

SCIENTIFIC SECTION

BOARD OF REVIEW ON PAPERS.—*Chairman*, L. W. Rowe; John C. Krantz, Jr.; F. J. Bacon.

STUDIES ON COMMERCIAL PSYLLIUM SEEDS.*

BY HEBER W. YOUNGKEN.

INTRODUCTION.

Psyllium Seed, a drug unknown to the majority of the people of this country 10 years ago, has suddenly loomed into popularity as a laxative and demulcent and its use appears to be gaining in momentum. The drug has been in use in Asia and Europe for more than a century, but until the past 4 or 5 years, only to a very limited extent in America. Its mucilage has been employed in Europe as a cosmetic and in the stiffening of fabrics.

The present commercial article has been varied and represents seed of various species of the genus *Plantago* which are related to our Common Plantain, *Plantago major*, a pestiferous weed in lawns and fields, the leaves of which have been used by the older people as a stimulating application to sores, frequently in the form of a poultice.

The chief commercial varieties in the American trade to-day are the French or black Psyllium Seed, the blonde or white Psyllium Seed also known as Indian Plantago Seed and the German Psyllium Seed. It is conservatively estimated that about 3000 tons of seed of these varieties were imported into this country during the past year, the bulk from France and India. The ratio of imports of the Black and Blonde varieties in 1931 were 5 to 1, in 1930 they were 2:1 (1).

In addition to the three chief commercial varieties, Spanish and Italian Psyllium Seeds have occurred in smaller amounts and a number of seeds purported to be Psyllium have been offered to the trade. The various commercial kinds can be purchased as "imported," "cleaned," "triple cleaned" and as "cleaned and sterilized."

On account of standards being lacking for Psyllium in the United States, considerable seed of inferior grade has been sporadically offered in trade, and even substitutes having no relationship to Psyllium have been marketed. One of the



Fig. 1.—*Plantago Psyllium* L., the source of French or Black Psyllium seed. (Photograph, the courtesy of Gray Herbarium.)

* Presented to the Scientific Section, Toronto meeting, 1932.

more recent of the latter has been the small fruit of a member of the *Labiatae* or Mint Family, namely, *Lallemantia royleana* Benth. Roasted German Psyllium has been offered under the label of French Psyllium Seed. The seed of a Plantain grown in New York State have been offered as Black Psyllium.

This investigation was made for the purpose of acquiring scientific data which would aid in the standardization of Psyllium Seed. It began over 2 years ago, since which time the writer has examined a large number of samples obtained from various trade sources. Seed of different types were planted in seed boxes and in the open and a number of seedlings brought to maturity and the plants and seed compared with authentic herbarium specimens in the Gray Herbarium of Harvard University, in order to establish the correct botanical identity of the commercial material.

Macroscopic and microscopic studies were also made on the French, Blonde and German varieties as well as determinations of the total ash, acid-insoluble ash and mucilage content of each of these, which will now be separately discussed.

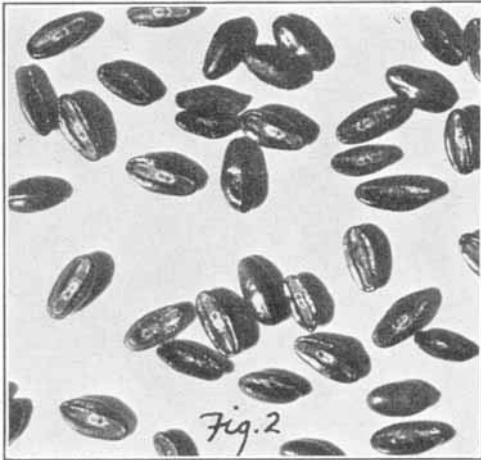


Fig. 2.—French Psyllium Seed $\times 4$ yielded by *Plantago Psyllium* L.

FRENCH PSYLLIUM SEED.

This represents the dried seed of *Plantago Psyllium* L. (Fam. *Plantaginaceae*). This seed is also called Flea Seed, Black Psyllium Seed and Brown Psyllium Seed.

Most of the commercial samples examined which were labeled "French Psyllium Seed" consisted in the main of the seed of *Plantago Psyllium* L., an annual caulescent, glandular, pubescent herb native to Mediterranean countries. It has been cul-

tivated extensively in France, which country to-day yields the bulk of our imports of this variety.

