

REPORT OF THE COMMITTEE ON UNOFFICIAL STANDARDS

The following portion of the report of the Committee on Unofficial Standards relates to certain crude drugs and chemicals suggested for inclusion in the next revision of the National Formulary, and by order of the Council is published in the Journal in order to afford opportunity for discussion before the standards proposed are finally adopted.

Manufacturers, importers, analysts, and others interested in any of the proposed standards, are requested to send their criticisms and comments to the chairman of the committee, George M. Beringer, 501 Federal Street, Camden, N. J.

ÆTHER ACETICUS

Acetic Ether Ethyl Acetate

A liquid containing not less than 96 percent by volume of Ethyl Acetate ($C_4H_8O_2$ or $CH_3CO.OC_2H_5 = 88.06$), the remainder consisting of alcohol and a little water. Keep it in well-stoppered bottles, in a cool and dark place, remote from lights or fire.

Acetic Ether is a transparent, colorless liquid, of a fragrant and refreshing, slightly acetous odor, and a peculiar acetous and burning taste.

Specific gravity: 0.883 to 0.895 at 25° C.

One mil of Acetic Ether is soluble in about 10 mils of water at 25° C.; miscible with alcohol, ether, fixed and volatile oils.

It does not immediately redden blue litmus paper.

Boiling-point: from 72° to 77° C.

Acetic Ether is readily volatilized, even at a low temperature. It is inflammable, burning with a yellowish flame and an acetous odor.

Upon evaporating 10 mils of Acetic Ether no weighable residue remains.

On allowing 10 mils of Acetic Ether to evaporate spontaneously from clean, odorless blotting paper, the final odor does not resemble that of pineapple (*butylic and amylic derivatives*).

Pour carefully 2 mils of Acetic Ether on 10 mils sulphuric acid; no dark ring is developed at the point of contact of the two layers within fifteen minutes (*readily carbonizable organic impurities*).

Mix 20 mils of Acetic Ether with a solution of 20 Gms. of potassium hydroxide in 50 mils of distilled water, allow the mixture to stand, agitating occasionally, or heat it gently under a reflux condenser, until a ho-

mogeneous liquid results, and then distil about 25 mils. The distillate complies with the U. S. P. requirements in the test for Methyl Alcohol described under Alcohol.

Shake 25 mils of Acetic Ether in a graduated tube, with 25 mils of water just previously saturated with Acetic Ether; upon separation the ethereal layer measures not less than 24 mils (undue proportion of *alcohol or water*).

CALCII LACTOPHOSPHAS

Calcium Lactophosphate

A mixture of calcium lactate, acid calcium lactate, and about 3 percent of calcium phosphate.

Calcium Lactophosphate occurs in white granules or as a white powder, without odor.

It is soluble in water, almost insoluble in alcohol.

Its aqueous solution is acid to litmus.

The addition of ammonium oxalate T. S. to an aqueous solution of Calcium Lactophosphate produces a white precipitate insoluble in acetic acid, but soluble in hydrochloric acid.

Add 10 mils of diluted sulphuric acid and 5 mils of potassium permanganate T. S. to 5 mils of an aqueous solution of Calcium Lactophosphate (1 in 20) and gently warm the mixture; the odor of acetaldehyde is evolved.

Silver nitrate T. S. or ammonium molybdate T. S. produces in an aqueous solution of Calcium Lactophosphate (1 in 20) yellow precipitates.

Mix 0.5 Gm. of Calcium Lactophosphate with 5 mils of sulphuric acid; not more than a slight coloration results (*readily carbonizable impurities*).

Ten mils of an aqueous solution of Calcium

Lactophosphate (1 in 100) mixed with 1 mil of diluted hydrochloric acid does not at once become turbid on the addition of a few drops of barium chloride T. S. (*sulphate*).

Another portion of 10 mils of the aqueous solution (1 in 100) when mixed with a few drops of nitric acid yields not more than an opalescence on the addition of a few drops of silver nitrate T. S. (*chloride*).

