AMPEROMETRIC TITRATION* (A REVIEW)

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O. A. Songina

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Our previous review of amperometric titration was published in 1958 [1], since when two† monographs on this subject [2, 3] have appeared, as well as individual chapters in textbooks of electrochemical analysis [4-9]. 27 percent of the articles in reviews of world literature in "Analytical Chemistry" [10-12] are by Soviet authors. These articles include reports on amperometric titration with two indicator electrodes [13].

We shall discuss the main trends in the development of amperometric titration (a.t.) and some of the most promising reports in this field.

The distinctive feature of this method at present is the use of organic reagents, particularly ethylenediaminetetraacetic acid (EDTA) or its salts (versene), and sulfur-containing compounds. Nearly 75 percent of all recent reports on a.t. deal with the use of organic compounds as titrating solutions.

Another characteristic feature is the use of solid electrodes, which are now displacing dropping mercury electrodes (about 35 percent of published reports deal with the latter). Together with platinum electrodes it is proposed to make increasing use of other types—tantalum [14-21], tungsten [22], and graphite [23-29]. Graphite electrodes are particularly promising because they are readily cleaned, and extraneous processes (particularly the reduction of dissolved oxygen) take place at far more negative potentials on them than on platinum. Kolthoff and Sambucetti [30] used a rotating aluminum electrode to determine the fluoride ion, while Harris used a palladium electrode [31]. However, the indicator electrode is then no longer "inert" and is anodically depolarized in the presence of fluoride ions. An anode current appears, decreasing as the fluoride ions are titrated with a solution of an aluminum or thorium salt. A similar method was proposed by Dumontier-Goureau and Tremillon [32].

Determination of Individual Elements

Most reports are devoted to the a.t. of silver [33-41] and gold [38, 41-49]. There is a distinct tendency towards replacement of the assay method of determining these elements in the presence of other elements by more up-to-date and simpler methods. Thiourea enables one to determine from 1 to 100 g/t Au in concentrates and crude copper [48]; zinc, nickel, cobalt, aluminum, lead, silver and several other elements do not interfere with the determination of gold by thioxine (in pure solutions) [48]. Unithiol is very sensitive, enabling one to determine 50 μg Au in 25 ml of solution; this method has been checked on ore concentrates containing 180-900 g/t Au [49]. Diethyldithiocarbamate [42] (enabling one to determine 0.4 mg Au in the titrant) and dithiobiuret [38] have also been recommended, as well as several organic reducing agents: metol, hydroquinone, p-aminophenol, and phenylenediamine [44]. These reducing agents are used for determining from 3 to 90 mg/liter Au in the presence of selenium, tellurium and metals of the platinum group. Mohr's salt [41] and potassium ferrocyanide [47] are among the inorganic reagents used for determining gold.

Titration with thiourea [37], dimercaptothiopyronecarboxylic acid [36], thionalide [40], piperidinedithiocarbamate [34], thioacetamide [33] and dithiobiuret [38] has been proposed for the determination of silver. In [2] a sufficiently sensitive and selective method of titrating silver with potassium iodide was developed; this proved suitable for determining silver in pharmaceutical preparations [35], crude copper, and various natural objects [39]; it is

*The selection of literature references was made with the valued assistance of I. S. Savitskaya (Cand. Chem. Sci.).

†When the review had been completed we received a report that a monograph entitled "Amperometric Titration", by J. Stock, had just been published in the USA (1965).

of particular interest that it can also be employed for the separate determination of silver and palladium when present together in a solution [50]. Many amperometric methods are devoted to palladium [36, 45, 51-59]. Methods described deal with the use of organic reagents for this purpose: thiourea [58], dithiocarbamate [51]; dithiobiuret [56], thioanilide [57], hexamethylenedithiocarbamate [55], and nioxime. In the latter case, titration is performed by means of the reduction current of $[PdCl_4]^{2-}$ on a platinum electrode [59]. Yu. I. Usatenko and V. I. Suprunovich developed the sequential titration of gold and palladium with thioxine [45] and the determination of palladium by this reagent in the presence of iridium, iron and copper [52-54].

