tablished December 31, 1970, leads to

$$\Delta H_f^\circ(298.15\ \mathrm{K}) = -237700 \pm 420\ \mathrm{J\ mol^{-1}}$$

However, we can note that these two tables do not include the same recent data after 1969.

Conclusion

The values which we have selected do not disagree with any other measurements, and we may conclude that the best value for the standard enthalpy of formation of solid cobaltous oxide is

$$\Delta H_f^\circ(\mathrm{CoO,\ s,\ 298.15\ K}) = -237500 \pm 400\ \mathrm{J\ mol^{-1}}$$

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Solubility of Hydrogen in 9-Methylanthracene, Dibenzofuran, and 2-Ethylanthraquinone at High Temperatures and Pressures

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Solubility of hydrogen was measured in 9-methylanthracene and dibenzofuran at 100, 150, 200, and 250 °C and for pressures up to ca. 258 atm. For ethylanthraquinone (mp ca. 110 °C), the solubility was measured at 125, 150, and 200 °C and up to ca. 250 atm. The solubility data were used to calculate Henry's constants of hydrogen in the three aromatic compounds.

Introduction

Solubility data are needed for efficient design of pressure blow down vessels in a chemical process. In coal liquefaction processes, the hydrogenation reactor is followed by a high-pressure and then a low-pressure separator. Hydrogen solubility data in polyaromatic hydrocarbons is of particular interest in the designing of these separators. This work was initiated to measure the effect of structure on the solubility of hydrogen in three-ring aromatic compounds, one of the major constituents of coal liquefaction products.

Experimental Section

The apparatus that was used to measure the solubility of hydrogen in the three polyaromatic compounds was described in an earlier publication (*2*). The only change was the replacement of a thermocouple with a calibrated platinum resistance thermometer for the measurement of liquid bath temperatures.

All three polyaromatic compounds were purchased from Aldrich Chemical Co. These were further purified to at least 99.9% purity by zone purification technique. Hydrogen, with a purity of 99.995%, was purchased from Matheson Gas Co.

Results and Discussion

The solubility data for hydrogen in 9-methylanthracene, 2-ethylanthraquinone, and dibenzofuran are tabulated in Tables I-III. Figure 1 shows hydrogen solubility as a function of temperature and pressure for all of the compounds. 2-Ethylanthraquinone was found to be unstable above 200 °C. Since it showed significant hydrogenation at 250 °C, solubility mea-

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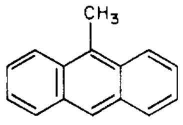
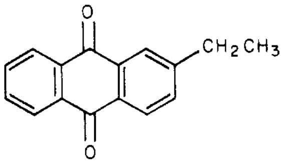
Table I. Solubility of H_2 in 9-Methylanthracene:



temp, °C	press., atm	x_{H_2}[a]	temp, °C	press., atm	x_{H_2}[a]
100.0	42.52	0.009844	200.0	50.07	0.02054
	67.62	0.01644		98.64	0.02814
	109.32	0.02671		144.90	0.0545
	164.42	0.03964		187.89	0.07016
	212.18	0.04952		252.72	0.09048
	257.14	0.05925	250.0	47.93	0.0230
150.0	50.27	0.0163		116.33	0.05766
	77.14	0.02373			
	132.38	0.0399			
	210.27	0.0618			
	257.69	0.07465			

[a] Mole fraction of H_2 in the liquid phase.

Table II. Solubility of Hydrogen in 2-Ethylanthraquinone:



temp, °C	press., atm	x_{H_2}[a]	temp, °C	press., atm	x_{H_2}[a]
125.0	49.49	0.0142	200.0	37.35	0.01478
	101.50	0.02972		73.74	0.02875
	150.61	0.04378		103.64	0.04222
	201.36	0.05853		150.27	0.05835
	250.68	0.07112		204.76	0.0776
150.0	33.57	0.0104		245.99	0.0908
	61.50	0.01822			
	100.10	0.03173			
	137.41	0.04347			
	168.64	0.05367			
	203.47	0.06393			
	247.69	0.07602			

[a] Mole fraction of H_2 in the liquid phase.

