which reproduces their results with considerable accuracy in this range. However, their equation gives values which are too low when extrapolated to the higher temperature range reported here, as shown in the third column of Table I. Consequently, an equation was derived which fits the data in the range 0–40° within our limit of error.

$$\log P_{\text{mm.}} = -(2018.37/T) - 6.0303 \log T + 24.91300 \qquad (1)$$

The deviations of the observed values from those calculated by means of this equation are shown in the fourth column of the table. The heat of vaporization calculated by means of this equation with suitable corrections[2] was found to be 5720 cal./mole at the normal boiling point, 276.03°K., as compared with the calorimetric value[2] of 5482. cal/mole.

The value found at the ice-point may be compared with other values in the literature. Wiberg and Sütterlin[3] report 681 mm., Simon and Huter[4] find a value of 679.1 (calcd.), Thompson and Linnett[5] 680 mm. The average is within our limit of error, equation (1) giving a value of 679 mm.

The authors wish to thank the Physics Department of Indiana University who loaned a cathetometer and to express their appreciation for a grant from the Graduate School Fund of Indiana University.

(3) E. Wiberg and W. Sütterlin, *Z. Elektrochem.*, **41**, 151 (1935).

(4) A. Simon and J. Huter, *ibid.*, **41**, 28 (1935).

(5) H. W. Thompson and J. W. Linnett, *Trans. Faraday Soc.*, **32**, 681 (1936).

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Ursolic Acid and Paraffin Hydrocarbons from Cryptostegia Leaves[2]

BY JONATHAN W. WHITE, JR., AND F. R. SENTI

During an investigation of methods for the recovery of rubber from the leaves of *Cryptostegia grandiflora*, a brief study of some of the constituents of the leaf-cuticle wax was made. A fermentation pretreatment for rubber recovery resulted in a leaf fraction consisting of all the epidermis, veins, and latex ducts, and representing about 18% of the original leaf weight.[3] From this leaf fraction was isolated ursolic acid and a mixture of *n*-paraffin C_{29}, C_{31} and C_{33} hydrocarbons.

The leaf fraction was exhaustively extracted with hot acetone. When the extract was cooled to room temperature a voluminous crystalline precipitate formed, representing 7.1–8.2% of the weight of the material extracted. This would correspond to 1.3–1.5% of the weight of the original leaves. After one recrystallization this crude wax had an acid number of 49.0, a saponification number of 79.3 and a melting range of 222–230°. Several components were isolated from this wax as described below.

Ursolic Acid.—Saponification by ethanolic potassium hydroxide yielded an acid fraction, representing 43% of the original wax. This was purified by the method of Sando[4] to yield a material identical with ursolic acid, as shown by the melting point (285°), X-ray powder patterns of the acid and of its sodium salt, and the optical properties of the crystals.[5] The sodium salt produced the sharper diffraction pattern; since this pattern has not been published, the interplanar spacings and relative intensities of reflection are presented in Table I.

TABLE I

X-RAY POWDER PATTERN OF SODIUM URSOLATE

Anal. Calculated for $C_{29}H_{47}OCO_2H$: C, 78.87; H, 10.60, neut. equiv., 465. Found: C, 78.94, 78.76; H, 10.44, 10.64; neut. equiv., 460.

Interplanar spacing, Å.	Intensity I/I_1	Interplanar spacing, Å.	Intensity, I/I_1
14.6	0.50	2.83	0.04
10.3	.03	2.71	.08
9.2	.50	2.63	.05
7.6	.03	2.54	.04
6.90	1.00	2.42	.05
6.49	0.50	2.35	.04
5.96	.50	2.28	.04
5.68	.50	2.23	.03
5.17	.08	2.18	.04
5.01	.18	2.13	.01
4.82	.13	2.10	.04
4.61	.14	2.05	.03
4.49	.12	2.00	.01
4.18	.10	1.98	.02
3.88	.07	1.95	.03
3.75	.03	1.91	.01
3.52	.09	1.87	.01
3.37	.06	1.79	.01
3.24	.03	1.71	.01
3.17	.06	1.66	.01
3.06	.04	1.59	.01
2.92	.06		

Ursolic acid has previously been reported to be a constituent in the skin of several fruits and of such leaves as *Arbutus unedo*, *Epigaea asiatica*, and mistletoe.[6]

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[1] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture.

