

The method I have used is as follows: One grain of ground capsicum is macerated over night in 100 cc. of alcohol. After thorough shaking, filtered. This alcoholic solution is then added to sweetened water in definite proportions until a distinct but weak pungency is perceptible on the tongue.

By this method, Japan Chillies tested 1 in 20,000 to 1 in 30,000, Zanzibar Chillies 1 in 40,000 and 1 in 45,000 (two lots), and Mombassa Chillies 1 in 50,000 to 1 in 100,000. From a limited number of tests the Mombassa brand appears to be decidedly stronger in capsaicin. We have not had it under observation long enough to decide on a limit of acceptability that will represent the average of the drug, but there appears to be no trouble in obtaining it of a strength of 1 in 50,000 or above.

Oleoresin of capsicum may test 1 in 150,000 and upwards. When used as a rubefacient, flavor is of no consequence, but a high capsaicin content is desirable.

It may be of interest to state that commercial capsicums vary also in fat-content and color to a marked degree. Oleoresins were examined which contained as little as 5 per cent. of fat insoluble in alcohol, while others contained above 50 per cent., yet the more pungent oleoresin (based on the entire mixture) were those containing considerable fat. The fat in some instances was a marked green—quite free from red; in others it was orange and in others a deep red; no relation of color or fat to pungency could be observed.

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DISCUSSION.

Mr. Beringer said that much fat would always be found in a well-developed fruit containing well-developed seeds, and that in the selection of capsicum we should avoid large matured fruits in which the seeds were fully developed.

Mr. Raubenheimer inquired of the author of the paper whether he had discovered any relation between the color of the ground capsicum and the finished tincture? In his experience he had not been able to discover any such relation. No matter what was the tint of the powdered drug the tincture always had a reddish color.

Mr. Eldred inquired if the quantity of fat in the oleoresin did not bear some relation to the pungency?

Mr. Scoville, in reply, stated that his work had begun as a study of the oleoresin, separating the fats insoluble in alcohol. He found the fats to vary from 5 to 50 percent. One containing 5 percent of fat was comparatively weak in pungency, those having the larger pods being more pungent, though he did not believe that this was due to their containing a large amount of fat; neither could he discover any relation between the pungency and the color of the capsicum.

The only test he had found to be satisfactory was the physiological test. He had examined a number of samples which tested 1 to 100,000.

THE ASH CONTENT OF DRUGS.

M. I. WILBERT.

In recent years there has been evidenced a growing disposition to place considerable reliance on the ash content of drugs as an aid in determining the nature and purity of the product under examination.

With a view of ascertaining what if any uniformity exists in the permissible

ash content of official drugs an analysis of the requirements made in 10 of the recently published pharmacopœias was made and the maximum ash content of some of the more widely used drugs is herewith presented in the form of a table.

Restricting the permissible quantity of ash in connection with vegetable or crude drugs is a comparatively modern requirement. It was introduced in the second edition of the German Pharmacopœia, published in 1882 and also appears in connection with a limited number of the drugs described in the U. S. P. of the same period. The number of official limitations for ash was increased but slowly and in the German Pharmacopœia for 1900 we find but twelve while in the corresponding U. S. P. VIII there are twenty such requirements in connection with the monographs for crude drugs.

The Netherlands Pharmacopœia published in 1905 appears to have been the first of the more widely known pharmacopœias to include an appreciable number of ash determinations; a total of 41.

In the Ph. Austr. VIII, published in 1906, this number is increased to 147, the maximum up to the present time, though the aggregate of the Ph. Helv. IV is nearly if not quite as great.

The Ph. Svec. IX, published in 1908, contains but a comparatively few definite figures, and the Ph. Hung. III, published in 1909, despite the fact that it follows the Austrian Pharmacopœia in many of the official requirements, includes but a limited number of limitations for ash.

The German Pharmacopœia which for some decades appears to have served as a model for the elaboration of our own U. S. P. has been continued within conservative lines and the new D. A. B. V. published in 1910 contains but a total of 34 requirements for ash content.

The impracticability of deducing any definite generalizations from the permissible limitations for ash included in the several pharmacopœias is well illustrated by the appended table. For many of the drugs the figures vary from 10 to 100 per cent. and in the limited number of cases where there is little or no variation, lupulin, for instance, the figures given have been found to be altogether too low for the commercially available product.

The variation in the actual ash content of drugs necessarily depends on many factors that are entirely beyond the control of the pharmacist or the dealer in drugs but the frequently observed variation in the ash content of the same sample, or lot of a drug is due to causes that are deserving of careful consideration on the part of the revisers of the Pharmacopœia. The fundamentally important factors for securing uniformity are to be sought in the method of incineration and the method of sampling employed therewith.

