CONCLUSION

The method described for the determination of traces of phenolic impurities in methyl salicylate is both rapid and accurate. It permits the detection of as little as 0.001 per cent free phenolic bodies and is reproducible to ±0.0003 per cent. This is a decided improvement over the customary method which is sensitive only to 0.02 per cent phenol.

REFERENCE

(1) Dodge, F. D., *Drug Markets*, 22 (1928), 609.

A Method for the Determination of Calomel in Tablets

By R. A. Bosee and L. A. Perlenfein†*

The well-known reaction which occurs when calomel is heated in the dry state with sodium carbonate or bicarbonate may be used to assay mercurous chloride in tablets. The method is especially useful for those tablets in which the presence of other ingredients renders the iodine method inapplicable, and makes filtration of the entire tablet mass difficult.

$$Hg_2Cl_2 + 2NaHCO_3 \rightarrow 2Hg\uparrow + 2NaCl + CO_2\uparrow + H_2O\uparrow$$

The method was first used in our laboratories for the assay of calomel and soda tablets.

EXPERIMENTAL

Assay for Calomel.—Weigh not less than 20 of the tablets, reduce them to a fine powder without an appreciable loss, transfer an aliquot portion equivalent to about 0.5 Gm. of mild mercurous chloride to a nickel crucible and ignite. Leach the carbonized mass with boiling water, add 30 cc. of tenth-normal silver nitrate, 5 cc. of nitric acid and filter. To the filtrate add ferric ammonium sulfate T.S. and titrate the excess of silver nitrate with tenth-normal ammonium thiocyanate.

Each cc. of tenth-normal silver nitrate is equivalent to 0.02361 Gm. of mild mercurous chloride, HgCl.

Assay for Sodium Bicarbonate.—It is also possible to obtain a figure for both sodium bicarbonate and calomel on the same sample. After igniting and leaching with boiling water, the filtrate can be first titrated with normal sulfuric acid in the presence of methyl orange, and then a chloride titration run.

The number of cc. of normal sulfuric acid multiplied by 0.08401 represents the amount of sodium bicarbonate not entering into the reaction with the calomel. The figure obtained for calomel multiplied by 0.356 represents the quantity of sodium bicarbonate entering into reacton. The sum of these two results gives the entire amount of sodium bicarbonate present in the sample.

If the method is to be used for assaying calomel in tablets not containing soda, carbonate or bicarbonate may be added and either titration used. If the alkalimetrical titration is used, a definitely measured quantity of reagent must be added. If the chloride titration is used, an excess of the reagent is all that is necessary. In either case, however, care must be exercised that the calomel does not volatilize before it has a chance to react with the carbonate. Best results were obtained by adding a few drops of water and mixing into a smooth, thick paste. In the case of the calomel and soda tablets the thorough mixing of the ingredients and the compression of the tablets make the addition of moisture before igniting unnecessary.

Procedures similar to both of those given have been used before for the determination of calomel in aqueous media. M. Kohn (1), (2), (3) showed that mercuric halides could be decomposed in alkaline solution and the halogens subsequently determined by the method of Volhard. D. Köszegi (4) pointed out that calomel could be determined by treating with a known quantity of normal NaOH, filtering and titrating the remaining NaOH with normal acid. However, for the usual calomel tablets, heating in the dry state with carbonate furnishes a quick and accurate method of eliminating troublesome fillers and at the same time volatilizes the metallic mercury formed during the reaction.

Results.—The following are examples of the results obtained by following these procedures:

Results of Assay of Calomel Tablets

Weight of Sample, Gm.	Gm. Calomel Calculated[a]	Found	Error, %
1.5056	0.4926	0.4925	0.02
1.5022	0.4914	0.4880	0.72
1.5040	0.4920	0.4904	0.32
Weight of Sample, Gm.	**Gm. Sodium Bicarbonate Calculated[a]**	**Found**	**Error, %**
1.5056	1.0017	0.9940	0.77
1.5022	0.9997	0.9966	0.31
1.5040	1.0009	0.9996	0.13

[a] Calculated amounts are based on assay of each separate ingredient by U. S. P. method.

REFERENCES

(1) Kohn, M., *Zeit. anorg. Chemie*, 59 (1908), 108–110.

(2) Kohn, *Ibid.*, 59 (1908), 271.

(3) Kohn, M., and Ostersetzer, A., *Ibid.*, 80 (1913), 218–220.

(4) Köszegi, D., *Pharm. Zeit.*, 76 (1931), 524.

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