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Abstract

Full Text

Chemistry

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QUANTITATIVE DETERMINATION OF SALTS AND THEIR MIXTURES WITH ACIDS AND BASES BY THE METHOD OF POTENTIOMETRIC TITRATION IN A MEDIUM OF METHYL ETHYL KETONE

(Presented by Academician I. V. Tananaev, November 27, 1961)

Differentiated acid-base titration of salts and their multicomponent mixtures with acids and bases cannot be carried out in aqueous solutions, because many salts in an aqueous medium are not titrated at all, and water has a leveling effect on acids and bases ^(1,2).

We have developed a potentiometric method for the analysis of salts in non-aqueous solutions, the advantages of which are well known ⁽³⁻⁵⁾. The method is based on the interaction of the analyzed mixture with perchloric acid in a methyl ethyl ketone medium and the subsequent differentiated titration with tetraethylammonium hydroxide (TEAH) of the mixture consisting of the acid formed as a result of the interaction of the salt with perchloric acid, the excess unreacted perchloric acid, and, in some cases, the acid contained in the analyzed mixture.

Experimental part

The objects of investigation were: 1) various individual salts and their binary mixtures; 2) binary and ternary mixtures of salts with acids; 3) binary and ternary mixtures of salts with bases.

The titration was carried out in a medium of chemically pure dried methyl ethyl ketone; changes in potentials were measured with an LP-5 potentiometer with glass and calomel electrodes; to prevent carbon dioxide from the air from entering the mixture being titrated, the titration was conducted in a stream of dry nitrogen.

Titration of individual salts. We titrated more than 100 different organic and inorganic salts of the type sodium acetate, potassium benzoate, sodium nitrate, etc. Upon the action of perchloric acid on the salt being analyzed, a

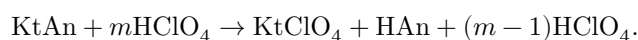
Fig. 1

Figure 1: Fig. 1

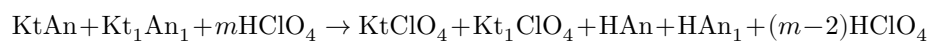
Fig. 2

Figure 2: Fig. 2

mixture of acids is formed, consisting of an excess of perchloric acid and the acid formed as a result of the interaction of the analyzed salt with HClO_4 :



Titration of binary mixtures of salts. As binary mixtures we titrated: sodium acetate + potassium salicylate, potassium salicylate + potassium benzoate, potassium benzoate + sodium oxalate, etc., in all more than 20 mixtures. Upon the action of perchloric acid on the analyzed mixture, a mixture of acids is formed, consisting of perchloric acid that has not entered into the reaction and acids released from the analyzed salts:



Analysis of the titration curves (Fig. 1) shows that the first titration jump corresponds to neutralization of the perchloric acid that has not entered into the reaction; the second, third, and fourth titration jumps (in the case of titration of salts of dibasic acids) correspond to titration of the acids HAn or $\text{HAn} + \text{HAn}_1$ released from the analyzed salts.

Titration of binary mixtures of salts with acids. Mixtures were titrated: sodium acetate + acetic acid, potassium benzoate + benzoic acid, sodium oxalate + oxalic acid, etc., more than 50 mixtures in all. As a result of the action of hydrochloric acid on the analyzed mixture, a mixture of acids is formed: hydrochloric acid that has not entered into the reaction, and the total amount of acid, i.e., that contained in the mixture and liberated from the salt:



Fig. 1. Curves of potentiometric titration of individual salts and their multi-component mixtures in methyl ethyl ketone medium.

1 –sodium acetate; 2 –potassium propionate; 3 –sodium oleate; 4 –potassium salicylate; 5 –sodium anthranilate; 6 –potassium benzoate; 7 –sodium oxalate; 8 –potassium salicylate + potassium benzoate; 9 –potassium salicylate + sodium acetate; 10 –sodium oxalate + potassium benzoate

Fig. 2. Curves of potentiometric titration of multicomponent mixtures of salts with acids in methyl ethyl ketone medium.

1 –sodium acetate + acetic acid; 2 –potassium propionate + propionic acid; 3 –potassium benzoate + benzoic acid; 4 –sodium oxalate + oxalic acid; 5 –potassium salicylate + salicylic acid + benzoic acid; 6 –sodium acetate + acetic acid + salicylic acid; 7 –potassium benzoate + benzoic acid + oxalic acid

Titration of ternary mixtures of salts with acids. The following mixtures were titrated: sodium acetate + acetic acid + salicylic acid, potassium salicylate + salicylic acid + benzoic acid, potassium benzoate + benzoic acid + oxalic acid, etc., more than 30 mixtures in all. When hydrochloric acid acts on the analyzed mixture, a mixture of acids is formed, consisting of an excess of hydrochloric acid, the acids contained in the analyzed mixture, and the acids liberated from the salts:



or



From an analysis of the titration curves (Fig. 2) it is seen that the first jump in titration corresponds to neutralization of the excess hydrochloric acid, while the second, third, and fourth jumps in titration (in the case of titration of dibasic acids or their salts) correspond to titration of the acids (2HAN_1 or $2\text{HAn} + \text{HAN}_1$) contained in the analyzed mixture and liberated from the salts.