The seeds were hemianatropous, elliptical to ovoid, dark brown to reddish brown, shining, from 2 to 2.5 mm. or occasionally 3 mm. in length and from 1 to 1.2 mm. in breadth, concavo-convex with a light brown longitudinal area extending lengthwise along the center of the upper surface and representing the embryo as viewed through the somewhat transparent coat. The lower surface showed a deep concavity in the center of the base of which was an oval, white scar representing the hilum. Some seeds showed a whitish raphe extending from the center of the base of the concavity to one end.

When this seed was soaked in water its seed coat swelled and the seeds became enveloped with a transparent mucilage which was odorless and tasteless.

One gram of seed, agitated occasionally in a cylinder with 8 cc. of water, formed a thick tasteless jelly with the seeds in suspension.

A mucilage determination was made on each of 2 samples of cleaned French

Psyllium Seed. Sample 1 yielded 12.470% and Sample 2 yielded 11.098% of mucilage.

Total ash and acid-insoluble ash determinations made on 5 of the samples of French Psyllium gave the following results: All were ashed whole.

Sample No.	Per Cent Total Ash.	Per Cent Acid-Insoluble Ash.
1 (cleaned)	2.61	0.715
2 (cleaned)	2.54	0.711
3 (clean-looking)	3.42	0.080
4 (clean-looking)	3.28	0.180
5 (clean-looking)	3.75	0.250

Histology.—Transverse sections of *P. Psyllium* seed cut through the center of the seed possess a reniform outline and present for examination a spermoderm, endosperm and embryo. The spermoderm shows (1) an outer epidermis of mucilaginous epidermal cells with more or less obliterated walls, forming a clear mucilage when coming into contact with water, (2) a hyaline middle layer seen only in parts of groove region and (3) a pigment layer with brown amorphous content. Directly beneath the spermoderm lies the broad endosperm composed of irregular shaped, thick-walled cells with walls of reserve cellulose, and intercellular-air-spaces. The outer layer of this region consists of palisade cells from about 18μ to 46μ in height. The contents of these cells consist of aleurone grains and fixed oil. The straight embryo lies in the center of the endosperm and consists of 2 plano-convex cotyledons and a cylindrical hypocotyl. The cells of the embryo contain aleurone grains and fixed oil. The aleurone grains measured from 2 to 8μ in diameter and were of rounded, oval, pyriform and irregular shapes. Most of them were under 4μ . Three plerome bundles extend through each of the cotyledons.

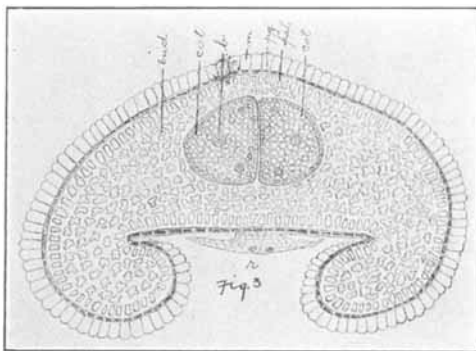


Fig. 3.—Transverse section of French Psyllium Seed (*Plantago Psyllium* L.) as observed in a glycerin mount ($\times 38$); *m*, mucilaginous epidermis and *pg*, pigment layer of seed coat; *end*, endosperm; *pal*, palisade layer of endosperm; *cot*, cotyledon; *b*, vascular bundle; *r*, raphe.

Impurities.—These consisted of foreign seeds, including those of the Cruciferae, membranous fragments of pyxes and tiny snail shells with sharp edges.

BLONDE PSYLLIUM SEED.

This represents the dried seed of *Plantago ovata* Forskall (Fam. *Plantaginaceae*), an annual acaulescent herb native to Asia and Mediterranean countries. It has also been termed in commerce White Psyllium Seed, Indian Plantago Seed and Ispaghula Seed. The plant has been extensively cultivated in India.

Most of the commercial samples labeled as above, which were examined, consisted in the main of this seed.

The seeds were broadly elliptical or ovate, cymbiform, pale grayish brown with a pinkish tinge and with a dull surface, light brown where abraded, averagely from 2 to 2.5 mm. occa-

sionally up to 3 mm. in length and from 1 to 1.5 mm. in width. On the convex surface there is a small, elongated, shining brown spot representing a portion of the embryo as seen through the seed coat. On the concave surface is a deep excavation at the base of which is the hilum, covered with a thin whitish membrane which extends to one of the edges. This represents the raphe, and the seed is thus hemianatropous.

