chloride-iodine titration method without preliminary oxidation.

The iron content is expressed in percentage by weight based upon the original weight of each portion of the mass. The dates of each series of assays are indicated.

SUMMARY AND CONCLUSIONS

1. All of the masses except the one made with sucrose retained good color and plasticity throughout the several months of study. The freshly prepared sucrose mass had a fine appearance. By the end of the second week it had developed a reddish brown color. After nine months the mass was hard and much oxidized at the surface. Internally the color was grayish green with brown spots throughout. The assays indicated that the ferrous iron had almost completely oxidized to the ferric iron.

2. The masses made with maltose and rhamnose showed some crystallization at the surface after several months.

3. Lucas and Stevens (1903) and Greenish (1904) observed that reducing sugars were capable of changing the ferric iron in Blaud's Pills to the ferrous state. Our observations seem to bear out this fact.

4. The variations between the ferrous and ferric iron content are consistent in most cases, as is indicated by the results for total iron except for galactose.

5. There is need for further investigation into the role played by reducing sugars in preventing the oxidation of ferrous iron in Blaud's Pills.

6. The ceric ammonium sulfate assay is, without doubt, more accurate than the dichromate procedure.

7. There seems to be no difference between *ortho*-phenanthroline-ferrous complex ion and phenylanthranilic acid as internal indicators for use with ceric ammonium sulfate.

8. Xylene cyanole F.F. was not wholly reliable as an internal indicator when used with ceric ammonium sulfate. In some cases the color change at the end-point failed to develop. This gave erratic results.

9. Potassium dichromate with diphenylamine T.S. as an internal indicator invariably gave high results on the estimation of ferrous iron.

REFERENCES

(1) Furman, N. H., *J. Am. Chem. Soc.*, 50 (1928), 755.

(2) Willard, H. H., and Young, P., *Ibid.*, 50 (1928), 1322.

(3) Ferrey, G. J. W., *Quart. J. Pharm. Pharmacol.*, 10 (1937), 351.

(4) Watt, L. A., *National Formulary Bull.* (Jan. 5, 1937), 2506.

(5) Lyons, C. G., and Appleyard, F. N., *Quart. J. Pharm. Pharmacol.*, 10 (1937), 343.

(6) Sutton, F., "Volumetric Analysis" (1935).

(7) Kolthoff, I. M., and Furman, N. H., "Volumetric Analysis," Vol. II (1929).

(8) Lucas, E. W., and Stevens, H. B., *Pharm. J.*, 71 (1903), 400.

(9) Greenish, H. G., *Ibid.*, 73 (1904), 734.

Phytosterol from the Buds and Fruit of the Tung Tree (*Aleurites fordii Hemsl.*)*

By Harold M. Sell and Albert H. Best

Work has been in progress at the U. S. Field Laboratory for Tung Investigations at Gainesville, Florida, on the physiological functions and nutrition of the tung tree at various periods of the year when major changes are taking place in the different tissues. In the course of this study, the waxlike substance found in the terminal bud was of interest. The literature (1, 2) does not report any work on the biochemical composition of the dormant tung bud. In this investigation a sterol has been isolated in a crystalline state from the bud and mature fruit and identified as phytosterol.

EXPERIMENTAL

Preparation of the Extract from Tung Buds.—In February of 1940, 7.1 lb. of buds[1] were collected from trees in the Alachua Tung Oil Orchards. The buds were cut into small pieces and extracted with petroleum ether for 24 hours. They were then ground to pass a 2-mm. mesh sieve and extracted for an additional 24 hours. Of this extracted material, 400 Gm. were saponified by refluxing with 350 ml. of 15% potassium hydroxide in 85% ethanol for 2 hours. After saponification the solution was diluted with 1200 ml. of water and extracted in a continuous

* Division of Fruit and Vegetable Crops and Diseases, Bureau of Plant Industry, United States Department of Agriculture, Gainesville, Florida.

[1] We are indebted to Mr. Rolf Buckley of the Alachua Tung Oil Company for this material.

extraction apparatus with petroleum ether. The petroleum ether extract was concentrated to dryness *in vacuo* at 50° C. The residue was taken up in 200 ml. of acetone and precipitated with 800 ml. of a 1% solution of digitonin (3) in ethanol. The sterol digitonide was collected on a Büchner funnel and washed with chloroform, ethanol and ether. The yield was 9 Gm.

