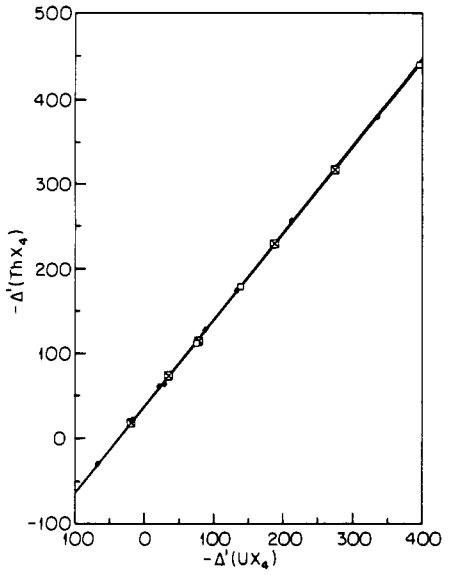

Figure 7. Relationship between $\Delta'(UX_4)$ and $\Delta'(ThX_4)$.

the univalent ions this would have the effect of reducing their a values by a factor of $1.2744a_{Li}$. This is a great help in making extrapolations to fluorides or iodides. A similar result would be obtained for the divalent metals by referencing $\Delta(M)$ values to Be, the element with the largest a value.

Registry No. $CeBr_3$, 14457-87-5; $TbBr_3$, 14456-47-4; $HoBr_3$, 13825-76-8; $TmBr_3$, 14456-51-0; $YbBr_3$, 13759-89-2; $CrBr_3$, 10031-25-1; $HfBr_4$, 13777-22-5; CuI_2, 13767-71-0; UO_2I_2, 13520-82-6; AuI_3, 13453-24-2; ScI_3, 14474-33-0; TbI_3, 13813-40-6; YbI_3, 13813-44-0; TlI_3, 13783-08-9; MnI_2, 7790-33-2; ZrI_4, 13986-26-0; MoI_2, 14055-74-4; MoI_4, 14055-76-6; TeI_4, 7790-48-9; PtI, 13779-77-6; PtI_2, 7790-39-8; PtI_3, 68220-29-1; MoF_2, 20205-60-1; ReI_3, 15622-42-1; WI_5, 13782-91-7; TaI_5, 14693-81-3; HfI_4, 13777-23-6; PaI_5, 17497-66-4; UI_5, 13775-20-7.

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Solubility of Four Amino Acids in Water and of Four Pairs of Amino Acids in Their Water Solutions

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Solubility is measured for L-serine, D-serine, DL-proline, and DL-arginine in water at 25–60 °C. The solubility of an amino acid A in a water solution of amino acid B, and the solubility of B in a solution of A, has been determined in the same temperature range for four pairs of A and B: L-glutamic acid + glycine, L-glutamic acid + L-aspartic acid, L-glutamic acid + L-serine, and L-aspartic acid + L-serine. The eutectic compositions of the water solution of the four pairs of amino acids are reported.

Introduction

Amino acids are valuable chemical substances that are the basic building blocks of all forms of life. For use in industrial processes and scientific laboratories and as food additives, amino acids have to be separated and purified from mixtures that are obtained from protein hydrolysis or an industrial synthetic process. Fractional precipitation and crystallization are suitable for separation of amino acids while other separation methods such as chromatography are also practiced. All separation methods stand to benefit from understanding of the solution and solubility behavior which is basic to the design of separation and purification processes.

The solubility data of some amino acids in water are tabulated in *CRC Handbook of Chemistry and Physics* (*1*) and Fasman's *Handbook of Biochemistry and Molecular Biology* (*2*) while the solubility data of L-threonine, L-cysteine, L-asparagine, L-glutamine, L-lysine, L-arginine, L-histidine, DL-proline, DL-tryptophan, DL-thrionine, DL-cysteine, DL-glutamine, DL-asparagine, DL-lysine, DL-arginine, and DL-histidine in water are still not found. The solubility data of an amino acid in a solution of other amino acids are scarce. Cohn and co-workers (*3*) reported the solubility of asparagine or cystine in solutions of alanine, glycine, or α-aminobutyric acid. Sexton and Dunn (*4*) reported that of glutamic acid or norvaline in glycine solution.