Upon soaking this seed in water the seed coat swelled and the seeds became enveloped with a transparent, colorless mucilage. Under the microscope the epidermal cells elongated and their outer and radial walls became transformed to mucilage.

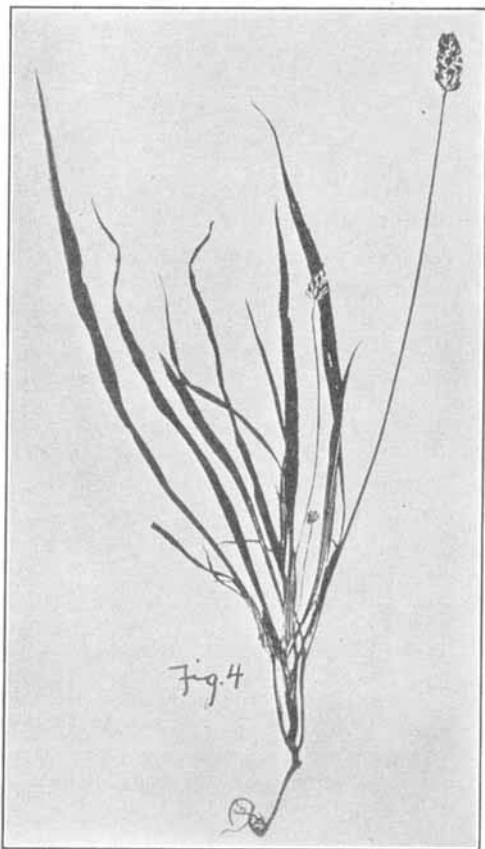


Fig. 4.—*Plantago ovata* Forskall, the source of typical Blonde Psyllium seed. Grown by author from commercial seed. $\times 1/3$.

One gram of this seed, occasionally agitated in a cylinder with 15 cc. of water, formed a thick, transparent, colorless mucilage which was devoid of taste or odor. Another sample yielded a thick mucilage when 1 Gm. of seed to 20 cc. of water were used.

Clevenger reports this seed for some time on the market as yielded by *Plantago Loeflingii* L. (2). The writer has grown mature plants from the commercial seed resembling that which he describes and compared them with authentic herbarium material in the Gray Herbarium of Harvard University and found these to represent *Plantago ovata* Forsk.

The main distinctions between *Plantago ovata* and *P. Loeflingii* plants can be determined through the dissection and examination of their flower parts. The petal lobes of *P. ovata* are oval with a mucronate summit whereas those of *P. Loeflingii* are lanceolate with an acute summit. The bracts of *P. ovata* are rounded ovate, glabrous, with a herbaceous midrib extending to the obtuse tip and scarious margined, whereas those

of *P. Loeflingii* are transversely ovate, broader than long, glabrous or ciliate at margin with a herbaceous midrib excurrent in a broad obtuse point. The sepals of *P. ovata* are marked with a distinct midrib extending from base to summit, whereas those of *P. Loeflingii* show only a rudimentary midrib in the basal portion. The foliage leaves of *P. Loeflingii* tend to develop longer marginal teeth than those of *P. ovata*.

Mucilage determinations were made on each of 2 samples of cleaned *Blonde Psyllium* Seed. Sample 1 yielded 32.08% and Sample 2 yielded 29.65% of mucilage.

Total ash and acid-insoluble ash determinations made on the following samples of Blonde Psyllium Seed gave the following results. All were ashed whole.

Sample No.	Per Cent Total Ash.	Per Cent Acid-Insoluble Ash.
1 (cleaned)	2.30	0.74
2 (cleaned)	2.31	0.72
3 (clean-looking)	2.70	0.36
4 (clean-looking)	2.63	0.38

Histology.—Cross sections of this seed exhibited a reniform outline and showed a spermo-derm composed of a colorless outer epidermis of radially elongated mucilage cells which greatly swell in water, becoming several times their normal size and their walls become more or less obliterated. In surface section, these cells are irregularly polygonal. Beneath this in sections cut through the center of the seed occurs a brown pigment layer. A somewhat obliterated, middle hyaline layer occurs in the ends and edges of the groove. Directly beneath the spermo-derm is a broad endosperm composed of a layer of palisade cells and many irregular shaped layers of reserve parenchyma cells with thick walls of reserve cellulose and large air-spaces. The cells contained aleurone grains mostly under 4μ in diameter and fixed oil droplets. The straight embryo, consisting of 2 plano-convex cotyledons and a hypocotyl, extends through the center of the endosperm region. Its more or less rounded parenchyma cells have contents similar to the endosperm. A perome bundle extends through the center of the hypocotyl and branches through each of the cotyledons.

