Wash down into the flask with 25 cc. of hydrochloric acid and 50 cc. of water, then add 8 Gm. of zinc (C. P. 10 mesh). Shake the flask until the amalgam which adheres together has been broken up into a granular form. Add an additional 2 Gm. of zinc and heat on a steam bath, shaking frequently until the solution becomes clear and colorless. Remove about 2 cc. of the supernatant liquid and saturate it with hydrogen sulfide; it should remain colorless. If, however, a brownish tinge develops (indicating the presence of mercuric salts), continue the digestion until the solution shows no reaction with hydrogen sulfide.

When all the mercury is reduced, wash the amalgam by decantation until the washings no longer show a reaction with silver nitrate solution. In the event that fragments of the amalgam are floating on the surface of the liquid, add a few cc. of alcohol to break the surface tension.

The amalgam is now dissolved by treating with a mixture of 25 cc. concentrated nitric acid and 100 cc. of water. If the reaction becomes too violent, the flask should be cooled by immersing in cold water. When the zinc has been dissolved out of the amalgam and the reaction has moderated, add 20 cc. more of concentrated nitric acid and heat on the steam bath until all the mercury is in solution; then add 20 cc. more of concentrated nitric acid and heat for 30 minutes on a steam bath. The solution should at this time be clear and colorless.

Dilute the solution to 500 cc. in a volumetric flask and after thorough mixing, remove about 10 cc. and add 4–5 drops of dilute hydrochloric acid. The solution should remain clear; if, however, mercurous salts are present (indicated by the turbidity of the solution), a new assay must be started using more nitric acid and a more prolonged heating time to insure complete oxidation.

Titrate 100 cc. of the above solution with 0.1*N* Potassium Thiocyanate V.S. using 2 cc. of ferric ammonium sulfate T.S. as indicator.

Each cc. 0.1*N* Potassium Thiocyanate V.S. is the equivalent of:

Mercuric Chloride, $HgCl_2$.....	0.013576 Gm.
Mercuric Iodide, HgI_2.....	0.02272 Gm.
Mercury, Hg.....	0.01003 Gm.

Note: (1) If the original sample contains mercurous salts, it should be oxidized with bromine before proceeding with the assay. (2) If the original sample contains sodium bicarbonate as in certain germicidal tablets of mercuric iodide, diluted hydrochloric acid should be added until there is an excess equivalent to about 25 cc. (Using ten tablets, each containing mercuric iodide $^3/_8$ gr., potassium iodide $^3/_4$ gr. and sodium bicarbonate 14 gr.)

The method has been found useful in the assay of Ammoniated Mercury Ointment U. S. P. and in ointments containing ammoniated mercury and zinc oxide in combination. In this case place 15 Gm. of the ointment, accurately weighed, in a 200-cc. beaker, warm slightly to soften the ointment and while stirring add 50 cc. ether and stir the mixture until the ointment base is dissolved. Transfer to a separatory funnel, washing the beaker with ether and diluted hydrochloric acid (10-cc. portions) until the ointment is completely transferred. Shake the mixture vigorously until all the inorganic compounds have been dissolved. Filter the aqueous layer into a 500-cc. Kjeldahl flask and wash the remaining ethereal solution with several portions of distilled water until the last washing produces no turbidity with silver nitrate T.S. Add the zinc to the acid solution in the Kjeldahl flask and proceed as outlined in the general method.

SUMMARY

In our hands this procedure has been found to yield accurate results and is more rapid than gravimetric methods. It has been successfully used in the assay of antiseptic tablets of mercuric chloride where the presence of organic dyes may lead to erroneous results if the sulfide precipitation method is employed. Other applications are evident and will be developed as opportunity arises.

