

as the burdock, little real damage was noted—it takes a good deal to damage a burdock—but *Carthamus*, for instance, was distorted almost beyond recognition, and quite failed to produce well-formed flowers. This species, by the way, was purchased from a seedsman as “Saffron,” but the akenes which fell out of the opened envelope were a sufficient demonstration of the substitution.

While on the subject of Composites it may here be noted that *Grindelia squarrosa*, of a genus usually listed as perennials, appears here to be a biennial only.

The pathology of drug plants, for the most part little studied, is full of absorbing problems, and our limited area and limited experience have presented several. Our first-year henbane, surviving heroically the onslaughts of the insects, succumbed ignominiously to what appeared to be a mosaic disease. The extensive studies which have been elsewhere made on the solanaceous mosaics have not, so far as we have noted, reported it on this plant, and correspondence with the Bureau of Plant Industry has not revealed previous record of it. Tobacco growing near-by showed mosaic, and it is not impossible that the infective agent may have been transferred by aphids, some species of which have proved capable of transmitting similar diseases. A second-year planting of henbane on the same spot, for the purpose of studying this disease further, was quite exterminated by insects. Mosaic, however, appeared on *Daturas* in another plot, and caused considerable injury. If the disease appeared in the previous plantings of these species, it escaped our notice. Investigations of the alkaloidal characteristics of infected leaves have proved of considerable interest, and will be reported in another paper.

From a monetary point of view, of course, such a plantation as ours cannot maintain itself, but, from that point of view, neither can a university! It may be said, however, that many of our small-scale products have found a use in the assaying and dispensing laboratories of the school, in our hospital practice, and are serving as a basis for several more purely scientific studies.

SOLUBILITY OF VOLATILE OILS IN AQUEOUS MEDIA.

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The experimental data recorded in this publication were obtained from investigations concerning the solubility of certain volatile oils in aqueous solution, with particular reference to the medicated waters of the Pharmacopœia.

The official type process for the preparation of aromatic waters¹ consists essentially of the three following consecutive operations briefly enumerated:

- (1) Trituration of the oil with talcum.
- (2) Addition of water with continued trituration.
- (3) Filtration.

Digression from the official method relative to the empirical selection of the requisite absorptive media, is provisionally conceded upon conformity of the finished preparation with the authoritative requirements.

The descent of a solvent through the oil-impregnated absorptive during filtration, presents the fundamental aspects commonly involved in the extraction (washing) of a partially soluble precipitate. The amount of oil remaining in the

¹ United States Pharmacopœia, Rev. IX, page 60.

talcum, proceeding the passage of the solvent, is totally dependent upon the number and volume of the aliquot portions of menstruum applied.

The vertical pressure, in funnel filtration through conoidal paper, varies at every instant, and the area decreases three times as fast as the rate of diminution in the volume of the liquid contents. For example, when $\frac{1}{2}$ of the liquid contents has been filtered through, the filtering area has decreased to $\frac{1}{6}$ of the original exposed area.

The quantity of oil removed with each unit volume of filtrate is fairly constant, providing the temperature is consistent with the volatility of the components ordinarily involved.

The infinitely small amounts of essential oils in solution in the finished product considered in conjunction with their characteristic degree of volatility, cannot be determined by the usual methods of physical quantitative procedure.

Quantitative data were subsequently obtained by resorting to the usual volumetric chemical methods, which procedure was conducive to concordant results.

EXPERIMENTAL.

The average specific gravity of the official aromatic waters was determined as 1.009—subject to uniform laboratory conditions. The insignificant quantity of essential oil or its water-soluble constituents is not totally responsible for the elevation of the gravity factor. The specific gravity varies primarily commensurate with the purity of the talc employed.

The presence of considerable suspended matter derived from purified talc was proved by microscopic examination of residues obtained by evaporation of small quantities of the waters immediately proceeding filtration. The residual impurities in U. S. P. IX Talc can be further eliminated to a greater degree by a third or fourth repetition of the general method of purification.¹

A saturated aqueous solution of oil of gaultheria was ultimately adopted as a criterion for future investigations along these lines. This was deemed advisable, in consideration of the distinct characteristic color reactions readily obtainable with the water-soluble constituent of the original oil.

