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Polarographic Determination of Traces of Metals in Organic Material. Determination of Pb, Cu, Cd, Ni, Zn, and Fe

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The most prominent features of polarography are rapidity and selectivity. Furthermore it is often possible to determine, simultaneously, several elements in the same sample. It will be shown that only two samples are necessary for the determination of the six elements mentioned in the heading, whereas when using colorimetric methods, the determination of each element consumes one sample. Unfortunately most previous work on polarographic determination of traces has been carried out without making full use of this favourable feature of polarography.

The determination of traces of metals in organic material is a routine problem in to-days' analytical laboratory. In this work cellulose, carboxymethyl cellulose (CMC) and dry yeast have been investigated. In cellulose it is important to know for example the copper and cadmium content because of the influence of copper on the bleaching of cellulose and of cadmium on the manufacture of viscose. CMC is nowadays used for pharmaceutical purposes and accordingly the lead content must be controlled, and for the same reason it is necessary to estimate the lead content of dry yeast. The other metals mentioned in the heading are usually ascertained in order to establish the degree of cleanliness of manufacture.

In order to prevent uncontrollable losses of the minute amounts of elements being determined, a leading principle in the present investigation has been to avoid achieving separations by the method of precipitation of interfering elements.

Another important aim in this work has been to device and combine the separate unit operations so that the working time is cut down to a minimum. As a result of this effort, the determination of the six elements described in this paper, including disintegration of the sample takes less than 60 minutes in routine work.

A. PROCEDURE

A method for wet combustion of cellulose, CMC, and dry yeast and subsequent polarographic determination of the amount of lead, copper, cadmium, nickel, zinc and iron in the organic material is described below. Two samples

are used. In one of them lead is determined in acid solution after separation by precipitation with ammonia, using ferric hydroxide as a collector. In the other sample, iron is determined in perchloric acid solution and copper, cadmium, nickel, and zinc after adding hydroxylammonium sulphate and pyridine.

Reagents of the highest grade of purity often contain too much lead and copper and need to be purified. Especially important is the purification of those reagents which are used in large amounts, *e.g.* the nitric and perchloric acids employed for wet combustion. The following reagents are used in this procedure.

Nitric acid is distilled in pyrex glass apparatus without stopcock grease, and stored in pyrex containers.

Perchloric acid is distilled in pyrex apparatus under reduced pressure, and stored in a pyrex container. Often "Baker Analyzed" perchloric acid can be used without purifying.

Hydrochloric acid is diluted with an equal volume of water and distilled in pyrex apparatus.

Pyridine: 100 ml pyridine are mixed with 100 ml conc. hydrochloric acid and distilled in a fractionating flask with a wide air-cooled condenser. The fraction boiling between 218 —222° C consists of pure pyridinium chloride, melting point 82° C. The distillate is mixed with 50 ml 50 % sodium hydroxide and pyridine is separated by decantation.

Hydroxyl ammonium chloride: Reagent grade.

Hydroxyl ammonium sulphate: Reagent grade.

Sodium chloride: Reagent grade.

Sodium carbonate: Reagent grade.

Iron (III) nitrate: Reagent grade.

Nitrogen gas.

Distilled water is redistilled in pyrex apparatus or purified by passing through a cation exchange resin, saturated with H^+ by thorough treatment with 4 *N* hydrochloric acid and subsequent washing with distilled water.

Wet combustion of cellulose and CMC. A 5.00 g sample is weighed out in a 250 ml pyrex beaker, and a mixture of 18 ml conc. nitric acid and 15 ml conc. perchloric acid is added. The beaker is covered with a watch glass with ribs and placed on a hot plate with a temperature of 250—300° C. The easily oxidized parts of the organic material first react with the nitric acid. When all this acid is gone and the temperature has risen to about 160° C, the perchloric acid begins to foam and the rest of the sample is oxidized. If charring occurs when the cellulose samples are combusted, the reaction can be accelerated by adding a few drops of conc. nitric acid. The perchloric acid is evaporated and, when only a salt cake remains, 5 ml of water are added. The solution is boiled for 2 mins. to drive off volatile oxidizing matter and hydrochloric acid formed during the reaction. Usually, enough perchloric acid remains on the walls of the beaker to dissolve the residue. If not, 1 ml 0.1 *N* perchloric acid is added for this purpose.

