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Progress in the Analytical Chemistry of Non-aqueous Solutions

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I. INTRODUCTION

With the rapid development of chemistry and chemical technology, it is becoming urgently necessary to improve and develop new and more effective methods for the analysis of inorganic and organic compounds. Among the most extensively applied analytical methods, a unique position is occupied by chemical and physicochemical methods based on the titration of various compounds in a medium of non-aqueous liquid solvents and in liquefied gases[1-25]. As a result, the analytical chemistry of non-aqueous solutions has developed into an important independent branch of chemical science. Reactions in non-aqueous solutions have begun to be used not only in the familiar classical

methods of alkalimetry and acidimetry, oxidation-reduction, precipitation, and complex formation, but also in specific methods of condensation, addition, substitution, oxomercuration, etc. As a result, the range of titrimetric methods has extended and there has been a marked increase in the number of substances whose quantitative determination has been found to be possible by volumetric methods[1,5,15,17,21].

It is sufficient to point out that methods involving the titration of non-aqueous solutions can be now used for the quantitative determination of a large number of individual compounds and that the number of mixtures which can be analysed by these methods is greater than the number of mixtures which analytical chemists have learned to titrate in the 140 years since Gay-Lussac introduced the methods of volumetric analysis in aqueous solutions into analytical practice. Potentiometric, conductometric, high-frequency, spectrophotometric, polarographic, chromatographic, and other physicochemical methods for the analysis of non-aqueous solutions have found the most extensive applications[25-43].

Methods involving the acid–base titration of non-aqueous solutions of various substances exhibiting acidic or basic properties in non-aqueous media are of the greatest practical and theoretical interest. This is due to the specific influence of non-aqueous solvents on many compounds whose aqueous solutions do not exhibit acidic or basic properties. As a result, the method of acid-base titration can be used successfully for the determination of the great majority of compounds, which cannot be titrated in aqueous media. At the present time non-aqueous titrametric methods are not only widely used in scientific research, but are also successfully employed in factory laboratories for chemical analysis.

In the last 4–5 years, since the publication in "Uspekhi Khimii" [Russian Chemical Reviews] of the first review on the application of non-aqueous solvents in analytical chemistry[8], research in this area has been in the following directions: (1) the development of the theoretical principles of the analytical chemistry of non-aqueous solutions—the quantitative estimation of the influence of non-aqueous solvents on the properties of electrolytes, the study of the differentiating action of solvents, etc.; (2) the use of new organic solvents as media for titration and of new reagents for the preparation of titrant solutions; (3) the development of methods for the analysis not only of individual compounds but also of mixtures of substances which exhibit analogous properties, for example mixtures of isomers, members of the same homologous series, etc.; (4) the development of methods for the analysis of compounds which do not exhibit acid-base properties in water, for example neutral salts, unsaturated compounds, ketones, aldehydes, etc.; (5) the extensive introduction of non-aqueous analytical methods for the analytical control of the composition of intermediates and the purity of finished products; and (6) the search for new methods for the determination of the equivalence point in titrations.

II. THEORY OF THE ACID–BASE TITRATION OF NON-AQUEOUS SOLUTIONS

In the theory of the analytical chemistry of non-aqueous solutions, a particularly important position is now held by ideas on the marked influence of the nature and physical properties of the solvent, the concentration of the solution, and the nature of the dissolved substance on the state of the dynamic equilibria between the ions and molecules of the dissolved substance and the solvent. This influence affects the physicochemical properties of the dissolved substance (electrical conductivity, solubility, ability to undergo dissociation, association, complex formation, etc.), and also the direction of ion exchange reactions[44-59].

Izmailov, Shkodin, Kolthoff, Bruckenstein, Hall, and a number of other workers previously carried out detailed studies of the acid–base equilibrium in anhydrous acetic acid. Work has recently been published on acid–base reactions in acetonitrile, ethylenediamine, t-butyl alcohol, dimethyl sulphoxide, and other solvents. The studies were carried out chiefly to estimate the strengths of acids and bases by measuring K_{diss} in non-aqueous solutions.

Acid–base equilibrium in acetonitrile has been studied by Kolthoff and coworkers[60-64]. It was found that in acetonitrile, perchloric acid is completely dissociated, whereas other acids, which are strong in water, are weak electrolytes. Sulphuric, nitric, hydrochloric, and hydrobromic acids dissociate according to the equation:

$$2HAn \rightleftarrows H^+ + AnHAn^-.$$

Picric acid at low concentrations (less than 0.1 M) dissociates to H^+ and Pi^-, but at higher concentrations it dissociates according to the equation

$$3HPi \rightleftarrows H^+ + Pi(HPi)_2^-.$$

Spectrophotometric and conductometric methods have been used to determine the dissociation constants

$$K_{AnHAn^-} = \frac{[AnHAn^-]}{[HAn]\,[An^-]} \text{ and } K_{HAn} = \frac{[H^+]\,[An^-]}{[HAn]}.$$

for the above acids. The acids can be arranged in the following order of decreasing strengths in a medium of acetonitrile: $HClO_4 > HBr > H_2SO_4 > HNO_3 > HCl > HPi$.

The authors cited compare the behaviour of the acids in acetic acid and in acetonitrile, by comparing the difference in the degree of dissociation of the acids with the solvation, dielectric constant, and basic nature of the solvent, with allowance for the formation and dissociation of ion pairs involving hydrogen bonding. The calculated curves for the conductometric titration of the bases in acetonitrile shows good agreement with the experimental curves[62-64]. Acetonitrile is an excellent differentiating solvent, due primarily to its very weak acidic and basic properties.

The behaviour of a number of acids and bases in acetonitrile has also been studied by other workers[65-69]. Measurement of the electrical conductivity of bases in acetonitrile showed that the dissociation of bases such as n-butylamine, diethylamine, triethylamine, 1,3-diphenylguanidine, and others in acetonitrile is less by four orders of magnitude than that in methanol[65]. The authors cited conclude that the low dissociation of the bases in acetonitrile is due chiefly to the very weak acidic properties of the solvent and the weak tendency for solvation to take place. At a concentration greater than 5×10^{-2} M, the dissociation of diphenylguanidine takes place according to the equation:

$$2B + CH_3CN \rightleftarrows (BH : B)^+ + CH_2CN^-;$$

at lower concentrations, the dissociation takes place according to the equation

$$B + CH_3CN \rightleftarrows BH^+ + CH_2CH^-.$$

The other bases are dimerised in acetonitrile and dissociate according to the equation:

$$B_2 + 2CH_3CN \rightleftarrows 2BH^+ + 2CH_2CN^-.$$

The determination of K_{diss} for perchloric acid in acetonitrile and other nitriles gave a value of about 10^{-3}, which does not agree with Kolthoff's data[70].

Schaap and coworkers[71] studied acid–base reactions in ethylenediamine. Because of the low value of the dielectric constant of ethylenediamine (DC = 12.5), none of the acids is completely dissociated in this medium. At a concentration greater than 10^{-3} M, even strong electrolytes are present in this solvent in the form of ion pairs. The association constants for the ion pairs formed by a number of acids which are strong acids in water (HCl, HBr, and HNO_3) have been determined. Values of K_{diss} for a number of derivatives of phenol, benzoic acid, and *p*-toluenesulphonic acid in ethylenediamine have been found by the electrical conductivity method[71]. Ethylenediamine exhibits a levelling action, since for all the acids studied $pK \simeq 4$.

Fritz and Marple[72,73] studied acid–base equilibria in t-butyl alcohol. On the basis of earlier studies in alcohols and other solvents with low dielectric constants, they suggest that the dissociation of acids in t-butyl alcohol takes place with the formation of ion pairs, although this process does not take place very extensively:

$$HAn \rightleftarrows H^+An^-;\ H^+An^- \rightleftarrows H^+ + An^-.$$

Potentiometric and conductometric methods were used to measure K_{diss} for perchloric, picric, and benzoic acids and 2,4-dinitrophenol.

Study of the acid–base properties of dimethyl sulphoxide[74,75] showed that in some respects this solvent behaves like water: perchloric, hydrochloric, and sulphuric acids are completely dissociated in this medium. The order of dissociation of the other acids is different from that in water. Thus picric acid is more extensively dissociated in dimethyl sulphoxide ($\Delta pK = -2.7$) than in water, whereas acetic, benzoic, and salicylic acids ($pK = 11.4$, 10, and 6.9) are less extensively dissociated in this solvent than in water; the dissociation constant of HSO_4^- in dimethyl sulphoxide is less than that in water.

