylpyridine (5-Ethyl-2-picoline, 3-Ethyl-6-methylpyridine, Aldehydine, Aldehydecollidine); $C_8H_{11}N$; [104-90-5] (2) Water; H_2O; [7732-18-5]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).
Variables:	**Prepared by:**
Temperature: 0–90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 5-ethyl-2-methylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	2.09	3.16×10^{-3}
10.0	1.54	2.32×10^{-3}
20.0	1.20	1.80×10^{-3}
30.0	1.05	1.58×10^{-3}
40.0	0.88	1.32×10^{-3}
50.0	0.85	1.27×10^{-3}
60.0	0.92	1.38×10^{-3}
70.0	0.92	1.38×10^{-3}
80.0	0.88	1.32×10^{-3}
90.0	0.89	1.33×10^{-3}

Solubility of water in 5-ethyl-2-methylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	27.0	0.713
10.0	24.9	0.690
20.0	19.7	0.623
30.0	15.6	0.554
40.0	15.2	0.547
50.0	14.0	0.523
60.0	14.0	0.523
70.0	13.0	0.501
80.0	12.1	0.481
90.0	12.3	0.485

Experimental Values

Solubility of 4-isopropylpyridine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	1.55	2.34×10^{-3}
10.0	1.38	2.08×10^{-3}
20.0	1.22	1.83×10^{-3}
30.0	1.09	1.64×10^{-3}
40.0	1.02	1.53×10^{-3}
50.0	0.97	1.45×10^{-3}
60.0	0.92	1.38×10^{-3}
70.0	0.97	1.45×10^{-3}
80.0	1.09	1.64×10^{-3}
90.0	1.08	1.62×10^{-3}

Solubility of water in 4-isopropylpyridine

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	17.2	0.583
10.0	15.7	0.556
20.0	15.5	0.552
30.0	13.1	0.503
40.0	13.4	0.510
50.0	12.7	0.495
60.0	12.0	0.478
70.0	11.7	0.471
80.0	11.5	0.466
90.0	12.0	0.478

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 98 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.04\%$ and mass fraction $w_2 = 0.3\%$ (standard deviation).

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 95 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.05\%$ and mass fraction $w_2 = 0.4\%$ (standard deviation).

2.23. 4-Isopropylpyridine + water

Components:	Original Measurements:
(1) 4-Isopropylpyridine (4-(1-Methylethyl)pyridine); $C_8H_{11}N$; [696-30-0] (2) Water; H_2O; [7732-18-5]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).

Variables:	Prepared by:
Temperature: 0−90.0 °C	B. Wisniewska-Goclowska

2.24. 2,4,6-Trimethylpyridine + water

Components:	Original Measurements:
(1) 2,4,6-Trimethylpyridine (2,4,6-Collidine); $C_8H_{11}N$; [108-75-8] (2) Water; H_2O; [7732-18-5]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).

Variables:	Prepared by:
Temperature: 6.5−90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2,4,6-trimethylpyridine in water

t/°C	$100\,w_1$	x_1 (compiler)
6.5	13.05	0.02183
7.5	9.73	0.01578
8.2	6.10	9.57×10^{-3}
20.0	3.57	5.48×10^{-3}
30.0	2.61	3.97×10^{-3}
40.0	2.10	3.18×10^{-3}
50.0	1.85	2.80×10^{-3}
60.0	1.71	2.58×10^{-3}
70.0	1.66	2.50×10^{-3}
80.0	1.54	2.32×10^{-3}
90.0	1.63	2.46×10^{-3}

Solubility of water in 2,4,6-trimethylpyridine

t/°C	$100\,w_2$	x_2 (compiler)
6.5	77.2	0.958
7.5	65.4	0.927
8.2	58.0	0.903
20.0	45.3	0.848
30.0	36.2	0.792
40.0	30.3	0.745
50.0	25.5	0.697
60.0	21.6	0.649
70.0	18.1	0.598
80.0	17.1	0.581
90.0	15.3	0.548

The lower critical solution temperature was reported to be 6.5 °C.

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 1.4\%$ (standard deviation).

2.25. Isoquinoline + water

Components:
(1) Isoquinoline; C_9H_7N; [119-65-3]
(2) Water; H_2O; [7732-18-5]

Evaluators:
A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland, January, 2012.

TABLE 31. Experimental solubility studies for isoquinoline (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Albert[28]	293	(1) in (2)	Zegalska[29]	283–484	Mutual

2.25.1. Experimental solubility studies for isoquinoline (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 31 together with the temperature range.

2.25.2. Upper critical point

The upper critical solution temperature $T_c = 483.5$ K was adopted from the data of Zegalska.[29] The corresponding mole fraction of the amine $x_c = 0.110$ was adjusted to the experimental data as described in Ref. 33, Sec. 1.2.

2.25.3. Critical evaluation of the solubility of isoquinoline (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2.1,

$$\ln x_1 = \ln x_c + a_1(T_c/T - 1) + a_2|(1 - T/T_c)|^{(1/3)} + a_3(1 - T/T_c), \qquad (39)$$

where x_c and T_c listed above were used. The coefficients a_1, a_2, and a_3 obtained from regression of the experimental solubilities are $a_1 = -11.38$, $a_2 = -6.366$, and $a_3 = 16.12$.

A single point reported by Albert[28] disagreed with the solubility curve measured by Zegalska.[29] The data are not

TABLE 32. Experimental values for solubility of isoquinoline (1) in water (2)

T/K	Experimental values x_1	Smoothed values x_1
293.2	6.7×10^{-5} (Ref. 28), 7.4×10^{-4} (Ref. 29), 7.6×10^{-4} (Ref. 29)	3.64×10^{-4}
313.2	9.3×10^{-4} (Ref. 29), 9.4×10^{-4} (Ref. 29)	7.35×10^{-4}
327.2	1.0×10^{-3} (Ref. 29)	1.11×10^{-3}
351.3	1.8×10^{-3} (Ref. 29)	2.00×10^{-3}
435.4	7.1×10^{-3} (Ref. 29)	8.14×10^{-3}
460.6	0.0146 (Ref. 29)	0.0134
475.7	0.0251 (Ref. 29)	0.0236
483.5	0.11 (Estimated upper critical point)	0.11