Time required for the above procedure: 40—60 mins. Note: If the sample contains only minute amounts of salts, a salt cake is formed in the beaker by the addition of 0.5 g sodium chloride before wet combustion.

Wet combustion of dry yeast. A 250 ml beaker is placed on a hot plate at about 150° C, and a mixture of 15 ml conc. perchloric acid and 35 ml conc. nitric acid is added. A 5.00 g sample of dry yeast is added in small portions to the acid. If too much of the sample is allowed to react, the foam may rise over the edges of the beaker. Even if sufficiently small amounts are added, it is necessary to stir with a glass rod to prevent the foam rising. When the

foaming has ceased, the beaker is covered with a watch glass with ribs and the procedure described above is followed. If the sample shows a tendency to char, nitric acid is added drop-wise till the charring disappears.

Determination of lead. The wet combusted sample is diluted with 50 ml water in the beaker and 3 ml conc. ammonia is added to precipitate ferric iron and lead. The solution is filtered through pyrex glass wool in a small funnel and washed with 10 ml water. The filtrate is discarded. (If the sample does not contain enough iron to give an easily filterable precipitate, 0.5 ml 0.1 *M* iron (III) nitrate should be added.) The precipitate is moistened with 2 *N* hydrochloric acid and iron and lead is washed out with 3 ml water. The filtrate is collected in a 10 ml measuring flask. The flask is heated on a steam bath and saturated sodium carbonate solution is added until ferric hydroxide begins to precipitate. The solution is acidified drop by drop with 1 *N* hydrochloric acid until the solution becomes clear. At the pH thus obtained iron (III) can easily be reduced by adding hydroxyl ammonium chloride (about 50 mg) and heating on steam bath for a few minutes. After cooling and diluting to the mark with water, the solution is transferred to the polarographic vessel. A blank is run all through the procedure. Note that in the blank, iron (III) nitrate should always be added in order to provide a collector for lead. The polarogram is run according to section B.

Determination of iron. Another sample, previously submitted to wet combustion and dissolved, is neutralized by adding saturated sodium carbonate solution to the hot sample until ferric hydroxide begins to precipitate. The precipitate is dissolved by acidifying with 1 ml 0.1 *N* perchloric acid and the solution is cooled and diluted to the mark in a 10 ml measuring flask.

The sample is transferred quantitatively by pouring it into a polarographic vessel without using any wash water. This operation is possible if the flask is previously made water-repellent with silicone [1].*

The solution is deaerated for 10 mins. by bubbling with nitrogen gas without submerging the dropping mercury electrode in the vessel. A polarogram must be run within a few minutes, as otherwise the ferric iron is reduced by mercury gathering on the bottom of the vessel. The sample is kept for the subsequent determination. The polarogram is run according to section B.

Determination of copper, cadmium, nickel and zinc. Hydroxyl ammonium sulphate, 0.1 g, is added to the polarographic vessel from the iron determination and ferric iron is reduced to ferrous iron by heating on a steam bath. After cooling, 1.00 ml pyridine is added and the solution is carefully deaerated with nitrogen. The polarogram is run according to section B.

B. RUNNING AND EVALUATING THE POLAROGRAMS

The polarograms of lead, copper, cadmium and nickel have the usual shape. The zinc wave, however, is not so good. The upper part is too steep and short to be described as approximating to a straight line (Fig. 3). The steepness results from the proximity of the Fe^{2+}-wave which follows. If the rate of increment of applied voltage is decreased, the

* The flask is impregnated with Desicote according to the description provided by Beckman Instruments, South Pasadena 3, California, USA.

wave becomes easier to evaluate. Evaluation of the polarograms is effected by measuring the heights of the waves at the half-wave potentials.

The different elements are polarographed between the following potentials referred to the saturated calomel electrode at 20° C.

Lead:	−0.15 volts	−0.65 volts
Copper:	−0.20 »	−0.70 »
Cadmium:	−0.50 »	−0.80 »
Nickel:	−0.50 »	−1.05 »
Zinc:	−0.75 »	−1.35 »

Note that if lead and copper are present in comparable quantities, a correction must be made when computing the copper content.