Study of acid–base equilibrium in glycols (HS represents the solvent)[76] showed that the dissociation of acids corresponds to the equation

$$HAn + HS \rightleftarrows H_2S^+ + An^-,$$

and the dissociation of bases to the equation

$$BH^+ + HS \rightleftarrows B + H_2S^+.$$

Shkodin and Sadovnichaya[77–81] measured K_{diss} for various bases by the electrical conductivity method and showed that anhydrous acetic acid increases the strength of all weak bases relative to water; one group of bases, with strengths close to that of the alkali metal acetates ($pK(H_2O) \simeq 4.3$–4.6) are levelled, and the other group of weaker bases ($pK(H_2O) > 5$) are differentiated. The bases are much weaker in propionic acid than in acetic acid. The strengths of bases which are very weak in water are increased in propionic acid, however. The differentiation of bases in propionic acid is much less than that in acetic acid. This confirms that in aliphatic acids the strength of electrolytes is inversely proportional to the magnitude of the dielectric constant.

Electrical conductivity data have been used to calculate pK for perchloric (2.85), hydrochloric (5.46), sulphuric (4.5), and hydrobromic (4.10) acids. The behaviour of acids and bases in a medium of acetic acid and its mixtures with other solvents has also been studied by a number of other workers[82–92].

Dulova and coworkers[93–99] studied the differentiating action of alcohols, esters, glycols, and ethylene chloride, and indicator and spectrophotometric methods were used to determine K_{diss} for a number of acids. The authors cited consider that the differentiating action of hydroxyl-containing solvents is related chiefly to the size of the radical and its three-dimensional structure.

A large number of determinations of K_{diss} for acids in methanol and ethanol and their mixtures with water have been made[100–122]. Sano[123,124] determined K_{diss} for a large number of acids in methanol and established that the values of K_{diss} decrease by a factor of approximately 80 000 relative to water. Tabagua[125–128] determined K_{diss} for a number of organic acids in methanol and ethanol and their mixtures with water, and showed that the values of K_{diss} for the acids decrease with increase in the concentration of alcohol in the mixture, and that at low concentrations of the alcohol the strength of the acids depends only on the dielectric constant of the medium. With increase in the alcohol concentration, the influence of the nature of the acids is observed, the changes in K_{diss} for weak acids being less marked than those for strong acids, due to the difference in the solvation energies of the ions and molecules.

Aleksandrov and coworkers[129] determined pK for a number of acids in methanol and its mixtures with water and benzene. Izmailov and Spivak[130] determined pK_{rel} for a number of acids in aprotic solvents. Data have been published on the dissociation of electrolytes of the acid–base type in formic acid[131,132], formamide[133–136], dimethylformamide[137], mixtures of dioxan and water[138–145], and other solvents[146–159]. A simple and rapid method for estimating solvent influence on the strengths of acids and bases involves the determination of the relative acidic or basic strength of electrolytes from the half-neutralisation potentials ($E^{1/2}$). When 50% of the weak acid or weak base is neutralised, pH = pK, so that $E^{1/2}$ can characterise the relative strength of electrolytes in non-aqueous solutions. Values of $E^{1/2}$ for derivatives of benzoic acid have been determined in pyridine, acetonitrile, methyl isobutyl ketone, 2-nitropropane, *o*-nitrotoluene, nitrobenzene, dimethylformamide, chlorobenzene, and bromobenzene[160]. A linear dependence of $E^{1/2}$ on $pK(H_2O)$ was obtained for all solvents. The solvents studied exhibit a strong differentiating action, and in these solvents it is possible to analyse a mixture of two acids with $\Delta pK(H_2O) = 0.94$. Determination of $E^{1/2}$ for derivatives of phenylurea in n-butylamine showed that the phenyl- and chlorophenyl-derivatives exhibit more strongly acidic properties in n-butylamine than urea and its alkyl derivatives[161].

The dependence of the half-neutralisation potentials on the values of $pK(H_2O)$ for electrolytes gives information on the differentiating action of non-aqueous solvents. The differentiating action of solvents is characterised by the slope of the straight line, which is determined by the number of mV per unit of $pK(H_2O)$. The steeper the straight line, the stronger the differentiating action shown by the solvent with respect to a given group of electrolytes. Comparison of the differentiating properties of nine non-aqueous solvents by measurement of the values of $E^{1/2}$ for the nitro- and halogeno-derivatives of phenol was carried out by Crabb and Critchfield[162]. They arranged the solvents studied in the following order of decreasing differentiating action: t-butyl alcohol > pyridine > tetrahydrofuran > > isopropyl alcohol > dimethyl sulphoxide > acetone > > acetonitrile > ethylene glycol > water. For derivatives of picric acid, the values of $E^{1/2}$, measured in acetone, were found to be linearly dependent on $pK(H_2O)$.[163]

We made a quantitative estimate of the differentiating action of alcohols, ketones, hydrocarbons, and mixed solvents with respect to monobasic aromatic acids, dicarboxy-

lic acids, and organic bases[1]. The observed dependence of $E^{1/2}$ on p$K(H_2O)$ showed that the strongest differentiating action with respect to monobasic aromatic acids is shown by ethyl methyl ketone, with respect to dicarboxylic acids by isopropyl alcohol and ethyl methyl ketone, and with respect to organic bases by mixed solvents: benzene or chloroform with ethyl methyl ketone or acetonitrile.

Measurement of the relative basic strength of phosphines in nitromethane showed that in the phosphine series the strongest electrolytes are tertiary phosphines, whereas in the amine series the strongest electrolytes are primary amines[164]. A linear dependence of $E^{1/2}$ on p$K(H_2O)$ was observed for derivatives of phenothiazine and sympathomimetic amines in acetone, acetonitrile, acetic acid, isopropyl alcohol, and nitromethane[165]. A common linear relationship was observed for all bases in a medium of acetic acid. In acetone, acetonitrile, and nitromethane, which exhibit effective differentiating properties, a characteristic relationship was observed for each group of bases. Isopropyl alcohol has a levelling action on amines. The dependence of $E^{1/2}$ on p$K(H_2O)$ makes it possible to estimate p$K(H_2O)$ for acids and bases for which these values have not previously been determined experimentally[1,89].

An important characteristic of a solvent, determining its scale of acidity in titrations, is the autoprotolysis constant (K_S). A number of authors have determined the values of K_S for acetic acid[166], acetic anhydride[167], ethanol, n-propanol, isopropyl alcohol[168,169], monoethanolamine[170,171], ethylene glycol and propylene glycol[76], dimethylformamide[172], dimethyl sulphoxide[74], and acetonitrile[66].

A number of studies have been devoted to the determination and use of Lewis acids in titrations in non-aqueous solvents[173-186].

Thus a survey of the theory of the analytical chemistry of non-aqueous solutions shows an increase in the number of papers devoted to the study of acid–base reactions in non-aqueous solutions. These studies, however, do not include such solvents as ketones, nitro-derivatives of hydrocarbons, and various mixed solvents, which have found extensive practical application as differentiating solvents. It is necessary to extend studies on the quantitative estimation of the influence of solvents on the dissociation of electrolytes, together with measurements of K_{diss}. Estimation of the relative strengths of electrolytes from half-neutralisation potentials gives extremely valuable information for the development of new methods for the analysis of mixtures of electrolytes. It is also necessary to extend studies on the determination of the autoprotolysis constants of non-aqueous solvents.

III. PRINCIPLES OF THE ACID–BASE TITRATION OF NON-AQUEOUS SOLUTIONS

In the selection of a method for the titration of acids, bases, and salts in non-aqueous solutions, particularly in the selection of a method for the differentiated analysis of mixtures of electrolytes, it is necessary to have a theoretical basis for the selection of the medium for the titration (solvent), the standard solution of the reagent (titrant), and the method for determining the equivalence point. Only a coordinated selection of these titration conditions permits a more rational and effective method for the differentiated determination of the individual compounds in multi-component mixtures[5,187].

1. Solvents used for Acid–Base Titration

In the determination of compounds which exhibit acid–base properties in non-aqueous solutions, various classes of organic solvent can be used as media for the titration: organic acids (formic, acetic, etc.), amines and heterocyclic bases (butylamine, ethylenediamine, pyridine, piperidine, etc.), glycols, alcohols (methaol, ethanol, propanol, isopropyl alcohol, etc.), ketones (acetone, ethyl methyl ketone, methyl isobutyl ketone, etc.), nitriles (acetonitrile), amides (dimethylformamide, formamide, etc.), esters and ethers (diethyl ether, dioxan, esters of acetic acid, etc.), hydrocarbons and their halogeno- and nitro-derivatives (benzene, chloroform, carbon tetrachloride, nitromethane, nitrobenzene, etc.), and various mixtures of these solvents.

In the last 4–5 years, a number of new organic solvents exhibiting valuable properties have been introduced into analytical practice, for example t-butyl alcohol, dimethyl sulphoxide, tetramethylguanidine, and various mixed solvents (mixtures of aprotic solvents with ketones and nitriles). t-Butyl alcohol[162,188-190] has proved to one of the best solvents for the titration of very weak acids; it has a lower acidity than the lower alcohols, and its acid solutions have a high stability. The differentiated titration of mixtures of various inorganic and organic acids can be carried out in t-butyl alcohol.

