NOTE

Crystal structure of (1R*,6S*,8R*,10R*)-8-(p-bromobenzensulfonyl)-1,10-dimethyltricyclo[4.4.0.0^3,8]decan-2-one

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The acid- or base-catalyzed isomerization of the cyclopropanol derivative (I) (Reusch, 1971), has provided a simple route to the perhydroindanedione, (II). Under similar conditions, another cyclopropanol derivative (III) was expected to yield (IV), a logical precursor in the synthesis of pinguisone (Sorm, 1969). However, base-catalysed isomerization of (III) gave a ketol as the major product.

A structural determination by standard chemical and instrumental methods did not permit an unambiguous structural assignment. Since the ketol was not suitably crystalline, we prepared and analyzed the p-bromobenzensulfonate derivative.

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A solution of 90 mg (0.22 mmol) of the unknown ketol in 2 ml of dry pyridine was treated at 0°C with a large excess of p-bromobenzenesulfonyl chloride. After complete dissolution, the resulting solution was stirred at room temperature for three days. The solution was then poured into water at 0°C, stirred and extracted with ether. The organic phase was washed sequentially with dilute hydrochloric acid, water and saturated sodium bicarbonate solution. After drying over anhydrous sodium sulfate, careful evaporation gave 133 mg (70%) of white crystals. A portion was dissolved in ether and placed in a closed vial, from which very slow evaporation of the solvent gave excellent single crystals appropriate for collecting three-dimensional X-ray data. An analytical sample had mp 124-125; ir(CCl₄) 1734, 1325-1380, 1178, 920, 868 cm⁻¹; nmr(CDC1₃) δ 8.88(d, 3H, J = 6Hz), 0.92(S, 3H), 1.20-2.86(m, 11H), 7.78(s, 4H); mass spectrum (70 eV) m/e (rel. intensity) 414(3), 412(3), 221(6), 219(6), 193(45), 176(24), 157(14), 155(14), 148(28), 133(12), 120(12), 110(100), 93(17), 81(14), 69(21), 55(21).

Analysis:

Calculated for C₁₈H₂₁BrO₄S:

\[ C, 52.31; H, 5.12\% . \]

Found:

\[ C, 52.29; H, 5.21\% . \]

Crystals of (1R*, 3R*, 8R*, 10R*)-8(p-bromobenzenesulfonyl)-1,10-dimethyltricyclo [4.4.0.0³,₈] deca-2-one (Figure 1) were in the form of clear, colorless prisms. Unit-cell dimensions, which are given with the crystal data, were obtained from the least-squares refinement of three instrument angle for 36 reflections that were carefully centered (Vandlen & Tulinsky, 1971). The three dimensional intensity and crystal data were measured with a Picker FACS-1 diffractometer which was equipped with a graphite monochromator (2θm = 12.16°). The intensity data were recorded by the ω-technique with a scan range of 1.5° and a scan rate of 1°/min. The data were corrected for Lorentz and polarization effects, and an empirical absorption correction (North, Phillips & Mathews, 1968) was applied to the data.

**Crystal Data**

\[ C₁₈H₂₁BrO₄S; \quad FW = 413.34 \]
\[ a = 11.949(2), \quad b = 12.475(2), \quad c = 6.682(1) \text{ Å} \]
\[ α = 102.49(1), \quad β = 100.86(1), \quad γ = 108.48(1) \]°
\[ V_c = 886.21 \text{ Å}^3; \quad Z = 2; \quad D_c = 1.545 \text{ g cm}^{-3} \]
\[ F(000) = 424; \quad μ(Mo Kα) = 25.90 \text{ cm}^{-1} \]
Space group: P1 (No. 2) (from structural analysis)
\[ 2θ_{max} = 50° (\text{Mo } Kα, \lambda = 0.70926 \text{ Å}) \]
Independent reflections: 3273; 2294 ≥ 2.5σ(I)
Tube current: 14 mA
Room temperature: 24°C