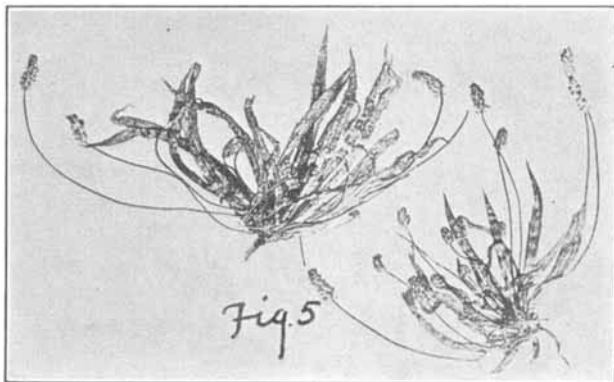


Fig. 5.—*Plantago Loeftingii* L. collected in Armenia. (Photograph, the courtesy of Gray Herbarium) $\times \frac{2}{3}$.

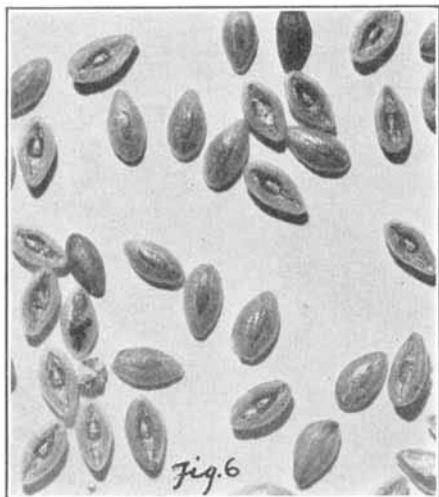


Fig. 6.—Blonde Psyllium or Indian Plantago Seed yielded by *Plantago ovata* Forskall. $\times 4$.

Impurities.—Foreign seeds, fragments of membranous pyxes and tiny snail shells.

GERMAN PSYLLIUM SEED.

This represents the dried ripe seed of *Plantago lanceolata* L. (Fam. *Plantaginaceae*), a perennial herb native to Europe and widely naturalized in grassy places and fields in this country. It occurs commonly as an adulterant of clover seed and is a constituent of some bird foods. This seed is also known as English Plantain Seed and the plant commonly as Ribgrass, English-Buck or Buckthorn Plantain and less commonly as Black

Jacks, Jack Straws, Dog's Ribs, Rattail, Chimney-sweeps and Snake, Lance-leaved or Ripple Plantain.

The seeds examined were hemianatropous, oval to elliptical to oblong ovate, glossy, light brown to dark brown, usually of both colors in commercial samples and no doubt representing different stages of maturity of the seed, from 2 to 2.5 mm. in length and from 1 to 1.2 mm. in breadth, concavo-convex, with a broad, longitudinal, yellowish brown area extending the length of the center of the upper surface and a deep excavation on the lower surface in the center of the base of which is an oval dark hilum from which in some seeds a raphe extends to one end.

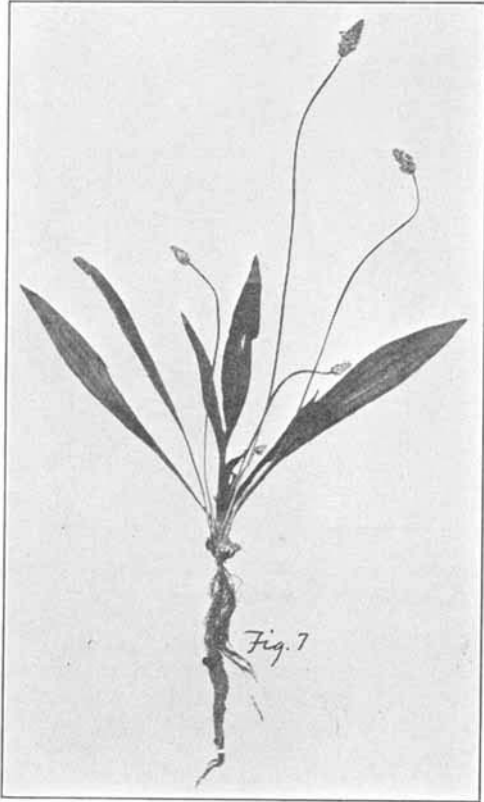


Fig. 7.—*Plantago lanceolata* L., the source of German Psyllium Seed. $\times \frac{1}{3}$.