Dimethyl sulphoxide is an effective solvent for many organic compounds[162 191-194], and unlike a number of solvents it is not toxic and has no odour. The presence of the semipolar bond between sulphur and oxygen in dimethyl sulphoxide gives it protophilic character and leads to an increase in the strength of weak organic acids dissolved in it. This solvent is characterised by a fairly large acidity scale (pK = 18) and high dielectric constant (40), and exhibits the same differentiating properties as dimethylformamide, but it does not contain acidic and basic impurities, and therefore does not require preliminary purification. Strong and weak organic acids can be titrated in dimethyl sulphoxide, and the titration of phenols is particularly effective (phenol, guaiacol, eugenol, etc.)[191]. Williams and coworkers[195,196] studied the properties of tetramethylguanidine and suggested its use as solvent without preliminary purification for the potentiometric and visual titration of phenols.

Solvents used for the titration of bases. Compounds which exhibit basic properties in non-aqueous solutions can be determined by titration in protolytic (protogenic and amphiprotic), aprotic, and various mixed solvents.

Among the protogenic solvents for the determination of weak bases, extensive use is still made of anhydrous acetic acid[197-243], first proposed for this purpose by Conant and Hall[8]. In acetic acid, the strengths of bases with p$K(H_2O)$ = 10–14 increase, but do not reach the value 4.3, and the bases exhibit their own individual properties. Thus it has been possible to carry out the differentiated titration of two-, three-, and four-component mixtures of bases in acetic acid[244-247].

Bases can be titrated in mixtures of acetic acid and acetic anhydride[248,249], or in pure acetic anhydride[220,250-256]. Acetic anhydride has found application as differentiating solvent for the differentiated titration of amines, sulphoxides, and amides[257]. The improvement in the conditions for the titration of bases in acetic anhydride compared with acetic acid may be attributed to its higher dielectric constant.

Unlike acetic acid, acetic anhydride differentiates bases over the entire scale of acidity[89]. The addition of hydrocarbons, their halogeno-derivatives, dioxan, or ketones to acetic acid or to acetic anhydride makes the end point of the titration sharper, due to a decrease in the ionic product of the medium[205,222,223,249,251,258-261]. The use of mixtures of formic acid with acetic anhydride[262] and propionic acid[263] as media for the titration of bases has also been described.

Of the amphiprotic solvents for the titration of bases, most extensive use has been made of alcohols, ketones, and nitriles. In the titration of bases in alcohols, the end points of the titrations are insufficiently sharp. Alcohols do not exhibit strong differentiating properties with respect to bases[244,264-267]. For the differentiated titration of mixtures of bases, extensive use is made of ketones, particularly acetone[208,233,268-270], ethyl methyl ketone[244,267,271-275], and isobutyl methyl ketone[268,276]. The differentiated titration of two-, three-, and four-component mixtures of strong and weak bases has been carried out in ethyl methyl ketone[267,271,275,277].

Acetonitrile has found extensive application as medium for the titration of bases; this solvent is used for the titration of individual bases[208,224] and for the differentiated titration of their mixtures[233,278]. The end points of the titration of bases in ketones and acetonitrile are sharper than those in protogenic solvents and alcohols, apparently due to the low values of the ionic products of these solvents.

Strong differentiating properties with respect to weak bases are also shown by nitrobenzene and nitromethane. In nitrobenzene it is possible to carry out the differentiated titration of various mixtures of bases whose $pK(H_2O)$ values differ by unity, and in nitromethane it is possible to carry out the differentiated titration of mixtures of components whose pK values differ by 0.3 unit[278]. The conditions of the titration in nitrobenzene are improved considerably when acidic solvents are added[279-281]. Study of the influence of the addition of organic acids on the titration of a mixture of diethylaniline and *m*-nitroaniline in nitrobenzene showed that the addition of 11.1% of a mixture of acetic and formic acids (1 : 3) increases the differentiating properties of the nitrobenzene[279].

Aprotic solvents used for the titration of bases include hydrocarbons (benzene, chlorobenzene, chloroform, dichloroethane, etc.)[268,282-292]. In titrations in aprotic solvents of low dielectric constant values (2–5), alcohols, ketones, and nitriles are added in proportions from 10 : 1 to 4 : 1, to increase the dielectric constant of the solution[267,293-296]. The end point of the titration is less sharp when alcohols are added than when ketones and nitriles are added[267]. Many diamines can be titrated as diacid bases in mixed solvents consisting of mixtures of chloroform or benzene with ethyl methyl ketone or acetonitrile (4 : 1)[267,294-296]. The production of the second inflection in the titration is due to the precipitation of the normal salt of the diamine and perchloric acid, sparingly soluble in aprotic solvents.

Bases can also be titrated in mixed solvents such as *m*-cresol + acetonitrile (1 : 1)[297], dioxan + chloroform[234], and dioxan + acetic acid + nitromethane (75 : 5 : 20)[298]. The titration of compounds with basic properties can also be carried out in acetonitrile[299,300], dimethyl sulphoxide[194], pyridine[301], ethyl acetate[302], and methylcellosolve[303]. New methods have been described for the purification and control of the purity of various non-aqueous solvents[304-309].

Solvents used for the titration of acids. The determination of compounds which exhibit acidic properties in non-aqueous solutions can be carried out by titration in aprotic and protolytic (protophilic, protogenic, and amphiprotic) solvents. The most widely used aprotic solvents are chloroform[310,311] and benzene[312,314]. In aprotic solvents, because of the low dielectric constant, it is possible to titrate not only individual strong, weak, and very weak acids, but also mixtures of acids differentially. Alcohols[190,315,316], ketones[317], and acetonitrile[318,319] in proportions from 3 : 1 to 10 : 1 are often used to increase the dielectric constant of the solution.

Amphiprotic solvents used for the same purpose include various alcohols, ketones, nitriles, esters, and other solvents. Alcohols, as the most readily available and inexpensive solvents, are widely used for the titration of strong and weak acids. Thus methanol[311,320,321], ethanol[311,321-325], propanol and isopropyl alcohol[162,311,326-328], butanol, isobutyl alcohol, and t-butyl alcohol[162,188,189,311], and mixtures of alcohols with one another[329] or with benzene[190,315,316], dioxan[324], pyridine[330], and ketones[331,332] can be used for the titration of higher aliphatic acids, dicarboxylic and inorganic acids, nitrophenols, and other compounds. Co-solvents are used to increase the solvent power of the alcohol and to obtain sharper end points in the titration. Mixtures of inorganic acids are readily titrated in alcohols, particularly lower alcohols (methanol and ethanol)[1]. Mixtures of carboxylic acids with phenols and mixtures of dicarboxylic acids are titrated in less acidic alcohols, such as isopropyl alcohol, isobutyl alcohol, and t-butyl alcohol[188,318,333].

Ketones are the best solvents for the titration of acids. Inorganic and organic acids, phenols, and a number of other substances can be titrated in acetone[193,310,311,332-348], ethyl methyl ketone[310-313,332-334,347,349-354], butyl methyl ketone and isobutyl methyl ketone[193,347] and mixtures of ketones with water[350,355,356], alcohol[331,332], dimethylformamide[350], and acetonitrile[318]. Ketones, because of their amphoteric character, low dielectric constants, and low K_S values, are excellent differentiating solvents. Ketones can be used successfully not only for the titration of two-, three-, and four-component mixtures of various acids, but also for the titration of five- and six-component mixtures of strong, weak, and very weak acids[311,333,353,354]. Nitriles[162,193,310-311,333,347,357] and esters of acetic acid[310,311,333] are effective differentiating solvents. Mineral and organic acids and their mixtures can be readily titrated in these media. High-frequency and potentiometric methods have been used for the titration of acids and their mixtures in dioxan and its mixtures with water[193,358-361].

Protophilic solvents are used as media for the titration of very weak acids. Phenols and weak carboxylic acids can be successfully titrated in ethylenediamine[193,362-366] and butylamine[161,316,363]. The titration of strong, weak, and very weak acids is carried out in pyridine[162,193,314,330,336,337,347,358,363,365,367-369] and piperidine[193,363]. Because of its low dielectric constant (12.5), pyridine is an effective differentiating solvent for the titration of mixtures of strong, weak, and very weak acids. In addition to amines, various amides can be used as media for the titration of acids. Various derivatives of phenols, carboxylic acids, and mineral acids can be effectively titrated in dimethylformamide[172,193,314,332,347,362,363,365,370-374], but the presence of water greatly distorts the results of the titration. Dimethylformamide exhibits weak basic properties, so that it can be used for the differentiated titration of mixtures of acids[347,371,375].

