Anal. Calcd. for $C_{18}H_{34}O_3$: C, 72.43; H, 11.48. Found: C, 72.96; H, 11.87.

B. *Caprylic palmitic anhydride.* To sodium palmitate (1 mole) which had been prepared from sodium methoxide and palmitic acid was added caproyl chloride (1.03 moles) and the reaction mixture was kept at 90° for 2 hr. The reaction product upon recrystallization twice from petroleum ether (b.p. 40–60°) gave caprylic palmitic anhydride in 85% yield, m.p. 58–59°.

Anal. Calcd. for $C_{24}H_{46}O_3$: C, 75.35; H, 12.12. Found: C, 75.71; H, 12.39.

C. *Benzoic myristic anhydride.* Benzoic myristic anhydride was prepared by the procedure reported by Ralston and Reck.[12]

D. *Acetic butyric anhydride.* Acetic butyric anhydride was obtained as a by-product in the preparation of the symmetrical anhydride,[10] b.p. 155–157°.

E. *The reactions with* D-*glucosamine.* To the methanolic solution of D-glucosamine was added an equivalent amount of the mixed acid anhydride, and the reaction mixtures were placed at ice-box temperature overnight. The crystals deposited were collected and recrystallized from ethanol. The reaction of acetic palmitic anhydride yielded *N*-palmitoyl-D-glucosamine (63%), m.p. 202–203°.

Anal. Calcd. for $C_{22}H_{44}O_6N$: C, 63.28; H, 10.38; N, 3.55. Found: C, 63.14; H, 10.42; N, 3.16.

The reaction of caprylic palmitic anhydride with D-glucosamine gave rise to *N*-palmitoyl-D-glucosamine in 79% yield, m.p. 201–202°.

Anal. Calcd. for $C_{22}H_{44}O_6N$: C, 63.28; H, 10.38; N, 3.55. Found: C, 63.24; H, 10.24; N, 3.40.

Benzoic myristic anhydride with D-glucosamine gave *N*-myristoyl-D-glucosamine in 86% yield, m.p. 208–209°, $[\alpha]_D^{16}$ +62° (*c* 1, water).

Anal. Calcd. for $C_{10}H_{19}O_6N$: C, 48.18; H, 7.68; N, 5.61. Found: C, 47.87; H, 7.60; N, 5.60.

Acetic butyric anhydride with D-glucosamine gave *N*-butyroyl-D-glucosamine in yields over 50%, m.p. 208–209°, $[\alpha]_D^{16}$ +62° (*c* 1, water).

Anal. Calcd. for $C_{10}H_{19}O_6N$: C, 48.18; H, 7.68; N, 5.61. Found: C, 47.87; H, 7.60; N, 5.60.

The reaction of benzoic phthalylglycine anhydride with D-*glucosamine.* Benzoic phthalylglycine anhydride was prepared by the procedure reported by Wieland, Kern, and Sehring.[14]

Benzoic phthalylglycine anhydride with D-glucosamine gave rise to *N*-(phthalylglycyl)-D-glucosamine in 65% yield, m.p. 218–219°, $[\alpha]_D^{18}$ +48° (*c* 1, water).

Anal. Calcd. for $C_{16}H_{18}O_8N_2 \cdot H_2O$: C, 50.00; H, 5.52; N, 7.29. Found: C, 49.83; H, 5.21; N, 7.36.

The water of crystallization was lost on drying for 1 hr. at 100° *in vacuo*.

Anal. Calcd. for $C_{16}H_{18}O_8N_2$: C, 52.46; H, 4.95; N, 7.65. Found: C, 51.91; H, 4.58; N, 7.83.

Refluxing phthalylglycine and acetic anhydride produced unstable acetyl phthalylglycine anhydride, which on reaction with D-glucosamine yielded *N*-(phthalylglycyl)-D-glucosamine.

*Tetra-O-acetyl-N-(phthalylglycyl)-*D-*glucosamine.* *N*-(Phthalylglycyl)-D-glucosamine (5 g.) was acetylated with the mixture of acetic anhydride (20 ml.) and pyridine (20 ml.). The acetylation product was treated in the usual manner. Recrystallization was effected from ethanol, yield, 4.0 g., m.p. 202–203°, $[\alpha]_D^{16}$ +29° (*c* 1, $CHCl_3$).

Anal. Calcd. for $C_{24}H_{26}O_{12}N_2$: C, 53.93; H, 4.90; N, 5.24. Found: C, 54.26; H, 5.19; N, 5.53.

BIOCHEMICAL LABORATORY
COLLEGE OF AGRICULTURE
KYOTO UNIVERSITY
KYOTO, JAPAN

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Constituents of the Saguaro (*Carnegiea gigantea*). I. Proximate Analysis of the Woody Tissues

JAMES W. BERRY, ANMIN HO, AND CORNELIUS STEELINK

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This report describes the results of a proximate chemical analysis of the "woody" portions of the giant Saguaro cactus, *Carnegiea gigantea, Br. and R.* This initial investigation is part of a larger study of the Saguaro, which has been undertaken in this laboratory to establish its relationship to other xerophytic plants with respect to identity and mode of formation of polysaccharides, lignin, suberin, and extractives.

The Saguaro, largest of the United States cacti, is endemic to the Sonoran desert. It thrives under conditions of high temperature, low rainfall, and loose rocky soil. Individual plants may attain a height of fifty feet, a weight of six tons and an age of 200 years. The ability of the cactus to accumulate and retain water enables it to flower and bloom during periods of prolonged drought. The cortex of the plant is unusually large, permitting a variety of studies not possible with the smaller cortex of other dicotyledons.

