INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

ANALYTICAL CHEMISTRY DIVISION
COMMISSION ON SOLUBILITY DATA

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## SOLUBILITY DATA SERIES

Volume 49

# ESTERS WITH WATER
# PART II: ESTERS 7-C TO 32-C

# SOLUBILITY DATA SERIES

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY
IUPAC Secretariat: Bank Court Chambers, 2-3 Pound Way,
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A complete list of volumes published in the Solubility Data Series will be found on p. 250.

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Volume 49

# ESTERS WITH WATER
# PART II: ESTERS 7-C TO 32-C

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# CONTENTS

# FOREWORD

*If the knowledge is*
*undigested or simply wrong,*
*more is not better.*

The Solubility Data Series is a project of Commission V.8 (Solubility Data) of the International Union of Pure and Applied Chemistry (IUPAC). The project had its origins in 1973, when the Analytical Chemistry Division of IUPAC set up a Subcommission on Solubility Data under the chairmanship of the late Prof. A. S. Kertes. When publication of the *Solubility Data Series* began in 1979, the Committee became a full commission of IUPAC, again under the chairmanship of Prof. Kertes, who also became Editor-in-Chief of the Series. The Series has as its goal the preparation of a comprehensive and critical compilation of data on solubilities in all physical systems, including gases, liquids and solids.

The motivation for the Series arose from the realization that, while solubility data are of importance in a wide range of fields in science and technology, the existing data had not been summarized in a form that was at the same time comprehensive and complete. Existing compilations of solubility data indeed existed, but they contained many errors, were in general uncritical, and were seriously out-of-date.

It was also realized that a new series of compilations of data gave educational opportunities, in that careful compilations of existing data could be used to demonstrate what constitutes data of high and lasting quality. As well, if the data were summarized in a sufficiently complete form, any individual could prepare his or her own evaluation, independently of the published evaluation. Thus, a special format was established for each volume, consisting of individual data sheets for each separate publication, and critical evaluations for each separate system, provided sufficient data from different sources were available for comparison. The compilations and, especially, the evaluation were to be prepared by active scientists who were either involved in producing new data, or were interested in using data of high quality. With minor modifications in format, this strategy has continued throughout the Series.

In the standard arrangement of each volume, the Critical Evaluation gives the following information:

(i) A text which discusses the numerical solubility information which has been abstracted from the primary sources in the form of compilation sheets. The text concerns primarily the quality of the data, after consideration of the purity of the materials and their characterization, the experimental method used, the uncertainties in the experimental values, the reproducibility, the agreement with accepted test values, and finally, the fitting of the data to suitable functions, along with statistical tests of the fitted data.

(ii) A set of recommended data, whenever possible, including weighted averages and estimated standard deviations. If applicable, one or more smoothing equations which have been computed or verified by the evaluator are also given.

(iii) A graphical plot of the recommended data, in the form of phase diagrams where appropriate.

The Compilation part consists of data sheets which summarize the experimental data from the primary literature. Here much effort is put into obtaining complete coverage; many good data have appeared in publications from the late nineteenth and early twentieth centuries, or in obscure journals. Data of demonstrably low precision are not compiled, but are mentioned in the Critical Evaluation. Similarly, graphical data, given the uncertainty of accurate conversion to numerical values, are compiled only where no better data are available. The documentation of data of low precision can serve to alert researchers to areas where more work is needed.

A typical data sheet contains the following information:

(i) list of components: names, formulas, Chemical Abstracts Registry Numbers;
(ii) primary source of the data;
(iii) experimental variables;
(iv) compiler's name;
(v) experimental values as they appear in the primary source, in modern units with explanations if appropriate;
(vi) experimental methods used;
(vii) apparatus and procédure used;
(viii) source and purity of materials used;
(ix) estimated error, either from the primary source or estimated by the compiler;
(x) references relevant to the generation of the data cited in the primary source.

Each volume also contains a general introduction to the particular type of system, such as solubility of gases, of solids in liquids, etc., which contains a discussion of the nomenclature used, the principle of accurate determination of solubilities, and related thermodynamic principles. This general introduction is followed by a specific introduction to the subject matter of the volume itself.

The Series embodies a new approach to the presentation of numerical data, and the details continue to be influenced strongly by the perceived needs of prospective users. The approach used will, it is hoped, encourage attention to the quality of new published work, as authors become more aware that their work will attain permanence only if it meets the standards set out in these volumes. If the Series succeeds in this respect, even partially, the Solubility Data Commission will have justified the labour expended by many scientists throughout the world in its production.

January, 1989

J. W. Lorimer,
London, Canada

# PREFACE

This volume, one of a pair in the Solubility Data Series devoted to esters in water, includes solubility data for binary systems containing an ester and water published up through the end of 1988. The first volume contains the esters having two through six carbon atoms with water and the second volume contains esters having seven or more carbon atoms with water. With only rare exceptions, the compiled data and evaluations involve measurements made at constant pressure over rather restricted ranges of temperature. The limited ranges of pressure and temperature involved are to be expected in view of the liquid-liquid condition prescribed for the systems.

For the most part, the data presented show general patterns of behavior expected for the topic systems. While rather extensive data have been reported for many of the systems under consideration, no effort was made to attempt any detailed correlations of behavior or descriptions of trends beyond those presented by the evaluator in the evaluation sections.

The critical evaluations were all prepared by one author and an introductory section has been included to elaborate the philosophy and methodology followed in the evaluations. No attempts were made to extract thermodynamic parameters such as activity coefficients, enthalpies of solution, and the like from the reported data although much of the published work is sufficiently precise for such activities.

Every attempt was made to include all published data for the subject systems through the end of 1988. The editors would be grateful for any information concerning material which was overlooked in the preparation of this work.

The editors would like to express their appreciation to all those who have provided assistance in the volume preparation, especially to fellow members of the IUPAC Commission on Solubility Data and to those who have served as reviewers of this work.

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November 1991.

# LIQUID-LIQUID SOLUBILITY: EVALUATION PHILOSOPHY AND METHODOLOGY

G.T. HEFTER

## INTRODUCTION

Because of the differing ways in which it is possible to critically evaluate published solubility data it is worthwhile to present a coherent statement on the philosophy and procedures which have been used in this volume. Although this is a personal statement concerning the present volume, many of the ideas have been developed by myself as an evaluator for previous liquid-liquid volumes in the Solubility Data Series [1-3] and are shared, to some degree, by other evaluators.

## EVALUATION PHILOSOPHY

The critical evaluation of data is, at least in part, a subjective process. Some physical scientists are uncomfortable with this notion but the history of science shows us it is undeniable. Because some subjective judgements are inevitably involved, it is vital that the evaluator's procedure should be as visible as possible. In this way, if the user does not agree with the evaluator's judgement or arguments or if new data become available it is possible, with a minimum of effort, to derive a new set of preferred or "best" values. For this reason the Critical Evaluations in this and those previous volumes to which I have contributed [1-3] have been, in essence, written like research papers with enough information provided for users to repeat or change the evaluation should they so desire.

The starting point for each Critical Evaluation is that the data compilation is complete, i.e., the data sheets include all the available data. This is an important premise because the addition of extra data may change the evaluation considerably. This is especially true of liquid-liquid systems since very few have been adequately studied over a wide temperature range and disagreements often exist between independent investigations.

In general, a "democracy of observers" is followed. That is, the data of all investigators are given equal weight irrespective of their reputation or location. This is justifiable in scientific terms because experience shows that real "errors" in liquid-liquid solubility data are much greater than individual authors' reported precisions. Thus, the benzene-water system, which is relatively simple and probably the most widely studied of all liquid-liquid systems [2], has solubilities which are known to an accuracy of about ± 2% (relative). The same estimated error is obtained regardless of whether a simple average of the available data is taken or whether a complex weighting procedure is used. This suggests that, at the present stage of development of liquid-liquid solubility determinations, equal weighting for all data is the most appropriate course of action. The exceptions to this rule are discussed below.

## REJECTION OF DATA

The statement that all data are given equal weight must be qualified: some data are assigned zero weight, i.e., they are rejected. Data rejection is potentially dangerous, especially when few data are available. Nevertheless, since a critical evaluation is required, it has to be considered. The most important reasons for rejecting data are as follows.

1. The data disagree "significantly" from most other studies (especially if these are numerous). Significance is subjective but a criterion of

$$| s_{obs} - s_{av} | > 3\sigma_n$$

where $s_{obs}$ is the reported solubility datum being considered for rejection

$s_{av}$ is the arithmetic average (mean) of all other studies at the same temperature and pressure

$\sigma_n$ is the standard deviation from the mean of these studies

has been generally, but not rigidly, applied [1-3].

It should be noted that this procedure can be invoked only once. That is, having rejected some points in a particular data set it is not statistically valid to recalculate a new value of $\sigma_n$ and then reject further points. Strictly speaking this means that outlying data should be included in the original calculation of $\sigma_n$. However, the amount of available data does not always make this approach realistic and common sense must be applied.

2. Approximate data (one significant figure) are rejected when a reasonable amount of more precise data is available.

3. Data which are grossly inconsistent with chemical systematics or thermodynamic expectations are also rejected.

4. In keeping with the practice adopted in previous liquid-liquid solubility values, data originally presented in graphical form or in volume ratio units are also excluded. In the latter case, data are retained if appropriate density data are readily available to permit conversion to mass units. As density measurements are usually far more reliable than solubility data, the choice of density values is usually not critical.

It is appropriate to add here that data for a particular system coming from the same authors or the same laboratories have generally not been regarded as independent measurements and have therefore been subsumed into a single result (at each temperature).

## THE EVALUATION FORMAT

The Critical Evaluations in this volume follow the format evolved in previous liquid-liquid solubility volumes [1-3]. In essence each Evaluation is divided into three parts. In the first part Table 1 lists all the available publications containing quantitative solubility data for the particular system. Broad experimental details such as temperature range, type of solubility measured ((1) in (2), (2) in (1) or mutual) and the method used are also included in Table 1. After mention of any other special features such as critical points, or any special types of investigation, further discussion is usually divided into two parts: the solubility of the organic in the aqueous phase, and that of water in the organic-rich phase. Since these two sections are handled in the same way they will be discussed together.

After listing rejected data, anomalies and caveats, all the available data (usually expressed as g/100g sln) are collected into Tables (usually Tables 2 & 3). To facilitate comparisons, data are interpolated (if necessary) to specific temperatures at 5K intervals near 298K, then 10 or 20K intervals. These temperatures are exact, i.e. 298.15, 303.15 ... K but for convenience are written 298, 303, ... K in the text. The nature of most liquid-liquid solubility data makes graphical interpolation the most appropriate approach [4]. Interpolated values are always designated with an asterisk (*). Very occasionally data are extrapolated but only over a short range (<5K) and only if the solubility curve is not changing sharply.

All the retained values at each temperature are then averaged and the mean and $\sigma_n$ recorded in the Tables. Few of the systems in this volume have sufficient data to permit meaningful statistical analysis. The value of $\sigma_n$ is therefore included only as a convenient measure of the spread of the experimental data.

## "BEST" VALUES

The word "best", almost always in inverted commas (quotation marks), is used throughout the Critical Evaluations in this and previous liquid-liquid solubility volumes [1-3] in a specific way: to mean "best available estimate". It follows from what has been said above that "best" values are simply the arithmetic average (mean) of all the accepted data, including those obtained by interpolation and excluding rejected data. "Best" values are given in both g/100g sln and mole fraction units (x).

## RECOMMENDED VALUES

Solubilities are recommended only if two or more independent studies (including rejected data) agree to within ± 5%(relative), i.e.,

$$| 100\sigma_n/s_{av} | \le 5$$

and there are no other obvious problems. Recommended data are designated by (*R*) immediately following the numerical value.

For sparingly soluble systems this criterion may be too harsh and

$$| 100\sigma_n/s_{av} | \le 10 \text{ or } 20$$

may be more appropriate [3]. Very few high molecular weight esters have been sufficiently well characterized to enable even these less restrictive criteria to be applied.

Data which are not "Recommended" are regarded as "Tentative". The likely accuracy of these data varies from system to system and from temperature to temperature but may be inferred from

1. $\sigma_n$
2. $100\sigma_n/s_{av}$
3. n (the number of independent measurements).

Occasionally there are definite reasons to suspect the available data are **not** reliable. Such data are labelled "Doubtful" and the detailed reasons for this classification given. Considerable care should be exercised in the use of such data.

Note that the mole fraction "best" solubilities in each table should be regarded as having the same status ("Recommended", "Tentative" or "Doubtful") and the same (relative) percentage uncertainties (where given) as the corresponding mass % solubilities.

## GRAPHICS

In systems for which a reasonable amount of data exists, selected results are plotted graphically over the studied temperature range. Appropriate comments on the plots are made in the figure caption or in the text.

## FITTING EQUATIONS

The use of fitting equations to smooth solubility data as a function of temperature is controversial and a topic in its own right which will not be discussed here. As in previous liquid-liquid solubility volumes [1-3], fitting equations are not generally given in the printed version of this volume. There are several reasons for this.

1. There is no single equation form which can be used for all systems.
2. The data for some systems are too scattered or poorly defined for realistic curve fitting.
3. Without special care empirical fitting equations may give misleading results (spurious extrema, etc.) when interpolating and can almost never be reliably used for extrapolation.

For certain systems where reliable data appear evident, fitting equations have been provided. Such equations come directly from the original works or from the efforts of the compilers in the course of their examination of reported data and preparation of smoothed value tables.

Users who wish to derive their own fitting equations are advised to do so with caution. It is suggested that the "best" values be used for this purpose, but it should be noted that this may create anomalies because of the way in which the "best" values are derived (see above). Where fitting equations are given in this volume they should be used only over the stated range.

**THERMODYNAMIC CONSISTENCY**

Using standard relationships from thermodynamics it can be shown that:

$$\Delta_{sln}G = -RT \ln a_{sat}$$

$$\frac{\partial(\Delta_{sln}G/T)}{\partial T} = -\frac{\Delta_{sln}H}{RT^2}$$

and $$\frac{\partial^2(\Delta_{sln}G/T)}{\partial T^2} = \frac{\partial(\Delta_{sln}H)}{\partial T} = \Delta_{sln}C_p$$

where $\Delta_{sln}G$, $\Delta_{sln}H$ and $\Delta_{sln}C_p$ are respectively the Gibbs energy, enthalpy and heat capacity (at constant pressure) changes corresponding to the dissolution of the solute in the solvent and $a_{sat}$ is the activity of the saturated solute in the solvent. If the solute is only sparingly soluble the thermodynamic parameters may be equated with their standard state values, and it should be noted that $\Delta_{sln}C_p$ may itself be temperature dependent.

Both $\Delta_{sln}H$ and $\Delta_{sln}C_p$, at least in principle, can be measured directly by separate calorimetric procedures [5,6] and therefore can serve as an independent check on the temperature dependence of published solubility data. This approach has been used with significant effect in evaluating hydrocarbon solubilities in water [2-3]. However, very few calorimetric data have been reported for ester-water systems and so this approach has not been utilized in the present volume.

**REFERENCES**

1. G.T. Hefter, in A.F.M. Barton, Ed., *Solubility Data Series, Vol. 15, Alcohols with Water* (Pergamon Press, Oxford, 1984).

2. G.T. Hefter, in D.G. Shaw, Ed., *Solubility Data Series, Vol. 37, Hydrocarbons with Water and Seawater, Part I: Hydrocarbons C5 to C7* (Pergamon Press, Oxford, 1989).

3. G.T. Hefter, in D.G. Shaw, Ed., *Solubility Data Series, Vol. 38, Hydrocarbons with Water and Seawater, Part II: Hydrocarbons C8 to C36* (Pergamon Press, Oxford, 1989).

4. G.T. Hefter, unpublished paper presented at 1st Intl. *Symp. Soly. Phenom.*, London, Canada, August, 1984.

5. S.O. Nilsson and I. Wadso, *J. Chem. Thermodyn.* 1984, 16, 317.

6. P. Picker, P.A. Leduc, P.R. Philip and J.E. Desnoyers, *J. Chem. Thermodyn.* 7, 3, 641.

**COMPONENTS:**

(1) 2-Propenoic acid, 2-methyl-, 2-cyanoethyl ester
*(2-cyanoethyl methacrylate)*;
$C_7H_9NO_2$; [4513-53-5]

(2) Water; $H_2O$; [7732-18-5]

**ORIGINAL MEASUREMENTS:**

Frolov, A.F.; Maiorova, N.M.; Erykov, V.G.; Korshukov, M.A.

*Zh. Prikl. Khim.* 1977, *50*, 1318-21.

**VARIABLES:**

$T/K = 293$

**PREPARED BY:**

Z. Maczynska

**EXPERIMENTAL VALUES:**

The solubility of 2-methyl-2-propenoic acid 2-cyanoethyl ester in water at 20°C was reported to be 1.7 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.0022.

The solubility of water in 2-methyl-2-propenoic acid 2-cyanoethyl ester at 20°C was reported to be 2.9 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.19.

**AUXILIARY INFORMATION**

**METHOD/APPARATUS/PROCEDURE:**

The method was not specified.

**SOURCE AND PURITY OF MATERIALS:**

(1) Source not specified; b.p. 240.0°C, $d_4^{20}$ 1.0400, $n_D^{20}$ 1.4460.

(2) Not specified.

**ESTIMATED ERROR:**

Not specified.

**REFERENCES:**

COMPONENTS:

(1) Formic acid cyclohexyl ester (*cyclohexyl formate*); $C_7H_{12}O_2$; [4351-54-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 363

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of formic acid cyclohexyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | - | 98.90 | - | 0.9267 |
| 9.3 | 1.70 | 98.58 | 0.00242 | 0.9070 |
| 19.0 | 0.65 | 98.44 | 0.00092 | 0.8987 |
| 29.6 | 0.39 | 98.63 | 0.00055 | 0.9101 |
| 39.5 | - | 98.61 | - | 0.9088 |
| 49.9 | - | 98.36 | - | 0.8939 |
| 69.9 | - | 98.10 | - | 0.8789 |
| 80.0 | - | 97.87 | - | 0.8659 |
| 90.3 | - | 97.50 | - | 0.8457 |
| std. dev. | 0.01 | 0.02 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 95%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) 2-Propenoic acid butyl ester (*butyl acrylate*); $C_7H_{12}O_2$; [141-32-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Logutov, V.I.; Danov, S.M.; Chubarov, G.A.; Tomashchuk, V.I.

*Zh. Prikl. Khim.* 1983, *56(1)*, 214-6.

VARIABLES:

$T$/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 2-Propenoic acid butyl ester in water at 20°C was reported to be 0.18 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 2.6 x $10^{-4}$.

The solubility of water in 2-Propenoic acid butyl ester at 20°C was reported to be 0.65 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.044.

The densities of the water-rich phase and the ester rich phase were reported to be $d_{20}^{20}$ 0.9981 and $d_{20}^{20}$ 0.8987 respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. After mixing and separating, the two samples were analysed. The water in organic phase was determined by titration with Karl Fischer reagent. The 2-Propenoic acid butyl ester was measured by the gas-liquid chromatography method with an inert standard or by the bromate titration method. Both procedures gave similar results.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; used as received; b.p. 147.4°C, $d_4^{20}$ 0.9010, $n_D^{20}$ 1.4200.

(2) Twice distilled.

ESTIMATED ERROR:

Temp. ±0.5°C.
Soly. ±(2-3)% (relative) (2) in organic phase,
±3% (relative) (1) in water phase.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Propanedioic acid diethyl ester (*diethyl malonate*); $C_7H_{12}O_4$; [105-53-3]<br>(2) Water; $H_2O$; [7732-18-5] | Sobotka, H.; Kahn, J.<br>*J. Am. Chem. Soc.* 1931, *53*, 2935-8. |
| **VARIABLES:** | **PREPARED BY:** |
| $T$/K = 293 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of propanedioic acid diethyl ester in water at 20°C was reported to be 2.08 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 2.04 g(1)/100g sln and 0.0023.

Density of water $d_4^{20}$ = 0.9982 (ref 1) was used in the calculation.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets. | (1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 1.0550, $n_D^{20}$ 1.4144.<br>(2) Distilled. |
| | **ESTIMATED ERROR:** |
| | Not specified. |
| | **REFERENCES:** |
| | 1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973. |

COMPONENTS:

(1) Pentanedioic acid dimethyl ester (*dimethyl glutarate*); $C_7H_{12}O_4$; [1119-40-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of pentanedioic acid dimethyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 7.1 | 97.70 | 0.0085 | 0.8269 |
| 9.4 | 6.3 | 97.37 | 0.0075 | 0.8063 |
| 20.0 | 6.1 | 97.72 | 0.0072 | 0.8282 |
| 29.6 | 5.9 | 96.44 | 0.0070 | 0.7529 |
| 39.5 | 5.8 | 95.88 | 0.0069 | 0.7236 |
| 49.8 | 5.8 | 95.36 | 0.0069 | 0.6980 |
| 60.3 | 6.1 | 94.69 | 0.0072 | 0.6673 |
| 70.2 | 6.4 | 92.89 | 0.0076 | 0.5950 |
| 80.0 | 6.6 | 92.86 | 0.0079 | 0.5939 |
| 90.5 | 8.1 | 91.77 | 0.0098 | 0.5564 |
| std. dev. | 0.10 | 0.03 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 98%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Acetic acid 1-methyl-1-butyl ester (*1-methylbutyl acetate*); $C_7H_{14}O_2$; [626-38-0]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A., Australia
January, 1989

CRITICAL EVALUATION:

Quantitative solubility data for the acetic acid 1-methyl-1-butyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Acetic acid 1-methyl-1-butyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Park and Hopkins (ref 1) | 298 | (1) in (2) | unspecified |
| Doolittle (ref 2) | 293 | mutual | unspecified |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation.

As can be seen from Table 1 no data have been reported under comparable conditions and so no Critical Evaluation is possible. Furthermore, the datum of Park and Hopkins (ref 1) is reported only in v/v units and would normally be excluded from consideration. However, assuming a density of 0.86 g/mL for (1) (ref 2) and a density of 1.00 g/mL for the saturated solution of (1) in (2), the solubility reported by Park and Hopkins (ref 1) at 298 K is equivalent to 0.23 g(1)/100 g sln which is in good agreement with Doolittle's value of 0.19 g(1)/100 g sln at 293 K (ref 1). Doolittle (ref 1) also reports a value of 0.88 g(2)/100 g sln for the solubility of water in the ester. The interested user is referred to the relevant Data Sheets for additional information.

REFERENCES

1. Park, J. G.; Hopkins, M. B. *Ind. Eng. Chem.* 1930, *22*, 826-30.
2. Doolittle, A. K. *Ind. Eng. Chem.* 1935, *27*, 1169-79.

COMPONENTS:

(1) Acetic acid 1-methyl-1-butyl ester (*1-methylbutyl acetate*); $C_7H_{14}O_2$; [626-38-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Park, J.G.; Hopkins, M.B.

*Ind. Eng. Chem.* 1930, *22*, 826-30.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 1-methyl-1-butyl ester in water at 25°C was reported to be 0.2 mL(1)/100mL(2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial samples; used as received; 85-88% of ester, b.p. range 128-134°C.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid 1-methyl-1-butyl ester (*1-methylbutyl acetate*); $C_7H_{14}O_2$; [626-38-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T$/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 1-methyl-1-butyl ester in water at 20°C was reported to be 0.19 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $2.6 \times 10^{-4}$.

The solubility of water in acetic acid 1-methyl-1-butyl ester at 20°C was reported to be 0.88 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.060.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 92%, b.p. range 121-144°C, $d_4^{20}$ 0.862.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
January, 1989

CRITICAL EVALUATION:

Quantitative solubility data for the acetic acid 3-methyl-1-butyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Acetic acid 3-methyl-1-butyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Bancroft (ref 1) | 293 | mutual | titration |
| Othmer *et al.* (ref 2) | 297 | mutual | unspecified |
| Narasimhan *et al.* (ref 3) | 303 | mutual | titration, refractometric |
| Iguchi and Fuse (ref 4) | 298 | mutual | titration |
| Ramanarao *et al.* (ref 5) | 303 | mutual | turbidometric |
| Andreeva *et al.* (ref 6) | 367 | mutual | titration |
| Rao *et al.* (ref 7) | 303 | mutual | titration |
| Skrzecz (ref 8) | 288-359 | mutual | synthetic, Karl Fischer |
| Stephenson and Stuart (ref 9) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

## 1. SOLUBILITY OF ACETIC ACID 3-METHYL-1-BUTYL ESTER (1) IN WATER (2)

All the available data for the solubility of acetic acid 3-methyl-1-butyl ester (1) in water (2) are summaized in Table 2 with the following exceptions.

The value of Bancroft (ref 1) reported in v/v units is excluded from consideration. The data reported by Iguchi and Fuse (ref 4), Ramanarao *et al.* (ref 5) and Andreeva *et al.*, (ref 6) are substantially higher (> $3\sigma_n$) than other values and have been rejected. The datum of Othmer *et al.* (ref 2), which is considerably lower than other studies is also rejected.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

The remaining data, largely due to Skrzecz (ref 8) and Stephenson and Stuart (ref 9) are in excellent agreement, enabling the average "Best" values to be Recommended over a wide temperature range. Selected data are plotted in Figure 1.

TABLE 2: Recommended (*R*) and Tentative Solubilities of Acetic acid 3-methyl-1-butyl ester (1) in Water (2)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values ($\pm\sigma_n$)[a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 0.34 (ref 9) | 0.34 | 4.7 |
| 283 | 0.260* (ref 9) | 0.26 | 3.6 |
| 293 | 0.218* (ref 8), 0.220* (ref 9) | 0.219 ± 0.001 (*R*) | 3.03 |
| 298 | 0.207* (ref 8), 0.208* (ref 9) | 0.208 ± 0.001 (*R*) | 2.88 |
| 303 | 0.22 (ref 3), 0.19 (ref 7),<br>0.198* (ref 8), 0.196* (ref 9) | 0.201 ± 0.011 (*R*) | 2.78 |
| 313 | 0.185* (ref 8), 0.182* (ref 9) | 0.184 ± 0.002 (*R*) | 2.55 |
| 323 | 0.178* (ref 8), 0.174* (ref 9) | 0.176 ± 0.002 (*R*) | 2.44 |
| 333 | 0.178* (ref 8), 0.174* (ref 9) | 0.176 ± 0.002 (*R*) | 2.44 |
| 343 | 0.185* (ref 8), 0.179* (ref 9) | 0.182 ± 0.003 (*R*) | 2.52 |
| 353 | 0.199* (ref 8), 0.190* (ref 9) | 0.195 ± 0.005 (*R*) | 2.70 |
| 363 | 0.219* (ref 8), 0.203 (ref 8) | 0.211 ± 0.008 (*R*) | 2.92 |

[a] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance. Mole fraction solubilities ($x_1$) have the same status and (relative) percentage uncertainties as the mass % solubilities.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A.,<br>Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

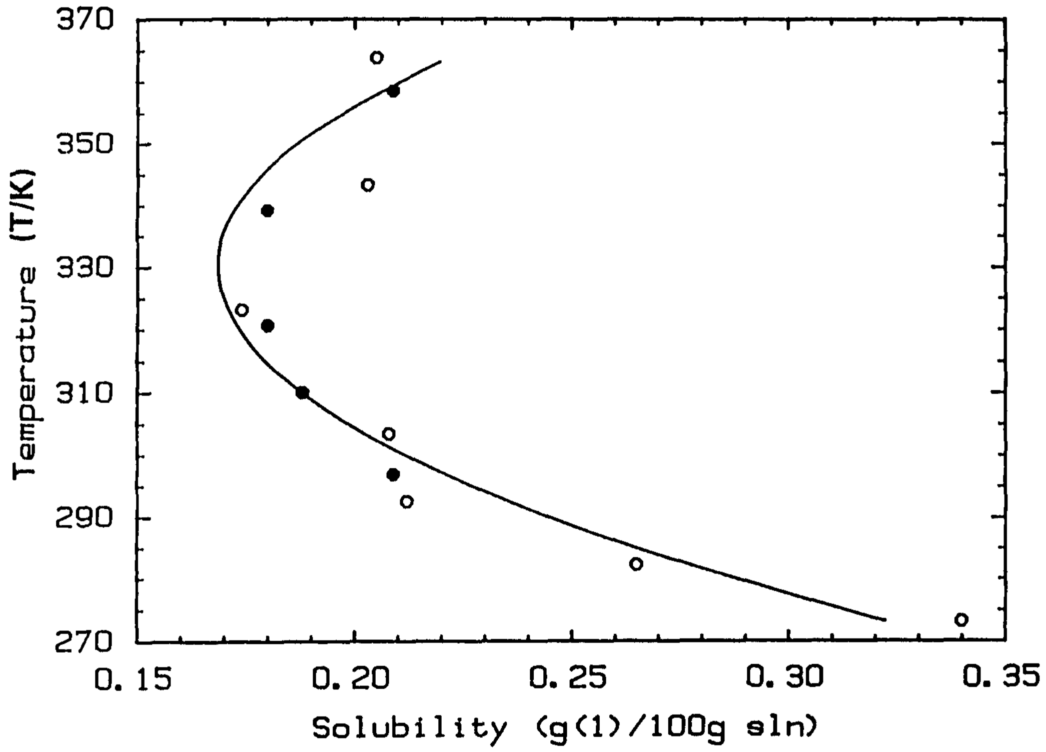

FIGURE 1. Selected data for the solubility of acetic acid 3-methyl-1-butyl ester (1) in water (2): ref 8 (●); ref 9 (○). Solid line is a least square polynomial fitted to the "Best" values from Table 2.

## 2. SOLUBILITY OF WATER (2) IN ACETIC ACID 3-METHYL-1-BUTYL ESTER (1)

All the available data for the solubility of water (2) in acetic acid 3-methyl-1-butyl ester (1) are summarized in Table 3 with the following exceptions.

The datum of Bancroft (ref 1) reported in v/v units has been excluded from consideration. The data reported by Othmer *et al.* (ref 2) and Andreeva *et al.* (ref 6) are significantly higher than other studies and have been rejected. The data of Iguchi and Fuse (ref 4) and Rao *et al.* (ref 7) are significantly lower than other studies and are also rejected.

The remaining data, mainly due to Skrzecz (ref 8) and Stephenson and Stuart (ref 9), are in general in good agreement although they differ significantly at $T$ > 343 K. Selected data are plotted in Figure 2.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

TABLE 3: Recommended (*R*) and Tentative Solubilities of Water (2) in Acetic acid 3-methyl-1-butyl ester (1)

| $T$/K | Solubilities | | | |
|---|---|---|---|---|
| | Reported values | "Best" values $(\pm\sigma_n)^a$ | | |
| | g(2)/100g sln | g(2)/100g sln | | $10^2x_2$ |
| 273 | 0.56 (ref 9) | 0.56 | | 3.9 |
| 283 | $0.67^*$ (ref 9) | 0.67 | | 4.7 |
| 293 | $0.67^*$ (ref 8), $0.78^*$ (ref 9) | 0.73 ± 0.06 | | 5.1 |
| 298 | $0.82^*$ (ref 8), $0.82^*$ (ref 9) | 0.82 | (*R*) | 5.6 |
| 303 | $0.58^b$ (ref 3), $0.545^b$ (ref 5)<br>$0.83^*$ (ref 8), $0.87^*$ (ref 9) | 0.85 ± 0.02 | (*R*) | 5.8 |
| 313 | $0.85^*$ (ref 8), $0.96^*$ (ref 9) | 0.90 ± 0.06 | (*R*) | 6.2 |
| 323 | $0.96^*$ (ref 8), $1.05^*$ (ref 9) | 1.00 ± 0.05 | (*R*) | 6.8 |
| 333 | $1.11^*$ (ref 8), $1.13^*$ (ref 9) | 1.12 ± 0.01 | (*R*) | 7.6 |
| 343 | $1.32^*$ (ref 8), $1.21^*$ (ref 9) | 1.26 ± 0.06 | (*R*) | 8.5 |
| 353 | $1.62^*$ (ref 8), $1.28^*$ (ref 9) | 1.5 ± 0.2 | | 10 |
| 363 | $2.03^*$ (ref 8), $1.33^*$ (ref 9) | 1.7 ± 0.4 | | 17 |

[a] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance. Mole fraction solubilities ($x_2$) have the same status and (relative) percentage uncertainties as the mass % solubilities.

[b] Not included in calculation of "Best" value.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

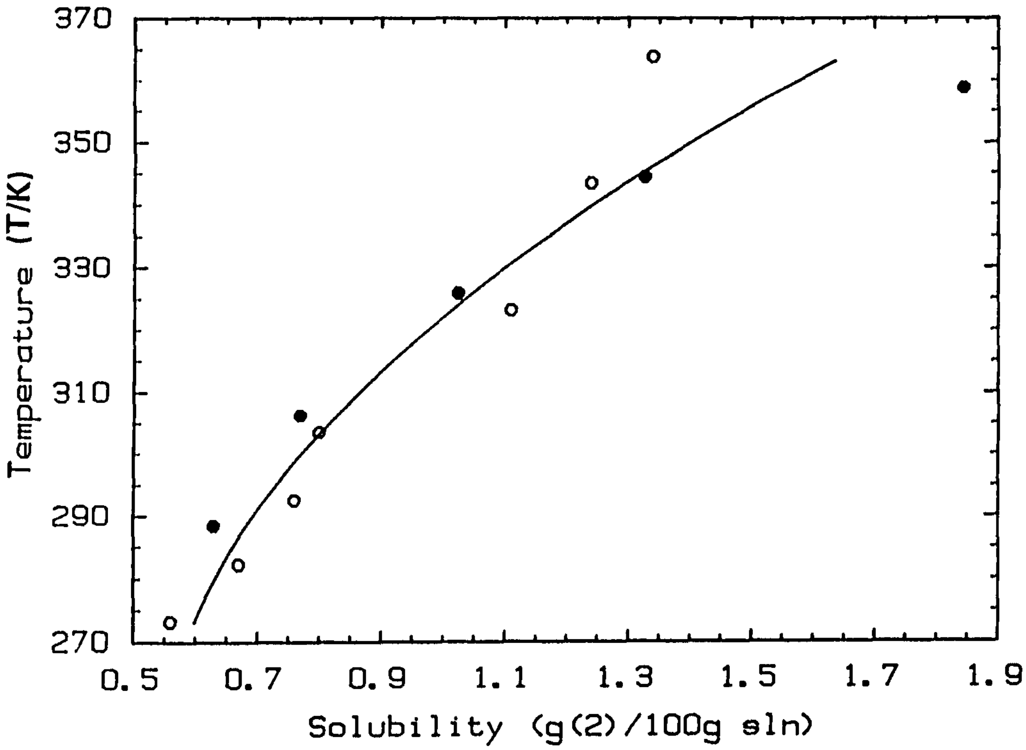

FIGURE 2. Selected data for the solubility of water (2) in acetic acid 3-methyl-1-butyl ester (1): ref 8 (●); ref 9 (○). Solid line is a least square polynomial fitted to the "Best" values from Table 3.

REFERENCES

1. Bancroft, W. D. *Phys. Rev.* 1895, *3*, 114-36.

2. Othmer, D. F.; White, R. E.; Trueger, E. *Ind. Eng. Chem.* 1941, *33*, 1240-8; *ibid.* 1513.

3. Narasimhan, K. S.; Reddy, C. C.; Chari, K. S. *J. Chem. Eng. Data* 1962, *7*, 457-60.

4. Iguchi, A.; Fuse, K. *Kagaku Kogaku* 1971, *35*, 1035-7.

5. Ramanarao, M. V.; Husain, A.; Chari, K. S. *Indian J. Technol.* 1964, *2*, 252-4.

6. Andreeva, N. G.; Komarova, L. F.; Garber, Yu. N. *Zh. Prikl. Khim.* 1978, *51*, 2031-6.

7. Rao, D. S.; Rao, K. V.; Prasad, A. R.; Chiranjivi, C. *J. Chem. Eng. Data* 1979, *24*, 241-4.

(continued next page)

COMPONENTS:

(1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A., Australia
January, 1989

CRITICAL EVALUATION: (continued)

8. Skrzecz, A. *Pol. J. Chem.* 1981, *55*, 1177-80; see also Skrzecz, A. *Thesis*, *I. Ch. F. PAN*, Warszawa, 1979.

9. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

ACKNOWLEDGEMENT

The Evaluator thanks Dr. Brian Clare for the graphics.

| | |
|---|---|
| COMPONENTS:<br>(1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]<br>(2) Water; $H_2O$; [7732-18-5] | ORIGINAL MEASUREMENTS:<br>Bancroft, W.D.<br>*Phys. Rev.* 1895, *3*, 114-36. |
| VARIABLES:<br>$T/K = 293$ | PREPARED BY:<br>A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of acetic acid 3-methyl-1-butyl ester in water at 20°C was reported to be 0.02 mL(1)/10mL(2).

The solubility of water in acetic acid 3-methyl-1-butyl ester at 20°C was reported to be 0.12 mL(2)/10mL(1).

AUXILIARY INFORMATION

| | |
|---|---|
| METHOD/APPARATUS/PROCEDURE:<br>The titration method was used. 10 mL of solvent in a test tube was titrated with the second component until the solution became cloudy. | SOURCE AND PURITY OF MATERIALS:<br>(1) Laboratory source; dried over $CaCl_2$, distilled.<br>(2) Not specified. |
| | ESTIMATED ERROR:<br>Soly. ±0.01 mL. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]<br>(2) Water; $H_2O$; [7732-18-5] | Othmer, D.F.; White, R.E.; Trueger, E.<br>*Ind. Eng. Chem.* 1941, *33*, 1240-8, 1513. |
| VARIABLES: | PREPARED BY: |
| $T$/K = 297 | Z. Maczynska |

EXPERIMENTAL VALUES:

The solubility of acetic acid 3-methyl-1-butyl ester in water at 23.5°C was reported to be 0.16 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $2.2 \times 10^{-4}$.