Protogenic solvents used for the titration of mineral acids include anhydrous formic and acetic acids[376-379]. In acetic acid, which has a low dielectric constant, it is possible to titrate differentially mixtures of inorganic acids

with one another and with organic acids[376-378]. Glycol solvents (mixtures of glycols with alcohols, ketones, hydrocarbons, and their halogeno-derivatives)[162,380-382] exhibit weak acidic properties. Mineral and organic acids and their mixtures can be determined in glycol solvents. Tetrahydrofuran[162,193,383], methylene chloride[382,384,386], acetyl chloride[386], and derivatives of aryl amides[387] are used successfully as solvents for the determination of individual carboxylic acids and their mixtures.

2. Titrants used for the Titration of Non-aqueous Solutions

Acidic titrants. The most widely used titrants for the titration of non-aqueous solutions of compounds of basic character are solutions of perchloric acid in acetic acid[198-200, 202-204,225-228,230-234,243-246], dioxan[204,210,241,251,255,257,259,266, 278,286-288,298,388], ethyl methyl ketone[267,273-275,277,294], mixtures of acetic acid and acetic anhydride (1 : 1)[389,390], ethanol[391], methylcellosolve[303], mixtures of acetonitrile and *m*-cresol[297], etc. The differentiated titration of mixtures of diamines, which can be titrated in a medium of benzene or chloroform with acetonitrile (4:1), has been carried out using solutions of $HClO_4$ in the same solvents[267,296]. The use of these titrants gives titration curves with sharper inflections than those obtained when a solution of $HClO_4$ in ethyl methyl ketone is used.

Various workers have used solutions of HCl in isopropyl alcohol[265] and isobutyl methyl ketone[276] for the determination of bases. Bases have been titrated with sulphonic acids, for example *p*-toluene-sulphonic acid in a mixture of ethanol and isopropyl alcohol (1 : 1) or in a mixture of chloroform with 1% ethanol, dioxan, and other solvents[268, 284,285,290,392], and also fluorosulphonic acid[393-396], trifluoromethyl sulphonic acid[397], and chlorosulphonic acid[398]. In a medium of acetic acid, fluorosulphonic acid is stronger than perchloric acid, and the stability of its solutions and the ease of preparation give grounds for considering it a better titrant than perchloric acid[393]. Study of the behaviour of standard methanol, ethanol, and mixed (glycol–methanol, glycol–isopropyl alcohol, and acetone–chloroform) solutions of fluorosulphonic acid[396] has shown that best results are achieved in the titration of bases with a methanol solution of fluorosulphonic acid.

Bases are also titrated with picric acid[399], trichloroacetic acid[392], acetic anhydride perchlorate[400], and an acetic acid solution of sulphur trioxide[401].

Solutions of $HClO_4$ in glacial acetic acid are standardised using tri(hydroxymethyl)methylamine[402]. An advantage of this reagent compared with potassium hydrogen phthalate is that it dissolves in acetic acid without heating, but it is a weaker base than potassium hydrogen phthalate and diphenylguanidine. $HClO_4$ solutions are also standardised using hexamethylenetetramine and pyramidone[241], and *p*-toluenesulphonic acid is standardised using strychnine[284].

Basic titrants. Methanol[320,321,343,345], ethanol[323, 331,335,340,341,375,384], isopropyl alcohol[380], and mixed[403] standard solutions of potassium hydroxide and sodium hydroxide are most widely used as titrants for the titration of compounds of acidic character in non-aqueous media. Extensive use is also made of alcoholic and mixed solutions of alkali metal alkoxides—sodium, potassium, and lithium methoxides[314,316,324,332,337,362,365,371,404] and isopropoxides[326]—and sodium aminoethoxide in ethylenediamine[366]. Organic bases used as titrants include triethanolamine[382], cyclohexylamine[383], diphenylguanidine[360,361], pyridine[377], and piperidine[381].

The strongest basic titrants in organic solvents are the quaternary ammonium hydroxides—tetramethylammonium[322,328], tetraethylammonium[310,312,334,344,347,349-354], and tetrabutylammonium[161,188-190,193,336,352,367,369,387,405,406] hydroxides—and various other derivatives of quaternary ammonium hydroxides, for example hexadecyltrimethylammonium[196], dodecyltrimethylammonium[327], and tributylethylammonium[368] hydroxides. Study of the properties of various derivatives of quaternary ammonium hydroxides, the factors influencing the stability of non-aqueous solutions of quaternary ammonium titrants[407], and the influence of the structure of the quaternary ammonium cation on the titration of various acids[408] by Harlow established that tetramethylammonium, tetrabutylammonium, and tetraethylammonium hydroxides are the most stable titrants, and trimethylbenzylammonium hydroxide the least stable.

In connection with the continuous extension of the range of application of quaternary ammonium bases, several new methods have been proposed for preparing solutions of these compounds. Harlow and Wyld[409] proposed a rapid and convenient method for preparing solutions of quaternary ammonium hydroxides, based on the reaction between potassium hydroxide and the quaternary ammonium chlorides in isopropyl alcohol. Gur'ev[410] used mercuric oxide in place of silver oxide for the preparation of solutions of tetramethylammonium and tetraethylammonium hydroxides. Kreshkov and coworkers[411] developed a very convenient method for preparing solutions of quaternary ammonium hydroxides by ion exchange; the titrants obtained by this method are highly pure.

3. Methods for Determining the Equivalence Point

Visual and physicochemical methods for determining the equivalence point are widely used in titrations in non-aqueous solutions. The potentiometric method[26,412-420] is the most widely used method of titration in non-aqueous solutions. The indicator electrodes used in the potentiometric titration include the hydrogen[413], glass[413,416], graphite[415], platinum[316], platinum oxide[417], antimony[418,421], and silver chloride[419] electrodes. A comparative study has been made of various electrode systems for titrations in acetic acid and dimethylformamide[420]. Comparison of platinum oxide and glass electrodes for acid–base titration in alcohols, ketones, acetonitrile, dimethylformamide, and mixed solvents[417] showed that the inflections observed are greater for the platinum oxide electrode than for the glass electrode.

A method for potentiometric titration at constant current strength[297], a dead-stop method[389], and a chronopotentiometric method which does not involve the use of a burette[422] have been described. Conductometric[25,423], high-frequency[25,27,87,424], and spectrophotometric[245,425] methods are widely used for titrations in non-aqueous media. Chronoconductometric titration in aqueous alcohol[426], thermometric titration in acetonitrile[357], acetic acid[427], and acetone[428], and coulometric[429], luminometric[430], dielectrometric[292], and amperometric[431,432] titrations have also been described. A number of papers have been devoted to the study of acid–base indicators for titrations in non-aqueous solutions[433-444].

IV. TITRATION IN NON-AQUEOUS SOLUTIONS OF BASES

1. Titration of Individual Compounds

Non-aqueous solvents can be used for the determination of the great majority of individual compounds which exhibit basic properties in non-aqueous solutions[1-5]. These compounds include aliphatic, aromatic, and heterocyclic amines[199,206,208,220-222,241,261,281,284,287,290,297,390,445], diamines[233,267,294-296,446], amides[230,250,251], imides[207], amine oxides, phosphine oxides, and aromatic *N*-oxides[255,447], compounds containing the mercapto-group[448], sulphonamides[449], pyrazolones[225,450], vitamins[204], antihistamines[205,223], antibiotics[259], alkaloids[226,228,229,234,249,285,292,293], and other compounds[198,200,224,232,234,240,256,258,286,288,289,451,452].

Shkodin and Karkuzaki[421] titrated very weak bases ($pK(H_2O) > 12$) such as urea, thiourea, caffeine, theobromine, etc. in a medium of absolute anhydrous acetic acid and its mixtures with acetic anhydride. They showed that the titration of these compounds becomes impossible when even small quantities of water are present.

Potentiometric titration at constant current strength in a medium of *m*-cresol–acetonitrile (1 : 1) has been used for the titration of monoacid and diacid aliphatic and aromatic amines and their nitro-derivatives[297], and methods have been described for the micro-titration of weak bases of $pK(H_2O) = 11$ (piperidine, benzidine, diphenylguanidine, pyridine, quinine, strychnine, etc.) in a medium of chloroform[284], dichloromethane, and chlorobenzene[290] with a solution of *p*-toluenesulphonic acid in a chloroform + ethanol medium.

In a 2 M benzene solution of nitrobenzene, modified by means of organic acids such as formic, acetic, or trichloroacetic, bases which are very weak in water (aniline, *p*- and *m*-chloroanilines, diethylaniline, *o*-, *m*-, and *p*-nitroanilines, and diphenylamine) behave as strong, intermediate, or weak bases, depending on the acid added[281].

Study of the possibility of titrating the amides of acids and various other weakly basic compounds (acetanilide and its derivatives) in a medium of acetic anhydride and its mixtures with acetone and chloroform (1 : 1)[251] has shown that the presence of electron-donating groups increases the basic strength and gives a sharper inflection in the titration. Alkaloids can be titrated in a mixture of acetic acid with acetic anhydride and other solvents. Mercuric acetate is added to the solvent to give a sharper inflection in the titration of alkaloids[249]. The conductometric titration of aldehydes and ketones as bases in a medium of H_2SO_4 with tetra(hydrogen sulphato)boric acid is possible[453].

