The Determination of Small Quantities of Lead, with Special Reference to Urine and Biological Materials.

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I. Introduction.—For the purpose of the experimental investigation that the Committee of Enquiry on Lead Ethyl Petrol decided to carry out, a method was needed to determine small quantities of lead in a variety of substances, including urine and biological materials. It was thought that a method of determining lead giving satisfactory results with urine could be applied, with suitable modifications, to the other substances likely to be met in the course of the Committee's programme of work. The method to be described has been in use for a year, and has given satisfactory results with urine and other materials containing organic matter; and, as the details of the method are of interest to chemists, the Committee has now given permission for it to be published.

When the work was begun the only valuable method of determining lead in urine was the modified Fairhall process (*J. Biol. Chem.*, 1924, **60**, 485)* adopted by the American investigators on the health hazards associated with the distribution and use of lead ethyl petrol; but, after the method to be described had been in use for several months, there came to our notice the methods of Taylor (*J. Proc. Roy. Soc., New South Wales*, 1927, **61**, 315), Cooksey and Walton (Analyst, 1929, 97), and Millet (*J. Biol. Chem.*, 1929, **82**, 265). Taylor's method depends on the absorption of the lead in urine by calcium oxalate precipitated directly therein, but no evidence is produced to show that

* See also: "A Study of the Health Hazards Associated with the Distribution and Use of Ethyl Gasoline," 1925, p. 16, by Kehoe and co-workers, Richberg Laboratory, University of Cincinnati, Ohio, U.S.A.

the filtrate from the calcium oxalate is free from lead. The method also involves the conversion of the oxalate into carbonate by gentle ignition, a process best avoided. Cooksey and Walton's method depends on the direct electrolysis of urine, but it is not known that the whole of the lead in urine is in a form capable of carrying the electric current. Beyond repeating the electrolysis, no evidence is afforded that the electrolyte finally discarded is free from lead. Millet's method in its earlier stages is the same as that of Fairhall and, therefore, some objections to Fairhall's method would apply to it. For these reasons, and also, because the method to be described was satisfactory in practice, the three processes mentioned above were not investigated.

FAIRHALL'S METHOD.— Fairhall's method, however, as it has been so largely used, requires fuller consideration. It depends in the first place on the precipitation of the lead, together with calcium phosphate, when the urine is made ammoniacal. The precipitate is filtered on paper, dried and ashed in a muffle at 500° C., the residue being extracted with nitric acid to remove the lead. The lead is subsequently precipitated under carefully controlled conditions, first, as lead sulphide; secondly, as lead sulphate; thirdly, as lead sulphide; and, finally, as lead chromate, the precipitate in each case being allowed to stand overnight before being filtered from its solution. Finally, the lead is determined colorimetrically by making use of the reaction between lead chromate and di-phenyl carbazide, which is oxidised by the chromate radicle, giving a pink colour. The process is long, taking about six days to complete.

A consideration of the Fairhall method suggested the following points for investigation or modification:

(1) It seemed improbable in a biological fluid, such as urine, that the whole of the lead would be in a form precipitable by ammonia. Moreover, Fairhall states that precipitation of the lead is complete only if the urine is fresh. This condition cannot always be satisfied.

(2) It seemed desirable to avoid the risk of loss of lead by ashing at any stage in the method.

(3) When the final lead solution is determined colorimetrically it seemed preferable to make use of a reaction dependent on the lead ion rather than on the chromate ion.

(4) The process was too long, and a shorter method consistent with the requisite degree of accuracy was desirable.

(5) No figures are given showing the magnitude of the error due to the presence of lead in the reagents used in the analysis, but it is stated on page 362 of "Experimental Studies on the Effect of Ethyl Gasoline and its Combustion Products" (*Bureau of Mines*, Washington, 1927) that "blank determinations should be made frequently on lead free material as a check against contamination from apparatus or reagents." Kehoe and Edgar, in *A Study of the Hazards Associated with the Sale and Distribution of Ethyl Gasoline*, 1925, p. 20, state that "lead-free reagents were employed throughout," but no mention of blank determinations is made.

The method to be described was devised, having regard to these five points. It has been tested, with satisfactory results, with urine, to which known proportions of lead in the form of lead hippurate have been added. The method was also used to show that lead is not always precipitated completely by ammonia from normal urine, even when the urine is quite fresh. This has also been shown by Taylor (*loc. cit.*).

