

DET KGL. DANSKE VIDENSKABERNES SELSKAB
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THE SOLUBILITY OF
CARBON MONOXIDE IN SOME LOWER
MONOVALENT ALCOHOLS

BY

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KØBENHAVN

I KOMMISSION HOS EJNAR MUNKSGAARD

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In an earlier paper (1), on the reaction velocity in the process between carbon monoxide and methyl- and ethyl alcoholate dissolved in the corresponding alcohols the solubility of carbon monoxide in these alcohols entered into the calculation of the velocity constant. A final calculation of the experiments was impossible owing to the lack of data on the solubility of carbon monoxide between 20° and 50°. The present paper deals with this solubility in methyl-, ethyl-, normal propyl- and isopropyl alcohol as well as in normal butyl- and isobutyl alcohol at the temperatures in question.

The solubility of carbon monoxide in methyl- and ethyl alcohol has previously been investigated by CARIUS (2), JUST (3) and SKIRROW (4), and a review of the results is given in Table 1. But the literature, as far as the author is aware, does not contain any data on the solubility of carbon monoxide in the propyl- and butyl alcohols. The solubilities recorded in Table 1 are expressed by means of Ostwald's absorption coefficient, i. e. the ratio between the concentration of the gas in the saturated solution and the concentration of the gas in the gas phase. CARIUS expresses the solubility of carbon monoxide in ethyl alcohol by means of Bunsen's absorption coefficient as 0.204 from 0° to 25°; this is in the table converted according to Ostwald's absorption coefficient.

Table 1.
Solubility (l = Ostwald's absorption coefficient) of carbon monoxide in alcohols.

	CH ₃ OH		C ₂ H ₅ OH	
	20°	25°	20°	25°
Carius, 1855.....			0.219	0.223
Just, 1901.....	0.1830	0.1955	0.1901	0.1921
Skirrow, 1902.....		0.196		0.192

Apparatus.

The solubility determinations described in the present paper were carried out in an apparatus which in principle is first described by ESTREICHER (5) and later improved by LANNUNG (6), whose paper on the subject contains a sketch and a detailed description. The particular advantage of the apparatus is that the experimental space is shut off by mercury, so that neither gas nor solvent during the experiment comes into contact with stopcocks and stopcock grease. During the experiment the apparatus was constantly shaken in an air thermostat the temperature of which between 20° and 50° could be kept constant with an accuracy of about 0.05° .

The procedure followed in the solubility determinations is described in detail by LANNUNG in the paper cited. In principle it is as follows: After the apparatus is completely filled with mercury a suitable volume of alcohol (about 20 ml) is sucked in and freed from air by keeping the apparatus evacuated for about 12 hours with repeated suction and shaking, whereupon the alcohol is confined between two mercury surfaces. Next about 6 ml of carbon monoxide is sucked into another part of the apparatus where it likewise is confined between mercury surfaces. The apparatus is now placed in the thermostat at 20° and the volume and pressure of the carbon monoxide read after temperature adjustment. Then the carbon monoxide is brought into contact with the alcohol by the separating string of mercury being allowed to drop down into the alcohol. The shaking is started and continued until the manometer reading shows no change for at least half an hour. Then the equilibrium adjustment is carried out at different temperatures. Finally the volume of liquid is determined by weighing the amount of mercury that can be drawn to a calibration mark. The volume of the manometer tube of the apparatus and the volume between the various marks are determined by weighing with mercury.

Materials.

Methyl-, ethyl-, and normal propyl alcohol were dehydrated in an apparatus made completely of glass, magnesium being used

according to H. LUND and J. BJERRUM (7). It was attempted to dehydrate isopropyl alcohol by the same method, but magnesium could not be made to react with this alcohol. The dehydration of isopropyl alcohol, normal butyl- and isobutyl alcohol was accomplished by slow distillation in a wire-gauze column (8) with 50 plates. Table 2 presents a summary of the boiling points