When soaked in water the seed coat swelled and became enveloped with a thin mucilage, not as stringy or as thick as in the case of that of the French or Blonde varieties of Psyllium. As with other *Plantago* seeds the mucilage formed is membrane mucilage from the epidermal cell walls.

One gram of seed, occasionally agitated in a cylinder with 4 cc. of water, formed a moderately thick tasteless mucilage. Clevenger (2) gives the swelling factor of *P. lanceolata* seeds as approximately 4. On two samples tested by his method I obtained a swelling factor of 3.5 and 4, respectively. Mucilage determinations were made upon 2 commercial samples of this seed. Sample 1 yielded 11.92% and Sample 2 yielded 10.87% of mucilage.

Total ash and acid-insoluble ash determinations made on the following samples of German Psyllium Seed gave the following results: All were ashed whole.

Sample No.	Per Cent Total Ash.	Per Cent Acid-Insoluble Ash.
1 (cleaned)	2.46	0.374
2 (cleaned)	2.64	0.333
3 (cleaned)	2.68	0.33
4 (cleaned)	2.64	0.33
5 (as imported)	3.65	0.54

Impurities.—The foreign organic matter in the samples studied included portions of spikes and pyxes of the same species, foenugreek seed and gramineous seed.

Histology.—Transverse sections of the seed of *P. lanceolata* showed a spermoderm consisting of an outer epidermis of tangentially elongated epidermal cells with mucilaginous walls

and contents and a pigment layer of thin-walled cells with brown, amorphous contents. . On the ends and in the edges of the excavation a middle layer of thin-walled cells is also evident as shown by Kraus (3) and Winton (4). Beneath the spermoderm a broad endosperm is to be found consisting of an outer palisade layer and of inner irregular shaped cells all with thick walls of reserve cellulose and with aleurone and fixed oil contents. Protoplasmic connections were evident between these cells. The embryo, consisting of 2 cotyledons of plano-convex appearance in cross sections and a terete hypocotyl, extends straight through the center of the endosperm. Its cells are thin-walled and contain aleurone and fixed oil.

SPANISH PSYLLIUM SEED.

Seven samples of commercial Spanish Psyllium were examined. Three of these showed the seed to be of the French type but of averagely lighter color and smaller. Three others were identical in every respect with French Psyllium Seed (*P. Psyllium*), previously described in this article. One of them proved to be identical with *Plantago lanceolata* seed.

The light colored Spanish Psyllium Seed was from 1.4 to 2.7 mm. in length, and 0.8 to 1 mm. in breadth; light brown in color becoming dark brown along the edges of the concave surface in some seeds, and in other seeds also showing a transitory darkening on parts of the convex surface. The seeds gave a silky sensation to the touch.

Although mature plants from seed samples of the lighter Spanish type of Psyllium have not as yet been reared by the writer, it seems likely that this light colored variety of Spanish Psyllium Seed may represent either immature seed of *Plantago Psyllium* L. or possibly the seed of *Plantago arenaria* Waldst. et Kit., a closely related species.

One gram of light colored Spanish Psyllium, occasionally agitated with 8 to 10 cc. of water, formed a thick odorless and tasteless jelly with imbedded seed. This jelly appeared thicker than that yielded by the French variety.

IDENTITY OF NEW YORK GROWN BLACK PSYLLIUM.

New York Black Psyllium Seed was found upon comparative study to be identical with *Plantago Rugelii* Dene.



Fig. 8.—German Psyllium Seed (*Plantago lanceolata*). × 2.5.



Fig. 9.—*Plantago Rugelii* Seed from a sample grown in New York State and marketed as Black Psyllium. × 3.

QUALITATIVE MUCILAGE TESTS.