In this work, we experimentally determine the solubility in water for four amino acids and the solubility of each of a pair of amino acids in their solutions for four pairs. We use the dry weight method for the determination.

Dry Weight Method

By the dry weight method, the solubility of a substance is determined by evaporating to dryness a saturated solution of the substance. The weight of the dissolved solute is obtained from the dried sample. The weight of the saturated solution having been determined prior to evaporation of the solution, the amount of solvent is obtained by difference.

The dry weight method has been used for the determination of the solubility of a single amino acid in water (*5*, *6*). In this work the method is adopted for this purpose and is extended to the determination of the solubility of an amino acid A in a mixed solution of amino acids A and B. For the latter purpose, a known unsaturated solution of B is brought in contact with solute A. Upon being saturated with A, the concentration of the total dissolved solutes A and B are determined by evaporating a sample of the solution to dryness. The solubility of A

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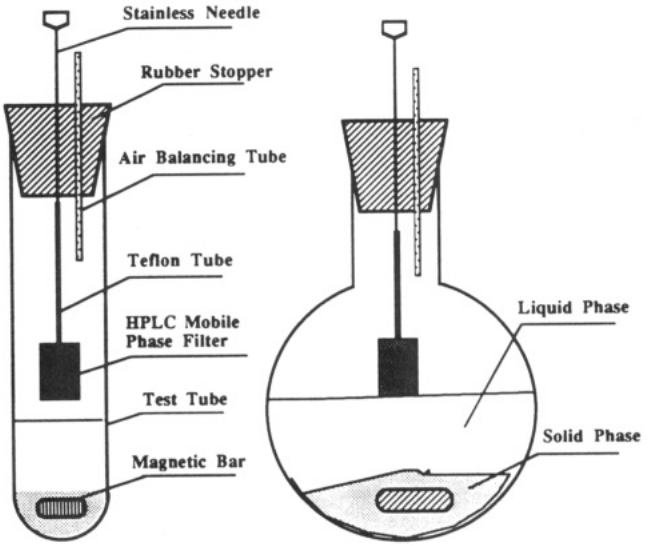

Figure 1. Equilibrium cells for solubility measurement.

is obtained by subtracting the known concentration of B from the total solutes. The method is valid for A and B that do not form a solid solution. The absence of a solid solution has been experimentally observed in this work for the amino acid pairs studied.

By starting an experiment with a concentrated solution of B, we determine the solubility of A in a series of solutions of A and B by successive dilution with water each time after withdrawing a solution sample from the equilibrium cell. By accounting for the size of the sample withdrawn and its concentrations of A and B, the remaining contents of water and B in the equilibrium cell are computed, enabling the experiment to be continued without having to empty the flask and recharge with fresh material upon the completion of a single solubility point.

A detailed description of our dry weight method for measurement of solubility in a solution of two amino acids has been presented by Jin (*7*).

Experimental Apparatus and Procedure

Figure 1 shows the equilibrium cells for solubility measurement. A solid amino acid and a liquid solution are brought in contact by continued stirring in the cell. The small cell made of a culture test tube of 50 mL is suitable for high solubility; the large cell made of a 250-mL flask is for low solubility.

A thermostated water bath keeps the temperature constant to ±0.05 °C. After being stirred overnight, the contents of the flask are allowed to settle. The supernatant liquid is sampled by being withdrawn through a HPLC mobile phase filter with 2-μ pores. The sampled liquid proceeds through a piece of Teflon tubing and a 20-gauge 3-in. long stainless needle into a syringe. The contents of the syringe are emptied into a weighed sample vial. The vial is capped, cooled to ambient temperature, and weighed. Subsequently the vial is uncapped and placed in an oven. The temperature and vacuum of the oven are adjusted for quiescent evaporation without foaming or bumping. The dried vial is again weighed.

