| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Fluorene; $C_{13}H_{10}$; [86-73-7] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia. |
| (2) Water; $H_2O$; [7732-18-5] | April 1986. |

CRITICAL EVALUATION:

Quantitative solubility data for fluorene (1) in water (2) have been reported in the publications listed in Table 1. No solubility data for water in fluorene have been reported.

TABLE 1: Quantitative Solubility Studies of Fluorene (1) in Water (2)

| Reference | $T$/K | Method |
|---|---|---|
| Wauchope and Getzen (ref 1) | 273-348 | spectrophotometric |
| Mackay and Shiu (ref 2) | 298 | spectrofluorometric |
| May *et al.* (ref 3) | 298 | chromatographic |

The original data in all of these publications are compiled in the Data Sheets immediately following this Critical Evaluation. The solubility values are also summarized in Table 2 and plotted in Figure 1.

At 298K, the only temperature where comparison is possible, the available data (ref 1-3) are in good agreement and the average value can be Recommended. At other temperatures only the values of Wauchope and Getzen (ref 1) are available and are therefore regarded as tentative.

TABLE 2: Recommended ($R$) and Tentative Solubility Values of Fluorene (1) in Water (2)

| $T$/K | Solubility values | | |
|---|---|---|---|
| | Reported values[a] $10^4 g(1)/100g$ sln | "Best" values ($\pm \sigma_n$)[b] $10^4 g(1)/100g$ sln | $10^6 x_1$ |
| 273 | 0.66 (ref 1) | 0.7 | 0.8 |
| 298 | 1.90 (ref 1), 1.98 (ref 2), 1.685 (ref 3) | 1.9 ± 0.1 ($R$) | 2.1 ($R$) |
| 303 | 2.4* (ref 1) | 2.4 | 2.6 |
| 313 | 3.8* (ref 1) | 3.8 | 4.1 |
| 323 | 6.29 (ref 1) | 6.3 | 6.8 |
| 333 | 10.4* (ref 1) | 10 | 11 |
| 343 | 18.5* (ref 1) | 19 | 21 |

[a] Values marked with an asterisk (*) were obtained by the Evaluator by graphical interpolation of the authors' original values.
[b] Obtained by averaging where appropriate; $\sigma_n$ has no statistical significance.

(continued next page)

| COMPONENTS: | EVALUATOR: |
|---|---|
| (1) Fluorene; $C_{13}H_{10}$; [86-73-7] | G.T. Hefter, School of Mathematical and Physical Sciences, Murdoch University, Perth, W.A., Australia. |
| (2) Water; $H_2O$; [7732-18-5] | April 1986. |

CRITICAL EVALUATION: (continued)

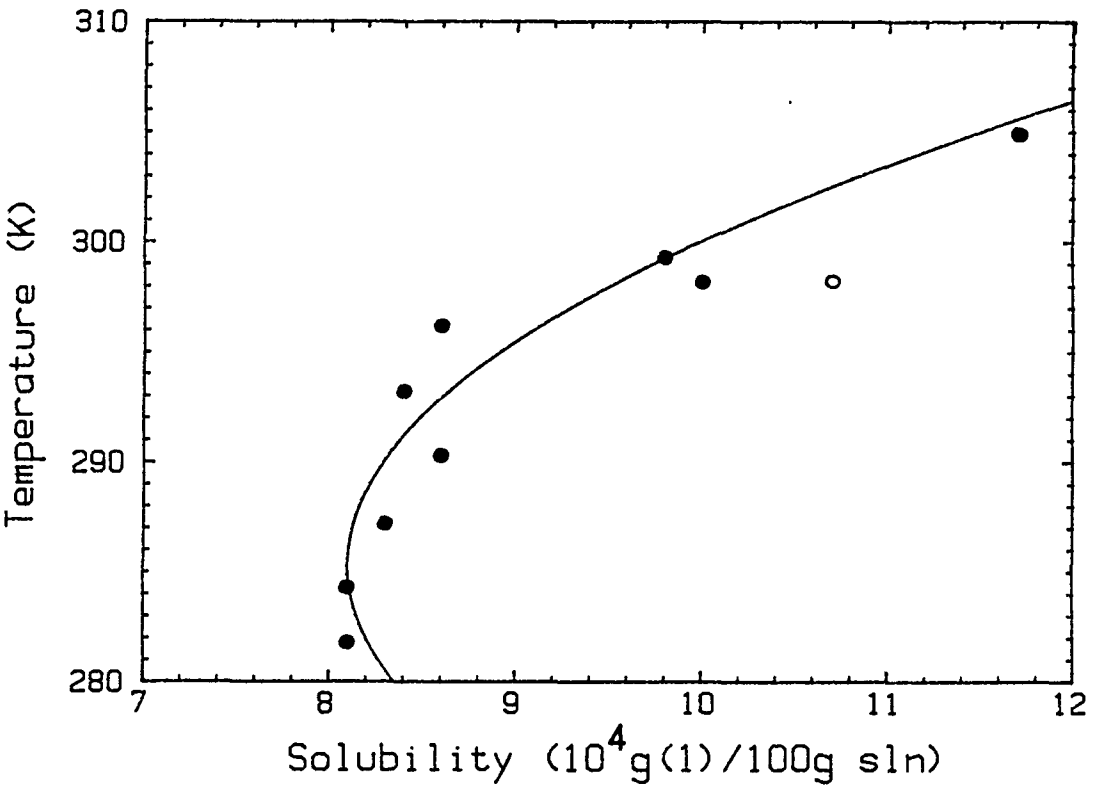


FIGURE 1. Solubility of fluorene in water: ref 1 (o); ref 2,3 (●).