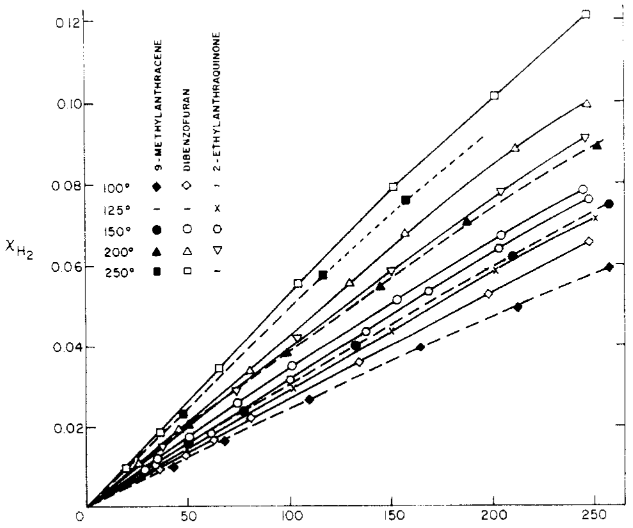
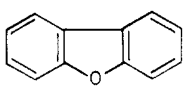


Figure 1. Solubility of hydrogen in 9-methylanthracene, dibenzofuran, and 2-ethylanthraquinone.

surements were discontinued at this temperature. NMR analysis of the hydrogenated compound showed almost complete hydrogenation of at least one of the double-bonded oxygens. The compound changed in color from light yellow to dark brown at the higher temperature. 9-Methylanthracene also showed some hydrogenation at 250 °C and pressures above ca. 100 atm. It also changed in color from light yellow to dark brown at 250 °C and pressures above ca. 100 atm. The melting point of the compound also decreased significantly. Therefore, in Figure 1, the solubility of H_2 in 9-methylanthracene at 250 °C is shown as a dotted line above ca. 100 atm. From the general trend at lower temperatures, it seems that at 250 °C the hydrogen solubility in 9-methylanthracene is apparently higher than expected. This should be expected if the compound has hydrogenated significantly (1).

Table III. Solubility of Hydrogen in Dibenzofuran:



temp, °C	press., atm	x_{H_2}[a]	temp, °C	press., atm	x_{H_2}[a]
100.0	35.97	0.00922	200.0	25.51	0.0108
	48.23	0.01280		45.5	0.01922
	62.24	0.01677		80.75	0.03382
	80.34	0.02222		129.66	0.05550
	133.95	0.03616		156.39	0.06786
	198.57	0.05294		211.7	0.0886
	247.82	0.06552		247.28	0.09906
150.0	28.91	0.00923	250.0	19.32	0.009546
	34.56	0.0119		36.05	0.01871
	50.54	0.01739		65.51	0.03456
	74.15	0.02582		104.29	0.05565
	101.22	0.03542		151.5	0.0792
	153.06	0.0515		201.19	0.1012
	204.76	0.06711		246.87	0.1214
	244.6	0.07838			

[a] Mole fraction of H_2 in the liquid phase.

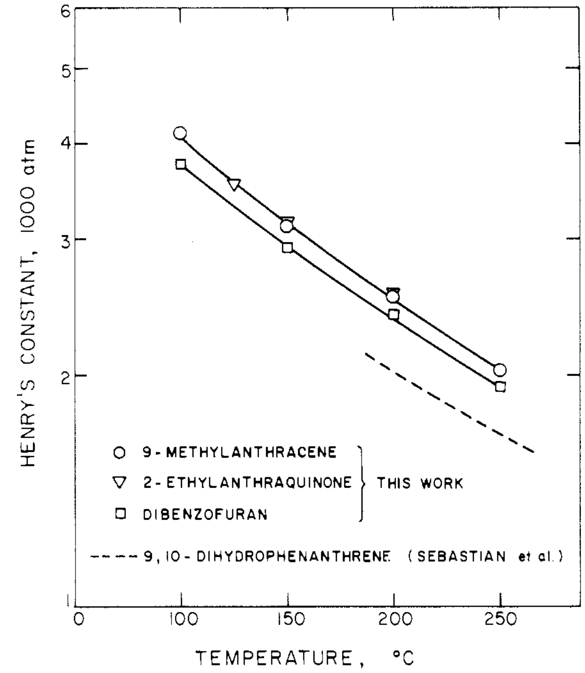


Figure 2. Henry's constants of hydrogen in 9-methylanthracene, 2-ethylanthraquinone, dibenzofuran, and 9,10-dihydrophenanthrene.

The solubility of hydrogen in dibenzofuran is moderately higher, at all temperatures, compared to the other two compounds. Dibenzofuran was found to be fairly stable even at 250 °C and up to 250 atm.