The solubility of water in acetic acid 3-methyl-1-butyl ester at 23.5°C was reported to be 2.8 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.172.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Nothing was specified in the paper. | (1) Not specified.<br>(2) Not specified. |
| | ESTIMATED ERROR: |
| | Temp. ±0.5°C (mean of reported range),<br>Soly. about ±0.04 g(1)/100g sln and ±2.1 g(2)/100g sln (compiler). |
| | REFERENCES: |

COMPONENTS:

(1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Narasimhan, K.S.; Reddy, C.C.; Chari, K.S.

*J. Chem. Eng. Data* 1962, *7*, 457-60.

VARIABLES:

$T$/K = 303

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 3-methyl-1-butyl ester in water at 30°C was reported to be 0.22 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $3.1 \times 10^{-4}$.

The solubility of water in acetic acid 3-methyl-1-butyl ester at 30°C was reported to be 0.58 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.040.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration and the analytical methods described by Narasimhan, Reddy and Chari (ref 1) were used. In the titration method, a known weight of mixture was taken in 100mL stoppered conical flask and kept in a thermostat for 1/2 h. which was found to be sufficient for equilibrium. Then, the selected component was added dropwise until a permanent turbidity appeared in the solution.
For the analytical procedure, the mixture was stirred for 1 h. in a thermostated equilibrium apparatus. After separation the refractive indexes of both layers were measured and the corresponding compositions were obtained graphically from a previously drawn plot relating the refractive index and composition. The data were reported together with the ternary system acetic acid 3-methyl-1-butyl ester-water-phenol.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; twice fractionated; purity 100%, b.p. 140.1-142.1°C, $d_4^{24}$ 0.8670, $n_D^{20}$ 1.4005.

(2) Distilled.

ESTIMATED ERROR:

Temp. ±0.1°C by (ref 1).
Soly. about ±0.02 g(1)/100g sln and ±0.18 g(2)/100g sln (compiler).

REFERENCES:

1. Narasimhan, K.S.; Reddy, C.C.; Chari, K.S. *J. Chem. Eng. Data* 1962, *7*, 340.

COMPONENTS:

(1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Iguchi, A.; Fuse, K.

*Kagaku Kogaku* 1971, *35(9)*, 1035-7.

VARIABLES:

$T/K = 298$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 3-methyl-1-butyl ester in water at 25°C was reported to be 0.43 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $6.0 \times 10^{-4}$.

The solubility of water in acetic acid 3-methyl-1-butyl ester at 25°C was reported to be 0.32 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.0227.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The samples were titrated with the second component to obtain turbidity. The method and data were reported together with the ternary system acetic acid 3-methyl-1-butyl ester-water-acetic acid. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial grade of high purity, used as received.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.1°C.
Soly. about ±0.2 g(1)/100g sln and ±0.4 g(2)/100g sln (compiler).

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]<br>(2) Water; $H_2O$; [7732-18-5] | Ramanarao, M.V.; Husain, A.; Chari, K.S.<br>*Indian J. Technol.* 1964, *2*, 252-4. |
| **VARIABLES:** | **PREPARED BY:** |
| $T$/K = 303 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of acetic acid 3-methyl-1-butyl ester in water at 30°C was reported to be 0.2500 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $3.467 \times 10^{-4}$.

The solubility of water in acetic acid 3-methyl-1-butyl ester at 30°C was reported to be 0.5445 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.03806.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The turbidity end point method described by Narasimhan, Reddy and Chari (ref 1) was used. Only the method for the ternary system was described. A known weight of mixture was placed into a 100 mL stoppered conical flask and kept in a thermostat for 1/2 h. which was found to be sufficient for equilibrium. Then, the selected component was added dropwise until a permanent turbidity appeared in the solution.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; twice distilled; b.p. 142.1°C.

(2) Distilled.

ESTIMATED ERROR:

Temp. ±0.1°C (authors).
Soly. about ±0.05 g(1)/100g sln and ±0.2 g(2)/100g sln (compiler).

REFERENCES:

1. Narasimhan, K.S.; Reddy, C.C.; Chari, K.S. *J. Chem. Eng. Data* 1962, *7*, 340.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]<br>(2) Water; $H_2O$; [7732-18-5] | Andreeva, N.G.; Komarova, L.F.; Garber, Yu.N.<br>*Zh. Prikl. Khim.* 1978, *51*, 2031-6. |
| **VARIABLES:** | **PREPARED BY:** |
| *T*/K = 367 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of acetic acid 3-methyl-1-butyl ester in water at 94.0°C[a] was reported to be $x_1$ = 0.011. The corresponding mass per cent value calculated by the compiler is 7.4 g(1)/100g sln.

The solubility of water in acetic acid 3-methyl-1-butyl ester at 94.0°C[a] was reported to be $x_2$ = 0.179. The corresponding mass per cent value calculated by the compiler is 2.93 g(2)/100g sln.

[a] Boiling temperature at atmospheric pressure.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method of Mozzhukhin, Tyurikov and Mitropolskaya (ref 1) was used. The data were reported together with the ternary system acetic acid 3-methyl-1-butyl ester-water-acetic acid butyl ester (*isopentyl acetate-water-butyl acetate*). No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Soly. about ±7.2 g(1)/100g sln and ±0.8 g(2)/100g sln (compiler).

REFERENCES:

1. Mozzhukhin, A.S.; Tyurikov, I.D.; Mitropolskaya, V.A. in *Fiz. Khim. Osn. Rektifikatsii*, Moskva, 1970.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]<br>(2) Water; $H_2O$; [7732-18-5] | Rao, D.S.; Rao, K.V.; Prasad, A.R.; Chiranjivi, C.<br>*J. Chem. Eng. Data* 1979, *24*, 241-4. |
| VARIABLES: | PREPARED BY: |
| $T$/K = 303 | Z. Maczynska |

EXPERIMENTAL VALUES:

The solubility of acetic acid 3-methyl-1-butyl ester in water at 30°C was reported to be 0.19 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $2.6 \times 10^{-4}$.

The solubility of water in acetic acid 3-methyl-1-butyl ester at 30°C was reported to be 0.15 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.011.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. An accurately weighed amount of one component was placed in a conical flask, the second component was titrated into the flask from a burette until the solution became turbid, and the amount titrated was recorded. The conical flask was kept in a constant-temperature bath. The data were reported together with the ternary system acetic acid 3-methyl-1-butyl ester-water-ethanenitrile (*isopentyl acetate-water-acetonitrile*).

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, reagent grade; used as received.

(2) Distilled; free from $CO_2$.

ESTIMATED ERROR:

Temp. ±0.1°C (authors).
Soly. about ±0.01 g(1)/100g sln and ±0.6 g(2)/100g sln (compiler).

REFERENCES:

COMPONENTS:

(1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Skrzecz, A.

*Pol. J. Chem.* 1981, 55, 1177-80.

Skrzecz, A.

*Thesis*, Inst. Phys. Chem., Pol. Acad. Sci., Warszawa, 1979.

VARIABLES:

$T$/K = 288 - 359

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid 3-methyl-1-butyl ester and water

| $T$/K | $x_1$ | | g(1)/100g sln | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 288.4[a] | - | 0.9562 | - | 99.370 |
| 296.9 | 0.00029 | - | 0.209 | - |
| 306.3 | - | 0.9470 | - | 99.231 |
| 310.1 | 0.00026 | - | 0.188 | - |
| 320.7 | 0.00025 | - | 0.180 | - |
| 325.9 | - | 0.9304 | - | 98.975 |
| 339.3 | 0.00025 | - | 0.180 | - |
| 344.4 | - | 0.9114 | - | 98.673 |

[a] By the Karl Fischer method.

(continued next page)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The synthetic method of Alexejew and the analytical method were used. An ampoule with the solution of fixed concentration was placed in a glass tube connected with a thermostat filled completely with distilled water. During the measurements the temperature of the bath was changed continuously and the appearance and disappearance of turbidity within the ampoule was observed visually. For the analytical method, The amount of water in the saturated organic phase was determined using a Karl Fischer titration procedure. The amount of water in the pure ester was taken into account.

SOURCE AND PURITY OF MATERIALS:

(1) Prolabo-Rhone Poulene, pure grade; distilled; purity 99.97% by glc, 0.08 wt% water by the Karl Fischer method.

(2) Distilled.

ESTIMATED ERROR:

Temp. ±(0.2-0.6)°C.
Soly. see above.

REFERENCES:

COMPONENTS:

(1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Skrzecz, A.

*Pol. J. Chem.* 1981, *55*, 1177-80.

Skrzecz, A.

*Thesis*, Inst. Phys. Chem., Pol. Acad. Sci., Warszawa, 1979.

EXPERIMENTAL VALUES: (continued)

Mutual solubility of acetic acid 3-methyl-1-butyl ester and water

| $T$/K | $x_1$ | | g(1)/100g sln | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 345.5 | 0.00026 | - | 0.188 | - |
| 358.6 | 0.00029 | - | 0.209 | - |
| 358.8 | - | 0.8804 | - | 98.155 |

Author's smoothing equations:

$x_1$ = $3.680\text{x}10^{-4} - 4.37\text{x}10^{-6}\ (T/K - 273.15) + 3.98\text{x}10^{-8}\ (T/K - 273.15)^2$

st.dev. = $1.82 \times 10^{-6}$ (2)-rich phase

$x_2$ = $0.038754 + 2.150\text{x}10^{-4}\ (T/K - 273.15) + 6.82\text{x}10^{-6}\ (T/K - 273.15)^2$

st. dev. = $6.68 \times 10^{-4}$ (1)-rich phase

COMPONENTS:

(1) Acetic acid 3-methyl-1-butyl ester (*isopentyl acetate*); $C_7H_{14}O_2$; [123-92-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid 3-methyl-1-butyl ester and water

| $t/°C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.340 | 99.44 | 0.000472 | 0.9609 |
| 9.1 | 0.265 | 99.33 | 0.000367 | 0.9535 |
| 19.4 | 0.212 | 99.24 | 0.000294 | 0.9475 |
| 30.3 | 0.208 | 99.20 | 0.000288 | 0.9449 |
| 39.7 | 0.184 | 99.15 | 0.000255 | 0.9416 |
| 50.0 | 0.174 | 98.89 | 0.000241 | 0.9250 |
| 60.1 | 0.152 | 98.84 | 0.000210 | 0.9218 |
| 70.2 | 0.203 | 98.76 | 0.000281 | 0.9168 |
| 80.3 | 0.182 | 98.80 | 0.000252 | 0.9193 |
| 90.7 | 0.205 | 98.66 | 0.000284 | 0.9106 |
| std. dev. | 0.002 | 0.01 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Acetic acid pentyl ester *(pentyl acetate)*; $C_7H_{14}O_2$; [628-63-7]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical
and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
January, 1989

CRITICAL EVALUATION:

Quantitative solubility data for the acetic acid pentyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Acetic acid pentyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Hemptinne (ref 1) | 298 | (1) in (2) | analytical |
| Park and Hopkins (ref 2) | 298 | (1) in (2) | unspecified |
| Doolittle (ref 3) | 293 | mutual | unspecified |
| Rao and Rao (ref 4) | 303 | mutual | titration |
| Venkataratnam *et al.* (ref 5) | 303 | mutual | titration |
| Rao and Rao (ref 6) | 303 | mutual | titration |
| Krupatkin and Shcherbakova (ref 7) | 298 | mutual | titration |
| Skrzecz (ref 8) | 286-365 | mutual | synthetic, Karl Fischer |
| Richon and Viallard (ref 9) | 298 | mutual | calorimetric, refractometric |
| Stephenson and Stuart (ref 10) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

## 1. SOLUBILITY OF ACETIC ACID PENTYL ESTER (1) IN WATER (2)

All the available data for the solubility of acetic acid pentyl ester (1) in water (2) are summarized in Table 2 with the following exclusions. The datum of Park and Hopkins (ref 2) reported in v/v units has been excluded from consideration. The datum of Krupatkin and Shcherbakova (ref 7) and the approximate values of Rao *et al.* (ref 4-6) appear to be high and have been rejected. Selected data are plotted in Figure 1.

(continued next page)

COMPONENTS:

(1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical
and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
January, 1989

CRITICAL EVALUATION: (continued)

TABLE 2: Recommended (*R*) and Tentative Solubilities of Acetic acid pentyl ester (1) in Water (2)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values ($\pm\sigma_n$)[a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 0.29 (ref 10) | 0.29 | 4.0 |
| 283 | 0.224* (ref 8), 0.25* (ref 10) | 0.24 ± 0.01 (*R*) | 3.3 |
| 293 | 0.195* (ref 8), 0.22* (ref 10) | 0.21 ± 0.01 (*R*) | 2.9 |
| 298 | 0.162* (ref 1), 0.183* (ref 8),<br>0.178*[b] (ref 9), 0.19* (ref 10) | 0.178 ± 0.010 (*R*) | 2.46 |
| 303 | 0.172* (ref 8), 0.18* (ref 10) | 0.176 ± 0.004 (*R*) | 2.44 |
| 313 | 0.157* (ref 8), 0.15* (ref 10) | 0.154 ± 0.004 (*R*) | 2.13 |
| 323 | 0.150* (ref 8), 0.10 (ref 10) | 0.13 ± 0.02 | 1.8 |
| 333 | 0.151* (ref 8), 0.10* (ref 10) | 0.13 ± 0.02 | 1.8 |
| 343 | 0.161* (ref 8), 0.17* (ref 10) | 0.166 ± 0.005 (*R*) | 2.30 |
| 353 | 0.181* (ref 8), 0.17* (ref 10) | 0.176 ± 0.006 (*R*) | 2.44 |
| 363 | 0.211* (ref 8) | 0.21 | 2.9 |

[a] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance. Mole fraction solubilities ($x_1$) have the same status and (relative) percentage uncertainties as the mass % solubilities.

[b] Average of two independent results reported by the original authors.

## 2. SOLUBILITY OF WATER (2) IN ACETIC ACID PENTYL ESTER (1)

All the available data for the solubility of water (2) in acetic acid pentyl ester (1) are summarized in Table 3 with the following exceptions.

The approximate values of Rao *et al.* (ref 4-6) are widely scattered and do not add any extra information to the more precise studies (ref 8-10) available and so have been rejected. The datum of Krupatkin and Shcherbakova (ref 7) at 298 K is significantly higher than all other studies (ref 8-10) and has also been rejected. Selected data are plotted in Figure 2.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

Although the remaining data (ref 8-10) are in reasonable agreement over a wide temperature range the differences are generally too large to permit many values to be Recommended without further studies.

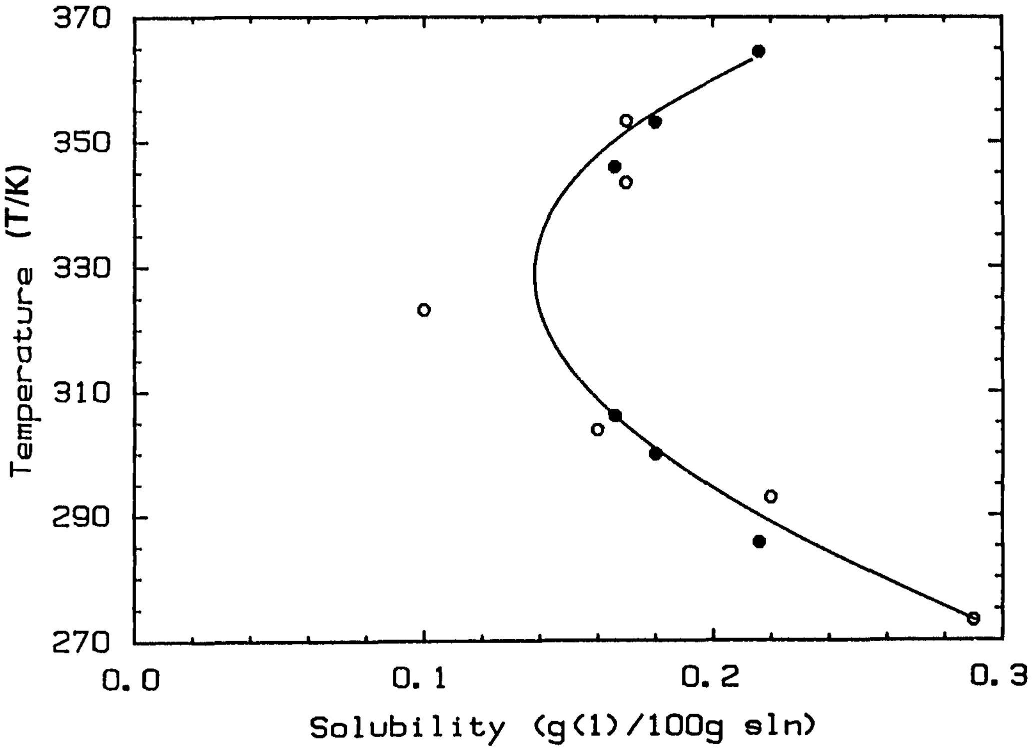

FIGURE 1. Selected data for the solubility of acetic acid pentyl ester (1) in water (2): ref 8 (●); ref 10 (O). Solid line is a least square polynomial fitted to the "Best" values from Table 2.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

TABLE 3: Recommended (*R*) and Tentative Solubilities of Water (2) in Acetic acid pentyl ester (1)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values $(\pm\sigma_n)^a$ | |
| | g(2)/100g sln | g(2)/100g sln | $10^2x_2$ |
| 273 | 0.79 (ref 10) | 0.79 | 5 |
| 283 | 0.64* (ref 8), 0.81* (ref 10) | 0.73 ± 0.09 | 5.1 |
| 293 | 0.63* (ref 8), 0.87* (ref 10) | 0.75 ± 0.12 | 5.2 |
| 298 | 0.65* (ref 8), 0.91 (ref 9), 0.91* (ref 10) | 0.82 ± 0.12 | 5.6 |
| 303 | 0.67* (ref 8), 0.96* (ref 10) | 0.82 ± 0.14 | 5.6 |
| 313 | 0.75* (ref 8), 1.06* (ref 10) | 0.9 ± 0.2 | 6 |
| 323 | 0.88* (ref 8), 1.16* (ref 10) | 1.02 ± 0.14 | 6.9 |
| 333 | 1.04* (ref 8), 1.26* (ref 10) | 1.15 ± 0.11 | 7.8 |
| 343 | 1.25* (ref 8), 1.34* (ref 10) | 1.30 ± 0.05 (*R*) | 8.7 |
| 353 | 1.51* (ref 8). 1.44* (ref 10) | 1.48 ± 0.04 (*R*) | 9.8 |
| 363 | 1.80* (ref 8), 1.55* (ref 10) | 1.68 ± 0.13 | 11 |

[a] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance. Mole fraction solubilities ($x_2$) have the same status and (relative) percentage uncertainties as the mass % solubilities.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

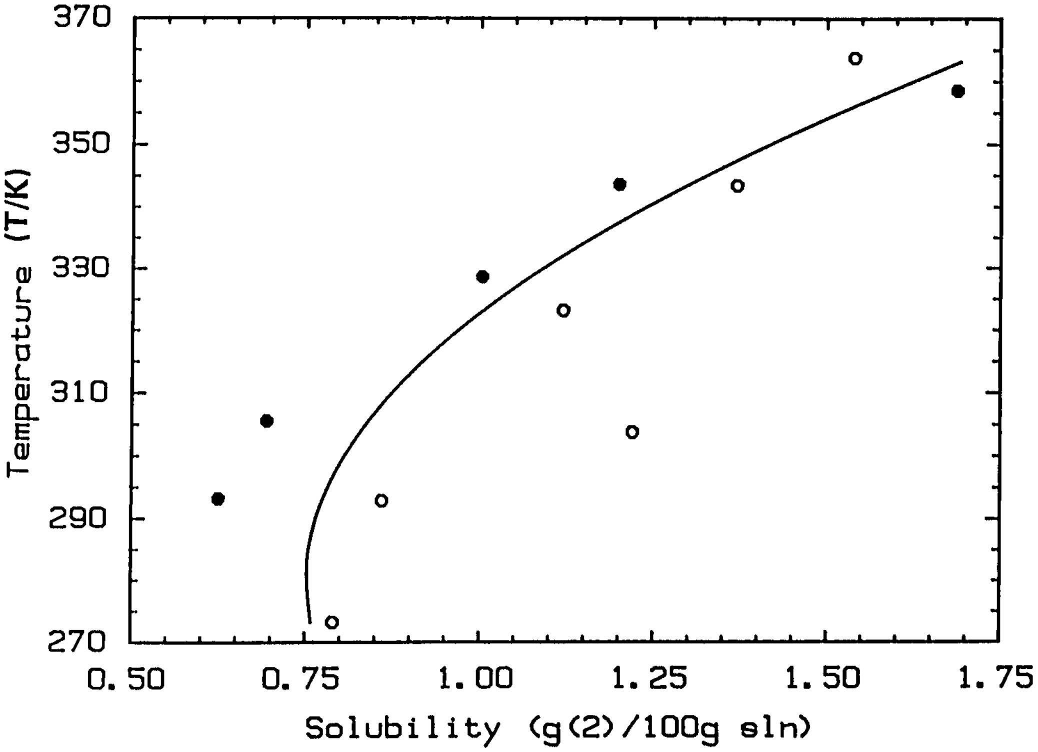

FIGURE 2. Selected data for the solubility of water (2) in acetic acid pentyl ester (1): ref 8 (●); ref 10 (O). Solid line is a least square polynomial fitted to the "Best" values from Table 3.

REFERENCES

1. Hemptinne, A. *Z. Phys. Chem.* 1894, *13*, 561-9.
2. Park, J. G.; Hopkins, M. B. *Ind. Eng. Chem.* 1930, *22*, 826-30.
3. Doolittle, A. K. *Ind. Eng. Chem.* 1935, *27*, 1169-79.
4. Rao, J. R.; Rao, C. V. *J. Appl. Chem.* 1957, *7*, 435-9.
5. Venkataratnam, A.; Rao, J. R.; Rao, C. V. *Chem. Eng. Sci.* 1957, *7*, 102-10.
6. Rao, J. R.,; Rao, C. V. *J. Appl. Chem.* 1959, *9*, 69-73.
7. Krupatkin, I. L.; Shcherbakova, T. A. *Zh. Prikl. Khim.* 1971, *44*, 307-11.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

8. Skrzecz, A. *Pol. J. Chem.* 1980, *54*, 1101-4; see also Skrzecz, A. *Thesis*, *I. Ch. F. PAN*, Warszawa, 1979.

9. Richon, D.; Viallard, A. *Fluid Phase Equil.* 1985, *21*, 279-93.

10. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

ACKNOWLEDGEMENT

The Evaluator thanks Dr. Brian Clare for the graphics.

COMPONENTS:

(1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Hemptinne, A.

*Z. Phys. Chem.* 1894, *13*, 561-9.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid pentyl ester in water at 25°C was reported to be 1.622 g(1)/L sln.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The mixture of water with excess ester was heated for some time in a water bath and the ester phase was filtered. A sample of known volume was then transferred to a smaller flask, heated with the known amount of baryta until complete saponification was obtained and then titrated. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]<br>(2) Water; $H_2O$; [7732-18-5] | Park, J.G.; Hopkins, M.B.<br>*Ind. Eng. Chem.* 1930, *22*, 826-30. |
| VARIABLES: | PREPARED BY: |
| $T/K = 298$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of acetic acid pentyl ester in water at 25°C was reported to be 0.2 mL(1)/100mL(2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial samples; used as received.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid pentyl ester in water at 20°C was reported to be 0.17 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $2.4 \times 10^{-4}$.

The solubility of water in acetic acid pentyl ester at 20°C was reported to be 1.15 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.0776.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 84%, mixed isomers; b.p. range 127-155°C, $d_4^{20}$ 0.862.

(2) Not specified.

ESTIMATED ERROR:

Soly. about ±0.03 g(1)/100g sln and ±0.5 g(2)/100g sln (compiler).

REFERENCES:

COMPONENTS:

(1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rao, J.R.; Rao, C.V.

*J. Appl. Chem.* 1957, *7*, 435-9.

VARIABLES:

$T/K = 303$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid pentyl ester in water at 30°C was reported to be 0.4 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $6 \times 10^{-4}$.

The solubility of water in acetic acid pentyl ester at 30°C was reported to be 1.7 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.111.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The data and method were reported together with the ternary system acetic acid pentyl ester-water-methanol.

SOURCE AND PURITY OF MATERIALS:

(1) May and Baker, reagent grade; 95% was distilled between 120 and 150°C, $d^{30}$ 0.8630, $n_D^{30}$ 1.3937.

(2) Distilled; free from $CO_2$.

ESTIMATED ERROR:

Temp. ±0.02°C (authors).
Soly. about ±0.2 g(1)/100g sln and ±1.0 g(2)/100g sln (compiler).

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]<br>(2) Water; $H_2O$; [7732-18-5] | Venkataratnam, A.; Rao, J.R.; Rao, C.V.<br>*Chem. Eng. Sci.* 1957, 7, 102-10. |
| VARIABLES: | PREPARED BY: |
| $T$/K = 303 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of acetic acid pentyl ester in water at 30°C was reported to be 0.2 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $3 \times 10^{-4}$.

The solubility of water in acetic acid pentyl ester at 30°C was reported to be 0.4 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.03.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method described by Othmer, White and Trueger (ref 1) was used. The data were reported together with the ternary system acetic acid pentyl ester-water-2-propanone (*pentyl acetate-water-acetone*).

SOURCE AND PURITY OF MATERIALS:

(1) May and Baker, analytical grade; used as received; 95% distilled between 120 and 150°C. $d^{30}$ 0.8630, $n^{30}$ 1.3937.

(2) Distilled; free of $CO_2$.

ESTIMATED ERROR:

Soly. about ±0.03 g(1)/100g sln and ±0.3 g(2)/100g sln (compiler).

REFERENCES:

1. Othmer, D.F.; White, R.E.; Trueger, E. *Ind. Eng. Chem.* 1941, *33*, 1240.

COMPONENTS:

(1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rao, J.R.; Rao, C.V.

*J. Appl. Chem.* 1959, *9*, 69-73.

VARIABLES:

$T$/K = 303

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid pentyl ester in water at 30°C was reported to be 0.4 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $6 \times 10^{-4}$.

The solubility of water in acetic acid pentyl ester at 30°C was reported to be 0.9 g(2)/100g sln and 99.2 g(1)/100g sln[a]. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.06.

[a] The sum of mass percent values is not equal 100%.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Presumably the titration method described by Rao and Rao (ref 1) was used. The data were reported together with the ternary system acetic acid pentyl ester-water-1-propanol.

SOURCE AND PURITY OF MATERIALS:

(1) May and Barker, analytical grade; 95% had distilled between 120 and 150°C, $d^{30}$ 0.8630, $n_D^{30}$ 1.3937.

(2) Distilled.

ESTIMATED ERROR:

Soly. about ±0.2 g(1)/100g sln and ±0.2 g(2)/100g sln (compiler).

REFERENCES:

1. Rao, J.R.; Rao, C.V. *J. Appl. Chem.* 1957, *7*, 435.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]<br>(2) Water; $H_2O$; [7732-18-5] | Krupatkin, I.L.; Shcherbakova, T.A.<br>*Zh. Prikl. Khim.* 1971, *44*, 307-11. |
| VARIABLES: | PREPARED BY: |
| $T/K = 298$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of acetic acid pentyl ester in water at 25°C was reported to be 0.22 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $3.05 \times 10^{-4}$.

The solubility of water in acetic acid pentyl ester at 25°C was reported to be 1.20 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.0807.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The amount of water in pure ester was taken into account. The data were reported together with the ternary system acetic acid pentyl ester-water-phosphoric acid.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, analytical grade; b.p. range 147-150°C.

(2) Twice distilled.

ESTIMATED ERROR:

Temp. ±0.1°C (authors).
Soly. about ±0.03 g(1)/100g sln and ±0.5 g(2)/100g sln (compiler).

REFERENCES:

COMPONENTS:

(1) Acetic acid pentyl ester
(*pentyl acetate*); $C_7H_{14}O_2$;
[628-63-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Skrzecz, A.

*Pol. J. Chem.* 1980, *54*, 1101-4.

Skrzecz, A.

*Thesis*, Inst. Phys. Chem., Pol. Acad. Sci., Warszawa, 1979.

VARIABLES:

$T$/K = 286 - 365

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid pentyl ester and water

| $T$/K | $x_1$ |  | g(1)/100g sln |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 285.6 | 0.00030 | - | 0.216 | - |
| 293.2 | - | 0.9565[a] | - | 99.375 |
| 299.9 | 0.00025 | - | 0.180 | - |
| 305.6 | - | 0.9519 | - | 99.306 |
| 306.1 | 0.00023 | - | 0.166 | - |
| 328.6 | - | 0.9318 | - | 98.997 |
| 343.6 | - | 0.9193 | - | 98.800 |
| 345.9 | 0.00023 | - | 0.166 | - |
| 353.1 | 0.00025 | - | 0.180 | - |
| 358.6 | - | 0.8896 | - | 98.312 |
| 364.5 | 0.00030 | - | 0.216 | - |

[a] By the Karl Fischer method.

Author's smoothing equations:

$$x_1 = 3.583\text{x}10^{-4} - 5.48\text{x}10^{-6}\ (T/K - 273.15) + 5.22\text{x}10^{-8}\ (T/K - 273.15)^2$$

st. dev. = $3.39 \text{ x } 10^{-6}$ (2)-rich phase

$$x_2 = 0.045710 - 3.4441\text{x}10^{-4}\ (T/K - 273.15) + 1.267\text{x}10^{-4}\ (T/K - 273.15)^2$$

st. dev. = $3.41 \text{ x } 10^{-3}$ (1)-rich phase

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The synthetic method of Alexejew and the analytical method were used. An ampoule with the solution of fixed concentration was placed into a glass tube connected with a thermostat filled completely with distilled water. The temperature of the bath was changed continuously during the measurements and the appearance and disappearance of turbidity within the ampoule was observed visually. For the analytical method, The amount of water in the saturated organic-phase was determined using a Karl Fischer titration procedure. The amount of water in the pure ester was taken into account.

SOURCE AND PURITY OF MATERIALS:

(1) POCH Gliwice, pure for analysis grade; distilled; purity 99.92% by glc, 0.27 wt% water by the Karl Fischer method.

(2) Distilled.

ESTIMATED ERROR:

Temp. ±(0.2-0.6)°C.
Soly. see above.

REFERENCES:

COMPONENTS:

(1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Richon, D.; Viallard, A.

*Fluid Phase Equilib.* 1985, *21*, 279-93.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid pentyl ester and water

| $T$/K | mol(1)/100g(2) | $x_1$ (2)-rich ph. | $x_1$ (1)-rich ph. | g(1)/100g sln (compiler) (2)-rich ph. | g(1)/100g sln (compiler) (1)-rich ph. |
|---|---|---|---|---|---|
| 298.1[a] | - | $2.33 \times 10^{-4}$ | 0.938 | 0.1681[d] | 99.09[e] |
| 298.1[b] | 0.00144 | $2.59 \times 10^{-4}$[c] | - | 0.1871[d] | - |

[a] Calorimetric method.
[b] Refractometric method.
[c] Compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The calorimetric and differential refractometric methods were used. The calorimetric method was described in the thesis of Richon (ref 1) and by Richon and Villard (ref 2). For the refractometric measurements, a Phoenix model 1-2000T differential refractometer from Texas Instruments was used, and the solubility was determined from a characteristic calibration curve as described in the thesis of Richon (ref 1).

SOURCE AND PURITY OF MATERIALS:

(1) Merck (for analysis); purified by preparation gas chromatographic method; purity >99.5%, water content was negligible.

(2) Distilled.

ESTIMATED ERROR:

Soly. about ±0.01[d] and ±0.2[e] g(1)/100g sln (compiler).

REFERENCES:

1. Richon, D. *Thesis*, University de Clermont-Ferrand, 1974.

2. Richon, D.; Villard, A. *Can. J. Chem.* 1976, *54*, 2584.

COMPONENTS:

(1) Acetic acid pentyl ester (*pentyl acetate*); $C_7H_{14}O_2$; [628-63-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid pentyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.29 | 99.21 | 0.00040 | 0.9456 |
| 9.8 | - | 99.19 | - | 0.9443 |
| 19.7 | 0.22 | 99.14 | 0.00030 | 0.9410 |
| 30.6 | 0.16 | 98.78 | 0.00022 | 0.9180 |
| 39.5 | 0.16 | 98.91 | 0.00022 | 0.9262 |
| 50.0 | 0.10 | 98.88 | 0.00014 | 0.9243 |
| 60.3 | 0.10 | 98.73 | 0.00014 | 0.9149 |
| 70.2 | 0.17 | 98.63 | 0.00023 | 0.9088 |
| 80.1 | 0.17 | 98.62 | 0.00023 | 0.9082 |
| 90.5 | - | 98.46 | - | 0.8984 |
| std. dev. | 0.01 | 0.02 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid 1-methylethyl ester (*isopropyl butyrate*); $C_7H_{14}O_2$; [638-11-9]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION:

Quantitative solubility data for the butanoic acid 1-methylethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Butanoic acid 1-methylethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Bomshtein *et al.* (ref 1) | 283-363 | mutual | titration |
| Stephenson and Stuart (ref 2) | 273-363 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF BUTANOIC ACID 1-METHYLETHYL ESTER (1) IN WATER (2)

All the available data (ref 1,2) for the solubility of butanoic acid 1-methylethyl ester (1) in water (2) are summarized in Table 2 and plotted in Figure 1. The data are in serious disagreement differing by up to an order of magnitude. Interestingly, the data of Bomshtein *et al.* (ref 1) are lower than those reported by Stephenson and Stuart (ref 2) whereas for most other systems studied by the same authors the reverse is true. In the absence of any other studies it is not possible to prefer one data set over the other and no "Best" values are proposed, however, the data of Stephenson and Stuart (ref 2) are usually reliable. Clearly this system warrants further investigation.

TABLE 2: Reported Solubilities of Butanoic acid 1-methylethyl ester (1) in Water (2)

| *T*/K | Reported solubilities | |
|---|---|---|
| | g(1)/100g sln | $10^3x_1^a$ |
| 273 | 3.12 (ref 2) | 4.4 |
| 283 | 0.20 (ref 1), 2.91* (ref 2) | 4.1 |
| 293 | 0.22 (ref 1), 2.74* (ref 2) | 3.8 |
| 298 | 0.24* (ref 1), 2.67* (ref 2) | 3.8 |

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid 1-methylethyl ester (*isopropyl butyrate*); $C_7H_{14}O_2$; [638-11-9]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

| $T$/K | Reported solubilities g(1)/100g sln | $10^3x_1$[a] |
|---|---|---|
| 303 | 0.25 (ref 1), 2.61* (ref 2) | 3.7 |
| 313 | 0.30 (ref 1), 2.53* (ref 2) | 3.6 |
| 323 | 0.40 (ref 1), 2.49* (ref 2) | 3.5 |
| 333 | 0.50 (ref 1), 2.56* (ref 2) | 3.7 |
| 343 | 0.65 (ref 1), 2.66* (ref 2) | 3.8 |
| 353 | 0.88 (ref 1), 2.82* (ref 2) | 4.0 |
| 363 | 1.40 (ref 1), 3.00* (ref 2) | 4.3 |

[a] Values of ref 2, see text.

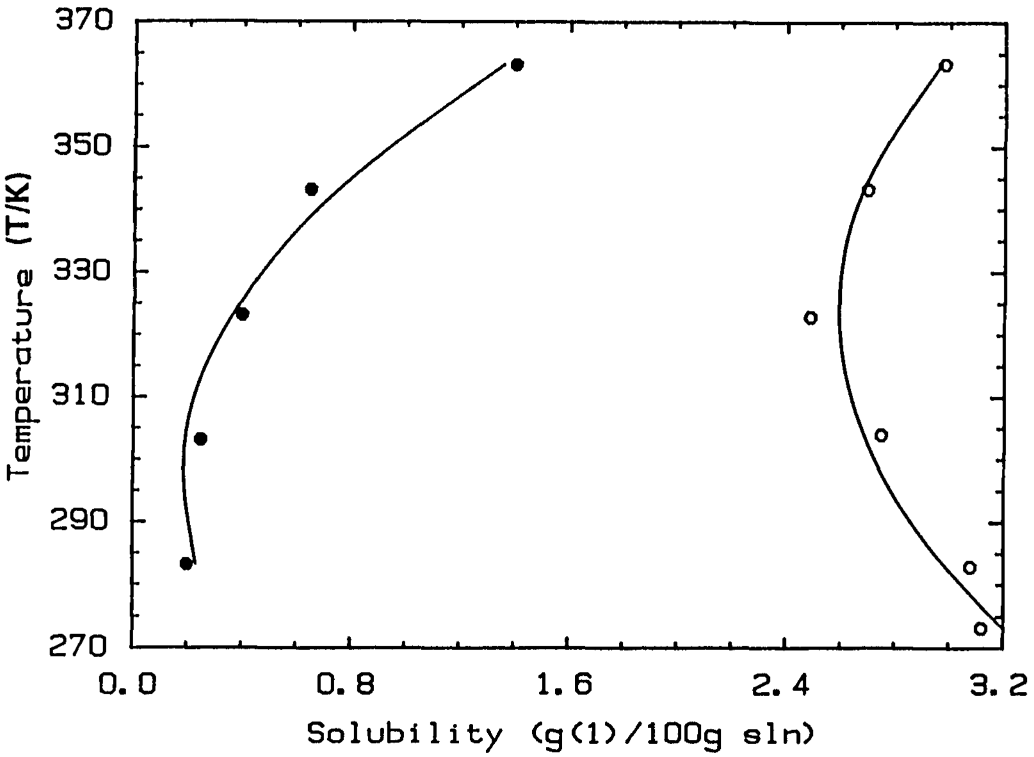

FIGURE 1. Data for the solubility of butanoic acid 1-methylethyl ester (1) in water (2): ref 1 (●); ref 2 (○). Solid lines are least squares polynomial fits and are included only for illustrative purposes.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid 1-methylethyl ester (*isopropyl butyrate*); $C_7H_{14}O_2$; [638-11-9]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

## 2. SOLUBILITY OF WATER (2) IN BUTANOIC ACID 1-METHYLETHYL ESTER (1)

All the available data for the solubility of water (2) in butanoic acid 1-methylethyl ester (1) are summarized in Table 3 and plotted in Figure 2.