II. Outline of the New Method.—An outline of the method indicates how the five considerations stated above were met. The stages of the method are:

(1) The whole of the urine is reduced to the state of a solution of inorganic salts by a process of wet combustion. This avoids the precipitation of the lead in a solution containing organic matter. Since approximately 90 per cent. of the organic matter in urine is urea, and as the salts of urea are comparatively stable, it was realised that this substance should be destroyed before attempting to remove the other organic matter by hot strong acids. This can be done by nitrous acid or an alkali nitrite, but the use of these substances was attended by some experimental inconveniences, such as an excessive volume of solution or difficulty in procuring alkali nitrites free from lead. We are indebted to Dr. Fox, Deputy Government Chemist, for the suggestion to use for this purpose, nitrosyl-sulphuric acid. The suggestion was adopted and proved entirely satisfactory in practice.

(2) After the volatilisation of silica, separated by the process of wet combustion, the lead is precipitated from the solution as lead sulphide, together with copper sulphide, the copper having been added to aid the precipitation of the lead.

(3) After the mixed sulphides have been washed with a solution of sodium sulphide, they are dissolved in nitric and sulphuric acids and the solution is evaporated to dryness. The mixed sulphates are dissolved in dilute nitric acid, and the solution is electrolysed, the lead being deposited on the anode as lead peroxide. This process shortens the duration of the analysis.

(4) The lead peroxide is dissolved from the anode, and the lead is precipitated from the solution as lead sulphate, leaving behind traces of bismuth, manganese and platinum.

(5) The lead sulphate is dissolved in a solution of ammonium acetate, and the lead in solution is determined colorimetrically as lead sulphide. This reaction depends on the lead ion.

(6) Blank determinations were made with the actual quantities of the reagents used in each set of experiments. The magnitude of the very small "blanks" obtained is given later.

At no stage in the process is there any "ashing" of a precipitate or filter paper, these being invariably destroyed by wet combustion. Normally, the process takes three and a half days to complete.

III. TESTING OF THE METHOD.—The method has been tested and found satisfactory, both as regards its individual stages and as a whole. Experiment has shown that no loss of lead occurs at those stages in the method involving filtration, washing of precipitates or electrolysis. These stages comprise:

(1) The precipitation of lead sulphide and the washing of it with water saturated with hydrogen sulphide. When the filtrate and wash water, to which a further quantity of copper nitrate has been added, is again saturated with hydrogen sulphide, no further precipitate of lead sulphide is obtained.

(2) Washing of the mixed sulphides with sodium sulphide. When the sodium sulphide washings are heated with sulphuric acid and the acid solution is subsequently electrolysed, no lead peroxide is deposited on the anode.

(3) The electrolysis. When small quantities of lead, varying from 0·015 to 1·1 mgrm., are electrolysed under the conditions to be described, the whole quantity of the lead is deposited on the anode and no lead is found on the cathode. A second electrolysis does not yield any further lead, and no lead can be detected in the electrolyte.

(4) The precipitation of the lead as lead sulphate. The filtrate from the lead sulphate obtained from normal urines gives a coloration with sodium sulphide corresponding with quantities of lead varying from 0·003 to 0·007 mgrm. But this coloration is, in part, due to traces of platinum, and possibly also to bismuth. The possible loss of lead at this stage is, on the average, less than 0·005 mgrm.—a negligible quantity. That no lead sulphate remains on the filter paper after it has been washed with ammonium acetate, has been shown by the destruction of the filter paper by means of sulphuric acid and the examination of the resulting solution by the sulphide colorimetric process.

The whole process has been tested by the addition of known quantities of lead hippurate to urine, with the following result:

Lead (Pb) added to one litre of urine. Mgrm.	Lead (Pb) found. Mgrm.
0·021	0·024
0·021	0·015
0·042	0·040
0·063	0·067
0·106	0·116
0·265	0·255
0·530	0·535

Since the lead is determined finally by means of the colour produced in alkaline solution on the addition of sodium sulphide, it is essential that the solution to be

examined shall be free from all metals that give coloured sulphides. This condition is met in the following manner. All these metals, except traces of bismuth, are removed by the sulphide precipitation or by electrolysis. The lead is separated from the last traces of bismuth by precipitation as lead sulphate. Under the conditions to be described the lead can be completely separated from 1·0 mgrm. of bismuth, but this quantity of bismuth will never be present at that stage of the process, since most of it has been removed during electrolysis. The greatest quantity of bismuth found on the electrode with the lead during the progress of the work was 0·03 mgrm.