Henry's constant is defined as

$$H_{2,1} = \lim_{x_2 \to 0} (f_2/x_2)$$

Table IV. Henry's Constants of H_2 in 9-Methylanthracene, 2-Ethylanthraquinone, and Dibenzofuran

	Henry's constant, atm		
temp, °C	9-methylanthracene	2-ethylanthraquinone	dibenzofuran
100	4140		3750
125		3520	
150	3240	3175	2925
200	2525	2550	2395
250	2025		1920

where subscript 2 stands for the solute and 1 for the solvent; x_2 is the mole fraction of the solute in the solvent; f_2 is the fugacity of the solute in the gas phase. Henry's constants for hydrogen in the three aromatic compounds were evaluated by extrapolating a plot of f_{H_2}/x_{H_2} to $x_H = 0$. The fugacity of hydrogen in the gas phase, f_{H_2}, was calculated from the Soave–Redlich–Kwong equation of state (*4*). Figure 2 shows the plot of Henry's constants vs. temperature. The values of these constants are tabulated in Table IV. For comparison purposes, the Henry's constant of hydrogen in 9,10-dihydrophenanthrene, calculated from data of Sebastian et al. (*3*), are also shown in Figure 2.

Acknowledgment

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Density and Surface Tension of 83 Organic Liquids

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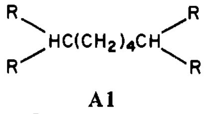
Densities and surface tensions of organic liquids of known purity were measured in the temperature range of 20–80 °C. In many cases literature data from apparently reliable sources conflicted with each other. Our measurements were conducted with the aim of making a choice. Also included are data of a few compounds of higher molecular weight measured in a broader temperature range.

In the present paper densities and surface tensions are given for 75 pure liquids and 8 purified polymers. The surface tensions of about half of the substances have been reported in the excellent critical review of Jasper (*1*), and his recommended values are compared with our data in Table II. In most cases the surface tensions agree within the experimental error, but, where significant deviations were found, the substances were reexamined with special care in order to obtain the most reliable values.

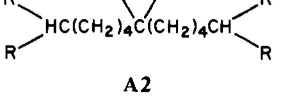
1. Compounds

In Table I information is given about origins, methods of purification, purities, refractive indexes, and melting points of the compounds examined. Compounds **16–24** are branched hydrocarbons of the general structures **A1** and **A2** with following formulas:



A1

A1-C_6, R = hexyl
A1-C_{10}, R = decyl
A1-C_{14}, R = tetradecyl
A1-C_{18}, R = octadecyl



A2

A2-C_{11}, R = hendecyl
A2-C_{13}, R = tridecyl
A2-C_{15}, R = pentadecyl
A2-C_{18}, R = octadecyl
A2-C_{22}, R = docosyl

Squalane (**15**) is a mixture of the diastereomers of 2,6,10,15,19,23-hexamethyltetracosane. Compounds **69–76** are poly(ethylene glycols) (PEGs) and their methoxylated derivatives (PEG-M) with nominal molecular masses of 600, 1000, 2000, and 20 000. The average molecular masses of the

$HO(CH_2CH_2O)_xH$ PEG

$H_3CO(CH_2CH_2O)_xCH_3$ PEG-M

PEGs determined by the method of ref *2* are as follows: PEG-600, 6.10 × 10^2; PEG-1000, 1.13 × 10^3; PEG-2000, 2.15 × 10^3; PEG-20000, 1.71 × 10^4. The derivatives PEG-M were prepared from the PEGs; consequently, their average molecular masses are MW(PEG-M) = MW(PEG) + 24. Under the synthetic conditions no degradation is to be expected.

1.1. ***Origin.*** In the third column of Table I, the origin of the compounds is indicated by using the following abbreviations: Fl, Fluka AG, Buchs, Switzerland; EGA, EGA-Chemie, Steinheim, Germany; Sh, Shell Research Laboratories, Amsterdam, Holland; Me, Merck AG, Darmstadt, Germany; Sy1, Synthetized in our laboratory, see ref *3*; Sy2, Synthetized in our laboratory, see ref *2*.

1.2. ***Purification.*** Commercially available research-grade compounds have been further purified. The method of purification is indicated in the fourth column of Table I by using the following abbreviations.

Di: Distillation of the product in a Vigreux column discarding a forerun and a residue, each representing 25% of the distilled material. This operation was capable of removing other members of homologous series to less than 0.2%.

Di/Na: Distilled over sodium.

Di/Hg: Distilled over mercury.

Cr: Recrystallization from diethyl ether (DE), propanol (Pr), cyclohexane (CH), ethanol (E), or *n*-hexane (H) or from a mixture of them. For example, the symbol "Di; 3×Cr(E/DE)" means that the middle fraction of the distillation was recrystallized three times from a mixture of ethanol and diethyl ether.

Fi: Filtration on a silica gel column (Merck: Kieselgel 60)

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