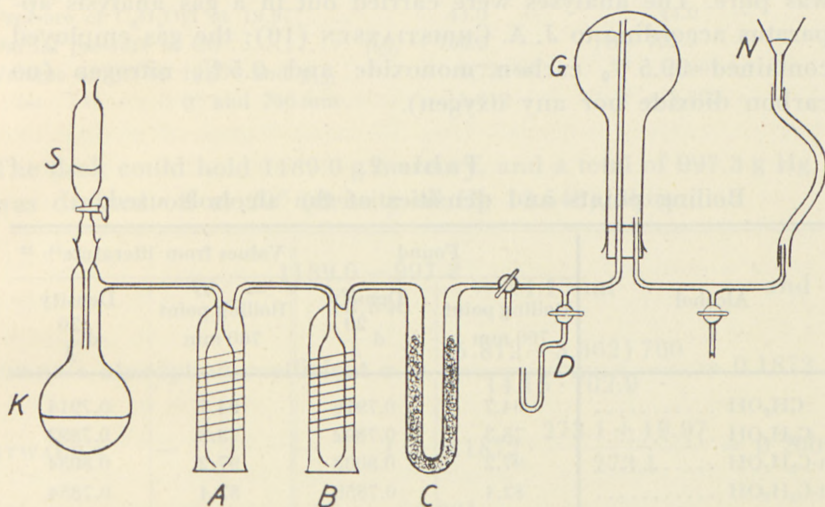


Fig. 1. S: Formic acid. K: Sulphuric acid. A: Sodium hydroxide solution. B: Sulphuric acid. C: Cotton-wool filter. G: Gasometer with mercury. N: Levelling container.

and densities found, the last two columns giving the corresponding constants from the literature. The boiling points were determined by distillation of 20 ml of alcohol in a boiling point apparatus described by H. BAGGESGAARD RASMUSSEN and F. REIMERS (9). Calibrated thermometers were placed so that the whole mercury column was in alcohol vapour. The boiling points are converted to 760 mm pressure. The densities were determined at $20.00 \pm 0.02^\circ$ in a 50 ml pycnometer with two graduated capillary tubes provided with ground-glass caps. In the calculation of the density correction is made for the buoyancy of the air.

Carbon monoxide was prepared in an apparatus like the one sketched in Fig. 1. From the separating funnel (S) anhydrous formic acid dripped into concentrated sulphuric acid in the flask (K). The carbon monoxide was led through glass spiral washing

bottles containing sodium hydroxide solution (A) and concentrated sulphuric acid (B) and a cotton-wool filter (C) into a 500 ml Berzelius gasometer (G) with levelling container (N). The carbon monoxide production was made very slow so that the washing could be effective. The gasometer was filled and emptied until sampling (stopcock D) showed that the carbon monoxide was pure. The analyses were carried out in a gas analysis apparatus according to J. A. CHRISTIANSEN (10); the gas employed contained 99.5 % carbon monoxide and 0.5 % nitrogen (no carbon dioxide nor any oxygen).

Table 2.
Boiling points and densities of the alcohols used.

Alcohol	Found		Values from literature ^{11, 12}	
	Boiling point 760 mm	Density $d \frac{20}{4}$	Boiling point 760 mm	Density $d \frac{20}{4}$
CH ₃ OH	64.7	0.7916	64.7	0.7914
C ₂ H ₅ OH	78.3	0.7892	78.3	0.7893
n-C ₃ H ₇ OH	97.2	0.8038	97.2	0.8034
i-C ₃ H ₇ OH	82.4	0.7859	82.4	0.7854
n-C ₄ H ₉ OH	117.8		117.7	
i-C ₄ H ₉ OH	107.9		107.9	

Experimental results.

The densities and vapour pressures used in the calculations are given in Table 3. The solubilities are expressed by Ostwald's absorption coefficient (l) and calculated from

$$l = \frac{(W - w) \cdot 760 \cdot T}{W_L \cdot P \cdot 273.1},$$

where W is the volume (0°, 760 mm) of the gas introduced, w the volume (0°, 760 mm) of the gas that remains after the absorption. W_L is the volume of the alcohol and P the partial pressure of the carbon monoxide, both at the prevailing absolute temperature T. The following survey shows an example of the calculation of the solubility

of carbon monoxide in ethyl alcohol:

	Before the absorption	After the absorption
Temperature $t^{\circ} \text{C}$	19.97°	19.97°
$1 + \frac{t}{273.1}$	1.0732	1.0732
Total pressure in the container ...	749.3 mm Hg	746.9 mm Hg
Pressure of $\text{C}_2\text{H}_5\text{OH}$ at 19.97°	44.0 —	44.0 —
Partial pressure of CO	$p_{\text{CO}} = 705.3$ —	$P = 702.9$ —
Volume of CO at 19.97° and p_{CO} .	6.721 ml	3.901 ml
— - - - 0° and 760 mm .	5.812 -	$w = 3.362$ -

The flask could hold 1189.0 g mercury, and a total of 997.3 g Hg was drained off at 20° (density of Hg 13.546), thus

$$W_L = \frac{1189.0 - 997.3}{13.546} = 14.15 \text{ ml} \quad \text{and}$$

BUNSEN'S absorption coefficient $\alpha = \frac{(5.812 - 3.362) 760}{14.15 \cdot 702.9} = 0.1872$

OSTWALD'S — — — $l = 0.1872 \cdot \frac{273.1 + 19.97}{273.1} = 0.2009.$

$$l_{20.0^{\circ}} = 0.201.$$

Table 3.

Values of densities and vapour pressures of the alcohols used in the calculations.

	Temp.	CH_3OH	$\text{C}_2\text{H}_5\text{OH}$	$n\text{-C}_3\text{H}_7\text{OH}$	$i\text{-C}_3\text{H}_7\text{OH}$	$n\text{-C}_4\text{H}_9\text{OH}$	$i\text{-C}_4\text{H}_9\text{OH}$
Density $\frac{t}{d \frac{4}{}}$	20.0	0.7915	0.7894	0.8035	0.7851	0.8098	0.8018
	30.0	0.7825	0.7810	0.7960	0.7769	0.8026	0.7941
	40.0	0.7740	0.7722	0.7875	0.7686	0.7954	0.7864
	50.0	0.7650	0.7633	0.7785	0.7603	0.7882	0.7785
	Lit.	(13)	(13)	(13)	(12)	(12)	(12)
Vapor pressure mm Hg	20.0	95.1	44.0	14.5	32.4	4.39	8.6
	30.0	160.3	78.1	27.6	59.1	9.52	17.0
	40.0	260.4	133.4	50.2	105.6	18.6	31.6
	50.0	409.4	219.8	87.2	176.8	33.7	56.2
	Lit.	(13)	(13)	(12)	(14)	(12)	(15)

In the numerical treatment of the experiments at other temperatures consideration was given to the various alcohol vapour pressures and to the coefficients of expansion of the alcohols, the mercury and the glass. The alcohol vapour pressures were not corrected for the reduction which is attributable to the dissolved carbon monoxide, nor was the volume of the alcohols corrected for changes due to evaporation or to carbon monoxide dissolved. The three last mentioned corrections are small in proportion to the experimental errors.

Tables 4—9 show the solubilities found. Values in the same vertical column originate from the same charge of alcohol and carbon monoxide. The experiments with methyl alcohol (Table 4) were difficult to reproduce, undoubtedly because of the very high vapour pressure. At 20° l was found to be 0.224, thus a value about 23% higher than that found by JUST (Table 1). An experiment with methyl alcohol to which had been added 1% of water gave at 20° $l = 0.220$. When the carbon monoxide pressure was varied the following values were found for the solubility in anhydrous methyl alcohol at 20° : 538 mm (0.227), 680 mm (0.224), 754 mm (0.224), thus the same within the experimental accuracy. The difference between JUST's results and those of the present investigation may perhaps be explained by the circumstance that different values of the vapour pressure of methyl alcohol have been used. JUST found that the solubility between 20° and 25° rises 0.0021 per degree, while the present experiments give a rise of 0.0008 per degree. A comparison of JUST's temperature coefficient with the temperature coefficient for the solubility of carbon monoxide in the other alcohols (Table 10) shows JUST's coefficient to be incredibly high.