The polarogram of Fe^{3+} is run in quite a different way. As can be seen in Fig. 2 no part of the curve is horizontal before the rise of the Fe^{3+}-wave, and therefore the polarogram is run by recording the diffusion current at a constant voltage of 0.0 v. The zero line of the instrument is then recorded. The distance along the current axis between these two lines is proportional to the ferric ion concentration.

C. STANDARD CURVES AND BLANKS

The standard samples are made in the following way: Filter paper is thoroughly washed with 4 *N* hydrochloric acid to displace cations present in the paper. Since filter paper absorbs metal ions, it should not be washed with distilled water. A 5 g sample is weighed out and known amounts of the elements concerned are added. The analysis is then carried out in the manner described for the samples. After correction for the blanks, standard curves (diffusion current against concentration) are plotted. All standard curves are approximately straight lines. If a blank amounts to more than 10 % of the element concerned, the reagents should be purified further.

It is also possible to use the "pilot ion" and standard addition technique, but in the present investigation the method with standard curves for each element was found more reliable. The computation of the cadmium content, however, was made by comparison with the height of the nickel wave, as the cadmium content was so low, that precision was not very important. Cadmium ions were shown to give approximately the same wave-height as nickel ions at the same molarity. In Fig. 1 are given examples of the standard curves obtained.

D. APPARATUS AND EXPERIMENTAL CONDITIONS

The *polarograph* used in the present investigation was an electronic pen-recording instrument (Radiometer, Copenhagen, Type: PO 3e). Maximum sensitivity: $2.7 \cdot 10^{-4}$ μA/mm. It was provided with a device for prewave compensation and another device for compensation of condenser current. The former is necessary when polarographing several ions in the same solution and the latter is indispensable when employing the high sensitivity necessary in trace analysis.

The capillary. Drop time at 1.0 V in the sample solutions 4.1 sec./drop. The mercury flow was 2.2 mg/sec. It is advantageous to have a capillary with a longer drop time than 4 sec. because otherwise maxima of the second type may develop [2,3]. Moreover, when using short drop time the capillary often drops irregularly at elevated potentials.

The *polarographic vessel* was a short test-tube provided with a stopper in which were bored holes for capillary, agar bridge and deaeration tube. The agar bridge connected the polarographic vessel with a saturated calomel electrode. The electrolyte in the agar bridge was 3 *M* sodium nitrate. Potassium chloride or ammonium nitrate cannot be used

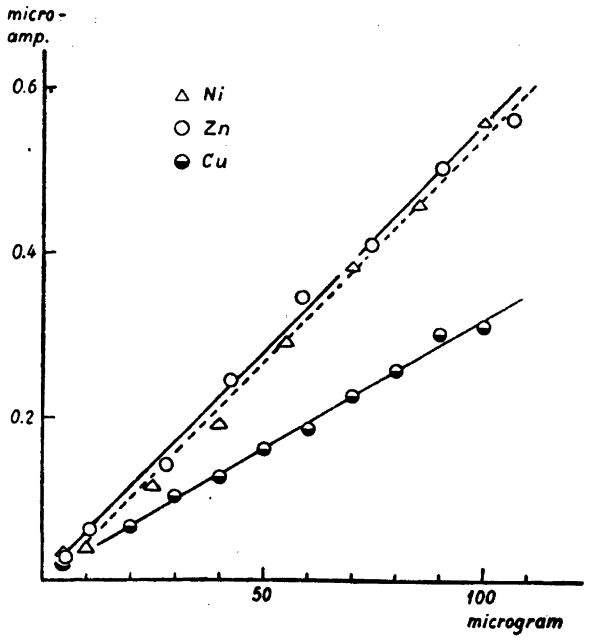

Fig. 1. Standard curves for copper, nickel, and zinc.

in the bridge because potassium and ammonium ions precipitate with perchlorate. Moreover, chloride interferes in the iron and copper determination.

The *temperature* was measured in the sample after the determination, and if not within 20° ± 0.5, the theoretical factor 1.6 % change of diffusion current per degree was used to correct the results.

E. DISCUSSION

1. Decomposition of sample.

Dry ashing is a lengthy process and during the ashing random losses of the elements to be determined can hardly be avoided. Moreover it is often difficult to dissolve the ash without previously melting it with, for example potassium pyrosulphate. Intractable residues are often produced when iron and aluminium are present in the samples. Another defect that is particularly important when traces of metals are involved is the danger of loss by combination with the material of the crucible.