REFERENCES

1. Wauchope, R.D.; Getzen, F.W. *J. Chem. Eng. Data* 1972, *17*, 38-41.

2. Mackay, D.; Shiu, W.Y. *J. Chem. Eng. Data* 1977, *22*, 399-402.

3. May, W.E.; Wasik, S.P.; Freeman, D.H. *Anal. Chem.* 1978, *50*, 997-1000.

ACKNOWLEDGEMENT

The Evaluator thanks Dr Brian Clare for the graphics.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Fluorene; $C_{13}H_{10}$; [86-73-7] | Wauchope, R.D.; Getzen, F.W. |
| (2) Water; $H_2O$; [7732-18-5] | *J. Chem. Eng. Data* 1972, *17*, 38-41. |
| **VARIABLES:** | **PREPARED BY:** |
| Temperature: 0-75°C | A. Maczynski |

**EXPERIMENTAL VALUES:**

Solubility of fluorene in water

| $t$/°C | mg(1)/kg(2) experiment | mg(1)/kg(2) smoothed with (std dev) | $10^4$ g(1)/100 g sln (compiler) | $10^7 x_1$ (compiler) |
|---|---|---|---|---|
| 0.0 | | 0.66(0.01) | 0.66 | 7.2 |
| 24.6 | 1.93, 1.87, 1.88 | 1.86 | 1.86 | 20.2 |
| 25.0 | | 1.90(0.03) | 1.90 | 20.6 |
| 29.9 | 2.41, 2.33, 2.34 | 2.37 | 2.37 | 25.7 |
| 30.3 | 2.10, 2.25, 2.23 | 2.41 | 2.41 | 26.1 |
| 38.4 | 3.72, 3.73 | 3.53 | 3.53 | 38.2 |
| 40.1 | 3.88, 3.84, 3.85 | 3.84 | 3.84 | 41.6 |
| 47.5 | 5.59, 5.62, 5.68 | 5.54 | 5.54 | 60.0 |
| 50.0 | | 6.29(0.05) | 6.29 | 68.2 |
| 50.1 | 6.31, 6.42, 6.54 | 6.32 | 6.32 | 68.5 |
| 50.2 | 6.27 | 6.35 | 6.35 | 68.8 |
| 54.7 | 8.31, 8.41, 8.56 | 8.02 | 8.02 | 86.9 |
| 59.2 | 10.5, 10.5 | 10.2 | 10.2 | 110 |
| 60.5 | 10.7, 11.0, 11.6 | 10.9 | 10.9 | 118 |
| 65.1 | 14.2, 14.1, 14.2 | 14.1 | 14.1 | 153 |
| 70.7 | 18.5, 18.5, 18.9 | 19.3 | 19.3 | 209 |
| 71.9 | 18.8 | 20.6 | 20.6 | 223 |
| 73.4 | 21.5 | 22.5 | 22.5 | 244 |
| 75.0 | | 24.7(0.4) | 24.7 | 268 |

AUXILIARY INFORMATION

**METHOD/APPARATUS/PROCEDURE:**

Approximately 20 g of (1) was placed in each of three 250-mL glass-stoppered flasks with (2). The flasks were suspended in an open water bath and shaken gently from one to three weeks between measurements. Samples of the replicate were extracted with cyclohexane. In all cases, spectra taken of second extracts or of the aqueous layer after extraction indicated complete extraction.
Standard solutions were prepared either by direct weighing using a Cahn electrobalance, or by weighing 0.1-0.2 g of samples followed by serial dilution in calibrated glassware.

**SOURCE AND PURITY OF MATERIALS:**

(1) Baker reagent; recrystallized three times from ether; vacuum-sublimed twice; purity not specified.

(2) distilled and deionized.

**ESTIMATED ERROR:**

temp. ± 0.5°C
soly. see experimental values above

**REFERENCES:**

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Fluorene; $C_{13}H_{10}$; [86-73-7] | Mackay, D.; Shiu, W.Y. |
| (2) Water; $H_2O$; [7732-18-5] | *J. Chem. Eng. Data* 1977, *22*, 399-402. |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 25°C | M.C. Haulait-Pirson |

**EXPERIMENTAL VALUES:**

The solubility of fluorene in water at 25°C was reported to be 1.98 mg(1) $dm^{-3}$ sln and $x_1 = 2.14 \times 10^{-7}$.