Of the numerous volatile oils examined, gaultheria was found to possess the maximum volume of water-soluble matters, consisting of, from 85 to 99 per cent. of methyl salicylate $C_6H_4(OH).COOCH_3[1:3]$. A solution of ferric chloride produces a rich bluish violet coloration with water previously agitated with the oil.

The reaction affords ample facilities for the direct colorimetric quantitative method which was subsequently discarded in preference to the more adequate volumetric procedure.

The official assay process for gaultheria is based upon the determination of the intensity of the acid reaction by neutralization to phenolphthalein with alcoholic V. S. potassium hydroxide. A saturated aqueous solution of the oil was prepared according to the official type process, using a highly purified form of talcum. The methyl salicylate contents of the oil were previously determined by the U. S. P. IX Assay² to be 99 per cent. The filtrate was repeatedly percolated

¹ Remington, "Practice of Pharmacy," 1892, page 1171.

² United States Pharmacopœia, Rev. IX, page 272.

through the oil-talc mixture upon a strong porous filter medium to insure maximum saturation. The specific gravity of the filtrate was found to be 1.0065.

A $\frac{25}{100}$ -cc portion of a representative specimen of the product was removed to an Erlenmeyer flask of $\frac{240}{100}$ -cc capacity and $\frac{20}{100}$ cc of *N*/10 V. S. alcoholic potassium hydroxide added. Phenolphthalein T. S. was then added and the contents of the flask were thoroughly agitated, and then titrated with *N*/10 V. S. hydrochloric acid. Each cc of *N*/10 alkali consumed, corresponds to 0.015206 gram of methyl salicylate.

The filtrate was found to contain 0.060824 per cent. of methyl salicylate in aqueous solution, corresponding to 0.0001980 per cent. of gaultheria.

RESORCINOL AND PHLOROGLUCINOL AS COLOR REAGENTS.

BY E. V. LYNN AND F. A. LEE.

The polyhydric phenols have long been used in numerous reactions as agents for qualitative detection of a variety of compounds. The identification of methyl alcohol through its oxidation product, formaldehyde, the detection of sucrose in various food products, and the use of Seliwanoff's reagent in physiological chemistry might be cited as illustrations. We have been unable, however, to find any reference in the literature to a systematic record of comparative results with these compounds. Michael and Ryder¹ many years ago investigated the reaction of various phenols with aldehydes, but neglected to report the specific appearance in each case. During an extensive search for some means of identifying small quantities of cinnamic aldehyde in volatile oils, we had occasion to try the effect of an acid solution of resorcinol. The reaction was so striking that we were induced to investigate it further and, after quite a long series of tests, are much impressed with the possibilities of using phloroglucinol and resorcinol as agents for the detection and identification of a wide variety of substances.

The reagents which we finally selected as most satisfactory are one per cent. solutions of the phenols in concentrated hydrochloric acid. Most of the compounds tried were nearly insoluble in water so that alcoholic solution of the reagent might have seemed more promising. Nevertheless, we have found that the reactions are so sensitive that saturated aqueous solutions of the compound in question give just as reliable results. Both of these phenols of course absorb oxygen from the air and in time are rendered inert, hence the reagents used should be freshly prepared. We have found, however, that they will keep well if ordinary caution is used in protection. In order to make the tests equal volumes of the substance and of the reagents were mixed and allowed to stand. Most of the substances were tested in saturated aqueous solutions but formic, acetic and butyric aldehydes were used in 1% solution and vanillin, heliotropin, chloral hydrate and salicin were about one-tenth this strength, while dimethylaminobenzaldehyde was a 0.1% alcohol-water solution. The following table gives the description of the reactions observed.

¹ *Am. Chem. Jour.*, 1887., p. 133.