In the routine work of the ordinary analytical laboratory it is impracticable to incinerate more than 1 or 2 gm. of a sample of crude drug and it is quite apparent that it would be difficult indeed to secure a representative sample of a root, bark, leaf or herb that could be relied upon without resorting to comminution and subsequent mixing of an appreciable quantity of the drug.

This difficulty of securing representative samples of many crude drugs has no doubt deterred the revisers of some of the more recent pharmacopœias from adopting the ash content factor more freely.

It is generally agreed that the exact method of determining the residual ash should be described so as to obviate, if possible, the likelihood of the residue retaining an undue amount of unconsumed carbon.

The Ph. Austr. VIII despite the fact that it includes upwards of 150 limitations for the ash content of drugs does not provide a method for determining this rather important requirement, and the several critics of this Pharmacopœia have not failed to assert that the commission in charge of the revision adopted theoretic rather than practical standards for many of the pharmacopœial drugs.

The Ph. Helv. IV directs that ash determinations are to be made by heating from 1 to 2 gm. of the substance at first moderately, with a low flame, and then gradually increasing the temperature until the residual ash is free from carbon.

The nature of the container in which the substance is to be incinerated is not specified and no provisions are made for aiding the combustion of protected carbon particles.

The new German Pharmacopœia process is much more complete. It directs that a suitable quantity of the substance is to be incinerated in a recently heated and tared crucible and in the event that complete combustion of the carbon particles is not brought about by continued, moderate, heating the material is to be leached out with hot water and the residual carbon again heated, as before. The resulting solution is subsequently evaporated and the weight of the dry residue is added to that of the ash.

This, Ph. Germ. V, method has been liberally criticised, many pharmacists believing that the leaching out method is much more time consuming than the methods which involve the use of clean sand for distributing the particles of carbon or the use of oxygen carriers such as nitric and oxalic acids for facilitating combustion.

Considerable difference of opinion appears to exist regarding the desirability of determining the ash, and other analytical factors, on the air dried drug or on the drug dried to constant weight in an exsiccator.

In view of the fact that it is the air dry drug that appears in commerce and is generally used in the making of galenical preparations as well as dispensing it would appear preferable to base pharmacopœial requirements on the commercial drug and to add such other restrictions as may be found necessary to limit the percentage of contained moisture.

This is apparently the view taken by the revisers of the German Pharmacopœia as that authority now requires that the official tests are to be applied to the air dried substances unless otherwise directed.

From the available evidence it would appear that the determination of the ash content of official drugs is practicable and important in connection with non-structural drugs, like gums and resins, pollen grains, seeds, spices and powdered drugs generally.

It is not generally applicable to leaf drugs, barks or roots in the uncomminuted form because of the difficulty of sampling.

To insure correlating results the method to be employed must be described, and, other things being equal, this method should be one that can be easily followed by retail druggists ordinarily well equipped for work of this kind.

TABLE SHOWING THE MAXIMUM ASH CONTENT OF SOME WELL-KNOWN DRUGS INCLUDED IN 10 OF THE RECENTLY PUBLISHED PHARMACOPŒIAS.