As the proportion of lead in urine is very small, it is essential to avoid, as far as possible, any adventitious gain of lead during the procedure, and, as this cannot be avoided altogether, it becomes necessary to know exactly how much lead is gained. The work was carried out in new silica vessels, and special precautions were adopted to prevent the access of dust during the process. All the reagents were specially prepared, and, wherever possible, from materials purifiable by volatilisation. A "blank" determination was made on all the reagents used for the process with each set of determinations, these being usually four in number, sometimes two, and occasionally six. Normally these "blanks" have varied from 0·002 to 0·005 mgrm. of lead (Pb), and averaged 0·004 mgrm.; in the early stages of the work three "blanks" of greater magnitude were obtained. These were 0·012, 0·013 and 0·010 mgrm., bringing the average for all the 24 "blanks" to 0·005 mgrm.

IV. Preparation of the Reagents.—*Water.*—The distilled water was tested periodically in portions of 1 litre, and found to be free from lead. If a metal condenser is used, care should be taken to avoid soldered joints, otherwise lead will be found in the distillate.

Hydrochloric Acid.
Nitric Acid.
Sulphuric Acid.

These were redistilled from a still made completely of clear silica ware and were stored in stoppered flasks of clear silica.

Hydrofluoric Acid.—This was redistilled from a still made completely of platinum and stored in a platinum bottle.

Nitrosyl Sulphuric Acid.—This reagent was prepared as follows:—Four hundred ml. of redistilled nitric acid (sp. gr. 1·4) were placed in a silica beaker of 800 ml. capacity and cooled in a bath of crushed ice. Gaseous sulphur dioxide from a syphon of the liquid substance was led into the cooled acid through a large trap consisting of an empty flask of 1500 ml. capacity, the tube dipping into the nitric acid being made of silica. The gas was bubbled slowly into the acid at such a rate that it was absorbed completely. When crystals began to form at the bottom of the beaker, the silica tube was progressively raised to prevent the incoming sulphur dioxide from coming into contact with them, since they react with sulphur dioxide to form sulphuric acid. The reaction is complete in 16–24 hours, when the liquid becomes pale green in colour. The liquor and crystals

were well mixed and divided into 5 equal portions, each of which was sufficient to destroy the urea of 1 litre of urine.

Ethyl and Amyl Alcohols.—These were redistilled from a glass still, the first and last runnings being rejected.

Citric Acid Solution.—A 10 per cent. solution of citric acid in water was made from citric acid that had been tested and found free from lead.

Ammonia (Selected).—Fifty ml. of ammonia (sp. gr., 0·88) must show no coloration on the addition of 2 drops of 10 per cent. sodium sulphide solution. It must also be free from sulphide.

Copper Nitrate Solution.—Electrolytic copper containing 0·02 per cent. of lead was dissolved in nitric acid and re-electrolysed. The deposited copper was dissolved in the minimum quantity of nitric acid, and the solution diluted until 1 ml. contained approximately 2 mgrm. of copper (Cu).

Sodium Sulphide Solution.—A 20 per cent. solution in water was made, allowed to stand for some days and then filtered. This strong solution was diluted with four volumes of water to make a 4 per cent. solution.

Ammonium Acetate Solution.—A 10 per cent. solution was made from selected specimens of the crystallised solid that were free from lead, copper, iron, and sulphide.

"Masked" Methyl Orange Indicator. (Hickman and Linstead, *J. Chem. Soc.*, 1922, p. 2502.)—One grm. of methyl orange and 1·4 grms. of Xylene Cyanol F.F. were dissolved in 500 ml. of 50 per cent. alcohol (by vol.).

Potassium Cyanide (Selected).—Ten ml. of a 10 per cent. solution must give no reaction for lead and sulphide.

Ten Per Cent. Sodium Sulphide Solution, for the colorimetric determination of lead:

Strong solution for stock: { Pure Na_2S crystals, 50 grms. / Pure glycerin, 50 grms. / Water to 250 ml. }

This stock solution, which keeps well, was diluted with an equal volume of water before use.

