

when the relapse immediately follows than when its onset is delayed one or two weeks.

We are still ignorant of the factors concerned in the production of this re-infection, but I think some peculiarity in the individual patient or in the micro-organism will yet be shown to play a prominent part, since there seems to be a great tendency in families to one type of attack, and the prevalence of different complications, including relapses, seems to vary considerably in successive epidemics.

I think that from a review of these 200 cases it must be concluded that a careful system of dieting, such as I have mentioned, has no injurious consequences and when one considers the benefits obtained—viz., more rapid recovery, diminished risk of surreptitious feeding with possibly harmful substances, lessened tendency to bolt food when allowed without proper mastication, and, finally, lessened tendency to asthenic complications as post-typhoid anæmia, gangrene, &c.—one must admit they there is no justification for resisting a craving appetite in the manner at present in vogue.

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A SIMPLE AND TRUSTWORTHY METHOD FOR THE QUANTITATIVE ESTIMATION OF MERCURY IN URINE.

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IN 1895 A. Jolles in Vienna published a memoir¹ entitled "A Simple and Delicate Method of Qualitative and Quantitative Estimation of Mercury in Urine." In the introduction Jolles gives an account of the methods hitherto proposed and adopted and comes to the conclusion that none of them fulfil the conditions which are essential in a practically useful analytical method—namely, the combined properties of simplicity and speed in working, with trustworthiness and accuracy in the results. Jolles then details a method for which he makes the claim that it combines these advantages. He recommends the use of metallic gold (which he obtains in granular form by an ingenious process) so as to form an amalgam with the mercury, when the latter is precipitated by the addition of stannous chloride. The use of the granular gold Jolles holds to be a great advance on former methods in which zinc, brass, wool, copper wire gauze, &c., were used. It readily takes up the mercury when this is precipitated by stannous chloride, and is then present in finely divided particles and in the "nascent" state. The whole operation according to Jolles takes at most 20 minutes.² In order to test these statements we prepared granular gold strictly according to the directions given by Jolles and with it made experiments, by his method, on mercurial solutions of different strengths. It was evident, however, that in no instance did complete amalgamation take place. As soon as the stannous chloride was added to the solution of bichloride of mercury, which was heated to 70° or 80° C., a more or less distinct clouding occurred, or a greyish precipitate of metallic mercury. The mixture was thoroughly stirred, shaken, and gently heated for a long time in order to bring the gold and mercury as much as possible into intimate contact, but in the end it always remained turbid, an evidence that the mercury had not been fully taken up by the gold. The mixture was allowed to settle and we tried to amalgamate with the granular gold the grey to black-coloured mercury which had all settled on the bottom. This only took place partly. A comparatively large quantity of free mercury always remained over.

We therefore consider it certain that it is impossible to effect a complete amalgamation of small quantities of mercury with gold in fluid media. Firstly, it was evident that the grey to black turbidity did not depend upon combinations with the tin; moreover, we took care that there was an excess of hydrochloric acid always present both in the clear, filtered solution of stannous chloride and in the

mercurial solution. In many trials, which we varied in every possible way by the use of gold in smaller or larger granules, in the finest spongy form, and finally in "leaf" form, we obtained no better result. After our experiments we therefore cannot agree with Jolles's conclusions. L. Böhn³ recommended another method of quantitatively estimating mercury in the urine which Rud. Winternitz published⁴ and improved upon.⁵ Winternitz obtained good results with his method, but it is hardly practical, both on account of the technical details in carrying it out and also on account of the length of time, from three to four days, which it requires. As our object was the estimation of the amount of mercury in the urine of syphilitic patients we needed a method which would combine the accuracy of Winternitz's method with the practicability and rapidity of that of Jolles. We therefore thought that we should attain our purpose if we could suitably combine the two methods mentioned. We then tried to obtain a complete union of the two metals by bringing the metallic gold for as long a time and as thoroughly as possible in contact with the solution containing the precipitated mercury. With this object in view we made a U tube of hard (potash) glass and partly filled it with the granular spongy gold. On one limb of the tube a bulb was blown to prevent small particles of gold from being carried over mechanically with the fluid. With the like object we placed two little glass rods in both limbs of the tube. We poured in corrosive sublimate solutions of different strengths, mixed with hydrochloric acid and stannous chloride. We tried it both warm and cold and poured it in both quickly and quite slowly, and even drop by drop, but we always found that the fluid which came through was more or less clouded with fine particles of precipitated mercury.