T/K	Experimental values x_1	Smoothed values T/K
481.2	0.0368 (Ref. 29)	480.9
482.4	0.0562 (Ref. 29)	483.0
483.5	0.078 (Ref. 29)	483.4
483.6	0.0750 (Ref. 29)	483.4
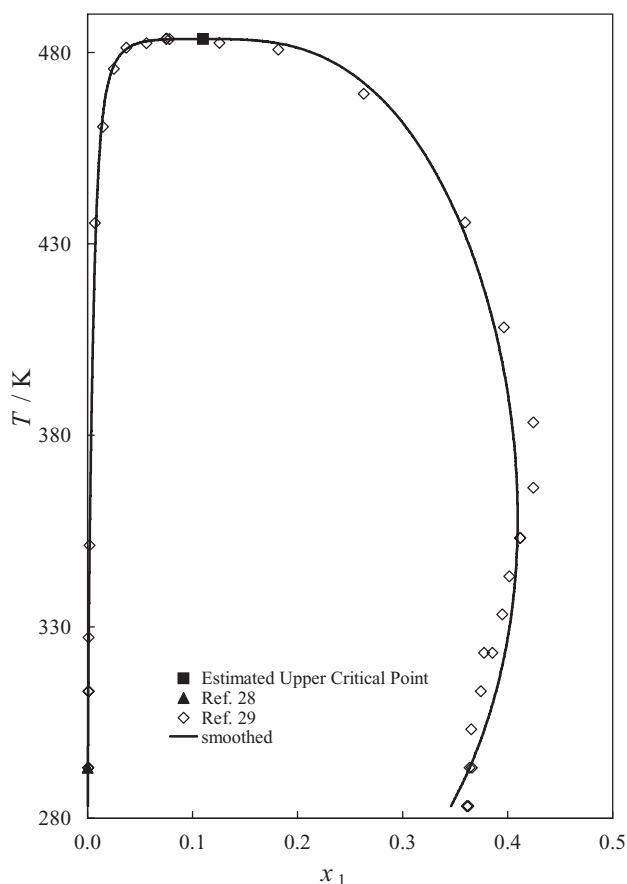
FIG. 11. Mutual solubility data for isoquinoline (1) + water (2).

TABLE 33. Experimental values for solubility of water (2) in isoquinoline (1)

T/K	Experimental values x_2	Smoothed values x_2
283.2	0.638 (Ref. 29), 0.639 (Ref. 29)	0.654
293.2	0.634 (Ref. 29), 0.636 (Ref. 29)	0.636
303.2	0.635 (Ref. 29)	0.622
313.2	0.626 (Ref. 29)	0.611
323.2	0.615 (Ref. 29), 0.623 (Ref. 29)	0.603
333.2	0.605 (Ref. 29)	0.597
343.2	0.598 (Ref. 29)	0.593
353.2	0.588 (Ref. 29), 0.588 (Ref. 29)	0.591
366.3	0.576 (Ref. 29)	0.591
383.4	0.576 (Ref. 29)	0.596
408.2	0.604 (Ref. 29)	0.613
435.6	0.641 (Ref. 29)	0.645
469.2	0.737 (Ref. 29)	0.725
480.7	0.819 (Ref. 29)	0.794

T/K	Experimental values x_2	Smoothed values T/K
482.5	0.875 (Ref. 29)	483.5

classified. All the experimental data and smoothed values are listed in Table 32 and shown in Fig. 11.

2.25.4. Critical evaluation of the solubility of water (2) in isoquinoline (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2.1,

$$\ln x_2 = \ln (1 - x_c) + b_1(T_c/T - 1) \\ + b_2|(1 - T/T_c)|^{(1/3)} + b_3(1 - T/T_c), \quad (40)$$

where $x_{c,1}$ and T_c listed above were used. The coefficients b_1, b_2, and b_3 obtained from regression of the experimental solubilities are $b_1 = 1.20$, $b_2 = -0.621$, and $b_3 = -1.68$.

The solubility of (2) in (1) was reported by Zegalska[29] only, and therefore it is not classified. All the experimental data and smoothed values are listed in Table 33 and shown in Fig. 11.

Components:
(1) Isoquinoline; C_9H_7N; [119-65-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[28]A. Albert, Chem. Ind. **1956**, 252.

Variables:
One temperature: 20 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of quinoline in water

t/°C	ml (2)/g (1)	100 w_1 (compiler)	x_1 (compiler)
20	2100	0.048	6.7×10^{-5}

Auxiliary Information

Method/Apparatus/Procedure:
The spectroscopic method was used. A sample of 0.1 g (1) was dissolved in 8 ml of hot water, and the resulting milky emulsion left for 10 h in a thermostat. The aqueous layer was decanted and centrifuged. A sample of 5 ml was diluted 800-fold with 0.1 N hydrochloric acid and the absorbance of the quinolinium cation peak at 313 μm was measured.

Source and Purity of Materials:
(1) Not specified.
(2) Not specified.

Estimated Error:
Temperature: ±0.02 °C.

Components:
(1) Isoquinoline; C_9H_7N;
[119-65-3]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[29]B. Zegalska, Bull. Acad. Pol. Sci., Ser. Sci. Chim. **7**, 357 (1968).

Variables:
Temperature: 10–210.4 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of isoquinoline in water

$t/°C$	$100\,w_1$	x_1
20[a]	0.53	7.4×10^{-4}[b]
20[c]	0.54	7.6×10^{-4}[b]
40[a]	0.67	9.4×10^{-4}[b]
40[c]	0.66	9.3×10^{-4}[b]
54.05[d]	0.71[b]	1.00×10^{-3}
78.10[d]	1.28[b]	1.80×10^{-3}
162.20[d]	4.88[b]	7.10×10^{-3}
187.45[d]	9.60[b]	0.0146
202.50[d]	15.58[b]	0.0251
208.05[d]	21.50[b]	0.0368
209.20[d]	29.91[b]	0.0562
210.40[d,e]	36.76[b]	0.0750

Solubility of water in isoquinoline

$t/°C$	$100\,w_2$	x_2
10[a]	19.7	0.638[b]
10[c]	19.8	0.639[b]
20[a]	19.6	0.636[b]
20[c]	19.6	0.636[b]
30[c]	19.5	0.635[b]
40[c]	18.9	0.626[b]
50[c]	18.2	0.615[b]
50[a]	18.7	0.623[b]
60[c]	17.6	0.605[b]
70[c]	17.2	0.5983[b]
80[c]	16.6	0.5880[b]
80[a]	16.6	0.5880[b]
93.15[d]	15.92[b]	0.5757
110.20[d]	15.92[b]	0.5757
135.05[d]	17.52[b]	0.6035
162.40[d]	19.92[b]	0.6406
196.00[d]	28.11[b]	0.7370
207.55[d]	38.62[b]	0.8185
209.35[d]	49.32[b]	0.8746
210.35[d]	62.25[b]	0.9220
210.40[d,e]	63.24[b]	0.9250

[a]Titration method.
[b]Calculated by compiler.
[c]Refractometric method.
[d]Synthetic method.
[e]Upper critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:

Three methods were used. Below 80 °C, the titration method was used. A known amount of component (1) in a thermostated vessel with stirrer was titrated with component (2). Next, the vessel was agitated and a permanent turbidity marked the coexistence point. Above 80 °C, the synthetic method was used. The ampoules were filled with mixtures of (1) and (2), sealed and placed in a silicon-oil bath. The samples were first overheated and cooled slowly with observation of the appearance of turbidity and of the meniscus. Additionally, the refractometric method was used. Component (1) was placed in the thermostatic vessel at known temperature. Next, component (2) was added and agitated for 15 min. After phase separation, samples were drawn from the lower layer and analyzed by standard refractive index curve.