V. METHOD OF SAMPLING.—The urine was collected in selected Winchester quart bottles. Seventy-two bottles, all of the same make and delivery, were chosen after tests had been made on three of them, selected at random from the 72. The tests were:—(1) In each of the three bottles were placed 50 ml. of hydrochloric acid of sp. gr. 1·1, the bottles and their contents were heated for 8 hours in a boiling water-bath and agitated at half-hour intervals. The lead dissolved by the hot acid was found to be 0·004 mgrm. (2) Fifty ml. of ammonia, of sp. gr. 0·88, was allowed to remain in each of the bottles for 8 hours, the bottles being agitated at half-hour intervals. At the end of that time no lead was detected in the ammonia.

A similar number of glass funnels, 4½ inches in diameter, were tested in the same manner by having hot acid and concentrated ammonia poured through them repeatedly over a period of 1 hour, but no lead was detected in the acid or alkaline solutions.

The bottles and funnels were thoroughly cleansed before use by rinsing first with 250 ml. of hot dilute nitric acid (1 to 10), next with a copious stream of tap-water, and finally with distilled water. The clean, well-drained bottles and funnels were placed in padded baskets and dispatched from time to time to the source of origin of the samples.

The urine from males only was examined. It was collected directly into the bottles, transference of the urine from one vessel to another being thus avoided. A full 24-hour supply was collected in each case, usually from 3 p.m. on one day to 3 p.m. the next.

VI. Details of the Method of Analysis.—(1) *For Urine.*—The total volume of the sample is measured. The drained bottle is washed with 50 ml. of hot dilute nitric acid (1 vol. of acid, sp. gr. 1·4 diluted with 9 vols. of water) until every trace of deposit has been removed from the sides and bottom of the bottle. Whenever possible, 1000 ml. of urine are taken for analysis, and to it is added its appropriate fraction of the nitric acid washings of the bottle.

(*a*) *Destruction of the Organic Matter.*—To 1 litre of the urine and its appropriate fraction of washings contained in a two-litre silica beaker, nitrosyl sulphuric acid, prepared from 80 ml. of concentrated nitric acid, is added gradually (*i.e.* one of the five portions obtained as described above). Repeated addition of small quantities (10 drops) of redistilled amyl alcohol is necessary to prevent frothing caused by the gaseous products of the reaction between the nitrous acid and urea. To minimise local action and consequent loss of nitrous acid, the nitrosyl sulphuric acid should be stirred frequently during its addition. The urine must, of course, be stirred constantly during the addition of the nitrosyl sulphuric acid.

When the vigorous reaction is complete, a few more drops of amyl alcohol are added, and the mixture is boiled down to about one-third of its original volume. After cautious addition of 20 ml. of redistilled conc. nitric acid (sp. gr. 1·4), the beaker is covered and the boiling continued until charring occurs, when a further 3 ml. of nitric acid are added. The heating is continued, with further additions of small quantities of nitric acid, as may be necessary, until the liquid is in a sufficiently clean state for transference to a 600 ml. beaker (any silica adhering to the large beaker should be removed by means of dilute hydrofluoric acid, as described later). After transference, a little more nitric acid is added to the diluted solution, and the boiling down is continued until sulphuric acid fumes are again evolved.

The final destruction of traces of organic matter is completed by boiling the concentrated acid solution vigorously for some time in the covered beaker, adding a few drops of concentrated nitric acid to the hot mixture, if necessary.

To ensure decomposition of residual traces of nitrosyl sulphuric acid, the warm, almost colourless, sulphuric acid solution is diluted, and again boiled

down until fumes of sulphuric acid appear. If this final dilution is omitted, the residual nitrous and nitric acids will interfere with the indicator used in neutralisation, and may prevent the complete precipitation of the lead as sulphide.

The excess of sulphuric acid is now expelled by heating until crystallisation of calcium sulphate just commences. Heating must not be continued beyond this stage, or the calcium sulphate will be rendered insoluble in water.

(*b*) *Removal of the Separated Silica.*—Before actual solidification takes place, the warm syrupy acid solution is diluted cautiously, first with a little cold water, and then with hot water to a volume of about 400 ml. At this stage all the calcium sulphate should be in solution, but it may be necessary to add a little hydrochloric acid and to continue heating for a short time.

The silica is now filtered off on an 11 cm. acid-washed filter paper, and washed once or twice with hot water. Any silica that remains adhering to the silica beaker is removed by means of hot water containing a few drops of hydrofluoric acid, and this solution is transferred to a platinum dish, which is covered and set aside.

