might be a mixture consisting of diosgenin along with a considerable amount of (II).

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## Studies on Digitalis Glycosides. Acetylgitoxin-a from Gitoxin

Acetylgitoxin- $\alpha$  had been obtained from lanatoside-B by means of enzymatic decomposition by Stoll *et al.*<sup>1)</sup> We prepared the same compound from gitoxin by means of partial acetylation.

Gitoxin was acetylated for 16 hours with equimolar amount of acetic anhydride in pyridine solution at room temperature, the crude acetate was submitted to silicagel chromatography, and the main fraction eluted with CHCl<sub>3</sub>•MeOH (25:1) was recrystallized from EtOH•Et<sub>2</sub>O to colorless plates, m.p.  $190\sim202^{\circ}/246\sim249^{\circ}$ . Analytical values corresponded to those of monoacetylgitoxin (*Anal.* Calcd. for C<sub>43</sub>H<sub>66</sub>O<sub>15</sub>•H<sub>2</sub>O:C, 61.40; H, 8.15; COCH<sub>3</sub>, 5.11. Found: C, 60.99; H, 8.25; COCH<sub>3</sub>, 5.52). The results of comparison of this product with authentic sample of acetylgitoxin– $\alpha$ , kindly furnished by Prof. A. Stoll, indicated the identity of the two glycosides as follows:

| $Acetylgitoxin-\alpha$                           | Monoacetylgitoxin obtained here             | Mixture           |
|--|---|-------------------|
| m.p. 190~203°/245~248° 2)                        | $190 \sim 202^{\circ}/246 \sim 249^{\circ}$ | 190~202°/245~249° |
| Rf (HCONH <sub>2</sub> , MeCOEt·Xylene=1:1) 0.51 | 0.51  | 0.51              |
| $(HCONH_2, BuOH \cdot Toluene = 1 : 6)$ 0.53     | 0.53  | 0.53              |
| $(\alpha)^{30}_{11} + 16^{\circ 1}$ (pyridine)   | $+17.5^{\circ}$ (pyridine)                  |                   |

The infrared spectrum of both also agreed well. It is presumable that acetyl-digitoxin- $\alpha$  and acetyldigoxin- $\alpha$  could be prepared by a similar procedure from digitoxin and digoxin, respectively, and the studies about it are in progress.

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<sup>1)</sup> A. Stoll, W. Kreis: Helv. Chim. Acta, 17, 592(1934); A. Stoll, A. von Wartburg, W. Kreis: *Ibid.*, 35, 1324(1952).

<sup>2)</sup> Though m.p. of acetylgitoxin-α had been reported as 203~204° 1), we found it showed the above-mentioned double melting point.