A high rate of evaporation is favored by high temperature, but too high a temperature can cause decomposition of the amino acid. For all the amino acids of this work, except glutamic acid, we have found 80 °C to be suitable. Evaporation of glutamic acid solution is accomplished at 60 °C as the acid undergoes a reaction at higher temperatures to form pyrrolidonecarboxylic acid (*8*).

For determining the solubility in solution of two amino acids, our experimental method rests on the assumption that the amino acid crystals do not form a solid solution. To find out if a solid solution is formed, we place crystals of amino acids A and B and water in an equilibrium flask and stir at constant temperature for several days. A saturated liquid sample is withdrawn, and the total concentration of the solution is determined by the dry weight method. An equal weight of water is added and the total concentration of solutes again determined for a sample of the saturated liquid. The procedure is repeated several times.

For the amino acid pairs of this study, the total concentration of the successive liquid samples stays constant till one amino acid becomes depleted.

The constancy of the liquid phase shows the system to be invariant. For a three-component system at a specified temperature and pressure, invariance indicates the coexistence of three phases: one liquid and two solids. The solids are in separate phases, not a solution.

Table I. Comparison of Solubility of Amino Acids of This Work and Literature Sources (g of acid/g of water)

amino acid		solubility				
		25.0 °C	30.0 °C	40.0 °C	50.0 °C	60.0 °C
L-Glu	literature[a]	0.008 64		0.015 10		0.0317
	this work	0.008 61		0.014 64		0.0312
L-Asp	literature[a]	0.005 00		0.008 38		0.0170
	this work	0.004 95		0.008 45		0.0164
L-Ser	literature[b]			0.592 0		
	this work			0.592 0		
DL-Ser	literature[c]	0.050 23		0.078 43		0.1341
	this work	0.050 20		0.077 8		0.1325
L-Ala	literature[a]	0.166 5	0.1757	0.195 7	0.2179	0.2426
	this work	0.165 0	0.1743	0.195 3	0.2165	0.2411
DL-Ala	literature[a]	0.167 2	0.1783	0.202 9	0.2309	0.2627
	this work	0.169 3	0.1803	0.205 5	0.2308	0.2632

[a] Reference 5. [b] Reference 2. [c] Reference 6.

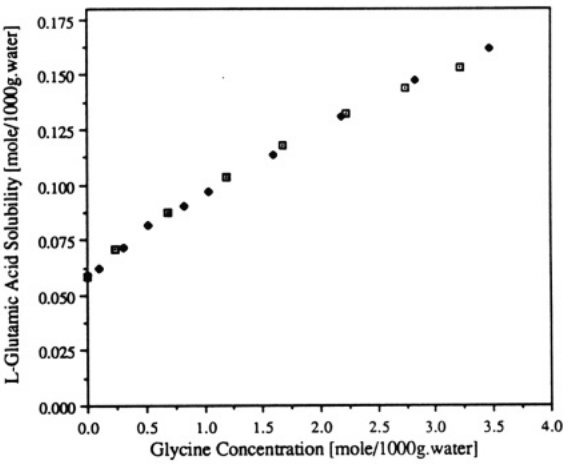

Figure 2. L-Glutamic acid solubility in aqueous glycine solutions at 25 °C: ⊡, this work; ♦, ref 4.

Experimental Uncertainties

To test our apparatus and procedure, we measure the solubility of several amino acids in water and the solubility of L-glutamic acid in aqueous glycine solutions for comparison with literature values. Table I and Figure 2 show the results. For the solubility in pure water, agreement with literature to about 1% is generally obtained although the largest deviation amounted to 3.6% for L-aspartic acid at 60 °C.