Although the two studies show a rather different dependence on temperature the results, unlike the aqueous phase solubilities, are in reasonable agreement and the average values may be regarded as Tentative pending further studies.

TABLE 3: Tentative Solubilities of Water (2) in Butanoic acid 1-methylethyl ester (1)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values ($\pm\sigma_n$)[a] | |
| | g(2)/100g sln | g(2)/100g sln | $x_2$ |
| 273 | 1.71 (ref 2) | 1.7 | 0.11 |
| 283 | 0.90 (ref 1), 1.81* (ref 2) | 1.4 ± 0.5 | 0.09 |
| 293 | 1.20 (ref 1), 1.92* (ref 2) | 1.6 ± 0.4 | 0.11 |
| 298 | 1.35* (ref 1), 2.00* (ref 2) | 1.7 ± 0.3 | 0.11 |
| 303 | 1.50 (ref 1), 2.06* (ref 2) | 1.8 ± 0.3 | 0.12 |
| 313 | 1.80 (ref 1), 2.20* (ref 2) | 2.0 ± 0.2 | 0.13 |
| 323 | 2.10 (ref 1), 2.37* (ref 2) | 2.2 ± 0.2 | 0.14 |
| 333 | 2.30 (ref 1), 2.57* (ref 2) | 2.4 ± 0.2 | 0.15 |
| 343 | 2.60 (ref 1), 2.78* (ref 2) | 2.7 ± 0.1 | 0.17 |
| 353 | 3.40 (ref 1), 3.00* (ref 2) | 3.2 ± 0.2 | 0.19 |
| 363 | 5.50 (ref 1), 3.20* (ref 2) | 4.4 ± 1.3 | 0.25 |

[a] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance. Mole fraction solubilities ($x_2$) have the same status and (relative) percentage uncertainties as the mass % solubilities.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid 1-methylethyl ester (*isopropyl butyrate*); $C_7H_{14}O_2$; [638-11-9]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

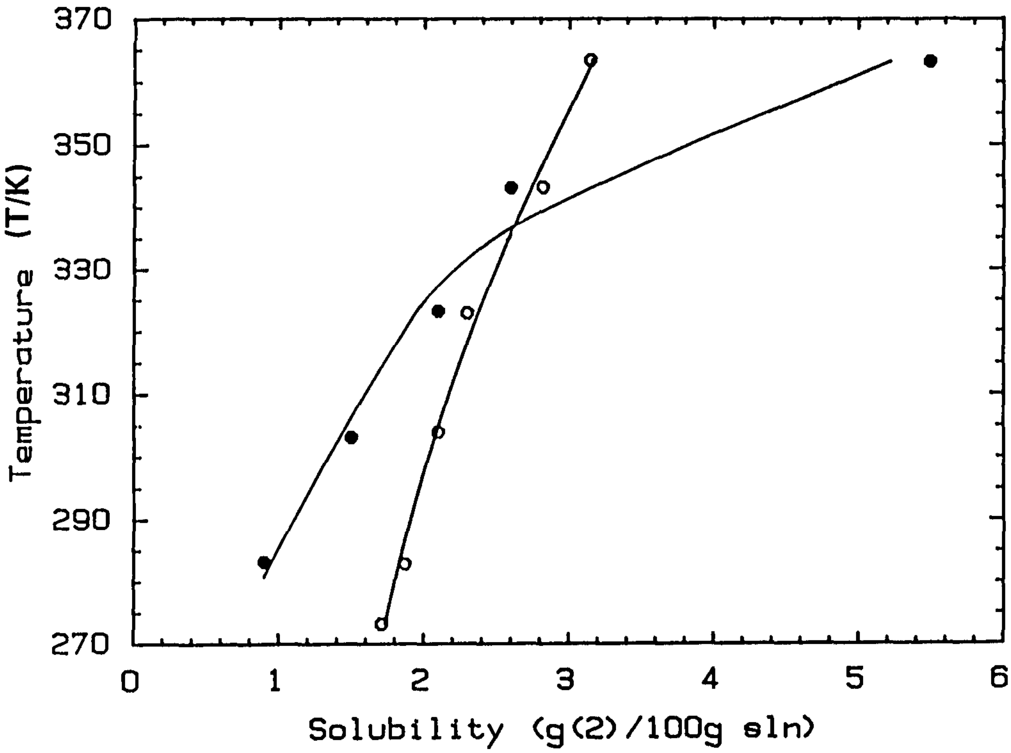

FIGURE 2. Data for the solubility of water (2) in butanoic acid 1-methylethyl ester (1): ref 1 (●); ref 2 (O). Solid lines are least squares polynomial fits and are included only for illustrative purposes.

REFERENCES

1. Bomshtein, A. L.; Trofimov, A. N.; Serafimov, L. A. *Zh. Prikl. Khim.* <u>1978</u>, *51*, 1280-2.

2. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* <u>1986</u>, *31*, 56-70.

ACKNOWLEDGEMENT

The Evaluator thanks Dr. Brian Clare for the graphics.

COMPONENTS:

(1) Butanoic acid 1-methylethyl ester (*isopropyl butyrate*); $C_7H_{14}O_2$; [638-11-9]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Bomshtein, A.L.; Trofimov, A.N.; Serafimov, L.A.

*Zh. Prikl. Khim.* 1978, *51*, 1280-2.

VARIABLES:

$T$/K = 283 - 363

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of butanoic acid 1-methylethyl ester and water

| $t$/°C | $x_1$ | | g(1)/100g sln (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 10 | 0.00028 | 0.9384 | 0.20 | 99.10 |
| 20 | 0.00031 | 0.9193 | 0.22 | 98.80 |
| 30 | 0.00035 | 0.9009 | 0.25 | 98.50 |
| 40 | 0.00042 | 0.8831 | 0.30 | 98.20 |
| 50 | 0.00056 | 0.8659 | 0.40 | 97.90 |
| 60 | 0.00069 | 0.8547 | 0.50 | 97.70 |
| 70 | 0.00091 | 0.8384 | 0.65 | 97.40 |
| 80 | 0.00123 | 0.7973 | 0.88 | 96.60 |
| 90 | 0.00196 | 0.7041 | 1.40 | 94.50 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used at constant temperature. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; distilled; without impurities by glc, b.p. 125.0°C, $n_D^{20}$ 1.394.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Butanoic acid 1-methylethyl ester (*isopropyl butyrate*); $C_7H_{14}O_2$; [638-11-9]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 363

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of butanoic acid 1-methylethyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 3.12 | 98.29 | 0.00444 | 0.8883 |
| 9.7 | 3.08 | 98.13 | 0.00438 | 0.8789 |
| 19.5 | 2.65 | 98.18 | 0.00375 | 0.8819 |
| 30.8 | 2.75 | 97.90 | 0.00390 | 0.8658 |
| 39.5 | 2.57 | 97.91 | 0.00364 | 0.8663 |
| 49.7 | 2.49 | 97.70 | 0.00352 | 0.8546 |
| 60.2 | 2.49 | 97.39 | 0.00352 | 0.8377 |
| 70.1 | 2.70 | 97.18 | 0.00382 | 0.8266 |
| 80.0 | 2.84 | 96.93 | 0.00403 | 0.8137 |
| 90.2 | 2.98 | 96.85 | 0.00423 | 0.8097 |
| std. dev. | 0.02 | 0.02 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 98%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid, 3-methyl-, ethyl ester (*ethyl isovalerate*); $C_7H_{14}O_2$; [108-64-5]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION:

Quantitative solubility data for the 3-methylbutanoic acid ethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the 3-Methylbutanoic acid ethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Bancroft (ref 1) | 293 | mutual | titration |
| Chang and Moulton (ref 2) | 298 | mutual | titration |
| Stephenson and Stuart (ref 3) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF 3-METHYLBUTANOIC ACID ETHYL ESTER (1) IN WATER (2)

All the available data for the solubility of 3-methylbutanoic acid ethyl ester (1) in water (2) are summarized in Table 2 except for the datum of Bancroft which is reported in v/v units and hence excluded from this Evaluation.

At 298 K, the only temperature where comparison is possible, the datum of Chang and Moulton (ref 2), is in quite good agreement with the interpolated value of Stephenson and Stuart (ref 3), lending confidence to the latter's values at other temperatures. Nevertheless, pending further studies, the data of Stephenson and Stuart must be regarded as Tentative although it may be noted that these authors' data are usually reliable.

TABLE 2: Tentative Solubilities of 3-Methylbutanoic acid ethyl ester (1) in Water (2)

| *T*/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 0.34 (ref 3) | 0.3 | 4 |
| 283 | 0.24 (ref 3) | 0.2 | 3 |
| 293 | $0.21^*$ (ref 3) | 0.2 | 3 |
| 298 | 0.16 (ref 2), $0.20^*$ (ref 3) | 0.20 | 2.8 |

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid, 3-methyl-, ethyl ester *(ethyl isovalerate)*; $C_7H_{14}O_2$; [108-64-5]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

| *T*/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 303 | 0.19* (ref 3) | 0.2 | 3 |
| 313 | 0.17* (ref 3) | 0.2 | 3 |
| 323 | 0.16* (ref 3) | 0.2 | 3 |
| 333 | 0.16* (ref 3) | 0.2 | 3 |
| 343 | 0.16* (ref 3) | 0.2 | 3 |
| 353 | 0.16* (ref 3) | 0.2 | 3 |
| 363 | 0.16* (ref 3) | 0.2 | 3 |

[a] Rounded values of ref 3.

2. SOLUBILITY OF WATER (2) IN 3-METHYLBUTANOIC ACID ETHYL ESTER (1)

The situation with regard to the solubility of water (2) in 3-methylbutanoic acid ethyl ester (1) is identical to that of the $H_2O$-rich phase and the comments under Part 1. of this Evaluation are equally applicable and should be consulted in conjunction with Table 3.

TABLE 3: Tentative Solubilities of Water (2) in 3-Methylbutanoic acid ethyl ester (1)

| *T*/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(2)/100g sln | g(2)/100g sln | $10^2x_2$ |
| 273 | 0.41 (ref 3) | 0.4 | 3 |
| 283 | 0.48* (ref 3) | 0.5 | 3 |
| 293 | 0.52* (ref 3) | 0.5 | 3 |
| 298 | 0.47 (ref 2), 0.61* (ref 3) | $0.54^b$ | 3.8 |
| 303 | 0.69* (ref 3) | 0.7 | 5 |
| 313 | 0.71* (ref 3) | 0.7 | 5 |
| 323 | 0.79* (ref 3) | 0.8 | 6 |

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid, 3-methyl-, ethyl ester<br>*(ethyl isovalerate)*; $C_7H_{14}O_2$; [108-64-5]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

| $T$/K | Solubilities | | | |
|---|---|---|---|---|
| | Reported values | | "Best" values [a] | |
| | g(2)/100g sln | | g(2)/100g sln | $10^2x_2$ |
| 333 | 0.83* | (ref 3) | 0.8 | 6 |
| 343 | 0.85* | (ref 3) | 0.9 | 6 |
| 353 | 0.94* | (ref 3) | 0.9 | 6 |
| 363 | 1.11* | (ref 3) | 1.1 | 8 |

[a] Rounded values of ref 3.

[b] Average value.

REFERENCES

1. Bancroft, W. D. *Phys. Rev.* 1895, *3*, 114-36.
2. Chang, Y. C.; Moulton, R. W. *Ind. Eng. Chem.* 1953, *45*, 2350-61.
3. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

COMPONENTS:

(1) Butanoic acid, 3-methyl-, ethyl ester (*ethyl isovalerate*); $C_7H_{14}O_2$; [108-64-5]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Bancroft, W.D.

*Phys. Rev.* 1895, 3, 114-36.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 3-methylbutanoic acid ethyl ester in water at 20°C was reported to be 0.02 mL(1)/10mL(2).

The solubility of water in 3-methylbutanoic acid ethyl ester at 20°C was reported to be 0.04 mL(2)/10mL(1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. 10 mL of solvent in a test tube was titrated with the second component until the solution became cloudy.

SOURCE AND PURITY OF MATERIALS:

(1) Laboratory source; dried over $CaCl_2$, distilled.

(2) Not specified.

ESTIMATED ERROR:

Soly. ±0.01 mL.

REFERENCES:

COMPONENTS:

(1) Butanoic acid, 3-methyl-, ethyl ester (*ethyl isovalerate*); $C_7H_{14}O_2$; [108-64-5]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Chang, Yi-C.; Moulton, R.W.

*Ind. Eng. Chem.* 1953, *45*, 2350-61.

VARIABLES:

$T/K = 298$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 3-methylbutanoic acid ethyl ester in water at 25°C was reported to be 0.16 g(1)/100g(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.16 g(1)/100g sln and $2.2 \times 10^{-4}$.

The solubility of water in 3-methylbutanoic acid ethyl ester at 25°C was reported to be 0.47 g(2)/100g(1). The corresponding mass per cent and mole fraction, $x_2$, values calculated by the compiler are 0.47 g(2)/100g sln and 0.033.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The 100g portion of solvent in a thermostated container was titrated with the second component from a 1 mL pipette calibrated to 0.01 mL. An average of two closest values (definitely cloud and definitely clear) was presented. The amount of solute (about 0.2 mL) was very difficult to judge and it was accurate only to 0.05 mL. The data were reported together with the ternary system 3-methylbutanoic acid ethyl ester-water-benzene.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; washed with dilute KOH and $H_2O$, dehydrated with anhydrone, fractionally distilled; purity 99.8% by a saponification test, b.p. range 133-134°C at 755 mm Hg, $n_D^{20}$ 1.3968.

(2) Distilled (distillation from $KMnO_4$ caused no change in the refractive index).

ESTIMATED ERROR:

Temp. ±0.1°C.
Soly. ±25% (probably relative error).

REFERENCES:

COMPONENTS:

(1) Butanoic acid, 3-methyl-, ethyl ester (*ethyl isovalerate*); $C_7H_{14}O_2$; [108-64-5]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of 3-methylbutanoic acid ethyl ester and water

| $t$/°C | g(1)/100g sln<br>(2)-rich phase | <br>(1)-rich phase | $x_1$ (compiler)<br>(2)-rich phase | <br>(1)-rich phase |
|---|---|---|---|---|
| 0 | 0.34 | 99.588 | 0.00047 | 0.97097 |
| 9.0 | 0.24 | 99.527 | 0.00033 | 0.96679 |
| 19.4 | 0.22 | 99.442 | 0.00030 | 0.96103 |
| 30.6 | 0.20 | 99.355 | 0.00028 | 0.95519 |
| 39.8 | 0.17 | 99.293 | 0.00023 | 0.95106 |
| 49.5 | 0.16 | 99.211 | 0.00022 | 0.94565 |
| 60.1 | 0.15 | 99.173 | 0.00021 | 0.94316 |
| 70.0 | 0.16 | 99.155 | 0.00022 | 0.94199 |
| 80.3 | 0.16 | 99.057 | 0.00022 | 0.93563 |
| 90.5 | 0.16 | 98.877 | 0.00022 | 0.92415 |
| std. dev. | 0.01 | 0.004 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Butanoic acid propyl ester (*propyl butyrate*); $C_7H_{14}O_2$; [105-66-8]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
January, 1989

CRITICAL EVALUATION:

Quantitative solubility data for the butanoic acid propyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Butanoic acid propyl ester (1) - Water (2) System

| Reference | $T$/K | Solubility | Method |
|---|---|---|---|
| Fuehner (ref 1) | 290 | (1) in (2) | titration |
| Stephenson and Stuart (ref 2) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF BUTANOIC ACID PROPYL ESTER (1) IN WATER (2)

All the available data are summarized in Table 2. At 290 K, the only temperature where comparison is possible, the values of Fuehner (ref 1) and Stephenson and Stuart (ref 2) are in good agreement giving confidence in the remaining data of ref 2. However, in the absence of independent studies the data of Stephenson and Stuart must be regarded as Tentative although it may be noted that the solubilities of these authors are usually reliable.

TABLE 2: Tentative Solubilities of Butanoic acid propyl ester (1) in Water (2)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 0.30 (ref 2) | 0.3 | 4 |
| 283 | 0.22* (ref 2) | 0.2 | 3 |
| 290 | 0.162 (ref 1), 0.19* (ref 2) | 0.18 | 2.5 |
| 293 | 0.19* (ref 2) | 0.2 | 3 |
| 298 | 0.18* (ref 2) | 0.2 | 3 |
| 303 | 0.17* (ref 2) | 0.2 | 3 |

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid propyl ester (*propyl butyrate*); $C_7H_{14}O_2$; [105-66-8]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

| $T/K$ | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 313 | 0.16* (ref 2) | 0.2 | 3 |
| 323 | 0.16* (ref 2) | 0.2 | 3 |
| 333 | 0.16* (ref 2) | 0.2 | 3 |
| 343 | 0.16* (ref 2) | 0.2 | 3 |
| 353 | 0.16* (ref 2) | 0.2 | 3 |
| 363 | 0.16* (ref 2) | 0.2 | 3 |

[a] Rounded values of ref 2 except at 290 K.

2. SOLUBILITY OF WATER (2) IN BUTANOIC ACID PROPYL ESTER (1)

Only the data of Stephenson and Stuart (ref 2) are available for the solubility of water (2) in propyl butyrate (1) and so no Critical Evaluation is possible. The interested user is referrd to the relevant Data Sheet for the experimental solubilities but it may be noted that the data of Stephenson and Stuart (ref 2) are usually reliable.

REFERENCES

1. Fuehner, H. *Ber. Dtsch. Chem. Ges.* 1924, *57*, 510-5.

2. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

COMPONENTS:

(1) Butanoic acid propyl ester (*propyl butyrate*); $C_7H_{14}O_2$; [105-66-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Fuehner, H.

*Ber. Dtsch. Chem. Ges.* 1924, 57, 510-5.

VARIABLES:

$T/K = 290$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of butanoic acid propyl ester in water at 17°C was reported to be 0.195 vol%, 0.162 g(1)/100g sln and 0.0124 mol(1)/L sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $2.24 \times 10^{-4}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added from pipette to the flask with a constant amount of water (50, 100 or 1000 mL) so long as, on shaking, the mixture remained transparent.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Butanoic acid propyl ester (*propyl butyrate*); $C_7H_{14}O_2$; [105-66-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of butanoic acid propyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.30 | 99.36 | 0.00042 | 0.9555 |
| 9.6 | 0.23 | - | 0.00032 | - |
| 20.1 | 0.19 | 99.37 | 0.00026 | 0.9562 |
| 29.6 | 0.15 | 99.36 | 0.00021 | 0.9555 |
| 39.5 | 0.14 | 99.34 | 0.00019 | 0.9542 |
| 49.8 | 0.17 | 99.16 | 0.00023 | 0.9423 |
| 60.1 | 0.21 | 98.52 | 0.00029 | 0.9021 |
| 70.1 | 0.15 | - | 0.00021 | - |
| 80.1 | 0.14 | 99.01 | 0.00019 | 0.9326 |
| 90.5 | 0.17 | 98.19 | 0.00023 | 0.8824 |
| std. dev. | 0.01 | 0.01 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Formic acid hexyl ester (*hexyl formate*); $C_7H_{14}O_2$; [629-33-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of formic acid hexyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | - | 99.474 | - | 0.96319 |
| 8.7 | 0.17 | 99.475 | 0.00024 | 0.96326 |
| 19.7 | 0.15 | 99.492 | 0.00021 | 0.96441 |
| 29.7 | 0.13 | 99.401 | 0.00018 | 0.95826 |
| 39.6 | 0.13 | 99.340 | 0.00018 | 0.95418 |
| 49.8 | 0.11 | 99.346 | 0.00015 | 0.95458 |
| 60.2 | 0.11 | 99.281 | 0.00015 | 0.95027 |
| 70.3 | 0.12 | - | 0.00017 | - |
| 80.2 | 0.13 | 99.153 | 0.00018 | 0.94185 |
| 90.6 | 0.14 | 99.201 | 0.00019 | 0.94499 |
| std. dev. | 0.01 | 0.003 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 96%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Hexanoic acid methyl ester (*methyl caproate*); $C_7H_{14}O_2$; [106-70-7]<br>(2) Water; $H_2O$; [7732-18-5] | Stephenson, R.; Stuart, J.<br>*J. Chem. Eng. Data* <u>1986</u>, *31*, 56-70. |
| VARIABLES: | PREPARED BY: |
| $T$/K = 273 - 364 | Z. Maczynska |

EXPERIMENTAL VALUES:

Mutual solubility of hexanoic acid methyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.241 | 99.62 | 0.000334 | 0.9732 |
| 9.3 | 0.177 | 99.54 | 0.000245 | 0.9677 |
| 20.0 | 0.166 | 99.45 | 0.000230 | 0.9616 |
| 29.7 | 0.167 | 99.40 | 0.000231 | 0.9582 |
| 39.5 | 0.129 | 99.32 | 0.000179 | 0.9528 |
| 50.0 | 0.137 | 99.25 | 0.000190 | 0.9482 |
| 60.0 | 0.132 | 99.19 | 0.000183 | 0.9442 |
| 70.2 | 0.128 | 99.20 | 0.000177 | 0.9449 |
| 80.2 | 0.138 | 99.17 | 0.000191 | 0.9429 |
| 90.5 | 0.147 | 99.11 | 0.000204 | 0.9390 |
| std. dev. | 0.001 | 0.01 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Pentanoic acid ethyl ester (*ethyl valerate*); $C_7H_{14}O_2$; [539-82-2]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A., Australia
January, 1989

CRITICAL EVALUATION:

Quantitative solubility data for the pentanoic acid ethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Pentanoic acid ethyl ester (1) - Water (2) System

| Reference | $T$/K | Solubility | Method |
|---|---|---|---|
| Hemptinne (ref 1) | 298 | (1) in (2) | analytical |
| Bomshtein *et al.* (ref 2) | 283-353 | mutual | titration |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

## 1. SOLUBILITY OF PENTANOIC ACID ETHYL ESTER (1) IN WATER (2)

For the solubility of pentanoic acid ethyl ester (1) in water (2) all the available data are summarized in Table 2. At 298 K, the only temperature where comparison is possible, the datum of Hemptinne (ref 1) is in good agreement with the interpolated value of Bomshtein *et al.* (ref 2). However, it should be noted that the solubilities reported by Bomshtein *et al.* often differ significantly from the "Best" values in well-characterized systems. For this reason the data in Table 2 should be regarded as very Tentative.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Pentanoic acid ethyl ester (*ethyl valerate*); $C_7H_{14}O_2$; [539-82-2]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

TABLE 2: Tentative Solubilities of Pentanoic acid ethyl ester (1) in Water (2)

| *T*/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 283 | 0.24 (ref 2) | 0.2 | 3 |
| 293 | 0.25 (ref 2) | 0.25 | 3.5 |
| 298 | 0.237 (ref 1), 0.255* (ref 2) | 0.25 | 3.5 |
| 303 | 0.26 (ref 2) | 0.3 | 4 |
| 313 | 0.27 (ref 2) | 0.3 | 4 |
| 323 | 0.32 (ref 2) | 0.3 | 4 |
| 333 | 0.37 (ref 2) | 0.4 | 6 |
| 343 | 0.55 (ref 2) | 0.6 | 8 |
| 353 | 0.78 (ref 2) | 0.8 | 11 |

[a] Rounded values of ref (2), but see text.

2. SOLUBILITY OF WATER (2) IN PENTANOIC ACID ETHYL ESTER (1)

As the only data available for the solubility of water (2) in pentanoic acid ethyl ester (1) are those of Bomshtein *et al.* (ref 2) no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheet but the comments made under part 1. of this Evaluation above should be noted.

REFERENCES

1. Hemptinne, A. *Z. Phys. Chem.* 1894, *13*, 561-9.
2. Bomshtein, A. L.; Trofimov, A. N.; Serafimov, L. A. *Zh. Prikl. Khim.* 1978, *51*, 1280-2.

| | |
|---|---|
| COMPONENTS:<br>(1) Pentanoic acid ethyl ester (*ethyl valerate*); $C_7H_{14}O_2$; [539-82-2]<br>(2) Water; $H_2O$; [7732-18-5] | ORIGINAL MEASUREMENTS:<br>Hemptinne, A.<br>*Z. Phys. Chem.* 1894, *13*, 561-9. |
| VARIABLES:<br>$T$/K = 298 | PREPARED BY:<br>A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of pentanoic acid ethyl ester in water at 25°C was reported to be 2.366 g(1)/L sln.

AUXILIARY INFORMATION

| | |
|---|---|
| METHOD/APPARATUS/PROCEDURE:<br>The analytical method was used. The mixture of water with excess ester was heated for some time in a water bath and the ester phase was filtered. A sample of known volume was then transferred to a smaller flask, heated with the known amount of baryta until complete saponification was obtained and then titrated. No further details were reported in the paper. | SOURCE AND PURITY OF MATERIALS:<br>(1) Not specified.<br>(2) Not specified.<br>ESTIMATED ERROR:<br>Not specified.<br>REFERENCES: |

COMPONENTS:

(1) Pentanoic acid ethyl ester (*ethyl valerate*); $C_7H_{14}O_2$; [539-82-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Bomshtein, A.L.; Trofimov, A.N.; Serafimov, L.A.

*Zh. Prikl. Khim.* 1978, *51*, 1280-2.

VARIABLES:

$T/K$ = 283 - 353

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of pentanoic acid ethyl ester and water

| $t/°C$ | $x_1$ (2)-rich phase | $x_1$ (1)-rich phase | g(1)/100g sln (compiler) (2)-rich phase | g(1)/100g sln (compiler) (1)-rich phase |
|---|---|---|---|---|
| 10 | 0.00033 | 0.9928 | 0.24 | 99.90 |
| 20 | 0.00034 | 0.9650 | 0.25 | 99.50 |
| 30 | 0.00036 | 0.9257 | 0.26 | 98.90 |
| 40 | 0.00038 | 0.9009 | 0.27 | 98.50 |
| 50 | 0.00044 | 0.8715 | 0.32 | 98.00 |
| 60 | 0.00051 | 0.8492 | 0.37 | 97.60 |
| 70 | 0.00077 | 0.8226 | 0.55 | 97.10 |
| 80 | 0.00109 | 0.7828 | 0.78 | 96.30 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used at constant temperature. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; distilled; without impurities by glc, b.p. 145.2°C, $n_D^{20}$ 1.400.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Propanoic acid butyl ester (*butyl propionate*); $C_7H_{14}O_2$; [590-01-2]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
January, 1989

CRITICAL EVALUATION:

Quantitative solubility data for the propanoic acid butyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Propanoic acid butyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Bridgman (ref 1) | 280-303 | (2) in (1) | synthetic |
| Park and Hopkins (ref 2) | 298 | (1) in (2) | unspecified |
| Doolittle (ref 3) | 293 | mutual | unspecified |
| Bomshtein *et al.* (ref 4) | 368 | mutual | titration |
| Stephenson and Stuart (ref 5) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

## 1. SOLUBILITY OF PROPANOIC ACID BUTYL ESTER (1) IN WATER (2)

Apart from the single temperature values of Doolittle (ref 3) and Bomshtein *et al.* (ref 4), only the data of Stephenson and Stuart (ref 5) are available for the solubilities of propanoic acid butyl ester (1) in water (2) over a range of temperatures. The datum of Doolittle (ref 3) is in reasonable agreement with that of Stephenson and Stuart (ref 5) but Bomshtein *et al.*'s (ref 4) value is an order of magnitude higher. In the absence of confirmatory studies it is not possible to assess the quality of these data although it may be noted that the data of Stephenson and Stuart (ref 5) are generally reliable and those of Bomshtein *et al.* (ref 4) are often high.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Propanoic acid butyl ester (*butyl propionate*); $C_7H_{14}O_2$; [590-01-2]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>January, 1989 |

CRITICAL EVALUATION: (continued)

TABLE 2: Reported Solubilities of Propanoic acid butyl ester (1) in Water (2)

| $T$/K | Reported Solubilities g(1)/100g sln | $10^4x_1$[a] |
|---|---|---|
| 273 | 0.298 (ref 5) | 4.1 |
| 283 | 0.228* (ref 5) | 3.2 |
| 293 | 0.15 (ref 3), 0.193* (ref 5) | 2.7 |
| 298 | 0.180* (ref 5) | 2.5 |
| 303 | 0.169* (ref 5) | 2.3 |
| 313 | 0.155* (ref 5) | 2.1 |
| 323 | 0.146* (ref 5) | 2.0 |
| 333 | 0.143* (ref 5) | 2.0 |
| 343 | 0.141* (ref 5) | 1.9 |
| 353 | 0.142* (ref 5) | 2.0 |
| 363 | 1.4[b] (ref 4), 0.142* (ref 5) | 2.0 |

[a] Data of Stephenson and Stuart (ref 5).

[b] 368 K (boiling point of mixture).

2. SOLUBILITY OF WATER (2) IN PROPANOIC ACID BUTYL ESTER (1)

All the available data for the solubility of water (2) in propanoic acid butyl ester (1) are summarized in Table 3. The situation for the organic-rich phase is better than for the aqueous phase, with the values of Bridgman (ref 1) being in excellent agreement with those of Stephenson and Stuart (ref 5) from 283-303 K. At other temperatures only the data of Stephenson and Stuart are available and in the absence of confirmatory studies must be regarded as Tentative.

(continued next page)

COMPONENTS:

(1) Propanoic acid butyl ester (*butyl propionate*); $C_7H_{14}O_2$; [590-01-2]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
January, 1989

CRITICAL EVALUATION: (continued)

TABLE 3: Recommended (*R*) and Tentative Solubilities of Water (2) in Propanoic acid butyl ester (1)

| $T$/K | Solubilities |  |  |
|---|---|---|---|
|  | Reported values | "Best" values ($\pm\sigma_n$)[a] |  |
|  | g(2)/100g sln | g(2)/100g sln | $10^2x_2$ |
| 273 | 0.48 (ref 5) | 0.5 | 4 |
| 283 | 0.470* (ref 1), 0.53* (ref 5) | 0.50 ± 0.03 (*R*) | 3.5 |
| 293 | 0.546* (ref 1), 0.80[b] (ref 3), 0.62* (ref 5) | 0.58 ± 0.04 (*R*) | 4.0 |
| 298 | 0.606* (ref 1), 0.68* (ref 5) | 0.64 ± 0.04 (*R*) | 4.5 |
| 303 | 0.686 (ref 1), 0.73* (ref 5) | 0.71 ± 0.02 (*R*) | 4.9 |
| 313 | 0.82* (ref 5) | 0.8 | 6 |
| 323 | 0.86* (ref 5) | 0.9 | 6 |
| 333 | 0.89* (ref 5) | 0.9 | 6 |
| 343 | 0.92* (ref 5) | 0.9 | 6 |
| 353 | 0.94* (ref 5) | 0.9 | 6 |
| 363 | 3.76[c] (ref 4), 1.00* (ref 5) | 1.0 | 7 |

[a] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance. Mole fraction solubilities ($x_2$) have the same status and (relative) percentage uncertainties as the mass % solubilities.

[b] Not included in calculation of "Best" value, see text.

[c] 368 K; not included in calculation of "Best" value, see text.

## REFERENCES

1. Bridgman, J. A. *Ind. Eng. Chem.* 1928, *20*, 184-7.
2. Park, J. G.; Hopkins. M. B. *Ind. Eng. Chem.* 1930, *22*, 826-30.
3. Doolittle, A. K. *Ind. Eng. Chem.* 1935, *27*, 1169-79.
4. Bomshtein, A. L.; Trofimov, A. N.; Serafimov, L. A. *Zh. Prikl. Khim.* 1984, *57*, 18-23.
5. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

COMPONENTS:

(1) Propanoic acid butyl ester (*butyl propionate*); $C_7H_{14}O_2$; [590-01-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Bridgman, J.A.

*Ind. End. Chem.* 1928, *20*, 184-7.

VARIABLES:

$T$/K = 280 - 303

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Solubility of water in propanoic acid butyl ester

| $t$/°C | g(2)/100g(1) | g(2)/100g solution (compiler) | $x_2$ (compiler) |
|---|---|---|---|
| 7.0 | 0.452 | 0.450 | 0.0316 |
| 16.0 | 0.513 | 0.510 | 0.0357 |
| 22.5 | 0.576 | 0.573 | 0.0400 |
| 30.0 | 0.691 | 0.686 | 0.0475 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The synthetic method similar to that described by Groschuff (ref 1) was used. A glass tube of about 100 mL capacity with a stopcock was filled with weighed (1) and (2). By shaking the tube after it had been warmed somewhat, the water was dissolved completely in the ester, after which the tube was placed in a 2-liter beaker filled with water and equipped with an agitator. By alternate slow cooling and warming, it was possible to determine, within about 0.5°C, the temperature at which water was precipitated from the solution, as shown by the clouding of the liquid which would clear again when the temperature was slightly increased.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; distilled; b.p. range 144-146°C; boiled for some time in open flask before used.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.25°C.

REFERENCES:

1. Groschuff, E. *Z. Elektrochem.* 1911, *17*, 348.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Propanoic acid butyl ester (*butyl propionate*); $C_7H_{14}O_2$; [590-01-2]<br>(2) Water; $H_2O$; [7732-18-5] | Park, J.G.; Hopkins, M.B.<br>*Ind. Eng. Chem.* 1930, *22*, 826-30. |
| **VARIABLES:** | **PREPARED BY:** |
| $T/K = 298$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of propanoic acid butyl ester in water at 25°C was reported to be 0.2 mL(1)/100mL(2).

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The method was not specified. | (1) Source not specified, commercial samples; used as received.<br>(2) Not specified. |
| | **ESTIMATED ERROR:** |
| | Not specified. |
| | **REFERENCES:** |

COMPONENTS:

(1) Propanoic acid butyl ester (*butyl propionate*); $C_7H_{14}O_2$; [590-01-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of propanoic acid butyl ester in water at 20°C was reported to be 0.15 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $2.1 \times 10^{-4}$.

The solubility of water in propanoic acid butyl ester at 20°C was reported to be 0.80 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.055.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 99%, b.p. range 124-171°C, $d_4^{20}$ 0.874.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Propanoic acid butyl ester (*butyl propionate*); $C_7H_{14}O_2$; [590-01-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Bomshtein, A.L.; Trofimov, A.N.; Serafimov, L.A.

*Zh. Prikl. Khim.* 1984, *57* 18-23.

VARIABLES:

$T/K = 368$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of propanoic acid butyl ester in water at 94.8°C[a] was reported to be $x_1 = 0.002$. The corresponding mass per cent value calculated by the compiler is 1.4 g(1)/100g sln.

The solubility of water in propanoic acid butyl ester at 94.8°C[a] was reported to be $x_2 = 0.220$. The corresponding mass per cent value calculated by the compiler is 3.76 g(2)/100g sln.

[a] Boiling temperature at 101.32 kPa.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The data and method were reported together with the ternary system propanoic acid butyl ester-water-acetic acid. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; distilled; without impurities by glc, b.p. 145.3°C, $n_D^{20}$ 1.4011.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Propanoic acid butyl ester (*butyl propionate*); $C_7H_{14}O_2$; [590-01-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of propanoic acid butyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.298 | 99.52 | 0.000413 | 0.9663 |
| 9.4 | 0.230 | 99.47 | 0.000319 | 0.9629 |
| 19.8 | 0.197 | 99.38 | 0.000273 | 0.9568 |
| 30.6 | 0.167 | 99.14 | 0.000231 | 0.9410 |
| 40.2 | 0.144 | 99.15 | 0.000199 | 0.9417 |
| 50.0 | 0.146 | 99.15 | 0.000202 | 0.9417 |
| 59.9 | 0.142 | 99.11 | 0.000197 | 0.9390 |
| 70.1 | 0.140 | 99.08 | 0.000194 | 0.9371 |
| 80.2 | 0.147 | 99.07 | 0.000204 | 0.9365 |
| 90.4 | 0.142 | 99.00 | 0.000197 | 0.9320 |
| std. dev. | 0.003 | 0.01 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Propanoic acid, 2,2-dimethyl-, ethyl ester (*ethyl trimethylacetate*); $C_7H_{14}O_2$; [3938-95-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of 2,2-dimethylpropanoic acid ethyl ester and water

| $t/^\circ C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.304 | 99.65 | 0.000422 | 0.9752 |
| 9.5 | 0.298 | 99.57 | 0.000413 | 0.9697 |
| 20.0 | 0.183 | 99.49 | 0.000253 | 0.9643 |
| 30.7 | 0.168 | 99.39 | 0.000233 | 0.9575 |
| 40.1 | 0.135 | 99.33 | 0.000187 | 0.9535 |
| 50.0 | 0.152 | 99.29 | 0.000210 | 0.9508 |
| 60.2 | 0.126 | 99.24 | 0.000174 | 0.9475 |
| 70.3 | 0.120 | 99.22 | 0.000166 | 0.9462 |
| 80.4 | 0.124 | 99.18 | 0.000172 | 0.9436 |
| 90.4 | 0.112 | 99.19 | 0.000155 | 0.9443 |
| std. dev. | 0.004 | 0.01 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 98%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Acetic acid 3-methoxy-1-butyl ester (*3-methoxybutyl acetate*); $C_7H_{14}O_3$; [4435-53-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T$/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 3-methoxy-1-butyl ester in water at 20°C was reported to be 6.46 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.00844.

