## EXAMINATION OF INTSIA (AFZELIA) BIJUGA BARK\*

## By W. Korytnyk†

Intsia bijuga (Leguminosae) is a commercial timber also known as Cairns teak in Queensland. It also occurs in New Guinea and is probably identical with the commercial timber kwila. Its sawdust and bark have been found to be devoid of alkaloids (Dr. H. J. Rodda, personal communication). The bark has now been examined for neutral and acid constituents and was found to contain 0.37 per cent. of  $\beta$ -amyrin and 0.10 per cent. of a saturated fatty acid material having the properties of the "cerotic" acid mentioned in Beilstein.

## $_{L}Experimental$

Analyses were carried out by the C.S.I.R.O. Microanalytical Laboratory.

 $4\cdot 5$  kg of ground bark (collected at Clump Point, Queensland, during 1951) was percolated with cold light petroleum (b.p. 40 °C) and yielded an oily green residue A. Subsequent percolation with cold methanol gave a dark brown residue B.

Residue A was shaken with light petroleum (b.p. 40 °C) and filtered. The solid residue was dissolved in benzene and adsorbed on an alumina column. The major fraction was eluted by chloroform and when recrystallized from ethyl acetate yielded  $3 \cdot 5$  g of β-amyrin, m.p. 197-198 °C,  $[\alpha]_D^{18}$  88·9° (c, 1·1 in chloroform) (Found: C, 84·3; H, 11·9%. Calc. for  $C_{80}H_{50}O$ : C, 84·4; H, 11·8%). Its infra-red spectrum in carbon tetrachloride solution was very similar to the published spectrum for β-amyrin (Allsop et al. 1956). The acetate prepared by reaction with acetic anhydride and pyridine had m.p. 237-238 °C,  $[\alpha]_D^{18}$  81·5° (c, 0·9 in chloroform) (Found: C, 81·75; H, 11·2%. Calc. for  $C_{33}H_{52}O_2$ : C, 82·0; H, 11·2%). Mixed m.p. with authentic β-amyrenyl acetate (kindly supplied by the Division of Industrial Chemistry, C.S.I.R.O., Melbourne) was undepressed and the infra-red spectra of the two specimens ("Nujol" mull) were identical. White (1956) records for β-amyrin, m.p. 199·5–200 °C,  $[\alpha]_D$  88° and its acetate, m.p. 241 °C,  $[\alpha]_D$  81°.

Residue B was suspended in water and extracted with chloroform. This extract was shaken with aqueous sodium hydroxide, when a considerable amount of water-insoluble sodium salt was formed. The neutral chloroform-extract was chromatographed as described above, and yielded another  $13\cdot 1$  g of  $\beta$ -amyrin.

<sup>\*</sup> Manuscript received December 2, 1957.

<sup>†</sup> Department of Organic Chemistry, University of Adelaide; present address: Chemistry Department, Purdue University, Lafayette, Indiana, U.S.A.

The sodium hydroxide-extract was filtered and the insoluble sodium salt was obtained as a partial gel. After washing with water, it was dissolved in hot ethanol, treated with charcoal, and filtered while hot. The free acid was precipitated by the addition of dilute hydrochloric acid.

The dried acid  $(4\cdot6\text{ g})$  was converted to its methyl ester by means of diazomethane, dissolved in light petroleum (b.p.  $40\text{-}60\,^{\circ}\text{C}$ ) and carefully chromatographed on an alumina column. The major fraction was eluted by benzene light petroleum (1:10) and, after being recrystallized from methanol the waxy plates had m.p.  $61\text{-}62\,^{\circ}\text{C}$ . The infra-red spectrum in carbon tetrachloride solution was typical of an ester of a saturated fatty acid and was characterized by a triplet absorption at  $8\cdot0$ ,  $8\cdot33$ , and  $8\cdot51\,\mu$  (Shreve et al. 1950) (Found: C,  $78\cdot8$ ; H,  $13\cdot2\%$ . Calc. for  $C_2$ ,  $H_{54}O_2$ : C,  $79\cdot0$ ; H,  $13\cdot3\%$ ). Methyl ester of "cerotic" acid is recorded as having m.p. 60 or  $62\,^{\circ}\text{C}$  (Beilstein). Hydrolysis of the ester by methanolic sodium hydroxide yielded the acid, which was recrystallized several times in methanol and light petroleum to a constant m.p. of  $78\text{-}79\,^{\circ}\text{C}$  (Found: C,  $78\cdot6$ ; H,  $13\cdot2\%$ . Calc. for  $C_{26}H_{52}O_2$ : C,  $78\cdot7$ ; H,  $13\cdot2\%$ ). Various workers (Beilstein) report the m.p. of "cerotic" acid, a mixture of straight chain saturated carboxylic acids, in the range  $77\cdot5\text{-}79\,^{\circ}\text{C}$ .

The author wishes to thank Dr. P. R. Jefferies and Dr. H. J. Rodda for their interest and advice, and Mr. L. J. Webb and Mr. R. G. Tracey, C.S.I.R.O., for arranging the collection of plant material. Infra-red spectra were done by Mr. A. G. Moritz.

## References

- Allsop, I. L., Cole, A. R. H., White, D. E., and Willix, R. L. S. (1956).—J. Chem. Soc. 1956: 4868.
- SHREVE, O. D., HEETHER, M. R., KNIGHT, H. B., and SWERN, D. (1950).—Analyt. Chem. 22: 1498.
- WHITE, D. E. (1956).—Rev. Pure Appl. Chem. 6: 191.