For the solubility of carbon monoxide in ethyl alcohol (Table 5) l was found to be 0.200 at 20° , thus a value lying between those found by CARIUS and JUST (Table 1). The experiments reported under "Apparatus A" were carried out in an apparatus having a flask volume of about 300 ml. About one year later (a new dehydrated alcohol and a newly prepared carbon monoxide being used) experiments were made in "Apparatus B", with a flask volume of about 90 ml. Since only less than 20 ml of alcohol can be introduced because of the high solubility, and the rest of the flask is filled with mercury, Apparatus B makes the method so much more rapid and more satisfactory in producing tem-

perature equilibrium. As Table 5 shows, the two apparatuses gave the same value for the solubilities. In the case of the other alcohols only Apparatus B has been used. Ethyl alcohol with 1% of water gave $l = 0.190$ at 20° . From CARIUS' results one arrives at a temperature coefficient of 0.0007. JUST's results give no reliable temperature coefficient (0.0004) since the deviations of the solubilities found are so small. The present experiments show an increase in solubility of 0.00053 per degree, which is of the same order of magnitude as for the rest of the alcohols investigated.

Table 10 presents a survey of the solubilities found, interpolated to the temperatures 20° , 35° and 50° , as well as the corresponding temperature coefficients.

Table 4.
Solubility in methyl alcohol.

t	l	l	l
19.9	0.226		
20.0			0.222
20.1		0.224	
29.0	0.225		
34.2		0.236	
35.2			0.228
39.5	0.233		
48.8	0.241		
49.8			0.250

Table 5.
Solubility in ethyl alcohol.

Apparatus A			Apparatus B		
t	l	l	t	l	l
20.0	0.200	0.200	20.0	0.201	
35.1		0.206	20.3		0.203
40.0	0.210		20.7	0.201*	
40.6		0.208	28.9	0.204	
50.2	0.215		29.8		0.206
			33.7	0.208*	
			37.9	0.211	
			38.8		0.211
			47.9	0.214	
			50.2		0.217

* The equilibrium entered from higher to lower temperature.

Table 6.
Solubility in normal propyl
alcohol.

t	l	l
20.0	0.177	0.177
34.8	0.182	
35.0		0.182
50.0	0.191	0.188

Table 7.
Solubility in isopropyl
alcohol.

t	l
20.0	0.190
20.0	0.190
35.1	0.196
51.2	0.207

Table 8.
Solubility in normal butyl alcohol.

t	l	l
19.8.....		0.165
19.9.....	0.164	0.164
20.0.....		0.164
20.0.....		0.165
35.1.....	0.168	
35.7.....		0.170
35.7.....		0.169
49.8.....	0.173	
49.9.....		0.173

Table 9.
Solubility in isobutyl alcohol.

t	l	l
20.0.....	0.174	0.173
35.0.....	0.180	0.181
49.8.....		0.185
50.0.....	0.186	

Table 10.
Solubility (l) of carbon monoxide in the alcohols.

Temp.	20°.0	35°.0	50°.0	$\frac{dl}{dt}$ (20°—50°)
CH ₃ OH	0.224	0.230	0.248	0.0008
C ₂ H ₅ OH	0.200	0.207	0.216	0.00053
n-C ₃ H ₇ OH	0.177	0.182	0.189	0.00040
i-C ₃ H ₇ OH	0.190	0.196	0.206	0.00053
n-C ₄ H ₉ OH	0.164	0.169	0.173	0.00030
i-C ₄ H ₉ OH	0.174	0.180	0.186	0.00040

Discussion.

Fig. 2 shows corresponding values of $\frac{1}{T} \cdot 10^5$ (abscissae) and $\log l$ (ordinates). The degrees centigrade corresponding to the reciprocal absolute temperature are plotted on the upper hori-