Source and Purity of Materials:

(1) Midland Tar Distillers or Schuchardt's; purified by distillation and crystallization; purity by cryometry 99.8%; b.p. 243.25 °C, m.p. 24.7 °C, $d_4^{25} = 1.62077$.
(2) Not specified.

Estimated Error:

Temperature: ± 0.1 °C at 80–200 °C and ± 0.4 °C at 200–230 °C (by the synthetic method).

2.26. Quinoline + water

Components:
(1) Quinoline (Benzo[*b*]pyridine); C$_9$H$_7$N; [91-22-5]
(2) Water; H$_2$O; [7732-18-5]

Evaluators:
A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland, January, 2012.

2.26.1. Experimental solubility studies for quinoline (1) + water (2)

The experimental solubility data for (1) + (2) have been investigated as listed in Table 34 together with the temperature range.

2.26.2. Upper critical point

The Upper Critical Temperature $T_c = 497.2$ K was adopted from the data of Zegalska.[29] The corresponding mole fraction of the amine $x_c = 0.110$ was adjusted to the experimental data as described in Ref. 33, Sec. 1.2.

2.26.3. Critical evaluation of the solubility of quinoline (1) in water (2)

The solubility of (1) in (2) was smoothed by the equation discussed in Ref. 33, Sec. 1.2.1,

$$\ln x_1 = \ln x_c + a_1(T_c/T - 1) + a_2|(1 - T/T_c)|^{(1/3)} + a_3(1 - T/T_c), \quad (41)$$

where x_c and T_c listed above were used. The coefficients a_1, a_2, and a_3 obtained from regression of the experimental solubilities are $a_1 = 5.10$, $a_2 = -4.309$, and $a_3 = -12.39$.

Following the criteria discussed in Ref. 33, Sec. 1.3, all the data were classified as tentative or doubtful. All the experimental data and smoothed values are listed in Table 35 and shown in Fig. 12.

TABLE 34. Experimental solubility studies for quinoline (1) + water (2)

Author(s)	T/K	Solubility	Author(s)	T/K	Solubility
Albert[28]	293	(1) in (2)	Stephenson[20]	281–363	Mutual
Letcher and Deenadayalu[30]	298	(1) in (2)	Zegalska[29]	293–497	Mutual

TABLE 35. Experimental values for solubility of quinoline (1) in water (2)

T/K	Experimental values x_1 (T = tentative, D = doubtful)	Smoothed values x_1
273.2	1.0×10^{-3} (T; Ref. 20)	9.9×10^{-4}
283.2	1.08×10^{-3} (D; Ref. 20)	9.7×10^{-4}
293.2	1.18×10^{-3} (D; Ref. 20),	9.6×10^{-4}
	9.1×10^{-4} (T; Ref. 28), 8.8×10^{-4}	
	(T; Ref. 29), 9.0×10^{-4} (T; Ref. 29)	
293.3	9.0×10^{-4} (T; Ref. 29)	9.6×10^{-4}
303.2	9.3×10^{-4} (T; Ref. 20)	9.8×10^{-4}
313.2	1.12×10^{-3} (D; Ref. 20), 1.00×10^{-3}	1.02×10^{-3}
	(T; Ref. 29), 1.01×10^{-3} (T; Ref. 29)	
323.2	1.18×10^{-3} (T; Ref. 20)	1.07×10^{-3}
325.5	1.0×10^{-3} (T; Ref. 29)	1.09×10^{-3}
333.2	1.10×10^{-3} (T; Ref. 20)	1.16×10^{-3}
339.6	1.2×10^{-3} (T; Ref. 29)	1.22×10^{-3}
343.2	1.12×10^{-3} (D; Ref. 20)	1.26×10^{-3}
353.2	1.31×10^{-3} (T; Ref. 20)	1.40×10^{-3}
356.4	1.3×10^{-3} (D; Ref. 29)	1.46×10^{-3}
361.3	1.6×10^{-3} (T; Ref. 29)	1.54×10^{-3}
363.2	1.82×10^{-3} (D; Ref. 20)	1.58×10^{-3}
375.5	2.0×10^{-3} (T; Ref. 29)	1.87×10^{-3}
437.0	6.7×10^{-3} (D; Ref. 29)	5.87×10^{-3}
470.3	0.0150 (T; Ref. 29)	0.0147
492.6	0.0351 (D; Ref. 29)	0.0416
496.2	0.0612 (T; Ref. 29)	0.0623
497.2	0.11 (Estimated upper critical solution point)	0.11

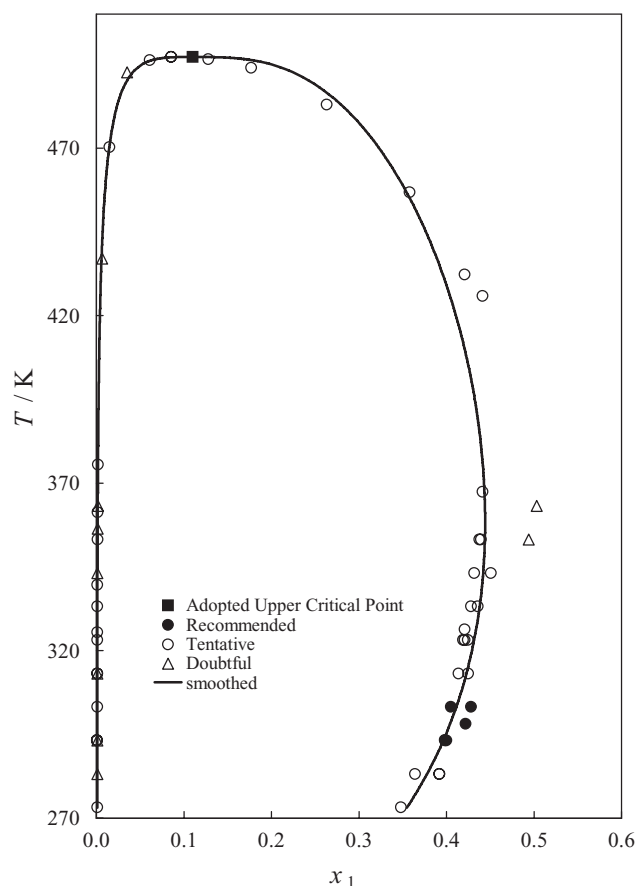| T/K	Experimental values x_1 (T = tentative)	Smoothed values T/K
497.2	0.0857 (T; Ref. 29)	497.2

FIG. 12. Mutual solubility data for quinoline (1) + water (2).