When the compounds to be determined do not exhibit basic properties in non-aqueous solutions, they are treated beforehand with various reagents which convert them into basic compounds[233,237,252]. Thus carbonyl compounds react with various salts of hydroxylamine or hydrazine derivatives to form oximes or phenylhydrazones, which exhibit the properties of weak bases in a medium of acetic anhydride[237,252]. Diamines are oxidised with chloranil, and diimines are reduced with hydroquinone to form basic compounds which can be determined by titration[233]. A method has been described for the determination of acetic anhydride in acetylating mixtures, based on its reaction with aniline, excess of which is titrated in a medium of diethylene glycol[454].

2. Titration of Primary, Secondary, and Tertiary Amines

Mixtures of primary, secondary, and tertiary amines are still analysed by acetylation of the primary and secondary amines with acetic anhydride[201,235,303] and binding of the primary amines with salicylaldehyde[210,264,269,298,455] or phthalic anhydride[202,456].

Strepikheev and coworkers[269] determined primary, secondary, and tertiary amino-groups in polynuclear polyamines in an acetone + acetic acid medium, using salicylaldehyde to bind the primary amines. The authors cited[269] showed that phthalic anhydride combines not only with primary but also with secondary amino-groups. Other workers also established that when solutions of primary and secondary amines with a high concentration of secondary amines are boiled for a prolonged period, the phthalic anhydride reacts to form neutral compounds. In order to hydrolyse the products, the reaction is carried out in 98% acetic acid or in the presence of perchloric acid[202]. Pyromellitic anhydride in a medium of dimethyl sulphoxide has been used to bind primary and secondary amines[194]. The reaction is more rapid when this acetylating reagent is used. A method has been proposed for the determination of primary, secondary, and tertiary amines, based on the difference in the rates of formation of thiourea by the reaction of the amines with isothiocyanates in acetic acid. Samples are removed at measured time intervals and the unreacted amine is titrated[236].

A method has been developed for the determination of tertiary amines in the presence of primary and secondary amines, based on the binding of the primary and secondary amines in the form of thiourea derivatives by the reaction with phenyl isothiocyanate followed by titration of the tertiary amine in isobutyl methyl ketone. This method is not applicable, however, when the secondary amines are N-secondary alkylanilines or ortho-substituted *N*-alkylanilines, where there is steric hindrance to the reaction with phenyl isothiocyanate[276]. In the determination of amine oxides and tertiary amines in the presence of one another, the latter are bound with alkyl halides, which convert them into neutral quaternary ammonium salts[265].

In the quantitative analysis of mixtures of higher aliphatic amines in the products of the hydrogenation of the nitriles of the higher carboxylic acids, the primary amines are bound with acetylacetone and the secondary amines are titrated acidimetrically; the sum of the primary and secondary amines is determined by titration after the amines have been converted into the dithiocarbamic acids[457]. Chloroform and methanol are used as solvents.

To determine piperazine, hydroxyethylpiperazine, and dihydroxyethylpiperazine in the presence of one another, the first two components of the mixture are separated from the dihydroxyethylpiperazine by precipitation with carbon disulphide from a solution of the mixture in chloroform[299], and the dihydroxyethylpiperazine is titrated. The precipitate is dissolved in acetonitrile, and the hydroxyethylpiperazine is determined by titration, after acetylation.

In the titration of amines by a solution of *p*-toluenesulphonic acid using the dielectrometric method, it was found that the titration curves for primary and secondary amines differ from those for tertiary amines[392]. This feature was used for the differentiated titration of a mixture of primary, secondary, and tertiary amines. The determination of tertiary amines in the presence of n-octanol[227] and of primary aliphatic amines in the presence of secondary and tertiary amines by photometric titration with a solution of

2-ethylhexanal[458], and also a method for determining primary and secondary diamines in a medium of ketones[459], have been described.

3. The Differentiated Titration of Mixtures of Bases

Methods which make possible the separate determination of each component in complex mixtures of bases with very similar $pK(H_2O)$ values are of particular interest. Two-, three-, and four-component mixtures of bases have been titrated, for example tetraethylammonium hydroxide + n-butylamine + diethylaniline, piperidine + 1,10-phenanthroline, piperidine + benzimidazole + methylbenzimidazole, etc. in ethyl methyl ketone[271,275-277], quinoline + aniline in acetonitrile, nitrobenzene, and nitromethane[278], diethylaniline + aniline or *m*-nitroaniline in a benzene solution of nitrobenzene, modified with acetic and formic acids[279-281], mixtures of amines, sulphoxides, and amides, and mixtures of pharmaceutical products in acetic anhydride[89,257], and mixtures of diamines in mixed solvents consisting of an aprotic solvent and an amphiprotic solvent[267,294-296]. Mixtures of weak bases are also titrated in acetic acid[233,244-247] and other solvents[260,262].

A method has been developed for the analysis of two-, three-, and four-component mixtures of aliphatic, aromatic, or heterocyclic amines, sulphoxides, and amides of carboxylic acids by titration in a medium of acetic anhydride. In the titration of four-component mixtures the first inflection in the titration corresponds to the neutralisation of the aliphatic amine, the second to the aromatic amine, the third to the sulphoxides, and the fourth to the amides[257]. The differentiated titration of a mixture of weak bases with $pK(H_2O) = 10-14$ (amidopyrine + antipyrine, caffeine + phenacetin, etc.) has been carried out in acetic anhydride[89].

We developed a method for the analysis of two- and three-component mixtures of diamines and of monoamines with diamines in mixed solvents—chloroform or benzene with ethyl methyl ketone or acetonitrile (4 : 1)[267,294-296]. The method can be used to analyse two-component mixtures of the *p*- and *o*- and *p*- and *m*-isomers of phenylenediamine, hexamethylenediamine + *N*-isopropyl-*N'*-phenyl-*p*-phenylenediamine + *p*-phenylenediamine, tyramine + *o*- + *m*-phenylenediamines, and a number of other mixtures.

Kreshkov and Vasil'ev[244,245] developed a method for the spectrophotometric titration of three- and four-component mixtures of weak and very weak bases ($pK(H_2O) > 13.5$) in a medium of acetic acid. Titration of *o*-nitroaniline + 4-methyl-2-nitroaniline + *p*-bromoaniline, *o*-nitroaniline + *m*-nitroaniline + 4-methyl-2-nitroaniline + *p*-nitrosodimethylaniline, and other mixtures showed that the spectrophotometric method can be used to analyse mixtures with a difference in $pK(H_2O)$ values of 0.8–1.5. Mixtures of the isomers of nitro-*o*-toluidine and nitro-*p*-toluidine, nitroaniline and nitro-*o*-anisidine have been titrated in a medium of acetic acid and its mixtures with acetone (1 : 4)[260]. Two-, three-, and four-component mixtures of weak bases with $pK(H_2O) = 10-13.81$ have been titrated in acetic acid by a conductometric method[78,247].

Lorenz and Parks[233] showed that it is possible to analyse mixtures of *NN'*-diaryl-*p*-phenylenediamines and *NN'*-diaryl-*p*-phenylenediimines in a medium of acetic acid. Chloranil was added when the mixture contained an excess of *NN'*-diaryl-*p*-phenylenediamine, and hydroquinone was added when the mixture contained an excess of *NN'*-diaryl-*p*-phenylenediimine. The analysis of mixtures of acylamidines, such as *N*-benzoylbenzamidine, *N*-thiobenzoylbenzamidine, dibenzamide, etc.[460]

V. TITRATION IN NON-AQUEOUS SOLUTIONS OF ACIDS

All organic and inorganic compounds which exhibit acidic properties can be determined in non-aqueous solvents. These compounds include phenols, mono-, di-, and tri-basic aliphatic and aromatic carboxylic acids, and also mono- and di-basic inorganic acids, acid anhydrides and chlorides, enols, imides, amides, and other compounds such as esters and alcohols[1-5].

1. Titration of Compounds Exhibiting Weakly Acidic Properties

Enols and their derivatives are successfully titrated by the potentiometric method in tetramethylguanidine[195,196], dimethyl sulphoxide[74,75,191], t-butyl alcohol[162,188,189,405], and *NN*-dimethylarylamides[387]. A number of papers have been devoted to the titration of phenols in ethylenediamine[362,364], dimethylformamide[172,362,370,372], acetone[347], ethyl methyl ketone[347,349], alcohols[188,328], and other solvents[331,347]. Phenols, cresols, and their derivatives are determined by high-frequency, conductometric[190,339,345,363], and spectrophotometric[329] methods. Polycyclic phenolic compounds are titrated in a medium of pyridine and acetone[337], substituted phenylureas in a medium of butylamine[161], and acylamidines in a medium of pyridine[367]. The differentiated titration of phenol and its nitro-derivatives has been carried out in ethyl methyl ketone[333] and in other solvents[347] by a potentiometric method, and mixtures of isomers of cresol and xylenone[339,345] have been titrated in a medium of acetone by a high-frequency method.