The filter paper containing the silica is now transferred to a 250 ml. tall silica beaker and treated with a mixture containing 10 ml. of concentrated nitric acid, 10 ml. of water, and 3 ml. of concentrated sulphuric acid. The covered beaker is heated on the hot plate until copious fumes of sulphuric acid are evolved, when the cover is removed for two or three minutes. (*Note.*—If charring occurs, a few drops of concentrated nitric acid are added and heating continued.) The contents of the beaker are now transferred to the platinum dish, previously mentioned, a few drops of hydrofluoric acid again being used, if necessary, to ensure complete transference of silica. After the addition of 1–2 ml. of hydrofluoric acid to the solution in the platinum dish, the silica is volatilised by evaporation of the solution on the hot plate to the fuming stage. (*Note.*—In cases where difficulty has occurred in bringing about complete solution of calcium sulphate, some of which may have been filtered off with the silica, the heating with concentrated sulphuric acid in the dish must be continued until all the sulphate passes into solution in the hot concentrated acid.) Upon transference to the beaker containing the original solution, there should be no further difficulty in obtaining a perfectly clear solution.

(*c*) *Precipitation of the Lead Sulphide.*—To the clear colourless solution 5 ml. of a 10 per cent. solution of citric acid and 5 ml. of a solution containing approximately 2 mgrms. of pure copper per ml. are added, followed by a few drops of "masked methyl orange" indicator. Concentrated ammonia (sp. gr. 0·880) is now run in, with constant stirring, when the colour of the solution will gradually change from red to purple, and finally to a neutral greyish tint. At this point the addition of one or two more drops of ammonia will cause a change to green (P_H about 4–5), the solution being now only slightly acid to litmus. This is the degree of acidity which has been found to be suitable for the precipitation of the sulphides. Hydrogen sulphide is now passed through the solution for one hour.

After the precipitated sulphides have been allowed to settle (15–30 minutes—not longer), the supernatant liquid is decanted through an 11 cm. acid-washed

filter paper, which must be free from pinholes. Finally, the sulphides are transferred to the paper and washed twice with hydrogen sulphide water, and then with 10–15 ml. of warm (40–50° C.) 4 per cent. sodium sulphide solution to remove sulphides of arsenic, antimony and tin. If the filtrate is not quite bright, it must again be passed through the filter.

The paper containing the sulphides is now destroyed by wet combustion in a tall 250 ml. silica beaker, as described previously for the silica separation, and, when all organic matter is destroyed, the excess of sulphuric acid is expelled, the beaker being removed from the hot plate when practically all the acid has evaporated, and the remaining acid being driven off by gentle blowing, while the beaker is still hot. The lead and copper are now in the form of sulphates.

(*d*) *Electrolytic Deposition of Lead Peroxide.*—To the tall 250 ml. silica beaker containing the dry sulphates, 10 ml. of water are added and then concentrated (0·88) ammonia, drop by drop, until any free sulphuric acid is neutralised, this being shown by the appearance of the blue colour due to copper. Fifteen ml. of water containing 1 ml. of concentrated redistilled nitric acid are then added, and the resulting solution electrolysed, under the following conditions:—Temperature, 70–80° C.; voltage, 1½–2 volts; current density, 0·3–0·4 amps./100 sq. cm.; speed of rotation of anode, 1500–2000 revolutions per minute.

The anode is a cylinder of platinum iridium (25 per cent. of iridium) foil, 1 cm. deep and 1 cm. diameter, joined centrally to a platinum iridium wire, about 12 cm. long, and is rotated at the above-mentioned speed. The cathode is a plate of platinum iridium foil, 1½ cm. square, also joined to a wire. Before electrolysis the cathode has a thin coating of copper deposited on it electrolytically. The beaker is fitted through the lid of a water bath so that it remains suspended by the rim, the temperature of the bath being kept between 70° and 80° C. By means of a sliding resistance the voltage from a 4 V accumulator is cut down until the current through the electrolyte is 20 milliamps, this giving the correct current density. The dimensions of the beaker are such that when the electrodes are at opposite sides and this current passes, the voltage drop between the electrodes is 1·6 volts. The distance between the electrodes is then about 4 cm. The beaker is covered with a split clock-glass, and the electrolysis continued for 1 hour, after which the motor rotating the anode is stopped, and the anode removed from the solution and washed.