2.26.4. Critical evaluation of the solubility of water (2) in quinoline (1)

The solubility of (2) in (1) was smoothed by the equation discussed in Ref. 33, Sec. 1.2.1,

$$\ln x_2 = \ln(1 - x_c) + b_1(T_c/T - 1) \\ + b_2|(1 - T/T_c)|^{(1/3)} + b_3(1 - T/T_c), \quad (42)$$

where $x_{c,1}$ and T_c listed above were used. The coefficients b_1, b_2, and b_3 obtained from regression of the experimental solubilities are $b_1 = 1.32$, $b_2 = -0.626$, and $b_3 = -2.06$.

Following the criteria discussed in Ref. 33, Sec. 1.3, the data were classified as recommended, tentative, or doubtful. All the experimental data and smoothed values are listed in Table 36 and shown in Fig. 12.

Components:	Original Measurements:
(1) Quinoline (Benzo[b]pyridine);	[28]A. Albert, Chem. Ind. **1956**, 252.
C$_9$H$_7$N; [91-22-5]	
(2) Water; H$_2$O; [7732-18-5]	

Variables:	Prepared by:
One temperature: 20 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of quinoline in water

$t/°C$	ml (2)/g (1)	$100 w_1$	x_1 (compiler)
20	153	0.65	9.1×10^{-4}

Auxiliary Information

Method/Apparatus/Procedure:
The spectroscopic method was used. A sample of 0.1 g (1) was dissolved in 8 ml of hot water, and the resulting milky emulsion left for 10 h in a thermostat. The aqueous layer was decanted and centrifuged. A sample of 5 ml was diluted 800-fold with 0.1 N hydrochloric acid and the absorbance of the quinolinium cation peak at 313 µm was measured.

Source and Purity of Materials:
(1) Not specified.
(2) Not specified.

Estimated Error:
Temperature: ±0.02 °C.

TABLE 36. Experimental values for solubility of water (2) in quinoline (1)

T/K	Experimental values x_2 (R = recommended, T = tentative, D = doubtful)	Smoothed values x_2
273.2	0.652 (T; Ref. 20)	0.645
283.2	0.636 (T; Ref. 20), 0.608 (T; Ref. 29)	0.623
293.2	0.600 (R; Ref. 29), 0.602 (R; Ref. 29)	0.604
293.3	0.602 (R; Ref. 29)	0.604
298.2	0.578 (R; Ref. 30)	0.596
303.2	0.572 (R; Ref. 20), 0.595 (R; Ref. 29)	0.589
313.2	0.575 (T; Ref. 20), 0.586 (T; Ref. 29)	0.577
323.2	0.581 (T; Ref. 20), 0.576 (T; Ref. 29), 0.579 (T; Ref. 29)	0.568
326.3	0.579 (T; Ref. 29)	0.566
333.2	0.564 (T; Ref. 20), 0.572 (T; Ref. 29)	0.562
343.2	0.549 (T; Ref. 20), 0.568 (T; Ref. 29)	0.558
353.2	0.506 (D; Ref. 20), 0.561 (T; Ref. 29), 0.562 (T; Ref. 29)	0.556
363.2	0.497 (D; Ref. 20)	0.556
367.4	0.559 (T; Ref. 29)	0.557
425.9	0.559 (T; Ref. 29)	0.596
432.3	0.579 (T; Ref. 29)	0.604
456.8	0.642 (T; Ref. 29)	0.645
483.0	0.737 (T; Ref. 29)	0.720
494.0	0.823 (T; Ref. 29)	0.788
496.5	0.872 (T; Ref. 29)	0.828

Components:	Original Measurements:
(1) Quinoline (Benzo[b]pyridine); C$_9$H$_7$N; [91-22-5] (2) Water; H$_2$O; [7732-18-5]	[30]T. M. Letcher, S. Ravindran, and S. E. Radloff, Fluid Phase Equilib. **69**, 251 (1991).
Variables: One temperature: 298.2 K	**Prepared by:** B. Wisniewska-Goclowska

Experimental Values

Solubility of quinoline in water

T/K	100 w_1 (compiler)	x_1
298.2	84.0	0.422

Auxiliary Information

Method/Apparatus/Procedure:
The cloud-point method was used. Solubilities were determined by formation of a cloudy mixture after the addition of a known mass of one component to a known mass of the other component. The results are part of various ternary solubility measurements.

Source and Purity of Materials:
(1) Source not specified; dried by addition of anhydrous potassium iodide and distilled; purity by glc >99.5 mol %.
(2) Not specified.

Estimated Error:
Solubility: mole fraction $x_1 = \pm 0.005$.

Components:	Original Measurements:
(1) Quinoline (Benzo[b]pyridine); C$_9$H$_7$N; [91-22-5] (2) Water; H$_2$O; [7732-18-5]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).
Variables: Temperature: 0−90.0 °C	**Prepared by:** B. Wisniewska-Goclowska

Experimental Values

Solubility of quinoline in water

t/°C	100 w_1	x_1 (compiler)
0.0	0.71	1.00×10^{-3}
10.0	0.77	1.08×10^{-3}
20.0	0.84	1.18×10^{-3}
30.0	0.66	9.3×10^{-4}
40.0	0.80	1.12×10^{-3}
50.0	0.84	1.18×10^{-3}
60.0	0.78	1.10×10^{-3}
70.0	0.80	1.12×10^{-3}
80.0	0.93	1.31×10^{-3}
90.0	1.29	1.82×10^{-3}

Solubility of water in quinoline

t/°C	100 w_2	x_2 (compiler)
0.0	20.70	0.652
10.0	19.60	0.636
30.0	15.70	0.572
40.0	15.90	0.575
50.0	16.20	0.581
60.0	15.30	0.564
70.0	14.50	0.549
80.0	12.50	0.506
90.0	12.10	0.497

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ± 0.1 °C.
Solubility: mass fraction $w_1 = 0.04\%$ and mass fraction $w_2 = 0.7\%$ (standard deviation).

