

## A MODIFICATION OF THE U. S. P. ASSAY PROCESS FOR OPIUM PREPARATIONS.

---

S. L. HILTON.

---

The method of assay for preparations of opium as given in the U. S. Pharmacopœia, while giving good results, is far from satisfactory for the reason that it is exceedingly tedious in carrying out owing to slow filtration, consequently more time is consumed than necessary. Again the method as given for handling the dried crystals, transferring them from the filter paper to a tared watch-glass, will invariably result in a loss of some of the crystals either by adhering to the filter paper so closely that they cannot be removed entirely, or by the finer crystals flying and being carried off in the air, the result being that the final assay will show results lower than what they should be. The transferring of the crystals to a flask and agitating them with lime water causes another loss, due to some of the finer particles flying in the air and also by the frothing in the flask, the frothing is very troublesome and the filtration following is very slow.

In looking for some method to overcome these difficulties the Gooch crucible suggested itself to me about a year or so ago. It seemed feasible for assaying opium preparations and I thought it would possibly solve the problem and eliminate the difficulties cited above. After numerous trials and noting the results obtained I found the process practicable and rapid so that now I never use the process as outlined in the U. S. P., but follow a modified process using the Gooch crucible with a corresponding saving of time and invariably obtain higher and more concordant results.

Before giving the method in detail permit me to state the apparatus necessary, all of which is inexpensive. A porcelain Gooch Crucible, 25 cc. capacity; a filter tube; two side-neck flasks, known as filtering flasks; about 500 cc. capacity; a good filter pump; some heavy walled rubber tubing, for connecting the flasks and the flask to the pump; some pure gum tubing, to make a tight joint between the crucible and filter tube and some C. P. asbestos, shredded by scraping with a knife and suspending same in distilled water. This should be kept on hand ready for use.

With this method I transfer 100 cc. of Tincture of Opium or other liquid opium preparation to an evaporating dish and evaporate it on a waterbath to about 20 cc., add 40 cc. of distilled water, mix thoroughly and set the liquid aside for one hour, occasionally stirring to disintegrate the resinous flakes adhering to the dish. Having set up a 25 cc. Gooch crucible and prepared a matrix of asbestos by pouring some of the solution suspended in water in the crucible, after starting the filter pump, wash the matrix with alcohol and ignite the crucible, when cold, connect the crucible, after emptying the filter flask and rinsing the same with distilled water, pour carefully the contents of the dish into the crucible after starting the filter pump, when all of the liquid has passed through, disconnect the flask and reserve the filtrate for the final evaporation. Connect the flask again and wash the mass on the filter until completely exhausted (indicated by almost a colorless filtrate and the absence of bitterness). Evaporate the washings in a tarred dish

to a small volume, then add the first filtrate, rinsing the vessel with several small portions of distilled water, and evaporate the whole to a weight of 14 gm.

Proceed as directed under Opium, U. S. P. VIII, page 329, beginning rotate the concentrated solution about in the dish until the rings of extract are re-dissolved, etc., After standing six hours or over night as directed, proceed as follows:

Having set up the Gooch crucible, prepared the matrix as previously indicated, wash with alcohol and ignite, cool in the desiccator and weigh, noting the weight of the crucible. Remove the stopper carefully from the flask, and should any crystals adhere to it, brush them into the flask. Wet the matrix in the crucible well with ether, and decant the ethereal solution in the flask as completely as possible upon the matrix in the crucible, after starting the filter pump. Add 10 cc. of ether to the flask and proceed as directed in the U. S. P. VIII, using the Gooch crucible for the purpose of collecting and washing the morphine crystals, as therein directed. When this has been completed, remove the Gooch crucible and dry same in the oven at a moderate temperature not exceeding 60 C. (140 F.), until the weight of the crucible and its contents remains constant. Note the weight and deduct the weight of the crucible previously obtained. This gives the weight of the impure Morphine.

Replace the Gooch crucible and pass lime water through the crucible (10 cc. for each 0.1 gm. of Morphine), reducing the vacuum in the filter flask so that the lime water comes through slowly, dissolving out the morphine, then wash the crucible with more lime water until after acidulation, the washings no longer yields a precipitate with mercuric potassium iodide, disconnect the crucible and dry in the oven at a temperature of 100 C. (212 F.) to a constant weight, deduct the weight of the crucible, this gives the weight of the insoluble matter in the crucible to be deducted from the weight of the impure morphine previously obtained, this difference represents the percentage of crystallized morphine in 100 cc. of the tincture or liquid preparation assayed.

For liquid preparations of opium and powdered opium this process works well, is a decided saving of time and gives a trifle higher but more concordant results; whether it will work satisfactorily on gum opium I am unable to say as I have not had occasion to try it.

The pharmacist who will equip himself with this apparatus will find that he has something that not only will prove of decided value to him in assaying opium preparations, but will be of service in expediting work at his prescription counter when it is necessary to filter prescriptions containing pepsin in solution, or a prescription like fluidextract of ergot diluted with distilled water and many others of a similar nature, with the loss of practically no time or unnecessary waiting on the part of a customer and at the same time dispensing the prescription in a presentable condition, more satisfactory to the physician and more presentable to the patient.

Many things will suggest themselves when possessing the necessary apparatus as for instance a short time ago it was necessary to make 500 cc. Elix. Pepsin and Bismuth, N. F., quickly and send it out right away. With a 25 cc. Gooch crucible I was able to filter it perfectly clear and dispense same in less than fifteen minutes.