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Isolation and Recovery of Dicarboxylic Acids

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NOTE

Isolation and Recovery of Dicarboxylic Acids

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A means of separating and isolating certain dibasic acids from solution was found during the course of a systematic investigation of stoichiometric urea–dicarboxylic acid complexes. The first publication (*1*) resulting from this work described the structure and the method of preparation for the insoluble urea–dicarboxylic acid complexes from the soluble dibasic acids in solution. In the present report we wish to describe a method for recovering these acids from their complexes.

The complexes included in this study were $HOOC(CH_2)_nCOOH \cdot 2H_2NCONH_2$ for $n = 0$, 2 to 5 and $HOOCCH_2COOH \cdot H_2NCONH_2$ These stoichiometric complexes or molecular compounds of urea should not be confused with the nonstoichiometric urea inclusion compounds (*2*). Using a standardized procedure, we were able to recover the dibasic acids from their complexes. Each complex, after being dissolved in water and acidified, was continuously extracted with ether. The organic matter isolated from the ether was tested for nitrogen and its neutralization equivalent was determined. The results obtained appear in Table 1. Table 1 shows that only the recovered oxalic and malonic acids were contaminated by urea. In both cases the percentage of urea was less than 0.05%. The presence of urea in these two cases is due to the fact that both acids are much stronger than the higher homologues and both form salts with urea in aqueous solution. It appears that when the aqueous solution is acidified with HCl, a small quantity of this salt persists in the solution and is slowly extracted by the ether. The higher homologues which form H-bonded complexes rather than salts are easily broken apart by HCl, so that the dibasic acid–urea complex is for all practical purposes absent.

The recoveries of acid from the complex were high enough to make such a procedure practical for isolating dibasic acids (*1*). Although the original procedure was directed toward obtaining complexes of maximum purity, the method of preparation could easily be modified using larger quantities of urea to increase the yield of insoluble acid complex. This urea could also be recycled.

TABLE 1

Recovery of Acids from Complexes

Complex, m.p.	Recovered acid: % by wt.[a]	% Urea[b]	M.p. (lit.)[c]
Oxalic acid·2 urea, 140–151[d]	86.8	.02	155(189)
Malonic acid·urea, 89–91	98.2	.04	148–155(135.6)
Succinic acid·2 urea, 134–40	97.9	0.00	182–185(185)
Glutaric acid·2 urea, 127–30	96.8	0.00	85–93(95–96)
Adipic acid·2 urea, 107–12	96.2	0.00	136–148(151–153)
Pimelic acid·2 urea 127–132	97.7	0.00	92–100(103)

[a] The percent by weight of recovered acids was determined by titration of an aliquot of the recovered acid dissolved in water with *N*/10 sodium hydroxide with a phenolphthalein indicator.

[b] The percent urea was calculated from the percent nitrogen assuming all nitrogen came from urea.

[c] All the literature-reported values of the melting point of the dibasic acids were obtained from either the *Handbook of Chemistry and Physics*, 43rd ed., or *Langes Handbook of Chemistry*, 6th ed.

[d] Uncorrected melting points were determined on a Fisher-Johns melting-point block.

One gram of complex was dissolved in 50 ml of water and acidified with 3 ml of concentrated hydrochloric acid. The solution was extracted with ether in a continuous extractor for 24 hours. The ether solution was divided in half and each portion was evaporated to dryness. One portion was dissolved in water and titrated with *N*/10 sodium hydroxide using a phenolphthalein indicator. The second portion was used to determine nitrogen, if present.

The procedure used to prepare the complexes was reported previously (*1*).

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