is the calculated 1:1 ratio. A light orange, crystalline precipitate formed; m. p. 205–210° (decomp.); yield, 1.4 g. The product was readily soluble in water.

Anal. Calcd. for $C_{10}H_{15}N_5 \cdot 2H_2O \cdot HCl$: Cl, 12.8. Found: Cl, 12.07.

In the case of the bis-guanidine derivative a solution of the monohydrochloride was prepared by dissolving a weighed amount in the calculated volume of standard hydrochloric acid and diluting to a definite concentration. This solution was used for the pharmacological tests which were carried out according to the international methods adopted for the standardization of insulin.[10]

Summary

Two derivatives of aminoguanidine have been prepared and their hypoglycemic action studied. Neither compound exerted an insulin-like action. Hypoglycemia was produced only when the amount of the compound administered approached the toxic dose, but this drop in blood sugar was due to shock to the rabbit's system as shown by the fact that injection of glucose did not cause recovery.

(10) "The Biological Standardization of Insulin," League of Nations, III, Health Report, 7 (1926).

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Beta-Ergostenol. II

BY FREDERICK W. HEYL, MERRILL C. HART AND HAROLD EMERSON

In a recent publication[1] from this Laboratory we described the separation of β-ergostenol from the mixture of isomeric sterols obtained by the saponification of α-ergostenol acetate which had been treated in chloroform solution with dry hydrogen chloride. Later Heilbron and Wilkinson[2] reported a better yield of β-ergostenol by the isomerization of the α-benzoate.

In our study[1] we obtained evidence of the presence of a third isomer; this was more soluble and had a higher rotation than β-ergostenol. We have now carried out a rather comprehensive survey of the esters of both α- and β-ergostenol.

Experimental

The β-ergostenol used had a melting point of 141° and $[\alpha]_D$ 21.2°. It was our purpose in this study to accumulate data indicating the best ester to use in the separation of α- and β-ergostenol from each other and the third isomer present in the isomerized mixture. From the data summarized in Table I it is seen that the *p*-nitrobenzoates show in alcohol the greatest differential in solubilities between the α- and β-esters, and consequently crystallization of the *p*-nitrobenzoates from this solvent should afford a satisfactory separation of these two isomers. This experiment was

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[1] Hart and Emerson, THIS JOURNAL, **54**, 1070 (1932).

[2] Heilbron and Wilkinson, *J. Chem. Soc.*, 1708 (1932).

tried on 5 g. of isomerized ergostenol but the results obtained were not appreciably better than those obtained by the fractionation of the sterol mixture itself.

TABLE I

DERIVATIVES OF α- AND β-ERGOSTENOL

	Ester[a]	Solvent	M. p., °C.	[α] 5461	Recovered ergostenol M. p., °C.	Recovered ergostenol [α] 5461	Solubilities G. in 100 cc. at 20° Alcohol, g.	Solubilities G. in 100 cc. at 20° Ethyl acetate, g.
1	β-Propionate	AcOH	110		138	18.0		
2	α-Propionate	AcOH	88	0	131	10.5		
3	β-Phthalate	EtOH	152–4		139			
4	α-Phthalate[b]	EtOH	185	1.45	127–8			
5	β-Benzoate	AcOEt	158	22.5	140	21.7		
6	α-Benzoate[b]	AcOEt	118	0	133			
7	β-*p*-Nitrobenzoate	AcOEt	180	25.0	141	22.0	0.112	1.521
8	α-*p*-Nitrobenzoate[b]	EtOH	178	0	132	10.2	.011	0.288
9	β-*m*-Nitrobenzoate	AcOEt	148	21.5	140	22.0	.063	2.062
10	α-*m*-Nitrobenzoate[b]	EtOH	165	−2.3	132	10.2	.035	0.585
11	β-3,5-Dinitrobenzoate	AcOEt	164	15.0	141	21.6	.174	4.190
12	α-3,5-Dinitrobenzoate	AcOEt	197	−2.8	133	10.3	.032	1.985
13	β-Phenylurethan[c]	EtOH	146–7	16.1				
14	α-Phenylurethan[c]	EtOH	136–7	−6.2				

	Formula	Carbon, % Calcd.	Carbon, % Found	Hydrogen, % Calcd.	Hydrogen, % Found	Mol. weight Calcd.	Mol. weight Found
5	$C_{35}H_{52}O_2$	83.3	83.1	10.4	10.1	504	519
6	$C_{35}H_{52}O_2$	83.3	83.1	10.4	10.4	504	489
7	$C_{35}H_{51}NO_4$	76.4	76.0	9.3	9.3	549	574
8	$C_{35}H_{51}NO_4$	76.4	76.2	9.3	9.2		
9	$C_{35}H_{51}NO_4$	76.4	76.0	9.3	9.4	549	524
10	$C_{35}H_{51}NO_4$	76.4	76.5	9.3	9.2	549	555
11	$C_{35}H_{50}O_6N_2$	70.7	70.4	8.5	8.3		
12	$C_{35}H_{50}O_6N_2$	70.7	70.5	8.5	8.5		
13	$C_{35}H_{53}O_2N$	N, 2.70	N, 2.96				
14	$C_{35}H_{53}O_2N$	N, 2.70	N, 2.97				

[a] Esterification by the usual method with pyridine as a solvent. [b] Without pyridine as a solvent. [c] Reaction in dry benzene. We wish to correct an error in our previous paper, THIS JOURNAL, **52,** 2015 (1930). The correct melting point of ergosteryl phenylurethan should be 185°.

Summary

1. The individuality of β-ergostenol has been established by the preparation of the following esters: propionate, phthalate, benzoate, *p*-nitrobenzoate, *m*-nitrobenzoate, 3,5-dinitrobenzoate and phenylurethan. (2). The corresponding esters of α-ergostenol have been prepared. Of these the 3,5-dinitrobenzoate melting at 197° is new, as is the phthalate melting at 185°.

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