A number of causes contribute to experimental uncertainty for amino acid solubility in a ternary solution: (a) impurity of amino acids, (b) incomplete separation of solution from the solid phase, (c) sample loss during drying, and (d) inconsistency between the actual amount of water in the solution phase and the amount of water calculated by mass balance.

Experimental error caused by all sources except for d did not exceed 2% whereas the error introduced by source d alone,

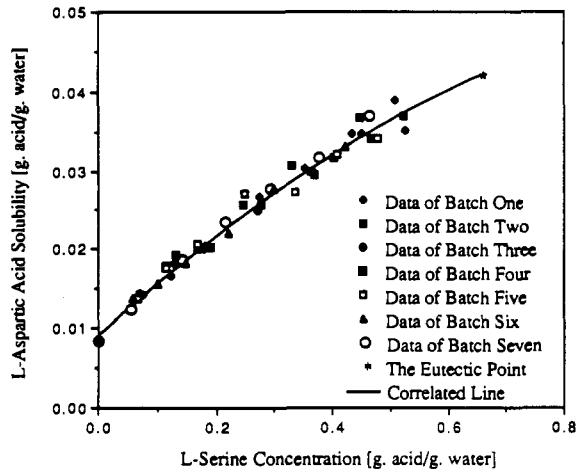

Figure 3. Solubility of L-aspartic acid in L-serine solutions at 40 °C.

Table II. Solubility of Amino Acids in Water (g of acid/g of water)

amino acid	solubility				
	25.0 °C	20.0 °C	40.0 °C	50.0 °C	60.0 °C
L-Ser	0.4217		0.5920		0.7960
D-Ser	0.4214		0.6000		0.8020
DL-Pro	1.319	1.401	1.641	1.967	2.397
DL-Arg	0.1972	0.2407	0.3445	0.4844	0.6709

if not corrected, could be quite large for systems where A has a very low solubility while B is in high concentration (*7*). Water condensing on the upper part of the equilibrium cell away from the solution phase could make the difference. The higher the experimental temperature, the more water could condense on the upper part of the cell. A blank test showed that the water condensed in the cell made of the 50 cm^3 test tube amounted to 0.07, 0.22, and 0.36 g at 25, 40, and 60 °C, respectively (*7*). With account made for the condensation, the experimental uncertainty is estimated to be less than 6%. Figure 2 shows the agreement with literature values for L-glutamic acid solubilities in aqueous glycine solution at 25 °C. The experimental uncertainty for the ternary system is shown in Figure 3. The direct experimental data from seven independent batch measurements and the best smoothed values based on the experimental data are both presented.

The data reported in this work are the average values of three independent determinations for single amino acid solubilities in water, and are the smoothest data set from batch experiments for amino acid solubilities in solutions of another amino acid.

Experimental Material

All the amino acids are used as purchased from Aldrich Chemical Co., Inc., with an indicated purity of 98% for L-aspartic acid and glycine, and 99% for the rest of the amino acids. The water used in the experiment is double distilled and deionized.

Solubility of Single Amino Acids

Table II presents the solubility in water of L-serine, D-serine, DL-proline, and DL-arginine determined in this work. The solubilities of L-serine and D-serine are found to be the same within the uncertainties of the experiment; the difference between the two serves to indicate the accuracy of our results. The solubility of L-serine was reported at 0.0, 10.0, 20.0, 30.0, and 40.0 °C (*2*). The 40.0 °C value is shown in Table I for comparison with our measurement. In Table II, we extend the solubility to 60.0 °C.