Wet ashing with a mixture of perchloric and nitric acids is a rapid procedure that has been found suitable for trace analysis. The main objection to this method has been that the large volumes of reagents employed might introduce significant amounts of impurities, but in the present investigation purified reagents have been used and difficulties from this source have been easily overcome. The methods of purifying the reagents have been very simple.

The oxidation of organic material by means of this mixture consists of two separate processes. First the nitric acid reacts with the easily oxidized parts

of the organic material present, and not until most of the nitric acid is volatilized does the perchloric acid react (at about 160°) with the more resistant parts. The final oxidation is then carried through by the constant boiling acid (72 %) at about 200° C. This process proceeds smoothly and without bumping. At the temperatures employed the volatility of the perchlorates of the metals with which this work is concerned is insignificant.

A mixture of perchloric and nitric acids has been used for oxidation of the organic parts of many very different materials, *e.g.* rubber [4,5], carbon [6], skin and leather [7], blood [8], plants *etc.* [9,10], textiles [11], sulfite waste liquor and lignosulphonates [12], slate and kolm [13], copper pyrite [14].

According to Kahane [15] there is risk of explosion if the oxidizable substance does not mix well with the acid, *e.g.* mineral oils and fats. Almost all accidents reported with perchloric acid have had the same cause, *viz.*, the evaporation of the alcoholic filtrate from potassium determinations [16]. The general use of perchloric acid has been discouraged by the view that all kinds of perchloric acid are dangerous. This opinion, however, stems from confusion of constantboiling perchloric acid with waterfree acid, which is unstable and explosive. Constant-boiling perchloric acid (72 %) is commercially available, and may be distilled without risk and is stable indefinitely.

In the laboratory, nitric and perchloric acids for wet combustion should be stored ready mixed to avoid the risk of using perchloric acid alone, which might be dangerous. Wet combustion with perchloric acid should not be applied to previously uninvestigated materials without thorough testing. A careful study of the literature is recommended.

Whilst working out this analysis about 1 500 wet combustions have been carried out without any sample having reacted with unreasonable violence. According to Hamlin [11] the tendency to char is diminished by prolonged oxidation with nitric acid. However, the CMC samples in the present investigation did not show any tendency to char when submitted to a very brisk oxidation with nitric acid, whereas when oxidation was done at lower temperatures charring was often observed. This can be explained by the fact that there is, during the heating of the perchloric acid, a period of reduction just after the nitric acid has been evaporated and before the reaction with the perchloric acid begins and this period should be as short as possible. The charring disappears slowly when the perchloric acid reacts, but the reaction is a lengthier process than usual.

The losses are sometimes measurable when using the present method of wet combustion, mainly because of mechanical loss during reaction in the beaker, but usually the agreement is good. (See Table 1.)

Table 1. Determinations of lead and cadmium before and after wet combustion.

	Before		After	
Lead	28.5	27.0 p.p.m.	27.0	28.5 p.p.m.
	15.0	16.5	15.0	17.0
Cadmium	54.3	54.8	55.5	54.0
	26.3	26.4	26.3	26.0

If instead of a beaker a flask provided with an air-cooled reflux condenser is used, losses are less frequent but evaporation of the acids after removing the condenser takes about twice as long. However, as the method is sufficiently precise for determination of traces, the more rapid procedure was chosen. It was found advantageous to add sodium chloride to the samples before wet combustion, because the salt cake left at the bottom of the beaker then became easier to dissolve and losses due to undissolved residues were avoided.

2. Polagraphy.

Table 2. Half-wave potentials (volts) against saturated calomel electrode.

	Aqueous solution of perchloric acid pH 0—2	Aqueous solution of pyridine and hydroxylammonium sulphate pH 5—6
Fe (III)	+ 0.47	—
Fe (II)	— 1.32	— 1.4
Cu (II)	— 0.3	—
Cu (I)	—	— 0.43
Pb (II)	— 0.43	— 0.45
Cd (II)	— 0.62	— 0.73
Ni (II)	— 1.1	— 0.82
Zn (II)	— 1.02	— 1.06

Lead. If a perchloric acid solution containing iron, copper, lead, cadmium, nickel and zinc in the proportions usual in the samples is polarographed, the resulting curve has the appearance shown in Fig. 2. As can be seen in Fig. 2 only Fe^{3+} can be determined with accuracy.

