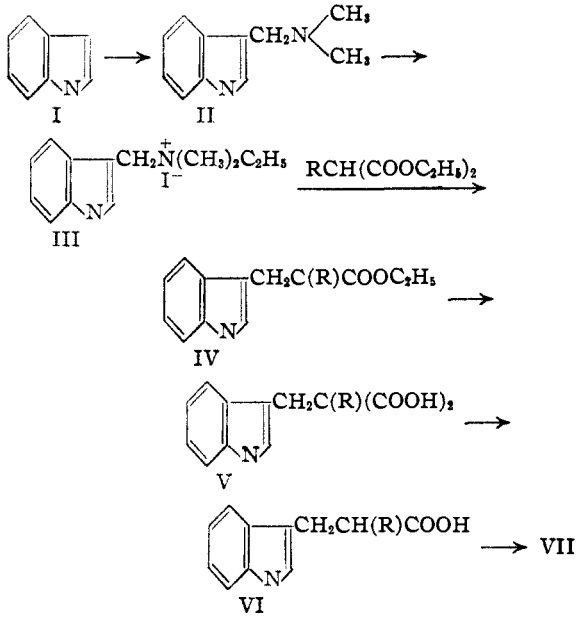
COMMUNICATIONS TO THE EDITOR

A NEW SYNTHESIS OF TRYPTOPHAN

Sirs:

In view of the recent communication of Snyder, *et al.*,[1] on the use of gramine methiodide as an alkylating agent, we wish at this time to record our experience in this field.

Tryptophan (VII) was synthesized by the procedure outlined in the following equations



The condensation was carried out successfully when R = H, $HNCOCH_3$ or $HNCOC_6H_5$. The ethyl α-carbethoxy-β-(3-indolyl)-propionate, (R = H), was hydrolyzed to the malonic acid which melted at 187–189° when pure.[2] The free acid was easily converted to β-(3-indolyl)-propionic acid, which melted at 128–130°.[2]

Ethyl α-carbethoxy-α-acetamido-β-(3-indolyl)-propionate, (R = $HNCOCH_3$), melted at 157° (Calcd. N, 8.09. Found: N, 8.27). Ethyl α-carbethoxy - α - benzamido - β - (3 - indolyl) - propionate, (R = $HNCOC_6H_5$), melted at 142° (Calcd. N, 6.86. Found: N, 7.20).

Hydrolysis of IV in sodium hydroxide solution proceeded quite smoothly. The free acetamido and benzamidomalonic acids (V), m. p. 135–137° (dec.), 85–90° (dec.), respectively, were used inthe crude form since some decarboxylation accompanied purification, thus rendering the preparation of analytical samples difficult. Complete decarboxylation was effected by heating at 180–200° until the evolution of carbon dioxide ceased.[2] The structures of the acetyl and benzoyltryptophan so prepared were confirmed by direct comparison with authentic specimens. The free amino acid was obtained from the above derivatives by hydrolysis according to procedures described in the literature. The over-all yield of tryptophan based on indole as the starting material was as high as 35% of the theoretical. Details of the procedure will be published at some future date.

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(1) Snyder, Smith and Stewart, This Journal, **66**, 200 (1944).

(2) Maurer and Moser, *Z. physiol. Chem.*, **161**, 131 (1926). The authors report 188° as the m. p. for the malonic acid and 134° for the decarboxylated product.

LOW ANGLE X-RAY SCATTERING FROM CHRYSOTILES

Sir:

It is becoming increasingly evident that studies of low angle scattering of monochromatic X-rays will prove valuable in the study of sub-optical–microscopic structures. Bernal and Fankuchen in their virus studies,[1] Bear in his collagen studies,[2] Warren for carbon black[3] and Kratky in studies of proteins and of fibers,[4,5] found such significant small angle scattering. We have now studied various chrysotiles with X-rays using a standard technique for the wide angle scattering and the apparatus and technique employed in the study of dry gels of tobacco mosaic virus[1] for the low angle work. Copper characteristic radiation filtered through nickel foil was used. In the low angle work a fine slit system to define the X-ray beam and a 30 cm. specimen to film distance permitted the study of the small angle scattering down to angles corresponding to 500 Å. The specimens were stationary thin slabs of chrysotile mounted so that the X-rays passed through the thin direction of the specimen. The wide angle diagrams are substantially alike and all showed an undifferentiated low angle equatorial scattering. The use of a fine slit system[1] resolved this scattering into clearly differentiated lines. In each of the four samples studied to date, at least two lines were always visible, the ratio of their Bragg spacings being $\sqrt{3}$. This suggests strongly that these chrysotiles are composed of parallel fundamental fibrils—hexagonally packed in cross section. The diameters of the fibrils can be computed from the Bragg spacings (on the hypothesis

(1) Bernal and Fankuchen, *J. Gen. Physiol.*, **25**, 111–165 (1941).

(2) Bear, This Journal, **64**, 727 (1942).

(3) Biscoe and Warren, *J. Applied Phys.*, **13**, 364–371 (1942).

(4) Kratky, Sekora and Treer, *Z. Elektrochem.*, **48**, 587–601 (1942).

(5) Kratky and Sekora, *Naturwissenschaften*, **31**, 46–47 (1943).