We arranged some quantitative analyses as follows. We weighed the carefully dried tube before running in a solution containing a known weight of mercury. After the solution had been slowly passed through we washed out the tube with water, alcohol, and ether, then dried and again weighed it. The difference of weights gave the weight of the metallic mercury which the gold had taken up. It was then again evident that a complete amalgamation of the two metals never took place as the weighings showed a loss of from 30 to 50 per cent. This method, then, as well as that of Jolles seems only useful for qualitative and not for quantitative analyses.

It may be mentioned that we used yet another form of gold, the so-called "true gold leaf," instead of the spongy granular gold of Jolles for filling the tube, but with this also we obtained no better result. It yet remained to prove that the turbidity of the fluid which came over was really due to mercury. We allowed the fluid to settle, and brought the grey to black deposit into prolonged contact with some granular gold, and then poured off the liquid. The gold amalgam was washed with water, alcohol, and ether, and then dried and heated in a test-tube with a small piece of iodine. A reddish-yellow ring of mercuric iodide was formed. We finally succeeded better when we combined R. Winternitz's method of amalgamation with mechanical filtration in the following manner. We prepared a small tube thus: a piece of hard glass tubing (Jena potash glass), with walls that were not too thick, was drawn out at one end in the flame of the blowpipe into a narrower part, and in this portion a thorough mixture of asbestos and "true gold leaf" was placed. We chose true gold leaf so as to get the gold in the finest possible state of division. The mixture was made by rubbing together pure white asbestos and pure gold leaf, washing out with hot hydrochloric acid, hot water, alcohol, and ether, and drying. In the lower portion of the wide part of the tube immediately above the narrow part we put a thick plug of asbestos to prevent any fragments of gold getting through and over it the layer of the mixture. The tube was then connected with a suction bottle, which was in turn connected with a water exhaust-pump. After these preparations, followed by a further thorough washing out with hydrochloric acid, water, alcohol, and ether successively, the apparatus was dried by gently heating with a small Bunsen flame and aspirating a constant stream of air dried by passage through concentrated sulphuric acid. The tube was weighed and the following three experiments were made.

¹ Monatshefte für Chemie, Band xvi., 1885, pp. 684-692.

² Op. cit., p. 689.

³ Zeitschrift für Physiologische Chemie, Band xv., pp. 1-36.

⁴ Archiv für Experimentelle Pathologie und Pharmakologie, 1889,

⁵ Archiv für Dermatologie und Syphilis, 1889, pp. 783-828.

In the first experiment a solution of corrosive sublimate containing six milligrammes of mercury was poured into about 200 cubic centimetres of a heated mixture of distilled water, stannous chloride, and hydrochloric acid, and after allowing it to stand a little the resulting cloudy fluid was aspirated through the filter-tube. The filtrate was clear and remained so after 12 hours, with the exception of some little shreds of asbestos, which had escaped. The tube was carefully washed with warm water, alcohol, and ether, and with hydrochloric acid to get rid of the tin. By means of a suction pump a stream of dry air was drawn through for some time, and by very gentle warming the tube freed from all moisture. The weighing gave 5.2 milligrammes mercury instead of the six milligrammes which had been passed through. On heating the tube to redness a deposit of mercury formed in the narrow neck, which gave the typical reaction with the vapour of iodine. In order to test whether we could get a good result by simple filtration through asbestos we made an experiment in every way similar to the foregoing, but without using gold. We obtained only 3.4 milligrammes of mercury from the six milligrammes passed through. The loss was evidently due to the long continued passage through the tube of a stream of air heated gently in order to dry it. Some pieces of asbestos that were black to start with became obviously lighter in colour: thus a partial volatilisation of the mercury took place. An explanation of the ready volatility of the mercury in this experiment is that here the mercury existed as a free metal instead of being present when gold was used in the amalgam which the latter forms at once, and which requires a greater degree of heat to become decomposed, consequently the mercury present in the free uncombined condition in the asbestos filter is more easily volatilised at a proportionately lower temperature than the mercury contained in the gold amalgam.