Tests were made on the filtered mucilage of cleaned seed of the first three varieties with litmus paper, 95% alcohol, lead acetate T.S., saturated solution of borax, iodine T.S., fuchsin-sulphurous acid, Millon's reagent and concentrated sulphuric acid with the following results:

Seed Variety.	Litmus.	Lead Acetate T.S.	95% Alcohol.	Sat. Sol. Borax.	Iodine T.S.	Fuchsin-Sulphurous Acid T.S.
French	Blue	No immediate change; slight ppt. later	Stringy precipitate	No change	Negative	Negative
Blonde	Blue	Slightly bluish cloudy ppt.	Precipitate	No change	Negative	Negative
German	Pink	Very cloudy, and if reagent is floating on mucilage, a line of ppt. is evident	Slight precipitate	No change	Negative	Negative (Upon heating a clear pink solution in all three)

Millon's reagent produced a white flocculent precipitate in all three. Cold concentrated sulphuric acid formed a clear solution with all three.

The mucilage of The French and Blonde varieties is colorless, while that of the German Psyllium is light brown.

SWELLING FACTORS OF SEEDS.

The following test was made upon lots of the different varieties of commercial seeds in order to obtain their swelling factors:

One gram of each sample was placed in a 50-cc. graduated cylinder and tap water added to the 20-cc. mark (to the 50-cc. mark with *Lallemantia royeleana*). The cylinder was shaken at intervals during a period of 24 hours, at the expiration of which time the seeds were allowed to settle, and the total volume occupied by the swollen seeds noted. The final reading in cubic centimeters was taken as the swelling factor. The results of this test for the several lots was as follows:

French Psyllium	8
Blonde Psyllium	11 to 13
German Psyllium	4
Spanish Psyllium	12 to 14
<i>Lallemantia royeleana</i>	40

This modification of the method proposed by J. F. Clevenger (2), appears to have the advantage of permitting more mucilage formation by the seeds. It is suggested as a better index of their mucilage forming capacity.

CONCLUSIONS.

(1) Typical French and the Dark Spanish varieties of Psyllium Seeds of the American market are yielded by *Plantago Psyllium* L.

(2) A sample of Black Psyllium collected and marketed as from plants grown in New York State was found to be yielded by *Plantago Rugelii* Dene.

(3) Typical Blonde Psyllium is the product of *Plantago ovata* Forsk., rather than of *P. Loeflingii* as previously reported.

(4) Of all the Commercial Psyllium seeds examined, those yielded by *Plantago ovata* rank highest in mucilage content and therefore in demulcent qualities with the seeds of *Plantago Psyllium* ranking second. *Lallemania royeleana* fruits yield more mucilage than Psyllium seeds but must not be regarded as even a variety of Psyllium.

(5) The mucilage yielded by the seeds of *Plantago Psyllium* and *Plantago ovata* is of superior quality to that yielded by the seeds of *Plantago lanceolata*.

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THE EFFECT OF VARIOUS CONDITIONS OF STORAGE ON THE POTENCY OF TINCTURE OF DIGITALIS.*

BY HERBERT M. EMIG.¹

I. DEFINITION.

One of the most important preparations appearing in the United States Pharmacopœia is tincture of digitalis. When this drug is prescribed a definite action upon the heart is expected. Often this characteristic digitalis action—slowing of the rate, decrease in conductivity, increase in irritability, lengthening in diastole and more effective systole, is not obtained, or is only weakly exhibited by old preparations. Although such tinctures at the time of manufacture assay full strength, at the end of the period of storage biological tests show that they have undergone a loss of all or part of their potency.

This work represents a study of the influence of several factors upon the keeping qualities of the tincture during storage. The factors concerned were temperature, hydrogen-ion concentration of the extracting menstruums, exclusion of air by sealing and displacement of air in container with carbon dioxide.

II. INTRODUCTION.

Numerous investigations considering the effect of changes in the hydrogen-ion concentration upon the potency of the tincture as well as the infusion of digitalis have been performed. Pittenger (1), as well as Wokes (2), found that a normal tincture lost 30% of its potency during a sixteen months' period. Wokes also observed that a variation in p_H value of the tincture over a range of 5.47 to 5.93 decreased its potency, and when a tincture was made distinctly alkaline (p_H 8) an increase in the rate of deterioration occurred. Contrary to the findings of Wokes,

* Scientific Section, A. P. H. A., Toronto meeting, 1932. No discussion.

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