The aqueous solution of Calcium Lactophosphate (1 in 50) does not respond to the U. S. P. test for Heavy Metals.

Mix 0.2 Gm. of Calcium Lactophosphate with 1 Gm. of potassium nitrate, ignite the mixture cautiously in small portions at a time until the carbonaceous matter is all or nearly all burned off, then add to the residue 20 mils of diluted sulphuric acid and evaporate the mixture until fumes of sulphuric anhydride are evolved. The residue complies with the U. S. P. requirement for arsenic.

Dissolve 1 Gm. of Calcium Lactophosphate and 2 Gms. of ammonium chloride in 75 mils of distilled water, heat the solution to boiling, and add an excess of ammonium oxalate T. S. (about 20 mils). Allow the precipitate to subside completely, then filter and supersaturate the filtrate with ammonia water; not more than a slight turbidity is produced (*magnesium*).

LITHII SALICYLAS

Lithium Salicylate
Lith. Sal.

It contains, when dried to constant weight at 120° C., not less than 98.5 percent of ($\text{LiC}_7\text{H}_5\text{O}_2 = 143.98$). Preserve it in well-closed containers, protected from heat and light.

Lithium Salicylate occurs as white or grayish-white powder; deliquescent in a moist atmosphere; odorless; taste sweetish.

Very soluble in water and alcohol.

When heated, the salt is decomposed, emitting the odor of phenol, and finally leaving a residue of lithium carbonate and carbon. It imparts a crimson color to a non-luminous flame.

The aqueous solution (1 in 20) is neutral or slightly acid to litmus, and when freshly made is colorless (*iron and organic coloring matters*), and does not effervesce upon the addition of diluted acids (*carbonate*).

Copper sulphate T. S. added to an aqueous solution of the salt (1 in 20) produces a bright green color.

Add a few drops of ferric chloride T. S. to an excess of an aqueous solution of the salt (1 in 4), a deep red color is produced. In dilute solution of the salt (1 in 100) ferric chloride T. S. produces a deep violet-blue color.

Hydrochloric acid added to an aqueous solution of the salt (1 in 20) produces a voluminous white precipitate of salicylic acid.

Dissolve 0.7 Gm. of the salt in 10 mils of distilled water, add an excess of hydrochloric acid and filter; evaporate the filtrate almost to dryness on a water-bath in a 50-mil Erlenmeyer flask, and then add 10 mils of amyl alcohol and cautiously heat until the lower aqueous layer is evaporated; then add 3 drops of hydrochloric acid and boil the liquid for three minutes; the insoluble residue weighs not more than 0.005 Gm. (limit of *other alkalies*). The removal of the water from the amyl alcohol mixture is facilitated by passing a current of air through the hot solution.

Dissolve 1 Gm. of the salt in 50 mils of distilled water, precipitate the salicylic acid by the addition of hydrochloric acid, and filter; the filtrate does not respond to the U. S. Pharmacopœia Test for Heavy Metals.

Assay.—Weigh accurately about 2 Gms. of Lithium Salicylate, previously dried to constant weight at 120° C., incinerate it in a porcelain crucible, and proceed by the method directed by the U. S. Pharmacopœia for the Assay of Alkali Salts of the Organic Acids. It shows not less than 98.5 percent $\text{LiC}_7\text{H}_5\text{O}_2$.

Each millilitre of half-normal sulphuric acid V. S. used corresponds to 0.07199 Gm. of $\text{LiC}_7\text{H}_5\text{O}_2$. Each gramme of Lithium Salicylate, previously dried at 120° C., corresponds to not less than 13.7 millilitres of half-normal sulphuric acid V. S.

MALVÆ FOLIA

Mallow Leaves
Malv. Fol.

The dried leaves of *Malva sylvestris* Linné or *Malva neglecta* Wallroth (Fam. *Malvaceæ*).