Large amounts of iron (III) and copper interfere in the determination of lead, since their waves precede that of lead. Generally, in the samples employed in the present investigation, the quantities of iron and copper are much larger than those of lead, and these interfering substances must be rendered harmless.

With this purpose in view it was decided that lead should be separated before determination. For various reasons methods involving extraction of lead from the ash were not attempted [17,18]. The main objection to this procedure is that it is always difficult to ensure complete extraction. It was considered more convenient to get all the sample into solution and then make the separation.

Lead may also be separated by electrolysis [19] which is a rapid procedure. The method finally chosen, however, is equally rapid and the equipment required is somewhat simpler.

Kolthoff and Matsuyama [20] recommend the precipitation of copper by potassium thiocyanate. When testing their method in this laboratory, using very low concentrations of lead (10^{-6} *M*), a small wave coinciding with the lead wave was always found, even when the pH was closely adjusted to the value recommended by these authors. Another procedure — especially common when colorimetric methods are being used — is the extraction of the lead dithizonate with chloroform [21]. This procedure was found to take too long and was therefore rejected.

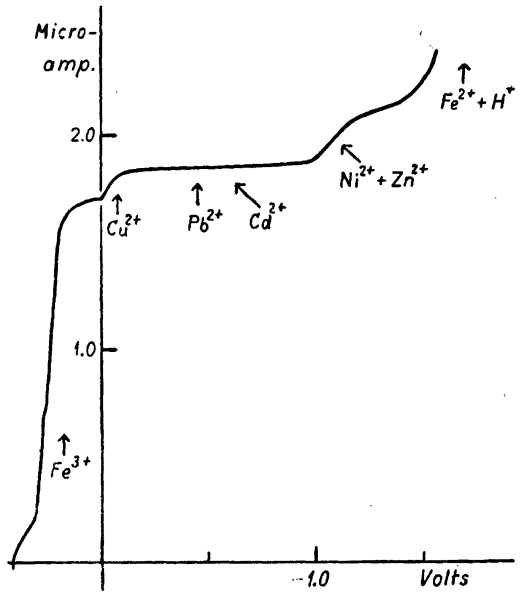

Fig. 2. Characteristic polarogram of metallic impurities in carboxymethyl cellulose after wet combustion with perchloric acid. pH 0—2.

Precipitation of lead with ammonia in the presence of ferric iron [22] and subsequent dissolution in acid makes quantitative recovery of traces of lead possible, as can be seen below.

Lead added (microgram)		20.0			50.0	
Lead found (microgram)	21.1	20.1	19.2	46.2	46.2	50.1

Taking up ferric hydroxide on glass wool instead of filter paper speeds up the procedure, and loss of lead by absorption in the paper is avoided. However, when large amounts of aluminium are present, as was the case with the dry yeast samples, the filtration of the combined ferric and aluminium hydroxides becomes rather tedious. Furthermore it is difficult to dissolve the precipitate in the limited amount of acid that may be used in order to keep the volume of the sample well within 10 ml.

When large amounts of copper are present, some of it is coprecipitated in the ferric hydroxide. The waves of copper and lead, however, are well separated in the acid medium used and thus copper does not interfere when present in the small quantities possible after separation.

The only metal giving a wave partly coinciding with the lead wave is thallium. No attempt was made to detect this element because of the improbability of thallium occurring in the samples.

No maximum was observed on the lead wave in the samples, which is an advantage, because the presence of maximum supressors like gelatin and methylcellulose may decrease the size of the lead wave [23].

Iron. Iron is usually determined in complexing solutions like tartrate [24], citrate [25], oxalate [26] and triethanol amine [27] where the ferric iron wave has the conventional shape of a polarographic wave. In the present investigation, however, it was necessary to consider the fact that copper, cadmium, nickel, and zinc should preferably be determined in the same sample, and the number of possible methods was accordingly restricted. If all methods involving precipitation of the excess iron were excluded, only one procedure was satisfactory *i.e.*, that of determining the iron in a non-complexing solution, where copper cannot interfere [20]. After reducing the ferric iron to ferrous iron, it is possible to determine the other metals simultaneously in a suitable complexing solution where the half wave potentials are well separated.