Much attention has been paid to the use of thioxine [60, 61] and dimethyldithiocarbamate [62, 63] for the determination of copper. The use of anthranilic acid labeled with hydrogen [64], and a number of other reagents [65-68], has been proposed for radiometric determinations with amperometric control.

Thiourea and its derivatives [38, 69-71], ethyl xanthogenate [72] and unithiol [73, 74] have been proposed for the determination of mercury. Unithiol enables one to determine separately mono and bivalent mercury [74]. Since this reagent forms a very stable complex with mercury, it can be used for determining as little as 1 μg Hg in the titrant. The anodic iodide method of determining mercury is also very sensitive [75]. Reference [76] gives a rather interesting method based on titration of mercury with Reinecke salt by means of the reduction current of the latter on a dropping mercury electrode. However, the practical application of this method, which involves the use of a mercury electrode, is less convenient than work with a platinum electrode (vide supra).

In recent years a.t. methods have been developed for several rare elements. For the determination of titanium, zirconium, gallium, and scandium, [77] proposes the use of p-benzoylphenylhydroxylamine, which gives with these elements poorly soluble complexes and is oxidized on a platinum electrode.

A simple method has been proposed for germanium, based on direct titration of a germanium (IV) salt in ammonium chloride solutions against magnesium sulfate by means of the reduction current of germanium on a mercury dropping electrode, giving a magnesium germanate precipitate [78]. The method enables one to determine tenths of a milligram of germanium in ~15 ml. Boron, arsenic and silicon do not interfere with the titration. It would appear that this method is simpler and more convenient than titration with pyrocatechol [79]. For the determination of germanium and other elements which form complex heteropolyacids (silicon, phosphorus and arsenic), [80] recommends titration with nitron on a dropping mercury electrode. Hafnium is determined by tartrazine and flavazine [81].

Several methods have been proposed for determining rare earths. The total rare earths can be titrated with permanganate on a platinum electrode in the form of oxalates after dissolving in sulfuric acid [82]. Cupferron is employed for determining both the total and the individual elements by the precipitation method [83, 84]. The determination of europium is based on the formation of a complex of europium with the sodium salt of diethylenetriaminepentaacetic acid [85] on a dropping electrode. Indian authors [86-88] used for the determination of cerium and lanthanum a solution of sodium molybdate, performing the titration at pH ≈ 6 on a dropping mercury electrode against the reduction current of the elements determined. The precipitate has the composition $Me_2O_3 \cdot 3MoO_3$, where Me is cerium or lanthanum.

Several methods have been proposed for the determination of cerium, based on reduction of cerium (IV) by solutions of ferrous iron, ferrocyanide, iodide [89], ascorbic acid [90, 91], oxalic acid [82] and hydroquinone [91].

For determining lanthanum, [92] proposed precipitation with potassium ferrocyanide and titration with a mercury electrode by means of the reduction current of lanthanum at −1.58 V. The titration is made simpler and more convenient by using the oxidation current of ferrocyanide on a platinum electrode at potential +1.0 V.

The a.t. method is used for determining plutonium: the PuO_2^{2+} ion is titrated with a ferrous iron solution on a platinum electrode [93, 94], reducing Pu(VI) to Pu(IV). By using a gravimetric buret and a microapparatus, 0.1-0.05 μg Pu in a milliliter can be determined.

Interest is attached to the use of iodine chloride as an oxidizing agent which can undergo reduction on a platinum electrode in the range +0.2-0.2 V/normal calomel half-cell [95]. For determining arsenic in the presence of antimony, the authors of [96] proposed titration with iodine on a platinum electrode after selective oxidation of antimony with bichromate in certain conditions. A method has also been developed for determining As(III) and As(V) when present together in ores. In alkaline solutions, arsenic and antimony (in trivalent form) can be titrated with a solution of mercuric chloride in the presence of tin (II), which in alkaline solutions gives an oxidation current

and reacts with this chloride; the titration curve has two sharp breaks [97]. Reference [98] recommends determination of arsenic with tartaric acid, with which it gives a complex, reduced on a dropping mercury electrode at −1.16 V.