Preparation of Sterol Acetate.—The sterol acetate (4) was obtained by refluxing the digitonide with 60 ml. of acetic anhydride for 2 hours. The acetate separated from the cooled solution and was recrystallized from acetic anhydride until a constant melting point was obtained. The product was dried in an Abderhalden drier for 4 hours at 100° C. and 4 mm. The final yield was 1.2 Gm. The following properties were observed:

Melting point—121.8–123° C. A mixed melting point with a known sample of phytosterol acetate gave no depression.

Rotation $[\alpha]_D^{25}$——33.3 in U. S. P. chloroform where C = 0.1 Gm. in 10 ml.

Color reactions—Positive test with Whitby's test B; Whitby and Salkowski; Liebermann and Burchard; Rosenheim and Page.

Preparation of Free Sterol.—The free sterol was obtained by refluxing 0.7 Gm. of sterol acetate with 50 ml. of a 4% solution of potassium hydroxide in 85% ethanol. The sterol crystallized from the solution upon cooling. It was recrystallized from absolute ethanol until a constant melting point was obtained. The product was dried in the Abderhalden drier for 4 hours at 100° C. and 4 mm. The yield was 0.6 Gm. of the purified sterol. The following properties were observed:

Melting point—136.7–137.7° C. A mixed melting point with known phytosterol gave no depression.

Rotation $[\alpha]_D^{25}$——32.7 in U. S. P. chloroform where C = 0.1 Gm. in 10 ml.

Color reactions—Positive test with Whitby's test B; Whitby and Salkowski; Liebermann and Burchard; Rosenheim and Page.

Preparation of Sterol Benzoate.—The sterol benzoate (5) was prepared by dissolving 0.1 Gm. of the sterol in 4 ml. of anhydrous pyridine and then adding 1 ml. of benzoyl chloride. The mixture was shaken and then warmed. The reaction product was poured into 10 ml. of water and shaken. The supernatant liquid was decanted and the residue stirred with 5 ml. of 5% sodium carbonate. The precipitate was filtered on a Büchner funnel and recrystallized from absolute ethanol until a constant melting point was obtained.

Melting point—146–147.2° C. A mixed melting point with a known sample of phytosterol benzoate gave no depression.

From these constants it is apparent that the sterol is phytosterol.

The sterol from the fruit was obtained from the non-saponifiable portion of the kernel. This sterol was found to be identical with the sterol from the buds, since its derivatives had the same constants as listed above.

SUMMARY

A sterol has been isolated from the buds and the mature fruit of the tung tree and identified as phytosterol.

REFERENCES

(1) Wehmer, C., "Die Planzenstoffe," Jena, 2nd Edition (1931).

(2) *Chemical Abstracts*, Vol. 1 (1907) to Vol. 34 (1940).

(3) Klein, G., *Handbuch der Pflanzen Analyze*, Wien, 2 (1932), 724.

(4) Lifschutz, *Zeit. Physiol. Chem.*, 101 (1918), 89–98.

(5) Shriner, R. L., and Fusion, R. C., "Identification of Organic Compounds," John Wiley and Sons, 2nd Edition (1940).

Pharmaceutical Emulsions. IV. Mixtures of Acacia and Tragacanth as Emulsifying Agents*,†

*By William J. Husa‡ and Charles H. Becker***

INTRODUCTION

In previous papers (1, 2, 3) detailed studies were made of the Continental and English methods of making emulsions; these older methods of emulsification were compared in efficiency with the use of various mechanical stirrers and a hand homogenizer for making emulsions. The present investigation is devoted to a study of acacia-tragacanth mixtures as emulsifying agents, using various methods of emulsification.

EXPERIMENTAL

Materials Used.—The oils selected for study were cod liver oil, linseed oil, castor oil and heavy mineral

* Presented before the Scientific Section, A. PH. A., Richmond meeting, 1940.

† This paper is based on part of a thesis presented to the Graduate Council of the University of Florida in partial fulfilment of the requirements for the degree of Doctor of Philosophy.

‡ Head Professor of Pharmacy, University of Florida.

** Graduate Scholar, University of Florida, 1939–1940.