THE ANALYST.

JULY, 1892.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The usual Monthly Meeting of the Society was held on Wednesday, 1st June, the President (Mr. Hehner) occupying the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed as members:—A. P. Aitken, D.Sc., Chemist to the Highland and Agricultural Society of Scotland, 8, Clyde Street, Edinburgh; Frederick Wm. Tompson, F.C.S., Chemist in the employ of Messrs. Bass & Co., Brewers, Burton-on-Trent, Stapenhill Road, Burton-on-Trent; and John Williams, Private Assistant to Professor J. B. Harrison, Government Analytical Chemist, British Guiana, Georgetown, Demerara, was proposed as an associate.

Messrs. Samuel Francis Burford, F.C.S.; James William Westmoreland, F.I.C., were duly elected members of the Society; and Messrs. G. H. Gabb and W. N. Yarrow, were duly elected associates.

Mr. H. Droop Richmond read the following "Notes from the Khedivial Laboratory, Cairo":—(7) "On a Sample of Milk yielded by an Arab Woman." (8) "Egyptian Flour." (9) "Some Egyptian Waters." (10) "The Manganese beds of Assiout." The publication of these papers is unavoidably held over until next month.

In the absence of Dr. Leather, Mr. Dyer read the following paper:—

THE DETECTION OF CASTOR OIL SEED AND OF CROTON SEED IN FEEDING STUFFS.

By J. WALTER LEATHER, Ph.D., F.I.C.

As is well known, both castor oil seed (*Ricinus communis*) and the seeds of croton (*Croton tiglium*) are very poisonous.

A small admixture, even considerably less than one per cent., of either of these seeds, when present in a cattle food, is sufficient to cause harm and even death. An ordinary

search with the microscope for any seed existing in smaller proportion than one per cent. would, in any case, be not only very tedious, but might fail to detect the constituent.

The difficulties attending a microscopical search for either of these seeds are, however, considerably increased on account of the "seed case" of each being quite impervious to light, and the structure being, in consequence, indiscernable.

The method which I have employed for some time past for the microscopical examination of foods, so far as the recognition of the testa of seeds is concerned, is, firstly, to digest a portion of the material with dilute HCl or H_2SO_4 (1 to 2 per cent.) for half-an-hour on the water-bath; then to remove the acid liquid by decantation and to wash the material several times in the same manner; secondly, to digest it with equally dilute KHO or NaHO and to repeat the washing by decantation. In this way I have found that the testæ of all those seeds which one generally meets with in cattle foods, and also the impurities which frequently accompany them, are rendered sufficiently transparent to be readily recognized under the microscope. After washing the alkaline liquid away, it is sometimes an advantage to wash the material with dilute HCl, which reduces the strong colour produced by the action of the alkali. I do not lay any claim to this process, for I believe that many chemists employ both acid and alkali for the above purpose, but I might mention that I have not met in text-books with any directions for their use in the manner I have indicated.

Now, if either castor oil-seed or croton seed be subjected to this process, the outer integument of each is washed away, and the principal part of the testa remains black and opaque. At the edges some indication of the structure can frequently be seen.

In any case, owing, as I have said, to the fact that it requires but a very small quantity of either of these seeds, probably 3 or 4 seeds, or less, per lb., to cause serious illness or death to stock, the examination with the microscope of one or two grammes of a material could not be expected to expose their presence with certainty.

During a search, some months ago, for these seeds in some decorticated cotton cake, I subjected a number of pieces which had been picked out with the aid of a pocket lens, to the action of a solution of sodium hypochlorite. I had previously employed this agent to bleach similar materials for another purpose, and I had always found that it removed the colour entirely after a few hours. I therefore hoped that I should in this way obtain a view of the structure of castor oil seed and of the pieces above referred to. To my surprise, however, they remained perfectly unbleached, even at the end of several days. I then subjected a number of pieces of the testæ of both castor oil seed and croton seed (1) to the action of dilute acid and alkali and (2) to sodium hypochlorite. The result was that the seeds remained entirely unbleached. At the same time I treated in the same manner the testæ of cotton seed, *Bassia*, *Polygonum convolvulus*, and locust-bean, all of which, especially the first-named, are very dark-coloured. In every case they bleached quite readily. Free chlorine has a more energetic action, and will gradually bleach both

castor oil seed and croton seed ; the time required amounted, however, in my experiments to some three or four days, and even then the colour was not completely discharged. I need not mention other seeds, which are likewise dark-coloured, and which frequently occur in cattle foods, for they present no difficulty in their recognition. I had now in my hands a method by which either castor oil seed or croton seed might be readily separated from other materials, even when present in quite small quantity. If the suspected feeding stuff be first digested with acid and alkali, and be then subjected for a few hours to the action of bleaching powder, any castor oil seed or croton seed would remain black amongst the bleached mass. I may mention here that preliminary experiments had shown me that this agent acted exactly in the same manner as sodium hypochlorite. In order to put the method to a test, I chose uncorticated cotton cake as the material with which to mix the castor oil seed or croton seed. I made this selection because the testa of the cotton seed is just as black after treatment with acid and alkali, as that of either castor or croton.

