Enantioselective Total Synthesis of Decytospolide A and Decytospolide B
Using an Achmatowicz Reaction

Arun K. Ghosh,* Hannah M. Simpson, and Anne Veitschegger
Department of Chemistry and Department of Medicinal Chemistry, Purdue University, 560 Oval Drive, West Lafayette, Indiana, 47907, USA

Table of Contents: Page:

General experimental conditions..............................................S1

$^1$H and $^{13}$C NMR Spectra of Reported Compounds..............................S2-S16

General experimental conditions
Chemicals and reagents were purchased from commercial suppliers and used without further purification. Anhydrous solvents were obtained as follows: dichloromethane and toluene from calcium hydride, diethyl ether and tetrahydrofuran from sodium/benzophenone. All other solvents were reagent grade. All moisture-sensitive reactions were either carried out in flame- or oven-dried (120 °C) glassware under an argon atmosphere. TLC analysis was conducted using glass-backed thin-layer silica gel chromatography plates (60 Å, 250 μm thickness, F254 indicator). Column chromatography was performed using silica gel, 230-400 mesh, 60 Å pore diameter. $^1$H and $^{13}$C NMR spectra were recorded on either Bruker ARX400, Bruker DRX-500, Bruker AV500HD. Chemical shift (δ values) are reported in parts per million and are referenced to the residual solvent signal (CDCl$_3$ $^1$H singlet = 7.26, $^{13}$C triplet = 77.16). Characteristic splitting patterns due to spin-spin coupling are identified as follows: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sep = septet, m = multiplet, dd = doublet of doublets, ddd = doublet of doublet of doublets, td = triplet of doublets, dq = doublet of quartets, brs = broad singlet, app = apparent. All coupling constants are measured in hertz (Hz). Optical rotations were recorded by a Perkin Elmer 341 polarimeter. LRMS and HRMS spectra were recorded at the Purdue University Department of Chemistry Mass Spectrometry Center. HPLC data was obtained on an Agilent 1290 Infinity II.
$^1$H NMR (500 MHz, CDCl$_3$) of Acetate 12

$^{13}$C NMR (125 MHz, CDCl$_3$) of Acetate 12
$^1$H NMR (500 MHz, CDCl$_3$) of Furanyl ketone 13

$^{13}$C NMR (125 MHz, CDCl$_3$) of Furanyl ketone 13
$^1$H NMR (500 MHz, CDCl$_3$) of Alcohol 10

$^{13}$C NMR (125 MHz, CDCl$_3$) of Alcohol 10
$^1$H NMR (500 MHz, CDCl$_3$) of tetrahydropyran 8

$^{13}$C NMR (125 MHz, CDCl$_3$) of tetrahydropyran 8
$^1$H NMR (500 MHz, CDCl$_3$) of Ketone 15

$^{13}$C NMR (125 MHz, CDCl$_3$) of Ketone 15
$^1$H NMR (400 MHz, CDCl$_3$) of Ketone 18

$^{13}$C NMR (100 MHz, CDCl$_3$) of Ketone 18
$^1$H NMR (400 MHz, CDCl$_3$) of Alcohol 19

$^{13}$C NMR (100 MHz, CDCl$_3$) of Alcohol 19
$^{1}H$ NMR (400 MHz, CDCl$_3$) of Alcohol 20

$^{13}C$ NMR (100 MHz, CDCl$_3$) of Alcohol 20
\(^1\text{H NMR (800 MHz, CDCl}_3\text{)}\) of Amide 21

\[^{13}\text{C NMR (200 MHz, CDCl}_3\text{)}\) of Amide 21
$^1$H NMR (500 MHz, CDCl$_3$) of Decytospolide A, 6

$^{13}$C NMR (125 MHz, CDCl$_3$) of Decytospolide A, 6
$^1$H NMR (500 MHz, CDCl$_3$) of Decytospolide B, 7

$^{13}$C NMR (125 MHz, CDCl$_3$) of Decytospolide B, 7
NMR (500 MHz, CDCl$_3$) of Synthetic (top) and Natural (bottom) Decytospolide A, 6
$^{13}$C NMR (125 MHz, CDCl$_3$) of Synthetic (top) and Natural (bottom) Decytospolide A, 6
$^1$H NMR (500 MHz, CDCl$_3$) of Synthetic (top) and Natural (bottom) Decytospolide B, 7
$^{13}$C NMR (125 MHz, CDCl$_3$) of Synthetic (top) and Natural (bottom) Decytospolide B, 7