Components:	Original Measurements:
(1) Quinoline (Benzo[b]pyridine); C$_9$H$_7$N; [91-22-5] (2) Water; H$_2$O; [7732-18-5]	[29]B. Zegalska, Bull. Acad. Pol. Sci., Ser. Sci. Chim. **7**, 357 (1968).
Variables: Temperature: 10–224.05 °C	**Prepared by:** B. Wisniewska-Goclowska

Experimental Values

Solubility of quinoline in water

$t/°C$	$100\,w_1$	x_1
20[a]	0.63	8.8×10^{-4}[b]
20[c]	0.64	9.0×10^{-4}[b]
20.10[d]	0.64[b]	9.0×10^{-4}
40[a]	0.71	1.00×10^{-3}[b]
40[c]	0.72	1.01×10^{-3}[b]
52.30[d]	0.71[b]	1.00×10^{-3}
66.45[d]	0.85[b]	1.20×10^{-3}
83.20[d]	0.92[b]	1.30×10^{-3}
88.15[d]	1.14[b]	1.60×10^{-3}
102.35[d]	1.42[b]	2.00×10^{-3}
163.80[d]	4.61[b]	6.70×10^{-3}
197.10[d]	9.84[b]	0.01500
219.45[d]	20.68[b]	0.03510
223.00[d]	31.85[b]	0.06120
224.05[d,e]	40.19[b]	0.08570

Solubility of water in quinoline

$t/°C$	$100\,w_2$	x_2
10[a]	17.8	0.608[b]
10[c]	17.8	0.608[b]
20[a]	17.3	0.600[b]
20[c]	17.4	0.602[b]
20.10[d]	17.41[b]	0.6018
30[c]	17.0	0.595[b]
40[c]	16.5	0.586[b]
50[a]	15.9	0.576[b]
50[c]	16.1	0.579[b]
53.10[d]	16.12[b]	0.5793
60[c]	15.7	0.5718[b]
70[c]	15.5	0.5681[b]
80[a]	15.1	0.5605[b]
80[c]	15.2	0.5624[b]
94.25[d]	15.01[b]	0.5587
152.75[d]	15.01[b]	0.5587
159.10[d]	16.12[b]	0.5793
183.60[d]	20.02[b]	0.6421
209.80[d]	28.09[b]	0.7368
220.80[d]	39.33[b]	0.8229
223.30[d]	48.73[b]	0.8720
224.05[d,e]	49.81[b]	0.9143

[a]Titration method.
[b]Calculated by compiler.
[c]Refractometric method.
[d]Synthetic method.
[e]Upper critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
Three methods were used. Below 80 °C, the titration method was used. A known amount of component (1) in a thermostatted vessel with stirrer was titrated with component (2). Next, the vessel was agitated and a permanent turbidity marked the coexistence point. Above 80 °C, the synthetic method was used. The ampoules were filled with mixtures of (1) and (2), sealed and placed in a silicon-oil bath. The samples were first overheated and cooled slowly with observation of the appearance of turbidity and of meniscus. Additionally, the refractometric method was used. The component (1) was placed in the thermostatic vessel at known temperature. Next, component (2) was added and agitated for 15 min. After phase separation, samples were drawn from the lower layer and analyzed by standard refractive index curve.

Source and Purity of Materials:
(1) Midland Tar Distillers or Schuchardt's; purified by distillation and crystallization; purity by cryometry 99.71%; b.p. 237.65 °C, m.p. −14.5 °C, $d_4^{25} = 1.6273$.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C at 80–200 °C and ±0.4 °C at 200–230 °C (by the synthetic method).

2.27. Benzyldimethylamine + water

Components:
(1) Benzyldimethylamine (*N,N*-Dimethylbenzylamine); C$_9$H$_{13}$N; [103-83-3]
(2) Water; H$_2$O; [7732-18-5]

Original Measurements:
[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:
Temperature: 0.0–90.0 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of benzyldimethylamine in water

$t/°C$	$100\,w_1$	x_1 (compiler)
0.0	2.59	3.53×10^{-3}
10.0	1.93	2.61×10^{-3}
20.0	1.41	1.90×10^{-3}
30.0	1.13	1.52×10^{-3}
40.0	0.92	1.23×10^{-3}
50.0	0.81	1.08×10^{-3}
60.0	0.70	9.3×10^{-4}
70.0	0.67	8.9×10^{-4}
80.0	0.61	8.1×10^{-4}
90.0	0.58	7.7×10^{-4}

Solubility of water in benzyldimethylamine

$t/°C$	$100\,w_2$	x_2 (compiler)
0.0	12.35	0.514
10.0	5.49	0.304
20.0	3.01	0.189
30.0	2.35	0.153
40.0	1.93	0.129
50.0	1.83	0.123
60.0	1.85	0.124
70.0	1.69	0.115
80.0	1.75	0.118
90.0	1.96	0.131

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99+ mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ± 0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 0.09\%$ (standard deviation).

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ± 0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 0.2\%$ (standard deviation).

2.28. 4-*tert*-Butylpyridine + water

Components:	Original Measurements:
(1) 4-*tert*-Butylpyridine (4-(1,1-Dimethylethyl)pyridine); $C_9H_{13}N$; [3978-81-2]	[20]R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
Temperature: 0–90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 4-*tert*-butylpyridine in water

t/°C	$100 w_1$	x_1 (compiler)
0.0	0.56	7.5×10^{-4}
10.0	0.44	5.9×10^{-4}
20.0	0.43	5.8×10^{-4}
30.0	0.40	5.3×10^{-4}
40.0	0.37	4.9×10^{-4}
50.0	0.35	4.7×10^{-4}
60.0	0.38	5.1×10^{-4}
70.0	0.39	5.2×10^{-4}
80.0	0.45	6.0×10^{-4}
90.0	0.47	6.3×10^{-4}

Solubility of water in 4-*tert*-butylpyridine

t/°C	$100 w_2$	x_2 (compiler)
0.0	11.4	0.491
10.0	11.9	0.503
20.0	10.8	0.476
30.0	10.6	0.471
40.0	9.6	0.443
50.0	9.2	0.432
60.0	9.3	0.435
70.0	9.1	0.429
80.0	9.1	0.429
90.0	8.9	0.423