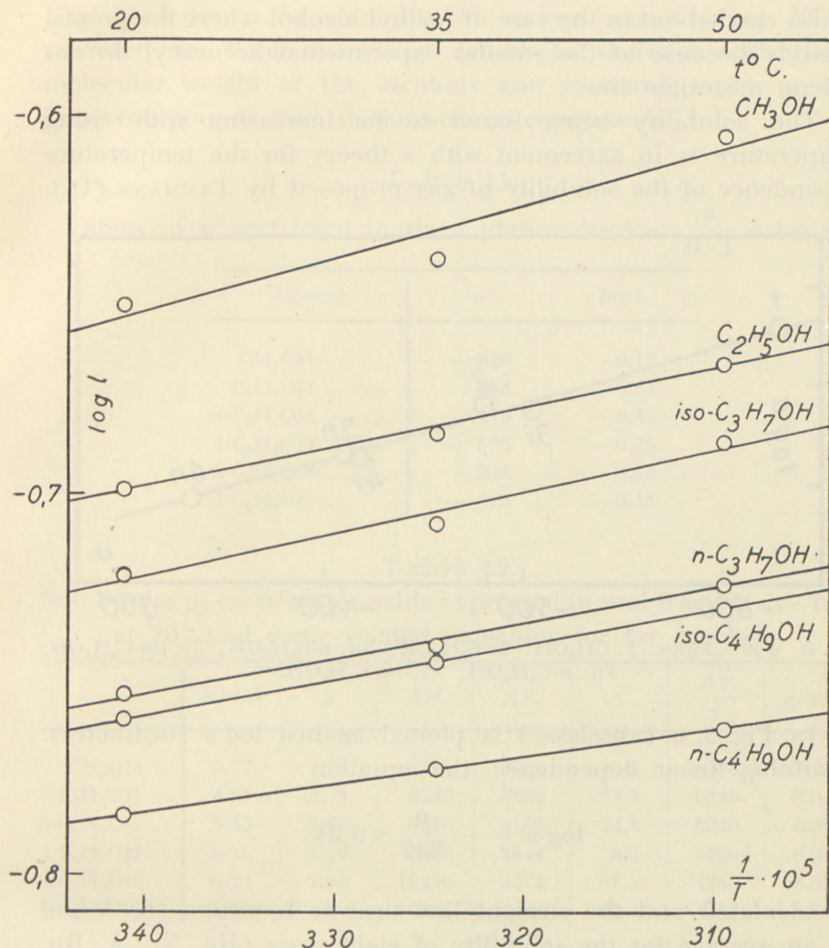


Fig. 2. Solubility-temperature curves.

zontal axis. Integration of the equation for the temperature dependence of the solubility, $\frac{d \ln l}{dT} = -\frac{u}{RT^2}$, where u is the heat of solution, gives, for constant u :

$$\ln l = \frac{u}{1.986 \cdot T} + \ln a, \text{ or } \log l = \frac{u}{4.57 \cdot T} + \log a.$$

The constants u and $\log a$ in the last equation are calculated by means of the method of least squares, Table 11. The calculation

is also carried out in the case of methyl alcohol where the points, possibly because of the smaller experimental accuracy, do not fall on a straight line.

The solubility being found to be increasing with rising temperature is in agreement with a theory for the temperature dependence of the solubility of gas proposed by TAMMANN (16).

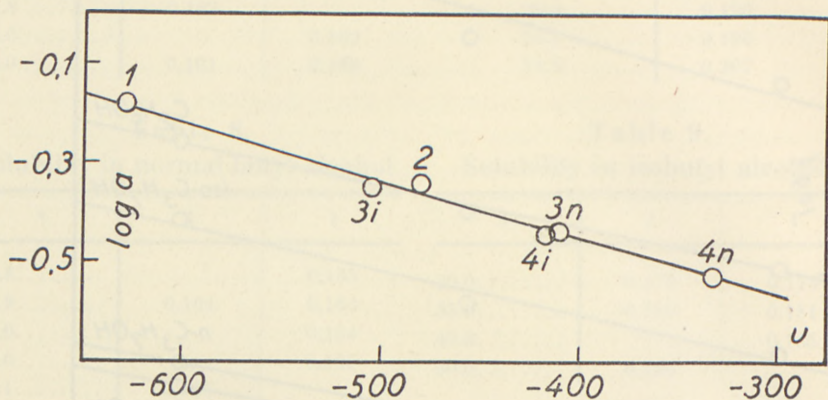


Fig. 3. u in calories. 1: CH₃OH; 2: C₂H₅OH; 3n: n-C₃H₇OH; 3i: iso-C₃H₇OH; 4n: n-C₄H₉OH; 4i: iso-C₄H₉OH.