[2] Natural Rubber from Domestic Sources, Paper.

[3] J. Naghski, J. W. White, Jr., S. R. Hoover and J. J. Willaman, in press.

(4) C. E. Sando, *J. Biol. Chem.*, **90**, 477 (1931).

(5) The crystals are blades which show biaxial positive interference figures with $2E$ approximately 130°. The index α, crosswise of the blades, was 1.551; β, along the blades, was 1.561; and γ, perpendicular to the blades, was undetermined. These values are in agreement with those reported by Sando.[4]

(6) K. S. Markley, S. B. Hendricks and C. E. Sando, *J. Biol. Chem.*, **111**, 133 (1935); K. S. Markley and C. E. Sando, *ibid.*, **119**, 641 (1937); W. A. Nealey, *Western Canner and Packer*, **33**, No. 12, 22 (1941); A. Sanna, *Atti IV Congr. Naz. chim. pura applicata* (1932) 595 (1933); K. Fujii, N. Shimada and T. Sasaki, *J. Pharm. Soc. Japan*, **55**, 650 (1935); E. I. van Itallie, *Pharm. Weekblad*, **58**, 824 (1921).

***n*-Paraffin Hydrocarbons.**—From the neutral fraction after saponification a waxy product was isolated which, after a thirty-minute treatment in concentrated sulfuric acid at 120°, was crystallized from a mixture (1:1) of Skellysolve B and 95% ethanol. Its physical properties and long spacing (d_{001}) are shown in Table II under total hydrocarbons. A combustion analysis of this sample shows C, 85.11%, H, 14.91%. This indicates a paraffin hydrocarbon, but the limits of error of the analysis do not permit a distinction between $C_{31}H_{64}$ and $C_{33}H_{68}$ or mixtures corresponding to this range of composition. Accordingly, the product was fractionally crystallized by cooling after another sulfuric acid treatment (in which no darkening was observed). Three fractions were obtained. They are shown as fractions 1, 2, and 3 in Table II in the order of increasing solubility in the Skellysolve B–ethanol solution, and hence shorter chain length. The best photographs of the paraffins were obtained with filtered Cu radiation, using a 10-mil collimating system and a sample-to-film distance of 10 cm.

TABLE II

PHYSICAL PROPERTIES OF HYDROCARBON MIXTURES

Sample	First transition point, heating, °C.	M. p., °C.	F. p., °C.	Crystal spacing d_{001}, Å.
Total hydrocarbons	61.7	68.3–68.8	68.3	43.0
Fraction 1	61.9–62.5	68.8–69.4	68.6	43.1
80% C_{31} + 20% C_{33}[a]	62.0	68.8	..	42.8
Fraction 2	61.0–61.6	67.9–68.5	68.3	42.2
95% C_{31} + 5% C_{33}[a]	62.0–62.2	67.9–68.0	67.4	42.0
90% C_{31} + 5% C_{33} + 5% C_{29}[a]	61.0	67.8	..	About 42.0
Fraction 3	59.7–60.0	67.0–67.5	67.0	41.4
80% C_{31} + 20% C_{29}[a]	60.0–60.5	66.6–66.8	66.0	41.5
$C_{31}H_{64}$[b]	62.0–62.5	67.6–67.8	67.3	41.55

[a] A. C. Chibnall, S. H. Piper, A. Pollard, E. F. Williams and P. N. Sahai, *Biochem. J.*, **28**, 2189 (1934). [b] S. H. Piper, A. C. Chibnall, S. J. Hopkins, A. Pollard, J. A. B. Smith and E. F. Williams, *ibid.*, **25**, 2072 (1931).