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

2.29. 2-Ethyl-6-methylaniline + water

Components:	Original Measurements:
(1) 2-Ethyl-6-methylaniline (6-Ethyl-*o*-toluidine); $C_9H_{13}N$; [24549-06-2]	[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).
(2) Water; H_2O; [7732-18-5]	

Variables:	Prepared by:
Temperature: 0.0–90.0 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of 2-ethyl-6-methylaniline in water

t/°C	$100 w_1$	x_1 (compiler)
0.0	0.46	6.1×10^{-4}
10.0	0.46	6.1×10^{-4}
20.0	0.36	4.8×10^{-4}
30.0	0.37	4.9×10^{-4}
40.0	0.36	4.8×10^{-4}
50.0	0.37	4.9×10^{-4}
60.0	0.33	4.4×10^{-4}
70.0	0.33	4.4×10^{-4}
80.0	0.41	5.4×10^{-4}
90.0	0.42	5.6×10^{-4}

Solubility of water in 2-ethyl-6-methylaniline

t/°C	$100 w_2$	x_2 (compiler)
0.0	0.52	0.038
10.0	0.58	0.042
20.0	0.66	0.048
30.0	0.73	0.053
40.0	0.99	0.070
50.0	0.88	0.063
60.0	0.86	0.062
70.0	0.81	0.058
80.0	0.81	0.058
90.0	0.89	0.064

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 0.02\%$ (standard deviation).

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99+ mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 0.03\%$ (standard deviation).

2.30. 2-Isopropylaniline + water

Components:
(1) 2-Isopropylaniline (2-Aminocumene); $C_9H_{13}N$; [643-28-7]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:
Temperature: 10.0–90.0 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 2-isopropylaniline in water

$t/°C$	$100\,w_1$	x_1 (compiler)
10.0	0.34	4.5×10^{-4}
20.0	0.34	4.5×10^{-4}
30.0	0.34	4.5×10^{-4}
40.0	0.47	6.2×10^{-4}
50.0	0.41	5.4×10^{-4}
60.0	0.49	6.5×10^{-4}
70.0	0.48	6.4×10^{-4}
80.0	0.37	4.9×10^{-4}
90.0	0.42	5.6×10^{-4}

Solubility of water in 2-isopropylaniline

$t/°C$	$100\,w_2$	x_2 (compiler)
10.0	0.90	0.064
20.0	1.27	0.088
30.0	0.93	0.066
40.0	1.39	0.096
50.0	1.13	0.079
60.0	1.29	0.090
70.0	1.42	0.098
80.0	1.42	0.098
90.0	1.52	0.104

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

2.31. 2-Methylquinoline + water

Components:
(1) 2-Methylquinoline (Quinaldine); $C_{10}H_9N$; [91-63-4]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[29]B. Zegalska, Bull. Acad. Pol. Sci., Ser. Sci. Chim. **7**, 357 (1968).

Variables:
Temperature: 20–265.4 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 2-methylquinoline in water

$t/°C$	$100\,w_1$ (compiler)	x_1
20.00	0.2	3×10^{-4}
180.15	2.7	3.5×10^{-3}
213.45	5.3	7.0×10^{-3}
240.45	12.1	0.0170
259.50	19.7	0.0300
264.30	30.1	0.0515
265.40[a]	41.2	0.0810

Solubility of water in 2-methylquinoline

$t/°C$	$100\,w_2$ (compiler)	x_2
21.50	14.61	0.5762
74.40	12.41	0.5297
160.00	12.60	0.5340
199.35	14.91	0.5820
225.10	19.51	0.6583
251.50	30.32	0.7757
264.10	49.64	0.8868
265.40[a]	58.81	0.9190

[a]Upper critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
Two methods were used. Below 80 °C, the titration method was used. A known amount of component (1) in a thermostated vessel with stirrer was titrated with component (2). Next, the vessel was agitated and a permanent turbidity marked the coexistence point. Above 80 °C, the synthetic method was used. The ampoules were filled with mixtures of (1) and (2), sealed and placed in a silicon-oil bath. The samples were first overheated and cooled slowly with observation of the appearance of turbidity and of meniscus.

Source and Purity of Materials:
(1) Midland Tar Distillers or Schuchardt's; purified by distillation and crystallization; purity by cryometry 99.00%; b.p. 247.75 °C, m.p. −2.9 °C, $d_4^{25} = 1.6128$.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C at 80–200 °C and ±0.4 °C at 200–230 °C, and ±0.8 °C above 230 °C (by the synthetic method).

2.32. 4-Methylquinoline + water

Components:
(1) 4-Methylquinoline (Lepidine, 4-Lepidine, γ-Methylquinoline); $C_{10}H_9N$; [491-35-0]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[29]B. Zegalska, Bull. Acad. Pol. Sci., Ser. Sci. Chim. **7**, 357 (1968).

Variables:
Temperature: 20–243.7 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 4-methylquinoline in water

$t/°C$	$100\,w_1$ (compiler)	x_1
20.00	0.16	2.0×10^{-4}
73.20	0.79	1.0×10^{-3}
163.95	3.24	4.2×10^{-3}
201.25	5.01	6.6×10^{-3}
226.45	11.57	0.0162
239.05	20.80	0.0320
242.20	29.66	0.0504
243.40	40.20	0.0780
243.70[a]	40.86	0.0800

Solubility of water in 4-methylquinoline

$t/°C$	$100\,w_2$ (compiler)	x_2
20.50	15.77	0.5980
47.50	14.51	0.5742
67.50	13.31	0.5496
102.40	11.91	0.5180
147.15	9.67	0.4596
177.90	14.51	0.5742
208.90	15.94	0.6010
225.25	22.20	0.6940
234.90	29.82	0.7715
240.25	39.61	0.8390
242.20	49.69	0.8870
243.70[a]	59.14	0.9200

[a]Upper critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
Two methods were used. Below 80 °C, the titration method was used. A known amount of component (1) in a thermostated vessel with stirrer was titrated with component (2). Next, the vessel was agitated and a permanent turbidity marked the coexistence point. Above 80 °C, the synthetic method was used. The ampoules were filled with mixtures of (1) and (2), sealed and placed in a silicon-oil bath. The samples were first overheated and cooled slowly with observation of the appearance of turbidity and of meniscus.

Source and Purity of Materials:
(1) Midland Tar Distillers or Schuchardt's; purified by distillation and crystallization; purity by glc 99%; b.p. 265.65 °C, m.p. 9.5 °C, $d_4^{25} = 1.6204$.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C at 80–200 °C and ±0.4 °C at 200–230 °C, and ±0.8 °C above 230 °C (by the synthetic method).

