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No. 5

OIL OF CLOVES

(A consideration of some recent literature on the subject)

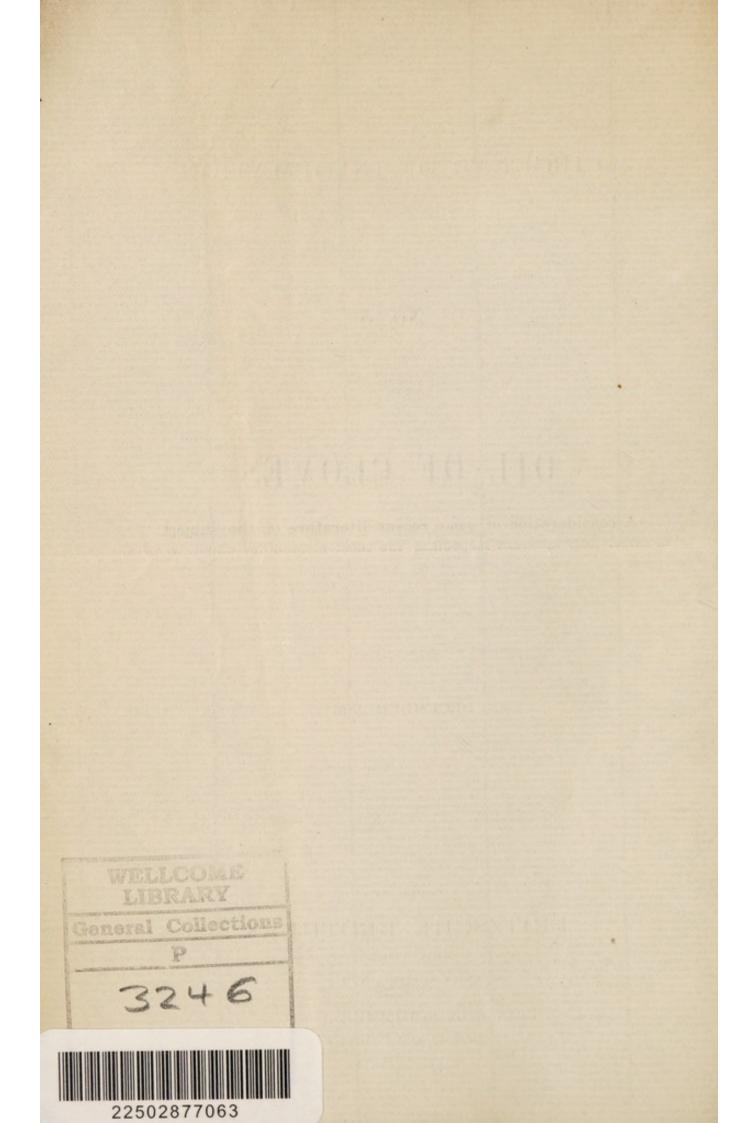
DECEMBER, 1894

ISSUED BY

FRITZSCHE BROTHERS

NEW YORK AND GARFIELD, N. J.

BRANCH OF SCHIMMEL & CO. LEIPSIC AND PRAGUE



OIL OF CLOVES.

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As large manufacturers of Oil of Cloves we feel called upon to express our views, somewhat briefly but forcibly, respecting a recent publication on this subject. Preliminary thereto, and by way of explanation, we deem it expedient to present some facts which have served as an incentive to the remarks here to follow.

At the meeting of the American Pharmaceutical Association held in Chicago in August, 1893, a paper entitled: "The value of the pharmacopocial requirements for Oil of Cloves" was read by Chas. T. P. Fennel, then Chairman of the Scientific Section of the Association. The number of glaring errors contained in that paper demonstrated not only that the writer of it knew nothing of the true character and composition of oil of cloves, but also that he was inconceivably ignorant respecting the most elementary chemical principles and manipulations. In the concluding paragraph of that paper¹ Mr. Fennel, furthermore, sought to impeach the integrity of manufacturers of oil of cloves by calling attention to an impure eugenol, obtained as a by-product in the manufacture of safrol, and by the remark: "This is in all probability obtained from the leaves (Illicium religiosum) of star anise, and used as the basis for the commercial oil of cloves."

The absurdity and incongruity of the statements made by Mr. Fennel in his first paper were such as to require no great amount of chemical knowledge for their discernment, and would seemingly at once have been apparent to any careful reader or intelligent pharmacist. Nevertheless, the article referred to not only made a quite complete circuit of the pharmaceutical trade journals, but, even after its discreditable character had been exposed, ² seems to have been regarded as one of the so-called "mooted questions," and was printed in full, without comment, in the Proceedings of the American Pharmaceutical Association for 1893, pages 135–139.

