from 95% ethanol until a constant melting point was obtained. The yield, in general, ranged from 40–75%.

DEPARTMENT OF PHARMACOLOGY
STATE UNIVERSITY OF IOWA
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Preparation of Methyl 2,3,4-Triacetyl-α-D-xyloside[1]

BY ROY L. WHISTLER, K. ANN KIMMELL AND DONALD F. DURSO

During the course of work in this Laboratory it became necessary to prepare methyl 2,3,4-triacetyl-α-D-xylopyranoside in fairly large quantities. Direct preparation of this compound from D-xylose involves the preparation of pure methyl α-D-xyloside and subsequent acetylation.[2] The procedure adopted in this work was to acetylate commercially available methyl β-D-xyloside to yield the corresponding triacetate,[3] from which the desired compound was easily prepared by isomerization with boron trifluoride using the procedure of Lindberg.[4] The yield of crystalline product was 85%.

Experimental

Methyl 2,3,4-Triacetyl-β-D-xyloside.—Commercially available crystalline methyl β-D-xyloside (15.00 g.) was acetylated at 110° with sodium acetate and acetic anhydride. The acetate was isolated in the usual manner and recrystallized from 95% ethanol. The yield was 23.85 g. of material whose m.p. 114.5–115.5° and $[\alpha]^{25}D$ −61.2 (*c*, 2.42 in chloroform) agreed with the constants previously reported.[3]

Anal. Calcd. for $C_6H_9O_5(CH_3CO)_3$: acetyl, 44.49. Found: acetyl, 44.3.

Methyl 2,3,4-Triacetyl-α-D-xyloside.—The β-compound (5.00 g.) was dissolved in 150 ml. of dry chloroform and the solution saturated with BF_3. Saturation was indicated by the formation of a white gelatinous precipitate in 10 min. The flow of gas was interrupted and the reaction flask stoppered. After 24 hours, the solution was treated with two 100-ml. portions of saturated sodium bicarbonate solution followed by three 150-ml. washes with water. The chloroform solution was dried over anhydrous sodium sulfate. Upon removal of the chloroform the product crystallized spontaneously. The yield was 4.29 g. After recrystallization from 95% ethanol, m.p. was 86–87° and $[\alpha]^{25}D$ +120.1 (*c*, 1.59 in chloroform). These values agreed with those previously reported for the desired product.[2]

Anal. Calcd. for $C_6H_9O_5(CH_3CO)_3$: acetyl, 44.49. Found: acetyl, 44.4.

(1) Paper No. 528 of the Purdue Agricultural Experiment Station.
(2) C. S. Hudson and J. K. Dale, THIS JOURNAL, **40**, 997 (1918).
(3) J. K Dale, *ibid.*, **37**, 2745 (1915).
(4) B. Lindberg, *Acta Chem. Scand.*, **2**, 426 (1948).

DEPARTMENT OF AGRICULTURAL CHEMISTRY
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β-Amyrin from *Chimaphila umbellata*[1a]

BY F. P. VEITCH, JR., AND PEARL ADAIR WELTON[1b]

During our investigation of the plant *Chimaphila umbellata* as a possible source of steroids having an oxygen function in the 11 or 12 position, a white crystalline material was isolated in 0.25% yield. From the physical constants of this compound and its derivatives (Table I) we have concluded that it is β-amyrin, a substance not previously reported as being present in this plant.

(1) (a) Obtained from S. B. Penick, New York. (b) Taken from a thesis submitted to the Graduate School of the University of Maryland in partial fulfillment of the requirements for the degree of Master of Science.

Experimental

Four kilograms of air dry *Chimaphila umbellata* was obtained in a finely divided state by the use of a Williams crusher, rollers, and a Wiley mill. The finely ground material was treated by essentially the same process as that described by Marker[2] for the isolation of sapogenins from plant sources. Upon concentration of the final ethereal extract and addition of acetone, about 20 g. of crude crystalline material precipitated. Purification of this material was effected by recrystallization from acetone followed by formation of the acetate which could be crystallized from ethyl acetate. Saponification of the acetate followed by crystallization of the regenerated compound from ethyl alcohol gave 8.5 g. of material (0.21% yield based on the weight of air-dry plant) having the following characteristics: m.p. 200°; $[\alpha]^{20}D$ +87.7° (in $CHCl_3$).

Anal. Calcd. for $C_{30}H_{50}O$: C, 84.43; H, 11.82. Found: C, 84.47, 84.58; H, 12.00, 12.00.

The acetate, benzoate and *p*-nitrobenzoate of this compound were prepared according to standard procedures. The physical constants of these derivatives and their analyses are summarized in Table I.

TABLE I[a]

Compound	M.p.,[b] °C.	$[\alpha]^{20}D$[d]	Analyses, % Found[c] Carbon	Found[c] Hydrogen	Calculated Carbon	Calculated Hydrogen
β-Amyrin	200	+87.7	84.47	12.00	84.43	11.82
Acetate	243	+80.66	82.02	11.22	82.00	11.18
Benzoate	232	.....	83.68	10.57	83.71	10.26
p-Nitrobenzoate	257	.....	...	...	...	...

[a] The values reported here are in agreement with the values reported by other workers: *cf.* L. C. King, *et al.*, THIS JOURNAL 65, 1168 (1943); A. Vesterburg, *Ber.*, **23**, 3186 (1890); N. H. Cohen, *Rec. trav. chim.*, **28**, 391; G. L. Powers, and W. E. Powers, *Pharm. Assoc.*, **29**, 175 (1940). [b] All melting points were determined on a Fisher-Johns melting point block. [c] We are indebted to Mrs. Mary Aldridge for the micro analyses reported here. [d] All rotations are in chloroform.

Values of 411 and 443 g. were obtained by the Rast method of molecular weight determination on the isolated compound, and 423 g. by the saponification method of the acetate. β-Amyrin has a molecular weight of 426 g.

(2) R. Marker, *et al.*, THIS JOURNAL, **69**, 2167 (1947).

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF MARYLAND
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The Melting Point and the Heat of Sublimation of Plutonium Trifluoride[1]

BY EDGAR F. WESTRUM, JR., AND JAMES C. WALLMANN

Careful measurements by an effusion technique of the equilibrium vapor pressures over plutonium trifluoride (PuF_3) have been reported.[2a,b] The slight deviation from linearity of the log *p versus* $1/T$ plot was represented by these authors without a stated reason as two straight lines intersecting at 1169 ± 9°, which temperature was interpreted as the melting point of plutonium trifluoride.

(1) Based on work reported in MB-IP 327, September 17, 1948, issued as Report UCRL-697 (May 19, 1950).
(2) (a) T. E. Phipps, G. W. Sears, R. L. Seifert and O. C. Simpson, *J. Chem. Phys.*, **18**, 713 (1950); (b) T. E. Phipps, G. W. Sears, R. L. Seifert, and O. C. Simpson, National Nuclear Energy Series, Plutonium Project Record, Vol. 14B, "The Transuranium Elements: Research Papers," Paper No. 6.1a (McGraw-Hill Book Co., Inc., New York, N. Y., 1949).