The solubility of water in acetic acid 3-methoxy-1-butyl ester at 20°C was reported to be 3.72 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.239.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 99%, b.p. range 135-173°C, $d_4^{20}$ 0.956.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Propanoic acid, 2-hydroxy-, 1-butyl ester (*butyl lactate*); $C_7H_{14}O_3$; [138-22-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 2-hydroxypropanoic acid 1-butyl ester in water at 20°C was reported to be 4.0 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.0051.

The solubility of water in 2-hydroxypropanoic acid 1-butyl ester at 20°C was reported to be 14.5 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.579.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 97%, b.p. range 145-230°C, $d_4^{20}$ 0.980.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid phenyl ester (*phenyl acetate*); $C_8H_8O_2$; [122-79-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Richon, D.; Viallard, A.

*Fluid Phase Equilib.* 1985, *21*, 279-93.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid phenyl ester in water at 298.1 K was reported to be 0.00416 mol(1)/100 g(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.558 g(1)/100g sln and 7.42 x $10^{-4}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The refractometric method was used. The Phoenix model 1-2000T differential refractometer from Texas Instruments was used and the solubility was determined from a characteristic calibration curve as described in the thesis of Richon (ref 1).

SOURCE AND PURITY OF MATERIALS:

(1) Merck (for synthesis); purified by preparation gas chromatographic method; purity >99.5%, water content was negligible.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. Richon, D. *Thesis*, University de Clermont-Ferrand, 1974.

COMPONENTS:

(1) Benzoic acid methyl ester (*methyl benzoate*); $C_8H_8O_2$; [93-58-3]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the benzoic acid methyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Benzoic acid methyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Gilbert and Lauer (ref 1) | 288-308 | mutual | titration |
| Richon and Viallard (ref 2) | 298 | (1) in (2) | refractometric |
| Stephenson and Stuart (ref 3) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF BENZOIC ACID METHYL ESTER (1) IN WATER (2)

All the available data for the solubility of benzoic acid methyl ester (1) in water (2) are summarized in Table 2 except that the approximate values of Gilbert and Lauer (ref 1) have been rejected because they are in general much higher than the other studies (ref 2,3).

At 298 K, the only temperature where comparison is possible, the data of Richon and Viallard (ref 2) and Stephenson and Stuart (ref 3) are in good agreement enabling the average value to be Recommended. At other temperatures only the data of Stephenson and Stuart (ref 3) are available and, pending further studies, they must be regarded as Tenative although it may be noted that the solubilities of these authors are usually reliable.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Benzoic acid methyl ester (*methyl benzoate*); $C_8H_8O_2$; [93-58-3]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Recommended (*R*) and Tentative Solubilities of Benzoic acid methyl ester (1) in Water (2)

| *T*/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 0.221 (ref 3) | 0.22 | 2.9 |
| 283 | 0.220* (ref 3) | 0.22 | 2.9 |
| 293 | 0.223* (ref 3) | 0.22 | 2.9 |
| 298 | 0.202 (ref 2), 0.227* (ref 3) | 0.215 ± 0.013[b] (*R*) | 2.85[b] |
| 303 | 0.233* (ref 3) | 0.23 | 3.0 |
| 313 | 0.245* ref 3) | 0.25 | 3.3 |
| 323 | 0.264* (ref 3) | 0.26 | 3.4 |
| 333 | 0.287* (ref 3) | 0.29 | 3.8 |
| 343 | 0.315* (ref 3) | 0.32 | 4.3 |
| 353 | 0.347* (ref 3) | 0.35 | 4.7 |
| 363 | 0.405* (ref 3) | 0.41 | 5.4 |

[a] Rounded values of ref 3 except at 298 K.

[b] Average value; $x_1$ has the same status and (relative) percentage uncertainty as the mass % solubility.

2. SOLUBILITY OF WATER (2) IN BENZOIC ACID METHYL ESTER (1)

All the data for the solubility of water (2) in benzoic acid methyl ester (1) are summarized in Table 3. The two data sets (ref 1,3) are in fair agreement but in the absence of other independent studies the reported solubilities must be regarded as Tentative since the aqueous phase data of Gilbert and Lauer (ref 1) were rejected (see above). However, it may be noted that the data of Stephenson and Stuart (ref 3) are usually reliable.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Benzoic acid methyl ester (*methyl benzoate*); $C_8H_8O_2$; [93-58-3]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 3: Tentative Solubilities of Water (2) in Benzoic acid methyl ester (1)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(2)/100g sln | g(2)/100g sln | $10^2x_2$ |
| 273 | 0.46 (ref 3) | 0.5 | 4 |
| 283 | $0.54^*$ (ref 3) | 0.5 | 4 |
| 293 | $0.4^*$ (ref 1), $0.62^*$ (ref 3) | 0.6 | 5 |
| 298 | $0.6^*$ (ref 1), $0.67^*$ (ref 3) | 0.6 | 5 |
| 303 | $0.9^*$ (ref 1), $0.73^*$ (ref 3) | 0.7 | 5 |
| 313 | $0.87^*$ (ref 3) | 0.9 | 6 |
| 323 | 1.02 (ref 3) | 1.0 | 7 |
| 333 | $1.18^*$ (ref 3) | 1.2 | 9 |
| 343 | $1.37^*$ (ref 3) | 1.4 | 10 |
| 353 | $1.60^*$ (ref 3) | 1.6 | 11 |
| 363 | $1.96^*$ (ref 3) | 2.0 | 14 |

[a] Rounded values of ref 3, see text.

REFERENCES

1. Gilbert, E. C.; Lauer, B. E. *J. Phys. Chem.* 1927, *31*, 1050-2.

2. Richon, D.; Viallard, A. *Fluid Phase Equil.* 1985, *21*, 279-93.

3. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

COMPONENTS:

(1) Benzoic acid methyl ester (*methyl benzoate*); $C_8H_8O_2$; [93-58-3]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gilbert, E.C.; Lauer, B.E.

*J. Phys. Chem.* 1927, *31*, 1050-2.

VARIABLES:

$T/K = 288 - 308$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of benzoic acid methyl ester and water

| $t/^\circ C$ | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 15 | 0.1 | 99.8 | 0.00013 | 0.985 |
| 25 | 0.4 | 99.4 | 0.00053 | 0.956 |
| 35 | 0.5 | 98.8 | 0.00066 | 0.916 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used as described by Walton and Jenkins (ref 1). A measured quantity of one component in a large test tube placed in a transparent constant temperature bath was titrated from a calibrated burette. The mixture was stirred rapidly with a motor stirrer and the equilibrium end point taken at the instant the solution became clear (with a lamp on the opposite side). The end points were sharp and easily duplicated. The temperature of the burette was recorded and from the density of the material the weight was computed. The solutions were carefully protected from moisture. The mutual solubility was determined from the densities of the respective saturated solutions on the assumption that mixing laws were valid in the range of low concentrations involved. Several runs were made at each temperature.

SOURCE AND PURITY OF MATERIALS:

(1) Eastman Kodak Company, chemically pure grade; redistilled under reduced pressure, a fraction boiling 88-91°C at 17mm Hg being collected.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. Walton, J.H.; Jenkins, J.D. *J. Am. Chem. Soc.* 1923, *45*, 2555.

COMPONENTS:

(1) Benzoic acid methyl ester (*methyl benzoate*); $C_8H_8O_2$; [93-58-3]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Richon, D.; Viallard, A.

*Fluid Phase Equilib.* 1985, *21*, 279-93.

VARIABLES:

$T/K = 298$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of benzoic acid methyl ester in water at 298.1 K was reported to be 0.00149 mol(1)/100 g(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.202 g(1)/100g sln and $2.68 \times 10^{-4}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The refractometric method was used. The Phoenix model 1-2000T differential refractometer from Texas Instruments was used and the solubility was determined from a characteristic calibration curve as described in the thesis of Richon (ref 1).

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; purified by preparation gas chromatographic method; purity >99.5%, water content was negligible.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. Richon, D. *Thesis*, University de Clermont-Ferrand, 1974.

COMPONENTS:

(1) Benzoic acid methyl ester (*methyl benzoate*); $C_8H_8O_2$; [93-58-3]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of benzoic acid methyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln<br>(2)-rich phase | <br>(1)-rich phase | $x_1$ (compiler)<br>(2)-rich phase | <br>(1)-rich phase |
|---|---|---|---|---|
| 0 | 0.221 | 99.54 | 0.000293 | 0.9662 |
| 9.7 | 0.221 | 99.46 | 0.000293 | 0.9606 |
| 20.1 | 0.213 | 99.35 | 0.000282 | 0.9529 |
| 29.6 | 0.282 | 99.27 | 0.000374 | 0.9473 |
| 40.2 | 0.247 | 99.16 | 0.000327 | 0.9398 |
| 49.8 | 0.258 | 99.02 | 0.000342 | 0.9304 |
| 60.1 | 0.286 | 98.76 | 0.000379 | 0.9133 |
| 70.2 | 0.325 | 98.62 | 0.000431 | 0.9043 |
| 80.3 | 0.358 | 98.44 | 0.000475 | 0.8930 |
| 90.5 | 0.408 | 97.96 | 0.000542 | 0.8640 |
| std. dev. | 0.002 | 0.02 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Benzoic acid, 2-hydroxy-, methyl ester (*methyl salicylate*); $C_8H_8O_3$; [119-36-8]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>March, 1990 |

CRITICAL EVALUATION:

Quantitative solubility data for the 2-hydroxybenzoic acid methyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the 2-Hydroxybenzoic acid methyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Seidell (ref 1) | 288-303 | (1) in (2) | titration |
| Stephenson and Stuart (ref 2) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF 2-HYDROXYBENZOIC ACID METHYL ESTER (1) IN WATER (2)

All the available quantitative solubility data for the solubility of 2-hydroxybenzoic acid methyl ester (1) in water (2) are summarized in Table 2.

At 298 K the data of Seidell (ref 1) and Stephenson and Stuart (ref 2) are in excellent agreement. Furthermore Seidell reported the solubility of (1) in (2) was constant over the range 288-303 K which is consistent, within the limits of his likely experimental errors, with the more recent study of Stephenson and Stuart. This gives confidence in the reliability of the data of Stephenson and Stuart at other temperatures although, in the absence of confirmatory studies, their solubilities at temperatures other than 298 K must be regarded as Tentative.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Benzoic acid, 2-hydroxy-, methyl ester (*methyl salicylate*); $C_8H_8O_3$; [119-36-8]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>March, 1990 |

CRITICAL EVALUATION: (continued)

TABLE 2: Recommended (*R*) and Tentative Solubilities of 2-Hydroxybenzoic acid methyl ester (1) in Water (2)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 0.12* (ref 2) | 0.12 | 1.4 |
| 283 | 0.12* (ref 2) | 0.12 | 1.4 |
| 293 | 0.12* (ref 2) | 0.12 | 1.4 |
| 298 | 0.12 (ref 1), 0.12* (ref 2) | 0.12 (*R*) | 1.4 |
| 303 | 0.13 (ref 2) | 0.13 | 1.5 |
| 313 | 0.15 (ref 2) | 0.15 | 1.8 |
| 323 | 0.17* (ref 2) | 0.17 | 2.0 |
| 333 | 0.20* (ref 2) | 0.20 | 2.4 |
| 343 | 0.22* (ref 2) | 0.22 | 2.6 |
| 353 | 0.25* (ref 2) | 0.25 | 2.9 |
| 363 | 0.27* (ref 2) | 0.27 | 3.2 |

[a] Values of ref 2, see text.

2. SOLUBILITY OF WATER (2) IN 2-HYDROXYBENZOIC ACID METHYL ESTER (1)

The only data available for the solubility of water (2) in 2-hydroxybenzoic acid methyl ester (1) are those of Stephenson and Stuart (ref 2) and so no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheet for the original solubility values but it may be noted that the data of these authors are usually reliable.

REFERENCES

1. Seidell, A. *Hygienic Lab. Bull.* 1910, No. 67, 98pp. (U.S. Govt. Printing Office, Washington, DC).
2. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

| | |
|---|---|
| COMPONENTS:<br>(1) Benzoic acid, 2-hydroxy-, methyl ester (*methyl salicylate*); $C_8H_8O_3$; [119-36-8]<br>(2) Water; $H_2O$; [7732-18-5] | ORIGINAL MEASUREMENTS:<br>Seidell, A.<br>*Hygienic Lab. Bull.* 1910, No. 67, 98 pp (US Govt. Printing Office, Washington, DC). |
| VARIABLES:<br>$T/K = 288 - 303$ | PREPARED BY:<br>G.T. Hefter |

EXPERIMENTAL VALUES:

The solubility of 2-hydroxybenzoic acid methyl ester in water at 25°C was reported to be 0.12 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $1.4 \times 10^{-4}$.

The solubility of 2-hydroxybenzoic acid methyl ester at other temperatures (15-30°C) was reported to be constant with an approximate value of 0.1 $cm^3$(1)/100$cm^3$(2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. Portions (50 $cm^3$) of (2) were placed in an Erlenmeyer flask and titrated with (1) to opalescence. The flask and its contents were then cooled to the lowest temperature desired and allowed to warm slowly. The temperature was continuously recorded with a thermometer and the last drop or two of (1) was added as the desired temperature was reached.

SOURCE AND PURITY OF MATERIALS:

(1) Source not stated; purified by fractional distillation; b.p. 220-221°C, $d_{25}^{25}$ 1.182 g/$cm^3$.

(2) Distilled (no details given).

ESTIMATED ERROR:

Temp. not stated.
Soly. not stated.

REFERENCES:

COMPONENTS:

(1) Benzoic acid, 2-hydroxy-, methyl ester (*methyl salicylate*); $C_8H_8O_3$; [119-36-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of 2-hydroxybenzoic acid methyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.116 | 99.84 | 0.000137 | 0.9866 |
| 9.5 | 0.131 | 99.83 | 0.000155 | 0.9858 |
| 19.9 | 0.120 | 99.74 | 0.000142 | 0.9784 |
| 29.7 | 0.124 | 99.70 | 0.000147 | 0.9752 |
| 39.5 | 0.145 | 99.66 | 0.000172 | 0.9720 |
| 50.0 | 0.175 | 99.59 | 0.000207 | 0.9664 |
| 60.1 | - | 99.52 | - | 0.9608 |
| 70.2 | 0.221 | 99.40 | 0.000262 | 0.9515 |
| 80.1 | 0.270 | 99.25 | 0.000320 | 0.9400 |
| 90.5 | 0.270 | 99.20 | 0.000320 | 0.9362 |
| std. dev. | 0.004 | 0.01 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Acetic acid cyclohexyl ester (*cyclohexyl acetate*); $C_8H_{14}O_2$; [622-45-7]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the acetic acid cyclohexyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Acetic acid cyclohexyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Doolittle (ref 1) | 293 | mutual | unspecified |
| Othmer *et al.* (ref 2) | 297 | mutual | unspecified |
| Stephenson and Stuart (ref 3) | 273-363 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF ACETIC ACID CYCLOHEXYL ESTER (1) IN WATER (2)

All the available data for the solubility of acetic acid cyclohexyl ester (1) in water (2) are summarized in Table 2 with the exception of the datum of Doolittle (ref 1) which is substantially lower than other data (ref 2,3) and has therefore been rejected.

At 296.7 K, the only temperature where comparison is possible, the data of Othmer *et al.* (ref 2) and Stephenson and Stuart (ref 3) are in good agreement and the average value is Recommended. At other temperatures only the data of Stephenson and Stuart (ref 3) are available and, pending further studies, must be regarded as Tentative, although it may be noted that solubilities reported by these authors are usually reliable.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid cyclohexyl ester (*cyclohexyl acetate*); $C_8H_{14}O_2$; [622-45-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Recommended ($R$) and Tentative Solubilities of Acetic acid cyclohexyl ester (1) in Water (2)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 0.393 (ref 3) | 0.39 | 5.0 |
| 283 | 0.325* (ref 3) | 0.33 | 4.2 |
| 293 | 0.292* (ref 3) | 0.29 | 3.7 |
| 296.7 | 0.29 (ref 2), 0.282* (ref 3) | 0.286 ± 0.006 ($R$)[b] | 3.63[b] |
| 298 | 0.277* (ref 3) | 0.28 | 3.6 |
| 303 | 0.264* (ref 3) | 0.26 | 3.3 |
| 313 | 0.242* (ref 3) | 0.24 | 3.0 |
| 323 | 0.225* (ref 3) | 0.23 | 2.9 |
| 333 | 0.217* (ref 3) | 0.22 | 2.8 |
| 343 | 0.224* (ref 3) | 0.22 | 2.8 |
| 353 | 0.247* (ref 3) | 0.25 | 3.2 |
| 363 | 0.290* (ref 3) | 0.29 | 3.7 |

[a] Rounded values of ref 3 except at 296.7 K.

[b] Average value; $x_1$ has the same status and (relative) percentage uncertainty as the mass % solubility.

2. SOLUBILITY OF WATER (2) IN ACETIC ACID CYCLOHEXYL ESTER (1)

All the available solubility data for water (2) in acetic acid cyclohexyl ester (1) are summarized in Table 3. Serious disagreement exists between the values reported (at slightly different temperatures) by Doolittle (ref 1) and Othmer *et al.* (ref 2) on the one hand and Stephenson and Stuart (ref 3) on the other. In the absence of confirmatory studies it is not possible to distinguish between these data and thus no Critical Evaluation is possible, although it may be noted that the data of Stephenson and Stuart (ref 3) are usually reliable.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid cyclohexyl ester (*cyclohexyl acetate*); $C_8H_{14}O_2$; [622-45-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 3: Reported Solubilities of Water (2) in Acetic acid cyclohexyl ester (1)

| $T$/K | Solubilities<br>Reported values<br>g(2)/100g sln | "Best" values [a]<br>$10^2x_2$[a] |
|---|---|---|
| 273 | 0.67 (ref 3) | 5.1 |
| 283 | 0.65* (ref 3) | 5.0 |
| 293 | 1.42 (ref 1), 0.70* (ref 3) | 5.3 |
| 298 | 1.45[b] (ref 2), 0.74* (ref 3) | 5.6 |
| 303 | 0.80* (ref 3) | 6.0 |
| 313 | 0.91* (ref 3) | 6.8 |
| 323 | 1.00* (ref 3) | 7.4 |
| 333 | 1.05* (ref 3) | 7.7 |
| 343 | 1.07* (ref 3) | 7.9 |
| 353 | 1.15* (ref 3) | 8.4 |
| 363 | 1.36* (ref 3) | 9.8 |

[a] Rounded values of ref 3, see text.

[b] 296.7 K.

REFERENCES

1. Doolittle, A. K. *Ind. Eng. Chem.* 1935, *27*, 1169-79.
2. Othmer, D. F.; White, R. E.; Trueger, E. *Ind. Eng. Chem.* 1941, *33*, 1240-8; *ibid.* 1513.
3. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

COMPONENTS:

(1) Acetic acid cyclohexyl ester (*cyclohexyl acetate*); $C_8H_{14}O_2$; [622-45-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* <u>1935</u>, *27*, 1169-79.

VARIABLES:

$T$/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid cyclohexyl ester in water at 20°C was reported to be 0.16 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $2.0 \times 10^{-4}$.

The solubility of water in acetic acid cyclohexyl ester at 20°C was reported to be 1.42 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.102.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 79%, b.p. range 165-193°C, $d_4^{20}$ 0.963.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid cyclohexyl ester (*cyclohexyl acetate*); $C_8H_{14}O_2$; [622-45-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Othmer, D.F.; White, R.E.; Trueger, E.

*Ind. Eng. Chem.* 1941, *33*, 1240-8, 1513.

VARIABLES:

$T/K = 297$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid cyclohexyl ester in water at 23.5°C was reported to be 0.29 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $3.7 \times 10^{-4}$.

The solubility of water in acetic acid cyclohexyl ester at 23.5°C was reported to be 1.45 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.104.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing was specified in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.5°C (mean of reported range).

REFERENCES:

COMPONENTS:

(1) Acetic acid cyclohexyl ester (*cyclohexyl acetate*); $C_8H_{14}O_2$; [622-45-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 363

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid cyclohexyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.393 | 99.33 | 0.000499 | 0.9494 |
| 9.4 | 0.317 | 99.35 | 0.000403 | 0.9509 |
| 19.8 | 0.304 | 99.29 | 0.000386 | 0.9466 |
| 29.7 | 0.260 | 99.21 | 0.000330 | 0.9408 |
| 39.6 | 0.251 | 99.10 | 0.000319 | 0.9331 |
| 49.8 | 0.229 | 99.01 | 0.000291 | 0.9268 |
| 60.1 | 0.218 | 98.95 | 0.000277 | 0.9227 |
| 70.0 | 0.224 | 98.93 | 0.000284 | 0.9213 |
| 80.1 | 0.257 | 98.85 | 0.000326 | 0.9159 |
| 90.3 | 0.286 | 98.64 | 0.000363 | 0.9018 |
| std. dev. | 0.002 | 0.01 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Butanedioic acid diethyl ester (*diethyl succinate*); $C_8H_{14}O_4$; [123-25-1]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the butanedioic acid diethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Butanedioic acid diethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Sobotka and Kahn (ref 1) | 293 | (1) in (2) | titration |
| Stephenson and Stuart (ref 2) | 274-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF BUTANEDIOIC ACID DIETHYL ESTER (1) IN WATER (2)

All the available data for the solubility of butanedioic acid diethyl ester (1) in water (2) are summarized in Table 2. At 293 K, the only temperature where comparison is possible, the data of Sobotka and Kahn (ref 1) and Stephenson and Stuart (ref 2) are in good agreement and the average value is Recommended. At other temperatures only the data of Stephenson and Stuart (ref 2) are available and must therefore be regarded as Tentative although it may be noted that the solubilities reported by these authors are generally reliable.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanedioic acid diethyl ester (*diethyl succinate*); $C_8H_{14}O_4$; [123-25-1]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Recommended (*R*) and Tentative Solubilities of Butanedioic acid diethyl ester (1) in Water (2)

| $T$/K | Solubilities |  |  |
|---|---|---|---|
|  | Reported values | "Best" values [a] |  |
|  | g(1)/100g sln | g(1)/100g sln | $10^3x_1$ |
| 273 | 2.86 (ref 2) | 2.9 | 3.0 |
| 283 | 2.50* (ref 2) | 2.5 | 2.6 |
| 293 | 1.89 (ref 1), 2.17* (ref 2) | 2.03 ± 0.14 (*R*)[b] | 2.14[b] |
| 298 | 2.15* (ref 2) | 2.2 | 2.3 |
| 303 | 1.95* (ref 2) | 2.0 | 2.1 |
| 313 | 1.81* (ref 2) | 1.8 | 1.9 |
| 323 | 1.75* (ref 2) | 1.8 | 1.9 |
| 333 | 1.75* (ref 2) | 1.8 | 1.9 |
| 343 | 1.78* (ref 2) | 1.8 | 1.9 |
| 353 | 1.86* (ref 2) | 1.9 | 2.0 |
| 363 | 2.05* (ref 2) | 2.1 | 2.2 |

[a] Rounded values of ref 2 except at 293 K.

[b] Average value; $x_1$ has the same status and (relative) percentage uncertainty as the mass % solubility.

2. SOLUBILITY OF WATER (2) IN BUTANEDIOIC ACID DIETHYL ESTER (1)

The only data available for the solubility of water (2) in butanedioic acid diethyl ester (1) are those of Stephenson and Stuart (ref 2) and so no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheet for the experimental solubilities but it may be noted that the data of these authors are usually reliable.

REFERENCES

1. Sobotka, H.; Kahn, J. *J. Am. Chem. Soc.* 1931, *53*, 2935-8.
2. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Butanedioic acid diethyl ester (*diethyl succinate*); $C_8H_{14}O_4$; [123-25-1]<br>(2) Water; $H_2O$; [7732-18-5] | Sobotka, H.; Kahn, J.<br>*J. Am. Chem. Soc.* 1931, *53*, 2935-8. |
| VARIABLES: | PREPARED BY: |
| $T$/K = 293 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of butanedioic acid diethyl ester in water at 20°C was reported to be 1.92 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 1.89 g(1)/100g sln and 0.00199.

Density of water $d_4^{20}$ = 0.9982 (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 1.0402, $n_D^{20}$ 1.4200.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Butanedioic acid diethyl ester (*diethyl succinate*); $C_8H_{14}O_4$; [123-25-1]<br>(2) Water; $H_2O$; [7732-18-5] | Stephenson, R.; Stuart, J.<br>*J. Chem. Eng. Data* 1986, *31*, 56-70. |
| **VARIABLES:** | **PREPARED BY:** |
| $T$/K = 273 - 364 | Z. Maczynska |

EXPERIMENTAL VALUES:

Mutual solubility of butanedioic acid diethyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 2.86 | 98.72 | 0.00303 | 0.8886 |
| 10.0 | 2.67 | 98.49 | 0.00283 | 0.8709 |
| 20.0 | 2.13 | 98.22 | 0.00225 | 0.8509 |
| 29.7 | 1.99 | 97.92 | 0.00209 | 0.8296 |
| 39.8 | 1.79 | 97.58 | 0.00188 | 0.8066 |
| 50.0 | 1.79 | 97.13 | 0.00188 | 0.7778 |
| 60.3 | 1.74 | 96.76 | 0.00183 | 0.7554 |
| 70.0 | 1.79 | 96.40 | 0.00188 | 0.7347 |
| 80.1 | 1.84 | 96.03 | 0.00193 | 0.7144 |
| 90.7 | 2.09 | 95.77 | 0.00220 | 0.7007 |
| std. dev. | 0.05 | 0.07 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) 2,3-Butanediol diacetate
*(2,3-butylene diacetate)*;
$C_8H_{14}O_4$; [1114-92-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Othmer, D.F.; Bergen, W.S.;
Shlechter, N.; Bruins, P.F.

*Ind. Eng. Chem.* 1945, *37*, 890-4.

VARIABLES:

$T/K = 299 - 348$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of 2,3-Butanediol diacetate and water

| $t/^{\circ}C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 26 | 3.62 | 96.3 | 0.00387 | 0.729 |
| 50 | 2.87 | 96.6 | 0.00305 | 0.746 |
| 75 | 3.7 | 95.9 | 0.00396 | 0.708 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Presumably the titration method reported by Othmer, White and Trueger (ref 1) was used. The data were reported together with the ternary system 2,3-Butanediol diacetate-water-2,3-butanediol (*2,3-butylene diacetate-water-butylene glycol*).

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized from meso-2,3-butylene glycol; b.p. range 1°C.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. Othmer, D.F.; White, R.E.; Trueger, E. *Ind. Eng. Chem.* 1941, *33*, 1240.

COMPONENTS:

(1) Hexanedioic acid dimethyl ester (*dimethyl adipate*); $C_8H_{14}O_4$; [627-93-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 282 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of hexanedioic acid dimethyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 9.2 | 3.81 | 98.06 | 0.00408 | 0.8394 |
| 19.6 | 3.07 | 97.70 | 0.00326 | 0.8146 |
| 30.9 | 2.85 | 97.33 | 0.00302 | 0.7903 |
| 39.5 | 2.77 | 96.93 | 0.00294 | 0.7655 |
| 49.8 | 2.70 | 96.34 | 0.00286 | 0.7313 |
| 60.0 | 2.73 | 95.90 | 0.00289 | 0.7075 |
| 70.5 | 2.87 | 95.27 | 0.00305 | 0.6756 |
| 80.2 | 3.50 | 94.59 | 0.00374 | 0.6439 |
| 90.5 | 3.23 | 94.28 | 0.00344 | 0.6302 |
| std. dev. | 0.06 | 0.06 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 98%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid 1,3-dimethylbutyl ester *(1,3-dimethylbutyl acetate)*; $C_8H_{16}O_2$; [108-84-9]<br>(2) Water; $H_2O$; [7732-18-5] | Doolittle, A.K.<br>*Ind. End. Chem.* 1935, *27*, 1169-79. |
| **VARIABLES:** | **PREPARED BY:** |
| $T/K = 293$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of acetic acid 1,3-dimethylbutyl ester in water at 20°C was reported to be 0.08 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $1.0 \times 10^{-4}$.

The solubility of water in acetic acid 1,3-dimethylbutyl ester at 20°C was reported to be 0.89 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.067.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 95%, b.p. range 140-147°C, $d_4^{20}$ 0.857, $n_D^{20}$ 1.4008.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid 2-ethyl-1-butyl ester (*2-ethylbutyl acetate*); $C_8H_{16}O_2$; [10031-87-5]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 2-ethyl-1-butyl ester in water at 20°C was reported to be 0.06 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $7 \times 10^{-5}$.

The solubility of water in acetic acid 2-ethyl-1-butyl ester at 20°C was reported to be 0.75 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.057.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 91%, b.p. range 157-164°C, $d_4^{20}$ 0.876, $n_D^{20}$ 1.4103.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid hexyl ester (*hexyl acetate*); $C_8H_{16}O_2$; [142-92-7]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the acetic acid hexyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Acetic acid hexyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Massaldi and King (ref 1) | 298 | (1) in (2) | GLC |
| Alvarez and Neila (ref 2) | 298 | mutual | titration |
| Stephenson and Stuart (ref 3) | 273-363 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

## 1. SOLUBILITY OF ACETIC ACID HEXYL ESTER (1) IN WATER (2)

All the available data for the solubility of acetic acid hexyl ester (1) in water (2) are summarized in Table 2 with the exception of the approximate value of Alvarez and Neila (ref 2) which is much higher than the other studies (ref 1,3) and is therefore rejected. At 298 K, the only temperature where comparison is possible, the data of Massaldi and King (ref 1) and Stephenson and Stuart (ref 3) are in excellent agreement and the average value is Recommended. At other temperatures only the values of Stephenson and Stuart (ref 3) are available. Although their results for this system are rather scattered, the solubilities reported by these authors are generally reliable and pending further studies can be considered as Tentative.

(continued next page)

COMPONENTS:

(1) Acetic acid hexyl ester (*hexyl acetate*); $C_8H_{16}O_2$; [142-92-7]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION: (continued)

TABLE 2: Recommended (*R*) and Tentative Solubilities of Acetic acid hexyl ester (1) in Water (2)

| *T*/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^5 x_1$ |
| 273 | 0.084 (ref 3) | 0.08 | 10 |
| 283 | 0.066 (ref 3) | 0.07 | 9 |
| 293 | 0.056* (ref 3) | 0.06 | 7 |
| 298 | 0.051 (ref 1), 0.053 (ref 3) | 0.052 ± 0.001 (*R*)[b] | 6.5 |
| 303 | 0.052* (ref 3) | 0.05 | 6 |
| 313 | 0.051* (ref 3) | 0.05 | 6 |
| 323 | 0.051* (ref 3) | 0.05 | 6 |
| 333 | 0.052* (ref 3) | 0.05 | 6 |
| 343 | 0.056* (ref 3) | 0.06 | 7 |
| 353 | 0.060* (ref 3) | 0.06 | 7 |
| 363 | 0.066* (ref 3) | 0.07 | 9 |

[a] Rounded values of ref 3 except at 298 K.

[b] Average value; $x_1$ has same status and (relative) percentage uncertainty as the mass % solubility.

2. SOLUBILITY OF WATER (2) IN ACETIC ACID HEXYL ESTER (1)

All the available data for the solubility of water (2) in acetic acid hexyl ester (1) are listed in Table 3. At 298 K, the only temperature where comparison is possible, the approximate value of Alvarez and Neila (ref 2) is not in very good agreement with the interpolated datum of Stephenson and Stuart (ref 3). Consequently, in the absence of other independent studies, all the data must be regarded as very Tentative. However, it may be noted that the solubilities reported by Stephenson and Stuart (ref 3) are generally reliable although the present values are rather scattered.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid hexyl ester (*hexyl acetate*); $C_8H_{16}O_2$; [142-92-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 3: Tentative Solubilities of Water (2) in Acetic acid hexyl ester (1)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(2)/100g sln | g(2)/100g sln | $10^2x_2$ |
| 273 | 0.494 (ref 3) | 0.5 | 4 |
| 283 | $0.54^*$ (ref 3) | 0.5 | 4 |
| 293 | $0.58^*$ (ref 3) | 0.6 | 5 |
| 298 | $1.1^b$ (ref 2), $0.61^*$ (ref 3) | 0.6 | 5 |
| 303 | $0.63^*$ (ref 3) | 0.6 | 5 |
| 313 | $0.66^*$ (ref 3) | 0.7 | 5 |
| 323 | $0.69^*$ (ref 3) | 0.7 | 5 |
| 333 | $0.72^*$ (ref 3) | 0.7 | 5 |
| 343 | $0.74^*$ (ref 3) | 0.7 | 5 |
| 353 | $0.76^*$ (ref 3) | 0.8 | 6 |
| 363 | $0.78^*$ (ref 3) | 0.8 | 6 |

[a] Rounded values of ref 3, see text.

[b] Not included in "Best" value.

REFERENCES

1. Massaldi, H. A.; King, C. J. *J. Chem. Eng. Data* 1973, *18*, 393-7.
2. Alvarez, J. R.; Neila, J. J. *An. Quim.* 1978, *74*, 326-32.
3. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

COMPONENTS:

(1) Acetic acid hexyl ester (*hexyl acetate*); $C_8H_{16}O_2$; [142-92-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Massaldi, H.A.; King, C.J.

*J. Chem. Eng. Data* 1973, *18*, 393-7.

VARIABLES:

$T/K = 298$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

The solubility of acetic acid hexyl ester in water at 25°C was reported to be 0.00354 mol(1)/L sln.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. A technique based on head-space analysis using gas-liquid chromatography was developed to determine solubilities of sparingly soluble organics. Saturated solutions need not be prepared in advance; thus, phase separation problems were avoided. Furthermore, the analysis of liquid samples was not required. This method is versatile enough to give determinations provided that the vapor pressure of the pure liquid substance is known. The gas chromatograph was a Varian Aerograph Model 1740 with a flame ionization detector.

SOURCE AND PURITY OF MATERIALS:

(1) Eastman Kodak Co., reagent grade; purity not specified; used as received.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.05°C.

REFERENCES:

COMPONENTS:

(1) Acetic acid hexyl ester *(hexyl acetate)*; $C_8H_{16}O_2$; [142-92-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Alvarez, J.R.; Neila, J.J.

*An. Quim.* 1978, *74*, 326-32.

VARIABLES:

$T/K = 298$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

The solubility of acetic acid hexyl ester in water at 25°C was reported to be 0.1 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.00013.

The solubility of water in acetic acid hexyl ester at 25°C was reported to be 1.1 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.082.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Presumably the titration method was used. The data were reported together with the ternary system acetic acid hexyl ester-water-phenol. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; used as received; $d_4^{21.3}$ 0.8681, $n_D^{20}$ 1.4093.

(2) Twice distilled over $KMnO_4$.

ESTIMATED ERROR:

Not specified.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid hexyl ester (*hexyl acetate*); $C_8H_{16}O_2$; [142-92-7]<br>(2) Water; $H_2O$; [7732-18-5] | Stephenson, R.; Stuart, J.<br>*J. Chem. Eng. Data* 1986, *31*, 56-70. |
| **VARIABLES:** | **PREPARED BY:** |
| $T$/K = 273 - 363 | Z. Maczynska |

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid hexyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.084 | 99.506 | 0.000105 | 0.96177 |
| 9.5 | 0.070 | 99.504 | 0.000087 | 0.96162 |
| 19.7 | 0.043 | 99.434 | 0.000054 | 0.95641 |
| 29.6 | 0.054 | 99.270 | 0.000067 | 0.94441 |
| 40.0 | 0.056 | 99.254 | 0.000070 | 0.94325 |
| 50.0 | 0.047 | 99.367 | 0.000059 | 0.95148 |
| 60.7 | 0.051 | 99.305 | 0.000064 | 0.94695 |
| 70.0 | 0.057 | 99.251 | 0.000071 | 0.94303 |
| 80.0 | 0.063 | 99.220 | 0.000079 | 0.94079 |
| 90.2 | 0.066 | 99.215 | 0.000082 | 0.94044 |
| std. dev. | 0.003 | 0.004 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Acetic acid 1-methylpentyl ester (*1-methylpentyl acetate*); $C_8H_{16}O_2$; [5953-49-1]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the acetic acid 1-methylpentyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Acetic acid 1-methylpentyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Park and Hopkins (ref 1) | 298 | (1) in (2) | unspecified |
| Doolittle (ref 2) | 293 | mutual | unspecified |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation.

Unfortunately, the data for the solubility of acetic acid 1-methylpentyl ester (1) in water (2) in both the publications listed in Table 1 are only semiquantitative. According to Park and Hopkins (ref 1) the solubility of (1) in (2) is less than 0.14 g(1)/100 g sln. Doolittle (ref 2) merely indicates (1) is "very slightly soluble" in (2).

For the solubility of water (2) in 1-methylpentyl acetate (1) only the datum of Doolittle (ref 2), 0.68 g(2)/100 g sln, is available and so no Critical Evaluation is possible. This system clearly requires further investigation.

REFERENCES

1. Park, J. G.; Hopkins, M. B. *Ind. Eng. Chem.* 1930, *22*, 826-30.
2. Doolittle, A. K. *Ind. Eng. Chem.* 1935, *27*, 1169-79.

COMPONENTS:

(1) Acetic acid 1-methylpentyl ester (*1-methylpentyl acetate*); $C_8H_{16}O_2$; [5953-49-1]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Park, J.G.; Hopkins, M.B.