In view of the criticisms made upon the first paper of Mr. Fennel on oil of cloves, it was, however, hardly to be expected that after an interval of a year he should feel encouraged to bring to public notice,

¹ Proc. Amer. Pharm. Assoc., 1896, p. 139.

² Pharm. Rundschau, N. Y., Oct., 1893, p. 228.

and before a national association of pharmacists, the results of his further researches on this subject. In this supposition we were obviously mistaken, for at the recent meeting of the American Pharmaceutical Association, held at Asheville, N. C., Sept. 3-8, 1894, another paper on "Oil Cloves," by Chas. T. P. Fennel, was presented. This paper is more extensive than the first, and comprises about 32 octavo pages of printed matter. If we except those portions of it which have been abstracted from various text books or chemical works, and consider only the results which ostensibly have been obtained by Mr. Fennel himself, together with his deductions from them, we must express our conviction that it stands without a parallel as an exposition of ignorance and audacity. It is, indeed, hardly conceivable that a person making claim to the character of a scientist could bring together such a mass of nonsense, and still less that he should venture such a bold assertion as this: "Every one should be convinced.....that oil of cloves, as found upon the American market, is not true oil of cloves, and consequently a substitute and an adulteration."

This second production of Mr. Fennel on oil of cloves we shall not attempt to criticise in extenso, for the reason that his statements are largely of such an irrational character as to place them quite beyond the limits of criticism. We shall therefore restrict ourselves to a few abstracts, which seem to us sufficient to indicate the general character of the paper.

Mr. Fennel begins his essay with a consideration of the methods commonly employed for determining boiling points, melting points and specific gravity, and then refers to the refraction and polarization of light. In a subsequent chapter he has brought together so-called "Historical Data," which comprise a number of citations from various text books or chemical works, dispensatories, etc., respecting the character of oil of cloves. Among the works from which these citations are drawn may be noted the following antiquated ones:

Deutsches Apotheker Buch, by Döbereiner, 1842,

Lehrbuch der Chemie, by Regnault, 1851,

Lehrbuch der Pharmaceutischen Chemie, by Gottlieb, 1857, and some others of more recent date.

Mr. Fennel then continues by bringing into review the previously considered subject matter, and his deductions therefrom are expressed in the following classical paragraphs:

"Epitomizing the facts revealed by the light of history we find statements which apparently are not in perfect accord with each other, and yet a careful analysis of these facts, the direct results of experimentation and observation, will indicate perfect harmony. The history clearly demonstrates that half a century ago our knowledge of oil of cloves was practically as complete as to-day.¹ The evidence further shows that the

¹ If Mr. Fennel intends this statement to apply simply to his own knowledge of the subject it is perfectly correct, but not otherwise. (Compare, for example, Liebig's Annalen, 1892, Bd. 271, pp. 285-299, and subsequently 1894, Bd. 279, pp. 391-393.)

results obtained were sought for the sake of truth and for the benefit of pharmacy by direct experimentation, and not, as to-day, at the expense of science, misapply-ing the knowledge gained by direct experimentation to supplant the products of nature. That oil of cloves should vary in color and fluidity is not remarkable, and but the result of the laws of nature. Age, the time during which the natural forces effect visible changes, constitutes the prime factor. The same causes have existed in the past as in the present, and experience and observation have shown the effects to be the same under all like conditions, a gradual evolution in condition until well defined limits are reached. The effects produced are made appreciable to our senses in the evolution of art and science by arbitrary standards and methods. The results obtained thereby must consequently vary upon such matters in state of change, between the limits of this matter in statu nascendi and the resultant of the two factors, heat and light. It is, therefore, not strange that the specific gravity of oil of cloves should vary between the limits 1.034 and 1.070; like-wise it is not remarkable that the boiling point should vary between the limits designated by 240°-255°." results obtained were sought for the sake of truth and for the benefit of pharmacy

"Truth sought for the sake of truth, and yet how profoundly ignorant were these experimenters in the eyes of our scientist.¹ All early experimenters agree that oil of cloves is a mixture of two components; a light oil, isomeric with turpen-tine oil C_{10} H₁₆, specific gravity 0.91 to 0.918, and boiling between 251°-254° C.?; and a heavy oil, an oxygenated oil, designated by various terms, acid oil, clove acid, eugenic acid and eugenol, specific gravity between 1.068 and 1.079, and boiling between 242°-252° C."