Titration of binary mixtures of salts with bases. As binary mixtures, the following were titrated: sodium acetate + sodium hydroxide, potassium benzoate + potassium hydroxide, sodium anthranilate + sodium hydroxide, etc., more than 30 mixtures in all. When hydrochloric acid acts on the analyzed mixture, a mixture of acids is formed: hydrochloric acid that has not entered into the reaction, and the acid liberated from the salt:



Titration of ternary salt mixtures with bases. The following were titrated: sodium acetate + potassium salicylate + caustic potash, potassium salicylate + potassium benzoate + caustic potash, potassium benzoate + sodium oxalate + caustic potash, etc., more than 20 mixtures in all.

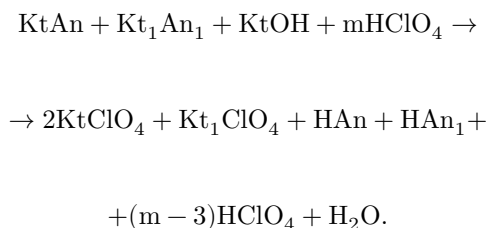
Fig. 3. Potentiometric titration curves of multicomponent salt mixtures with bases in methyl ethyl ketone medium.

Fig. 3. Potentiometric titration curves of multicomponent salt mixtures with bases in methyl ethyl ketone medium. 1 –sodium oleate + caustic soda; 2 –potassium salicylate + caustic potash; 3 –sodium anthranilate + caustic soda; 4 –sodium oxalate + caustic soda; 5 –potassium salicylate + potassium benzoate + caustic potash; 6 –sodium acetate + potassium salicylate + caustic soda; 7 –sodium oxalate + potassium benzoate + caustic potash

Figure 3: Fig. 3. Potentiometric titration curves of multicomponent salt mixtures with bases in methyl ethyl ketone medium. 1 –sodium oleate + caustic soda; 2 –potassium salicylate + caustic potash; 3 –sodium anthranilate + caustic soda; 4 –sodium oxalate + caustic soda; 5 –potassium salicylate + potassium benzoate + caustic potash; 6 –sodium acetate + potassium salicylate + caustic soda; 7 –sodium oxalate + potassium benzoate + caustic potash

1 –sodium oleate + caustic soda; 2 –potassium salicylate + caustic potash; 3 –sodium anthranilate + caustic soda; 4 –sodium oxalate + caustic soda; 5 –potassium salicylate + potassium benzoate + caustic potash; 6 –sodium acetate + potassium salicylate + caustic soda; 7 –sodium oxalate + potassium benzoate + caustic potash.

As a result of the action of perchloric acid on the mixture being analyzed, a mixture of acids is formed, consisting of an excess of perchloric acid and of acids liberated from the salts:



Analysis of the titration curves (Fig. 3) shows that the first titration jump corresponds to neutralization of the excess perchloric acid, while the second, third, and fourth titration jumps (in the case of titration of salts of dibasic acids) correspond to titration of the acids (HAn or HAn + HAn₁) liberated from the salts. Some results of quantitative determinations are presented in Table 1.

Thus, the proposed method makes it possible, very rapidly and with sufficient accuracy for quantitative determinations, to titrate differentially not only individual salts, but also their multicomponent mixtures with acids and bases from a single weighed sample of the substance being determined.

Table 1

Results of quantitative determination of some salts and their multi-component mixtures with acids and bases

Substance determined	Taken, g	Found, g	Relative error, %
Sodium acetate	0.0758	0.0758	0
Potassium benzoate + potassium salicylate	0.0197	0.0196	-0.50
Potassium benzoate + potassium salicylate	0.0185	0.0187	+1.06
Sodium anthranilate + anthranilic acid	0.0324	0.0318	-1.85
Sodium anthranilate + anthranilic acid	0.0366	0.0365	-0.27
Sodium acetate + acetic acid + salicylic acid	0.0386	0.0386	0
Sodium acetate + acetic acid + salicylic acid	0.0308	0.0312	+1.28
Sodium acetate + acetic acid + salicylic acid	0.0322	0.0309	-4.0
Potassium propionate + caustic potash	0.0468	0.0454	-2.98
Potassium propionate + caustic potash	0.0357	0.0362	+1.38

Substance determined	Taken, g	Found, g	Relative error, %
Potassium salicylate + potassium benzoate + caustic potash	0.0214	0.0216	+0.92
Potassium salicylate + potassium benzoate + caustic potash	0.0200	0.0197	-1.50
Potassium salicylate + potassium benzoate + caustic potash	0.0369	0.0375	+1.60

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Note: Figure translations are in progress. See original paper for figures.

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