In Fig. 3, u (abscissae) is plotted against $\log a$ (ordinates). Assuming linear dependence, the equation

$$\log a = -\frac{u}{837} - 0.94$$

is calculated and the straight line drawn. LANNUNG (6) found on an average for the solubility of eight gases (He, Ne, A, Rn, H₂, CO, CO₂) in methyl alcohol, ethyl alcohol, acetone, cyclohexane and cyclohexanol the equation:

$$\log a = -\frac{u}{3000} - 0.3.$$

When comparing the solubility of gases in different liquids it is the most reasonable to express the solubility in mol fraction (x) which for sparingly soluble gases can be calculated from

$$x = \frac{1 \cdot 273.1 \cdot \text{molecular weight}_{\text{alcohol}} \cdot 10^{-3}}{T \cdot 22.4 \cdot d_{T(\text{alcohol})}}$$

Expressed by x the solubility of carbon monoxide at 20° is recorded in Table 12. x is found to increase with rising molecular weight of the alcohols and is found to be greater in isocompounds than in the normal compounds.

Table 11.

Values of u and $\log a$ in the equation $\log l = \frac{u}{R \cdot T} + \log a$.

Alcohol	u	$\log a$
CH ₃ OH	- 630	- 0.18
C ₂ H ₅ OH	- 483	- 0.34
n-C ₃ H ₇ OH	- 410	- 0.45
i-C ₃ H ₇ OH	- 506	- 0.35
n-C ₄ H ₉ OH	- 334	- 0.54
i-C ₄ H ₉ OH	- 419	- 0.45

Table 12.

Solubilities of carbon monoxide expressed in mol fraction ($x \cdot 10^4$) at 20° and some related constants for the alcohols.

	$x \cdot 10^4$	μ	ΔH	ΔE	v	$\frac{\Delta E}{v}$	$\alpha \cdot 10^{24}$
CH ₃ OH ..	3.77	1.6	8520	7850	40.5	8010	3.24
C ₂ H ₅ OH ..	4.85	1.71	9580	8882	58.3	6240	5.04
n-C ₃ H ₇ OH ..	5.50	1.66	9840	9105	74.8	5040	6.90
i-C ₃ H ₇ OH ..	6.10	1.70	9550	8844	76.5	4790	6.91
n-C ₄ H ₉ OH ..	6.24	1.66	10450	9674	91.5	4380	8.72
i-C ₄ H ₉ OH ..	6.69	1.8	10220	9464	92.4	4250	8.74

μ : Dipole moment in Debye; ΔH : Heat of vaporization in $\frac{\text{cal}}{\text{mol}}$; ΔE : Energy of vaporization in $\frac{\text{cal}}{\text{mol}}$ ($= \Delta H - RT$); v : Molecular volume in ml; $\frac{\Delta E}{v}$: "Internal pressure" in atm.; α : Polarizability calculated from molecular refractivity (R), $\alpha = \frac{3R}{4\pi N}$. Values from Intern. Crit. Tables and Landolt-Börnstein's Tables.

Table 12 moreover records the dipole moments (μ), heats of vaporization (ΔH), molecular volumes (v), and polarizabilities (α) of the alcohols. Moreover, $\frac{\Delta E}{v}$ has been calculated (see the table), presumably giving quite valuable information regarding the relative internal pressures in the alcohols which are almost

Table 13.

Solubility of different gases in the alcohols expressed in mol fraction ($x \cdot 10^4$) at 20°. Ar at 0°.

	He ⁶⁾	Ne ⁶⁾	Ar ¹⁸⁾	Rn ²⁰⁾	H ₂ ³⁾	N ₂ ³⁾	O ₂ ²¹⁾	CO	CO ₂	N ₂ O ²²⁾
"Ideal"*		1.7	21	710**		10	16	11	178	202
CH ₃ OH . . .	0.57	0.78	4.8	94	1.5	2.3	3.1	3.77	60 ²²⁾	53
C ₂ H ₅ OH . . .	0.73	1.05	6.5	170	2.1	3.4	3.7	4.85	69 ²²⁾	72
n-C ₃ H ₇ OH . . .			7.3	266				5.50	77 ³⁾	
i-C ₃ H ₇ OH . . .				215				6.10		
n-C ₄ H ₉ OH . . .			8.6	338				6.24		
i-C ₄ H ₉ OH . . .			9.4	308				6.69	75 ³⁾	

* Calculated by HILDEBRAND (17).** Calculated by the author.

identical in polarity. Carbon monoxide belongs to the non-polar gases (dipole moment 0.12 Debye (11)). The fact that the solubility is found to increase with decreasing internal pressure is in agreement with a long series of earlier observations (see, for example, a review of the solubility of non-polar gases in different liquids, on p. 134 of HILDEBRAND's monograph (17)). The fact that the solubility varies in the same manner as the polarizability is in agreement with a series of observations by SISKIND and KASARNOWSKY (18).