Mr. Fennel subsequently remarks as follows :

"In view of all that has been written upon the subject of oil of cloves, the writer was induced to again submit samples of oil to examination that were bought in the open market from reliable and reputable firms. Each sample was submitted to pharmacopeial requirements and were not found wanting suffici-ently, in the opinion of the writer, to warrant a verdict discriminating against them. The writer's profound ignorance accounts for this lack of judgment, not-withstanding that the voluminous array of data were at his disposal. To insure accuracy, and to obtain comparable results, the writer was compelled to prepare a sample of oil of authentic origin and purity."

It is also stated that "this sample is found among the following list of samples tested, and constitutes the basis of comparison for the other oils." Strangely enough we do not find this oil among the list of samples tested by Mr. Fennel, nor is there any further reference to it whatsoever by which it may be recognized.

For the entertainment of those who are not particularly interested in the more serious consideration of this subject, we cannot refrain from presenting at least one example, among several similar ones, of the astonishing and totally incomprehensible results obtained by Mr. Fennel in his pretended examination of commercial oil of cloves. The following paragraphs are therefore quoted verbatim.

"Sample No. 2. Sp. gr. 1.046 $\frac{15^{\circ} \text{ C.}}{15^{\circ} \text{ C.}}$ Normal pressure. Commenced to boil at 80°C., rising rapidly to 95°C., accompanied with crackling noise and heavy bump-ing to 98° C.; remaining stationary, then slowly to 105° C., collecting 0.5 per cent., temperature rising rapidly to 245° C., collecting 42 per cent., gradual rise to 252° C., collecting 40 per cent.

1st distillate, 0.5 per cent. 2d distillate, 42 per cent., sp. gr. 1.035 $\frac{15^{\circ} \text{ C.}}{15^{\circ} \text{ C.}}$

p. 228.)

3d distillate, 40 per cent., sp. gr. 1.045 $\frac{15^{\circ} \text{ C.}}{15^{\circ} \text{ C.}}$ Residue dark brown." "Sample No. 2. Sp. grav. 1.046 $\frac{15^{\circ} \text{ C.}}{15^{\circ} \text{ C.}}$ Normal pressure. Treated with an equal volume of concentrated solution of potassium hydroxide, diluted with another volume of water and distilled. Temperature rose gradually to 85° C. liquid boiling, gradually to 98° C., slowly to 103° C., collecting 10 per cent.; when oily portion of distillate indicated a specific gravity heavier than water, distillation

1 This evidently refers to the critic of Mr. Fennel's former paper, who, however, made no allusion to the earlier experimenters, but simply denounced the falsity of Mr. Fennel's statements. (Compare Pharm. Rundschau, Oct., 1893, interrupted, the distillate set aside, oil portion separated by separatory funnel, collecting 80 per cent. = 8 per cent. of the original, sp. gr. 0.98 $\frac{15^{\circ} \text{ C.}}{15^{\circ} \text{ C.}}$ These 8 per cent. were submitted to distillation, collecting

50 per cent. at 103° C., sp. gr. 0.885 <u>15° C.</u> equal to 4 per cent. of the original.

Original residue neutralized with hydrochloric acid, washed with water, using separatory funnel, finally dried with bibulous paper, and submitted to distillation. Temperature rose rapidly to 106° C., brisk ebullition remaining continuous and constant, without increase in temp., liquid becoming darker but no distillate, finally resulting in the breaking of the flask."¹

The results of a number of distillations, conducted in a manner similar to the above, are then tabulated, and the grand climax finds expression in the following, to us unintelligible, paragraph:

"Generally the quantity of mass is limited, and yet this factor of quantity of mass is extremely important in analysis and synthesis. The consideration of this factor is not new and comes under the head of Physical Chemistry ; but so far but little has been accomplished in the generalization of the relationship of chemical phenomena. Notwithstanding all the difficulties that may be encountered and the objections that may be raised in this paper, every one should be convinced, on a review of the results obtained by analysis under alike conditions, temperature, pressure, force, tension, etc., supplemented by the data furnished by the literature on the subject, that oil of cloves, as found upon the American market, is not true oil of cloves, and consequently a substitute and an adulteration."

We do not wish to concern ourselves with the nonsense contained in the rhetorical effort of this final paragraph further than to declare that we cannot allow the dense ignorance displayed by Mr. Fennel to serve as a support for such a malicious and libelous statement that "oil of cloves, as found upon the American market, is not true oil of cloves, and consequently a substitute and an adulteration."

