A Simple and Inexpensive Method for the Preparation of $RCD_2 \cdot CO_2H$ and $RCD_2 \cdot OH$

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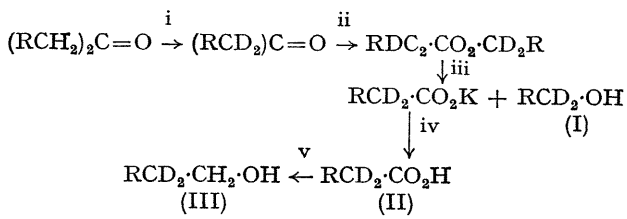
Summary The oxidation of α-deuteriated ketones, followed by hydrolysis, provides a simple and inexpensive method for the preparation of α-deuteriated acids and alcohols.

IN connection with some mass spectral studies, it was necessary to prepare as starting materials the labelled compounds $RCD_2 \cdot OH$ (I) and $RCD_2 \cdot CO_2H$ (II) in relatively large quantities (>10 g). Previous methods require the use of relatively expensive deuteriated reagents (*e.g.* $LiAlD_4$ or $NaBD_4$)[1] for (I), and exchanges in sealed tubes [for (II)];[2] these factors usually limit the scale of the preparation. The procedure outlined in the Scheme is inexpensive and is capable of large-scale preparation.

The ease of base-catalysed deuteriation of the α-positions of ketones is well known. Three successive exchanges (12—24 h) with D_2O give deuteriated compounds with > 90% deuteriation.[3] High yields (70—90%) are obtained for the oxidation of these ketones to the esters *via* the Baeyer–Villiger oxidation, using reagents such as commercially available trifluoroacetic anhydride and hydrogen peroxide.[4] Hydrolysis with base, followed by the usual work-up, gives (I) and (II). Alternatively, the ester can be reduced with $LiAlH_4$ to give the deuteriated alcohols (I) and



SCHEME. Reagents: i, Base–D_2O (3 exchanges); ii, $(CF_3 \cdot CO)_2$-O–H_2O_2; iii, KOH; iv, H^+; v, a, CH_2N_2; b, $LiAlH_4$.

(III). In our hands, 43 g (0·5 mol) of pentan-3-one gave 24 g (0·32 mol) of $CH_3 \cdot CD_2 \cdot CO_2H$, and 15 g (0·31 mol) of $CH_3 \cdot CD_2 \cdot OH$, an overall yield of 64% and 62%, respectively.

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