Title of Drug.	Ph. Germ. V	Ph. Hung. III	Ph. Ital. III	Ph. Fr. V	Ph. Svec. IX	Ph. Helv. IV	Ph. Aust. VIII	Ph. Belg. III	Ph. Ndl. IV	Ph. U.S.P. VIII
Acacia	5.0	5.0	4.0	...	5.0	4.0	3.0	5.0	4.0	4.0
Adeps Lanæ	0.1	0.05	0.05	0.10	0.30
Aloe	1.0	...	2.0	1.5	...	1.5	1.0	...	1.5	...
Althæa	6.0	6.0	7.5	7.0	...
Amylum	1.0	0.5	1.0	1.0	...	0.5	0.5	1.0	1.0	...
Anisum	10.0	10.0	10.0	12.0
Asafoetida	15.0	...	10.0	10.0	10.0	20.0	10.0	10.0	10.0	15.0
Belladonnæ Folia	15.0	15.0	15.0
Benzoinum	2.0	...	2.0	2.0	...	1.5	2.0	...	2.0	2.0
Calumba	8.0	6.0
Cantharis	8.0	...	7.0	8.0	8.0	...	9.0	8.0
Capsicum	6.5	5.0	6.5	6.5
Carbo Ligni	5.0	...	2.0	2.0	2.0	...
Cardamomum	10.0	8.0	...	8.0	4.0
Carum	8.0	8.0	7.0	8.0
Caryophyllus	8.0	7.0	8.0	...	6.0	8.0
Cinchona	6.0	6.0	6.0	...	8.0	...
Cinnamomum Zeylanicum	5.0	5.0	5.0	7.0	8.0	4.0
Coccus	6.0	6.0
Cubeba	8.0	...	9.0	8.0	9.0	...	10.0	...
Digitalis	10.0	10.0	12.0
Ergota	5.0	5.0	5.0	...	5.0	...
Foeniculum	10.0	10.0	10.0	12.0
Gelatina	2.0	...	2.0	2.0	...	2.0	2.0	2.0	3.0	...
Gentiana	6.0	5.0	7.0	6.0	...
Glycyrrhiza	6.0	6.0	7.5	6.0	...
Gossypium Purificatum	0.3	...	0.3	0.4	...	0.5	0.5	0.3	0.3	0.3
Granatum	15.5	10.0	...	15.0	...
Hydrastis	6.0	6.0	6.0	...	6.0	...
Hyoscyamus	24.0
Ipecacuanha	4.0	4.0	5.0	...	6.0	...
Jalapa	6.5	...	4.5	6.5	5.0
Linum	5.0	...	6.0	5.0	5.0
Lupulinum	10.0	10.0	10.0	10.0
Lycopodium	3.0	...	4.0	3.0	3.0	4.0	5.0	5.0
Manna	3.0	...	3.5	...	4.0	3.0	4.0
Mel	0.8	...	0.4	...	0.5	0.8	0.4	0.5	...	0.3
Myrrha	7.0	...	6.0	...	6.0	6.0	6.0	6.0	5.0	...
Nux Vomica	3.0	3.5	3.0
Opium	6.0	6.0	6.0
Rhamnus Purshiana	6.0	10.0	...
Rheum	12.0	...	12.0	13.0	12.0	...	12.0	...
Saccharum	0.1	0.075	0.1	...
Saccharum Lactis	0.25	0.2	0.1	0.25
Scilla	5.0	5.0	8.0
Senna	12.0	12.0	10.0	12.0	8.0	...
Sinapis	5.0	5.0	5.0	5.0	8.0	...
Stramonium	20.0
Valeriana	12.0	10.0	15.0
Zingiber	7.0	7.0	5.0	...	8.0	...

DISCUSSION.

Mr. Beringer stated that the figures for ash content in Mr. Wilbert's paper were very similar to other compilations, and inquired whether any of the results given had been confirmed by his own experiments or were taken from published results.

Mr. Wilbert stated that both the Netherlands and Austrian Pharmacopœias had been severely criticised on their generally low ash content requirement for drugs and that the figures cited were largely academic. Practically all the pharmacopœial standards are in sub-

stantial agreement for the ash content of Lupulin, but are not in accordance with actual commercial conditions, which are nearly twice as high as pharmacopœial standards.

Observations relating to ash content, in order to be of practical value, must cover hundreds of thousands of samples, be carried over a series of years and made by a number of observers.

In the Digest of Criticisms of the Hygienic Laboratory an effort had been made to compile ash determinations recorded by different individuals, and also the work done in European laboratories in connection with spices. For the majority of vegetable drugs he thought it necessary to be content with ash determinations, permitting rather wide limitations for drugs in the powdered form.

THE COMPOSITION OF GELSEMININE.

L. E. SAYRE.

The alkaloids of Gelsemium have been investigated at intervals since 1869, by Wormley, Gerrard, Robbins, Sonnenschein, Thompson and others. These valuable contributions have been referred to in previous papers, published in the proceedings of this association since 1907.

It was not until 1887 that F. A. Thompson announced the existence of a second alkaloid in the root which he named Gelseminine. The study of this second principle was left by Thompson for others who might in the future have the time and the inclination to do it. The result of my study of this principle seems to indicate, as stated in former papers, that the Gelseminine of Thompson is not a definite and simple body. Recent study confirms this opinion. Other confirmation, than my own work, has been given by a recent analyst, Charles Watson Moore, whose paper was published in the Jour. Chem. Soc'ty, Nov., 1910, No. LXXVII, p. 2223. This author, after mentioning the less important principles of the alcoholic extract states:

"The portion of the alcoholic extract soluble in water from which the resin has been removed contained scopoletin (a monomethyl ether of esculetin) which was present in a free state and also a glucoside, together with some sugar. It also yielded three alkaloidal products, one of which was obtained in a pure crystalline form corresponding to Gelsemine. The other two were amorphous and non-crystalline, the one to which the name Gelseminine has been given being more basic than the other."

This unexpected confirmation of my observations, previously made, is especially gratifying.

In the April issue of the Bulletin of the A. Ph. A. an article appears by Kimberly, Roberts and Vanderkleed, in which a suitable assay of Gelsemium is discussed. These men state that the activity of the drug depends primarily upon the so-called alkaloid Gelseminine, and only secondarily upon the more readily obtained Gelsemine, which exists in much larger proportion. These investigators fail to recognize the presence of three alkaloids, but this does not detract from their valuable contribution. The existence of three alkaloids, however, makes the assay of the drug still more complicated. A chemical assay can be made reliable only when we *know* the principles we have to consider.