6. Finally, we have "the names or initials of the physician with the date." In life, the Great Physician orders, and the date is from birth to death—three score and ten years, perhaps a little longer—and then "finis," the prescription of life is completed.

What the end-result will be, in any individual case, no man knoweth, because no one has ever returned from the Great Beyond to tell us. But with faith and courage and cheerfulness, coupled with the best thought of which we are capable, let us compound our prescription of life according to the "talents" given us, so that it shall be of lasting credit to ourselves, our profession and our Alma Mater.

IODINE OINTMENT—DATA AND METHOD OF ASSAY.

LEO H. FRIED, PH. G., PHAR. D.

Before explaining the method of assay, let us first consider the absorption of iodine by benzoinated lard. The free iodine in the ointment is readily absorbed by the lard, even though potassium iodide and glycerin are present to retard this. The figures which I have computed by various experiments will readily show this.

A. N. D. Pullen explains in "The Pharmaceutical Journal and Pharmacist," November 16, 1912, page 610, that if no glycerin or potassium iodide were present in the ointment there would only be one twentieth of iodine in the free state within a few hours. He also claims that in the presence of glycerin and potassium iodide the absorption is attenuated, so that after a lapse of four months there still is 2.92 percent of the original 4 percent in free state. However, I find in my experiments that within a period of ten days there is 1.16 percent of iodine absorbed, leaving 2.84 percent free iodine. This ointment which I experimented with was kept at ordinary room temperature on a shelf, so that the changes of temperature was that of any ordinary room. On examining this ointment ninety days later, I found that only a trace of absorption had taken place. After a period of eight months from the date of compounding, no more iodine was absorbed.

METHOD OF ASSAY.

I first made up one hundred grammes of iodine ointment according to the U. S. P. VIII, page 494, as follows:

Iodine	4 gms.
Potassium Iodide	4 gms.
Glycerin	12 gms.
Benzoinated Lard	80 gms.
	100 gms.

ASSAY FOR FREE IODINE.

Carefully clean, dry and tare 120 cc. Erlenmeyer glass stoppered flask and accurately weigh into it from 3.0 to 5.0 gms. of ointment, using a glass rod for the transfer of same. Add 30 cc. chloroform, shake the flask a few minutes until the ointment is apparently dissolved. Then add 30 cc. of distilled water and shake (this will dissolve the potassium iodide and glycerin which did not go into the chloroformic solution). Immediately, titrate the solution with N/10 sodium thiosulphate, shaking the flask well after every addition until a light yellow color

still remains, and add about 1 cc. cold starch T. S., and continue titration until the blue color of starch iodide disappears after vigorous shaking and does not return in one minute.

I specifically mention that the titration should be immediately performed so that the chloroformic solution will not aid in the absorption of iodine by the lard

CALCULATION.

$$\frac{\text{No. of cc. used} \times \text{factor} \times 100}{\text{Weight of sample}} = \text{percent of free Iodine}$$

On assaying as above I have the following figures of free iodine:

Immediately after making	3.89%
One hour " "	3.51%
One day " "	3.48%
Five days " "	3.06%
Ten " " "	2.84%
Thirty " " "	2.81%
Ninety " " "	2.8096%
Eight months " "	2.8095%

By these figures we see that the absorption goes on slowly until the maximum amount is absorbed.

ASSAY FOR POTASSIUM IODIDE.

Accurately weigh about 5 gms. of ointment into an ordinary 250 cc. Erlenmeyer flask and attach to a distillation apparatus, using as a receiver a 250 cc. glass stoppered Erlenmeyer flask, containing one gm. potassium iodide dissolved in 30 cc. distilled water and add to it 30 cc. of chloroform. Allow the end of the condenser tube to dip into this mixture. Make all connections air-tight, using rubber stoppers and not cork. Mix 5 cc. sulphuric acid with 150 cc. of distilled water, add the acid mixture quickly into the flask. Drop in a few pieces of pumice stone which have previously been heated to redness and dropped into cold water. Finally drop in a piece of ferric alum, about 5 gm. Place upon a wire gauze and apply direct flame very slowly at first, until the purple vapors of iodine have all distilled over. The iodine will sometimes condense in the tube and not go down into the receiving flask; if this happens move the flame and allow the liquid in the flask to suck up into the tube, whence it will dissolve the iodine. Replace the flame and continue distillation until an oily substance comes over. Discontinue the distillation by first removing the tube from the liquid in the receiving flask. Then take away the flame. Wash out the condenser tube and end with about 20 cc. distilled water. Immediately titrate the distillate with N/10 sodium thiosulphate, shaking the flask vigorously after every addition. Note the number of cc. of V. S. used, and calculate total iodine liberated.

CALCULATION.

$$\frac{\text{No. of cc. used} \times \text{factor} \times 100}{\text{Weight of sample}} = \text{percent total Iodine}$$

Total Iodine less *Free Iodine* equals *Iodine from KI*

$$\text{Iodine} : \text{Potassium Iodide} :: \text{Iodine from KI} : \text{Potassium Iodide} = \text{percent KI}$$

$$125.9 : 164.76 :: \text{Iodine from KI} : X$$

I will admit that this distillation process is not the speediest way of obtaining the percentage of potassium iodide in the ointment, but I can say that it is the most accurate. I have tried other processes such as, washing out the potassium

iodide by means of hot water, also using oxidizing agents such as ferric chloride, sodium nitrite, potassium permanganate, manganese dioxide, etc., to liberate iodine from the potassium iodide, and find that liberation is either incomplete or else the oxidizing agents interfere with the final titration. By the above method given, I got an average of 3.91 percent of KI from four titrations.

In conclusion, while this does not directly belong to the assay, I might state that from a therapeutical standpoint, many physicians I have spoken to on the subject, seem to prefer an ointment that is not freshly prepared as required by the U. S. P., but claim that an older ointment has the same effect with milder action.

DEPARTMENT OF PHARMACY, COLLEGE OF JERSEY CITY.

MAGNESIUM PHOSPHATE IN POPPY CAPSULES.

J. E. KIMLEL, PH. C.

Whether or not the knowledge of the existence of magnesium phosphate in poppy capsules is new, the writer is unable to state. The literature, so far as investigated, makes no mention of it.

Since the existence of the anti-narcotic law, preparations of poppy heads will undoubtely receive more attention from the chemist.

Morphine is the important constituent and varies, according to King, from 1 to 2 percent.

To the busy chemist with little time for research, the U. S. P. method for determination of morphine in opium preparations will probably be applied, which may lead to very serious error in the final result.

Magnesium phosphate being slightly soluble in the hydro or hydro-alcoholic menstruum used, is extracted from the ground capsules and remains in the finished product.

In the course of assay, using the fluidextract for example, the extract is concentrated, ether and alcohol added and ammonia water to liberate the morphine. The whole is then vigorously shaken and set aside to allow the morphine to crystallize.

The magnesium phosphate originally present in the capsules and again in the fluidextract is by this process converted into insoluble ammonium-magnesium phosphate, which crystallizes out along with the morphine, providing enough ammonia water has been added to render the whole alkaline, otherwise no morphine will be found in the residue.

The ammonium magnesium phosphate resembles minute crystals of morphine and with a little washing is rendered bright and clean, still retaining a yellowish color, imparted by the mother liquor.

If now the residue is weighed and calculated as morphine, without further purification, as is sometimes done in the assay of opium (the writer is not discussing the propriety of such an omission) the analyst's report will not show the correct morphine content.

From this it can easily be seen that any method for the determination of morphine in poppy capsules that depends upon the crystallization of the morphine