Anal. Calcd. for $C_{17}H_{19}O_2N$: C, 75.80; H, 7.11. Found: C, 75.77; H, 7.01.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF MICHIGAN
ANN ARBOR, MICHIGAN RECEIVED APRIL 5, 1945

Isolation of Betaine from Guayule[1]

BY C. W. MURRAY AND E. D. WALTER

In the processing of guayule, *Parthenium argentatum* Gray, for the isolation of natural rubber, a large amount of residual plant material is discarded. It is important to determine whether useful products can be found in and recovered from such residues. Also, since guayule is a satisfactory natural rubber for incorporation with synthetic rubber in building tires, knowledge of the composition of the non-rubber constituents of guayule is of great interest. It is possible that these constituents affect the quality of the rubber.

From the water soluble fraction of the leaves and from the defoliated shrub, betaine was isolated as the hydrochloride.[2]

We have isolated betaine from the acetone and benzene insoluble fraction of guayule rubber, thus proving the presence of betaine in the crude pebble-milled rubber. Betaine was also obtained from the product of each step in the extraction of the rubber, such as the dispersion prepared by cutting or comminuting the shrub in water and used for separation of rubber as latex, the solution obtained in parboiling of shrub to remove leaves, and the pebble mill slurry.

Isolation.—The dry, defoliated guayule shrub (2586 g.) was cut in a Ball and Jewell mill, a 1/4-inch screen being used. The material was placed in an 8-gallon container, and four gallons of cold distilled water was added. The mixture was mechanically stirred for about an hour and drained through a coarse filter paper. A second extraction was made with cold distilled water by allowing the mixture to stand overnight. The product was filtered as before, and the filtrate was added to the first. The combined extract was concentrated by passing steam through copper coils immersed in the liquor. After the extract was transferred to a 2-liter beaker, about 100 ml. of concentrated hydrochloric acid was added. A precipitate consisting of organic material and calcium sulfate formed was filtered off. The filtrate was further concentrated, and again precipitated inorganic salts and some humin-like material were filtered off. The residue was washed with warm methanol, the washings being added to the filtrate. The filtrate was clarified several times with Nuchar and was further concentrated until betaine hydrochloride began to crystallize. The mixture was cooled, and the betaine hydrochloride was filtered off, washed with cold absolute ethanol, and recrystallized from 95% ethanol: yield, 12 g. or about 0.4% of betaine hydrochloride.

The above experiment was repeated on 2893 g. (dry wt.) of defoliated shrub with hot instead of cold water; yield, 14 g. or about 0.5% of betaine hydrochloride.

A similar experiment with 1415 g. of dry, cut leaves yielded 13 g. or about 0.9% of betaine hydrochloride.

Optical Properties.—The betaine hydrochloride, crystallized from 95% ethanol, consisted of monoclinic prisms, tabular forms and triangular fragments. The refractive indices (determined in daylight by immersion in organic liquids) were: $n_\alpha = 1.515$, $n_\beta = 1.535$, $n_\gamma = 1.594$, all ± 0.003. In parallel polarized light (crossed nicols) many crystals did not show complete extinction on rotating the stage. These showed the emergence of a well centered optic axis interference figure by conoscopic observation in convergent polarized light (crossed nicols). The optic sign was positive. The double refraction was strong ($n_\gamma - n_\alpha = 0.079$). The extinction was usually parallel or symmetrical, and an occasional section had positive elongation with an extinction angle of about 35°: 2V = 62° 16′ calcd.; r > v weak. The optical properties of an authentic sample of synthetic betaine hydrochloride agreed in all respects with those of the material isolated from guayule. This comparison served to positively identify the isolated substance.

Anal.[3] Calcd. for $C_5H_{11}O_2N \cdot HCl$: C, 39.09; H, 7.87; N, 9.12; mol. wt., 153.6. Found: C, 39.0; H, 8.24; N, 8.94; equiv. wt. (when titrated with NaOH to phenolphthalein), 155.6.

(1) Not copyrighted.

(2) A. J. Haagen-Smit and co-workers, at the California Institute of Technology, previously stated, in a private communication, that they obtained betaine as the hydrochloride from alcohol extracts of guayule shrub.

(3) The authors are indebted to Norman L. Kaufman of this Laboratory for analytical results reported.

BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY
AGRICULTURAL RESEARCH ADMINISTRATION
GUAYULE EMERGENCY RUBBER PROJECT OF
U. S. DEPARTMENT OF AGRICULTURE
SALINAS, CALIFORNIA RECEIVED MARCH 10, 1945

Dielectric Constants of Ethylene Dichloride–Benzene Mixtures

BY ARTHUR A. VERNON, JEFFRIES WYMAN[1] AND ROBERT A. AVERY[2]

The dielectric constants of ethylene dichloride and of benzene are well known and the dielectric constants of *dilute* solutions of ethylene dichloride in benzene have been measured by Williams,[3] Gross,[4] and Muller.[5] In the course of some recent experimental work it was necessary to determine the dielectric constants of these solutions over the complete range and the results are given below.

Experimental

The benzene, C. P. thiophene-free, was stored for several days over anhydrous calcium chloride and then distilled from anhydrous aluminum oxide. The first fifth and last

TABLE I

DIELECTRIC CONSTANT OF BENZENE–ETHYLENE DICHLORIDE MIXTURES AT 25°

Mole fraction of benzene	Wave length in meters	Dielectric constant	$\frac{E-1}{E+2}$
1.000	6.800	2.274	0.29815
0.887	7.533	2.791	.3748
.789	8.215	3.318	.4359
.658	8.8825	3.879	.4897
.554	9.786	4.707	.5527
.395	10.969	5.914	.6209
.281	11.881	6.940	.6644
.143	13.115	8.455	.7131
.000	14.522	10.365	.7574

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(2) United States Public Health Service, St. Louis, Missouri.

(3) Williams, *Z. physik. chem.*, **A138**, 75 (1928).

(4) Gross, *Physik. Z.*, **32**, 567 (1931).

(5) Muller, *ibid.*, **34**, 689 (1933).