DOLEZALEK (19) has shown that for a number of liquid mixtures of two non-electrolytes, A and B, (e. g. benzene and ethylene chloride) one finds for the whole range of concentration that

$$p = p_0 \cdot x,$$

where p is the partial pressure of A (B), p_0 the vapour pressure of pure A (B) and x the concentration of A (B) calculated as mol fraction. If one of the components is associated (e. g. CCl₄ in benzene), or the components enter into additive combination with each other (e. g. acetone and chloroform), this circumstance must be taken into account in the calculation of x . On the basis of the following considerations, DOLEZALEK has proposed a method for calculating the ideal solubility of gases in liquids: A liquid which is saturated by a gas of the partial pressure p can be imagined to be formed by mixing into the liquid so much "liquid gas" that the mixture has a partial pressure of the gas

of the magnitude p . The solubility of the gas given at a partial pressure of one atmosphere then is

$$\text{solubility} = x = \frac{1}{p_0}.$$

where p_0 is the vapour pressure of the gas in liquid form at the temperature considered.

Most of the gases for which solubility measurements are available have critical temperatures considerably below the temperature at which the solubility determinations have been carried out. HILDEBRAND (17) has, however, for a number of gases calculated theoretical solubilities according to the above equation by extrapolating available vapour pressure measurements of the condensed gases to 20° . Some of the results of these calculations are reproduced in Table 13. The solubility of radium emanation has by the present author been calculated on the basis of vapour pressure measurements by GRAY and RAMSAY (23), by interpolating to a vapour pressure at 20° of 14.1 atmospheres, whence $x = \frac{1}{14.1} = 0.0710$. This calculation of solubilities is thus very approximate, but nevertheless gives a fair orientation regarding the relative magnitude of these quantities. This "ideal solubility", x , is independent of the solvent. In practice the solubilities are most generally found to depend on the solvent, and as a rule to be between one and ten times as small as the "ideal" one, though for the solubility in water one as a rule finds that it is about one hundred times smaller.

Table 13 surveys the solubility of different one-, two-, and three-atomic gases in six alcohols. The values are inter- or extrapolated to 20° and converted into mol fraction.

The present investigations have been carried out in the Department of Inorganic Chemistry, the Danish School of Pharmacy. The author wishes to thank the Head of the Department, Professor CARL FAURHOLT, for permission to do this work and for the excellent working conditions which were available. He is moreover indebted to Professor NIELS BJERRUM for the loan of the apparatus in which the solubility determinations were made, and to the Carlsberg Foundation for financial support in the purchase of a gas analysis apparatus.

Summary.

The solubility of carbon monoxide between the temperatures 20° and 50° is determined in methyl-, ethyl-, normal propyl-, isopropyl-, normal butyl-, and isobutyl alcohol. The results are expressed by means of Ostwald's solubility coefficient (l) and recorded in Table 10, where also dl/dt is calculated. A graphical presentation of the measurements is given in Fig. 2. Within the experimental accuracy the points found lie on a straight line for the equation of which ($\log l = \frac{u}{R \cdot T} + \log a$) the constants u and $\log a$ are calculated in Table 11.

Table 12 gives the solubility expressed in mol fraction ($x \cdot 10^4$). This table also includes a calculation of the ratio between the energy of vaporization and the molecular volume for each alcohol, $\frac{\Delta E}{v}$, which for the alcohols almost identical in polarity furnishes information regarding the relative internal pressure. It will be seen that the solubility of carbon monoxide increases with falling internal pressure for the alcohols, which rule is in agreement with that governing the solubility of other gases in various solvents.

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