2.33. 7-Methylquinoline + water

Components:
(1) 7-Methylquinoline (*m*-Toluquinoline); $C_{10}H_9N$; [612-60-2]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[29]B. Zegalska, Bull. Acad. Pol. Sci., Ser. Sci. Chim. **7**, 357 (1968).

Variables:
Temperature: 20–258.7 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 7-methylquinoline in water

$t/°C$	$100\,w_1$ (compiler)	x_1
20.00	0.20	2.5×10^{-4}
161.35	3.2	4.2×10^{-3}
187.20	4.2	5.5×10^{-3}
212.40	8.4	0.0114
226.60	12.1	0.0170
244.80	19.7	0.0300
256.50	36.3	0.0670
258.50	41.5	0.0820
258.70[a]	46.6	0.0990

Solubility of water in 7-methylquinoline

$t/°C$	$100\,w_2$ (compiler)	x_2
20.00	12.60	0.5340
84.00	10.51	0.4827
148.00	10.52	0.4830
198.20	12.52	0.5322
213.95	16.39	0.6090
235.00	21.88	0.6900
251.00	33.62	0.8010
258.30	49.22	0.8851
258.70[a]	53.39	0.9010

[a]Upper critical solution temperature.

Auxiliary Information

Method/Apparatus/Procedure:
Two methods were used. Below 80 °C, the titration method was used. A known amount of component (1) in a thermostated vessel with stirrer was titrated with component (2). Next, the vessel was agitated and a permanent turbidity marked the coexistence point. Above 80 °C, the synthetic method was used. The ampoules were filled with mixtures of (1) and (2), sealed and placed in a silicon-oil bath. The samples were first overheated and cooled slowly with observation of the appearance of turbidity and of meniscus.

Source and Purity of Materials:
(1) Midland Tar Distillers or Schuchardt's; purified by distillation and crystallization; b.p. 257.71 °C, m.p. 39.9 °C, $d_4^{25} = 1.6169$.
(2) Not specified.

Estimated Error:
Temperature: ± 0.1 °C at 80–200 °C and ± 0.4 °C at 200–230 °C, and ± 0.8 °C above 230 °C (by the synthetic method).

Auxiliary Information

Method/Apparatus/Procedure:
Two methods were used. Below 80 °C, the titration method was used. A known amount of component (1) in a thermostated vessel with stirrer was titrated with component (2). Next, the vessel was agitated and a permanent turbidity marked the coexistence point. Above 80 °C, the synthetic method was used. The ampoules were filled with mixtures of (1) and (2), sealed and placed in a silicon-oil bath. The samples were first overheated and cooled slowly with observation of the appearance of turbidity and of meniscus.

Source and Purity of Materials:
(1) Midland Tar Distillers or Schuchardt's; purified by distillation and crystallization; purity by glc 99.1%; b.p. 247.90 °C, m.p. –26.7 °C, $d_4^{25} = 1.6169$.
(2) Not specified.

Estimated Error:
Temperature: ± 0.1 °C at 80–200 °C and ± 0.4 °C at 200–230 °C, and ± 0.8 °C above 230 °C (by the synthetic method).

2.34. 8-Methylquinoline + water

Components:
(1) 8-Methylquinoline; $C_{10}H_9N$; [611-32-5]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[29] B. Zegalska, Bull. Acad. Pol. Sci., Ser. Sci. Chim. **7**, 357 (1968).

Variables:
Temperature: 20–305.5 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 8-methylquinoline in water

$t/$°C	$100\,w_1$ (compiler)	x_1
20.00	0.9	1.1×10^{-3}
195.60	2.1	2.7×10^{-3}
237.85	5.1	6.7×10^{-3}
264.00	10.1	0.014
296.00	22.9	0.036
303.00	36.0	0.066
305.50	46.9	0.100

Solubility of water in 8-methylquinoline

$t/$°C	$100\,w_2$ (compiler)	x_2
28.50	4.51	0.273
164.50	4.51	0.273
188.00	5.38	0.311
232.05	9.61	0.458
272.00	19.01	0.651
304.10	39.43	0.838
305.50	53.11	0.900

2.35. 5-Butyl-2-methylpyridine + water

Components:
(1) 5-Butyl-2-methylpyridine (3-Butyl-6-methylpyridine); $C_{10}H_{15}N$; [702-16-9]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[20] R. M. Stephenson, J. Chem. Eng. Data **38**, 428 (1993).

Variables:
Temperature: 0−90.0 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of 5-butyl-2-methylpyridine in water

$t/$°C	$100\,w_1$	x_1 (compiler)
0.0	0.42	5.5×10^{-4}
10.0	0.25	3.3×10^{-4}
20.0	0.18	2.4×10^{-4}
30.0	0.19	2.5×10^{-4}
40.0	0.23	3.0×10^{-4}
50.0	0.31	4.1×10^{-4}
60.0	0.21	2.8×10^{-4}
70.0	0.24	3.2×10^{-4}
80.0	0.20	2.6×10^{-4}
90.0	0.17	2.2×10^{-4}

Solubility of water in 5-butyl-2-methylpyridine

$t/$°C	$100\,w_2$	x_2 (compiler)
0.0	7.38	0.378
10.0	7.40	0.378
20.0	6.48	0.345
30.0	5.86	0.322
40.0	5.91	0.324
50.0	4.59	0.268
60.0	4.85	0.280
70.0	5.24	0.296
80.0	4.82	0.278
90.0	4.52	0.265

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.01\%$ and mass fraction $w_2 = 0.10\%$ (standard deviation).

Auxiliary Information

Method/Apparatus/Procedure:
The analytical method was used. Amine (1) and water (2) were brought into equilibrium at a given temperature in a thermostat, and samples of each layer were removed by syringe for analysis. To each sample, a higher boiling organic compound was added as a standard and the ratio of peaks was measured by gas chromatography, using a Gow-Mac Series 500 thermal conductivity GC with a Hewlett-Packard 3390A thermal integrator. Temperature was measured by a calibrated thermometer accurate to 0.1 °C.

Source and Purity of Materials:
(1) Aldrich Chemical Co. or TCI America; purity 99.7 mass%; used as received without further purification.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.
Solubility: mass fraction $w_1 = 0.001\%$ and mass fraction $w_2 = 0.002\%$ (standard deviation).

2.36. *N,N*-Diethylaniline + water

Components:
(1) *N,N*-Diethylaniline;
$C_{10}H_{15}N$; [91-66-7]
(2) Water; H_2O; [7732-18-5]

Original Measurements:
[26]R. M. Stephenson, J. Chem. Eng. Data **38**, 634 (1993).

