of calcium, aluminum and magnesium oxides and carbonates. There was also present small amounts of iron and silica which would be present from the wash water used in the preparation of the pulp or in many other ways.

"The fiber present in the pulp was found to be composed of linen, very well pulped except for a very few bunches of undigested or unbeaten fiber which it is impossible to overcome. No test for other fibers was obtained. No acids, sulphur compounds or chlorine was found which might have come from the process of manufacture. The paper gave an alkaline reaction due to the filler used which was composed of calcium, aluminum and magnesium carbonates and oxides. The carbonates present were equal to at least 75% of the ash and probably more, but it was impossible to determine this very accurately. No impurities other than those mentioned were found. The moisture found was of varying weight, due to the fact that paper of the variety examined has a tendency to hold moisture proportionate to the humidity of the atmosphere of the date of examination."

I wish to call particular attention to the small amount of ash shown by these analyses compared with the amount actually found by the writer in twenty-eight samples which yielded an average percentage of 14.93.

NITRATES IN PAPERS.

It appears that the majority of manufacturers object to paper containing nitrates.

Nitrates may be detected by applying the diphenylamine test as follows:—Take one drop diphenylamine test solution on a glass rod and draw the rod lightly and quickly across the paper. Hold the paper to the light and within five or ten seconds a blue coloration is produced in case nitrates are present. If a large quantity of the nitrate is present the coloration forms immediately. The diphenylamine test solution may be prepared after Withers & Gray's formula,[9] which is as follows:—0.7 gram of diphenylamine is dissolved in 60 cc. concentrated sulphuric acid and 28.8 cc. of distilled water. The mixture is thoroughly cooled and 11.3 cc. concentrated hydrochloric acid are added slowly.

The papers of only four brands out of twenty-eight tested, responded to this test for nitrates.

In closing, I wish to acknowledge the helpful suggestions of Prof. John U. Lloyd while experimenting with Lloyd's Reagent; also to those who kindly and promptly replied to my letters when seeking information on certain points and to the manufacturers in furnishing data in reference to cigarette papers.

ESTIMATION OF CALOMEL.

R. I. GRANTHAM.

The iodometric method for the estimation of calomel is the most expeditious and convenient one. This method, however, cannot be applied generally, especially in the analysis of calomel tablets or similar preparations which contain in addition to the mercury salt various other ingredients. In order to find a reliable method for estimating the calomel in such products the following methods were tried by which reliable results were obtained:—

METHOD I.

0.3 gm. of calomel or an amount of the powdered tablets equivalent to 0.3 gm. of calomel is transferred to a 6 ounce glass stoppered bottle. Any alkali present

9 J. Am. Chem. Soc. (1911), Vol. 33, 708.

is neutralized with acetic acid and about 5 cc. of chloroform, 20 cc. of N/10 iodine solution and 5 cc. of potassium iodide solution (1:1) are added.. The mixture is shaken occasionally for 15 minutes and the excess of iodine is then titrated with N/10 sodium thiosulphate solution. Each cc. of N/10 iodine solution corresponds to 0.0236 gm. or 0.364 grains of calomel.

METHOD II.

0.2 gm. of calomel or a corresponding amount of the powdered tablets is transferred to a 4 ounce Erlenmeyer flask, mixed with 0.5 gm. of potassium chlorate and 15 to 20 cc. of 10 *per cent.* hydrochloric acid. The mixture is digested on a steam-bath for 15 minutes and is then filtered into an 8 ounce glass stoppered bottle. The solution is neutralized with caustic alkali and a sufficient quantity of the alkali is then added to obtain an excess equivalent to about 20 cc. of normal caustic alkali. After the addition of 3 cc. of formaldehyde solution (37 *per cent.*), the mixture is shaken well and is then allowed to stand for 15 minutes. It is then made acid with acetic acid and the metallic mercury which is formed estimated iodometrically as given in Method I. Each cc. of the N/10 iodine solution corresponds to 0.0118 gm. or 0.182 grains of calomel.

METHOD III.