Titration of manganese (II) with permanganate in the presence of pyrophosphate, previously performed potentiometrically, is replaced [99] by a.t. on a platinum electrode, which is used for determining manganese in ores [99] and in zinc bath electrolytes [100]. Another method for determining manganese is a ferrocyanide method performed in a medium of water and alcohol in the presence of glycocoll and sodium chloride for stabilizing the precipitate's composition [101].

Titration of potassium with calcium ferrocyanide was used with good results by the author of [102] for the analysis of sylvinites.

Tellurium (IV) is readily titrated with permanganate in alkali on a platinum electrode by means of the reduction current of permanganate [10]; the method is interesting because it can be employed in the presence of selenium, which is not oxidized by permanganate in alkali.

The orthophosphoric acid ion can be determined by precipitation, by direct titration with a silver nitrate solution in an acetate medium containing 80% ethanol to reduce the precipitate's solubility [104]. The method has a coulometric version, with generation of the titrated ion by anodic dissolution of silver.

Organic and New Inorganic Reagents

In recent years very extensive use has been made of ethylenediaminetetraacetic acid, its sodium salt (versene) and other compounds of this type, for example nitrilotriacetic acid, diethylenetriaminepentaacetate and triethylenetetramine. All determinations based on the use of these compounds are performed by direct titration with respect to the reduction current of the ion determined (mainly on a dropping mercury electrode), or the anode oxidation current of the reagent on a platinum electrode, or by back-titration of the excess EDTA with any substance whose complex with EDTA is somewhat less stable than the ion being determined. The back-titration variant enables one to use versene for the a.t. of virtually any element giving a complex with this reagent.

Reference [105] describes the determination of molybdenum by direct titration on a tantalum electrode by the oxidation current of versene; other reports describe the titration of small amounts of thallium (III) by the oxidation current of versene on a tantalum electrode [21], the determination of microamounts of uranium on a graphite electrode [21], and determination of V (III) and (IV) on a platinum electrode [106]. The instability constants of vanadium (III) and (IV) complexes differ by a factor of seven; this enables one to titrate sequentially at pH 3-4, vanadium (III) being titrated first, followed by vanadium (IV).

Particular note should be made of the use of EDTA and the analogous new compound EGTA (ethylene glycol-bis) amino-2-ethyl ester (N-N'-tetraacetate) for determining calcium and magnesium [107]. The end point is determined from anodic oxidation of the mercury of the electrode in the presence of excess of the titrating reagent. The method may be used for determining any amounts of calcium and magnesium, even less than a microgram; in this case a stationary mercury electrode of diameter 12 cm is used. The current strength is measured by a potentiometer via a standard resistance. 0.01-ml microburets, polyethylene vessels and water purified by ion exchange and twice distilled from a quartz vessel are used for this work.

Two reviews [108, 109] deal with the use of organic reagents in amperometric titration, one of these being devoted to the amperometric determination of rare elements.

In recent years several new inorganic reagents have been employed for a.t., notably iodine chloride and selenious acid. The latter is recommended for determining thorium [110], vanadium [111], cobalt [112] and cadmium [113] on a dropping mercury electrode by the reduction current either of these ions or of the selenite ion.

Electrometric Indicators

Satisfactory progress is being made in the use of electrochemical indicators for determining the end point in cases where neither the titrant nor the determined ion give an electrode reaction, or the end point must be made sharper. For this purpose, Yu. I. Usatenko and his co-workers employed the difference between the strengths of the corresponding complexes and their reactivities on the electrode after the main reaction between the reagent and the ion has ended. For the example, in determining gold with diethyldithiocarbamate [42], they used lead salts; in the titration of palladium with hexamethylenedithiocarbamate, bismuth salts [55]; and in the titration of iridium,

palladium, iron and copper with thioxine [52] the elements themselves play in turn the role of amperometric indicators. Copper salts play a similar role in the determination of gold with unithiol [49]. In one of the complexometric methods of determining calcium and magnesium [114], thallium acts as the indicator, and mercury plays a similar role in the determination of uranium [23]. The authors of [115] discuss other cases of the use of electrochemical indicators.

