tannin being removed in considerable quantities, after extracting with three or four liters during eight to twelve hours.

This investigation will be continued, and extended to include other tanning materials.

[CONTRIBUTION FROM THE BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE.]

DETERMINATION OF SULPHUR AND PHOSPHORIC ACID IN FOODS, FECES AND URINE.

BY W. L. DUBOIS.

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IN a previous contribution[1] from the Bureau of Chemistry, the methods then in use for the determination of sulphur and phosphoric acid in connection with the metabolism experiments being conducted by Dr. Wiley and Dr. Bigelow were described. Since the appearance of that article, some important changes in the method of burning foods and feces have been made, to which I wish to call attention. Urines are still burned by Osborne's method, the facility of handling them in that way being all that could be desired. In this work all phosphoric acid determinations were made by Mr. C. W. Nelson.

For foods and feces we are now using a modification of a method recently described by Neumann and Meinertz.[2] In adapting this method to foods and feces we doubled the initial amount of sodium peroxide, owing to the larger sample used. It was found that moist foods, *e. g.*, meats and eggs, burned without difficulty, whereas dried foods and feces gave a good deal of trouble by frequent flashing. Since the most marked difference in these two classes of material is in their water content, we found that the addition of a small amount of water to the dried charges caused the combustion to proceed smoothly. Experience showed that the use of sodium carbonate alone, instead of a mixture of sodium and potassium carbonate, is more satisfactory, because the fusion with the former is more rapidly dissolved from the crucible by hot water, although as far as accuracy of results is concerned, we noticed no difference whether using sodium carbonate, potassium carbonate,

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[1] This Journal, **26**, 1108.

[2] *Z. physiol. Chem.*, **43**, 37.

or a mixture of the two. One charge is used for the determination of both sulphur and phosphoric acid, as formerly. The method of Neumann and Meinertz, as adapted to the burning of foods and feces and now in use in the Bureau, is as follows:

Four grams of fresh foods, 2 grams of dried foods or 1.5 grams of dried feces are brought into a nickel crucible of 100 cc. capacity. The dry charges are moistened with 2 cc. of water, 5 grams of anhydrous sodium carbonate, and 5 grams of sodium peroxide are added, and the charge mixed intimately. With feces, the addition of sodium peroxide can be made all at once, but with dried foods half is stirred in and then after standing a short time, the remainder is added. This is to avoid the sudden combustion which sometimes takes place, if all the peroxide be added at once, probably due to the more highly carbonaceous character of this latter class of materials. The crucible is placed over a low, alcohol flame, and heated until the material is dry, usually requiring about half an hour; the flame is then increased, and the charge heated until it disintegrates, and can be easily broken up into a powder. The crucible is removed from the flame, the powdered charge covered with sodium peroxide and heated until liquid, starting with a moderate flame and increasing to an intense blast. When the liquid state is reached, a little peroxide is added to insure complete oxidation, the crucible allowed to cool, and the fusion dissolved out with hot water, and treated exactly as described for the Osborne method.

The advantages of this method over that of burning with sodium peroxide alone, are the following:

(1) Combustion is more satisfactory. When burning by Osborne's method, charred material was often found on dissolving the fusion, making it necessary to repeat the combustion. By employing sodium carbonate with the peroxide, as described above, such cases of incomplete combustion are rare. That results agree with Osborne's method, as formerly used by us, is seen by the following table:

		Per cent. sulphur.		Per cent. phosphoric acid.	
Substance.	Laboratory number.	Osborne.	Modified Neumann.	Osborne.	Modified Neumann.
Dried feces	12154	0.707	0.715	4.32	4.32
" "	12159	0.506	0.517	5.68	5.60
" "	12161	0.612	0.606	5.72	5.68
" "	12164	0.546	0.527	5.48	5.40
" "	12170	0.561	0.546	6.44	6.52
" "	12172	0.621	0.608	4.04	4.08
" "	12173	0.561	0.569	5.92	5.88
" "	12176	0.563	0.575	5.24	5.20
" "	12178	0.583	0.586	4.32	4.28
" "	12183	0.531	0.546	5.16	5.08
" "	12184	0.610	0.611	4.24	4.28
Foods.					
		Per cent. sulphur.			
Pork chops......	.....	0.285	0.311	....	....
Veal............	.....	0.308	0.310	....	....
Beef steak.......	.....	0.303	0.294	....	....
Chicken.........	.....	0.321	0.313	....	....
Ice cream........	.....	0.031	0.029	....	....
Oysters (fried)...	.....	0.206	0.200	....	....
Fish............	.....	0.264	0.261	....	....
Eggs............	.....	0.211	0.204	....	....
—Bread.......	.....	0.163	0.183	....	....
—Potato......	.....	0.142	0.146	....	....
—Custard.....	.....	0.183	0.186	....	....

(2) Less time and attention are required. The sodium peroxide method demands close attention, owing to the constant danger of frothing and flashing, and is consequently, extremely tedious. Furthermore, the actual combustion is slower. With the sodium carbonate modification very little attention is required, more lamps can be used, and owing to the shorter time required for combustion, many more samples can be burned in a given time.

(3) Economy of material. The amount of sodium peroxide used is usually less than in the older method.

The type of the lamp which we find most satisfactory is Barthel's alcohol burner. This lamp burns alcohol vapor, is sparing of fuel, and is capable of adjustment, ranging from a very small flame to a powerful blast.

Kiester and Theil[1] published an article recently, in which they obtained a pure barium sulphate precipitate in the presence of much iron, by adding barium chloride to the hot alkaline solution, and

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[1] *Z. anorg. Chem.*, **19**, 100.

then making acid with hydrochloric acid. In this way no iron is occluded. We tried this method of precipitation with feces in order to avoid, if possible, the removal of phosphate by filtration, but the results obtained were not satisfactory.

IMPROVEMENTS IN GAS ANALYSIS APPARATUS.

BY ALFRED H. WHITE AND E. D. CAMPBELL.

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THE accuracy of gas analysis is so largely dependent on methods of manipulation and details of construction of apparatus, that it is worth while to record anything which adds to the simplicity or accuracy of existing methods. The apparatus described in this article differs only in details from the usual form of Hempel apparatus, but the changes that have been introduced have been prompted by experience, and have proven their value by actual use in our gas analysis laboratory for from four to twelve years. During this period, the glass parts of apparatus have been made from our sketches by Greiner and Friedrichs, of Ilmenau, in Thuringia, and the stands have been made in our own shops. Changes in detail have been gradually introduced in the effort to render the process simpler and more accurate, and the apparatus less liable to breakage. These sketches of our present apparatus are now presented in the belief that they represent slight improvements in simplicity, durability and accuracy.

The perspective view shows the apparatus as it appears in service, while some details are given in the other sketch. The burette-stand, it will be readily evident, is made from an ordinary iron stand, one of whose rings of external diameter slightly greater than the water jacket of the burette has been provided with a brass collar, thus making a cup in which the rubber stopper of the water jacket rests without binding. Another ring large enough to slip loosely over the water jacket, serves to keep the burette vertical. A segment is sawed out of the front of this ring to allow an uninterrupted view of the graduations, and it is wrapped with chamois skin until it fits as snugly as desired. By this simple arrangement the burette may be raised, lowered, or swung to one side at the convenience of the operator, and may be tipped in any position while carrying, without danger of breakage.