A number of ethers and esters have been titrated in non-aqueous solvents. Pathochornik and coworkers[324] established that in an ethanol + dioxan mixture, esters of *p*-nitrophenol and esters of thiophenol behave as acids and can be titrated with CH_3ONa solution. A method has been developed for the analysis of diethyl malonate and its derivatives, based on enolisation and subsequent titration of the resulting acid in a medium of ethylenediamine, dimethylformamide, or pyridine[365]. The titration of *p*-nitrophenylhydrazones of aldehydes, ketones, and hydrocarbons as acids in a medium of pyridine with tetrabutylammonium hydroxide solution has been studied. The nitrophenylhydrazones of dicarbonyl compounds can be titrated as dibasic acids[369].

Harlow[461] published an interesting study of the influence of potassium ions on the shape of the potentiometric titration curves for weak acids in pyridine. The presence of 10% potassium ions in the solution being titrated decreases considerably the magnitude of the potential jump at the equivalence point.

2. Titration of Monocarboxylic Acids.

Aliphatic and aromatic carboxylic acids and their hydroxo-, halogeno-, nitro-, and other derivatives can be successfully titrated in non-aqueous solvents[366,406,462]. Of particular importance is the titration of the water-insoluble

higher aliphatic acids[315,342,463], benzoic acid and its derivatives, α- and β-naphthalenecarboxylic acids and their derivatives, and a number of other acids[335,352,361]. All carboxylic acids can be titrated with sufficient accuracy in alcohols[342], ketones[193,332,338,342], chloroform[333], ethylenediamine[366], mixtures of dioxan and water[359,360], and a number of other solvents[315].

In differentiating solvents it is possible to titrate mixtures of benzoic and salicylic acids, and also the *o*- and *m*-, and *o*- and *p*-isomers of hydroxybenzoic acid[333]. The differentiated titration of mixtures of naphthalenecarboxylic acids and their hydroxy-derivatives[352] and naphthols[331] can be carried out in a medium of ketones. The differentiated titration of four-component mixtures of carboxylic acids and phenols can be carried out in t-butyl alcohol[188]. Vasil'ev and Tumovkii[212] have titrated two-component mixtures of amphoteric compounds: 1-naphthylamine + 4-nitro-1-naphthylamine, 4-aminophenol + 2-nitro-4-aminophenol, etc., as acids with sodium methoxide in a medium of acetone + methanol (2 : 1) and as bases with perchloric acid in a medium of acetic acid.

3. Titration of Dicarboxylic Acids

In connection with the vast technical importance of dicarboxylic acids, which are widely used in the production of plastics, paints, synthetic fibres, and other macromolecular products, a large number of papers have recently been published on the titration of dicarboxylic acids and their mixtures in non-aqueous solvents[318,335,349,350,464,465]. Non-aqueous solvents can be used as media for the titration of unsaturated dicarboxylic acids (maleic, fumaric, itaconic, and citraconic), saturated dicarboxylic acids (from oxalic to sebacic), and aromatic dicarboxylic acids (the isomers of phthalic acid), and also for various mixtures of dicarboxylic acids, including mixtures of isomers (maleic and fumaric, itaconic and citraconic), and mixtures of acids of the same homologous series (oxalic and succinic, sebacic and adipic). Dicarboxylic acids and their mixtures can be titrated by potentiometric[318] and high-frequency[374,466-468] methods.

The differentiated titration of mixtures of dicarboxylic acids can be carried out in isopropyl alcohol, acetone, ethyl methyl ketone, acetonitrile, and mixtures of aprotic solvents with alcohols, ketones, and acetonitrile[469-472]. For example, a mixture of maleic and phthalic acids can be titrated in ethyl methyl ketone, a mixture of maleic and fumaric acids can be titrated in acetonitrile, and a mixture of sebacic and azelaic acids can be titrated in a chloroform + acetonitrile (4 : 1) mixture.

We developed methods for the quantitative differentiated titration of aromatic dicarboxylic acids, for example mixtures of iso- and terephthalic acids[469], mixtures of phthalic, isophthalic, and terephthalic acids, etc.[470] For the determination of all three isomers, a method was proposed based on two parallel titrations of the mixtures of acids in ethyl methyl ketone, one specimen being titrated with $(C_2H_5)_4NOH$ solution, and the second with KOH solution. The curves obtained were used to calculate the concentration of each isomer in the mixture[351,470]. The method can also be used for the analysis of a three-component mixture of maleic, fumaric, and phthalic acids[318].

Methods have been developed for the titration of mixtures of dicarboxylic acids with one another[335] and with inorganic acids such as hydrochloric[318], nitric, etc.[343] in a medium of ketones.

4. Titration of Inorganic Acids

Non-aqueous solvents can be used as media for the rapid and sufficiently accurate determination of individual inorganic acids, for example phosphoric[377], nitric[188,321,377], sulphuric[188,355,377,382], perchloric[188,377,382], and others[188,377], and also for the differentiated titration of various acid mixtures. Thus mixtures of inorganic acids, for example sulphuric and perchloric[385], can be analysed by potentiometric titration in methylene chloride. Zarinskii and Gur'ev used a high-frequency method for the differentiated titration of mixtures of inorganic acids in acetic acid[378] and in glycol solvents[380]. The differentiated titration of two-component mixtures of inorganic acids, for example sulphuric and phosphoric, nitric and phosphoric, sulphuric and hydrochloric, etc. has been carried out by a conductometric method in ethanol[322] and in acetic acid[376]. Potentiometric titration can be used for the analysis of mixtures of inorganic and organic acids, for example hydrochloric and acetic acids in methylene chloride[384], sulphuric and phenolsulphonic acids in a dioxan + diethylene glycol mixture[381], sulphuric and sulphonic acids[344] and nitric and monochloroacetic acids[346] in acetone, and sulphuric and lactic acids in isopropyl alcohol[326].

Drozdov and Kolchina[473] developed a potentiometric method for the analysis of a three-component mixture of hydrochloric acid, methylphosphonic acid, and isobutyl hydrogen methylphosphonate, and also other mixtures of phosphoric acid derivatives of considerable practical importance.

We developed methods for the titration of two-, three-, four-, five-, and six-component mixtures of strong, weak, and very weak organic and inorganic acids in a medium of ketones and other differentiating solvents[310,353,354]. Methods have been proposed for the determination of mixtures of nitric acid with organic acids (nitrobenzoic acid, nitrophenols, and α-hydroxyisobutyric acid) in ethyl methyl ketone[334], and for the analysis of two- and three-component mixtures of nitric acid with monocarboxylic, dicarboxylic, and hydroxycarboxylic acids (nitric + adipic + oxalic) in methanol[320,321]. A method proposed for the analysis of mixtures of phosphoric and sulphuric acids, and of phosphoric and nitric acids in the presence of trioctylamine[1] is of considerable practical importance in phosphoric acid production.

5. Titration of Acid Anhydrides and Chlorides

A number of papers[313,330,474,475] have been devoted to the determination of the anhydrides and chlorides of carboxylic acids. The chlorides of aliphatic and aromatic carboxylic acids exhibit acidic properties in tetrahydrofuran and can be titrated with cyclohexylamine solution[383]. The method is suitable for the determination of all acid chlorides, including those containing readily hydrolysed groups, and also for the determination of aromatic compounds containing two acid chloride groups. A method has been proposed for the determination of an acid chloride in the presence of pyridine in a medium of dioxan; excess of water is removed with acetic anhydride, and the resulting pyridinium chloride is titrated with perchloric acid solution[358]. A method has been developed for the analysis of the actual concentration of the acid chlorides of carboxylic acids in mixtures with the acid and hydrochloric acid. The method is based on the titration of the acid chloride with sodium hydroxide in ethanol before and after heating, and can be used to

determine the concentrations of hydrochloric and carboxylic acids and the acid chloride[325].

Emelin and Svistunova have proposed a number of methods for the determination of the anhydrides and chlorides of carboxylic acids[341,348,356]. The determination of acetic anhydride in acetylating mixtures is based on the reaction with aniline, excess of which is titrated in diethylene glycol[454], and the determination of the chloride of isophthalic acid is based on the reaction of the chloride with aniline. The differentiated titration of the resulting anilinium chloride and isophthalic acid is carried out potentiometrically in a medium of acetone with alcoholic sodium hydroxide solution.

VI. TITRATION IN NON-AQUEOUS SOLUTIONS OF SALTS

The great majority of salts can be titrated in non-aqueous solvents, since many salts which are neutral substances in aqueous solution exhibit acidic or basic properties in non-aqueous solutions. Titration in non-aqueous solutions not only makes it possible to determine individual salts, but also to analyse multicomponent mixtures and mixtures of salts with acids or with bases[476].