0.3 gm. of calomel or an equivalent amount of the powdered tablets is treated with potassium chlorate and hydrochloric acid as just given. The mixture is filtered into a large beaker, the filter washed well with water and the filtrate is made alkaline with ammonia water. After the addition of a large excess of acetic acid and one to three grams of sodium or potassium oxalate to the filtrate, the mixture is boiled, stirring constantly in order to prevent bumping. After allowing the mixture to settle, it is filtered. The precipitate is washed three times with hot water by decantation, is transferred to a filter and is washed with hot water until free from chlorides, using for this purpose about 150 cc. of water in all. The filtrate and wash-water are then heated to about 80° and the mercury is precipitated with hydrogen sulphide gas and estimated as mercury sulphide in the usual way.

Although Method I, originated by Hempel, is a very expeditious and reliable one it cannot be applied as already pointed out in every case. It is essential that chloroform and acetic acid be used, as I have found that in the absence of chloroform, when tablets are made with mineral oil as a lubricant, the mercury globules are covered with a thin coating of the oil and the iodine solution is thus prevented from acting on the mercury. If such is the case the results naturally will be low. The acetic acid is used in order to dissolve any alkaline salts present.

Method II is devised as a check of Method I, and although it is somewhat more complicated it may be considered as giving more accurate results for general use. Most of the foreign matter present is oxidized by the chlorine liberated from the potassium chlorate by the hydrochloric acid.

In this process the calomel is converted into mercuric chloride, the latter reduced by the formaldehyde to metallic mercury, which in turn is converted by the iodine solution into mercuric iodide. From the amount of iodine solution used for this purpose, found by titrating back the excess of iodine solution, the amount of calomel can easily be estimated.

The third method is well adapted for examining tablets which contain coloring matter, or pills which contain resins, gums and similar substances. The sodium or potassium oxalate is added to eliminate the hypochlorite which is formed during the reaction and which would cause a precipitation of sulphur when conducting the hydrogen sulphide into the solution obtained. Naturally all insoluble matter should be eliminated by filtration before the mercury is precipitated by the hydrogen sulphide. Calcium phosphate which is frequently applied as a diluent in making tablets of this kind is also precipitated as oxalate.

These methods were applied to mixtures of various composition, and also to various kinds of tablets. The mixtures were the following, the amount of ingredients being expressed in grams. The following results were obtained:—

	Mixture No. 1	Mixture No. 2	Mixture No. 3	Mixture No. 4
Calomel	5 gms.	5 gms.	5 gms.	2.5 gms.
Sod. Bicarb.	5 gms.	10 gms.	25 gms.	2.5 gms.
Starch	1 gm.	2 gms.	5 gms.	5 gms.
Talcum	1 gm.	2 gms.	5 gms.	5 gms.
Glycolene	0.5 gm.	1 gm.	2 gms.	2 gms.
Prec. Calc. Phos. q. s. ad	25	50	100	100
Calomel in Mixtures	20%	10%	5%	2.5%

	Mixture No. 5	Mixture No. 6
Calomel	2.5 gms.	2.5 gms.
Milk Sugar q. s. ad	50 gms.	10 gms.
Calomel in Mixtures	5%	25%

	1	2	3	4	5	6
Method I	99.1%	98.0%	100 %	96 %	99.5%	99.1%
Method II	99.8%	100 %	100.9%	96.7%	99.8%	99.2%
Method III	98.3%	99.6%	97.2%	95.1%	100.1%	100 %

Three kinds of tablets were examined and the results obtained by the three methods represent the following amount of the theoretical amount present:—

Tablets	Method I	90 %	94 %	106%
	Method II	97.3%	100.1%	98%
	Method III	98.2%	101 %	99%

Analytical Laboratory of Sharp & Dohme, July, 1914.

SOME FACTORS IN DRUG ABSORPTION IN FROGS.

W. F. BAKER, M. S., M. D.

Among the objections that have been urged against the frog-heart assay method for the digitalis bodies, the principal ones are the factor of absorption and the time-limit. Of these, the factor of absorption is undoubtedly the more important, the time-limit being dependent upon the rate of absorption.

The manner in which various fluids are absorbed from the lymph sac or the absorption prevented, has not been determined. Experiments and experience have demonstrated, however, that the majority of healthy frogs will absorb from the ventral lymph sac, in a period of one hour, a quantity of fluid equal to about 0.015 cc. per gram of body weight.

Focke has demonstrated that enough of the digitalis bodies may be absorbed in five to twenty minutes to produce the characteristic digitalis heart and has taken twenty minutes as the time-limit for his method. Magnus, using another method, has extended the time to one-half hour, Famulener and Lyons to one hour, and