

tures in sols, Zocher has described strikingly similar observations (7). On standing, V_2O_5 sols tend to form ordered aggregates, and Zocher has also observed twinkling under crossed nichols. This similarity is of special interest because Zocher's ordered colloids belong to a type that has many properties in common with purified TMV solutions.

All in all, the observations reported in this paper are compatible with the hypothesis that the different large inclusion bodies and the small particles responsible for twinkling are forms of aggregation which TMV may assume inside the plant. They suggest that the conditions of stability are different for the different forms.

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Polymorphism of Pregnenolone Acetate

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In this laboratory rigorously purified samples of 5-pregnen-3 β -ol-20-one acetate from various sources showed a melting point lower than those reported by previous investigators. This behavior led us to examine the properties of this compound more carefully in order to determine whether polymorphic forms were involved.

Other workers have reported that pregnenolone acetate melts at 146°–147° when prepared from stigmasterol through 3 β -acetoxybisor-5-cholenic acid (1), and at 147°–149° when prepared through its semicarbazone after oxidation of cholesteryl acetate dibromide (2, 3). In the present work, a sample of pregnenolone prepared through selective reduction of 5,16-pregnadien-3 β -ol-20-one from diosgenin (4) was acetylated, and the crude product was crystallized from acetone. This was followed by crystallization from isopropanol, ethyl acetate, isopropanol, and acetone. The melting range did not change after the first two crystallizations. When heated from 135° at

0.5°/min, the pure material (prismatic needles, or parallelopipeds, dried *in vacuo* and powdered) partly liquefied at 144.8°–145.5°, solidified at 145.5°–146°, and fused again at 147.5°–150.0°. A sample inserted at 145.5° melted almost completely, then solidified at 146°–147°, and remelted at 147.8°–150.5°. Resolidified samples showed no change below 148°, then melted at 148°–150.5°. On powdering and drying *in vacuo* at 100° or 110° no change in range of fusion was observed, although 1–4% of samples sublimed. Continued high-temperature (145°) treatment caused decomposition as shown by lowered melting points and faulty analyses. The pure material (*Anal.* Calcd for $C_{23}H_{34}O_3$: C, 77.05; H, 9.56. Found: C, 76.2; H, 9.65) showed the following specific rotations: $[\alpha]_D^{21} = +18.9^\circ \pm 0.6^\circ$ (1% in EtOH); $[\alpha]_D^{21} = +13.8^\circ \pm 0.6^\circ$ (1% in $CHCl_3$); $[\alpha]_D^{18} = +11.1^\circ \pm 0.6^\circ$ (1% in dioxane).

Further samples of pregnenolone acetate showing the same phenomenon upon melting were prepared from diosgenin (4), 3-acetoxy-bisnorcholeonic acid (1), and cholesterol (2, 3). Each sample was recrystallized exhaustively from isopropyl alcohol, acetone, ethyl acetate, and finally benzene-ethyl ether (1:10) to constant melting range. Solubility analyses (5) indicated no impurity within the limits of the method. The soluble portions from the solubility analyses and the final recrystallizations were, within instrumental limitations, identical to the main crystal fractions in melting range, optical rotation ($[\alpha]_D^{20} = 10.6^\circ \pm 0.7^\circ$ in dioxane), molecular extinction coefficient at 281 m μ ($\epsilon = 42.09 \pm 0.31$, 2 mg/cc in absolute ethanol), and infrared absorption spectrum.

The semicarbazone of the purified pregnenolone acetate was prepared in quantitative yield (mp = 253°–255°C dec, inserted at 245°), and recrystallized to constant mp (256.5°–257°C) from chloroform. The pregnenolone acetate prepared by splitting with pyruvic acid (3) still exhibited the wide melting range characteristic of polymorphic forms.

On the basis of this physical and chemical evidence, the behavior upon fusion of pregnenolone acetate can be ascribed to the presence of one or more polymorphic forms.

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¹ All melting points are corrected.

Roots, herbs, leaves, and barks still used as healing agents by the contemporary Mayas have been turned over by the Knaggs Expedition to Sterling-Winthrop Research Institute for study. Headed by Nelson S. Knaggs, of Hilton-Davis Chemical Company, dye manufacturers of Cincinnati, the expedition visited Guatemala, Yucatan, and Honduras, testing new

tropical medicines developed by Sterling-Winthrop, observing Maya methods of weaving and dyeing textiles, and collecting natural-history specimens.

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