Note on Philippine Turtle Oil*

By Pura Villarica and Patrocinio Valenzuela†

"A giant leathery sea turtle, known in science as *Dermochelys schlegeli* (Garman), was recorded in Philippine waters for the second time when Lt. Col. Zerbee of the U. S. Army shot the reptile in Tayabas Bay, off the coast of Lucena on March 26, 1939. The monster was brought to Manila by the S.S. *Masbate* on which the colonel was a passenger when he sighted the reptile, which had some pilot fish, or remora, attached to its back. The dorsal shield or carapace of the animal measured 194 centimeters from tip to tip and the whole animal weighed about 300 kilograms. Leathery turtles are known to attain a length of about 320 centimeters and a weight of about 500 kilograms. The turtle is known to be dis-

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tributed between the tropics, but is scarce wherever it occurs. It is of no great commercial value, except for the oil, which is highly prized for use in cosmetics.

"The first recorded evidence of the presence of these huge reptiles in Philippine waters is a stuffed specimen exhibited in the old Ateneo de Manila. This specimen is said to have been caught in Manila Bay.

"Immediately upon arrival in the City of Manila, Lt. Col. Zerbee presented the dead turtle to Dr. Canuto Manuel of the former Division of Fish and Game Administration of the Bureau of Science, now of the Division of Fisheries of the Department of Agriculture and Commerce of the Commonwealth of the Philippines. After retaining the huge shell, Dr. Manuel offered to us the carcass for the extraction of turtle oil."[1]

As is well known in pharmacy, turtle oil has recently come into prominence in the cosmetic industry. Hence, the kind offer of Dr. Manuel has afforded us an opportunity to study the properties of Philippine turtle oil.

EXPERIMENTAL

Extraction of Oil.—To a small quantity of the fatty tissues of the turtle a little water was added, and then the mixture was heated until all available fats were extracted. A small amount of boric acid was added to the fatty oil thus obtained.

PROPERTIES OF THE OIL

The liquid fatty oil at 28° C. is lemon-yellow in color, with marked fishy odor. It is slightly soluble in cold alcohol, sparingly soluble in hot alcohol and ethyl acetate and very soluble in ether, chloroform, benzene, carbon disulfide and acetone. Other properties[2] follow:

Congealing point	6.03° C.
Refractive index (at 34° C.)	1.4664
Specific gravity (at 32° C.)	0.9222
Acid value	0.07 mg.
Iodine value	82.2
Saponification value	198.5
Unsaponifiable matter	2.46%

When examined five months later on September 23, 1939, after standing throughout at laboratory temperature in Manila, P. I., the same oil gave the following values:

Specific gravity at 30° C.	0.9143
Refractive index at 29° C.	1.4689
Acid value	0.105 mg.
Iodine value	106.2
Saponification value	227.7

COLOR TESTS

1. Color Test Given in "Turtle Oil Facts" (1).—One drop of concentrated HNO_3 was allowed to run down the side of a small evaporating dish to come in contact with a small quantity of turtle oil. It should have a brownish coloration—first a tan color and then a brown one was produced. No violet or lavender color was developed.

2. Color Test for Cod Liver Oil as Given in the U. S. P. XI, p. 261.—A solution of 1 drop of the cod liver oil in 1 cc. of $CHCl_3$ when shaken with 1 drop of conc. H_2SO_4 acquired a violet-red tint gradually changing to reddish brown. When this color test was tried for the turtle oil, 3 drops of the oil in 1 cc. of $CHCl_3$ were used instead of 1 only. A slightly violet-red tint was obtained, but it readily disappeared and turned to a slightly brown color.

TESTS FOR VITAMINS

1. Vitamin A.—Antimony trichloride test: Antimony trichloride washed with $CHCl_3$ and dried is dissolved in $CHCl_3$ to make a 30% solution (weight in volume). After standing, the clear solution is decanted and placed in a burette. The oil to be tested is dissolved in $CHCl_3$ (20%), and to 0.2 cc. delivered from a 1-cc. burette, 2 cc. of the antimony trichloride solution is added. The liquid is at once transferred to a cell and its blue color intensity is measured against standard glasses in a Lovibond tintometer.

The test was made for the turtle oil, cod liver oil containing vitamin A and rice oil also containing vitamin A.

In the last two a pale blue fluorescence was observed, which disappeared at once and developed into a purplish brown color.

The rice oil attained, as final color, a purplish red.