The second experiment was conducted in the same way as the first, except that five milligrammes of mercury in about 300 cubic centimetres of water, hydrochloric acid, and stannous chloride were used in order to have a dilute solution. We found 4.3 milligrammes of mercury as the result of the weighings. Here also the slight loss was accounted for by the fact that some little pieces of asbestos were carried through. We therefore altered our procedure in the following manner. Instead of weighing the tube twice, once before filtering the mercurial solution and once with the mercury, so as to weigh the mercury directly, we calculated it indirectly from the loss of weight of the tube after heating it to redness. The tube was weighed after passing the mercurial solution and drying as before. It was then heated to redness and again weighed.

In the third experiment four milligrammes of mercury were dissolved in 500 cubic centimetres, thus making a very dilute solution. The quantity found by this method was 3.7 milligrammes of mercury. From this experiment our method seemed practical, but nevertheless a slight loss occurred in our process which would be of importance in working with still smaller quantities of mercury.

In order to remedy this defect we endeavoured to obtain the gold in yet finer sub-division than we had in the mixture of asbestos and gold leaf and we discovered an excellent material for our purpose in the hitherto unknown "gold asbest," in which the gold is deposited in molecular sub-division on fibres of asbestos. To prepare this substance we dissolved chemically pure gold in aqua regia, and heated it to drive off the chlorine and oxide of nitrogen and until the solution of gold chloride contained only very little free acid. In this solution we placed clean, fine, fibres of asbestos and allowed them to drain after they had been soaked through with the somewhat concentrated solution of gold. Then they were dried in a porcelain dish on the sand bath. The dish was gradually heated over the flame, whilst from above a stream of hydrogen (which had been purified by being passed through permanganate of potash and liquor potassæ and dried with sulphuric acid) was introduced by means of a porcelain tube. After about 15 minutes the reduction of the gold chloride was complete and the asbestos appeared partly coated with finely divided particles of glistening gold. It was cleaned with diluted hydrochloric acid and then dried.

The preparation of "gold-asbest" by means of the double chloride of gold and ammonium which we tried, both with and without the use of hydrogen, is not to be recommended. We now used the "gold asbest" prepared after our method for filling our filter amalgam tube. We finally found that

the most practical way was to put a thick pad of asbestos in the narrowest part of the tube, over this a layer of "gold asbest," then a layer of granular spongy gold, and finally a second layer of "gold asbest." Experiments gave the following results.

| — | Experi- ment I. | Experi- ment II. | Experi- ment III. |
|--|--------------------|---------------------|----------------------|
| Decimilligrammes of mercury in the form of corrosive sublimate in each litre of water | 40 | 30 | 20 |
| Decimilligrammes of mercury found | 41 | 30 | 20 |

We afterwards worked with still more dilute solutions :—

| — | Experi- ment I. | Experi- ment II. |
|---|--------------------|---------------------|
| Decimilligrammes of mercury added to each one and a half litre of water | 20 | 20 |
| Decimilligrammes of mercury found | 21.5 | 20 |

These experiments show distinctly the usefulness of our method for quantitatively estimating mercury in very weak watery solutions of corrosive sublimate. They should be carried out in the following way.

The dilute mercury solution should be heated on the water bath to about 70° C., then mixed with about 50 cubic centimetres freshly prepared, clear stannous chloride solution. After the solution has cooled a little it is aspirated through the filter amalgam tube already described, which is then washed with moderately warm diluted hydrochloric acid and afterwards three or four times with warm water. Finally, the water is got rid of with alcohol and ether, and the tube, after a slight warming at about 40° C., is dried by drawing a stream of dry air through it. The mercury forms a grey ring varying in tint from light to dark in the "gold asbest" layer in the tube. After drawing the air through for a quarter of an hour the tube is perfectly dry and is then weighed. Afterwards it is again connected with the stream of dry air and heated to redness in the Bunsen flame. After a little heating a precipitate of metallic mercury forms in the narrow part of the tube. The tube is left in the dry air apparatus until cold and then again weighed. The difference between the two weights gives the weight of mercury which was present in the solution filtered.