*Ind. Eng. Chem.* 1930, *22*, 826-30.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 1-methylpentyl ester in water at 25°C was reported to be less than 0.1 mL(1)/100mL(2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial samples; used as received; 85-88% of ester, b.p. range 146-156°C, $n_D^{20}$ 1.4081.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid 1-methylpentyl ester (*1-methylpentyl acetate*); $C_8H_{16}O_2$; [5953-49-1]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 1-methylpentyl ester in water at 20°C was reported to be very slightly soluble.

The solubility of water in acetic acid 1-methylpentyl ester at 20°C was reported to be 0.68 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.052.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 98%, b.p. range 129-158°C, $d_4^{20}$ 0.861.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Butanoic acid butyl ester (*butyl butyrate*); $C_8H_{16}O_2$; [109-21-7]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A., Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the butanoic acid butyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Butanoic acid butyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Bridgman (ref 1) | 283-306 | (2) in (1) | synthetic |
| Doolittle (ref 2) | 293 | mutual | unspecified |
| Chebotaev *et al.* (ref 3) | 370 | (2) in (1) | analytical |
| Stephenson and Stuart (ref 4) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF BUTANOIC ACID BUTYL ESTER (1) IN WATER (2)

All the available data for the solubility of butanoic acid butyl ester (1) in water (2) are summarized in Table 2. At 293 K, the only temperature where comparison is possible, the data of Doolittle (ref 2) and Stephenson and Stuart (ref 4) are in serous disagreement. In the absence of independent studies it is impossible to prefer either value although it may be noted that the data of Stephenson and Stuart (ref 4) are generally reliable.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid butyl ester<br>(*butyl butyrate*); $C_8H_{16}O_2$;<br>[109-21-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical<br>and Physical Sciences,<br>Murdoch University, Perth, W.A.,<br>Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Reported Solubilities
of Butanoic acid butyl ester (1) in Water (2)

| *T*/K | Reported Solubilities | |
|---|---|---|
| | g(1)/100g sln | $10^4 x_1{}^a$ |
| 273 | 1.22 (ref 4) | 15.4 |
| 283 | $0.90^*$ (ref 4) | 11.3 |
| 293 | 0.05 (ref 2), $0.66^*$ (ref 4) | 8.3 |
| 298 | $0.60^*$ (ref 4) | 7.5 |
| 303 | $0.56^*$ (ref 4) | 7.0 |
| 313 | $0.52^*$ (ref 4) | 6.5 |
| 323 | $0.49^*$ (ref 4) | 6.1 |
| 333 | $0.47^*$ (ref 4) | 5.9 |
| 343 | $0.47^*$ (ref 4) | 5.9 |
| 353 | $0.49^*$ (ref 4) | 6.1 |
| 363 | $0.51^*$ (ref 4) | 6.3 |

[a] Data of ref 4, see text.

## 2. SOLUBILITY OF WATER (2) IN BUTANOIC ACID BUTYL ESTER (1)

All the available data for the solubility of water (2) in butanoic acid butyl ester (1) are summarized in Table 3 with the exception of the value of Chebotaev *et al.* (ref 3) which is much higher than the other (ref 4) study and is rejected. Unlike the $H_2O$-rich phase the agreement between independent studies over the range 283-303 is good and a number of values have been Recommended. At other temperatures only the data of Stephenson and Stuart (ref 4) are available, excluding the datum of Chebotaev *et al.* (ref 3) referred to above, and must be considered Tentative pending further studies.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid butyl ester (*butyl butyrate*); $C_8H_{16}O_2$; [109-21-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 3: Recommended (*R*) and Tentative Solubilities of Water (2) in Butanoic acid butyl ester (1)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values $(\pm\sigma_n)^a$ | |
| | g(2)/100g sln | g(2)/100g sln | $10^2x_2$ |
| 273 | 0.339 (ref 4) | 0.34 | 2.7 |
| 283 | 0.379 (ref 1), 0.370* (ref 4) | 0.37 ± 0.01 (*R*) | 2.9 |
| 293 | 0.450* (ref 1), 0.43 (ref 2),<br>0.412* (ref 4) | 0.43 ± 0.02 (*R*) | 3.3 |
| 298 | 0.490* (ref 1), 0.438* (ref 4) | 0.46 ± 0.03 (*R*) | 3.5 |
| 303 | 0.533* (ref 1), 0.466 (ref 4) | 0.50 ± 0.03 (*R*) | 3.9 |
| 313 | 0.53* (ref 4) | 0.53 | 4.1 |
| 323 | 0.59* (ref 4) | 0.59 | 4.5 |
| 333 | 0.63* (ref 4) | 0.63 | 4.8 |
| 343 | 0.66* (ref 4) | 0.66 | 5.1 |
| 353 | 0.67* (ref 4) | 0.67 | 5.1 |
| 363 | 0.68* (ref 4) | 0.68 | 5.2 |

[a] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance. Mole fraction solubilities ($x_2$) have the same status and (relative) percentage uncertainties as the mass % solubilities.

REFERENCES

1. Bridgman, J. A. *Ind. Eng. Chem.* 1928, *20*, 184-7.
2. Doolittle, A. K. *Ind. Eng. Chem.* 1935, *27*, 1169-79.
3. Chebotaev, V. F.; Balashov, M. I.; Serafimov, L. A. *Fiz. Khim. Osn. Rektifikatsii* 1970, 100-3.
4. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

COMPONENTS:

(1) Butanoic acid butyl ester (*butyl butyrate*); $C_8H_{16}O_2$; [109-21-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Bridgman, J.A.

*Ind. End. Chem.* 1928, *20*, 184-7.

VARIABLES:

$T$/K = 283 - 306

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Solubility of water in butanoic acid butyl ester

| $t$/°C | g(2)/100g(1) | g(2)/100g solution (compiler) | $x_2$ (compiler) |
|---|---|---|---|
| 10.0 | 0.380 | 0.379 | 0.0296 |
| 16.0 | 0.424 | 0.422 | 0.0328 |
| 25.5 | 0.497 | 0.494 | 0.0382 |
| 33.0 | 0.574 | 0.570 | 0.0439 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The synthetic method similar to that described by Groschuff (ref 1) was used. A glass tube of about 100 mL capacity with a stopcock was filled with weighed (1) and (2). By shaking the tube after it had been warmed somewhat, the water was dissolved completely in the ester, after which the tube was placed in a 2-liter beaker filled with water and equipped with an agitator. By alternate slow cooling and warming, it was possible to determine, within about 0.5°C, the temperature at which water was precipitated from the solution, as shown by the clouding of the liquid which would clear again when the temperature was slightly increased.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; distilled; b.p. range 164.5-165.5°C; boiled for some time in open flask before used.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.25°C.

REFERENCES:

1. Groschuff, E. *Z. Elektrochem.* 1911, *17*, 348.

COMPONENTS:

(1) Butanoic acid butyl ester *(butyl butyrate)*; $C_8H_{16}O_2$; [109-21-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of butanoic acid butyl ester in water at 20°C was reported to be 0.05 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $6 \times 10^{-5}$.

The solubility of water in butanoic acid butyl ester at 20°C was reported to be 0.43 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.033.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 99%, b.p. range 152-170°C, $d_4^{20}$ 0.874.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Butanoic acid butyl ester (*butyl butyrate*); $C_8H_{16}O_2$; [109-21-7]<br>(2) Water; $H_2O$; [7732-18-5] | Chebotaev, V.F.; Balashov, M.I.; Serafimov, L.A.<br>*Fiz. Khim. Osn. Rektifikatsii* 1970, 100-3. |
| VARIABLES: | PREPARED BY: |
| $T/K = 370$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of water in butanoic acid butyl ester at 97.2°C[a] was reported to be 2.56 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.1449.

[a] Boiling temperature of two-phase mixture equal to 97.2°C was taken from Ogrodnikov, Lesteva and Kogan (ref 2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The standard methods were used as described by Alders (ref 1). The phase composition was determined by chemical and refractometry methods. No chemical reaction was observed. The data and method were reported together with ternary system butanoic acid butyl ester-water-butanoic acid (*butyl butyrate-water-butyric acid*).

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; distilled on a 20 TP column.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. Alders,A. *Zhidkostnaya Ekstraktsiya*, I.L., Moskva, 1962.
2. Ogrodnikov, S.K.; Lesteva, T.M.; Kogan,V.B. *Azeotropnye Smesi - spravochnik*, Khimiya, Leningrad, 1971.

COMPONENTS:

(1) Butanoic acid butyl ester (*butyl butyrate*); $C_8H_{16}O_2$; [109-21-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of butanoic acid butyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.122 | 99.661 | 0.000152 | 0.97349 |
| 9.5 | 0.102 | 99.630 | 0.000127 | 0.97113 |
| 20.0 | 0.066 | 99.560 | 0.000082 | 0.96583 |
| 29.7 | 0.056 | 99.573 | 0.000070 | 0.96681 |
| 39.6 | 0.052 | 99.460 | 0.000065 | 0.95835 |
| 50.0 | 0.051 | 99.423 | 0.000064 | 0.95560 |
| 59.9 | 0.047 | 99.358 | 0.000059 | 0.95082 |
| 70.2 | 0.047 | 99.349 | 0.000059 | 0.95016 |
| 80.0 | 0.049 | 99.320 | 0.000061 | 0.94804 |
| 90.3 | 0.051 | 99.317 | 0.000064 | 0.94782 |
| std. dev. | 0.001 | 0.004 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Formic acid heptyl ester (*heptyl formate*); $C_8H_{16}O_2$; [112-23-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of formic acid heptyl ester and water

| $t/^\circ C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | - | 99.32 | - | 0.9480 |
| 9.1 | - | 99.37 | - | 0.9517 |
| 19.5 | 0.162 | 99.32 | 0.000203 | 0.9480 |
| 29.7 | 0.142 | - | 0.000178 | - |
| 39.6 | 0.078 | 99.20 | 0.000097 | 0.9393 |
| 50.0 | 0.071 | 99.15 | 0.000089 | 0.9358 |
| 60.1 | 0.063 | 98.88 | 0.000079 | 0.9168 |
| 70.1 | 0.087 | 98.94 | 0.000109 | 0.9210 |
| 80.0 | 0.075 | 98.92 | 0.000094 | 0.9196 |
| 90.6 | 0.056 | 98.81 | 0.000070 | 0.9120 |
| std. dev. | 0.002 | 0.01 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 97%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

**COMPONENTS:**

(1) Heptanoic acid methyl ester (*methyl enanthate*); $C_8H_{16}O_2$; [106-73-0]

(2) Water; $H_2O$; [7732-18-5]

**ORIGINAL MEASUREMENTS:**

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

**VARIABLES:**

$T/K = 273 - 364$

**PREPARED BY:**

Z. Maczynska

**EXPERIMENTAL VALUES:**

Mutual solubility of heptanoic acid methyl ester and water

| $t/°C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.105 | 99.45 | 0.000131 | 0.9576 |
| 9.5 | 0.122 | 99.42 | 0.000152 | 0.9554 |
| 19.3 | 0.094 | 99.33 | 0.000117 | 0.9488 |
| 30.0 | 0.090 | 99.30 | 0.000112 | 0.9466 |
| 39.6 | 0.063 | 99.36 | 0.000079 | 0.9509 |
| 49.8 | - | 99.24 | - | 0.9422 |
| 60.5 | 0.054 | 99.27 | 0.000067 | 0.9444 |
| 70.5 | 0.087 | 99.28 | 0.000109 | 0.9451 |
| 80.5 | 0.066 | 99.24 | 0.000082 | 0.9422 |
| 90.5 | 0.053 | 99.13 | 0.000066 | 0.9343 |
| std. dev. | 0.001 | 0.01 | | |

## AUXILIARY INFORMATION

**METHOD/APPARATUS/PROCEDURE:**

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

**SOURCE AND PURITY OF MATERIALS:**

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

**ESTIMATED ERROR:**

Accuracy of method 0.1 wt% or less, for solubility, see above.

**REFERENCES:**

COMPONENTS:

(1) Hexanoic acid ethyl ester (*ethyl caproate*); $C_8H_{16}O_2$; [123-66-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sobotka, H.; Kahn, J.

*J. Am. Chem. Soc.* 1931, *53*, 2935-8.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of hexanoic acid ethyl ester in water at 20°C was reported to be 0.063 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.063 g(1)/100g sln and $7.9 \times 10^{-5}$.

Density of water $d_4^{20} = 0.9982$ (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $n_D^{20}$ 1.4089.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) Hexanoic acid ethyl ester (*ethyl caproate*); $C_8H_{16}O_2$; [123-66-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of hexanoic acid ethyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.107 | 99.64 | 0.000134 | 0.9719 |
| 9.6 | 0.102 | 99.61 | 0.000127 | 0.9696 |
| 19.7 | 0.083 | 99.53 | 0.000104 | 0.9636 |
| 29.6 | 0.075 | 99.49 | 0.000094 | 0.9606 |
| 39.8 | 0.070 | 99.44 | 0.000087 | 0.9568 |
| 50.0 | 0.060 | 99.42 | 0.000075 | 0.9554 |
| 60.6 | 0.049 | 99.38 | 0.000061 | 0.9524 |
| 70.5 | 0.055 | 99.34 | 0.000069 | 0.9495 |
| 80.4 | 0.055 | 99.29 | 0.000069 | 0.9458 |
| 90.6 | 0.059 | 99.26 | 0.000074 | 0.9437 |
| std. dev. | 0.007 | 0.01 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Propanoic acid, 2-methyl-, butyl ester
*(butyl isobutyrate)*; $C_8H_{16}O_2$; [97-87-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of 2-methylpropanoic acid butyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.090 | 99.64 | 0.000112 | 0.9719 |
| 9.8 | 0.071 | 99.63 | 0.000089 | 0.9711 |
| 19.6 | 0.077 | 99.57 | 0.000096 | 0.9666 |
| 30.9 | 0.076 | 99.61 | 0.000095 | 0.9696 |
| 40.1 | 0.051 | 99.53 | 0.000064 | 0.9636 |
| 50.0 | 0.043 | 99.51 | 0.000054 | 0.9621 |
| 60.0 | 0.048 | 99.42 | 0.000060 | 0.9554 |
| 70.0 | 0.043 | 99.34 | 0.000054 | 0.9495 |
| 80.2 | 0.044 | 99.35 | 0.000055 | 0.9502 |
| 90.5 | 0.043 | 99.32 | 0.000054 | 0.9480 |
| std. dev. | 0.003 | 0.01 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 98%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Propanoic acid 3-methyl-1-butyl ester (*isopentyl propionate*); $C_8H_{16}O_2$; [105-68-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of propanoic acid 3-methyl-1-butyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.097 | 99.57 | 0.000121 | 0.9666 |
| 9.3 | 0.100 | 99.57 | 0.000125 | 0.9666 |
| 19.3 | 0.065 | 99.52 | 0.000081 | 0.9628 |
| 29.7 | 0.056 | 99.46 | 0.000070 | 0.9583 |
| 40.0 | 0.063 | 99.40 | 0.000079 | 0.9539 |
| 49.8 | 0.045 | 99.47 | 0.000056 | 0.9591 |
| 60.0 | 0.056 | 99.40 | 0.000070 | 0.9539 |
| 70.0 | 0.075 | - | 0.000094 | - |
| 79.7 | 0.057 | 99.22 | 0.000071 | 0.9408 |
| 90.6 | 0.064 | 99.68 | 0.000080 | 0.9032 |
| std. dev. | 0.001 | 0.01 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Propanoic acid, 2-methyl-, 2-methylpropyl ester (*isobutyl isobutyrate*); $C_8H_{16}O_2$; [97-85-8]<br>(2) Water; $H_2O$; [7732-18-5] | Stephenson, R.; Stuart, J.<br>*J. Chem. Eng. Data* 1986, *31*, 56-70. |
| **VARIABLES:** | **PREPARED BY:** |
| $T/K$ = 273 - 363 | Z. Maczynska |

EXPERIMENTAL VALUES:

Mutual solubility of 2-methylpropanoic acid 2-methylpropyl ester and water

| $t/^\circ C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.098 | 99.57 | 0.000122 | 0.9666 |
| 9.3 | - | 99.48 | - | 0.9598 |
| 19.4 | 0.050 | 99.46 | 0.000062 | 0.9583 |
| 29.6 | 0.049 | 99.29 | 0.000061 | 0.9458 |
| 39.5 | 0.050 | 99.43 | 0.000062 | 0.9561 |
| 50.0 | 0.051 | 99.40 | 0.000064 | 0.9539 |
| 59.8 | 0.046 | 99.18 | 0.000057 | 0.9379 |
| 70.2 | 0.045 | 99.26 | 0.000056 | 0.9437 |
| 80.1 | 0.041 | 99.24 | 0.000051 | 0.9422 |
| 90.3 | 0.056 | 99.20 | 0.000070 | 0.9393 |
| std. dev. | 0.001 | 0.02 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 98%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Propanoic acid pentyl ester (*pentyl propionate*); $C_8H_{16}O_2$; [624-54-4]<br>(2) Water; $H_2O$; [7732-18-5] | Hemptinne, A.<br>*Z. Phys. Chem.* 1894, *13*, 561-9. |
| VARIABLES: | PREPARED BY: |
| $T/K = 298$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of propanoic acid pentyl ester in water at 25°C was reported to be 0.908 g(1)/L sln.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The analytical method was used. The mixture of water with excess ester was heated for some time in a water bath and the ester phase was filtered. A sample of known volume was then transferred to a smaller flask, heated with the known amount of baryta until complete saponification was obtained and then titrated. No further details were reported in the paper. | (1) Not specified.<br>(2) Not specified. |
| | ESTIMATED ERROR:<br>Not specified. |
| | REFERENCES: |

COMPONENTS:

(1) Acetic acid 2-butoxyethyl ester (*2-butoxyethyl acetate*); $C_8H_{16}O_3$; [112-07-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 2-butoxyethyl ester in water at 20°C was reported to be 0.9 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.0010.

The solubility of water in acetic acid 2-butoxyethyl ester at 20°C was reported to be 1.9 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.15.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 99%, b.p. range 188-192°C, $d_4^{20}$ 0.943.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid phenylmethyl ester (*benzyl acetate*); $C_9H_{10}O_2$; [140-11-4]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION:

Quantitative solubility data for the acetic acid phenylmethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Acetic acid phenylmethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Goto *et al*. (ref 1) | 363 | (1) in (2) | GLC |
| Stephenson and Stuart (ref 2) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF ACETIC ACID PHENYLMETHYL ESTER (1) IN WATER (2)

All the available data for the solubility of acetic acid phenylmethyl ester (1) in water (2) are summarized in Table 2. At 363 K, the only temperature where comparison is possible, the data of Goto *et al*. (ref 1) and Stephenson and Stuart (ref 2) are in fair agreement. At other temperatures only the data of Stephenson and Stuart (ref 2) are available and in the absence of other studies must be regarded as Tentative, although it may be noted that the solubilities reported by these authors are usually reliable.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid phenylmethyl ester *(benzyl acetate)*; $C_9H_{10}O_2$; [140-11-4]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Tentative Solubilities of Acetic acid phenylmethyl ester (1) in Water (2)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 0.253 (ref 2) | 0.25 | 3.0 |
| 283 | 0.253 (ref 2) | 0.25 | 3.0 |
| 293 | 0.255* (ref 2) | 0.26 | 3.1 |
| 298 | 0.258* (ref 2) | 0.26 | 3.1 |
| 303 | 0.262* (ref 2) | 0.26 | 3.1 |
| 313 | 0.277* (ref 2) | 0.28 | 3.3 |
| 323 | 0.305* (ref 2) | 0.31 | 3.6 |
| 333 | 0.338* (ref 2) | 0.34 | 4.0 |
| 343 | 0.372* (ref 2) | 0.37 | 4.5 |
| 353 | 0.409 (ref 2) | 0.41 | 4.9 |
| 363 | 0.58 (ref 1), 0.447* (ref 2) | 0.45 | 5.4 |

[a] Rounded values of ref 2, see text.

2. SOLUBILITY OF WATER (2) IN ACETIC ACID PHENYLMETHYL ESTER (1)

The only data available for the solubility of water (2) in acetic acid phenylmethyl ester (1) are those of Stephenson and Stuart (ref 2) and so no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheet for the experimental results but it may be noted that solubilities reported by these authors are usually reliable.

REFERENCES

1. Goto, S.; Matsubara, M.; Washino, K. *Kagaku Kogaku* 1974, *38*, 869-73.

2. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid phenylmethyl ester *(benzyl acetate)*; $C_9H_{10}O_2$; [140-11-4]<br>(2) Water; $H_2O$; [7732-18-5] | Goto, S.; Matsubara, M.; Washino, K.<br>*Kagaku Kogaku* 1974, *38(12)*, 869-73. |
| VARIABLES: | PREPARED BY: |
| $T$/K = 363 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of acetic acid phenylmethyl ester in water at 90°C was reported to be 0.57 and 0.58 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 6.9 x $10^{-4}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The samples of total volume about 50 $cm^3$ containing acetic acid phenylmethyl ester, water and tetralin were thermostated at 90°C, mixed and separated over 1 h. Then, the phases were analysed by glc. The method and data were reported together with the ternary system acetic acid phenylmethyl ester-water-tetralin. Tetralin was not detected in the water phase.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; purity >99 wt%; used as received.

(2) Distilled.

ESTIMATED ERROR:

Soly. <±0.01 g(1)/100g sln (compiler).

REFERENCES:

COMPONENTS:

(1) Acetic acid phenylmethyl ester (*benzyl acetate*); $C_9H_{10}O_2$; [140-11-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid phenylmethyl ester and water

| $t$/°C | g(1)/100g sln (2)-rich phase | g(1)/100g sln (1)-rich phase | $x_1$ (compiler) (2)-rich phase | $x_1$ (compiler) (1)-rich phase |
|---|---|---|---|---|
| 0 | 0.253 | 99.38 | 0.000304 | 0.9506 |
| 9.7 | 0.253 | 99.36 | 0.000304 | 0.9490 |
| 20.0 | 0.266 | 99.22 | 0.000320 | 0.9385 |
| 30.7 | 0.240 | 99.08 | 0.000288 | 0.9281 |
| 40.3 | 0.278 | 98.94 | 0.000334 | 0.9180 |
| 49.8 | 0.305 | 98.79 | 0.000367 | 0.9073 |
| 60.1 | 0.336 | 98.61 | 0.000404 | 0.8948 |
| 70.4 | 0.375 | 98.41 | 0.000451 | 0.8813 |
| 80.1 | 0.409 | 98.17 | 0.000492 | 0.8655 |
| 90.4 | 0.448 | 97.83 | 0.000539 | 0.8439 |
| std. dev. | 0.002 | 0.03 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Benzoic acid ethyl ester (*ethyl benzoate*); $C_9H_{10}O_2$; [93-89-0]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION:

Quantitative solubility data for the benzoic acid ethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Benzoic acid ethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Krupatkin and Glagoleva (ref 1) | 298 | mutual | titration |
| Stephenson and Stuart (ref 2) | 273-363 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF BENZOIC ACID ETHYL ESTER (1) IN WATER (2)

All the available data for the solubility of benzoic acid ethyl ester (1) in water (2) are summarized in Table 2. At 298 K, the only temperature where comparison is possible, the data of Krupatkin and Glagoleva (ref 1) and Stephenson and Stuart (ref 2) are in only fair agreement. At other temperatures only the data of Stephenson and Stuart (ref 2) are available. Although the data reported by these authors are usually reliable their results for the present system are very scattered and, pending further studies, should be regarded as very Tentative.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Benzoic acid ethyl ester *(ethyl benzoate)*; $C_9H_{10}O_2$; [93-89-0]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Tentative Solubilities
of Benzoic acid ethyl ester (1) in Water (2)

| $T/K$ | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 0.108 (ref 2) | 0.1 | 1 |
| 283 | 0.095* (ref 2) | 0.1 | 1 |
| 293 | 0.085* (ref 2) | 0.09 | 1 |
| 298 | 0.15[b] (ref 1), 0.085* (ref 2) | 0.09 | 1 |
| 303 | 0.090 (ref 2) | 0.09 | 1 |
| 313 | 0.10* (ref 2) | 0.1 | 1 |
| 323 | 0.11* (ref 2) | 0.1 | 1 |
| 333 | 0.11* (ref 2) | 0.1 | 1 |
| 343 | 0.12* (ref 2) | 0.1 | 1 |
| 353 | 0.13* (ref 2) | 0.1 | 1 |
| 363 | 0.14* (ref 2) | 0.1 | 1 |

[a] Rounded values of ref 2, but see text.

[b] Not included in estimation of "Best" value.

2. SOLUBILITY OF WATER (2) IN BENZOIC ACID ETHYL ESTER (1)

All the available data for the solubility of water (2) in benzoic acid ethyl ester (1) are summarized in Table 3. The situation for the organic-rich phase is equivalent to that of the $H_2O$-rich phase and the user is referred to the comments in Part 1. of this Evaluation.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Benzoic acid ethyl ester (*ethyl benzoate*); $C_9H_{10}O_2$; [93-89-0]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 3: Tentative Solubilities of Water (2) in Benzoic acid ethyl ester (1)

| *T*/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(2)/100g sln | g(2)/100g sln | $10^2x_2$ |
| 283 | $1.1^*$ (ref 2) | 1.1 | 8 |
| 293 | $1.0^*$ (ref 2) | 1.0 | 7 |
| 298 | $0.57^b$ (ref 1), $1.0^*$ (ref 2) | 1.0 | 7 |
| 303 | $0.9^*$ (ref 2) | 0.9 | 7 |
| 313 | $0.9^*$ (ref 2) | 0.9 | 7 |
| 323 | $1.1^*$ (ref 2) | 1.1 | 8 |
| 333 | $1.0^*$ (ref 2) | 1.0 | 7 |

[a] Rounded values of ref 2, but see text.

[b] Not included in calculation of "Best" value.

REFERENCES

1. Krupatkin, I. L.; Glagoleva, M. F. *Zh. Prikl. Khim.* 1972, *45*, 1795-9.

2. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

COMPONENTS:

(1) Benzoic acid ethyl ester (*ethyl benzoate*); $C_9H_{10}O_2$; [93-89-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Krupatkin, I.L.; Glagoleva, M.F.
*Zh. Prikl. Khim.* 1972, *45*, 1795-9.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of benzoic acid ethyl ester in water at 25°C was reported to be 0.15 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $1.8 \times 10^{-4}$.

The solubility of water in benzoic acid ethyl ester at 25°C was reported to be 0.57 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.046.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used as described in the publication of Krupatkin and Glagoleva (ref 1). The samples were titrated up to turbidity. The data were reported together with the ternary system benzoic acid ethyl ester-water-2-furancarbonal (*ethyl benzoate-water-furfural*). No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; b.p. 212°C, $d_{25}^{25}$ 1.050, $n_D^{25}$ 1.5050.

(2) Twice distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. Krupatkin, I.L.; Glagoleva, M.F. *Zh. Prikl. Khim.* 1969, *42*, 880.

COMPONENTS:

(1) Benzoic acid ethyl ester (*ethyl benzoate*); $C_9H_{10}O_2$; [93-89-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 363

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of benzoic acid ethyl ester and water

| $t$/°C | g(1)/100g sln (2)-rich phase | g(1)/100g sln (1)-rich phase | $x_1$ (compiler) (2)-rich phase | $x_1$ (compiler) (1)-rich phase |
|---|---|---|---|---|
| 0 | 0.108 | 98.81 | 0.000130 | 0.9088 |
| 9.5 | - | 99.18 | - | 0.9355 |
| 19.6 | 0.085 | 99.01 | 0.000102 | 0.9230 |
| 30.5 | 0.081 | 99.08 | 0.000097 | 0.9281 |
| 40.0 | 0.106 | 99.02 | 0.000127 | 0.9238 |
| 50.0 | 0.108 | 98.88 | 0.000130 | 0.9137 |
| 60.1 | 0.117 | 98.97 | 0.000140 | 0.9202 |
| 70.5 | 0.121 | - | 0.000145 | - |
| 80.2 | 0.121 | - | 0.000145 | - |
| 90.3 | 0.143 | - | 0.000172 | - |
| std. dev. | 0.002 | 0.01 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 97%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Benzoic acid, 2-hydroxy-, ethyl ester (*ethyl salicylate*); $C_9H_{10}O_3$; [118-61-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of 2-hydroxybenzoic acid ethyl ester and water

| $t/°C$ | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | - | 99.80 | - | 0.9818 |
| 9.2 | 0.067 | 99.76 | 0.000073 | 0.9783 |
| 19.5 | 0.036 | 99.79 | 0.000039 | 0.9809 |
| 29.8 | 0.037 | 99.68 | 0.000040 | 0.9712 |
| 39.7 | 0.029 | 99.74 | 0.000031 | 0.9765 |
| 49.8 | 0.050 | 99.73 | 0.000054 | 0.9756 |
| 60.1 | 0.040 | 99.76 | 0.000043 | 0.9783 |
| 70.1 | 0.040 | 99.54 | 0.000043 | 0.9591 |
| 80.1 | 0.069 | 99.37 | 0.000075 | 0.9447 |
| 90.5 | 0.078 | 99.33 | 0.000085 | 0.9414 |
| std. dev. | 0.003 | 0.01 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 97%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) 1,2,3-Propanetriol triacetate (*glycerol triacetate*); $C_9H_{14}O_6$; [102-76-1]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the 1,2,3-propanetriol triacetate (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the 1,2,3-Propanetriol triacetate (1) - Water (2) System

| Reference | T/K | Solubility | Method |
|---|---|---|---|
| Othmer *et al.* (ref 1) | 297[a] | mutual | unspecified |
| Kraus *et al.* (ref 2) | 298 | mutual | analytical, Karl Fischer |

[a] 23.5 °C.

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation and are summarized in Tables 2 and 3 below. Unfortunately, these two independent studies are in poor agreement in both the $H_2O$-rich and the organic-rich phases (the slight temperature difference should not be important) and the average "Best" values must be regarded as very Tentative, pending further studies.

TABLE 2: Tentative Solubilities of 1,2,3-Propanetriol triacetate (1) in Water (2)

| T/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" value [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^3x_1$ |
| 298 | 5.5[b] (ref 1), 7.4 (ref 2) | 6.5 ± 1.0 | 5.7 |

[a] Average value, but see text. $x_1$ has the same (relative) percentage uncertainty as the mass % solubility.

[b] 296.7 K.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) 1,2,3-Propanetriol triacetate (*glycerol triacetate*); $C_9H_{14}O_6$; [102-76-1]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 3: Tentative Solubilities of Water (2) in 1,2,3-Propanetriol triacetate (1)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" value [a] | |
| | g(2)/100g sln | g(2)/100g sln | $x_2$ |
| 298 | 2.5[b] (ref 1), 4.60 (ref 2) | 3.5 ± 1.0 | 0.31 |

[a] Average value, but see text. $x_2$ has the same status and (relative) percentage uncertainty as the mass % solubility.

[b] 296.7 K.

REFERENCES

1. Othmer, D. F.; White, R. E.; Trueger, E. *Ind. Eng. Chem.* 1941, *33*, 1240-8.
2. Kraus, K. A.; Rardon, R. J.; Baldwin, W. H. *J. Am. Chem. Soc.* 1964, *86*, 2571-6.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) 1,2,3-Propanetriol triacetate (*glycerol triacetate*); $C_9H_{14}O_6$; [102-76-1]<br>(2) Water; $H_2O$; [7732-18-5] | Othmer, D.F.; White, R.E.; Trueger, E.<br>*Ind. Eng. Chem.* 1941, *33*, 1240-8. |
| VARIABLES: | PREPARED BY: |
| $T$/K = 298 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of 1,2,3-propanetriol triacetate in water at 24.5°C was reported to be 5.5 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.0048.

The solubility of water in 1,2,3-propanetriol triacetate at 24.5°C was reported to be 2.5 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.24.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Nothing was specified in the paper. | (1) Not specified.<br>(2) Not specified. |
| | ESTIMATED ERROR: |
| | Temp. ±0.5°C (mean of reported range). |
| | REFERENCES: |

COMPONENTS:

(1) 1,2,3-Propanetriol triacetate (*glycerol triacetate*); $C_9H_{14}O_6$; [102-76-1]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kraus, K.A.; Raridon, R.J.; Baldwin, W.H.

*J. Am. Chem. Soc.* 1964, *86*, 2571-6.

VARIABLES:

$T$/K = 298

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

The solubility of 1,2,3-propanetriol triacetate in water at 25°C was reported to be 7.4 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.0066.

The solubility of water in 1,2,3-propanetriol triacetate at 25°C was reported to be 4.60 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.369.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical methods were used. The mutual solubilities of (1) and (2) were determined by shaking mixtures in stoppered tubes in a constant temperature bath. The (1)-rich phases were analyzed for water by the use of Karl Fischer titrations. The (2)-rich phases were analyzed for acetate by a semimicro adaptation of the standard saponification technique whereby back titration of excess alkali is performed on a boiling solution to minimize carbonate error, and in a flask under a reflux condenser to minimize loss of acetic acid.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; commercial product; distilled under vacuum and middle fraction 60-80% collected.

(2) Not specified.

ESTIMATED ERROR:

Soly. ±5 g(1)/100g sln and
±1 g(2)/100g sln (precision).

REFERENCES:

COMPONENTS:

(1) Propanoic acid cyclohexyl ester (*cyclohexyl propionate*); $C_9H_{16}O_2$; [6222-35-1]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of propanoic acid cyclohexyl ester and water

| $t/°C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.109 | 99.17 | 0.000126 | 0.9323 |
| 9.5 | 0.096 | 99.29 | 0.000111 | 0.9416 |
| 20.2 | 0.079 | 99.21 | 0.000091 | 0.9354 |
| 30.6 | 0.073 | 99.06 | 0.000084 | 0.9239 |
| 39.6 | 0.065 | 99.18 | 0.000075 | 0.9331 |
| 50.0 | 0.061 | 99.32 | 0.000070 | 0.9439 |
| 60.1 | 0.073 | 99.28 | 0.000084 | 0.9408 |
| 70.2 | 0.066 | 99.06 | 0.000076 | 0.9239 |
| 80.2 | 0.080 | 98.91 | 0.000092 | 0.9127 |
| 90.6 | 0.075 | 98.78 | 0.000086 | 0.9032 |
| std. dev. | 0.001 | 0.05 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Pentanedioic acid diethyl ester (*diethyl glutarate*); $C_9H_{16}O_4$; [818-38-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sobotka, H.; Kahn, J.

*J. Am. Chem. Soc.* 1931, *53*, 2935-8.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of pentanedioic acid diethyl ester in water at 20°C was reported to be 0.882 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.876 g(1)/100g sln and $8.45 \times 10^{-4}$.

Density of water $d_4^{20} = 0.9982$ (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 1.0220, $n_D^{20}$ 1.4242.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Acetic acid heptyl ester (*heptyl acetate*); $C_9H_{18}O_2$; [112-06-1]<br>(2) Water; $H_2O$; [7732-18-5] | Stephenson, R.; Stuart, J.<br>*J. Chem. Eng. Data* 1986, *31*, 56-70. |
| VARIABLES: | PREPARED BY: |
| $T/K$ = 273 - 364 | Z. Maczynska |

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid heptyl ester and water

| $t/^\circ C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.023 | 99.66 | 0.000026 | 0.9709 |
| 9.2 | 0.018 | 99.62 | 0.000020 | 0.9676 |
| 19.6 | 0.020 | 99.55 | 0.000023 | 0.9618 |
| 30.6 | 0.023 | 99.52 | 0.000026 | 0.9593 |
| 40.3 | 0.021 | 99.41 | 0.000024 | 0.9504 |
| 50.0 | 0.020 | 99.41 | 0.000023 | 0.9504 |
| 60.2 | 0.019 | 99.37 | 0.000022 | 0.9472 |
| 70.2 | 0.017 | 99.35 | 0.000019 | 0.9456 |
| 80.4 | 0.015 | 99.33 | 0.000017 | 0.9440 |
| 90.6 | 0.019 | 99.33 | 0.000022 | 0.9440 |
| std. dev. | 0.001 | 0.01 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 98%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Butanoic acid 3-methyl-1-butyl ester (*isopentyl butyrate*); $C_9H_{18}O_2$; [106-27-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of butanoic acid 3-methyl-1-butyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | - | 99.75 | - | 0.9784 |
| 9.4 | 0.035 | 99.71 | 0.000040 | 0.9751 |
| 19.6 | 0.022 | 99.76 | 0.000025 | 0.9793 |
| 30.9 | 0.027 | 99.62 | 0.000031 | 0.9676 |
| 39.9 | 0.015 | 99.59 | 0.000017 | 0.9651 |
| 49.8 | 0.037 | 99.69 | 0.000042 | 0.9734 |
| 59.7 | 0.013 | 99.68 | 0.000015 | 0.9726 |
| 70.2 | 0.014 | 99.61 | 0.000016 | 0.9667 |
| 80.1 | 0.024 | 99.52 | 0.000027 | 0.9593 |
| 90.7 | 0.020 | 99.50 | 0.000023 | 0.9577 |
| std. dev. | 0.004 | 0.01 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Butanoic acid pentyl ester (*pentyl butyrate*); $C_9H_{18}O_2$; [540-18-1]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A., Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the butanoic acid pentyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Butanoic acid pentyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Hemptinne (ref 1) | 298 | (1) in (2) | analytical |
| Stephenson and Stuart (ref 2) | 273-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF BUTANOIC ACID PENTYL ESTER (1) IN WATER (2)

All the available data for the solubility of butanoic acid pentyl ester (1) in water (2) are summarized in Table 2. At 298 K, the only temperature where comparison is possible, the data of Hemptinne (ref 1) and Stephenson and Stuart (ref 2) are in only fair agreement. At other temperatures only the data of Stephenson and Stuart (ref 2) are available. Although the solubilities reported by these authors are usually reliable it should be noted that their results for this system are very scattered. Consequently, all the values in Table 2 should be regarded as very Tentative, pending further studies.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Butanoic acid pentyl ester (*pentyl butyrate*); $C_9H_{18}O_2$; [540-18-1]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Tentative Solubilities of Butanoic acid pentyl ester (1) in Water (2)

| *T*/K | Solubilities | | |
|---|---|---|---|
| | Reported values [a] | "Best" values [b] | |
| | g(1)/100g sln | g(1)/100g sln | $10^5 x_1$ |
| 273 | 0.03* (ref 2) | 0.03 | 3 |
| 283 | 0.03* (ref 2) | 0.03 | 3 |
| 293 | 0.03* (ref 2) | 0.03 | 3 |
| 298 | 0.050 (ref 1), 0.03* (ref 2) | 0.04 ± 0.01[c] | 5 |
| 303 | 0.03* (ref 2) | 0.03 | 3 |
| 313 | 0.03* (ref 2) | 0.03 | 3 |
| 323 | 0.03* (ref 2) | 0.03 | 3 |
| 333 | 0.03* (ref 2) | 0.03 | 3 |
| 343 | 0.03* (ref 2) | 0.03 | 3 |
| 353 | 0.02* (ref 2) | 0.02 | 2 |
| 363 | 0.02* (ref 2) | 0.02 | 2 |

[a] Data in ref 2 very scattered.