The corresponding mass percent calculated by the compiler is $1.98 \times 10^{-4}$ g(1)/100 g sln.

AUXILIARY INFORMATION

**METHOD/APPARATUS/PROCEDURE:**

A saturated solution of (1) in (2) was vigorously stirred in a 250 mL flask for 24 hrs. and subsequently settled at 25°C for at least 48 hrs. Then the saturated solution was decanted and filtered and 50-100 mL extracted with approximately 5 mL of cyclohexane in a separatory funnel. After shaking for 2 hrs. the cyclohexane extract was removed for analysis. An Aminco-Browman spectrophotofluorometer (American Instruments Ltd.) was used for analysis. Many details are given in the paper.

**SOURCE AND PURITY OF MATERIALS:**

(1) Aldrich Chemicals, Eastman Kodak, or K and K Laboratories, commercial highest grade; used as received.

(2) doubly distilled.

**ESTIMATED ERROR:**

soly. ± 0.04 mg(1) $dm^{-3}$ sln (maximum deviation from several determinations.)

**REFERENCES:**

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Fluorene; $C_{13}H_{10}$; [86-73-7]<br>(2) Water; $H_2O$; [7732-18-5] | May, W.E.; Wasik, S.P.; Freeman, D.H.<br>*Anal. Chem.* 1978, *50*, 997-1000. |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 25°C | A. Maczynski |

**EXPERIMENTAL VALUES:**

The solubility of fluorene in water at 25°C was reported to be 1.685 mg(1)/kg(2).

The corresponding mass percent and mole fraction, $x_1$, values calculated by compiler are $1.685 \times 10^{-4}$ g(1)/100 g sln and $1.826 \times 10^{-7}$.

AUXILIARY INFORMATION

**METHOD/APPARATUS/PROCEDURE:**

The dynamic coupled column liquid chromatography (DCCLC) method was based on generating saturated solutions by pumping water through a column packed with glass beads that have been coated with the component (1) (generator column). The concentration of (1) in the effluent of the generator column was measured by a modification of the coupled column liquid chromatographic process that has been described in ref 1.

**SOURCE AND PURITY OF MATERIALS:**

(1) commercial product; less than 3% impurities.

(2) distilled over $KMnO_4$ and NaOH and passed through a column packed with XAD-2 (Rohm and Hass, Philadelphia, Pa).

**ESTIMATED ERROR:**

temp. ± 0.05°C
soly. ± 0.005 mg(1)/100 kg(2) (standard deviation)

**REFERENCES:**

1. May, W.; Chesler, S.; Cram, S.; Gump, B.; Hertz, H.; Enagonio, D.; Dyszel, S. *J. Chromatogr. Sci.* 1975, *13*, 535.

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Fluorene; $C_{13}H_{10}$; [86-73-7]<br>(2) Sodium Chloride; NaCl; [7647-14-5]<br>(3) Water; $H_2O$; [7732-18-5] | May, W.E.; Wasik, S.P.; Freeman, D.H.<br>*Anal. Chem.* 1978, *50*, 997-1000. |
| **VARIABLES:** | **PREPARED BY:** |
| One temperature: 25°C<br>Salinity: 0-40 g(2)/kg sln | W.Y. Shiu and D. Mackay |

**EXPERIMENTAL VALUES:**

The solubility of fluorene in aqueous sodium chloride is reported in terms of the Setschenow equation:

$$\log(S_o/S) = K_s C_s$$

where;

$S_o$ is the solubility of (1) in water (mg/L)

S is the solubility of (1) in saline solution (mg/L)

$K_s$ is the Setschenow constant (L/mol)

$C_s$ in the concentration of sodium chloride (mol/L)

evaluating the equation for S over the range of $C_s$ 0-0.7 mol/L, $K_s$ = 0.267 with $S_o$ = 1.685.

The corresponding mass percent and mole fraction $x_1$, at salinity = 35 g(2)/kg sln calculated by the compilers are $1.20 \times 10^{-4}$ g(1)/100 g sln and $1.30 \times 10^{-7}$.

**AUXILIARY INFORMATION**

**METHOD/APPARATUS/PROCEDURE:**

A saturated solution of (1) was prepared by pumping salt water through a "generation column" which was packed with glass beads coated with 1% by weight of (1). The saturated solution was extracted with an "extractor column" packed with a superficially porous bonded $C_{18}$ stationary phase, then a water-acetonitrile solvent was passed through for extraction. The extract was introduced into a liquid chromatograph and the concentration of (1) was measured with a UV detector.

**SOURCE AND PURITY OF MATERIALS:**

(1) greater than 97% pure.

(2) reagent grade.

(3) distilled from potassium permanganate-sodium hydroxide and passed through an XAD-2 column.

**ESTIMATED ERROR:**

temp ± 0.05°C

**REFERENCES:**