Leaves of *Malva sylvestris*, petiolate with petioles up to 10 cm. long, orbicular or reniform, slightly truncate or cordate at the base, 10 to 11 cm. long, 15 to 20 cm. wide, with three to seven shallow, angular or rounded lobes, venation palmate, margins crenate-dentate pubescent on both sides.

Leaves of *Malva neglecta*, with petioles up

to 20 cm. in length, orbicular up to 8 cm. wide, base deeply cordate, five to seven shallow rounded lobes, venation palmate, margins with rounded blunt teeth, pubescence more scant than in *M. sylvestris*.

Inodorous; on chewing it becomes very mucilaginous, taste bland.

Leaves showing brown fungous growths of *Puccinia Malvacearum* Montagne must not be used.

Examined under the microscope, sections show the epidermis containing mucilage cells; numerous stomata on both surfaces, each with three or four neighboring cells; trichomes of three distinct types, small, short-stalked glandular hairs, single, large one-celled curved hairs with thick walls and compound hairs in groups of two- to six-celled stellate hairs (the latter especially numerous in *M. sylvestris*); palisade tissue of one or two rows of cells, the mesophyl of three or four rows; the parenchyma containing calcium oxalate in rosette crystals and numerous mucilage cells, which are likewise present in the tissue of the veins; veins with collateral bundles with the sieve tissue toward the lower surface and collenchyma above.

The powder is light green and under the microscope exhibits the characteristic hairs, mucilage cells, epidermis with stomata, and rosette crystals of calcium oxalate.

Malya leaves yield not more than 16 percent of ash.

MANGANI SULPHAS

Manganese Sulphate
Mang. Sulph.

Crystallized Manganese Sulphate containing not less than 62 percent nor more than 68 percent of ($MnSO_4 = 151$).

Manganese Sulphate occurs as pale rose-colored, translucent, tetrahedral prisms; slightly effervescent when exposed. Odorless; taste slightly bitter and astringent.

1 Gm. dissolves in about 1 mil of water at 25° C. and in about 0.6 mil of boiling water; insoluble in alcohol.

The aqueous solution of the salt (1 in 20) is neutral or slightly acid to litmus. It yields with ammonium sulphide T. S. a salmon-colored precipitate soluble in dilute acids; and with barium chloride T. S. a white precipitate insoluble in hydrochloric acid.

Mix a fragment of Manganese Sulphate with a small amount of sodium hydroxide and fuse the mixture, it yields a green mass which dissolves in water to a green solution.

To 20 mils of an aqueous solution of the salt (1 in 100) add a few drops of chlorine water, boil out the excess of chlorine and cool, it is not colored red by potassium sulphocyanate T. S. (*Iron*).

The addition of a few drops of silver nitrate T. S. to 10 mils of an aqueous solution of the salt (1 in 100) previously mixed with a few drops of nitric acid does not at once produce more than an opalescence (*chlorides*).

An aqueous solution of the salt (1 in 50), slightly acidulated with hydrochloric acid, does not respond to the U. S. Pharmacopeia Test for Heavy Metals.

Completely precipitate the manganese from an aqueous solution of 1 Gm. of the salt by the addition of ammonium carbonate T. S. and filter; the filtrate evaporated and gently ignited leaves a residue weighing not more than 0.005 Gm. (limit of other *salts* or of the *alkalies*).

A solution of 1 Gm. of the salt and 1 Gm. of sodium acetate in 10 mils of distilled water, to which a few drops of acetic acid have been added, is not affected by hydrogen sulphide T. S. (*zinc*).

Assay.—Dissolve about 0.5 Gm. of Manganese Sulphate, accurately weighed, in 100 mils of distilled water, add 10 mils of ammonia water and 30 mils of solution of hydrogen dioxide, and boil the mixture until the precipitation is complete. Transfer to a filter and wash the precipitate with distilled water until the filtrate ceases to give a precipitate with barium chloride T. S., then dry the filter and precipitate, and incinerate these, first at a low heat and then at a strong red heat, until the weight is constant. The residue of mangano-manganic oxide (Mn_2O_3) so obtained corresponds to not less than 31.3 percent nor more than 34.3 percent of the weight of the salt taken.