Table III. Solubility of L-Glutamic Acid and Glycine in Their Solutions

temperature/ °C	L-Glu solubility/ (g of acid/g of water)		Gly solubility/ (g of acid/g of water)	
	solb	Gly concn	solb	L-Glu concn
25.0	0.008 61	0.0000		
	0.010 38	0.0177		
	0.012 85	0.0515		
	0.015 22	0.0889		
	0.017 29	0.1261		
	0.019 45	0.1668		
	0.021 20	0.2056		
	0.022 59	0.2411		
40.0	0.014 64	0.0000		
	0.017 81	0.0271		
	0.022 78	0.0779		
	0.026 76	0.1218		
	0.029 88	0.1696		
	0.034 69	0.2226		
	0.039 60	0.2768		
	0.041 23	0.3260		
60.0	0.031 23	0.0000	0.4526	0.000 00
	0.037 69	0.0364	0.4543	0.010 89
	0.044 68	0.1043	0.4547	0.017 68
	0.052 90	0.1649	0.4561	0.026 99
	0.059 87	0.2323	0.4563	0.037 51
	0.064 43	0.3021	0.4590	0.047 46
	0.069 85	0.3730	0.4617	0.058 27
	0.071 42	0.4414	0.4668	0.070 50
	0.072 50[a]	0.4690[a]	0.4690[a]	0.072 50[a]

[a] Solubility value at the eutectic point.

Table IV. Solubility of L-Glutamic Acid and L-Aspartic Acid in Their Solutions

temperature/ °C	L-Glu solubility/ (g of acid/g of water)		L-Asp solubility/ (g of acid/g of water)	
	solb	L-Asp concn	solb	L-Glu concn
25.0	0.008 61	0.000 00	0.004 95	0.000 00
	0.008 61	0.000 64	0.005 02	0.000 68
	0.008 69	0.001 21	0.005 06	0.001 70
	0.008 80	0.001 90	0.005 14	0.003 22
	0.008 90	0.002 75	0.005 23	0.004 84
	0.009 08	0.003 77	0.005 30	0.006 57
	0.009 17	0.004 67	0.005 39	0.008 23
	0.009 20[a]	0.005 51[a]	0.005 51[a]	0.009 20[a]
40.0	0.014 64	0.000 00	0.008 45	0.000 00
	0.014 73	0.001 03	0.008 46	0.001 24
	0.001 86	0.002 16	0.008 61	0.002 79
	0.015 03	0.003 37	0.008 68	0.005 06
	0.015 15	0.004 90	0.008 87	0.008 47
	0.015 38	0.006 55	0.009 11	0.011 51
	0.015 47	0.008 50	0.009 29	0.014 27
	0.015 62[a]	0.009 38[a]	0.009 38[a]	0.015 62[a]
60.0	0.031 23	0.000 00	0.016 40	0.000 00
	0.031 77	0.002 59	0.016 76	0.004 90
	0.032 21	0.004 35	0.016 97	0.008 20
	0.032 70	0.007 62	0.017 31	0.013 69
	0.032 75	0.010 86	0.017 65	0.019 22
	0.033 25	0.015 30	0.018 32	0.026 76
	0.033 32[a]	0.018 70[a]	0.018 70[a]	0.033 32[a]

[a] Solubility value at the eutectic point.

Solubility in Mixed Solutions

The solubility of an amino acid A in a water solution of amino acid B, and the solubility of B in a solution of A, has been determined for four pairs of A and B: L-glutamic acid + glycine, L-glutamic acid + L-aspartic acid, L-glutamic acid + L-serine, and L-aspartic acid + L-serine.

Tables III–VI present the solubility data. The solubility of an amino acid is enhanced by the presence of another amino acid for all four pairs studied here. The enhancement effect is brought out in Figures 4–7. The enhancement effect is the strongest in L-aspartic acid + L-serine solutions shown in Figure