A unique feature of the plant is the inner framework of ligniferous ribs, the secondary xylem, which is its main structural member. The chemical composition of this woody rib material as related to the composition of typical heartwoods is of interest to organic chemists and taxonomists alike. Another interesting feature of this plant is its response to injury or bacterial infection following injury.[1] A hard callus tissue is formed in concentric layers around the injured part. This callus tisse may extend deep into the pulpy cortex, sometimes more than six inches and, like the ribs, it is highly ligniferous. The mechanism of callus formation, as well as the formation of related wound tissues and the mechanism of abscission, have been studied in other plants from an anatomical, physiological, and histochemical viewpoint.[3,4] Bonner[6,7] and

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TABLE I[a]

SAGUARO TISSUE ANALYSIS

Tissue	Extractives[b] Pet. Ether	Ethyl Ether	Benzene	Ethanol	Cold H_2O	Hot H_2O	1% NaOH	Steam Volatile	Lignin[c]	Holocellulose	Percent of Holocellulose Alpha	Beta	Gamma
Saguaro rib	0.16	0.11	0.05	2.85	3.41	1.68	11.7	0.08	21.90	68.56	49.35	14.36	36.31
Saguaro callus	3.16	2.20	1.02	4.24	2.92	1.46	13.5	0.08	30.40	53.30	52.95	15.00	32.00

TABLE II[d]

TYPICAL HEARTWOOD COMPOSITION

Hardwoods	0.5–2.0	1.8–4.0	1.5–7.0	14–21	19–24	71–78	62–73	
Conifers	0.4–5.6	1.4–8.0	0.4–5	9–15	25–29	60–74	66–75	
Conifer bark	4–34	1–33	5–41	20–44	27–55			

[a] All results in % of oven-dry (105°) unextracted samples. [b] Successive extractions with petroleum, ether, ether, benzene, 95% ethanol, cold H_2O, hot H_2O, and 1% NaOH. [c] TAPPI Standard T-13m Method. [d] These values represent a range from minimum to maximum percentages of ten species (see Ref. 2a).

others have investigated the effect of plant growth hormones on the rate of formation of these tissues. However, little is known concerning the chemical composition of these pathological excrescences or their co-occurring chemical precursors.

The Saguaro appeared to be ideally suited for a study of callus formation, lignification, and suberization because of its large cortex, its easy mechanical separation into distinct tissues, and its availability.

This preliminary investigation was concerned with a proximate chemical analysis of the ribs and callus tissue (the two ligniferous parts of the plant). In order to compare adequately these two tissues with one another and with other plants, we have determined the concentrations of lignin, polysaccharides, and extractives. Standard methods of heartwood analysis were used. The data obtained from Saguaro tissue are listed in Table I. To emphasize the relation of cactus wood to economic woods, data showing the composition ranges of some typical heartwoods are presented in Table II.

The above results may be summarized as follows:

1. The chemical composition of the ribs is quite similar to a number of representative hardwoods.

2. The extractive content of the callus tissue (particularly that portion which is soluble in organic solvents) is significantly greater than that for the rib tissue.

3. The lignin content of the callus tissue is approximately 50% greater than that for the rib tissue, whereas the holocellulose content is considerably lower.

The last two results are not entirely unexpected, since it is known[3,4] that the formation of plant callus tissue is accompanied by a large increase in cell-wall lignin, suberin, resinous substances, and compounds soluble in organic solvents.

Although most callus tissue is found deeply imbedded in the cortex of the Saguaro, no detectable concentration of lignin is present in the uninjured portion of the cortex. A clearer understanding of the mechanism of callus formation must await the determination of the nature of the callus lignin and the identities of some of the co-occurring extractives in the adjacent tissues. Work is now in progress in this laboratory to elucidate the structures of some of these constituents.

EXPERIMENTAL

Sampling. A 20-foot plant, which had been recently felled by wind, was cut 10 feet above the base. A 1-foot section was removed, the pulp stripped away from the ribs, and the latter dried at room temperature and finally ground in a Wiley mill to pass 40–60 mesh screen.

Lignin determination. Lignin in the callus and ribs was determined by the TAPPI Standard T-13m methods, as outlined by Browning (see Ref. 2b, p. 1218).

Holocellulose determination. The method of Wise[5] was used.

Alpha-, beta-, and gamma-cellulose determination. Alpha-cellulose content in the isolated holocellulose was determined by the TAPPI Standard T203m Method (Ref. 2b, p. 1240). Beta and gamma-cellulose were determined by the volumetric method outlined in Ref. 2b, p. 1242.

Extractives. A 30-g. sample of air-dried material, ground to pass a 40–60 mesh screen, was placed in an Soxhlet extractor and extracted continuously for 12 hr. with 500 ml. of appropriate solvent. Successive extractions were performed with petroleum ether (b.p. 60–75°), diethyl ether, benzene, and ethanol (95%). The residual meal was then triturated with cold water for 14 hr., after which it was extracted with boiling water in the Soxhlet apparatus. A final extraction was made with cold 1% sodium hydroxide. All fractions were evaporated to dryness (with the exception of the sodium hydroxide fraction which was neutralized and filtered) and the residues dried at 105°, after which they were weighed.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF ARIZONA
TUCSON, ARIZ.