Chibnall, *et al.*,[7] examined the cuticle constituents of a large number of plants and found that all paraffins present contained an odd number of carbon atoms. We assume this to be true for the paraffins of cryptostegia leaf wax and accordingly have compared our data with those given by Piper[8] and Chibnall[7] for the odd-paraffin hydrocarbons.

The data for fraction 1 agree fairly well with the data for a mixture of 80% $C_{31}H_{64}$ and 20% $C_{33}H_{68}$. The crystal spacing 43.1 Å. is slightly larger than that (42.8 Å.) given by the mixture, indicating that the content of $C_{33}H_{68}$ in this fraction is somewhat greater than 20%.

The melting point and crystal spacing of fraction 2 are in fair agreement with those of a mixture containing 95% $C_{31}H_{64}$ and 5% $C_{33}H_{68}$, but the low transition point of this fraction indicates the presence of a small percentage of $C_{29}H_{60}$. Chibnall[7] lists data for a mixture of 90% $C_{31}H_{64}$, 5% $C_{33}H_{68}$, and 5% $C_{29}H_{60}$, and in fair accord with the data for fraction 2.

The data for fraction 3 are consistent with those for a mixture of about 80% $C_{31}H_{64}$ and 20% $C_{29}H_{60}$. The melting point and crystal spacing of a 50–50 mixture of $C_{33}H_{68}$ and $C_{29}H_{60}$ would be close to the values observed for this fraction, but binary mixtures of paraffins differing in chain length by four or more carbon atoms crystallize in two phases,[8] each phase giving a separate d_{001} value. However, only one phase was observed in each of the fractions.

Acknowledgment.—Acknowledgment is made of the coöperation of C. L. Ogg, who made the microanalyses.

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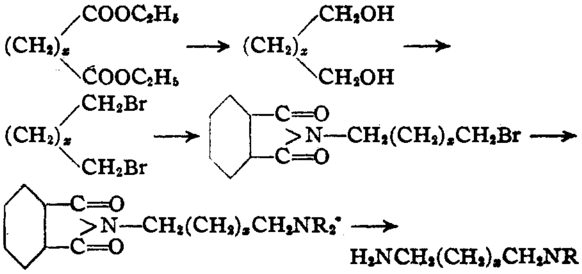
[7] A. C. Chibnall, S. H. Piper, A. Pollard, E. F. Williams and P. N. Sahai, *Biochem. J.*, **28**, 2189 (1934).

[8] S. H. Piper, A. C. Chibnall, S. J. Hopkins, A. Pollard, J. A. B. Smith and E. F. Williams, *Biochem. J.*, **25**, 2072 (1931).

NEW COMPOUNDS

Some Polymethylenediamines[1,2]

Incidental to another problem, a series of diamines of the general formula $H_2N(CH_2)_xNR_2$ has been prepared by the following reactions:



Ethyl adipate, ethyl sebacate and ethyl suberate were reduced to the corresponding polymethylene glycols either with sodium and *n*-butyl alcohol[3] or by catalytic reduction with copper chromium oxide.[4] Yields by both methods were 80–85%.

The polymethylene dibromides were prepared from the glycols by the action of dry hydrogen bromide.[5]

The 1-dialkylaminopolymethylene phthalimides were prepared from the polymethylene dibromides and potas-

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(1) The work described in this paper was done under a contract recommended by the Committee on Medical Research between the Office of Scientific Research and Development and the University of Missouri.

(2) Microanalyses by Lois May and Frances Marx, Microchemical Laboratory, Columbia University.

(3) "Organic Syntheses," Coll. Vol. II, p. 154 (1943).

(4) Ref. 3, p. 325.

(5) "Organic Syntheses," Vol. 20, p. 20.