Variables:
Temperature: 0.0–90.0 °C

Prepared by:
B. Wisniewska-Goclowska

Experimental Values

Solubility of *N,N*-diethylaniline in water

t/°C	$100\,w_1$	x_1 (compiler)
0.0	0.016	1.9×10^{-5}
10.0	0.017	2.0×10^{-5}
60.0	0.021	2.5×10^{-5}

Solubility of water in *N,N*-diethylaniline

t/°C	$100\,w_2$	x_2 (compiler)
0.0	0.028	2.3×10^{-3}
10.0	0.022	1.8×10^{-3}
20.0	0.026	2.2×10^{-3}
30.0	0.037	3.1×10^{-3}
40.0	0.047	3.9×10^{-3}
50.0	0.071	5.9×10^{-3}
60.0	0.073	6.0×10^{-3}
70.0	0.080	6.6×10^{-3}
80.0	0.108	8.9×10^{-3}
90.0	0.120	9.9×10^{-3}

2.37. Diphenylamine + water

Components:
(1) Diphenylamine
(*N*-Phenylaniline,
N-Phenylbenzenamine);
$C_{12}H_{11}N$; [122-39-4]
(2) Water; H_2O; [7732-18-5]

Evaluators:
A. Maczynski, M. Goral, and B. Wisniewska-Goclowska, Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland, January, 2012.

2.37.1. Experimental solubility studies for diphenylamine (1) in water (2)

The experimental solubility data for (1) in (2) have been investigated as listed in Table 37 together with the temperature range.

2.37.2. Critical evaluation of the solubility of diphenylamine (1) in water (2)

The data of Desvergnes[32] and Paul[31] at temperatures 298 K and 302 K are in good agreement and therefore these data were classified as tentative. All the experimental data are listed in Table 38 and shown in Fig. 13.

TABLE 38. Experimental values for solubility of diphenylamine (1) in water (2)

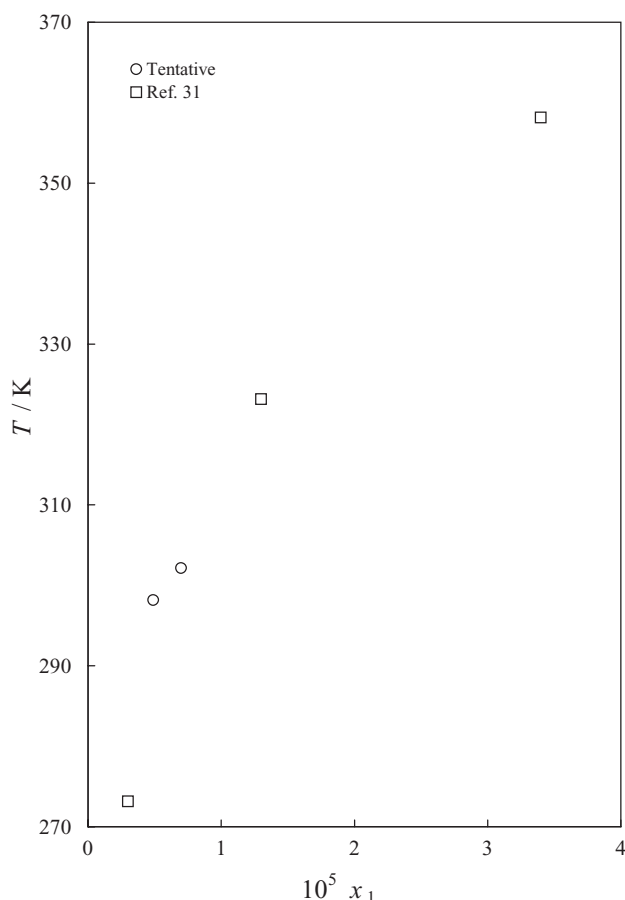| T/K	Experimental values x_1 (T = tentative)
273.2	3×10^{-6} (Ref. 31)
298.2	4.9×10^{-6} (T; Ref. 32)
302.2	7×10^{-6} (T; Ref. 31)
323.2	1.3×10^{-5} (Ref. 31)
358.2	3.4×10^{-5} (Ref. 31)

FIG. 13. Solubility data for diphenylamine (1) in water (2).

Components:	Original Measurements:
(1) Diphenylamine (*N*-Phenylaniline, *N*-Phenylbenzenamine); $C_{12}H_{11}N$; [122-39-4] (2) Water; H_2O; [7732-18-5]	[31]M. A. Paul, J. Am. Chem. Soc. **76**, 3236 (1954).

Variables:	Prepared by:
One temperature: 25 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of diphenylamine in water

$t/°C$	g (1)/l sln	$100 w_1$ (compiler)	x_1 (compiler)
25.0	0.046	4.6×10^{-3}	4.9×10^{-6}

Auxiliary Information

Method/Apparatus/Procedure:
The spectroscopic method was used. Approximately 0.1 g of (1) was introduced into 50-ml glass-stoppered flask containing about 30 ml of (2), and the flask was then agitated continuously for a period of at least 48 h in a thermostat operating at (25.0 ± 0.1) °C. A sample of the solution was then withdrawn through a cotton plug with a transfer pipet and accurately diluted for analysis by means of its absorbance as measured with the spectrophotometer.

Source and Purity of Materials:
(1) Eastman Kodak Co.; recrystallized from mixture of ethanol with water.
(2) Not specified.

Estimated Error:
Temperature: ±0.1 °C.

Components:	Original Measurements:
(1) Diphenylamine (*N*-Phenylaniline, *N*-Phenylbenzenamine); $C_{12}H_{11}N$; [122-39-4] (2) Water; H_2O; [7732-18-5]	[32]M. L. Desvergnes, Ann. Chim. Anal. Chim. Appl. **10**, 253 (1928).

Variables:	Prepared by:
Temperature: 0−85 °C	B. Wisniewska-Goclowska

Experimental Values

Solubility of diphenylamine in water

$t/°C$	$100 w_1$	x_1 (compiler)
0	0.003	3×10^{-6}
28	0.007	7×10^{-6}
50	0.012	1.3×10^{-5}
85	0.032	3.4×10^{-5}

Auxiliary Information

Method/Apparatus/Procedure:
The solubility of (1) in (2) was determined by evaporation of water from the mixture. No more details were reported in the paper.

Source and Purity of Materials:
(1) Source not specified; chemical purity; crystallized from ethanol; m.p. 53 °C.
(2) Twice distilled.

Estimated Error:
Not specified.

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