Each gramme of mangano-manganic oxide is equivalent to 1.979 Gm. of $MnSO_4$.

POTASSII SULPHAS

Potassium Sulphate.
Pot. Sulph.

It contains when dried to constant weight at 100° C. not less than 99 percent of ($K_2SO_4 = 174.27$).

Potassium Sulphate occurs as hard, colorless, translucent, six-sided, rhombic prisms terminated by pyramids, or a white powder; permanent in the air. Odorless; taste saline, somewhat bitter.

1 Gm. dissolves in 10 mils of water at 25° C., and in 5 mils of boiling water; insoluble in alcohol.

When heated the crystals decrepitate. At a bright red heat the salt fuses, and at a white heat partial decomposition occurs.

The aqueous solution of the salt is neutral to litmus.

A saturated aqueous solution of the salt yields a white, crystalline precipitate with excess of sodium bitartrate T. S.

An aqueous solution of the salt (1 in 20) yields with barium chloride T. S. a heavy white precipitate, insoluble in hydrochloric acid.

An aqueous solution of Potassium Sulphate (1 in 50), slightly acidulated with hydrochloric acid, does not respond to the U. S. Pharmacopœia Test for Heavy Metals.

Five mils of an aqueous solution of the salt (1 in 25) meets the requirement of the U. S. Pharmacopœia Test for Arsenic.

Assay.—Dissolve about 0.6 Gm. of Potassium Sulphate dried to constant weight at 100° C., accurately weighed, in 100 mils of distilled water, acidulate the solution with hydrochloric acid and heat it to boiling. Gradually add an excess of barium chloride T. S., and heat for thirty minutes on a water-

bath, collect the precipitate of barium sulphate on a filter, wash it with distilled water until silver nitrate T. S. fails to produce an opalescence in 2 mils of the filtrate previously acidulated with nitric acid. Dry the precipitate, ignite and weigh the residue. It corresponds to not less than 99 percent of K_2SO_4 .

Each gramme of Potassium Sulphate corresponds to not less than 1.328 Gm. of $BaSO_4$.

SCOPARIUS

Scoparius

Scopar. Broom Top

The dried top of *Cytisus Scoparius* (L) Link (Fam. *Leguminosæ*).

Thin, flexible, branched twigs, 2 to 3 mm. thick, with five angles; externally dark green, nearly glabrous, with numerous reddish-brown cork patches; internally yellowish; fracture short-fibrous, that of thicker pieces tough and splintery; upper leaves, sessile, often only 1-foliolate, obovate, present in small amount or absent.

Odor slight, on bruising more distinct and peculiar; taste disagreeable, bitter.

Scoparius yields not more than 5 percent of ash.

HARRISON LAW DECISION RELATING TO FILLING OF PRESCRIPTIONS ENDORSED

The officials of the Treasury Department, after further consideration of the controverted questions regarding Treasury decisions 2213 and 2244, involving details of the Harrison Anti-narcotic Law, advise that the Internal Revenue Bureau has received communications from high physicians and medical organizations in the country emphatically indorsing the position taken by the Internal Revenue office and the Treasury Department in the matter of refilling prescriptions, which was denied in Treasury decision 2213.

It is stated that these communications from medical men and organizations take the position that the authorizing of the refilling of prescriptions containing *even minimum* quantities of narcotic drugs would do more to en-

courage the drug habit than any other action that might be taken under the law. They protest, also, against the continuation of the exemption in section 6 of the law, and say that, next to permitting the refilling of prescriptions, the indiscriminate sale of medicinal compounds containing habit-forming quantities of these drugs is a weak feature of the law and will be helpful in encouraging the forming of drug habits among the people.

The department officials decline to make public the names of the organizations and physicians who take this position, stating that the communications were addressed to the department without any intimation that a public discussion of the matter is desired.