Table V. Solubility of L-Glutamic Acid and L-Serine in Their Solutions

temperature/ °C	L-Glu solubility/ (g of acid/g of water)		L-Ser solubility/ (g of acid/g of water)	
	solb	L-Ser concn	solb	L-Glu concn
25.0	0.008 61	0.0000	0.4217	0.000 00
	0.009 38	0.0349	0.4291	0.003 30
	0.013 30	0.0997	0.4312	0.005 40
	0.017 14	0.1531	0.4335	0.008 65
	0.021 77	0.2090	0.4355	0.012 48
	0.025 06	0.2690	0.4417	0.018 46
	0.029 83	0.3313	0.4453	0.024 20
	0.035 69	0.3919	0.4482	0.030 12
	0.039 50[a]	0.4601[a]	0.4554	0.035 64
			0.4601[a]	0.039 50[a]
40.0	0.014 64	0.0000	0.5920	0.000 00
	0.017 67	0.0532	0.6057	0.007 85
	0.024 04	0.1434	0.6097	0.011 35
	0.027 84	0.2144	0.6130	0.018 24
	0.033 24	0.2916	0.6220	0.027 86
	0.037 62	0.3748	0.6274	0.041 82
	0.043 30	0.4624	0.6341[a]	0.050 85[a]
	0.047 54	0.5401		
	0.050 85[a]	0.6341[a]		
60.0	0.031 23	0.0000	0.7960	0.000 00
	0.033 66	0.0521	0.8104	0.012 36
	0.036 08	0.1159	0.8125	0.020 05
	0.042 06	0.2198	0.8140	0.030 03
	0.046 53	0.3154	0.8162	0.038 63
	0.052 83	0.4292	0.8170	0.047 79
	0.061 25	0.5553	0.8192	0.057 82
	0.065 25	0.6655	0.8214	0.068 56
	0.071 01[a]	0.8224[a]	0.8224[4]a	0.071 01[a]

[a] Solubility value at the eutectic point.

Table VI. Solubility of L-Aspartic Acid and L-Serine in Their Solutions

temperature/ °C	L-Asp solubility/ (g of acid/g of water)		L-Ser solubility/ (g of acid/g of water)	
	solb	L-Ser concn	solb	L-Asp concn
25.0	0.004 95	0.0000	0.4217	0.000 00
	0.005 79	0.0128	0.4260	0.001 40
	0.008 17	0.0433	0.4268	0.001 93
	0.011 80	0.1052	0.4278	0.003 13
	0.015 88	0.1614	0.4302	0.005 40
	0.019 50	0.2200	0.4341	0.007 28
	0.023 08	0.2831	0.4385	0.009 67
	0.032 00[a]	0.4614[a]	0.4392	0.012 08
			0.4422	0.014 29
			0.4614[a]	0.032 00[a]
40.0	0.008 45	0.0000	0.5920	0.000 00
	0.013 16	0.0547	0.6056	0.005 66
	0.018 59	0.1413	0.6092	0.008 20
	0.023 44	0.2152	0.6152	0.012 03
	0.027 66	0.2935	0.6216	0.017 11
	0.031 76	0.3763	0.6280	0.024 72
	0.036 20	0.4639	0.6347	0.032 90
	0.041 24[a]	0.6425[a]	0.6425[a]	0.041 24[a]
60.0	0.016 40	0.0000	0.7960	0.000 00
	0.023 22	0.0579	0.8095	0.011 88
	0.029 43	0.1239	0.8177	0.019 84
	0.036 51	0.2216	0.8213	0.030 07
	0.042 46	0.3103	0.8231	0.036 34
	0.047 46	0.4163	0.8313	0.045 45
	0.056 24	0.5345	0.8342	0.054 23
	0.063 08	0.6587	0.8356	0.064 65
	0.070 92	0.7679	0.8440[a]	0.075 88
	0.075 88[a]	0.8440[a]		

[a] Solubility value at the eutectic point.

7; it is the lowest in L-aspartic acid + L-glutamic acid solutions shown in Figure 5.