The iron determination in perchloric acid solution can be made in the presence of large amounts of copper if the solution is free from chloride. Chloride interferes because an anodic wave from the reaction $2Hg + 2Cl^- = Hg_2Cl_2 + 2e^-$ is obtained at + 0.2 volts.

Molybdenum and vanadium, even at low concentrations, give very high steps, coinciding with the ferric wave. If these metals are present in the sample the iron determination should be made in another background solution.

The ferrous iron wave cannot be used because the upper part of the wave coincides with the hydrogen wave.

Copper, Cadmium, Nickel and Zinc. Pyridine buffers have been used in polarographic analysis, mainly because of their ability to precipitate ferric iron without any considerable coprecipitation [28]. Such a solution, however, has many other favourable features. As can be seen from Table 2 and Fig. 3, the half-waves are well separated. Nickel in noncomplexing solution gives an irreversible wave coinciding with the zinc wave. This wave moves to a more positive potential when pyridine is added and a halfwave potential is obtained that is well separated from those of zinc and cadmium [29].

Moreover, it was found that hydroxylamine easily reduced copper to a stable copper (I)-pyridine complex giving a wave consisting of one step instead of the double-wave usually obtained in complexing solutions. Here the height of the copper wave is, for the same molar concentration, about half that of the waves of the other ions concerned. This is an advantage, as copper gives the first wave (See Fig. 3) and may be determined with an adequate degree of accuracy, whereas the following ions cannot be determined with sufficient accuracy if too much copper is present. Furthermore the single copper wave is very easy to evaluate.

Another advantage of the pyridine-hydroxylamine solution is that no iron is precipitated and, in addition it was found that Fe (II) and Cu (I) are not oxidized by air at the pH obtained (pH = 5—6). The waves obtained are well developed and there are no maxima. The only irregularity observed was that if the molarity of copper exceeds 10^{-3} *M* the copper wave shows a peculiar minimum at potentials more negative than −0.5 v.

Many other background solutions have been suggested for the polarography of copper, cadmium, nickel and zinc, but the pyridine-hydroxylamine solution

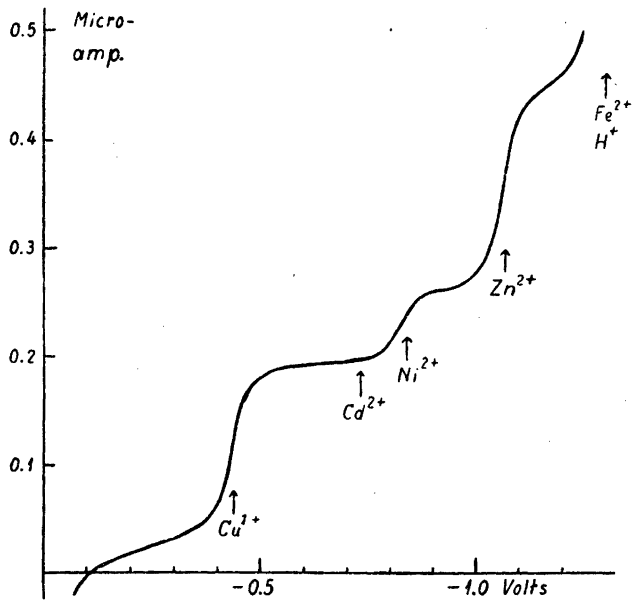

Fig. 3. Polarogram in aqueous solution containing pyridine and hydroxylammonium sulphate.

proved to be the most versatile. For example SCN^- is a complexing agent frequently used [30], but unfortunately copper precipitates and cannot be determined. Tartrate and citrate buffers are frequently employed for the same purpose, but if much iron is present it interferes with all the elements to be determined in the solution except copper, thus making more separations necessary [31,32].

The copper and lead waves coincide in pyridine buffered solution but, since lead is determined separately, a correction can be made for the lead content, which is usually small compared with copper. The amount of pyridine added to the solution is not very critical and as the samples may contain different amounts of perchloric acid after the wet combustion, it was decided to use an excess of pyridine to get the same pH from time to time. If too much perchloric acid is left after evaporation, pyridinium perchlorate precipitates. Care should be taken to avoid the presence of chloride, because copper is mono-valent in this solution and copper (I) chloride is precipitated.

