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A SIMPLE ONE-POT PREPARATION OF 1-ARYLSULFONYLBICYCLOBUTANES FROM γ,δ-EPOXYSULFONES

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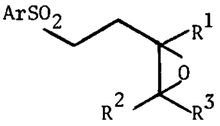
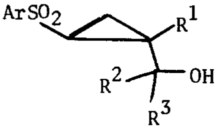
Abstracts: Sequential treatment of γ,δ-epoxysulfones with butyl lithium, methanesulfonyl chloride, and butyl lithium provides 1-arylsulfonylbicyclo[1.1.0]butanes in over 50% overall yields.

We have previously shown that 1-(arylsulfonyl)-2-(hydroxyalkyl)bicyclobutanes could be obtained from γ,δ-epoxysulfones (1) by dehydration of alcohols 2, epoxidation of the resultant 2-vinylcyclopropane derivatives and base treatment of the epoxide.[1] We report now on a simpler preparation of bicyclobutanes (4), devoid here of the hydroxyl function, by base treatment of the methanesulfonate esters (3) of alcohols 2:



1



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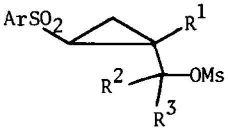

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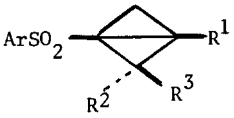

4

a $R^1=R^2=R^3=H$
b $R^1=R^2=H$, $R^3=CH(CH_3)_2$
c $R^1=H$, $R^2=R^3=CH_3$
d $R^1=CH_3$, $R^2=R^3=H$

(Ar=Ph,unless otherwise indicated)

The whole sequence can be conducted in one pot at 0°C during ten minutes and in over 50% overall yield. Thus, sequential treatment of 1a in tetrahydrofuran (THF) at 0°C (ice cooling) with one equivalent each of n-butyl lithium (BuLi; 1.95 M in hexane), methanesulfonyl chloride (MsCl; 2N in THF), and BuLi again furnished 4a,[2] mp 81-2°C, in 50-53% overall yield.[3] Each individual step required ca. three minutes for completion, including addition time of the reagent via syringe (3 to 6 mmol scale). The last step, in particular, had to be here of short duration in order to avoid extensive decomposition of 4a.

Similar treatment of 1b (Ar=p-tolyl) provided 4b (Ar=p-tolyl), mp 66-7°C, in 65% overall yield.

The sequence of reactions was also applicable to 1c, where the intermediate mesylate would be tertiary, without any concurrent elimination being observed. Bicyclobutane 4c was obtained as a liquid in 47-51% overall yield.

Compounds **4** could, of course, be alternatively prepared from alcohols **2**,[1,4] in a one-pot reaction, or from mesylates **3**. Thus, the isomeric **2d**[1] were separated as their mesylates and treated separately with one equivalent of BuLi to provide **4d**, mp 60-1°C, in over 80% yield. Treatment of **2b**-tosylate with BuLi furnished **4b** in 91% yield. Compound **4a** was obtained from **3a** in 55% yield, indicating that the last step in the above sequence was detrimental to the yield.

All compounds **4** were identified by their characteristic ^{1}H NMR spectra and by the ready formation of diiodides by addition of iodine across the central bond.[1,2,5]

A full account of this and of the previous work,[1] with detailed analytical and spectral data, will be submitted for publication in the near future.

References and Notes

1. Y. Gaoni, *Tetrahedron Lett.* 503 (1976).
2. All new solid compounds gave correct elemental analyses. All new compounds had spectral properties in accordance with the suggested structures.
3. Reported yields refer to chromatographically separated, mostly solid, spectroscopically pure compounds.
4. B. Corbel and T. Durst, *J.Org.Chem.* **41**, 3648 (1976).
5. Mp of diiodide derived from **4a**, 149-50°C; from **4b** — 117-8°C; from **4c** — 150-1°C; from **4d** — 136-7°C.

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