NOTES

The Solubility of Ethyl Acetate in Water

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In view of some uncertainties in the literature[2-6] it was found necessary to redetermine the solubility of ethyl acetate at various temperatures for an investigation which was in progress.

The experimental results are given in Table I where t is the turbidity temperature in °C. and s is the solubility in grams of ethyl acetate per 100 g. of water.

Table I

t	s	t	s	t	s
19.2	8.34	25.9	7.93	31.2	7.60
20.4	8.31	26.0	7.93	31.8	7.58
20.5	8.29	26.3	7.94	31.9	7.58
21.3	8.26	27.0	7.89	31.9	7.57
22.0	8.22	28.0	7.80	32.0	7.51
22.6	8.14	28.1	7.81	32.8	7.49
22.7	8.12	28.4	7.82	33.0	7.46
23.0	8.11	28.7	7.81	33.8	7.40
23.4	8.10	29.7	7.71	34.0	7.38
24.7	8.05	29.9	7.71	36.8	7.32
25.1	8.03	30.0	7.72	37.8	7.25
25.4	8.02	30.0	7.69	39.9	7.15
25.4	8.03	30.1	7.69	39.9	7.15
25.4	8.00				

By the application of the method of least squares the following equation is calculated

$$s = (9.552 \pm 0.018) - (0.0618 \pm 0.0006)t$$

The results of this investigation are compared with those previously given in the literature in Table II.

Table II

Comparison of the Solubility Results for Ethyl Acetate in Water

Investigator	20°	25°	30°	35°	40°	45°	50°
This invest.	8.32	8.01	7.70	7.39	7.08	..	..
Schles. and Kub.[3]	8.42	8.03	7.69	7.41	7.18	7.00	6.88
Merriman[4]	8.53	8.08	7.70	7.38	7.10	..	..
Seidel[5]	9.02	8.58	8.24	7.98	7.72	7.53	7.31
Gl. and P.[6]	..	7.39	..	..	..	..	6.04
B. and Gl.[7]	8.40	..	..	..	6.97	..	..

While the earlier results of Glasstone and Pound[5] are far lower than the other results, the more recent values of Beech and Glasstone,[6] determined by a different method, are in better agreement although the value given at 40° is still somewhat low. The high results of Seidel[4] may be caused by appreciable amounts of water and alcohol being present in the ethyl acetate thus increasing the solubility above the correct value.

Consideration of the data above leads to the following average values for the solubility of ethyl acetate (in g. of ethyl acetate per 100 g. of water) as reliable: 20°, 8.42; 25°, 8.04; 30°, 7.70; 35°, 7.39; 40°, 7.12.

Experimental

The solubilities were determined by the synthetic, turbidimetric method.[7] The solubilities of ethyl acetate agree within ±0.1° when determined by heating and then cooling. The appearance of turbidity may be detected within about 0.2° range usually. Temperatures were determined with a $^1/_{20}$° thermometer which gave agreement within $^1/_{100}$° with the sodium sulfate transition point. Each solubility was redetermined two or three times.

The ethyl acetate employed was Mallinckrodt analytical reagent grade ethyl acetate. The percentage of water as determined by Karl Fischer reagent[8] was 0.06%. The ethyl acetate was redistilled from a column of 24 theoretical plates. No change in refractive index ($n^{24.3}{}_{\mathrm{D}}$ 1.3697) was found.

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Determination of the Terminal Carboxyl Residues of Peptides and of Proteins[1]

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Recently, Fromageot, *et al.*,[3] and Chibnall and Rees[4] have described the reduction of the terminal carboxyl groups of insulin with subsequent identification of the resulting amino alcohols generated by the hydrolysis of the reduced protein. The present note reports a modification similar to that which Waley and Watson[5] have applied to insulin, of the method originally presented by Schlack and Kumpf[6] for the identification of the terminal carboxyl residues of peptides.

In outline, this method involves the creation of the thiohydantoin on the carboxyl end of the peptide chain in an acid medium, the amino end being blocked by acetylation. The thiohydantoin is then hydrolyzed from the peptide in an acid medium and isolated. The purified thiohydantoin is then hydrolyzed in an alkaline medium, thus producing the corresponding amino acid which is then identified and estimated.

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