1. Titration of Individual Salts

A number of salts exhibit acidic properties in protophilic and amphiprotic solvents and can be titrated as acids[476]. For example, inorganic salts such as the fluorides of multivalent metals (Mo, Ta, Ti, Nb, and W) can be successfully titrated in a mixed ethyl methyl ketone + benzene (1 : 1) solvent[317]; nitrates, bromides, and chlorides of the rare earth elements can be titrated in a medium of acetone + methanol (1 : 1); and lithium and sodium perchlorates can be titrated in pyridine[368]. Salts of organic acids can be titrated, for example zinc stearate in ethanol[323], and zinc styphnate in ethylenediamine[477]. Jasinski and coworkers titrated salts of organic bases, for example amine picrates in pyridine[403] and aromatic amine hexachlorotellurites in acetonitrile and dimethylformamide[478]. Salts of organic bases and mineral acids are readily titrated in methanol[1].

A number of salts exhibit basic properties in protogenic and amphiprotic solvents and can be titrated as bases[479-483,496-498]. Inorganic salts can be titrated, for example chlorides[484] in dioxan, nitrites in the presence of nitrates in ethyl methyl ketone[476], nitrates[485] and sulphates[486] in an acetic acid + acetic anhydride mixture, ammonium carbonate[487] in acetone, and salts of the rare earth elements in acetic acid, ethyl methyl ketone, and mixtures of glycol with alcohols[488,489].

Most salts of monobasic organic acids (the acetates, formates, benzoates, salicylates, lactates, etc. of lithium, sodium, calcium, zinc, cobalt, manganese, and other alkali and alkaline earth metals) can be successfully titrated in acetic acid and acetic anhydride, alcohols, and ketones[476,490-496]. The salts of polybasic organic acids (oxalic, tartaric, etc.) can be determined by titration in acetic acid[497]. Salts of organic bases[485,499] can also be titrated as bases in acetic acid[500,501], acetic anhydride[499], and acetic acid + acetic anhydride mixtures[485]. A method has been described for the determination of the combined acid in hydrazinium salts, based on the conversion of the hydrazine into the azine by reaction with acetone and titration of the resulting acid with NaOH solution[502].

2. Titration of Salt Mixtures

The use of non-aqueous differentiating solvents makes it possible to analyse salt mixtures[476,503,504]. Mixtures of salts, for example lithium and calcium acetates, potassium and zinc benzoates, sodium adipate and potassium hydrogen phthalate, etc., can be titrated as mixtures of bases with different strengths in ethyl methyl ketone[505] and in a dioxan + water (1 : 1) mixture[359]. Mixtures of salts can also be titrated as mixtures of acids with different strengths in pyridine[368] and methanol[1]. An interesting method has been developed for the analysis of two- and three-component mixtures of nitrates of the rare earth elements with the nitrates of other elements (Ni + La, Al + Eu, Cr + Tu, Fe + Zn + La, and Cr + Co + La) in a methanol + acetone medium[1]. A method has been developed for the analysis of multi-component mixtures of salts of organic acids, based on treatment of the mixture of salts to be analysed with perchloric or sulphuric acid in a medium of ethyl methyl ketone, followed by differentiated titration of the excess of perchloric or sulphuric acid and the acids liberated from the salts[312,506].

3. Titration of Mixtures of Salts with Acids or Bases

Mixtures of salts and acids can be analysed by direct potentiometric titration; for example, two-component mixtures of lithium chlorate and perchloric acid, or of sodium chlorate and benzoic acid, and also three-component mixtures of lithium and ammonium chlorates with perchloric acid can be titrated in pyridine[368], and a mixture of ammonium chloride and hydrochloric acid can be titrated in acetone[510-512].

Kreshkov, Yarovenko, and Milaev[1] developed a method for the analysis of mixtures of the rare earth elements, based on the conversion of the mixture to be analysed into the nitrates, bromides, or chlorides by the action of the corresponding acids followed by differentiated titration of the excess of acid and the mixtures of salts, which in an acetone + methanol medium behave as acids with different strengths (HNO_3 + Lu, HNO_3 + Lu + La, and HNO_3 + Dy + La).

Methods which have been developed for the analysis of salt mixtures, based on the preliminary treatment of the solution to be analysed with a stronger acid, can be used for the analysis of mixtures of salts with acids or bases[340-506-509]. This method can be used in a medium of ethyl methyl ketone for the titration of mixtures of sodium acetate and acetic acid, potassium benzoate and benzoic acid, and sodium acetate and acetic and salicylic acids, etc., and also mixtures of salts and bases, for example potassium benzoate and potassium hydroxide, sodium acetate, potassium salicylate, and sodium hydroxide, and other mixtures[505,508,509]. A method has been proposed for the analysis of mixtures of salts and acids, based on treatment of the mixture to be analysed with HCl solution followed by potentiometric titration of the resulting acids with an ethanolic solution of KOH[340].

A method has been proposed for the differentiated titration of mixtures of salts with hydrochloric, nitric, sulphuric, acetic, and other acids, based on the reaction of the mixture to be analysed with an excess of tetraethylammonium hydroxide solution; the excess of tetraethylammonium hydroxide and the hydroxide of the cation of the salt being analysed are then determined by potentiometric titration with a solution of perchloric acid in ethyl methyl ketone[513]. For the analysis of mixtures of salts of organic bases with

inorganic acids, we proposed a combined method[1], involving ion exchange and potentiometric titration of the mixture to be analysed in a medium of methanol.

4. Determination of Salts Using Ion Exchange

The use of ion exchange in non-aqueous solutions makes it possible to analyse various salt mixtures, and also mixtures of salts with acids or bases[514-521].

Yarovenko, Sayushkina, and Zelenina developed methods for the analysis of salt mixtures, based on the use of ion exchange in non-aqueous solutions. It is possible with H-cation exchange to determine mixtures of salts, whose acids can be determined by differentiated titration in non-aqueous solutions using the potentiometric method, for example, mixtures of the salts of strong inorganic acids such as sulphates + chlorides, sulphates + bromides, or sulphates + nitrates; mixtures of the salts of strong inorganic acids and weak organic acids such as nitrates (chlorides, bromides) + benzoates (salicylates, propionates, valerates, etc.); and mixtures of salts of organic acids such as salicylates + benzoates (propionates, etc.).

It is possible by OH-anion exchange to analyse mixtures of salts, whose bases can be estimated by differentiated titration in non-aqueous solutions. The use of anion exchange with Cl^- or NO_3^- forms can be used to determine mixtures of sulphates, chlorides, and nitrates.

VII. TITRATION OF MONOMERIC AND POLYMERIC COMPOUNDS IN NON-AQUEOUS SOLUTIONS

Titration in non-aqueous solutions has found extensive application for the analysis of monomeric and polymeric compounds.

1. Titration of Organoelemental Compounds

Non-aqueous solutions have found most extensive application in the analysis of organosilicon monomers and polymers. Kreshkov[522] developed the theoretical principles of the analysis of monomeric and polymeric organosilicon compounds in non-aqueous solutions and demonstrated the possibility of their practical application. Non-aqueous solutions can be used for the titration of alkylchlorosilanes[523,524], phosphorus-, boron-, sulphur-, and hydrogen-containing organosilicon compounds[525-535], alkylacetoxysilanes[536-538], silanols, and siloxanes[539]. A conductometric method has been described for the titration of trimethylchlorosilane, dimethyldichlorosilane, and methyltrichlorosilane in a medium of acetonitrile with organic bases containing tertiary nitrogen atoms (pyridine, quinoline, 8-quinolinol, dimethylaminoantipyrine, etc.)[523].

Drozdov and Tarasyants[531] developed a method for the potentiometric titration of alkylthiocyanatosilanes in the presence of alkylacetoxysilanes in a medium of acetonitrile. A method has been described for the analysis of boron-containing organosilicon compounds in the presence of organosilicon compounds containing chlorine and phosphorus[534].

The potentiometric and conductometric titration of alkylsilyl hydrogen phosphates in alcohols, ketones, and acetonitrile have been studied[527,529,530], and it has been established that triethylsilyl hydrogen alkylphosphonates can be determined quantitatively without preliminary separation in the presence of the corresponding triethylsilyl alkylphosphonate esters or alkylchlorosilanes. The behaviour of alkylsilanes, hexaalkyldisiloxanes, and sodium triethylsilanoxide in liquid hydrogen fluoride has been studied by an electrical conductivity method; the conductometric determination of individual alkylsilanols and hexaalkyldisiloxanes and their binary mixtures is possible[539].

Myshlyaeva and coworkers[540,541] developed a new method for the quantitative determination of silicon in organosilicon compounds, based on their decomposition with hydrofluoric acid followed by conductometric titration of the resulting hydrogen fluorosilicate in non-aqueous solvents with a solution of benzidine, tolidine, and other organic amines.