A eutectic point is obtained when the liquid solution is saturated with both amino acids. The eutectic is an invariant point at a given temperature. The eutectic point of the amino acid

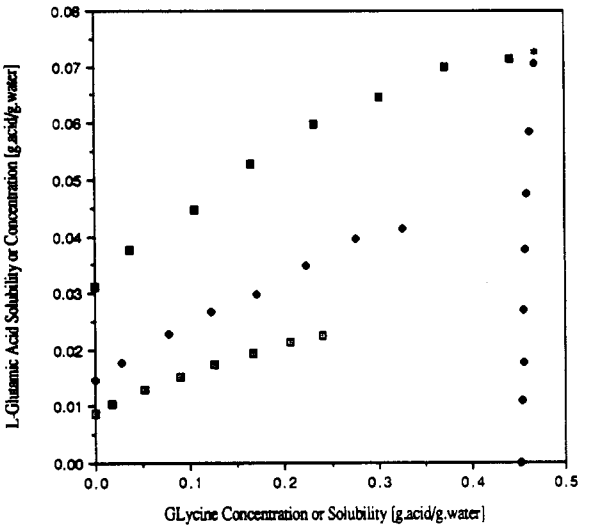

Figure 4. Solubility of L-glutamic acid and glycine in their solutions: ♦, L-Glu solubility at 40 °C; □, L-Glu solubility at 60 °C; ◊, Gly solubility at 60 °C; ⊡, L-Glu solubility at 25 °C.

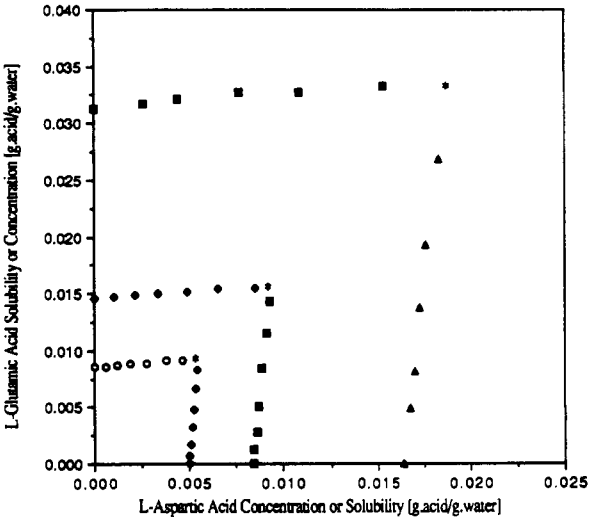

Figure 5. Solubility of L-glutamic acid and L-aspartic acid in their solutions: O, L-Glu solubility at 25 °C; ♦, L-Glu solubility at 40 °C; ■, L-Glu solubility at 60 °C; ◊, L-Asp solubility at 25 °C; □, L-Asp solubility at 40 °C; ▲, L-Asp solubility at 60 °C.

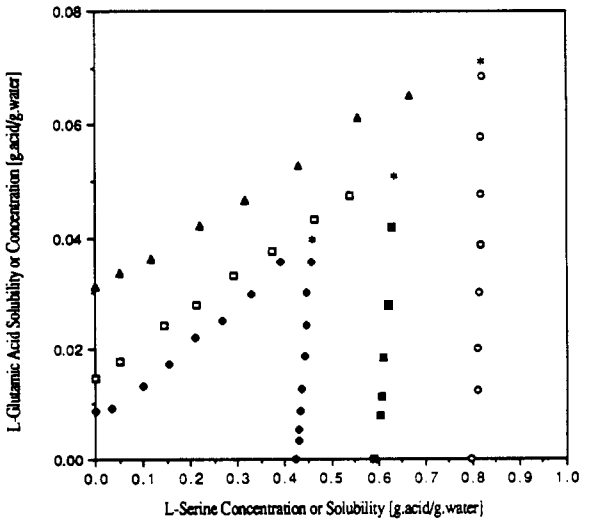

Figure 6. Solubility of L-glutamic acid and L-serine in their solutions: ♦, L-Glu solubility at 25 °C; ◊, L-Ser solubility at 25 °C; □, L-Glu solubility at 40 °C; ■, L-Ser solubility at 40 °C; ▲, L-Glu solubility at 60 °C; O, L-Ser solubility at 60 °C.