[b] Rounded values of ref 2, except at 298 K.

[c] Average value; $x_1$ has the same (relative) percentage uncertainty as the mass % solubility.

## 2. SOLUBILITY OF WATER (2) IN BUTANOIC ACID PENTYL ESTER (1)

Only the data of Stephenson and Stuart (ref 2) are available for the solubility of water (2) in butanoic acid pentyl ester (1) and so no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheet for the experimental solubilities. It should be noted, however, that whilst the solubilities reported by these authors are generally reliable, their results for this system are very scattered. Further studies are clearly required.

REFERENCES

1. Hemptinne, A. *Z. Phys. Chem.* 1894, *13*, 561-9.
2. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Butanoic acid pentyl ester (*pentyl butyrate*); $C_9H_{18}O_2$; [540-18-1]<br>(2) Water; $H_2O$; [7732-18-5] | Hemptinne, A.<br>*Z. Phys. Chem.* 1894, *13*, 561-9. |
| VARIABLES: | PREPARED BY: |
| $T/K = 298$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of butanoic acid pentyl ester in water at 25°C was reported to be 0.505 g(1)/L sln.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The mixture of water with excess ester was heated for some time in a water bath and the ester phase was filtered. A sample of known volume was then transferred to a smaller flask, heated with the known amount of baryta until complete saponification was obtained and then titrated. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Butanoic acid pentyl ester (*pentyl butyrate*); $C_9H_{18}O_2$; [540-18-1]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of butanoic acid pentyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.026 | 99.54 | 0.000030 | 0.9610 |
| 9.4 | 0.034 | 99.66 | 0.000039 | 0.9709 |
| 19.2 | 0.025 | 99.60 | 0.000028 | 0.9659 |
| 29.6 | 0.024 | 99.65 | 0.000027 | 0.9701 |
| 40.1 | 0.030 | 99.48 | 0.000034 | 0.9561 |
| 50.0 | 0.032 | 99.61 | 0.000036 | 0.9667 |
| 60.0 | 0.028 | 99.56 | 0.000032 | 0.9626 |
| 70.3 | - | 99.38 | - | 0.9480 |
| 80.0 | 0.019 | 99.52 | 0.000022 | 0.9593 |
| 90.5 | 0.025 | 99.44 | 0.000028 | 0.9529 |
| std. dev. | 0.002 | 0.01 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Formic acid octyl ester (*octyl formate*); $C_9H_{18}O_2$; [112-32-3]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of formic acid octyl ester and water

| $t/°C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | - | 99.579 | - | 0.96419 |
| 9.2 | 0.078 | 99.555 | 0.000089 | 0.96222 |
| 19.0 | 0.064 | 99.583 | 0.000073 | 0.96452 |
| 29.4 | 0.039 | 99.572 | 0.000044 | 0.96361 |
| 39.5 | 0.040 | 99.555 | 0.000045 | 0.96222 |
| 50.0 | 0.071 | 99.545 | 0.000081 | 0.96140 |
| 60.0 | 0.028 | 99.522 | 0.000032 | 0.95952 |
| 70.1 | 0.044 | 99.506 | 0.000050 | 0.95821 |
| 80.2 | 0.036 | 99.426 | 0.000041 | 0.95174 |
| 90.5 | 0.061 | 99.461 | 0.000069 | 0.95456 |
| std. dev. | 0.003 | 0.003 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Heptanoic acid ethyl ester (*ethyl heptanoate*); $C_9H_{18}O_2$; [106-30-9]<br>(2) Water; $H_2O$; [7732-18-5] | Sobotka, H.; Kahn, J.<br>*J. Am. Chem. Soc.* 1931, *53*, 2935-8. |
| VARIABLES: | PREPARED BY: |
| $T/K = 293$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of heptanoic acid ethyl ester in water at 20°C was reported to be 0.029 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.029 g(1)/100g sln and $3.3 \times 10^{-5}$.

Density of water $d_4^{20} = 0.9982$ (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indica-] tor particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $n_D^{20}$ 1.4137.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) Pentanoic acid 1-butyl ester (*butyl valerate*); $C_9H_{18}O_2$; [591-68-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Bomshtein, A.L.; Trofimov, A.N.; Serafimov, L.A.

*Zh. Prikl. Khim.* 1978, *51*, 1280-2.

VARIABLES:

$T/K = 293 - 363$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of pentanoic acid 1-butyl ester and water

| $t/^{\circ}C$ | $x_1$ | | g(1)/100g sln (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 20 | 0.00002 | 0.9037 | 0.02 | 98.80 |
| 30 | 0.00009 | 0.8821 | 0.08 | 98.50 |
| 40 | 0.00015 | 0.8751 | 0.13 | 98.40 |
| 50 | 0.00021 | 0.8514 | 0.18 | 98.05 |
| 60 | 0.00027 | 0.8319 | 0.24 | 97.75 |
| 70 | 0.00037 | 0.8163 | 0.32 | 97.50 |
| 80 | 0.00039 | 0.8011 | 0.34 | 97.25 |
| 90 | 0.00046 | 0.7865 | 0.40 | 97.00 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used at constant temperature. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; distilled; without impurities by glc, b.p. 185.3°C, $n_D^{20}$ 1.412.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Propanoic acid hexyl ester (*hexyl propionate*); $C_9H_{18}O_2$; [2445-76-3]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of propanoic acid hexyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.029 | 99.61 | 0.000033 | 0.9667 |
| 9.7 | 0.022 | 99.66 | 0.000025 | 0.9709 |
| 20.0 | 0.024 | 99.66 | 0.000027 | 0.9709 |
| 29.6 | 0.012 | 99.55 | 0.000014 | 0.9618 |
| 39.8 | 0.012 | 99.52 | 0.000014 | 0.9593 |
| 50.0 | 0.014 | 99.54 | 0.000016 | 0.9610 |
| 60.0 | 0.016 | 99.50 | 0.000018 | 0.9577 |
| 70.3 | 0.018 | 99.51 | 0.000020 | 0.9585 |
| 80.3 | 0.017 | 99.49 | 0.000019 | 0.9569 |
| 90.6 | 0.018 | 99.17 | 0.000020 | 0.9315 |
| std. dev. | 0.001 | 0.02 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Dibutylphosphinic acid methyl ester (*methyl dibutylphosphinate*); $C_9H_{21}O_2P$; [7163-67-9]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.

*Zh. Fiz. Khim.* 1966, *40*, 221-3.

VARIABLES:

$T$/K = 309 - 464

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of dibutylphosphinic acid methyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 35.5 LCST | 19.83 | 19.83 | 0.02265 | 0.02265 |
| 36.2 | 12.75 | - | 0.01351 | - |
| 36.6 | - | 31.05 | - | 0.04049 |
| 37.0 | - | 36.87 | - | 0.05189 |
| 38.0 | - | 42.02 | - | 0.06360 |
| 40.2 | 7.40 | - | 0.00743 | - |
| 41.0 | - | 46.14 | - | 0.07431 |
| 47.2 | - | 52.49 | - | 0.09382 |
| 50.0 | 4.85 | - | 0.00475 | - |
| 56.3 | - | 57.58 | - | 0.11285 |
| 58.0 | 3.79 | - | 0.00368 | - |
| 60.0 | 3.65 | - | 0.00354 | - |

(continued next page)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Alekseev's synthetic method was used. A water or glycerine bath was used for temperature control. Ester hydrolysis was not observed. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized; distilled 5-6 times under vacuum; $d_4^{25}$ 0.9501, $n_D^{25}$ 1.4441.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Dibutylphosphinic acid methyl ester<br>(*methyl dibutylphosphinate*);<br>$C_9H_{21}O_2P$; [7163-67-9]<br>(2) Water; $H_2O$; [7732-18-5] | Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.<br>*Zh. Fiz. Khim.* 1966, *40*, 221-3. |

EXPERIMENTAL VALUES: (continued)

Mutual solubility of dibutylphosphinic acid methyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 64.0 | 3.46 | - | 0.00335 | - |
| 68.0 | 3.29 | - | 0.00318 | - |
| 71.0 | - | 62.20 | - | 0.13360 |
| 90.5 | - | 65.14 | - | 0.14902 |
| 120.0 | 3.29 | - | 0.00318 | - |
| 133.0 | - | 65.14 | - | 0.14902 |
| 150.0 | 4.85 | - | 0.00475 | - |
| 155.0 | - | 62.20 | - | 0.13360 |
| 171.0 | - | 57.58 | - | 0.11285 |
| 172.0 | 7.40 | - | 0.00743 | - |
| 181.0 | - | 52.49 | - | 0.09382 |
| 186.0 | 12.75 | - | 0.01351 | - |
| 190.0 | 19.83 | 42.02 | 0.02265 | 0.06360 |
| 191.0 UCST | 31.05 | 31.05 | 0.04049 | 0.04049 |

COMPONENTS:

(1) Methylphosphonic acid dibutyl ester
(*dibutyl methylphosphonate*);
$C_9H_{21}O_3P$; [2404-73-1]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.

*Zh. Fiz. Khim.* 1967, *41*, 1815-7.

VARIABLES:

$T$/K = 275 - 348

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of methylphosphonic acid dibutyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 1.6 | - | 60.82 | - | 0.11840 |
| 4.3 | 3.49 | - | 0.00312 | - |
| 7.5 | 3.02 | - | 0.00269 | - |
| 8.4 | - | 64.88 | - | 0.13780 |
| 8.7 | 2.94 | - | 0.00261 | - |
| 18.2 | - | 69.20 | - | 0.16274 |
| 19.7 | 2.00 | - | 0.00176 | - |
| 28.0 | - | 72.09 | - | 0.18265 |
| 34.0 | 1.49 | - | 0.00131 | - |
| 44.5 | - | 75.30 | - | 0.20870 |
| 75.0 | - | 78.34 | - | 0.23833 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Alekseev's synthetic method was used. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized; distilled 5-6 times under vacuum; $d_4^{20}$ 0.9788, $n_D^{20}$ 1.4255.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid dimethyl ester (*dimethyl phthalate*); $C_{10}H_{10}O_4$; [131-11-13]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the 1,2-benzenedicarboxylic acid dimethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the 1,2-Benzenedicarboxylic acid dimethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Shanley and Greenspan (ref 1) | RT[a] | (2) in (1) | unspecified |
| Leyder and Boulanger (ref 2) | 293 | (1) in (2) | GLC,<br>spectrophotometric |

[a] RT - room temperature.

As can be seen from Table 1, there are no independent studies for solubilities in either the $H_2O$-rich or organic-rich phases and so no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheets immediately following this Evaluation for the experimental data.

REFERENCES

1. Shanley, E. S.; Greenspan, F. P. *Ind. Eng. Chem.* 1947, *39*, 1536-43.

2. Leyder, F.; Boulanger, P. *Bull. Environ. Contam. Toxicol.* 1983, *30*, 152-7.

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid dimethyl ester (*dimethyl phthalate*); $C_{10}H_{10}O_4$; [131-11-13]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Shanley, E.S.; Greenspan, F.P.

*Ind. Eng. Chem.* 1947, *39*, 1536-43.

VARIABLES:

$T$/K = room temperature

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of water in 1,2-benzenedicarboxylic acid dimethyl ester at room temperature was reported to be 1.6 g(2)/100g(1). The corresponding mass per cent and mole fraction, $x_2$, values calculated by the compiler are 1.6 g(2)/100g sln and 0.15.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid dimethyl ester (*dimethyl phthalate*); $C_{10}H_{10}O_4$; [131-11-13]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Leyder, F.; Boulanger, P.

*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7.

VARIABLES:

*T*/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid dimethyl ester in water at 20°C was reported to be 2.21 x $10^{-2}$ mol(1)/L and 4.290 g(1)/L.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent-flush" and "hot-needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) Fluka; purity>99>% ; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

COMPONENTS:

(1) Hexanedioic acid diethyl ester (*diethyl adipate*); $C_{10}H_{18}O_4$; [141-28-6]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the hexanedioic acid diethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Hexanedioic acid diethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Sobotka and Kahn (ref 1) | 293 | (1) in (2) | titration |
| Gross *et al.* (ref 2) | 303 | (1) in (2) | interferometric |
| Stephenson and Stuart (ref 3) | 273-363 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF HEXANEDIOIC ACID DIETHYL ESTER (1) IN WATER (2)

All the available data for the solubility of hexanedioic acid diethyl ester (1) in water (2) are summarized in Table 2. At 293 and 303 K, the only temperatures where comparison is possible, the studies of Sobotka and Kahn (ref 1) and Gross *et al.* (ref 2) respectively, are in only fair agreement with that of Stephenson and Stuart (ref 3). At other temperatures only the data of Stephenson and Stuart (ref 3) are available. Consequently, all data must be considered Tentative pending further studies although it may be noted that the data of Stephenson and Stuart (ref 3) are generally reliable.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Hexanedioic acid diethyl ester (*diethyl adipate*); $C_{10}H_{18}O_4$; [141-28-6]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Tentative Solubilities of Hexanedioic acid diethyl ester (1) in Water (2)

| $T$/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^4x_1$ |
| 273 | 1.07 (ref 3) | 1.0 | 9 |
| 283 | 0.87* (ref 3) | 0.9 | 8 |
| 293 | 0.422 (ref 1), 0.69* (ref 3) | 0.6 ± 0.1[b] | 5 |
| 298 | 0.64* (ref 3) | 0.6 | 5 |
| 303 | 0.424 (ref 2), 0.60* (ref 3) | 0.5 ± 0.1[b] | 4 |
| 313 | 0.54* (ref 3) | 0.5 | 4 |
| 323 | 0.51* (ref 3) | 0.5 | 4 |
| 333 | 0.50* (ref 3) | 0.5 | 4 |
| 343 | 0.50* (ref 3) | 0.5 | 4 |
| 353 | 0.51* (ref 3) | 0.5 | 4 |
| 363 | 0.55* (ref 3) | 0.6 | 5 |

[a] Rounded values of ref 3 unless otherwise indicated.

[b] Average value; $x_1$ values have the same (relative) percentage uncertainty as the mass % solubilities.

2. SOLUBILITY OF WATER (2) IN HEXANEDIOIC ACID DIETHYL ESTER (1)

As the only available data for the solubility of water (2) in hexanedioic acid diethyl ester (1) are those of Stephenson and Stuart (ref 3) no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheet for the experimental solubilities but it may be noted that the data of these authors are generally reliable (but see Part 1. above).

REFERENCES

1. Sobotka, H.; Kahn, J. *J. Am. Chem. Soc.* 1931, *53*, 2935-8.
2. Gross, P. M.; Saylor, J. H.; Gorman, M. A. *J. Am. Chem. Soc.* 1953, *55*, 650-2.
3. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Hexanedioic acid diethyl ester (*diethyl adipate*); $C_{10}H_{18}O_4$; [141-28-6]<br>(2) Water; $H_2O$; [7732-18-5] | Sobotka, H.; Kahn, J.<br>*J. Am. Chem. Soc.* 1931, *53*, 2935-8. |
| VARIABLES: | PREPARED BY: |
| $T/K = 293$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of hexanedioic acid diethyl ester in water at 20°C was reported to be 0.423 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.422 g(1)/100g sln and $3.77 \times 10^{-4}$.

Density of water $d_4^{20} = 0.9982$ (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 1.0086, $n_D^{20}$ 1.4278.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) Hexanedioic acid diethyl ester (*diethyl adipate*); $C_{10}H_{18}O_4$; [141-28-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gross, P.M.; Saylor, J.H.; Gorman, M.A.

*J. Am. Chem. Soc.* 1933, *55*, 650-2.

VARIABLES:

$T/K = 303$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

The solubility of hexanedioic acid diethyl ester in water at 30°C was reported to be 4.26 g(1)/kg(2) and 0.0211 mol(1)/kg(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.424 g(1)/100g sln and $3.79 \times 10^{-4}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The saturated solutions of (1) in (2) were prepared in thin-walled cylindrical flask in a large water thermostat. The saturated solutions were analyzed by means of a Zeiss combination liquid and gas interferometer. The procedure has been previously described in detail by Gross and Saylor (ref 1).

SOURCE AND PURITY OF MATERIALS:

(1) Eastman Kodak Co. best grade; distilled;
b.p. range 129.0-129.4°C.

(2) Distilled.

ESTIMATED ERROR:

Temp. ±0.02°C.
Soly. ±0.5% (from the concordance of duplicate determinations).

REFERENCES:

1. Gross, P.M.; Saylor, J.H. *J. Am. Chem. Soc.* 1931, *53*, 1744.

COMPONENTS:

(1) Hexanedioic acid diethyl ester (*diethyl adipate*); $C_{10}H_{18}O_4$; [141-28-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 363

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of hexanedioic acid diethyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 1.07 | 98.92 | 0.00096 | 0.8908 |
| 9.8 | 1.26 | 98.89 | 0.00113 | 0.8881 |
| 20.5 | 0.69 | - | 0.00062 | - |
| 39.5 | 0.54 | 98.05 | 0.00048 | 0.8175 |
| 50.0 | 0.52 | 97.89 | 0.00046 | 0.8051 |
| 60.1 | 0.50 | 97.62 | 0.00045 | 0.7851 |
| 69.7 | 0.50 | 97.56 | 0.00045 | 0.7808 |
| 80.2 | 0.49 | 97.17 | 0.00045 | 0.7536 |
| 90.3 | 0.56 | 97.12 | 0.00050 | 0.7502 |
| std. dev. | 0.01 | 0.03 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Acetic acid 2-ethylhexyl ester
*(2-ethylhexyl acetate)*;
$C_{10}H_{20}O_2$; [103-09-3]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

*T*/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 2-ethylhexyl ester in water at 20°C was reported to be very slightly soluble.

The solubility of water in acetic acid 2-ethylhexyl ester at 20°C was reported to be 0.39 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.036.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 99%, b.p. range 195-203°C, $d_4^{20}$ 0.873, $n_D^{20}$ 1.4300.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid octyl ester (*octyl acetate*); $C_{10}H_{20}O_2$; [112-14-1]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Carbide and Carbon Chemicals Corporation, Solvent Chart.

Othmer, D.F.; White, R.E.; Trueger, E.

*Ind. Eng. Chem.* 1941, *33*, 1240-8, 1513.

VARIABLES:

$T$/K = 296

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid octyl ester in water at 23°C was reported to be less than 0.3 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is less than 3 x $10^{-5}$.

The solubility of water in acetic acid octyl ester at 23°C was reported to be 0.55 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.050.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing was specified in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Nonanoic acid methyl ester (*methyl nonanoate*); $C_{10}H_{20}O_2$; [1731-84-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Tewari, Y.B.; Miller, M.M.; Wasik, S.P.; Martire, D.E.

*J. Chem. Eng. Data* 1982, *27*, 451-4.

VARIABLES:

$T/K = 298$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of nonanoic acid methyl ester in water at 25°C was reported to be $1.33 \times 10^{-4}$ mol(1)/L sln. The corresponding value on a mass/volume basis is 0.0229 g(1)/L sln (compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The aqueous phase was generated by pumping water into the inlet of coated generator column which was thermostated, either by using a minipump or by means of water reservoir using compressed air at 5 psi. The aqueous solution was extracted by the use of a known amount of immiscible with water solution and then analyzed by a gas chromatographic technique.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; purity >99% by glc.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.1°C.
Soly. ±1.0%.

REFERENCES:

COMPONENTS:

(1) Octanoic acid ethyl ester (*ethyl caprylate*); $C_{10}H_{20}O_2$; [106-32-1]

(2) Water; $H_2O$; [7732-18-5]

EVALUATOR:

G.T. Hefter, School of Mathematical
and Physical Sciences,
Murdoch University, Perth, W.A.,
Australia
December, 1988

CRITICAL EVALUATION:

Quantitative solubility data for the octanoic acid ethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Octanoic acid ethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Sobotka and Kahn (ref 1) | 293 | (1) in (2) | titration |
| Stephenson and Stuart (ref 2) | 283-364 | mutual | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF OCTANOIC ACID ETHYL ESTER (1) IN WATER (2)

All the available data for the solubility of octanoic acid ethyl ester (1) in water (2) are summarized in Table 2. At 293 K, the only temperature where comparison is possible, the data of Sobotka and Kahn (ref 1) and Stephenson and Stuart (ref 2) disagree by almost an order of magnitude (Table 2). In the absence of other independent studies it is not possible to determine which value is more reliable. At other temperatures, only the data of Stephenson and Stuart (ref 2) are available. Although the solubilities reported by these authors are usually reliable it should be noted that their results for this system are very scattered. For these reasons the data in Table 2 should be regarded as very Tentative pending further studies.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Octanoic acid ethyl ester (*ethyl caprylate*); $C_{10}H_{20}O_2$; [106-32-1]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Tentative Solubilities of Octanoic acid ethyl ester (1) in Water (2)

| *T*/K | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values [a] | |
| | g(1)/100g sln | g(1)/100g sln | $10^5x_1$ |
| 283 | 0.05* (ref 2) | 0.05 | 5 |
| 293 | 0.007 (ref 1), 0.05 (ref 2) | 0.05 | 5 |
| 298 | 0.04* (ref 2) | 0.04 | 4 |
| 303 | 0.04* (ref 2) | 0.04 | 4 |
| 313 | 0.04* (ref 2) | 0.04 | 4 |
| 323 | 0.04* (ref 2) | 0.04 | 4 |
| 333 | 0.04* (ref 2) | 0.04 | 4 |
| 343 | 0.04* (ref 2) | 0.04 | 4 |
| 353 | 0.04* (ref 2) | 0.04 | 4 |
| 363 | 0.04* (ref 2) | 0.04 | 4 |

[a] Rounded values of ref 2, but see text.

## 2. SOLUBILITY OF WATER (2) IN OCTANOIC ACID ETHYL ESTER (1)

As the only available data for the solubility of water (2) in octanoic acid ethyl ester (1) are those of Stephenson and Stuart (ref 2) no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheet for the experimental solubilities. However, it may be noted that the solubilities reported by these authors are generally reliable although the values reported for this particular system are rather scattered (see also Part 1. above).

REFERENCES

1. Sobotka, H.; Kahn, J. *J. Am. Chem. Soc.* 1931, *53*, 2935-8.
2. Stephenson, R.; Stuart, J. *J. Chem. Eng. Data* 1986, *31*, 56-70.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Octanoic acid ethyl ester (*ethyl caprylate*); $C_{10}H_{20}O_2$; [106-32-1]<br>(2) Water; $H_2O$; [7732-18-5] | Sobotka, H.; Kahn, J.<br>*J. Am. Chem. Soc.* 1931, *53*, 2935-8. |
| VARIABLES: | PREPARED BY: |
| $T/K = 293$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of octanoic acid ethyl ester in water at 20°C was reported to be 0.007 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.007 g(1)/100g sln and $7 \times 10^{-6}$.

Density of water $d_4^{20} = 0.9982$ (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 0.8674, $n_D^{20}$ 1.4197.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) Octanoic acid ethyl ester (*ethyl caprylate*); $C_{10}H_{20}O_2$; [106-32-1]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of octanoic acid ethyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | - | 99.18 | - | 0.9267 |
| 9.5 | 0.054 | 98.85 | 0.000056 | 0.8999 |
| 19.6 | 0.051 | 99.10 | 0.000053 | 0.9201 |
| 30.6 | 0.038 | 99.55 | 0.000040 | 0.9585 |
| 39.5 | - | 99.61 | - | 0.9639 |
| 50.0 | 0.037 | 99.35 | 0.000039 | 0.9411 |
| 60.1 | 0.044 | 99.49 | 0.000046 | 0.9533 |
| 70.2 | 0.040 | 99.57 | 0.000042 | 0.9603 |
| 80.0 | 0.042 | 99.59 | 0.000044 | 0.9621 |
| 90.6 | - | 99.61 | - | 0.9639 |
| std. dev. | 0.003 | 0.01 |  |  |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 99%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Propanoic acid, 2-methyl-, hexyl ester *(hexyl isobutyrate)*; $C_{10}H_{20}O_2$; [2349-07-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T$/K = 273 - 364

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of 2-methylpropanoic acid hexyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.0116 | 99.50 | 0.0000121 | 0.9541 |
| 9.4 | 0.0061 | 99.69 | 0.0000064 | 0.9711 |
| 19.5 | 0.0038 | 99.63 | 0.0000040 | 0.9657 |
| 29.7 | - | 99.74 | - | 0.9757 |
| 39.5 | 0.0032 | - | 0.0000033 | - |
| 50.0 | 0.0076 | 99.56 | 0.0000079 | 0.9594 |
| 59.8 | 0.0081 | - | 0.0000085 | - |
| 70.1 | - | 99.60 | - | 0.9630 |
| 80.0 | 0.0040 | 99.59 | 0.0000042 | 0.9621 |
| 90.6 | 0.0087 | 99.58 | 0.0000091 | 0.9612 |
| std. dev. | 0.0005 | 0.02 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator). A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 98%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid 2-(2-butoxyethoxy)ethyl ester *(2-(2-butoxyethoxy)ethyl acetate)*; $C_{10}H_{20}O_4$; [124-17-4]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION:

Quantitative solubility data for the acetic acid 2-(2-butoxyethoxy)ethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Acetic acid 2-(2-butoxyethoxy)ethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Doolittle (ref 1) | 293 | mutual | unspecified |
| Othmer *et al.* (ref 2) | 297 | mutual | unspecified |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. They are also summarized in Tables 2 and 3 below.

As there have been only two independent studies at slightly different temperatures, Critical Evaluation is difficult. If the temperature difference is assumed to be negligible (ester-water mutual solubilities do not generally vary dramatically *ca.* 295 K) then the data for the $H_2O$-rich phase are in poor agreement whilst the ester-rich phase values are in good agreement. The latter may be regarded as Tentative.

TABLE 2: Reported Solubilities of Acetic acid 2-(2-butoxyethoxy)ethyl ester (1) in Water (2)

| *T*/K | Reported Solubilities | |
|---|---|---|
| | g(1)/100g sln | $10^3x_1$ |
| 293 | 1.6 (ref 1) | 1.4 |
| 297 | 6.5 (ref 2) | 6.1 |

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Acetic acid 2-(2-butoxyethoxy)ethyl ester *(2-(2-butoxyethoxy)ethyl acetate)*; $C_{10}H_{20}O_4$; [124-17-4]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 3: Tentative Solubilities of Water (2) in Acetic acid 2-(2-butoxyethoxy)ethyl ester (1)

| *T*/K | Tentative Solubilities | |
|---|---|---|
| | g(2)/100g sln | $x_2$ |
| 293 | 3.4 (ref 1) | 0.29 |
| 297 | 3.7 (ref 2) | 0.30 |

REFERENCES

1. Doolittle, A. K. *Ind. Eng. Chem.* 1935, *27*, 1169-79.
2. Othmer, D. F.; White, R. E.; Trueger, E. *Ind. Eng. Chem.* 1941, *33*, 1240-8; *ibid.* 1513; see also Carbide and Carbon Chemical Corporation, Solvent Chart, before 1941.

COMPONENTS:

(1) Acetic acid 2-(2-butoxyethoxy)ethyl ester *(2-(2-butoxyethoxy)ethyl acetate)*; $C_{10}H_{20}O_4$; [124-17-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Doolittle, A.K.

*Ind. End. Chem.* 1935, *27*, 1169-79.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 2-(2-butoxyethoxy)ethyl ester in water at 20°C was reported to be 1.6 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.0014.

The solubility of water in acetic acid 2-(2-butoxyethoxy)ethyl ester at 20°C was reported to be 3.4 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.29.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial product; purity 99%, b.p. range 236-249°C, $d_4^{20}$ 0.987.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Acetic acid 2-(2-butoxyethoxy)ethyl ester *(2-(2-butoxyethoxy)ethyl acetate)*; $C_{10}H_{20}O_4$; [124-17-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Carbide and Carbon Chemicals Corporation, Solvent Chart.

Othmer, D.F.; White, R.E.; Trueger, E.

*Ind. Eng. Chem.* 1941, *33*, 1240-8, 1513.

VARIABLES:

$T$/K = 297

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of acetic acid 2-(2-butoxyethoxy)ethyl ester in water at 23.5°C was reported to be 6.5 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.00609.

The solubility of water in acetic acid 2-(2-butoxyethoxy)ethyl ester at 23.5°C was reported to be 3.7 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.303.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nothing was specified in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.5°C (mean of reported range).

REFERENCES:

COMPONENTS:

(1) Dibutylphosphinic acid ethyl ester
(*ethyl dibutylphosphinate*);
$C_{10}H_{23}O_2P$; [7100-92-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.

*Zh. Fiz. Khim.* 1966, *40*, 221-3.

VARIABLES:

$T/K = 287 - 599$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of dibutylphosphinic acid ethyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 14.0 LCST | 23.36 | 23.36 | 0.02593 | 0.02593 |
| 14.6 | - | 34.57 | - | 0.04411 |
| 15.4 | 9.99 | - | 0.00960 | - |
| 18.0 | - | 46.94 | - | 0.07172 |
| 18.6 | 5.96 | - | 0.00550 | - |
| 22.5 | - | 53.17 | - | 0.09022 |
| 23.4 | 4.43 | - | 0.00403 | - |
| 26.5 | 3.88 | - | 0.00351 | - |
| 28.0 | - | 58.65 | - | 0.11023 |
| 29.5 | 3.58 | - | 0.00323 | - |
| 41.0 | 2.44 | - | 0.00218 | - |
| 52.0 | 2.02 | - | 0.00180 | - |

(continued next page)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Alekseev's synthetic method was used. A water or glycerine bath was used for temperature control. Ester hydrolysis was not observed. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized; distilled 5-6 times under vacuum; $d_4^{20}$ 0.9394, $n_D^{20}$ 1.4420.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Dibutylphosphinic acid ethyl ester<br>(*ethyl dibutylphosphinate*);<br>$C_{10}H_{23}O_2P$; [7100-92-7]<br>(2) Water; $H_2O$; [7732-18-5] | Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.<br>*Zh. Fiz. Khim.* 1966, *40*, 221-3. |

EXPERIMENTAL VALUES: (continued)

Mutual solubility of dibutylphosphinic acid ethyl ester and water

| $t/^\circ C$ | g(1)/100g sln (2)-rich phase | g(1)/100g sln (1)-rich phase | $x_1$ (compiler) (2)-rich phase | $x_1$ (compiler) (1)-rich phase |
|---|---|---|---|---|
| 54.8 | - | 70.28 | - | 0.17118 |
| 58.0 | 1.79 | - | 0.00159 | - |
| 89.0 | - | 74.91 | - | 0.20683 |
| 127.0 | 1.79 | - | 0.00159 | - |
| 159.0 | 2.44 | - | 0.00218 | - |
| 168.0 | - | 70.28 | - | 0.17118 |
| 189.0 | 4.43 | - | 0.00403 | - |
| 211.0 | - | 58.65 | - | 0.11023 |
| 216.0 | 9.99 | - | 0.00960 | - |
| 217.0 | - | 53.17 | - | 0.09022 |
| 223.0 | - | 46.94 | - | 0.07172 |
| 225.0 | - | 34.57 | - | 0.04411 |
| 226.0 UCST | 23.36 | 23.36 | 0.02593 | 0.02593 |

COMPONENTS:

(1) Dipropylphosphinic acid butyl ester
*(butyl dipropylphosphinate)*;
$C_{10}H_{23}O_2P$; [16984-10-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.

*Zh. Fiz. Khim.* 1967, *41*, 1815-7.

VARIABLES:

$T$/K = 273 - 448

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of dipropylphosphinic acid butyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0.0 | 3.3[b] | 13.1[b] | 0.003[a] | 0.013[a] |
| 0.3 | - | 14.36 | - | 0.01443 |
| 2.4 | 3.12 | - | 0.002805 | - |
| 3.15 | - | 15.76 | - | 0.01608 |
| 4.2 | - | 16.62 | - | 0.01711 |
| 5.8 | 2.57 | - | 0.002299 | - |
| 7.4 | - | 19.70 | - | 0.02098 |
| 8.3 | - | 19.95 | - | 0.02130 |
| 13.6 | - | 30.08 | - | 0.03621 |
| 14.2 | 1.88 | - | 0.001671 | - |

[a] Experimental value.

[b] Calculated by the compiler.

(continued next page)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Alekseev's synthetic method was used. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized; distilled 5-6 times under vacuum; $d_4^{20}$ 0.9372, $n_D^{20}$ 1.4420.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Dipropylphosphinic acid butyl ester
(*butyl dipropylphosphinate*);
$C_{10}H_{23}O_2P$; [16984-10-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.;
Yakovlev, I.I.; Durasov, V.B.;
Yakovleva, N.I.; Khol'kina, I.D.

*Zh. Fiz. Khim.* 1967, *41*, 1815-7.

EXPERIMENTAL VALUES: (continued)

Mutual solubility of dipropylphosphinic acid butyl ester and water

| $t$/°C | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 17.2 | 1.69 | - | 0.001499 | - |
| 18.1 | - | 39.70 | - | 0.05438 |
| 20.8 | 1.56 | - | 0.001382 | - |
| 23.9 | - | 49.44 | - | 0.07869 |
| 33.8 | 1.04 | - | 0.000917 | - |
| 34.0 | - | 59.62 | - | 0.11423 |
| 53.0 | 0.651 | - | 0.000572 | - |
| 57.0 | - | 69.98 | - | 0.16916 |
| 60.7 | - | 70.76 | - | 0.17448 |
| 89.8 | - | 74.49 | - | 0.20321 |
| 139.0 | - | 74.49 | - | 0.20321 |
| 175.0 | - | 70.76 | - | 0.17448 |

COMPONENTS:

(1) Ethylphosphonic acid dibutyl ester
(*dibutyl ethylphosphonate*);
$C_{10}H_{23}O_3P$; [2404-58-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.

*Zh. Fiz. Khim.* 1967, *41*, 1815-7.

VARIABLES:

$T/K = 278 - 346$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of ethylphosphonic acid dibutyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 5.2 | - | 73.28 | - | 0.1819 |
| 8.6 | 1.51 | - | 0.001241 | - |
| 9.8 | - | 74.87 | - | 0.1945 |
| 16.2 | - | 77.06 | - | 0.2140 |
| 19.5 | 1.02 | - | 0.000835 | - |
| 29.5 | - | 79.94 | - | 0.2441 |
| 34.5 | 0.703 | - | 0.000574 | - |
| 44.4 | - | 83.31 | - | 0.2880 |
| 53.0 | 0.504 | - | 0.000410 | - |
| 73.0 | - | 84.62 | - | 0.3084 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Alekseev's synthetic method was used. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized; distilled 5-6 times under vacuum; $d_4^{20}$ 0.9658, $n_D^{20}$ 1.4290.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Heptanedioic acid diethyl ester (*diethyl heptanedioate*); $C_{11}H_{20}O_4$; [2050-20-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sobotka, H.; Kahn, J.

*J. Am. Chem. Soc.* 1931, *53*, 2935-8.

VARIABLES:

$T$/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of heptanedioic acid diethyl ester in water at 20°C was reported to be 0.199 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.199 g(1)/100g sln and $1.66 \times 10^{-4}$.

Density of water $d_4^{20}$ = 0.9982 (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 0.9945, $n_D^{20}$ 1.4303.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) Acetic acid isononyl ester (*isononyl acetate*); $C_{11}H_{22}O_2$; [40379-24-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Stephenson, R.; Stuart, J.

*J. Chem. Eng. Data* 1986, *31*, 56-70.

VARIABLES:

$T/K = 273 - 364$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

Mutual solubility of acetic acid isononyl ester and water

| $t/^\circ C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0 | 0.018 | 99.666 | 0.000017 | 0.96651 |
| 9.2 | 0.023 | 99.687 | 0.000022 | 0.96855 |
| 19.4 | 0.020 | 99.708 | 0.000019 | 0.97061 |
| 29.6 | 0.018 | 99.628 | 0.000017 | 0.96282 |
| 39.8 | 0.021 | 99.608 | 0.000020 | 0.96089 |
| 49.8 | 0.021 | 99.629 | 0.000020 | 0.96292 |
| 60.3 | 0.013 | 99.631 | 0.000012 | 0.96311 |
| 70.2 | 0.018 | 99.613 | 0.000017 | 0.96138 |
| 80.3 | 0.026 | 99.637 | 0.000025 | 0.96369 |
| 90.5 | 0.021 | 99.598 | 0.000020 | 0.95993 |
| std. dev. | 0.002 | 0.003 | | |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Component (1) was equilibrated with component (2) at a given temperature in a thermostat. Each layer was sampled with a syringe; (1) was determined by adding a weighed amount of acetonitrile (or sometimes propanol) to the organic layer sample and measuring by a Gow-Mac thermal conductivity gc the (1)/acetonitrile peak ratio (Chromosorb 101 packing and a HP 3390 A recorder-integrator).
A similar procedure but a higher boiling material (e.g. 1-hexanol) was used to determine (2) in the water layer.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercial sample; purity 98%; used as received.

