



**100**

**[1H $\beta$ ,6H $\beta$ ]-7 $\alpha$ -Acetoxy-3-methyl-11-thiabicyclo[4.4.1]undeca-2,4,8-triene-**

**11,11-dioxide (100).** A mixture of chromium complex **23** (0.200 g, 0.690 mmol), 1-acetoxy-1,3-butadiene (0.327 mL, 2.75 mmol), and 1,2-dichloroethane (250 mL) was irradiated (medium-pressure Hg lamp, uranium glass filter) until complete consumption of the starting materials was indicated by TLC analysis (~45 min). The solvent was removed *in vacuo* and the resulting residue was stirred in MeOH (50 mL) under a blanket of CO gas (balloon) until decomplexation was observed (red-orange color disappears and the mixture turns green). The mixture was filtered through a pad of celite, concentrated *in vacuo*, and purified by flash column chromatography (silica gel, hexanes/ethyl acetate, 5 : 1), which provided 0.146 g (79%) of cycloadduct **100** as white needles: mp (hexanes) 147–149 °C;  $R_f$  0.23 (hexanes/ethyl acetate, 5 : 1); IR (neat)  $\nu$  1735, 1299, 1242, 1123, 1030  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.97–5.99 (m, 1H), 5.90 (d,  $J = 13.5$  Hz, 1H), 5.71–5.77 (m, 1H), 5.57–5.61 (m, 1H), 5.54 (dd,  $J = 12.8, 7.3$  Hz, 1H), 5.38 (d,  $J = 7.5$  Hz, 1 H), 4.05–4.07 (m, 1H), 3.74–3.78 (m, 1H), 2.94 (ddd,  $J = 18.8, 5.6, 3.2$  Hz, 1H), 2.66 (ddd,  $J = 15.5, 9.0, 6.5$  Hz, 1H), 2.13 (s, 3H), 1.91 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.4 (C), 135.3 (C), 135.0 (CH), 134.1 (CH), 126.3 (CH), 119.4 (CH), 118.7 (CH), 70.2 (CH), 68.5 (CH), 63.2 (CH), 28.3 ( $\text{CH}_2$ ), 26.7 ( $\text{CH}_3$ ), 20.9 ( $\text{CH}_3$ ); MS (EI)  $m/e$  (rel. intensity) 268 ( $\text{M}^+$ , 0.3), 226 (3), 191 (8), 178 (31), 144 (70), 129 (100), 91 (93), 65 (29); HRMS calcd for  $\text{C}_{11}\text{H}_{14}\text{O}_3\text{S}$  ( $\text{M}^+ - \text{C}_2\text{H}_2\text{O}$ ) 226.0664, found 226.0665. Anal. calcd for  $\text{C}_{13}\text{H}_{16}\text{O}_4\text{S}$ : C, 58.19; H, 6.02. Found: C, 57.86; H, 6.11.

### Data is reported in this order:

mp/bp (solvent) data °C;  $[\alpha]_D^{26}$  +/- data (*c* conc in g/100 mL, solvent);  $R_f$  data (solvent);

IR (neat or solvent)  $\nu$  data  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  data;  $^{13}\text{C}$  NMR (100

MHz,  $\text{CDCl}_3$ )  $\delta$  data (dept data); MS (EI) *m/e* (rel. intensity) data; HRMS calcd for

$\text{C}_{11}\text{H}_{14}\text{O}_3\text{S}$  ( $\text{M}^+$ ) X.XXXX, found X.XXXX. Anal. calcd for  $\text{C}_{13}\text{H}_{16}\text{O}_4\text{S}$ : C, 58.19; H,

6.02. Found: C, 57.86; H, 6.11.

### Common errors:

- The number of sig. figs in the reagents should correspond to the sig. figs in the yield.
- mL, mp, bp, mmol, mol, min, and h do not have periods.
- *in vacuo* is in italics
- If you used saturated aqueous ammonium chloride solution, then write it out.
- mp, bp,  $R_f$  and  $[\alpha]_D^{26}$  do not have equal '=' signs.
- For  $-78$  °C. Note: en dash for minus sign, and space between 78 and °
- $[\alpha]_D^{26}$ :
  - See the web page for directions on creating/editing this symbol.
  - The number of sig. figs. in the conc. of the rotation should correspond to the sig. figs. in the rotation.
  - There are no units reported for the rotation.
- $R_f$ : note that the f is in italics and is subscripted.
- IR: You only need to list 6–8 diagnostic bands.
- $^1\text{H}$  NMR:
  - Chemical shifts are reported in highest to lowest.
  - Chemical shifts are reported to two decimal places.
  - *J* values are in hertz (Hz) and have one decimal place. The *J* is in italics.
  - There are spaces between the *J*, the equal sign, and the number (*J* = 7.2).
- $^{13}\text{C}$  NMR: Chemical shifts have one decimal point.
- Low res MS: You only need to have 8–10 peaks, and these should be the most intense with those of higher mass taking precedence. The  $\text{M}^+$  peak should be included if at all possible.
- HRMS: reported to four decimal places.