[1Hβ,6Hβ]-7α-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) ν 1735, 1299, 1242, 1123, 1030 cm −1; 1H NMR (400 MHz, CDCl 3 ) δ 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, 1H), 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); 13C NMR (100 MHz, CDCl 3 ) δ 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 (CH 2 ), 26.7 (CH 3 ), 20.9 (CH 3 ); MS (EI) m/e (rel. intensity) 268 (M + , 0.3), 226 (3), 191 (8), 178 (31), 144 (70), 129 (100), 91 (93), 65 (29); HRMS calcd for C 11 H 14 O 3 S (M + –C 2 H 2 O) 226.0664, found 226.0665. Anal. calcd for C 13 H 16 O 4 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; \([\alpha]^{26}_D\) data (conc in g/100 mL, solvent); Rf data (solvent);

IR (neat or solvent) ν data cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) δ data; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) δ data (dept data); MS (EI) m/e (rel. intensity) data; HRMS calcd for

C\(_{11}\)H\(_{14}\)O\(_3\)S (M\(^+\)) X.XXXX, found X.XXXX. Anal. calcd for C\(_{13}\)H\(_{16}\)O\(_4\)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, Rf and \([\alpha]^{26}_D\) do not have equal ‘=’ signs.
- For −78 °C. Note: en dash for minus sign, and space between 78 and °
- \([\alpha]^{26}_D\):
  - 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.
- Rf: note that the f is in italics and is subscripted.
- IR: You only need to list 6–8 diagnostic bands.
- \(^1\)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 (\(J = 7.2\)).
- \(^{13}\)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 M\(^+\) peak should be included if at all possible.
- HRMS: reported to four decimal places.