2. Titration of Monomeric Compounds

One method for the determination of unsaturated monomeric compounds is based on oxomercuration:

$$R_1R_2C=CR_3R_4 + Hg(OCOCH_3)_2 + CH_3OH \rightarrow$$
$$\rightarrow R_1R_2(OCH_3)C-CR_3R_4(HgOCOCH_3) + CH_3COOH.$$

Balyatinskaya and Tur'yan[542-544] used this reaction to determine the purity of vinyl derivatives of the pyridine series such as 2-methyl-5-vinylpyridine, styrene, methylstyrene, and 2,4-dimethylstyrene. The acetic acid liberated in the reaction in quantities equivalent to the concentration of the monomer being determined is titrated with a methanolic solution of KOH. The differentiated determination of cis- and trans-isomeric acids (fumaric and maleic, mesaconic and citraconic, and oleic and elaidic is possible[545]. The determination of the *cis*- and *trans*-isomeric forms of the acids involves determination of the total acidity and determination of the concentration of the *cis*-isomer by oxomercuration, followed by potentiometric titration of the unreacted mercury(II) acetate in non-aqueous medium. The rate of reaction of the *trans*-isomer with mercury(II) acetate is smaller by a factor of 20 than the rate of reaction of the *cis*-isomer.

A micro-method has been described for the determination of acetylenic hydrogen, based on the reaction of monosubstituted acetylenes with $AgClO_4$. The $HClO_4$ liberated is titrated with trihydroxymethylaminomethane solution[546].

Non-aqueous titration is finding application for the determination of the concentration of residual monomer in a polymer. Thus a method has been proposed for the determination of methylamine in melamine resins by titration of a specimen of the resin with an acetic acid solution of $HClO_4$ in an acetic acid + acetic anhydride (8 : 2) mixture using Crystal Violet as indicator. An analogous method can be used to determine the concentration of benzoguanamine in benzoguanamine resins. Guretskii and Toryanik[547] determined combined dibasic acids in polyester resins by potentiometric titration, in non-aqueous solutions, of the resin hydrolysis products.

3. Titration of Functional Groups in Organic Compounds

The concentration of carboxyl[371], alkoxy[548-551], hydroxyl[552-554], epoxy[291], dinitrophenyl[555] and other functional groups[556] in organic compounds can be determined in non-aqueous solvents.

Cundiff and Marcunas[549] developed a method for the determination of alkoxy groups, based on their reaction with HI to form the alkyl iodide, which in a medium of pyridine forms the alkylpyridinium halide; the differentiated titration of the latter and the excess of HI can be carried out with tetrabutylammonium hydroxide solution. For the determination of alkoxy groups in higher alcohols, a method has been proposed for the determination of the quantity of alkyl iodide formed, without its distillation[548].

A method has been described for the determination of hydroxyl groups[552] in organic substances in the presence of carbonyl compounds, ethers, amines, alkoxy compounds, and complex hydrocarbon mixtures by direct titration with aluminium and lithium amide in a medium of tetrahydrofuran. Hydroxyl groups can also be determined using 3,5-dinitrobenzoyl chloride[553], which reacts quantitatively with hydroxyl groups to form dinitrobenzoate, which can be titrated as a weak acid in a medium of pyridine.

A method has been proposed for the determination of compounds containing epoxy or ethyleneimine groups by titration in a medium of chloroform with a solution of perchloric acid in the presence of an excess of a quaternary ammonium bromide or iodide[291]. The concentration of acetoxy groups in various substituted acetoxysilanes can be determined by potentiometric titration in ethyl methyl ketone with a methanolic solution of sodium methoxide[536,537].

Titration in non-aqueous solutions is widely used for the determination of terminal functional groups in macromolecular compounds. This method is the most rapid and convenient for the determination of the molecular weights of linear condensation polymers whose molecular weight usually does not exceed 20 000.[557-559] Carboxyl[391,560,561], hydroxyl[562,563], and amino groups[254,391,564] can be determined. Carboxyl groups in polyamides can be determined by titration in a hot 2,6-xylenol + chloroform mixture[560] and in propargyl alcohol[391]. The chief disadvantage of these methods is the necessity to dissolve the polyamides at high temperatures, at which some of them decompose. For the determination of amino groups, the polyamides are dissolved in phenol + ethanol[564] and *m*-cresol + isopropyl alcohol (2 : 1)[391] mixtures. A method has been developed for the analysis of polymeric compounds of the benzimidazole series, based on the determination of the NH-group of the elementary unit of the polymer by titration in an ethyl methyl ketone + formic acid (4 : 1) medium[565]. Primary, secondary, and tertiary amino groups in polynuclear polyamines can be determined in non-aqueous solvents[269,456].

A method has been proposed for the determination of hydroxyl groups in macromolecular alcohols, the products of the addition of ethylene oxide to higher alcohols, and complex mixtures containing alcohols, alkanes, alkenes, esters, acids, and carbonyl compounds, based on acetylation with acetic anhydride in a mixed ethyl acetate + n-hexane solvent with the addition of perchloric acid as catalyst[562]. A similar method has been used to determine hydroxyl groups in polyoxyalkylene ethers[563]. Hydroxyl groups have also been determined in polynuclear phenolic compounds[566].

Guretskii and Rybal'chenko[567] proposed a rapid method for the determination of epoxy-groups in epoxy resins modified by organosilicon compounds. The method is based on the titration of the epoxy resins in a medium of acetic acid saturated with KBr solution, using an acetic acid solution of HBr as titrant. The addition of KBr to the solution makes it possible to increase considerably the rate of hydrobromination of the epoxy groups, Epoxy-groups have also been determined in non-aqueous solvents by other workers[568].

VIII. ANALYSIS OF INDUSTRIAL MATERIALS

Non-aqueous titration is being used successfully for the analysis of raw materials, intermediates, and finished products in the pharmaceutical and food industries, and also in the production of plastics, in the control of the various stages of industrial sulphonation, in the processing of coal, and in other industries. Methods for the titration of pharmaceutical products are particularly widely ployed[198,200,204,206,209,223,225,232,234,242,259,261,266,268,286,288,289,569-577]. Non-aqueous solvents can be used for the titration of not only individual pharmaceutical products with acidic or basic properties, but also their mixtures. Thus Izmailov and coworkers[571] analysed mixed products consisting of mixtures of weak acids (luminal, veronal, and aspirin) with weak bases (pyramidone, caffeine, and codeine). The bases were determined in a mixture of acetic acid and acetic anhydride (1 : 4) and the acids were determined in a mixture of dimethylformamide and benzene (1 : 3).

Titration in non-aqueous solutions is used for the determination of the concentration of antioxidants (*NN'*-disubstituted *p*-phenylenediamines) and polymerisation accelerators (diphenylguanidine) introduced into rubbers and rubber mixtures[233,446,578]. The bases to be determined are first extracted from the rubber mixtures using acetone. The concentration of aliphatic acids in rubbers is determined by a polarographic method[579].

Gribova and Khmel'nitskaya[264] developed a method for the analysis of an industrial mixture of mono-, di-, and tri-ethanolamines. Gribova[338] developed a method for the production control of 2,4-dichlorophenoxyacetic acid and also a method for the analysis of a sulphonation mixture containing sulphuric and sulphonic acids[344]. A method has been described for the analysis of explosive powders in non-aqueous solutions. The specimen is treated with dichloromethane and the concentrations of diphenylamine, 2-nitrodiphenylamine, 2,4-dinitrodiphenylamine, and N-nitrosodiphenylamine in the presence of one another are determined[220].

Emelin and coworkers[580] proposed a method for the determination of pyridine and nitrile nitrogen in the copolymer of acrylonitrile with methylvinylpyridine. Pyridine nitrogen in the form of the amine is determined in nitromethane by titration with perchloric acid solution, and the nitrile nitrogen is determined in dimethylformamide by titration with piperidine solution. The same authors also determined acetic anhydride in acetylating mixtures[454]. Rapid methods have been developed for the analytical control of the quality of crude phthalic anhydride obtained by the catalytic oxidation of naphthalene[581], and for the determination of n-butylamine in a caprolactam melt[270] and of tertiary amines in kerosene solutions[227].

A method has been described for the determination of sulphur in a gas by combustion in an atmosphere of oxygen followed by conductometric titration of the resulting sulphuric acid in aqueous ethanol[582]. Carbon dioxide obtained by the combustion of organic compounds is absorbed in dimethylformamide and titrated with a benzene–methanol solution of CH_3OK[583]. A method has been developed for the determination of traces of carbon in metals, alloys, and ceramics, based on the action of heat on the specimen in a current of oxygen, absorption of the resulting carbon dioxide in acetone, and subsequent titration[584].

The differentiated titration of ammonium nitrogen in ammonium salts and ammonium fertilisers can be carried out by back titration in acetone and other non-aqueous solvents[585-587].

Non-aqueous titrations were also used for the determination of pyridine bases in phenols[238] and of traces of acid in ethyl silicate[588] and surface-active substances[327,340].

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