No wave coincides with the cadmium wave and thus the cadmium determination is very selective.

If the pyridine is not purified by distillation as pyridinium chloride as described above, there is interference through some impurities in the pyridine having their waves at the same potentials as nickel and zinc. Distillation of analytically pure pyridine in a column does not yield satisfactory results. After a few days the pyridine then gives the same characteristic interfering waves.

Cobalt gives a wave partly coinciding with the zinc wave. If the former is present, determination of these two metals should be made, for example according to Gagliardo [33].

The sample for determination of iron, copper, cadmium, nickel, and zinc is transferred quantitatively from the measuring flask to the polarographic vessel, the reason being that when pyridine is added after the determination of iron, the volume of the sample must be known exactly. Transferring of liquid by using pipettes in this case is not so rapid and gives less precision than the former procedure. Thus it has been found very useful to render the vessel water-repellent when transferring small volumes of liquid quantitatively from one vessel to another without diluting with washwater [1].

F. ANALYSES

About 200 determinations of each element have been made, and below a few of them are given.

Carboxymethyl Cellulose. Three subsequent analyses of a typical sample

Pb	Fe	Cu	Cd	Ni	Zn
4.6	180	22.4	0	4.8	7.6 p.p.m.
4.7	180	24.3	0	5.0	7.5
4.4	211	23.8	0	5.0	7.4

An homogenous lot of CMC was divided in two parts. One of them was analysed in its original condition and the other after addition of known amounts of metals (Table 3). In this table, each value represents a mean of three determinations.

Table 3.

	Original p.p.m.	Added p.p.m.	Found p.p.m.	Theoretical p.p.m.
Pb	0.8	10.0	12.2	10.8
	1.3	10.0	12.4	11.3
Fe	132	99.5	230	232
	136	99.5	224	236
Cu	7.0	24.2	27.5	31.2
	8.0	24.2	29.2	32.2
Cd	0.0	20.1	17.0	20.1
	0.0	20.1	17.6	20.1
Ni	3.6	17.8	21.6	21.4
	4.0	17.8	22.4	21.8
Zn	11.4	20.2	36.0	31.6
	15.6	20.2	33.4	35.8

Cellulose. Three subsequent analyses of a typical sample

Pb	Fe	Cu	Cd	Ni	Zn
4.7	180	24.3	0.0	5.0	7.5 p.p.m.
4.4	180	24.8	0.0	5.0	7.4
5.0	174	24.4	0.0	5.0	6.9

The lead and copper contents of this sample were determined by dithizone methods at the Institute of Wood research, Stockholm, and were found to be 25, 23, and 25 p.p.m. for copper and 7, 7, and 6 p.p.m. for lead in good, respectively fair correspondence with the values given above.

Dry Yeast. For reasons mentioned in section E 2 only lead was determined in these samples. Results for determination of lead in three different samples:

1:st sample	2:nd sample	3:rd sample
1.7	0.8	3.0 p.p.m.
1.6	0.8	3.4
1.5	0.5	3.2

G. PRECISION

The precision of the method as derived from 180 determinations is about $\sigma = \pm 2$ at 50—100 p.p.m., and $\sigma = \pm 0.4$ at 2 p.p.m. The main sources of error are to be found in the wet combustion and above all in the subsequent chemical processes preceding the polarographic determination. Care should be taken, that impurities are not introduced from the vessels.

The polarographic measurements are made rapidly, and even when the concentrations are as low as 10^{-5} M the precision is 4—5 %. However, it should be observed that the precision is considerably influenced by the presence of preceding waves [34,35].

SUMMARY

A rapid method is described for wet combustion of cellulose, carboxymethyl cellulose and dry yeast and subsequent polarographic determination of the amount of lead, copper, cadmium, nickel, zinc and iron in the organic material. Two samples are used. In one of them lead is determined in acid solution after separation by precipitation with ammonia, using ferric hydroxide as a collector. In the other sample, iron is determined in perchloric acid solution and copper, cadmium, nickel, and zinc after adding hydroxyl ammonium sulphate and pyridine. The time required for the complete analysis in routine work is about 60 minutes. Precision: $\sigma = \pm 2$ at 50—100 p.p.m. and $\sigma = \pm 0.4$ at 2 p.p.m.

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