

rapid final solution of the remaining details of triterpene structure.

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<sup>1</sup> For summaries, see Spring, F. S., *Ann. Rep. Chem. Soc.*, **38**, 192 (1941). Noller, C. R., *Ann. Rev. Biochem.*, **14**, 383 (1945).

<sup>2</sup> Barton, D. H. R., and Jones, E. R. H., *J. Chem. Soc.*, 659 (1944).

<sup>3</sup> Haworth, R. D., *Ann. Rep. Chem. Soc.*, **34**, 327 (1937).

<sup>4</sup> Cf. Meisek, A., Jeger, O., and Ruzicka, L., *Helv. Chim. Acta*, **32**, 1075 (1949).

<sup>5</sup> Winterstein, A., and Stein, G., *Ann.*, **502**, 223 (1933). (On repetition of this work, we find for  $\beta$ -amyrin-III, m.p. 190–191° corr.  $[\alpha]_D^{20} = -28^\circ$ .)

### Molecular Rotation Contribution of C(17) in the Steroid Skeleton

Fieser and Fieser<sup>1</sup> have recently deduced molecular rotation contributions for the various asymmetric centres in the steroid skeleton. Their values for the contribution of C(17) were obtained, in the absence of data permitting direct comparison, from the molecular rotation of cholestane by subtracting the contributions of all the other asymmetric centres, some of which (those of C(9), C(10) and C(13)) appear to be unreliable.

The rotation contributions ( $\Delta$ ) of C(17) can now be estimated more simply and reliably by comparing the molecular rotations,  $[M]_D$ , of androstane, 5-*allo*-pregnane and 5-*allo*-17-*isopregnane*, the last-named compound having recently been prepared by Casanova and Reichstein<sup>2</sup>.

Compound	Grouping at C(17)	$[M]_D$	$\Delta$	Ref.
5- <i>allo</i> Pregnane	CHEt ( $\beta$ )	+ 52°		2, 3
Androstane	CH <sub>3</sub>	+ 5°		4
Contribution of C(17) carrying a ( $\beta$ )-ethyl group				
5- <i>allo</i> -17- <i>iso</i> Pregnane	CHEt ( $\alpha$ )	- 95°	+ 47°	2
Androstane	CH <sub>3</sub>	+ 5°		4
Contribution of C(17) carrying an ( $\alpha$ )-ethyl group				
			- 100°	

The contributions of C(17) thus calculated are not equal in magnitude, presumably owing to vicinal action of the asymmetric centres at C(13) and C(14). The values for the contributions of C(17) calculated

indirectly by Fieser and Fieser ( $\pm 35^\circ$  for  $\beta$ - and  $\alpha$ -substituents respectively) are of the same sign and the same order of magnitude as those given above.

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<sup>1</sup> Fieser, L. F., and Fieser, M., "Natural Products related to Phenanthrene", 211 (3rd edit., New York; Reinhold, 1949).

<sup>2</sup> Casanova, R., and Reichstein, T., *Helv. Chim. Acta*, **32**, 647 (1949).

<sup>3</sup> Ruzicka, L., Goldberg, M. W., and Hardegger, E., *Helv. Chim. Acta*, **22**, 1294 (1939).

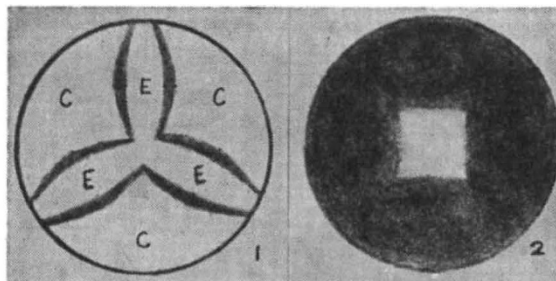
<sup>4</sup> Prelog, V., Ruzicka, L., and Wieland, P., *Helv. Chim. Acta*, **27**, 66 (1944).

### Reactivity of Different Faces of a Copper Single Crystal

By using single-crystal metal spheres, Hausser<sup>1</sup>, Tamman and Sartorius<sup>2</sup>, and in particular A. T. Gwathmey<sup>3</sup> and his collaborators, have demonstrated the marked differences in the physico-chemical properties of the surfaces of a crystal. I have extended their techniques to reactions in which crystals may be etched by vapours. A single crystal of the substance under examination is allowed to react with gaseous reagent under conditions of temperature and pressure such that the product is volatilized away as fast as it is formed. For example, a single crystal of pure copper was electrolytically polished and subjected to the action of a halogen vapour at 444° C. and about 1 mm. pressure. Under the conditions of the experiment the cuprous halide sublimed away continuously, leaving a clean, etched copper surface. Etch pits were produced with their reflecting surfaces (micro-facets) parallel to certain crystallographic planes, so that, on rotating the sphere in a parallel beam of light with the eye looking along the beam, reflexions were seen at certain orientations. It was thus possible to identify easily the facets produced and, by roughly comparing the intensities of the reflected beams, to judge which predominated.

In general, the facets exposed were (111), (011), (012) and (001). In all cases the reflexions from (111) and (011) were strong and that from (001) very weak. With iodine and bromine the (012) reflexion was also strong; but with chlorine it was weak.

Normally the facets were not uniformly distributed, but arranged in patterns as with etching by acids, etc. These patterns were related to the symmetry of the crystallographic axes concerned. Two typical examples are shown diagrammatically.



Copper single-crystal spheres, electrolytically polished, then etched by bromine at 444° C. (1) View along (111) direction with illumination by beam of light parallel to that direction. The regions marked E are the 'cube edge' areas and are centred on the (011) poles. The regions marked C are 'cube face' areas and are centred on the (001) poles. Both reflect light back along the (111) direction. The dark areas, which do not reflect in this direction, are centred on the (012) poles. (2) View along (001) direction similarly illuminated. Only a small square diffuse patch reflects light back