(2) Not specified.

ESTIMATED ERROR:

Accuracy of method 0.1 wt% or less, for solubility, see above.

REFERENCES:

COMPONENTS:

(1) Decanoic acid methyl ester (*methyl decanoate*); $C_{11}H_{22}O_2$; [110-42-9]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Tewari, Y.B.; Miller, M.M.; Wasik, S.P.; Martire, D.E.

*J. Chem. Eng. Data* 1982, *27*, 451-4.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of decanoic acid methyl ester in water at 25°C was reported to be $2.05 \times 10^{-5}$ mol(1)/L sln. The corresponding value on mass/volume basis is 0.00382 g(1)/L sln (compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The aqueous phase was generated by pumping water into the inlet of a coated generator column which was thermostated, either by using a minipump or by means of a water reservoir using compressed air at 5 psi. The aqueous solution was extracted by the use of a known amount of immiscible-with-water solution and then analyzed by a gas chromatographic technique.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; purity >99% by glc.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.1°C.
Soly. ±1.0%.

REFERENCES:

COMPONENTS:

(1) Nonanoic acid ethyl ester (*ethyl nonanoate*); $C_{11}H_{22}O_2$; [123-29-5]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sobotka, H.; Kahn, J.

*J. Am. Chem. Soc.* 1931, *53*, 2935-8.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of Nonanoic acid ethyl ester in water at 20°C was reported to be 0.003 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.003 g(1)/100g sln and $3 \times 10^{-6}$.

Density of water $d_4^{20} = 0.9982$ (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $n_D^{20}$ 1.4223.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) Dibutylphosphinic acid propyl ester
(*propyl dibutylphosphinate*);
$C_{11}H_{25}O_2P$; [7100-93-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.

*Zh. Fiz. Khim.* 1966, *40*, 221-3.

VARIABLES:

$T$/K = 279 - 516

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of dibutylphosphinic acid propyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 6.1 | - | 65.22 | - | 0.13296 |
| 10.0 | 3.01 | - | 0.00253 | - |
| 13.7 | - | 69.63 | - | 0.15789 |
| 15.2 | - | 70.48 | - | 0.16336 |
| 20.5 | 2.01 | - | 0.00167 | - |
| 27.5 | - | 74.76 | - | 0.19499 |
| 29.0 | 1.50 | - | 0.00124 | - |
| 29.8 | - | 75.31 | - | 0.19964 |
| 41.0 | 1.01 | - | 0.00083 | - |
| 64.0 | 0.75 | - | 0.00062 | - |
| 66.5 | - | 80.58 | - | 0.25336 |
| 143.0 | - | 80.58 | - | 0.25336 |

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AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Alekseev's synthetic method was used. A water or glycerine bath was used for temperature control. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized; distilled 5-6 times under vacuum; $d_4^{20}$ 0.9298, $n_D^{20}$ 1.4439.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Dibutylphosphinic acid propyl ester<br>*(propyl dibutylphosphinate)*;<br>$C_{11}H_{25}O_2P$; [7100-93-8]<br>(2) Water; $H_2O$; [7732-18-5] | Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.<br>*Zh. Fiz. Khim.* 1966, *40*, 221-3. |

EXPERIMENTAL VALUES: (continued)

Mutual solubility of dibutylphosphinic acid propyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 176.0 | 1.41 | - | 0.00117 | - |
| 189.0 | - | 75.31 | - | 0.19964 |
| 197.0 | 2.24 | - | 0.00187 | - |
| 208.0 | - | 70.48 | - | 0.16336 |
| 217.0 | 4.05 | - | 0.00344 | - |
| 221.0 | - | 65.22 | - | 0.13296 |
| 230.0 | 6.86 | - | 0.00599 | - |
| 242.0 | 17.09 | 50.32 | 0.01658 | 0.07650 |
| 243.0 UCST | 35.08 | 35.08 | 0.04232 | 0.04232 |

The ester hydrolysis began at temperature 150-170°C.

COMPONENTS:

(1) Propylphosphonic acid dibutyl ester
(*dibutyl propylphosphonate*);
$C_{11}H_{25}O_3P$; [4628-12-0]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.

*Zh. Fiz. Khim.* 1967, *41*, 1815-7.

VARIABLES:

$T$/K = 274 - 316

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of propylphosphonic acid dibutyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 1.2 | - | 81.62 | - | 0.2529 |
| 9.5 | - | 83.43 | - | 0.2774 |
| 11.5 | 0.596 | - | $4.569 \times 10^{-4}$ | - |
| 15.7 | 0.446 | - | $3.414 \times 10^{-4}$ | - |
| 19.4 | 0.386 | - | $2.953 \times 10^{-4}$ | - |
| 22.4 | - | 85.38 | - | 0.3081 |
| 23.8 | 0.324 | - | $2.478 \times 10^{-4}$ | - |
| 33.0 | - | 86.36 | - | 0.3256 |
| 34.5 | 0.230 | - | $1.757 \times 10^{-4}$ | - |
| 43.0 | - | 87.06 | - | 0.3390 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Alekseev's synthetic method was used. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized; distilled 5-6 times under vacuum; $d_4^{20}$ 0.9569, $n_D^{20}$ 1.4309.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid diethyl ester
*(diethyl phthalate)*;
$C_{12}H_{14}O_4$; [84-66-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Shanley, E.S.; Greenspan, F.P.

*Ind. Eng. Chem.* 1947, *39*, 1536-43.

VARIABLES:

$T$/K = room temperature

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of water in 1,2-benzenedicarboxylic acid diethyl ester at room temperature was reported to be 1.0 g(2)/100g(1). The corresponding mass per cent and mole fraction, $x_2$, values calculated by the compiler are 0.99 g(2)/100g sln and 0.11.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid diethyl ester
*(diethyl phthalate)*;
$C_{12}H_{14}O_4$; [84-66-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Leyder, F.; Boulanger, P.

*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid diethyl ester in water at 20°C was reported to be $4.18 \times 10^{-3}$ mol(1)/L and 0.928 g(1)/L.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent flush" and "hot needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) Merck; purity 99% ; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

COMPONENTS:

(1) Octanedioic acid diethyl ester (*diethyl octanedioate*); $C_{12}H_{22}O_4$; [2050-23-9]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sobotka, H.; Kahn, J.

*J. Am. Chem. Soc.* 1931, *53*, 2935-8.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of octanedioic acid diethyl ester in water at 20°C was reported to be 0.068 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.068 g(1)/100g sln and $5.3 \times 10^{-5}$.

Density of water $d_4^{20} = 0.9982$ (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 0.9822, $n_D^{20}$ 1.4334.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) Decanoic acid ethyl ester (*ethyl decanoate*); $C_{12}H_{24}O_2$; [110-38-3]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sobotka, H.; Kahn, J.

*J. Am. Chem. Soc.* 1931, *53*, 2935-8.

VARIABLES:

$T$/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of decanoic acid ethyl ester in water at 20°C was reported to be 0.0015 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.0015 g(1)/100g sln and $1.3 \times 10^{-6}$.

Density of water $d_4^{20}$ = 0.9982 (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 0.8637, $n_D^{20}$ 1.4269.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) Dibutylphosphinic acid butyl ester
(*butyl dibutylphosphinate*);
$C_{12}H_{27}O_2P$; [2950-47-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakolvlev, I.I.; Durasov, V.B.; Mironova, Z.N.

*Izv. Sib. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* 1965, *(3)*, 27-31.

VARIABLES:

$T$/K = 273 - 429

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of dibutylphosphinic acid butyl ester and water

| $t$/°C | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0. | - | 73.65[a] | - | 0.1769 |
| 3.7 | 1.30 | - | $10.12 \times 10^{-4}$ | - |
| 6.1 | - | 75.46 | - | 0.1912 |
| 7.7 | 1.03 | - | $8.00 \times 10^{-4}$ | - |
| 12.0 | - | 77.27 | - | 0.2072 |
| 14.4 | 0.716 | - | $5.54 \times 10^{-4}$ | - |
| 17.5 | - | 78.54 | - | 0.2196 |
| 22.9 | 0.490 | - | $3.78 \times 10^{-4}$ | - |
| 24.4 | - | 79.95 | - | 0.2346 |
| 25.0 | 0.49[b] | 80.45[a] | $3.78 \times 10^{-4}$ | 0.2403 |
| 25.0 | 0.445 | - | $3.44 \times 10^{-4}$ | - |
| 26.5 | 0.409 | - | $3.16 \times 10^{-4}$ | - |

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AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The synthetic method proposed by Aleksejev was used. Light filters and a high intensity light source were used to measure the cloudiness point with good accuracy. As a check, two analytical methods were used: the Karl Fischer method for water in the organic phase and the extraction of dibutylphosphinic acid butyl ester with diethyl ether, stripping, and drying over $P_2O_5$.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized and purified as given by Nikolaev, Dyadin, Yakovlev and Mironova (ref 1); b.p. 138°C at 6 mm Hg, $d_4^{20}$ 0.9262, $n_D^{20}$ 1.4440.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±(0.2-0.6)°C below 30°C,
±(1-3)°C at high temperature.

REFERENCES:

1. Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Mironova, Z.N. *Dokl. Akad. Nauk SSSR* 1963, *153*, 118.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Dibutylphosphinic acid butyl ester<br>(*butyl dibutylphosphinate*);<br>$C_{12}H_{27}O_2P$; [2950-47-2]<br>(2) Water; $H_2O$; [7732-18-5] | Nikolaev, A.V.; Dyadin, Yu.A.; Yakolvlev, I.I.; Durasov, V.B.; Mironova, Z.N.<br>*Izv. Sib. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* 1965, *(3)*, 27-31. |

EXPERIMENTAL VALUES: (continued)

Mutual solubility of dibutylphosphinic acid butyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln |  | $x_1$ (compiler) |  |
|---|---|---|---|---|
|  | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 31.5 | 0.341 | - | $2.63 \times 10^{-4}$ | - |
| 32.5 | - | 81.19 | - | 0.2492 |
| 41.0 | 0.259 | - | $2.00 \times 10^{-4}$ | - |
| 43.2 | - | 82.38 | - | 0.2644 |
| 55.0 | - | 83.29 | - | 0.2771 |
| 57.5 | 0.200 | - | $1.54 \times 10^{-4}$ | - |
| 69.5 | 0.180 | - | $1.39 \times 10^{-4}$ | - |
| 70.7 | - | 84.12 | - | 0.2894 |
| 87.0 | 0.180 | - | $1.39 \times 10^{-4}$ | - |
| 124.0 | - | 84.12 | - | 0.2894 |
| 141.0 | - | 83.29 | - | 0.2771 |
| 156.0 | - | 82.38 | - | 0.2644 |

[a] Karl Fischer method.

[b] Extraction of dibutylphosphinic acid butyl ester with diethyl ether.

| | |
|---|---|
| COMPONENTS:<br>(1) Dibutylphosphinic acid butyl ester<br>(*butyl dibutylphosphinate*);<br>$C_{12}H_{27}O_2P$; [2950-47-2]<br>(2) Water; $H_2O$; [7732-18-5] | ORIGINAL MEASUREMENTS:<br>Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Durasov, V.B.; Yakovleva, N.I.; Khol'kina, I.D.<br>*Zh. Fiz. Khim.* 1966, *40*, 221-3. |
| VARIABLES:<br>*T*/K = 526 | PREPARED BY:<br>A. Skrzecz |

EXPERIMENTAL VALUES:

The UCST was reported to be 253°C.

The dibutylphosphinic acid butyl ester hydrolysis began at temperature 150-170°C. The hydrolisis measured in the sample containing 19.89wt% of ester after heating at 260°C through 20 min was 5-10%.

AUXILIARY INFORMATION

| | |
|---|---|
| METHOD/APPARATUS/PROCEDURE:<br>The Alekseev's synthetic method was used. A water or glycerine bath was used for temperature control. No further details were reported in the paper. | SOURCE AND PURITY OF MATERIALS:<br>(1) Synthesized; distilled 5-6 times under vacuum; $d_4^{25}$ 0.9179, $n_D^{25}$ 1.4442.<br>(2) Not specified. |
| | ESTIMATED ERROR:<br>Not specified. |
| | REFERENCES: |

COMPONENTS:

(1) Butylphosphonic acid dibutyl ester (*dibutyl butylphosphonate*); $C_{12}H_{27}O_3P$; [78-46-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakolvlev, I.I.; Durasov, V.B.; Mironova, Z.N.

*Izv. Sib. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* 1965, *(3)*, 27-31.

VARIABLES:

$T$/K = 275 - 419

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Mutual solubility of butylphosphonic acid dibutyl ester and water

| $t$/°C | g(1)/100g sln<br>(2)-rich phase | <br>(1)-rich phase | $x_1$ (compiler)<br>(2)-rich phase | <br>(1)-rich phase |
|---|---|---|---|---|
| 2.2 | - | 86.00 | - | 0.3066 |
| 6.0 | 0.247 | - | $17.82 \times 10^{-5}$ | - |
| 13.3 | - | 87.47 | - | 0.3344 |
| 13.4 | 0.168 | - | $12.11 \times 10^{-5}$ | - |
| 20.5 | - | 88.12 | - | 0.3480 |
| 21.0 | 0.121 | - | $8.72 \times 10^{-5}$ | - |
| 24.1 | - | 88.44 | - | 0.3551 |
| 28.6 | 0.0985 | - | $7.10 \times 10^{-5}$ | - |
| 35.0 | 0.0853 | - | $6.14 \times 10^{-5}$ | - |
| 39.5 | - | 88.29 | - | 0.3750 |

(continued next page)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The synthetic method proposed by Aleksejev was used. Light filters and a high intensity light source were used to measure the cloudiness point with good accuracy. No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized and purified as given by Nikolaev, Dyadin, Yakovlev and Mironova (ref 1); b.p. 133-5°C at 6 mm Hg, $d_4^{20}$ 0.9472, $n_D^{20}$ 1.4323.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±(0.2-0.6)°C below 30°C,
±(1-3)°C at high temperature.

REFERENCES:

1. Nikolaev, A.V.; Dyadin, Yu.A.; Yakovlev, I.I.; Mironova, Z.N. *Dokl. Akad. Nauk SSSR* 1963, *153*, 118.

COMPONENTS:

(1) Butylphosphonic acid dibutyl ester
*(dibutyl butylphosphonate)*;
$C_{12}H_{27}O_3P$; [78-46-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakolvlev, I.I.; Durasov, V.B.; Mironova, Z.N.

*Izv. Sib. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* 1965, *(3)*, 27-31.

EXPERIMENTAL VALUES: (continued)

Mutual solubility of butylphosphonic acid dibutyl ester and water

| $t/^\circ C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 43.9 | 0.0688 | - | $4.95 \times 10^{-5}$ | - |
| 48.0 | 0.0591 | - | $4.26 \times 10^{-5}$ | - |
| 51.8 | - | 89.76 | - | 0.3868 |
| 96.0 | - | 89.76 | - | 0.3868 |
| 100.0 | 0.0591 | - | $4.26 \times 10^{-5}$ | - |
| 106.0 | 0.0688 | - | $4.95 \times 10^{-5}$ | - |
| 109.0 | - | 88.29 | - | 0.3750 |
| 126.0 | - | 88.44 | - | 0.3551 |
| 146.0 | - | 87.47 | - | 0.3344 |

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION:

Quantitative solubility data for the phosphoric acid tributyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the Phosphoric acid tributyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Alcock *et al.* (ref 1) | 289-298 | mutual | analytical |
| Burger and Forsman (ref 2) | 293 | mutual | unspecified |
| Higgins *et al.* (ref 3) | 277-323 | (1) in (2) | analytical |
| Hasegawa (ref 4) | 298 | (1) in (2) | titration |
| Mikhailov *et al.* (ref 5) | 279-299 | (2) in (1) | Karl Fischer, synthetic |
| Hardy *et al.* (ref 6) | 273-333 | (2) in (1) | Karl Fischer |
| Nikolaev *et al.* (ref 7) | 273-460 | mutual | Karl Fischer, synthetic |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

1. SOLUBILITY OF PHOSPHORIC ACID TRIBUTYL ESTER (1) IN WATER (2)

All the available data for the solubility of phosphoric acid tributyl ester (1) in water (2) are summarized in Table 2 with the following exceptions. The data of Alcock *et al.* (ref 1) are much lower, and that of Hasegawa (ref 4) much higher, than all other studies and have therefore been rejected.

Agreement among the remaining data (Table 2) is in general good and a number of the average "Best" values have been Recommended. At higher temperatures ($T$ > 323 K) only the data of Nikolaev (ref 7) are available and must therefore be considered as Tentative. Selected data are plotted in Figure 1.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Recommended (*R*) and Tentative Solubilities of Phosphoric acid tributyl ester (1) in Water (2)

| $T/K$ | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values $(\pm\sigma_n)^a$ | |
| | $10^2$ g(1)/100g sln | $10^2$ g(1)/100g sln | $10^5x_1$ |
| 283 | 7.5* (ref 3), 7.5* (ref 7) | 7.5 (*R*) | 5.1 |
| 293 | 4.8* (ref 3), 4.6* (ref 7) | 4.7 ± 0.1 (*R*) | 3.2 |
| 298 | 3.9 (ref 2), 4.2* (ref 3), 3.95* (ref 7) | 4.0 ± 0.1 (*R*) | 2.7 |
| 303 | 3.8* (ref 3), 3.5* (ref 7) | 3.7 ± 0.2 (*R*) | 2.5 |
| 313 | 3.4* (ref 3), 2.9* (ref 7) | 3.2 ± 0.3 | 2.2 |
| 323 | 2.9* (ref 3), 2.6* (ref 7) | 2.8 ± 0.2 | 1.9 |
| 333 | 2.5* (ref 7) | 2.5 | 1.7 |
| 343 | 2.6* (ref 7) | 2.6 | 1.8 |
| 353 | 2.9* (ref 7) | 2.9 | 2.0 |
| 363 | 3.6* (ref 7) | 3.6 | 2.5 |
| 373 | 5.0* (ref 7) | 5.0 | 3.4 |

[a] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance. Mole fraction solubilities ($x_1$) have the same status and (relative) percentage uncertainties as the mass % solubilities.

## 2. SOLUBILITY OF WATER (2) IN PHOSPHORIC ACID TRIBUTYL ESTER (1)

All the available data for the solubility of water (2) in phosphoric acid tributyl ester (1) are summarized in Table 3 with the exception of the datum of Alcock *et al.* (ref 1) which is lower than all other studies and has therefore been rejected.

The remaining data are in good agreement (Table 3) enabling a number of average "Best" values to be Recommended. At higher temperatures ($T$ > 343 K) only the data of Nikolaev (ref 7) are available and must therefore be considered as Tentative. Selected data are plotted in Figure 2.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

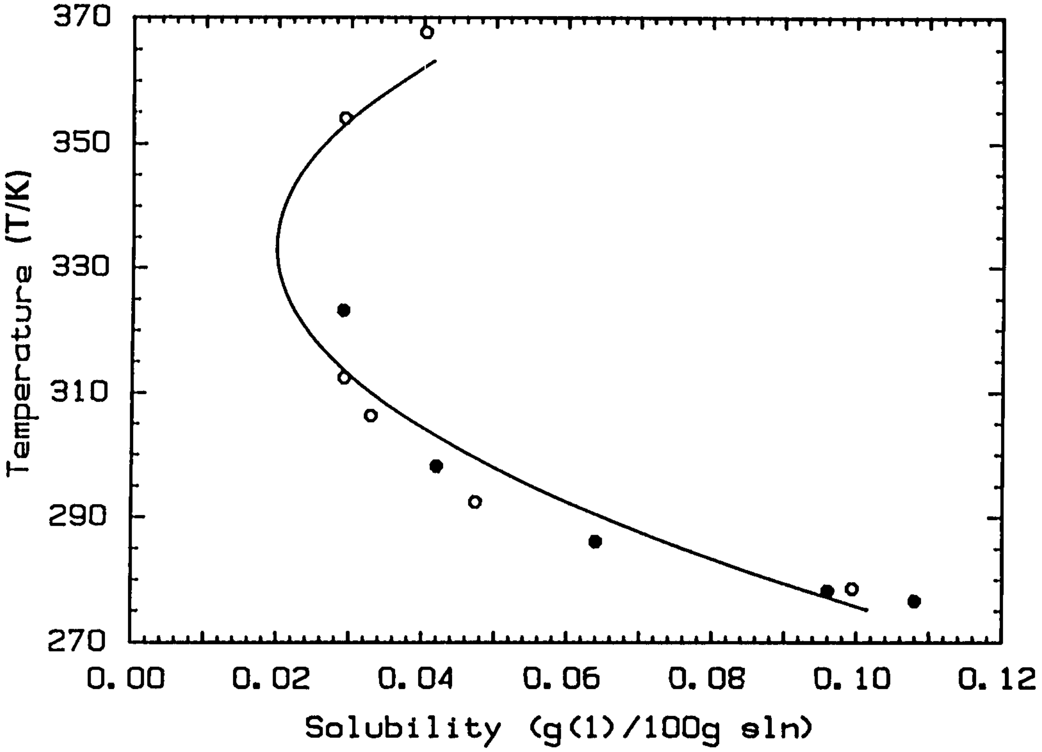

FIGURE 1. Selected data for the solubility of phosphoric acid tributyl ester (1) in water (2): ref 3 (●); ref 7 (O). Solid line is a least square polynomial fitted to the "Best" values from Table 2.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 3: Recommended (*R*) and Tentative Solubilities of Water (2) in Phosphoric acid tributyl ester (1)

| $T/\mathrm{K}$ | Solubilities | | |
|---|---|---|---|
| | Reported values | "Best" values $(\pm\sigma_n)^a$ | |
| | g(2)/100g sln | g(2)/100g sln | $x_2$ |
| 273 | 7.09 (ref 6), 7.52 (ref 7) | 7.3 ± 0.2 (*R*) | 0.54 |
| 283 | 6.38$^*$ (ref 5), 6.92$^*$ (ref 6), 7.05$^*$ (ref 7) | 6.8 ± 0.3 (*R*) | 0.52 |
| 293 | 6.24$^*$ (ref 5), 6.73$^*$ (ref 6), 6.70$^*$ (ref 7) | 6.6 ± 0.2 (*R*) | 0.51 |
| 298 | 6.24 (ref 5), 6.64 (ref 6), 6.56 (ref 7) | 6.5 ± 0.2 (*R*) | 0.51 |
| 303 | 6.53$^*$ (ref 6), 6.45$^*$ (ref 7) | 6.49 ± 0.04 (*R*) | 0.507 |
| 313 | 6.31$^*$ (ref 6), 6.40$^*$ (ref 7) | 6.36 ± 0.05 (*R*) | 0.501 |
| 323 | 6.08 (ref 6), 6.37$^*$ (ref 7) | 6.2 ± 0.1 (*R*) | 0.49 |
| 333 | 5.82 (ref 6), 6.40$^*$ (ref 7) | 6.1 ± 0.3 (*R*) | 0.49 |
| 343 | 6.60$^*$ (ref 7) | 6.6 | 0.51 |
| 353 | 6.85$^*$ (ref 7) | 6.9 | 0.52 |
| 363 | 7.17$^*$ (ref 7) | 7.2 | 0.53 |
| 373 | 7.55$^*$ (ref 7) | 7.6 | 0.55 |
| 393 | 8.40$^*$ (ref 7) | 8.4 | 0.58 |
| 413 | 9.3$^*$ (ref 7) | 9 | 0.59 |
| 433 | 10.1$^*$ (ref 7) | 10 | 0.62 |
| 453 | 10.9$^*$ (ref 7) | 11 | 0.65 |

[a] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance. Mole fraction solubilities ($x_2$) have the same status and (relative) percentage uncertainties as the mass % solubilities.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

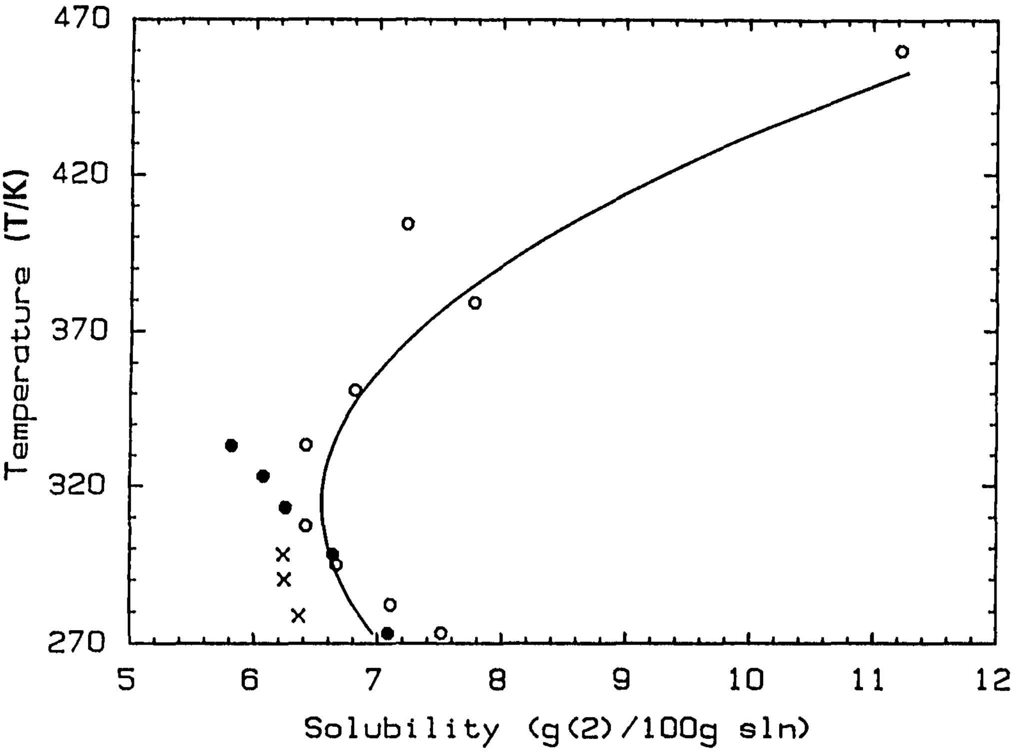

FIGURE 2. Selected data for the solubility of water (2) in phosphoric acid tributyl ester (1): ref 5 (X); ref 6 (●); ref 7 (O). Solid line is a least square polynomial fitted to the "Best" values from Table 3.

REFERENCES

1. Alcock, K.; Grimley, S. S.; Healy, T. V.; Kennedy, J.; McKay, H. A. C. *Trans. Faraday Soc.* 1956, *52*, 39-47.

2. Burger, L. L.; Forsman, R. C. Report HW-20936, 1951; quoted in Bruce, F. R. *et al. Progress in Nuclear Energy, Series 3, Process Chemistry*, Vol. 2, Pergamon Press, 1958, 546-56.

3. Higgins, C. E.; Baldwin, W. H.; Soldano, B. A. *J. Phys. Chem.* 1959, *63*, 113-8.

4. Hasegawa, T. *Kogyo Kagaku Zasshi* 1961, *64*, 1239-41.

5. Mikhailov, V. A.; Kharachenko, S. K.; Nazin, A. G. *Izv. Sib. Otd. Akad. Nauk SSSR* 1962, (7), 50-6.

6. Hardy, C. J.; Fairhurst, D.; McKay, H. A. C.; Willson, A. M. *Trans. Faraday Soc.* 1964, *60*, 1626-36.

7. Nikolaev, A. V.; Dyadin, Yu. A.; Yakovlev, I. I.; Durasov, V. B.; Mironova, Z. N. *Izv. Sib. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* 1965, (3), 27-31.

COMPONENTS:

(1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Alcock, K.; Grimley, S.S.; Healy, T.V.; Kennedy, J.; McKay, H.A.C.

*Trans. Faraday Soc.* 1956, *52*, 39-47.

VARIABLES:

$T$/K = 289 - 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Solubility of phosphoric acid tributyl ester in water

| $t$/°C | g(1)/dm$^3$ | mol(1)/dm$^3$ sln (compiler) |
|---|---|---|
| 16. | 0.420 | 0.001577 |
| 17. | 0.410 | 0.001540 |
| 19. | 0.397 | 0.001491 |
| 22. | 0.380 | 0.001427 |

Solubility of water in phosphoric acid tributyl ester

| $t$/°C | mol(1)/dm$^3$ sln | g(2)/dm$^3$ sln (compiler) |
|---|---|---|
| 25. | 3.22 | 58.0 |
| 25. | 3.43 | 61.8 |

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. 0.5 cm$^3$ of $^{32}$P-labelled organic phase was shaken with 50 cm$^3$ of aq. phase for 20 min. After a short standing period, the aqueous phase was separated and centrifuged. A sample was diluted 10-fold, and 10 cm$^3$ were placed in a GM6 liquid counter for counting and comparison with a suitable standard. The method was reported together with the systems phosphoric acid tributyl ester-water-diluent-nitric acid.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; purified by boiling with dilute NaOH solution to distill volatile impurities; the remaining portion washed repeatedly with water. $^{32}$P-labelled (1) supplied by Radiochemical Center, Amersham; purified as described above.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Burger, L.L.; Forsman, R.C.
*Report HW-20936*, 1951.

Data taken from: Bruce, F.R.;
Fletcher, J.M.; Hyman, H.H.
*Progress in Nuclear Energy*, ser.3,
Process Chemistry vol.2,
Pergamon Press, 1958, 546-56.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of phosphoric acid tributyl ester in water at 25°C was reported to be 0.39 g(1)/$dm^3$ sln. The corresponding value on a mole/volume basis is 0.00146 mol(1)/$dm^3$.

The solubility of water in phosphoric acid tributyl ester at 25°C was reported to be 64 g(2)/$dm^3$ sln. The corresponding value on a mole/volume basis is 3.55 mol(2)/$dm^3$ sln.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method was not specified.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Not specified.

REFERENCES:

COMPONENTS:

(1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Higgins, C.E.; Baldwin, W.H.; Soldano, B.A.

*J. Phys. Chem.* 1959, *63*, 113-8.

VARIABLES:

$T/K = 277 - 323$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Solubility of phosphoric acid tributyl ester in water

| $t/^{\circ}C$ | mg(1)/dm$^3$ sln | mol(1)/dm$^3$ sln |
|---|---|---|
| 3.4 | 1075 | 0.0040 |
| 4.0 | 1012 | 0.0038[b] |
| 5.0 | 957 | 0.0036[b] |
| 13.0 | 640 | 0.0024[b] |
| 25.0 | 422[a] | 0.0016 |
| 50.0 | 285 | 0.0011 |

[a] The average of 30 measurments.

[b] Calculated by the compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The mixture of water and ester labeled with $^{32}P$ were tumbled in a constant temperature bath for 1/2 to 1 h. and stored overnight to ensure complete separation. The aqueous phase was analysed by counting a dilution of an aliquot containing sufficient activity in the solution counter as described by Higgins and Baldwin (ref 1). The specific activity of the labeled ester was measured at the same time for comparison. The averages of at least two separated determinations were presented.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, C.P. grade; tributyl phosphate-$^{32}P$ prepared in the laboratory as described by Higgins and Baldwin (ref 2); specific activity 40-50(micro)c/mmole.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.2°C.
Soly. ±1.8% (the largest average deviation of a single determination at 25°C).

REFERENCES:

1. Higgins, C.E.; Baldwin, W.H. *Anal. Chem.* 1955, *27*, 1780.
2. Higgins, C.E.; Baldwin, W.H. *J. Org. Chem.* 1956, *21*, 1156.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]<br>(2) Water; $H_2O$; [7732-18-5] | Hasegawa T.<br>*Kogyo Kagaku Zasshi* 1961, *64(7)*, 1239-41. |
| VARIABLES: | PREPARED BY: |
| $T$/K = 298 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of phosphoric acid tributyl ester in water 25°C was reported to be 6.8 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 0.0049.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The titration method was used. The cloudiness was observed visually. The data and method were reported together with the ternary systems phosphoric acid tributyl ester-water-2-furancarbonal (*tributyl phosphate-water-furfural*). No further details were reported in the paper. | (1) Source not specified; boiled with 5 volumes of 0.4% NaOH(aq) washed several times with water, distilled at reduced pressure; b.p. 132°C at 9 mm Hg, $d_4^{25}$ 0.9538.<br>(2) Not specified. |
| | ESTIMATED ERROR: |
| | Not specified. |
| | REFERENCES: |

COMPONENTS:

(1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Mikhailov, V.A.; Kharchenko, S.K.; Nazin, A.G.

*Izv. Sib. Otd. Akad. Nauk SSSR* 1962, *(7)*, 50-6.

VARIABLES:

$T$/K = 279 - 299

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Solubility of water in phosphoric acid tributyl ester

| $t$/°C | g(2)/100g sln | $x_2$ (compiler) | |
|---|---|---|---|
| 5.5[b] | 6.37 | 0.501 | |
| 17.[b] | 6.25 | 0.496 | |
| 25.[a] | 6.244(0.014)[d] | 0.4961(0.0005) | (authors) |
| 25.[c] | 6.38 | 0.502 | |
| 26.[b] | 6.22 | 0.495 | |

[a] Analytical method.

[b] Alekseev's synthetic method.

[c] Isopiestic method.

[d] The mean of 8 experiments.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical, Alekseev's synthetic, and the isopieztic methods were used. In the analytical method, the phases were separated, after thermostating 2-3 h., using a centrifuge for 20 sec. (for most accurate results), and the water was titrated with Karl Fischer reagent. For the Alekseev's method, sealed ampoules containing a known sample composition along with a magnetic stirrer were placed into a water bath and the temperature of cloudiness was observed. In the isopiestic procedure, the assumption that the ester phase was in equilibrium with pure water was made. No further details were provided in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; distilled at vacuum over solid $Na_2CO_3$; $d_4^{25}$ 0.9728, $n_D^{25}$ 1.4244.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.05°C[a] and ±(1-2)°C[b].

REFERENCES:

COMPONENTS:

(1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Hardy, C.J.; Fairhurst, D.; McKay, H.A.C.; Willson, A.M.

*Trans. Faraday Soc.* 1964, *60*, 1626-36.

VARIABLES:

$T/K = 273 - 333$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

Solubility of water in phosphoric acid tributyl ester

| $t/^{\circ}C$ | mol(2)/dm$^3$ sln | density g/cm$^3$ | g(2)/100g sln (compiler) | $x_2$ (compiler) |
|---|---|---|---|---|
| 0.(0.2) | 3.92 | 0.9982 | 7.09 | 0.5296 |
| 25.(0.2) | 3.59(0.03)[a] | 0.9760 | 6.64 | 0.5120 |
| 40.(0.1) | 3.34 | 0.9640 | 6.26 | 0.4960 |
| 50.(0.1) | 3.23 | 0.9573[b] | 6.08 | 0.4889 |
| 60.(0.1) | 3.07 | 0.9508[b] | 5.82 | 0.4773 |

[a] The average of 17 determinations (each in duplicate): 5 by shaking, 5 by "very fast" stirring, 3 by "fast" stirring and 4 by saturation with water vapour.

[b] Calculated from authors' equation:

$$d_t = 0.9982\ (1.0 - 9.6 \times 10^{-4}\ (t/^{\circ}C) + 2.8 \times 10^{-6}\ (t/^{\circ}C)^2)$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The solutions were prepared by shaking together known weights of components for 15 min or by saturating ester with steam. The ester was equilibrated with water at 25°C by:

(i) shaking in a mechanical shaker for 15 minutes and centrifuged,

(ii) stirring 10 cm$^3$ of each phase in a 40 cm$^3$ centrifuge tube for 15-60 min at one of 3 different speeds (slow, fast, very fast)

(iii) bubbling nitrogen saturated with steam through the tube containing the pure ester; water concentration becoming constant after 24 h.

The phases at 0°C were separated by centrifugation and at 40-60°C they were settled for 1 h. in a stoppered tube at the required temperature. The water concentration was determined by the standard Karl Fischer reagent using the dead-stop end-point method.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, commercially available; washed and heated with 0.1 N alkali, volatile matter distilled in a stream of nitrogen, the remaining alkaline solution washed six times with water, dried under vacuum at 40-50°C with a nitrogen bleed, stored in a dark glass stoppered bottle.

(2) Not specified.

ESTIMATED ERROR:

See above.

REFERENCES:

### COMPONENTS:

(1) Phosphoric acid tributyl ester
*(tributyl phosphate)*;
$C_{12}H_{27}O_4P$; [126-73-8]

(2) Water; $H_2O$; [7732-18-5]

### ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakolvlev, I.I.; Durasov, V.B.; Mironova, Z.N.

*Izv. Sib. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* 1965, *(3)*, 27-31.

### VARIABLES:

$T$/K = 273 - 460

### PREPARED BY:

A. Skrzecz

### EXPERIMENTAL VALUES:

Mutual solubility of phosphoric acid tributyl ester and water

| $t/^{\circ}C$ | g(1)/100g sln | | $x_1$ (compiler) | |
|---|---|---|---|---|
| | (2)-rich phase | (1)-rich phase | (2)-rich phase | (1)-rich phase |
| 0.$^{a}$ | - | 92.48 | - | 0.4541 |
| 4.6 | - | 92.70 | - | 0.4621 |
| 5.4 | 0.0994 | - | $6.73 \times 10^{-5}$ | - |
| 9.1 | - | 92.89 | - | 0.4692 |
| 13.9 | 0.0620 | - | $4.20 \times 10^{-5}$ | - |
| 14.6 | - | 93.09 | - | 0.4756 |
| 16.3 | - | 93.18 | - | 0.4803 |
| 19.3 | 0.0474 | - | $3.21 \times 10^{-5}$ | - |
| 21.6 | - | 93.33 | - | 0.4863 |
| 24.2 | - | 93.38 | - | 0.4883 |
| 24.7 | 0.0403 | - | $2.73 \times 10^{-5}$ | - |
| 25.0$^{a}$ | - | 93.44 | - | 0.4907 |

(continued next page)

### AUXILIARY INFORMATION

### METHOD/APPARATUS/PROCEDURE:

The synthetic method proposed by Aleksejev was used. Light filters and a high intensity light source were used to measure the cloudiness point with good accuracy. The Karl Fischer analytical method was used as a check. No further details were reported in the paper.

### SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, pure grade; washed several times with NaOH(aq), diluted HCL, $H_2O$, three times distilled under vacuum; b.p. 132-3°C at 3 mm Hg, $d_4^{25}$ 0.9723, $n_D^{25}$ 1.4224.

(2) Not specified.

### ESTIMATED ERROR:

Temp. ±(0.2-0.6)°C below 30°C,
±(1-3)°C at high temperature.

### REFERENCES:

COMPONENTS:

(1) Phosphoric acid tributyl ester (*tributyl phosphate*); $C_{12}H_{27}O_4P$; [126-73-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Nikolaev, A.V.; Dyadin, Yu.A.; Yakolvlev, I.I.; Durasov, V.B.; Mironova, Z.N.

*Izv. Sib. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk* 1965, *(3)*, 27-31.

EXPERIMENTAL VALUES: (continued)

Mutual solubility of phosphoric acid tributyl ester and water

| t/°C | g(1)/100g sln<br>(2)-rich phase | <br>(1)-rich phase | $x_1$ (compiler)<br>(2)-rich phase | <br>(1)-rich phase |
|---|---|---|---|---|
| 25.1 | - | 93.41 | - | 0.4895 |
| 25.3 | - | 93.46 | - | 0.4915 |
| 29.5 | - | 93.52 | - | 0.4940 |
| 33.1 | 0.0329 | - | $2.23 \times 10^{-5}$ | - |
| 34.2 | - | 93.58 | - | 0.4965 |
| 39.2 | 0.0292 | - | $1.98 \times 10^{-5}$ | - |
| 60.2 | - | 93.58 | - | 0.4965 |
| 65.0 | - | 93.52 | - | 0.4940 |
| 69.5 | - | 93.41 | - | 0.4895 |
| 71.0 | - | 93.38 | - | 0.4883 |
| 78.0 | - | 93.18 | - | 0.4803 |
| 80.8 | 0.0292 | - | $1.98 \times 10^{-5}$ | - |
| 82.0 | - | 93.09 | - | 0.4756 |
| 86.0 | 0.0329 | - | $2.23 \times 10^{-5}$ | - |
| 94.5 | 0.0403 | 92.70 | $2.73 \times 10^{-5}$ | 0.4621 |
| 106.0 | - | 92.22 | - | 0.4450 |
| 117.0 | - | 91.72 | - | 0.4284 |
| 131.0 | - | 92.77 | - | 0.4646 |
| 134.0 | - | 89.02 | - | 0.3542 |
| 187.0 | - | 88.77 | - | 0.3484 |

[a] Karl Fischer analytical method.

COMPONENTS:

(1) Phosphoric acid tris(2-methylpropyl) ester (*tri(2-methylpropyl) phosphate*); $C_{12}H_{27}O_4P$; [126-71-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Apelblat, A.

*J. Chem. Soc.* B 1969, 175-7.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of water in phosphoric acid tris(2-methylpropyl) ester at 25°C was reported to be 3.50 mol(2)/L sln. The corresponding value on a mole/volume basis is 63.1 g(2)/L sln (compiler).

The density of the ester-phase was reported was to be $d^{25}$ 0.9655.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The mixture was equilibrated by vigorous stirring for 30 min. and settling for 12 h. The organic phase was analyzed with the Karl Fischer reagent. Preliminary experiments showed that stirring and settling times were sufficient.

SOURCE AND PURITY OF MATERIALS:

(1) Albright and Wilson Co; purified by method described in (ref 1); $d_4^{25}$ 0.9614, $n_D^{25}$ 1.4168.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.1°C.

REFERENCES:

1. Alcock, K.; Grimley, S.S.; Healy, T.V.; Kennedy, J.; McKay, H.A.C. *Trans. Faraday Soc.* 1956, *52*, 39.

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Benzoic acid, 2-hydroxy-, phenyl ester (*phenyl salicylate*); $C_{13}H_{10}O_3$; [118-55-8]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>March, 1990 |

CRITICAL EVALUATION:

Quantitative solubility data for the 2-hydroxybenzoic acid phenyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the 2-Hydroxybenzoic acid phenyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility<br>g(1)/100 g sln | Method |
|---|---|---|---|
| Seidell (ref 1) | 298 | 0.015 | gravimetric |
| Dehn (ref 2) | RT[a] | 0.04 | gravimetric |

[a] Room temperature (293-298 K).

As can be seen from Table 1, the data of Seidell (ref 1) and Dehn (ref 2) for the solubility of phenyl salicylate (1) in water (2) are not in particularly good agreement. In the absence of further information it is not possible to prefer either value although it may be noted that Seidell's data in other systems are generally close to Recommended values.

REFERENCES

1. Seidell, A. *Hygienic Lab. Bull.* 1910, No. 67, 98pp. (U.S. Govt. Printing Office, Washington, DC).
2. Dehn, W. M. *J. Am. Chem. Soc.* 1917, *39*, 1399-404.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Benzoic acid, 2-hydroxy-, phenyl ester (*phenyl salicylate*); $C_{13}H_{10}O_3$; [118-55-8]<br>(2) Water; $H_2O$; [7732-18-5] | Seidell, A.<br>*Hygienic Lab. Bull.* 1910, No. 67, 98 pp (US Govt. Printing Office, Washington, DC). |
| VARIABLES: | PREPARED BY: |
| $T$/K = 298 | G.T. Hefter |

EXPERIMENTAL VALUES:

The solubility of 2-hydroxybenzoic acid phenyl ester in water at 25°C was reported to be 0.015 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $1.3 \times 10^{-5}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Appropriate quantities of (1) and (2) were shaken together for two days. The amount of dissolved (1) was determined by evaporation of a known quantity of the saturated solution. The residue was dried to a constant weight in a vacuum desiccator.

SOURCE AND PURITY OF MATERIALS:

(1) Source not stated; purity determined to be practically 100% by saponification.

(2) Distilled (no details given).

ESTIMATED ERROR:

Temp. not stated.
Soly. not stated.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Benzoic acid, 2-hydroxy-, phenyl ester (*phenyl salicylate*); $C_{13}H_{10}O_3$; [118-55-8]<br>(2) Water; $H_2O$; [7732-18-5] | Dehn, W.M.<br>*J. Am. Chem. Soc.* 1917, *39*, 1399-1404. |
| VARIABLES: | PREPARED BY: |
| $T$/K = room temperature | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of 2-hydroxybenzoic acid phenyl ester in water at room temperature[a] was reported to be 0.04 g(1)/100g(1). The corresponding mass percentage and mole fraction, $x_1$, values calculated by the compiler are 0.040 g(1)/100g sln and $3.4 \times 10^{-5}$.

[a] Room temperature was reported to be 20-25°C.

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| Excess of the substance and 5 mL of water were enclosed in small vials which, when stoppered, were shaken or were left until equilibrium was established. The solutions were filtered into weighed crucibles and reweighed. After drying in a vacuum desiccator or on a water bath, the crucibles were again weighed and the loss of solvent calculated. The measurements were made at room temperatures from 20 to 25°C. | (1) Source not specified, commercial grade; used as received.<br>(2) Not specified. |
| | ESTIMATED ERROR: |
| | Not specified. |
| | REFERENCES: |

COMPONENTS:

(1) Phosphoric acid methyl diphenyl ester (*methyl diphenyl phosphate*); $C_{13}H_{13}O_4P$; [115-89-9]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Apelblat, A.

*J. Chem. Soc.* B 1969, 175-7.

VARIABLES:

$T/K = 298$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of water in phosphoric acid methyl diphenyl ester at 25°C was reported to be 1.49 mol(2)/L sln. The corresponding value on a mass/volume basis is 26.8 g(2)/L sln (compiler).

The density of the ester-phase was reported was to be $d^{25}$ 1.2195.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The mixture was equilibrated by vigorous stirring for 30 min. and settling for 12 h. The organic phase was analyzed with the Karl Fischer reagent. Preliminary experiments showed that stirring and settling times were sufficient.

SOURCE AND PURITY OF MATERIALS:

(1) Aldrich Chemical Co.; purified by method described by Alcock, Grimley, Healy, Kennedy and McKay (ref 1); $d_4^{25}$ 1.2258, $n_D^{25}$ 1.5356.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.1°C.

REFERENCES:

1. Alcock, K.; Grimley, S.S.; Healy, T.V.; Kennedy, J.; McKay, H.A.C. *Trans. Faraday Soc.* 1956, *52*, 39.

COMPONENTS:

(1) Nonanedioic acid diethyl ester (*diethyl nonanedioate*); $C_{13}H_{24}O_4$; [624-17-9]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sobotka, H.; Kahn, J.

*J. Am. Chem. Soc.* 1931, *53*, 2935-8.

VARIABLES:

$T$/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of nonanedioic acid diethyl ester in water at 20°C was reported to be 0.025 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.025 g(1)/100g sln and 1.8 x $10^{-5}$.

Density of water $d_4^{20}$ = 0.9982 (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 0.9729, $n_D^{20}$ 1.4358.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid di-2-propenyl ester (*diallyl phthalate*); $C_{14}H_{14}O_4$; [131-17-9]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Leyder, F.; Boulanger, P.

*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid di-2-propenyl ester in water at 20°C was reported to be $7.39 \times 10^{-4}$ mol(1)/L and 0.182 g(1)/L.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent flush" and "hot needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) CEPEA; purity not specified; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid bis(1-methylethyl) ester (*diisopropyl phthalate*); $C_{14}H_{18}O_4$; [605-45-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Leyder, F.; Boulanger, P.

*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid bis(1-methylethyl) ester in water at 20°C was reported to be $1.33 \times 10^{-3}$ mol(1)/L and 0.332 g(1)/L.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent flush" and "hot needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) Chrompack, stationary phase for gas chromatography; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) 1,2-Benzenedicarboxylic acid dipropyl ester (*dipropyl phthalate*); $C_{14}H_{18}O_4$; [131-16-8]<br>(2) Water; $H_2O$; [7732-18-5] | Leyder, F.; Boulanger, P.<br>*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7. |
| **VARIABLES:** | **PREPARED BY:** |
| $T$/K = 293 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid dipropyl ester in water at 20°C was reported to be $4.32 \times 10^{-4}$ mol(1)/L and 0.108 g(1)/L.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent flush" and "hot needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) Chrompack, stationary phase for gas chromatography; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

COMPONENTS:

(1) Decanedioic acid diethyl ester (*diethyl decanedioate*); $C_{14}H_{26}O_4$; [110-40-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sobotka, H.; Kahn, J.

*J. Am. Chem. Soc.* 1931, *53*, 2935-8.

VARIABLES:

$T$/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of decanedioic acid diethyl ester in water at 20°C was reported to be 0.008 g(1)/100mL(2). The corresponding mass per cent and mole fraction, $x_1$, values calculated by the compiler are 0.008 g(1)/100g sln and 6 x $10^{-6}$.

Density of water $d_4^{20}$ = 0.9982 (ref 1) was used in the calculation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used. The ester was added dropwise from a micro-burette with a capillary tip to 100, 250, or 500 mL of water in a narrow-mouthed stock bottle with a well-ground glass stopper. The bottle was shaken after each addition of ester. 1-5 mg of Sudan IV dye was put into the water to improve the end-point of the titration. At saturation, one additional drop of ester was sufficient to convert the floating rough indicator particles into dark transparent droplets.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified (Eastman Kodak Laboratories or synthesized); twice-distilled under reduced pressure; $d_4^{20}$ 0.9646, $n_D^{20}$ 1.4368.

(2) Distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. *Selected Values of Properties of Hydrocarbons and Related Compounds*, API Research Project 44, Thermodynamics Research Center, Texas A and M University, Texas, 1973.

COMPONENTS:

(1) Phosphoric acid dibutyl methylphenyl ester (*dibutyl tolyl phosphate*); $C_{15}H_{25}O_4P$; [26446-69-5]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Apelblat, A.

*J. Chem. Soc.* B 1969, 175-7.

VARIABLES:

$T/K = 298$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of water in phosphoric acid dibutyl methylphenyl ester at 25°C was reported to be 1.50 mol(2)/L sln. The corresponding value on a mass/volume basis is 27.0 g(2)/L sln (compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The mixture was equilibrated by vigorous stirring for 30 min. and settling for 12 h. The organic phase was analyzed with the Karl Fischer reagent. Preliminary experiments showed that stirring and settling times were sufficient.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized at the Plastics Research Laboratory of the Weizmann Institute of Science; used as received; b.p. 132-5°C at 0.3 mm Hg, $n_D^{25}$ 1.4722.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.1°C.

REFERENCES:

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid bis(2-methylpropyl) ester (*diisobutyl phthalate*); $C_{16}H_{22}O_4$; [84-69-5]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Leyder, F.; Boulanger, P.

*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7.

VARIABLES:

$T/K = 293$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid bis(2-methylpropyl) ester in water at 20°C was reported to be $7.30 \times 10^{-5}$ mol(1)/L and 0.0203 g(1)/L.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent flush" and "hot needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) BASF "Palatinol IC"; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) 1,2-Benzenedicarboxylic acid dibutyl ester (*dibutyl phthalate*); $C_{16}H_{22}O_4$; [84-74-2]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION:

Quantitative solubility data for the 1,2-benzenedicarboxylic acid dibutyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the 1,2-Benzenedicarboxylic acid dibutyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Krupatkin and Glagoleva (ref 1) | 298 | mutual | titration |
| Schwarz (ref 2) | 297[a] | (1) in (2) | chromatographic |
| Leyder and Boulanger (ref 3) | 293 | (1) in (2) | GLC |

[a] 23.5 °C.

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

## 1. SOLUBILITY OF 1,2-BENZENEDICARBOXYLIC ACID DIBUTYL ESTER (1) IN WATER (2)

All the available data for the solubility of 1,2-benzenedicarboxylic acid dibutyl ester (1) in water (2) are summarized in Table 2. The value of Krupatkin and Glagoleva (ref 1) is much higher than the other studies and is rejected. Pending further independent studies the remaining data (ref 2,3) must be regarded as very Tentative.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) 1,2-Benzenedicarboxylic acid dibutyl ester<br>(*dibutyl phthalate*); $C_{16}H_{22}O_4$; [84-74-2]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

TABLE 2: Reported Solubilities of 1,2-Benzenedicarboxylic acid dibutyl ester (1) in Water (2)

| $T$/K | Reported Solubilities | |
|---|---|---|
| | $10^3$ g(1)/100g sln | $10^7 x_1$ |
| 293 | 1.01 (ref 3) | 6 |
| 297[a] | 1.83 (ref 2) | 12 |
| 298 | 40 (ref 1)[b] | 260[b] |

[a] 23.5 °C.

[b] Rejected value.

## 2. SOLUBILITY OF WATER (2) IN 1,2-BENZENEDICARBOXYLIC ACID DIBUTYL ESTER (1)

As the only available datum for the solubility of water (2) in 1,2-benzenedicarboxylic acid dibutyl ester (1) is that of Krupatkin and Glagoleva (ref 1) no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheet for the experimental solubility but it may be noted that solubilities reported by these authors often differ from reliable values.

REFERENCES

1. Krupatkin, I. L.; Glagoleva, M. F. *Zh. Prikl. Khim.* 1972, *45*, 1317-20.
2. Schwarz, F. P. *Anal. Chem.* 1980, *52*, 10-15.
3. Leyder, F.; Boulanger, P. *Bull. Environ. Contam. Toxicol.* 1983, *30*, 152-7.

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid dibutyl ester (*dibutyl phthalate*); $C_{16}H_{22}O_4$; [84-74-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Krupatkin, I.L.; Glagoleva, M.F.

*Zh. Prikl. Khim.* 1972, *45*, 1317-20.

VARIABLES:

$T$/K = 298

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid dibutyl ester in water at 25°C was reported to be 0.04 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $3 \times 10^{-5}$.

The solubility of water in 1,2-benzenedicarboxylic acid dibutyl ester at 25°C was reported to be 0.62 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.088.

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 1,2-benzenedicarboxylic acid dibutyl ester-water-2-furancarbonal (*dibutyl phthalate-water-furfural*). No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; b.p. 340°C, $d_4^{25}$ 1.0485, $n_D^{25}$ 1.4882.

(2) Twice distilled.

ESTIMATED ERROR:

Temp. ±0.1°C.

REFERENCES:

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid dibutyl ester (*dibutyl phthalate*); $C_{16}H_{22}O_4$; [84-74-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Schwarz, F.P.

*Anal. Chem.* 1980, *52*, 10-5.

VARIABLES:

$T$/K = 297

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid dibutyl ester in water at 23.5°C was reported to be 0.00183 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $1.18 \times 10^{-6}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The elution chromatographic method was used. The column was filled with Chromosorb P (80-120 mesh, 4-6 $m^2/g$ by BET) coated with the solute and prepared in a special way as described in the paper. The water passing through the solute column was collected in graduated vessels. The measurements were initiated after the appearance of the solute depleted light pink colored zone (solvent zone) on Chromosorb P. Each measurement consisted of turning off the flow, measuring the length of the zone and the total volume of the water passed through the column. Measurements were initiated after the solvent zone extended 0.3 cm down the length of the column and were continued until the solvent zone extended down at least 25% of the column length.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified, reagent grade; used as received; $d^{20}$ 1.047.

(2) Distilled.

ESTIMATED ERROR:

Temp. ±1.5°C.
Soly. ±0.00006 g(1)/100g sln (standard deviation).

REFERENCES:

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid dibutyl ester (*dibutyl phthalate*); $C_{16}H_{22}O_4$; [84-74-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Leyder, F.; Boulanger, P.

*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7.

VARIABLES:

*T*/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid dibutyl ester in water at 20°C was reported to be 3.63 x $10^{-5}$ mol(1)/L and 0.0101 g(1)/L.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent flush" and "hot needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) Fluka; purity >98%; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

| | |
|---|---|
| COMPONENTS:<br>(1) Phosphoric acid butyl bis(methylphenyl) ester (*butyl ditolyl phosphate*); $C_{18}H_{23}O_4P$; [25657-10-7]<br>(2) Water; $H_2O$; [7732-18-5] | ORIGINAL MEASUREMENTS:<br>Apelblat, A.<br>*J. Chem. Soc.* B 1969, 175-7. |
| VARIABLES:<br>$T/K = 298$ | PREPARED BY:<br>A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of water in phosphoric acid butyl bis(methylphenyl) ester at 25°C was reported to be 0.69 mol(2)/L sln. The corresponding value on a mass/volume basis is 12.4 g(2)/L sln (compiler).

The density of the ester-phase was reported to be $d^{25}$ 1.1097.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The mixture was equilibrated by vigorous stirring for 30 min. and settling for 12 h. The organic phase was analyzed with the Karl Fischer reagent. Preliminary experiments showed that stirring and settling times were sufficient.

SOURCE AND PURITY OF MATERIALS:

(1) Synthesized at the Plastics Research Laboratory of the Weizmann Institute of Science; used as received; b.p. 180°C at 0.25 mm Hg, $d_4^{25}$ 1.1118, $n_D^{25}$ 1.5160.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.1°C.

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) 1,2-Benzenedicarboxylic acid dipentyl ester *(dipentyl phthalate)*; $C_{18}H_{26}O_4$; [131-18-0]<br>(2) Water; $H_2O$; [7732-18-5] | Leyder, F.; Boulanger, P.<br>*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7. |
| **VARIABLES:** | **PREPARED BY:** |
| $T$/K = 293 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid dipentyl ester in water at 20°C was reported to be 0.3 x $10^{-6}$ [a], 2.6 x $10^{-6}$ [b] mol(1)/L and 0.0001[a], 0.0008[b] g(1)/L.

[a] Gas chromatography analysis.

[b] UV spectrophotometry.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent flush" and "hot needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) Kodak, practical grade; purity >90% ; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

| | |
|---|---|
| COMPONENTS:<br>(1) Phosphoric acid tris(butoxyethyl) ester (*tri(butoxyethyl) phosphate*); $C_{18}H_{39}O_7P$; [78-51-3]<br>(2) Water; $H_2O$; [7732-18-5] | ORIGINAL MEASUREMENTS:<br>Apelblat, A.<br>*J. Chem. Soc.* B 1969, 175-7. |
| VARIABLES:<br>$T$/K = 298 | PREPARED BY:<br>A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of water in phosphoric acid tris(butoxyethyl) ester at 25°C was reported to be 4.26 mol(2)/L sln. The corresponding value on a mass/volume basis is 76.7 g(2)/L sln (compiler).

The density of the ester-phase was reported to be $d^{25}$ 1.0204.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The mixture was equilibrated by vigorous stirring for 30 min. and settling for 12 h. The organic phase was analyzed with the Karl Fischer reagent. Preliminary experiments showed that stirring and settling times were sufficient.

SOURCE AND PURITY OF MATERIALS:

(1) Albright and Wilson Co; purified by method described by Alcock, Grimley, Healy, Kennedy and McKay (ref 1); $d_4^{25}$ 1.0181, $n_D^{25}$ 1.4358.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.1°C.

REFERENCES:

1. Alcock, K.; Grimley, S.S.; Healy, T.V.; Kennedy, J.; McKay, H.A.C. *Trans. Faraday Soc.* 1956, *52*, 39.

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) 1,2-Benzenedicarboxylic acid butyl phenylmethyl ester (*butyl benzyl phthalate*); $C_{19}H_{20}O_4$; [85-68-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION:

Quantitative solubility data for the 1,2-benzenedicarboxylic acid butyl phenylmethyl ester (1) - water (2) system have been reported in the publications listed in Table 1.

TABLE 1: Quantitative Solubility Studies of the 1,2-Benzenedicarboxylic acid butyl phenylmethyl ester (1) - Water (2) System

| Reference | *T*/K | Solubility | Method |
|---|---|---|---|
| Krupatkin and Glagoleva (ref 1) | 298 | mutual | titration |
| Leyder and Boulanger (ref 2) | 293 | (1) in (2) | GLC |

The original data in these publications are compiled in the Data Sheets immediately following this Critical Evaluation. For convenience, further discussion of this system will be divided into two parts.

## 1. SOLUBILITY OF 1,2-BENZENEDICARBOXYLIC ACID BUTYL PHENYLMETHYL ESTER (1) IN WATER (2)

All the available data for the solubility of 1,2-benzenedicarboxylic acid butyl phenylmethyl ester (1) in water (2) are summarized in Table 2. The data are in serious disagreement differing by three orders of magnitude! The more recent value of Leyder and Boulanger (ref 2) using a more appropriate technique is probably more reasonable but, in the absence of further studies, it is not possible to reject the datum of Krupatkin and Glagoleva (ref 1).

TABLE 2: Reported Solubilities of 1,2-Benzenedicarboxylic acid butyl phenylmethyl ester (1) in Water (2)

| *T*/K | Reported Solubilities | |
|---|---|---|
| | $10^4$ g(1)/100g sln | $10^7x_1$ |
| 293 | $2.82^a$ (ref 2) | $1.63^a$ |
| 298 | $2100^b$ (ref 1) | $1200^b$ |

[a] Preferred value, see text.

[b] Unlikely value, see text.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) 1,2-Benzenedicarboxylic acid butyl phenylmethyl ester (*butyl benzyl phthalate*); $C_{19}H_{20}O_4$; [85-68-7]<br>(2) Water; $H_2O$; [7732-18-5] | G.T. Hefter, School of Mathematical and Physical Sciences,<br>Murdoch University, Perth, W.A., Australia<br>December, 1988 |

CRITICAL EVALUATION: (continued)

## 2. SOLUBILITY OF WATER (2) IN 1,2-BENZENEDICARBOXYLIC ACID BUTYL PHENYLMETHYL ESTER (1)

The only available datum for the solubility of water (2) in 1,2-benzenedicarboxylic acid butyl phenylmethyl ester (1) is that of Krupatkin and Glagoleva (ref 1) and thus no Critical Evaluation is possible. The interested user is referred to the relevant Data Sheet for the experimental solubility but it should be noted that solubilities reported by these authors often differ from reliable values in well characterized systems.

REFERENCES

1. Krupatkin, I. L.; Glagoleva, M. F. *Zh. Prikl. Khim.* 1972, *45*, 1795-9.

2. Leyder, F.; Boulanger, P. *Bull. Environ. Contam. Toxicol.* 1983, *30*, 152-7.

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid butyl phenylmethyl ester (*butyl benzyl phthalate*); $C_{19}H_{20}O_4$; [85-68-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Krupatkin, I.L.; Glagoleva, M.F.
*Zh. Prikl. Khim.* 1972, *45*, 1795-9.

VARIABLES:

$T/K = 298$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid butyl phenylmethyl ester in water at 25°C was reported to be 0.21 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $1.21 \times 10^{-4}$.

The solubility of water in 1,2-benzenedicarboxylic acid butyl phenylmethyl ester at 25°C was reported to be 0.07 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.012.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used as given by Krupatkin and Glagoleva (ref 1). The samples were titrated up to turbidity. The data were reported together with the ternary system 1,2-benzenedicarboxylic acid butyl phenylmethyl ester-water-2-furancarbonal (*butyl benzyl phthalate-water-furfural*). No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; b.p.236°C, $d_{25}^{25}$ 1.1065, $n_D^{25}$ 1.5180.

(2) Twice distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. Krupatkin, I.L.; Glagoleva, M.F. *Zh. Prikl. Khim.* 1969, *42*, 880.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) 1,2-Benzenedicarboxylic acid butyl phenylmethyl ester (*butyl benzyl phthalate*); $C_{19}H_{20}O_4$; [85-68-7]<br>(2) Water; $H_2O$; [7732-18-5] | Leyder, F.; Boulanger, P.<br>*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7. |
| **VARIABLES:** | **PREPARED BY:** |
| $T/K = 293$ | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid butyl phenylmethyl ester in water at 20°C was reported to be $9.02 \times 10^{-6}$ mol(1)/L and 0.00282 g(1)/L.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent flush" and "hot needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) Bayer Unimoll BB; purity not specified; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid butyl octyl ester (*butyl octyl phthalate*); $C_{20}H_{30}O_4$; [84-78-6]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Krupatkin, I.L.; Glagoleva, M.F.
*Zh. Prikl. Khim.* 1972, *45*, 1795-9.

VARIABLES:

$T/K = 298$

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid butyl octyl ester in water at 25°C was reported to be 0.04 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $2.2 \times 10^{-5}$.

The solubility of water in 1,2-benzenedicarboxylic acid butyl octyl ester at 25°C was reported to be 0.02 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.0037.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The titration method was used as given by Krupatkin and Glagoleva (ref 1). The samples were titrated up to turbidity. The data were reported together with the ternary system 1,2-benzenedicarboxylic acid butyl octyl ester-water-2-furancarbonal (*butyl octyl phthalate-water-furfural*). No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; $d_{25}^{25}$ 1.0097, $n_D^{25}$ 1.4868.

(2) Twice distilled.

ESTIMATED ERROR:

Not specified.

REFERENCES:

1. Krupatkin, I.L.; Glagoleva, M.F. *Zh. Prikl. Khim.* 1969, *42*, 880.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) L-Ascorbic acid 6-hexadecanoate (*L-ascorbyl palmitate*); $C_{22}H_{38}O_7$; [137-66-6]<br>(2) Water; $H_2O$; [7732-18-5] | Swern, D.<br>*J. Am. Chem. Soc.* 1949, *71*, 3256. |
| **VARIABLES:** | **PREPARED BY:** |
| $T$/K = 298 | A. Skrzecz |

EXPERIMENTAL VALUES:

The solubility of L-ascorbic acid 6-hexadecanoate in water at 25°C was reported to be 0.56 g(1)/100g[a,b] and 0.31 g(1)/100g[a,c]. The corresponding mass per cent and mole fractions, $x_1$, values calculated by the compiler are 0.56 g(1)/100g sln, 0.31 g(1)/100g sln and $2.4 \times 10^{-4}$, $1.4 \times 10^{-4}$.

The units g(1)/100g were reported as above.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical methods were used. An excess of ester was shaken with water until an equilibrium was obtained. The dissolved ester was determined both by titration with 0.1 N NaOH and by evaporation of water. At least two determinations were made.

SOURCE AND PURITY OF MATERIALS:

(1) Not specified.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.10°C.
Soly. ±0.5% (precision of duplicates).

REFERENCES:

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid bis(2-ethylhexyl) ester (*bis(2-ethylhexyl) phthalate*); $C_{24}H_{38}O_4$; [117-81-7]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Leyder, F.; Boulanger, P.

*Bull. Environ. Contam. Toxicol.* 1983, *30(2)*, 152-7.

VARIABLES:

$T$/K = 293

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid bis(2-ethylhexyl) ester in water at 20°C was reported to be 1.05 x $10^{-7}$ mol(1)/L and 4.1 x $10^{-5}$ g(1)/L.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method as described in the *OECD Guidelines for Testing of Chemicals* (ref 1) was used. Gas chromatography analyses were conducted on a Girdel 3000 FFLE apparatus with flame-ionization detection. A 4 mm x 180 m stainless steel column packed with 3% OV-1 on 80-100 mesh Chromosorb W-AW-DMCS and helium at a 20mL/min flow rate were used. Samples were fortified with an internal standard, extracted, and concentrated to 1 mL of hexane solution. "Solvent flush" and "hot needle" injection techniques were used. The two methods gave similar results. Peak heights were measured throughout. UV measurements were made by using 1-cm cells in a Perkin-Elmer model 552 uv-Visible spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

(1) Essochem DOP; purity not specified; used as received.

(2) Deionized and distilled from $KMnO_4$.

ESTIMATED ERROR:

Temp. ±1°C.

REFERENCES:

1. *OECD Guidelines for Testing of Chemicals*, Paris, OECD, 1981. (methods 101, 105).

COMPONENTS:

(1) Phosphoric acid tris(2-ethylhexyl) ester (*tri(2-ethylhexyl) phosphate*); $C_{24}H_{51}O_4P$; [78-42-2]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Apelblat, A.

*J. Chem. Soc.* B 1969, 175-7.

VARIABLES:

$T$/K = 298

PREPARED BY:

A. Skrzecz

EXPERIMENTAL VALUES:

The solubility of water in phosphoric acid tris(2-ethylhexyl) ester at 25°C was reported to be 0.81 mol(2)/L sln. The corresponding value on a mass/volume basis is 14.6 g(2)/L sln (compiler).

The density of the ester-phase was reported to be $d^{25}$ 0.9235.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. The mixture was equilibrated by vigorous stirring for 30 min. and settling for 12 h. The organic phase was analyzed with the Karl Fischer reagent. Preliminary experiments showed that stirring and settling times were sufficient.

SOURCE AND PURITY OF MATERIALS:

(1) Albright and Wilson Co; purified by method described by Alcock, Grimley, Healy, Kennedy and McKay (ref 1); $d_4^{25}$ 0.9220, $n_D^{25}$ 1.4417.

(2) Not specified.

ESTIMATED ERROR:

Temp. ±0.1°C.

REFERENCES:

1. Alcock, K.; Grimley, S.S.; Healy, T.V.; Kennedy, J.; McKay, H.A.C. *Trans. Faraday Soc.* 1956, *52*, 39.

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid dinonyl ester
(*dinonyl phthalate*);
$C_{26}H_{42}O_4$; [84-76-4]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Krupatkin, I.L.; Glagoleva, M.F.

*Zh. Prikl. Khim.* 1972, *45*, 1317-20.

VARIABLES:

*T*/K = 298

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid dinonyl ester in water at 25°C was reported to be 0.02 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $9 \times 10^{-6}$.

The solubility of water in 1,2-benzenedicarboxylic acid dinonyl ester at 25°C was reported to be 0.09 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.02.

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 1,2-benzenedicarboxylic acid dinonyl ester-water-2-furancarbonal (*dinonyl phthalate-water-furfural*). No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified;
b.p. 240°C, $d_4^{25}$ 0.9819,
$n_D^{25}$ 1.4852.

(2) Twice distilled.

ESTIMATED ERROR:

Temp. ±0.1°C.

REFERENCES:

COMPONENTS:

(1) Benzoic acid, 2-hydroxy-, cinchonan-9-ol, 6'-methoxy,(8*α*,9*R*) ester (*quinine salicylate*); $C_{27}H_{30}N_2O_5$; [750-90-3]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Seidell, A.

*Hygienic Lab. Bull.* 1910, No. 67, 98 pp (US Govt. Printing Office, Washington, DC).

VARIABLES:

$T$/K = 298

PREPARED BY:

G.T. Hefter

EXPERIMENTAL VALUES:

The solubility of 2-hydroxybenzoic acid 6'-methoxycinchonan-9-ol (8*α*,9*R*) ester in water at 25°C was reported to be 0.065 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is 2.5 x $10^{-5}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The analytical method was used. Appropriate quantities of (1) and (2) were shaken together. The amount of dissolved (1) was determined by evaporation of a known quantity of the saturated solution. The residue was dried to a constant weight in a vacuum desiccator.

SOURCE AND PURITY OF MATERIALS:

(1) Merck, m.p. ca. 195°C.

(2) Distilled (no details given).

ESTIMATED ERROR:

Temp. not stated.
Soly. not stated.

REFERENCES:

COMPONENTS:

(1) 1,2-Benzenedicarboxylic acid didodecyl ester (*didodecyl phthalate*); $C_{32}H_{54}O_4$; [2432-90-8]

(2) Water; $H_2O$; [7732-18-5]

ORIGINAL MEASUREMENTS:

Krupatkin, I.L.; Glagoleva, M.F.

*Zh. Prikl. Khim.* 1972, *45*, 1317-20.

VARIABLES:

$T/K = 298$

PREPARED BY:

Z. Maczynska

EXPERIMENTAL VALUES:

The solubility of 1,2-benzenedicarboxylic acid didodecyl ester in water at 25°C was reported to be 0.07 g(1)/100g sln. The corresponding mole fraction, $x_1$, value calculated by the compiler is $3 \times 10^{-5}$.

The solubility of water in 1,2-benzenedicarboxylic acid didodecyl ester at 25°C was reported to be 0.02 g(2)/100g sln. The corresponding mole fraction, $x_2$, value calculated by the compiler is 0.006.

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 1,2-benzenedicarboxylic acid didodecyl ester-water-2-furancarbonal (*didodecyl phthalate-water-furfural*). No further details were reported in the paper.

SOURCE AND PURITY OF MATERIALS:

(1) Source not specified; $d_4^{25}$ 0.9548, $n_D^{25}$ 1.4839.

(2) Twice distilled.

ESTIMATED ERROR:

Temp. ±0.1°C.

REFERENCES:

# SYSTEM INDEX

Pages preceded by E refer to evaluation texts whereas those not preceded by E refer to compiled tables.

# REGISTRY NUMBER INDEX

Pages preceded by E refer to evaluation texts whereas those not preceded by E refer to compiled tables.

# AUTHOR INDEX

Pages preceded by E refer to evaluation texts whereas those not preceded by E refer to compiled tables.

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| | |
|---|---|
| Volume 1 | H. L. Clever, *Helium and Neon* |
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| Volume 25 | C. Hirayama, Z. Galus and C. Guminski, *Metals in Mercury* |
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| Volume 27/28 | H. L. Clever and C. L. Young, *Methane* |
| Volume 29 | H. L. Clever, *Mercury in Liquids, Compressed Gases, Molten Salts and Other Elements* |
| Volume 30 | H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid* |
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| | |
|---|---|
| Volume 32 | P. G. T. Fogg and C. L. Young, *Hydrogen Sulfide, Deuterium Sulfide and Hydrogen Selenide* |
| Volume 33 | P. Franzosini, *Molten Alkali Metal Alkanoates* |
| Volume 34 | A. N. Paruta and R. Piekos, *4-Aminobenzenesulfonamides. Part I: Non-cyclic Substituents* |
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| Volume 38 | D. G. Shaw, *Hydrocarbons with Water and Seawater. Part II: Hydrocarbons* $C_8$ *to* $C_{36}$ |
| Volume 39 | C. L. Young, *Cumulative Index: Volumes 20-38* |
| Volume 40 | J. Hala, *Halides, Oxyhalides and Salts of Halogen Complexes of Titanium, Zirconium, Hafnium, Vanadium, Niobium and Tantalum* |
| Volume 41 | C.-Y. Chan, I. N. Lepeshkov and K. H. Khoo, *Alkaline Earth Metal Perchlorates* |
| Volume 42 | P. G. T. Fogg and W. Gerrard, *Hydrogen Halides in Non-aqueous Solvents* |
| Volume 43 | R. W. Cargill, *Carbon Monoxide* |
| Volume 44 | H. Miyamoto, E. M. Woolley and M. Salomon, *Copper and Silver Halates* |
| Volume 45/46 | R. P. T. Tomkins and N. P. Bansal, *Gases in Molten Salts* |
| Volume 47 | R. Cohen-Adad and J. W. Lorimer, *Alkali Metal and Ammonium Chlorides in Water and Heavy Water (Binary Systems)* |
| Volume 48 | F. Getzen, G. Hefter and A. Maczynski, *Esters with Water. Part I: Esters 2-C to 6-C* |
| Volume 49 | F. Getzen, G. Hefter and A. Maczynski, *Esters with Water. Part II: Esters 7-C to 32-C* |