Although the reputation of all American manufacturers of oil of cloves is alike traduced by the bold charges made in the published statements of Mr. Fennel, we have decided to take independent action in exposing their falsity. This would hardly seem necessary were it not for the lamentable fact that such papers are permitted to be discussed at the meetings of the Association before which they are read, and that, in the case of the previous papers of a like character by Mr. Fennel, they were actually printed in the Proceedings of the Association after their discreditable character had been exposed, thus receiving to a certain extent an official indorsement.

With a desire to give Mr. Fennel an opportunity to express himself a little more definitely on this subject, we directed to him the following inquiries:

I. Were you aware at the time of writing your paper that we are large manufacturers of oil of cloves, and that we supply a large portion of the oil found on the American market?

II. Have you ever examined a sample of oil of cloves which you knew to be a product of our house, and do any of the samples of oil of

¹ It is unfortunate that the flask broke at this juncture, for we should like to have been informed regarding the further behavior of this remarkable liquid.

cloves examined and referred to in your recent paper presented to the American Pharmaceutical Association represent an oil of our manufacture? If so, will you kindly inform us by what number it is there designated?

To these inquiries we received from Mr. Fennel the following reply: "The paper in question was written after a thorough investigation of the subject, and while it may inure against the reputation of some, you must also recollect that others likewise have reputation at stake. I presume, from the interest you manifest in this matter, that you are manufacturers of essential oils, and therefore sincerely in vite broad scientific criticisms to a paper written on a broad scientific basis." We received, however, no answer to our questions.

Although the oil of cloves produced by us always represents an article of the highest attainable quality and absolute purity, and, being in every respect a perfectly normal distillate, is not at all in need of any defense, we have thought it of interest to place the results of our examination of this product, which is regularly supplied in large quantities to the American market, in comparison with the characters attributed to commercial oil of cloves in the papers published by Mr. Fennel.

A sample of oil taken from our stock, without any special selection or preference, we find to have the following characters:

Specific gravity 1.067 at 15° C.

On subjecting 100 grammes of the oil to distillation in a fractionating flask there was obtained:

I.	From	100°	to 250.5	° C. (corr.)	1.9 g	rammes.
II.	"	250.5°	to 264°	C. (corr.)	92.4	"
III.	Residu	ue in f	lask		5.6	"

99.9 grammes.

I. The first fraction contained some water, which is always present in small amount in such products, together with a few drops of oil carried over by the aqueous vapor.

II. The second fraction includes the essential constituents of the oil, namely, eugenol and sesquiterpene (caryophyllene) $C_{15} H_{24}$. (The corrected boiling points of these fractions denote that the mercurial column of the thermometer was entirely in the vapor, and they are therefore slightly higher than would be obtained under other variable conditions of distillation.)

III. The residue in the flask consists in part of decomposition products of the oil, which are unavoidably formed at the high temperature by distillation with direct heat, and partly also of the liquid formed by the condensation of the vapor remaining in the flask.

The original oil forms a perfectly clear solution with twice its volume of a mixture of 2 volumes of official alcohol and 1 volume of water. The U. S. Pharmacopoeia requires for Oil of Cloves a specific gravity of 1.060 to 1.067; the German Pharmacopoeia a specific gravity of at least 1.06. Mr. Fennel seems to have found in the specimens he examined a spec. gravity ranging from 1.037 to 1.0567.

The U. S. Pharmacopœia makes no requirement as to the boiling point, but the German Pharmacopœia requires that the oil shall come to full ebullition at 247° C., which evidently refers to an uncorrected boiling point, such as is frequently stated when special accuracy is not required. Mr. Fennel, in his publication, has recorded almost every possible and impossible variation of boiling point from 60° to 276° C.

Having now presented a sufficient number of facts to demonstrate the falsity of Mr. Fennel's statements and of the conclusions based upon them, so far at least as they may be construed to refer to the oil of cloves of our manufacture on the American market, we may make a few final comments.

It is our conviction that no such results as have been recorded by Mr. Fennel can be obtained with any oil of cloves produced by a reputable manufacturer, and that they were either brought about by an almost inconceivable ignorance of the simplest chemical and physical processes or something worse. We furthermore indignantly repudiate the unqualified assertion that the oil of cloves of the American market is "a substitute and an adulteration," and challenge Mr. Fennel to produce any confirmation of this statement by any reputable and properly qualified chemist. We also beg to state that, having considered it our duty to present the character of his recent publications on this subject in their true light, we shall not deem it necessary to take any notice of similar articles which he may cause to be published in the future. We may, however, express the hope that the spirit of true scientific inquiry and research shall not continue to be defamed and perverted by such shallow pretensions.

II. The second fraction in fudes the classiful constituents of the

FRITZSCHE BROTHERS.