To one pound of the coarsely ground cotton cake I added, in each of the following experiments, the weighed and coarsely broken up testa of *one seed* of castor and croton respectively, and, after mixing roughly, the whole was digested in dilute HCl and boiled for one hour by passing a current of steam through it. After washing by decantation, the mass was boiled with dilute alkali for a similar period, and again washed by decantation until the washing water was colourless. Bleaching powder, about $\frac{1}{2}$ lb. to 1 lb. (the exact quantity does not appear to be material) was then added, and sufficient water to keep the temperature from rising. This was usually three or four litres. The mass was stirred occasionally and left over night, although the bleaching action was frequently complete at the end of four hours. Washing by decantation now followed, by which means most of the lime could be removed. It remained now merely to pick over, by hand, the bleached material and select the unbleached pieces for examination. This operation requires, for a pound of material, some considerable time, varying from one half to a whole day, but it is so simple that any careful laboratory servant can perform it. Care must be taken not to have anything thrown away. The picking over of the material is most readily performed by spreading small quantities on the bottom of a flat porcelain dish, which is about half full of water; any dark-coloured or black pieces can then be easily seen, and can be taken out. After the unbleached pieces have been separated they are readily examined, and anything other than castor or croton again separated. I may mention here that in a pound of bleaching powder a number of bits of coal or stone may often be found, and, further, in the case of the cotton seed, the hilum does not always bleach so readily as the other portions, so that these would likewise be separated with any castor or croton seed.

EXPERIMENTS —

(1.) Weight of testa of *castor oil seed*, 0.223 grm., mixed with 1lb. of cotton cake : weight of separated castor oil seed testa, 0.200 grm.

(2.) *Castor oil seed testa*, 0.122 grm., mixed with 1lb. of cotton cake : recovered 0.087 grm. castor oil seed testa.

(3.) *Castor oil seed testa*, 0.065 grm., mixed with 1lb. of cotton cake : recovered 0.042 grm.

(4.) *Croton seed testa*, weight 0.074 grm., mixed with 1lb. of cotton cake : recovered 0.060 grm.

(5.) *Croton seed testa*, weight 0.080 grm : recovered 0.078 grm.

The method, accordingly, is not merely a qualitative one, but is, at the same time, a valuable quantitative one.

Only in one experiment did I fail to recover the greater part of the seed *testa* which was put into the cotton seed cake. In this case 0.071 grm. of the *testa* of croton seed was added, and only 0.02 grm. was recovered. An explanation is, however, easily found for this loss. In a preliminary bleaching experiment with castor oil bean, part of a *mouldy* seed was subjected to the action of sodium hypochlorite, and about one-half of the piece became bleached after the lapse of a day. A microscopical examination revealed, however, the fact that this seed-case had been penetrated through and through with a fungus.

In the experiment in question, the croton seed happened to be a mouldy one, and no doubt a part of it became bleached. With regard to the amount of the seeds which were not recovered in the other quantitative experiments, I believe that the loss was due to very minute pieces of the *testæ* being broken off during the process of boiling, the *testa* in each case being extremely brittle, and these minute fragments might naturally be overlooked. Usually, I found that the *testa* of the seed had been broken into some 25 or 30 pieces, by rough pounding with a pestle.

Regarding the hilum of the cotton seed, referred to before as not being completely bleached, there is no difficulty in distinguishing it, for the pieces are round and have no sharp edges ; moreover, they are marked by a series of rings alternating grey and black on the surface.

It now remains for me to speak of the peculiarities of the *testæ* of these two poisonous seeds. The outer covering of each is quite distinct, and, in the case of castor seed, very peculiar. If any pieces of either the one or the other seed be found intact no difficulty will be experienced in recognising them. The outer integuments are, however, extremely thin and brittle, and it must not be expected that they will necessarily be present. Of the two, I have as yet only found castor oil seed in any sample of feeding material, and I therefore cannot give any experience as to the other. In the case of castor oil seed, it may very readily happen that this characteristic integument has disappeared, and as a matter of fact I have often found it to be so.

But in the event of no pieces of either of these seeds being found intact, and if they should have been separated by the bleaching process, they are simply black and opaque and extremely similar. One of their chief characteristics is their extraordinary brittle-

ness, and if one endeavours to cut the testa across with the knife, the two parts fly away some considerable distance, and the broken pieces of either have invariably sharp and frequently ragged edges.

Regarding their thickness, I have measured a number of pieces, and find that of the castor oil seed testa to be from 0.25 m.m. to 0.30 m.m., and that of the croton seed testa to be from 0.20 m.m. to 0.25 m.m.; the latter being thus distinctly thinner than the former.

After passing through the bleaching process, the castor oil seed testa has a much smoother and more shiny exterior than the croton seed, and herein lies one point of distinction.

I have examined the microscopical appearance of sections taken transversely through the testa of each. For the greater part, the structure of each is very similar, each being built up of closely packed bundles of fibres of the same thickness. One difference I have noticed is, that on the outermost edge of the section of the croton testa there is a distinct thickening of each bundle of fibres, whereas this is quite absent from the section of the castor oil seed testa.