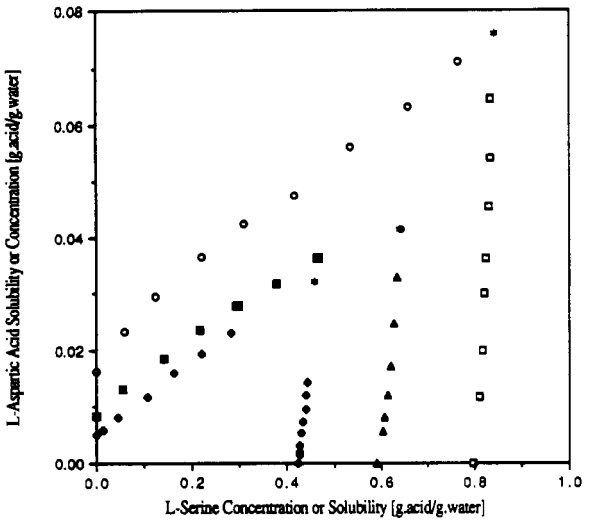

Figure 7. Solubility of L-aspartic acid and L-serine in their solutions: ♦, L-Asp solubility at 25 °C; ◇, L-Ser solubility at 25 °C; ■, L-Asp solubility at 40 °C; ▲, L-Ser solubility at 40 °C; O, L-Asp solubility at 60 °C; □, L-Ser solubility at 60 °C.

Table VII. Total Liquid-Phase Concentration of Amino Acids at Eutectic Point

system	total solubility/ (g of acid/g of water) 25.0 °C	40.0 °C	60.0 °C
L-Glu + Gly			0.5415
L-Glu + L-Asp	0.1471	0.2500	0.5202
L-Glu + L-Ser	0.4996	0.6850	0.8934
L-Asp + L-Ser	0.4934	0.6837	0.9200

mixtures of this work has been cited in the preceding discussion in support of the existence of pure amino acid solids in preference to a solid solution. Experimentally we determine by the dry weight method the total solute concentration of the eutectic liquid solution that is saturated with both amino acids. Table VII presents the results for the four pairs of amino acids of this work.

By graphically extrapolating the experimental solubility of the two amino acids (Tables IV–VII) to intersect the measured total solubility within the uncertainty range, we determine the solubility of each amino acid in the eutectic solution and the precise total solubility reported in Table VII. The eutectic compositions indicated with an asterisk are included in Tables III–VI and Figures 4–6.

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Registry No. L-Serine, 56-45-1; D-serine, 312-84-5; DL-proline, 609-36-9; DL-arginine, 7200-25-1; L-glutamic acid, 56-86-0; glycine, 56-40-6; L-aspartic acid, 56-84-8.

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Vapor–Liquid Equilibria of 2,3-Dimethylbutane + Methanol or Ethanol at 101.3 kPa

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Isobaric vapor–liquid equilibria were measured for 2,3-dimethylbutane + methanol or ethanol at 101.3 kPa. The experimental data were correlated with the nonrandom two-liquid (NRTL) and Wilson equations.

Introduction

In the present study, vapor–liquid equilibria (VLE) were measured for two binary systems, 2,3-dimethylbutane + methanol or + ethanol, at 101.3 kPa pressure using a vapor and liquid recirculate still. For 2,3-dimethylbutane + methanol, two sets of data are available in the literature (*1*, *2*), but those data are not consistent according to Gmehling and Onken (*3*). New reliable data seem, therefore, to be required for this system. No VLE data have been reported previously for 2,3-dimethylbutane + ethanol.

Experimental Section

Materials. 2,3-Dimethylbutane, methanol, and ethanol were special grade reagents. Methanol and ethanol were used after their minute water content was removed with molecular sieves 3A. A gas-chromatographic analysis on all three materials indicated that each had a purity of at least 99.9 mol %. Table I compares some of the measured properties with literature data.

Procedure. The equilibrium still used to obtain VLE data was a modified Rogalski–Malanowski (*4*) still with a provision for

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