(*e*) *Precipitation of Lead Sulphate.*—The lead peroxide is dissolved from the anode in a 100 ml. tall silica beaker by heating for 30 minutes in a boiling water-bath with a mixture containing 25 ml. of water, 0·5 ml. of concentrated nitric acid, and 5–10 drops of alcohol. The anode should now be visibly free from lead peroxide, and should give no coloration when tested with Trillat's reagent (*Compt. rend.*, 1903, **136**, 1205). Redistilled concentrated sulphuric acid (0·5 ml.) is now added to the solution, and the mixture evaporated on the hot plate to complete dryness (to remove all nitric acid). A further 0·5 ml. of concentrated sulphuric acid is now added, and the beaker is covered and again heated to the fuming point for a few seconds. After cooling, 15 ml. of a mixture containing 1 vol. of alcohol (about 94 per cent. by

volume) to two volumes of water are added, and *thoroughly mixed with the acid.* The mixture is allowed to stand overnight, when the lead sulphate is filtered off on a 5 cm. acid-washed, close filter paper, and washed twice with a mixture containing alcohol, water, and concentrated sulphuric acid in the respective proportions by volume 10, 20 and 1. Ten ml. of 10 per cent. ammonium acetate solution are now boiled in the beaker in which the sulphate precipitation was carried out, and the hot solution is passed through the filter into a similar beaker. This operation is repeated, the same 10 ml. of ammonium acetate being again raised to boiling, passed through the filter, and collected in the first beaker. The filter is finally washed three times with about 5 ml. of hot water containing a little ammonium acetate.

(*f*) *Colorimetric Determination as Lead Sulphide.*—An opinion as to the approximate quantity of lead present having been formed by inspection of the electrode after electrolysis, the whole, or a suitable proportion of the ammonium acetate solution, is transferred to a tall 50 ml. Nessler cylinder. The best quantity to be used for the matching as lead sulphide under these conditions has been found to be 0·05 mgrm. of lead. An exactly similar cylinder is used for the solution with which it is to be compared, and 10 ml. of the ammonium acetate reagent are placed in this cylinder. To each cylinder are added 2 ml. of 10 per cent. potassium cyanide solution, 5 ml. of approximately 6 *N* ammonia, water to the 50 ml. graduation, and, finally, with constant stirring, 2 drops of the special 10 per cent. sodium sulphide solution. A solution containing 0·01 mgrm. of lead per ml. is now run from a burette into the cylinder containing the control solution, until a match is obtained.

The solution in the cylinder containing the control is now rejected, and a fresh control prepared containing all the reagents (except the sulphide) and a volume of the standard lead solution which is less by 1 ml. than that added in the first comparison. The sulphide is added last, followed by a little more lead solution as may be necessary to produce a perfect match. The burette reading may then be recorded. The cylinders should be viewed both with the cylinder containing the control on the left and also on the right of the cylinder containing the solution to be examined.

By working with 0·05–0·10 mgrm. of lead, the results obtained colorimetrically are within 5 per cent. of the exact amount present.

(2) For Biological Materials.—The method has been applied, with slight modification, to biological materials, the essential difference being the omission of the nitrosyl sulphuric acid. The material is first heated in a covered silica beaker with dilute nitric acid, containing 10 per cent. by volume of nitric acid (sp. gr. 1·4) until most of the solid matter has been disintegrated. The destruction of the organic matter is then completed by the action of concentrated sulphuric and nitric acids, and the process is then continued as described above.

(3) For Miscellaneous Materials.—Organic matter, if present, is first destroyed by wet combustion with strong nitric and sulphuric acids, the method

then being continued as described in VI (1) (*b*) above. It should be noted, however, that the presence in the electrolyte of relatively large quantities of iron salts causes an incomplete deposition of the lead during electrolysis. If, on neutralisation of the electrolyte during the process of adjusting the acidity of the solution prior to electrolysis, a red precipitate of ferric hydroxide should be observed, it is advisable to reverse the sequence of the electrolysis and the separation of the deposited lead as lead sulphate.

VII. RESULTS.—Fifty-five samples of normal urine from persons residing in London and the surrounding country districts gave quantities of lead varying from nil to 0·133 mgrm. of lead (Pb) per litre, the average value being 0·040 mgrm. of lead per litre.

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Official Appointment.

THE Minister of Health has confirmed the following appointment :

MR. A. E. JOHNSON, B.Sc., F.I.C., as Public Analyst for the County Borough of Wolverhampton (November 11, 1929).