After obtaining such accurate results with watery solutions of corrosive sublimate by our method we did not hesitate to apply it to urine. Two milligrammes of mercury in the form of corrosive sublimate were mixed with a litre of normal urine free from mercury and the whole was warmed on the inverted condenser with hydrochloric acid and chlorate of potash till steam was given off in order to destroy the organic substances. The somewhat cooled urine was passed through a folded filter and stannous chloride and hydrochloric acid were added to the clear filtrate. From this point the working was exactly as described above. It appeared, however, that some organic substances which had not been destroyed were thrown down with the mercury in the asbestos. On ignition empyreumatic smelling gases escaped and the loss on heating showed 6.4 milligrammes instead of two milligrammes. We had then to devise some means of getting rid of the organic substances which remained after the first treatment with chlorate of potash and hydrochloric acid. We succeeded by repeated thorough treatment with chlorate of potash and hydrochloric acid, by which, it is true, some loss often occurred on account of the copious frothing. At times an almost explosive compound of the chlorate of potash and the organic substance was formed on warming.

Two experiments in which care was taken that there was no loss gave the following results: (1) 40 decimilligrammes of mercury were added to one litre of normal urine, and 36 decimilligrammes of mercury were found; and (2) 20 decimilligrammes of mercury were added to one litre of normal urine and 19 decimilligrammes of mercury were found. We now made extensive experiments with urine containing mercury from syphilitic patients, and finally employed our method, after many technical alterations and improvements, in the following way. One litre of urine in a two-litre retort flask of potash

glass was placed on the water bath and from 15 to 20 grammes of chlorate of potash were added together with about 100 cubic centimetres of strong hydrochloric acid. The mixture was warmed until the development of the chlorine was made apparent by the mixture becoming lighter in colour. It is advisable to attach a short inverted condenser. By the action of the nascent chlorine the organic constituents are for the most part destroyed and the mercury which is presumed to be in combination with them is changed into bichloride of mercury. The retort was then taken from the water bath and allowed to stand for 12 hours in order that the chlorine might act on the organic substances for a sufficiently long time. Afterwards it was again gently warmed and about 100 cubic centimetres of clear stannous chloride solution (prepared by warming excess of tin in concentrated hydrochloric acid and filtering through a hard filter) were added. After cooling a little it was filtered through an asbestos filter by means of the exhaust-pump and slightly washed. The asbestos filter was prepared from pure shredded asbestos on a porcelain filter which was placed in a moderately sized ordinary glass funnel. What adhered to the sides of the funnel was washed down with slightly warmed water from the wash-bottle, otherwise it adhered very firmly. The deposit, which along with the organic substances containing the mercury, together with a little hydrate of potash and some water, was placed in a retort holding about 300 cubic centimetres, into which it was entirely transferred by washing the funnel with warm liquor potassæ. The retort was placed in the water bath, slightly warmed, and allowed to cool. The organic substance was thus brought into solution and the action of the chlorine later was more thorough and took place quite quietly. Then (after the cooling) some crystals of chlorate of potash and concentrated hydrochloric acid were added. The chlorine formed destroyed the remainder of the organic substances entirely, and the mercury was all brought into solution as chloride. The solution was then filtered, best with the suction-pump (through a small filter plate on which there was a round close-fitting filter-paper, washed with as little water as possible). Afterwards the warm solution was mixed with from 10 to 20 cubic centimetres of stannous chloride and filtered through a filter amalgam tube filled with "gold asbest" and finely divided granular gold. The smallest particles of mercury were retained by this. The filter tube was then washed with diluted hydrochloric acid and water and afterwards three times with alcohol, then three times with ether, well dried in the dry air stream by which it was warmed a little at the start, and weighed till the weight was constant. Then the mercury was volatilised, the air current being again passed through. It had to be heated strongly as the "gold asbest" was a very bad conductor of heat, and the heating had to go on till the gold amalgam was thoroughly decomposed and the mercury entirely driven off. The tube was then weighed again till the weight was constant. The weight of the mercury contained in the sample of urine was the difference between the two weights. Three test experiments made by this method gave the following results:—

| | Experiment I. | Experiment II. | Experiment III. |
|--|---------------|----------------|-----------------|
| Mercury (decimilligrammes) added to each litre of normal mercury-free urine | 25 | 44 | 0 |
| Mercury (decimilligrammes) found in urine | 28 | 37 | 5 |

