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OXIDATION OF OZONIDES WITH CHROMIC ACID

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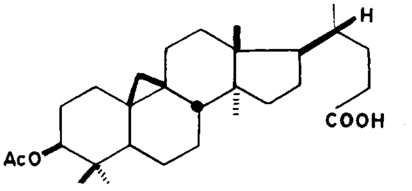
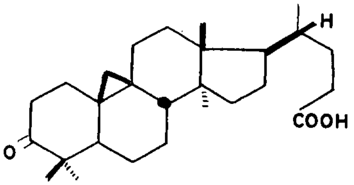
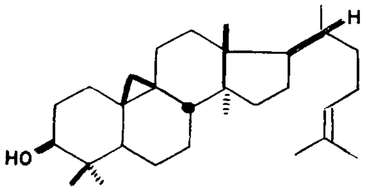
IN connection with a research project, rather large quantities of acids I, II, conceivably readily accessible from cycloartenol (III), were required. Surprisingly, conventional ozonolysis (decomposition of ozonide with hot water) of cycloartenyl acetate is reported[1] to give a complex reaction product, from which trisnor-acid (I) could not be isolated. We now find that if the crude ozonide is directly subjected to oxidative cleavage with chromic acid (Jones reagent[2]), acid I can be obtained in an excellent yield. Likewise, the crude ozonide from cycloartenol, on cleavage (with concomitant oxidation of C_3-OH) with Jones reagent, directly gave II in over 80% yield. It may be mentioned here that chromic acid oxidative cleavage of ozonides has not been reported so far, though several other oxidising agents[3] have been employed and chromic acid oxidation of products from usual cleavage of ozonides has been resorted to[4].



I



II



III

The above oxidative work-up of ozonides has been applied to a few other olefins and the results (Table 1) demonstrate the usefulness of this procedure.

TABLE 1. CHROMIC ACID OXIDATION OF OZONIDES

Olefin	Product(s) isolated	Yield (%)
Cyclohexene	adipic acid	80-85
Oleic acid	pelargonic acid) and azelaic acid)	85-90
Undecylenic acid	sebacic acid	95
Δ^3-Carene	keto acid[5]	55-60

In the case of cycloartenyl acetate it was demonstrated that the crude ozonide consumed ~ 2 atoms of oxygen per mole.

A typical experimental procedure for the chromic acid oxidation of ozonides is given below:

Oxidation of cycloartenol (III) to the *trisnor*-keto acid (II)

Cycloartenol (1.0 g) in dry EtOAc (200 ml) was ozonised at -10°, in the usual manner. The solvent was flashed off to give the crude ozonide, which was taken up in acetone (15 ml). This was cooled (0°) and Jones reagent[2] introduced dropwise with cooling and stirring till a brown colour persisted (2 ml). The reaction mixture was allowed to stand at room temp. for 5 min. and then diluted with ice-water (100 ml). The product was taken up in ether (70 ml x 3) and separated with NaOH aq. (5%) into acidic and neutral parts. The acidic part (0.82 g, m.p. 145-170°) was crystallised from CH_3CN to give pure II (correct analytical data and the expected IR and PMR characteristics), m.p. 168-175°, $[\alpha]$ +35.8 ($CHCl_3$, *c* 0.95%).

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