2. Test for Vitamin B_1[3].—A 1:10 dilution of the oil in alcohol was made and the latter tested for its vitamin B_1 content using the McCollum and Prebluda qualitative test.[4]

No pink color was developed. This test shows that the oil does not contain vitamin B_1.

[1] Data furnished by Dr. Canuto Manuel, formerly of the Division of Fisheries, now of the Division of Natural History Museum, Department of Agriculture and Commerce, Commonwealth of the Philippines.

[2] Analyzed in April 1939.

[3] The test for vitamin B_1 was made owing to various requests made in our laboratory for the detection of vitamin B_1 in some fixed oils.

[4] Private communication from Dr. E. V. McCollum. Also see McCollum, E. V., and Prebluda, H. A., *Science*, 84 (1936), 433; Manalo, G. D., Concha, J., and Valenzuela P., paper read at the U. S. P. Revision Conferences, May 13, 1940.

PREPARATION OF TURTLE OIL CREAMS

The following formulas were tried:

Formula A	
Turtle oil	50
Almond oil	27
Lanolin	15
Beeswax	8

Formula B	
Turtle oil	30
Almond oil	46
Lanolin	16
Beeswax	8

Both formulas gave creams which were pale yellow and soft, the odor of lanolin being well marked in both, notwithstanding the previous addition of perfume oil.

To prepare a cream with better consistency than that of the formulas previously prepared, the following was devised:

Turtle oil	15 Gm.
Almond oil	20 Gm.
Lanolin	8 Gm.
Beeswax	10 Gm.
Borax	0.02 Gm.
Distilled water	10 cc.

The cream obtained had, besides a better consistency, a creamy opaque appearance, although the odor of lanolin still persisted. It had a paler color.

The following formula was also tried:

Turtle oil	8 Gm.
Beeswax	20 Gm.
Paraffin wax	15 Gm.
Mineral oil	80 Gm.
Borax	2 Gm.
Distilled water	50 Gm.
Perfume oil	1.5 Gm.

The cream thus prepared had a consistency suitable for the Philippine climate. It is creamy, opaque and white.

A study of the compiled data on turtle oils especially with reference to the data given by Brown (1) shows that Philippine turtle oil compares favorably with the turtle oils obtained in other countries.

SUMMARY

A liquid fatty oil was obtained from a Philippine turtle. Its physical and chemical properties were studied. Its behavior under some color tests, under the antimony trichloride test for vitamin A and under the McCollum and Prebluda test for vitamin B_1 was observed. The results of the analysis show that the oil examined compared favorably with turtle oils obtained in other countries. Using some of the formulas given in the literature on turtle oil, creams were prepared from the Philippine sample.

REFERENCE

(1) Brown, F. W., *Drug and Cosmetic Ind.*, 32 (1933), 211.

Studies on Viburnum

IX. The Pharmacognosy and Pharmacology of Viburnum Alnifolium*

By Heber W. Youngken† and James C. Munch‡

During the summer of 1939, while engaged in field work in the southern United States, the senior author ascertained that large amounts of the bark of the Hobble-bush, *Viburnum alnifolium* Marsh., were being collected in the southern Blue Ridge district and marketed as "Southern Cramp Bark" and as "Cramp Bark." Later, it was ascertained that this practice has been going on for some time, the bark being admixed occasionally by collectors with bark offered to dealers as Black Haw Tree Bark and frequently substituted for genuine Cramp Bark or Viburnum Opulus, N. F. Since certain pieces of these barks bear a striking superficial resemblance one to the other, it is not strange that this substitute has eluded identity.

The chief purposes of this investigation were to study the physical characteristics, histology and pharmacology of the stem bark of *Viburnum alnifolium* and to develop means of distinguishing it from genuine Cramp Bark.

EXPERIMENTAL

Materials and Methods.—The materials used in the pharmacognostical portion of this investigation consisted of representative parts of plants of *Viburnum alnifolium* gathered on Mt. Mitchell, N. C., and in Aroostook Co., Maine, and of representative parts of plants of *Viburnum opulus* var. *americanum* gathered in the Arnold Arboretum, Jamaica Plain, Mass., and in Aroostook Co. and Orono, Maine, by the senior author, several samples of bark labeled

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