Amperometric Titration of Organic Compounds

Amperometric titration plays an important part in the analysis of organic substances, particularly pharmaceutical preparations (this forms the subject of Zyka and Dolezal's monograph [3]). As before, silver and mercury compounds are used for determining compounds containing mercapto groups [116-118], particularly for the determination of thiourea [37, 119, 120]. Aldehydes and ketones are titrated with dinitrophenylhydrazine [121-123] or hydroxylamine [124] on a dropping mercury electrode. Salts of hydroquinone sulfonic acids [125, 126], polysaccharides [127], and starch fractions [128] are titrated with iodine on a platinum electrode; phenolic groups [129] and vinyl monomers are titrated with bromide-bromate on a platinum electrode. Various amines are titrated with tetraphenylboron sodium on a dropping mercury electrode or graphite electrode [24]. Acetylene can be titrated with silver or mercury salts [131, 132].

Particular note should be made of the use of a.t. in nonaqueous media, particularly for determining organosilicon compounds [133-136]. Compounds containing the $\equiv Si-H$ group are titrated in methanol with a solution of mercuric chloride in a methanol-benzene solution, while alkylchlorosilanes are titrated in the same medium or in anhydrous acetic acid with corresponding solutions of lead or cadmium salts. The medium is made conductive by adding lithium salts soluble in organic solvents. A dropping mercury electrode acts as the cathode, the mercury pool as the anode. Unsaturated compounds oxidized by a solution of bromine in methanol or in anhydrous acetic acid are titrated on a platinum electrode against the oxidation current of bromine [135].

Titration with Two Indicator Electrodes

Recent years have seen a marked heightening of interest in a.t. with two indicator electrodes (dead-stop end point) [137-141]. American chemists recently suggested a new name for this method, "biamperometric titration"; this is a convenient term, but essentially incorrect. Although the conventional name is longer, we shall retain its use because it is more correct (strictly speaking, as pointed out by the author in [2], it would be even more correct to refer to "polarometric" rather than "amperometric" titration).

As a result of the method's simplicity and convenience, its use is widening in the Soviet Union. N. L. Udal'tsova [142] has used it for titrating uranium (IV) in the presence of iron with oxidizing agents such as solutions of cerium (IV) sulfate, ammonium vanadate or iron alum. Indian authors have published a series of reports [143] on the determination of cerium, iron, uranium, cobalt, and tungsten by ascorbic acid (by the reduction method). An interesting point is that tungsten can be determined in the presence of vanadium and molybdenum. If all three elements are to be determined, molybdenum is extracted from 4N HCl, vanadium is titrated in the acid medium, and tungsten then titrated at pH 4.6. Molybdenum can also be back-titrated with ascorbic acid at pH 1.5 after removing the ether and dissolving the residue in dilute H_2SO_4.

Redox reactions are the most convenient for titrating with two electrodes, although precipitation and complexing reactions may also be used. V. D. Vasilenko titrates the total rare earths, precipitating them with ferrocyanide in the presence of alcohol [144]. I. A. Tserkovnitskaya and E. Ju-Tsiu [145] titrate thorium with versene, using a mixture of ferrous and ferric iron as indicator. EDTA is also used for titrating iron itself [146]. The authors of [147] recommend titration of cerium (IV) by the reduction method, using Mohr's salt, cupferron or oxalic acid. Several Soviet [149-151] and foreign [152, 153] reports are devoted to the determination of vanadium by Mohr's salt. Where vanadium (IV) and (V) are present together, the latter can be titrated with Mohr's salt, and then vanadium (IV) with permanganate [150]. Reference [154] recommends determining gold by ascorbic acid. Potassium is precipitated with tetraphenylboron sodium and the excess precipitant is titrated with a solution of thallium nitrate [155]. Amalgamated silver electrodes are used for this work, in place of the platinum electrodes employed in all the cases described hitherto.

A method has been developed for the sequential argentometric and mercurimetric titration of a mixture of sulfides, polysulfides and thiosulfate. The method is used for analyzing industrial solutions of ammonium persulfate [156].

