Easy Synthesis of 2-Hydroxy-3-methylcyclopent-2-enone

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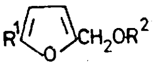
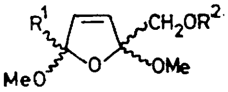
Summary A new, easy synthetic route to 2-hydroxy-3-methyl cyclopent-2-enone from 5-methylfurfuryl alcoho is described, in which the key intermediates are derivatives of 2,5-dimethoxy tetrahydrofuran prepared from the anodic oxidation of the corresponding derivatives of furan, followed by hydrogenation.

The synthesis of 2-hydroxy-3-methylcyclopent-2-enone (**4a**) is of interest since it has been found to be a key compound in the flavour of various foods.[1] We describe herein a new, easy synthetic route to (**4**) from derivatives of furan.


(1)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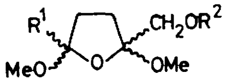
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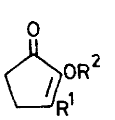
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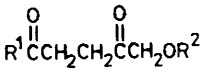

$R^1\overset{O}{\overset{\|}{C}}CH_2CH_2\overset{O}{\overset{\|}{C}}CH_2OR^2$

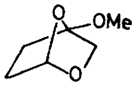
(5)

a; $R^1 = Me$, $R^2 = H$
b; $R^1 = R^2 = Me$
c; $R^1 = R^2 = H$

Electrochemical oxidation of commercially available 5-methylfurfuryl alcohol (**1a**) in methanol gave a stereoisomeric mixture of the dimethoxy compounds (**2a**) (70% yield),[2] which were hydrogenated over Raney nickel in methanol to give (**3a**) in good yield (>90%). A solution of (**3a**) (0·05 mol) in water (30 ml) was treated with an acidic ion-exchange resin (Dowex 50W, 1 g) for 15 min at room temperature, the solution was mixed with aqueous sodium carbonate (100 ml, 1%), and was heated to reflux for 3 h. Extraction of the reaction mixture with chloroform, followed by removal of the solvent, gave crystals of (**4a**) (70—80% yield).

The formation of the linear diketone (**5a**) as an intermediate was confirmed by its isolation and cyclization to (**4a**) by treatment with sodium carbonate.

The trimethoxy compound (**3b**), prepared from (**1b**), was similarly converted into (**4b**) (73% yield). The intermediate (**5b**) was also isolated. Dimethoxytetrahydrofurfuryl alcohol (**3c**), prepared from (**1c**), however, could not be converted into (**5c**) by treatment with acid; instead, compound (**6**) was obtained in this case, which was stable towards base.


(6)

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[1] C. M. Leir, *J. Org. Chem.*, 1970, **35**, 3203; K. Sato, S. Inoue, T. Kitagawa, and T. Takahashi, *ibid.*, 1973, **38**, 551.

[2] N. Elming, *Adv. Org. Chem.*, 1960, **2**, 67.