The most serious objection to the tintometer method as described by Fuller is that in measuring the yellow color of the ether solution no account is taken of the presence of the yellow non-anthraquinone material. It would be actually possible in this way to obtain a positive value on material free from anthraquinone. Where this determination is afterwards checked colorimetrically by the alkali salt method, the difficulty is avoided in part, but the presence of this yellow material now in the water solution, does not contribute to a simplification of the colorimetric method.

Neither does it seem proper to use as a colorimetric standard a solution of aloe emodin for comparison with solutions the principal color of which is due to frangula emodin. We are of the opinion that the only satisfactory color standard can be obtained from a specimen of the species of drug under examination from which the anthraquinones have been extracted and determined gravimetrically with the greatest quantitative accuracy possible. The residue so obtained might be dissolved in alkali and used as the basis of a color standard.

Finally attention should be called to the various published analyses of drugs of this class, including those reported in this paper in which will be noted the excellent agreement between duplicate determinations, although they may be out of all bounds of reason as solubility data is considered. The extraction and removal of non-anthraquinone material is very evidently largely influenced by certain physical conditions which are established by each operator in his duplicate analyses.

The authors are continuing this work, first, with a view to establishing conditions which will lead to accuracy of results regardless of the time involved, and then to an elimination of procedure by which the method may be sensibly shortened without an appreciable loss in accuracy.

NOTES ON REDUCED IRON.*

BY GEORGE L. KEENAN.

Some time ago it was suggested to the writer that a comparative microscopical study of products appearing on the market as reduced iron or iron by hydrogen would be of interest in determining any diagnostic microscopical differences that might exist in the products, thereby eventually aiding in differentiating any non-pharmacopœial articles from the pharmacopœial. The purpose of this paper is to call attention to these differences.

Reduced iron is formed by the action of hydrogen upon ferric oxide, resulting in a product which should contain, according to the U. S. P. X (abstract), not less than 90 per cent of metallic iron (Fe). In general appearance the resulting product should consist of an odorless, grayish black, lusterless powder, which should pass through a No. 100 sieve. As an additional test of its genuineness, the Pharmacopœia stipulates that when 1 Gm. of reduced iron is heated in a porcelain crucible with a small Bunsen flame until a bluish black color appears without glowing, the particles of the material should glow brightly as they fall through the air when poured from the crucible.

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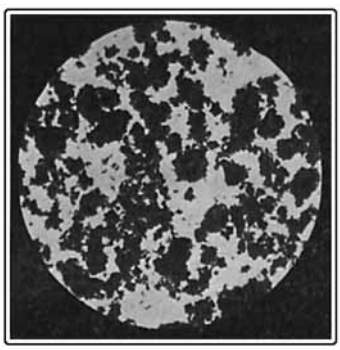
* Scientific Section, A. PH. A., Des Moines meeting, 1925.

Reduced iron made by the pharmacopœial method[1] consists, as already indicated, of a lusterless grayish black powder, usually so fine that a microscopical examination fails to reveal individual particles of any appreciable size. A macroscopical examination of a number of commercial samples labeled "reduced iron," however, showed them to be of quite different character, in that they consisted of a steel-gray granular substance, the individual particles of which are much larger than those of pharmacopœial reduced iron.

The microscopical examination of the two products reveals, moreover, even more striking differences than mere size. Under the microscope, the pharmacopœial material consists of small irregular particles aggregated into structureless masses. (Fig. 1.) The commercial material referred to, on the other hand, consists of fragments with definite outlines, usually taking the form of particles resembling leaflike structures. (Fig. 2.) These differences in the microscopical appearance of the two products afford a ready means for differentiating them.



Pharmacopœial reduced iron. (60.)

Non-pharmacopœial reduced iron. (60.)

The usual assay for metallic iron reveals no significant differences in the two products and therefore is valueless for determinative purposes. The official glow test, however, although it leaves much to be desired as far as accuracy is concerned, affords additional proof that the substances are of different nature. In carrying out the glow test the directions were somewhat varied for the purpose of drawing a sharper line between the two products. A small amount of the pharmacopœial reduced iron (about 1 Gm.) was placed in a small porcelain crucible and heated over the Bunsen flame until it became black and finally glowed. On being poured into a dish the material broke up into chunks resembling glowing charcoal. The non-pharmacopœial material, treated in the same manner, did not coalesce into chunks when poured into the dish but remained a granular powder and did not glow.

The pharmacopœial reduced iron is amorphous in microscopical appearance and the ready reactivity which it shows in the glow test confirms this view of its state of aggregation. On the other hand, the non-pharmacopœial article has the appearance of having crystallized in a more or less definite form, and its failure to give the glow test points in the same direction. There is reason to believe, then, from a microscopical study of these two products as well as from the glow test, that they have not been subjected to the same sort of treatment in their preparation.

BUREAU OF CHEMISTRY,
U. S. DEPARTMENT OF AGRICULTURE.

[1] Mr. Raymond M. Hann prepared the reduced iron from a commercial sample of amorphous iron oxide.