There is a distinct trend towards use of the a.t. method with two electrodes for analyzing organic substances. For example, [157] describes the iodometric titration of the C=C double bond (determination of the diene number) in technical products, [158] the bromatometric titration of oximes, and [159] diphenyldimethylpyrazolone hexylresorcinol and sulfadiazine; in the presence of carbamide, the ammonium ion is precipitated with tetraphenylboron sodium and the excess of the latter is titrated with silver nitrate [160].

As before, this method is widely used for determining water with Fisher's reagent [161-166], and for determining water-dissolved oxygen by the conventional method of Winkler, but with amperometric titration of the liberated iodine with thiosulfate, using two platinum electrodes [167-169]. Reference [168] shows that tenths of a microgram of oxygen can be determined with absolute error ~ 0.06 μg/liter.

The same method is used for indicating the end point during coulometric titration.

A distinctive variant of the method with two electrodes is used in Beyermann's work [170-172]. He imposes a specific emf (~ 60 mV) on two identical electrodes of a noble metal, carries out electrolysis of an appropriate electrolyte for ~ 30 sec, and adds to the electrolyzer a solution of the substance to be determined; during this process the current increases as a result of the electrode reaction of the added ion. By measuring this current in strictly defined conditions, 10^{-7} m/liter gold, platinum metals, and chromium (in the form of bichromate) can be determined from calibration curves [170]. The same principle is recommended by Beyermann for determining similarly small amounts of fluorine; in this case, one of the electrodes should be of aluminum: the anodic dissolution current of an aluminum anode will be proportional to the fluorine concentration. It would appear that this method deserves a considerable amount of interest, particularly when determining ultrasmall amounts of a substance.

Within this field we may include [173], in which McCloskey proposed the determination of small amounts of cyanide ions on a silver anode coupled with a platinum cathode at 0.15 V, with anode rotation rate 850-1550 rpm. Blaedel and Olson's paper [174] is of interest: when glucose is reacted with oxidase the hydrogen peroxide evolved oxidizes ferrocyanide, which is added to the solution; as a result of formation of a ferri-ferro system a current is produced, its value being proportional to the amount of reacted glucose. The electrodes are platinum tubes through which the solution investigated is passed, allowing continuous monitoring of the process.

Catalytic Reactions

Catalytic reactions enable one to determine 10^{-6} to 10^{-7} g of a substance. The molybdenum-catalyzed reaction between H_2O_2 and KI has been closely studied. It was performed in an amperometric variant by several authors at virtually the same time: Jedrzejewskii in Poland [175], A. M. Bulgakova, N. I. Zalyubovskaya, and L. S. Manzhelii [176, 177], and by the present author [178]. Reference [177] describes the determination of both molybdenum and tungsten, both elements being determined in very pure substances and monocrystals of cadmium sulfide and lithium fluoride. The reaction between H_2O_2 and KI is also catalyzed by many other substances, for example, zirconium salts [179] and potassium bichromate [180]. Jedrzejewskii [18] proposed the use of the latter reaction for determining hydrogen peroxide. Titration is performed with one or with two platinum electrodes. Michalskii and his co-workers [181] used two electrodes for determining iron (II) catalyzing the reaction between persulfate and iodide, and for determining germanium as catalyst of the reaction between molybdenum (VI) and iodide [183], and thiocyanate, sulfide and thiosulfate ions from their catalytic influence on the iodine-azide reaction [184].

The author of [185] proposes titration of a niobium peroxide compound on a dropping mercury electrode with cupferron. By reducing the niobium concentration, cupferron reduces the catalytic reduction current of niobium, and the titration curve acquires a distinct L-shape. The use of the amperometric method can obviously greatly increase the sensitivity of niobium determination from the reaction between niobium peroxide and permanganate, described by Babko in [186]: the excess permanganate gives a distinct reduction current on a platinum electrode at any solution pH.

