Supporting Information for
Tandem Dihydroxylation / Hemiketalization / Conjugate Addition Leading to a Singly Anomeric Spiroketal
Synthesis

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NMR Spectra for Synthesized Compounds
$^1$H NMR Spectrum for 9:

$^{13}$C NMR Spectrum for 9:
$^1$H NMR Spectrum for 10:

$^{13}$C NMR Spectrum for 10:
$^1$H NMR Spectrum for 11:

$^{13}$C NMR Spectrum for 11:
$^1$H NMR Spectrum for 12:

$^{13}$C NMR Spectrum for 12:
$^1$H NMR Spectrum for 13:

$^{13}$C NMR Spectrum for 13:
$^1$H NMR Spectrum for *trans*-14 (major observed diastereomer):

$^{13}$C NMR Spectrum for *trans*-14 (major observed diastereomer):
$^1$H NMR Spectrum for *trans*-14 (mixture of minor diastereomers):

$^1$H NMR Spectrum for *cis*-14 (major observed diastereomer):
$^1$H NMR Spectrum for *cis*-5:

$^1$H NMR Spectrum for a mixture of *cis*-5 (one diastereomer) and *trans*-5 (mixture of two atropisomers):
$^{13}$C NMR Spectrum for a mixture of *cis*-5 (one diastereomer) and *trans*-5 (mixture of two atropisomers):

DEPT-135 NMR Spectrum for a mixture of *cis*-5 (one diastereomer) and *trans*-5 (mixture of two atropisomers):
$^1$H NMR Spectrum for 4:

$^{13}$C NMR Spectrum for 4:
$^1$H NMR Spectrum for 7a:

![NMR Spectrum Image]

1D TOCSY Spectra for 7a:

![TOCSY Spectrum Images]
1D NOE NMR Spectra for 7a:

1D NOE Spectrum for 7a
irradiating from 3.00 to 3.78 ppm

1D NOE Spectrum for 7a
irradiating from 4.05 to 3.94 ppm
HSQC NMR Spectrum for 7a:
**$^1$H NMR Spectrum for 17:**

![1H NMR Spectrum](image)

**$^{13}$C NMR Spectrum for 17:**

![13C NMR Spectrum](image)
$^1$H NMR Spectrum for 18:

1D TOCSY NMR Spectra for 18:

1D TOCSY Spectrum for 18
irradiating from 4.75 to 4.73 ppm

1D TOCSY Spectrum for 18
irradiating from 4.84 to 4.82 ppm
1D NOE NMR Spectra for 18:

1D NOE Spectrum for 18
irradiating from 4.77 to 4.70 ppm

1D NOE Spectrum for 18
irradiating from 4.86 to 4.79 ppm