Lastly, if the edges of the pieces be examined with a low power objective (about 1 in.) with reflected light, the peculiar curvature of the bundles of fibres on the inner edge is readily seen. This is common to both of the seeds.

DISCUSSION.

Mr. Bernard Dyer said that castor seed found in feeding stuffs was sometimes in the decorticated form. One small piece of husk might then represent a large amount of castor seed.

The President asked Mr. Dyer if this impurity was accidental, or was the seed added intentionally?

Mr. Dyer replied that he was of opinion that sometimes it was added accidentally and sometimes intentionally. In a good many cases it had been found in Brazilian cotton meal and cake; and in that case he believed the castor oil seed had been accidentally introduced, through the castor oil plant growing as a weed among the cotton; but in some cases he was sure that it was deliberately added through ignorance. There was an enormous quantity of castor cake produced, especially in tropical countries, which ought all to be used as manure. It was sometimes bought up by people who were ignorant of its poisonous properties. He had recently had a sample of Niger seed cake sent to him for the purpose of examination, with the view of ascertaining whether it contained castor; and he found that it did. Since then he had found a little castor seed in a sample of linseed cake. He also found a considerable quantity of Niger cake in it, with which the linseed cake had been adulterated, and it seemed not at all improbable that the Niger cake had contained the castor.

Mr. F. H. Perry Coste pointed out with regard to the question that Dr. Leather had referred to at the beginning of his paper—the difficulty of rendering some of those objects transparent—that some microscopical journals had recently given an account of a new process, just discovered, for effecting that purpose. The process was to place the leaf or other object in a drop of glycerin on a slide, cover with a cover-glass, and gently heat, replacing the glycerin from time to time. That method rendered a thick object so transparent that the whole structure of it could be seen. It was, of course, quite different in character from the bleaching process, the colour not being entirely removed. The secret of the process is “the use of glycerin and the saturation of the cells.”*

Mr. Bertram Blount wished to ask Mr. Dyer if he could inform him whether any attempt had been made to detect castor oil seed by chemical means? He supposed both it and croton seed would be susceptible of detection by recognising the poisonous principles they contained. He believed it was a fact that a poisonous principle had been definitely recognised in castor. As Mr. Dyer had said, there might be only a small quantity of the husk to a comparatively large quantity of the seed, and thus any method of detection, based on the recognition of the husk, was necessarily imperfect.

Mr. Dyer said that he did not think that the definite poisonous principle of castor had been isolated. Professor Tuson, years ago, isolated an alkaloid, which was described in Watt's *Dictionary of Chemistry*, but if he (Mr. Dyer) recollected rightly, that alkaloid did not appear to have been the active ingredient of the seed.

Sir Charles Cameron said that he had some experience in the examination of oil cakes, and on one or two occasions he detected what he believed to be the impurity referred to by Mr. Dyer. He would say, however, that he did not feel great confidence in the results he had arrived at.

Mr. Cassal asked whether it had been attempted to make use of chlorate of potash and hydrochloric acid in the bleaching process described? Mr. Dyer had stated that it took a very considerable time to carry out the bleaching with hypochlorite. He (Mr. Cassal) had found the chlorate of potash and hydrochloric acid method very rapid for vegetable tissue generally.

Sir Charles Cameron said that on one occasion he extracted, by purely chemical treatment, a substance such as that referred to from croton seed, which had strong blistering properties. Its ether extract had acted as a vesicant upon his wrist. It was believed that the cake, in that case, had been very injurious to animals.

Mr. Dyer, replying to Mr. Cassal, said he had not tried the chlorate of potash and hydrochloric acid method. He would expect that method to bleach almost anything. The process Dr. Leather had described was not at all tedious. It was necessary to get rid of the mealy, farinaceous or albuminous part of the meal. That was easily done in an hour or so by the alkali and acid treatment. If the bleaching were

See “Microscope,” xi. (1891), pp. 265-7; and Royal Microscopic Society's Journal, 1892, pp. 287-8.

set going at night, the sample was always ready the next morning. He believed it would be advantageous to use a solution of sodium hypochlorite, which would be free from many of the impurities which were indicated in the paper likely to be present in ordinary bleaching powder. Dr. Leather found that sodium hypochlorite would act in every respect as well as bleaching powder. He thought that the point mentioned by Dr. Leather, with reference to the hilum of the cotton seed, should be carefully noted. The part of the cotton-seed husk which included the hilum did not bleach as a rule, and at first sight looked a little like "unskinned" castor husk; but it was always soft and fleshy at the edges, and if looked at with a low-power magnifying glass, bore no resemblance to castor husk. The latter, when magnified, looked very like a much-diminished fragment of smooth cocoa-nut shell.

Sir Charles Cameron asked Mr. Dyer what he had found certain small particles in decorticated cotton-cake to consist of? The particles in question were very hard and brittle.

Mr. Dyer replied that, if the particles occurred in Brazilian cotton-cake, they were most likely castor.

This terminated the proceedings of the Society.