Innovations in Apparatus

Michalski et al. proposed for the performance of a.t. without an external emf comparison electrodes consisting of solutions of mercuric nitrate [187], cerium perchlorate [188], mixtures of solutions of iodine and potassium iodide, and a saturated solution of thallium iodide [189] in which a stationary platinum wire is immersed. It is rather surprising to find that Michalskii has elsewhere [190] stated that no author has previously described a.t. without an external emf; in fact, previous reports on this subject had been given by Kolthoff and others (1945-1946) and by the present author in 1949 (in Izv. AN KazSSR, seriya khim., No. 3).

Japanese authors use manganese dioxide and lead dioxide as comparison electrodes [191]. The present author recommends a rotating platinum electrode in saturated solutions of various oxidants [192-194]. The solutions may be selected so as to obtain [194] a continuous series of electrodes with different potentials in the range from +1.6 V (permanganate in sulfuric acid) to +0.02 V (normal m.i.c.c [metal-ion concentration cell]).

Titration without an external emf is convenient from the practical aspect because it enables one to simplify the setup. Furthermore, both Michalski and the author have noted that the titration curves become clearer if the difference between the cathode and anode potentials is made sufficiently small. This can be done by selecting a comparison electrode whose potential matches that of the process on the indicator electrode.

Several new indicator electrode designs have appeared: a screened dropping mercury electrode for titration with constant stirring or aeration [195], a rotating dropping mercury electrode [196], a rotating mercury electrode [197], etc. References [196, 198] describe vibrating platinum electrodes, and [199] a "convective" platinum electrode located in the electrolyzer together with a rapidly rotating disk; its sensitivity is considered to be higher. The authors of [200] have designed a platinum electrode giving polarograms with a shape very similar to that obtained on a mercury dropping electrode. Reference [174] describes a setup for continuous observations on current strength.

For a.t. with two electrodes [204] recommends the use of two hanging drop electrodes, [151] a setup without a stirrer but with one rotating electrode, and [202] a current amplifier based on transistors, giving a 275-fold current amplification.

A. M. Gabovich [203] has shown that a pulse oscillographic polarograph can be used for a.t.

To increase the measurement accuracy [204] proposes a piston buret and a minumum current detector consisting of a recorder with microswitch, operated when the recorder has passed through the minimum point. Reference [205] recommends a thermostated buret with automatic zeroing. Automation of titration is examined in [107] and by Japanese authors [206] whose articles deal with automatic titration of highly radioactive solutions.

General Investigations

The a.t. method is frequently used for research work, particularly for determining the composition of complexes. This method enables one to establish with sufficient accuracy the ratio between reactants as a precipitate or soluble complex is formed. Yu. I. Usatenko et al. studied complexes of diethyldithiocarbamate with gold [42] and complexes of indium [207] and gold [45] with thioxine. N. N. Kuz'mina studied complexes of silver with thiourea [209], and the authors of [49] complexes of gold with unithiol. Reference [209] discusses the formation of complexes of uranium (VI) and aluminum in a citrate medium, [210] the composition of complexes of vanadium (IV) with ethanolamine, etc.

In the organic field, the authors of [211] studied the addition of bromine at the double bond in barbiturates (phanodorn and evipal sodium) by titration with potassium bromate.

Amperometric methods are also used for studying the rate of formation of precipitates, for example, thallium sulfide during the reaction of thallium ions with thioacetamide [212].

The present author used the amperometric method for studying redox processes during titration of iron by the Zimmermann-Weingardt method [213]. Analysis of the amperometric titration curves and polarograms obtained on a platinum electrode shed some light on the complex reactions taking place during titration.

Studies have also been made of changes at the electrode surface due to processes on the solid electrode: reduction of solid particles (precipitates) formed during titration and producing the anomalous shape of the titration curves; the shape of the polarograms in the presence of two reducing substances as a function of the concentration ratio of these substances; the conditions in which redox processes take place on the electrode and in solution, and their influence on the a.t. curve shape [214-218]. Michalski and Pawluk [219, 220] examined cases of titration of reversible systems by irreversible systems, and reversible by reversible systems, without imposing an external emf.

Recent years have seen a return of interest in the mathematical processing of a.t. curves and their theoretical study [221-223].

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