A series of three experiments was then made with pathological urine, each litre of which on an average of many analyses contained 20 decimilligrammes of mercury.

| | Experiment I. | Experiment II. | Experiment III. |
|--|---------------|----------------|-----------------|
| Decimilligrammes of mercury added per litre | 30 | 40.5 | 67 |
| Decimilligrammes of mercury already present | 20 | 20 | 20 |
| Totals | 50 | 60.5 | 87 |
| Decimilligrammes of mercury found | 60 | 55 | 77 |

We think that these figures sufficiently prove the usefulness of our method with respect to accuracy, and it can be carried out in a proportionately quick and easy way when once one has the apparatus for its performance, so that, with sufficient practice, three or four analyses can be made in 24 hours. Our method is very well suited to carrying out an extensive series of experiments which we have undertaken on the amount of mercury eliminated in the urine of persons who were undergoing a course of inunctions.

In conclusion we can confidently state that the method in the case of urine containing at least one milligramme of mercury per litre, by proper and careful work, according to our directions, gives absolutely accurate results. The results, as shown in the figures supplied, in cases where the urine contained less than one milligramme per litre, are only approximate. Our method has the advantage over that of Winternitz of being considerably quicker and easier in carrying out, and over the method of Jolles of being more certain and accurate with almost the same readiness and facility in working. The presence of an iodide does not interfere with the results obtained.

PROTRACTED GESTATION.

BY JOHN PHILLIPS, M.B., C.M., L.R.C.P. EDIN.

THE question of the duration of pregnancy being of considerable interest, not only from a medico-legal point of view, but also from that of general practice, I venture to report what I believe to be an authentic case in which it was prolonged to much beyond the usual period.

My patient was a primipara and unmarried. Intercourse was declared to have taken place on one occasion only—Sept. 6th, 1898—the girl's age at that time being only 16 years and four months. Her menstrual periods had occurred at regular intervals and they usually lasted about a week. Her last period had occurred in August, 1898, and had entirely ceased by the 24th of that month, this being the first day on which she considered herself "well," and it was from this date that I calculated the probable duration of pregnancy. In consequence of her missing her next two periods in September and October her aunt with whom she lived took her on Oct. 24th to a medical man in London, to whom the girl confessed, when questioned, that she had had intercourse, and he expressed the opinion that she was most probably pregnant. This proved to be the case and in April of the present year she came under my observation, and I was asked to attend her in her confinement, which I calculated would take place somewhere about May 29th, this being the usual average period of 278 days from the cessation of her last menstrual period (August 24th, 1898), from which I reckoned. Instead, however, of the confinement occurring at the expected time she not only went through the whole of June without a single pain, but labour did not set in until July 13th, on which date the pains began at 6 P.M.—323 days after the cessation of her last period and 310 days after the date of coitus, the child being born on the following day at 4.30 P.M. There is not the slightest doubt that no menstrual period occurred subsequently to that in August, as in addition to the girl's own statement her aunt, who always looked after her linen and who consequently always knew when her niece was unwell, declares that there "was not a stain" subsequently to the August period. During the entire pregnancy the girl had excellent health and there were no pains of any description prior to the onset of labour, which was perfectly normal and the presentation the usual cephalic one. Unfortunately at the time I had no opportunity of having the child (a female) weighed as it was sent away to be nursed almost immediately after its birth (within three hours), but although well proportioned and of good size it did not seem at all unusually large.

The late Dr. Matthews Duncan in his book, "Fecundity, Fertility, Sterility, and Allied Topics" (p. 348), is inclined to disbelieve in any case of supposed protracted gestation unless the size and weight of the child are above the average, considering that increased growth must follow lengthened gestation, but this cannot be taken as proved and, as Dr. Playfair points out, even if it be admitted the foetus may have been originally small, so that at the end of the protracted gestation it may be of no more than the average weight and