Supporting Information for the Paper “A Versatile Stereocontrolled Approach to Chiral Tetrahydrofuran and Tetrahydropyran Derivatives by Use of Sequential Asymmetric Horner-Wadsworth-Emmons and Ring Closure Reactions”

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NMR data for compounds **41** and **42**

The absolute configurations of compounds **15a** and **17a** were assigned on the basis of NMR analyses¹ of the corresponding Mosher ester derivatives **41** and **42**.

¹H NMR data (250 MHz) for MTPA-esters **41**² derived from **20a**:

	(<i>S</i>)- 41	(<i>R</i>)- 41	$\Delta\delta = \delta(S) - \delta(R)$	
Proton	δ (ppm)	δ (ppm)	$\Delta\delta$ (ppm)	$\Delta\delta$ (Hz)
1a	4.367	4.286	+0.081	+20.3
1b	4.055	4.033	+0.022	+5.5
5	5.204	5.280	-0.076	-19.0
6	6.384	6.446	-0.062	-15.5
7	5.344	5.388	-0.044	-11.0

¹H NMR data (250 MHz) for MTPA-esters **42**² derived from **23a**:

	(<i>S</i>)- 42	(<i>R</i>)- 42	$\Delta\delta = \delta(S) - \delta(R)$	
Proton	δ (ppm)	δ (ppm)	$\Delta\delta$ (ppm)	$\Delta\delta$ (Hz)
1a	4.333	4.417	-0.084	-21.0
1b	4.083	4.111	-0.028	-7.0
5	6.053	6.026	+0.027	+6.8
6	5.784	5.709	+0.075	+18.8
7	5.034	5.015	0.019	+4.8

- (1) For a description of this method, see: (a) Ohtani, I.; Kusumi, T.; Kashman, Y.; Kakisawa, H. *J. Am. Chem. Soc.* **1991**, *113*, 4092. See also: (b) Dale, J. A.; Mosher, H. S. *J. Am. Chem. Soc.* **1973**, *95*, 512.
- (2) The notations (*S*) and (*R*) refer to the stereocenter of the Mosher acid unit.

Preparation of, and analytical data for, compound **43**

In order to prove the stereochemical assignment of compound **30**, it was converted to **43** in four straightforward steps.

According to ^1H and ^{13}C analysis, **43** is a *meso*-compound (and not pseudo- C_2 -symmetric) which implies that the stereochemistry of **30** is the one shown. **43**: ^1H NMR (250 MHz) δ 7.37-7.27 (m, 5H), 4.58 (app s, 2H), 4.12 (dd, $J = 11.6, 4.5$ Hz, 2H), 4.06 (dd, $J = 11.6, 5.8$ Hz, 2H), 3.68-3.52 (m, 3H), 2.06 (br d, $J = 4.6$ Hz, 2H), 2.01 (br d, $J = 4.5$, 2H), 1.20 (s, 18H); ^{13}C NMR (62.9 MHz) δ 178.4, 138.2, 128.5, 127.7, 127.6, 73.9, 73.7, 69.8, 66.5, 38.8, 34.2, 27.2; IR 2963, 1731, 1285, 1148 cm^{-1} .

General NMR data for the 8-phenylmenthyl unit

Note: $\text{R}^* = (1R,2S,5R)$ -8-phenylmenthyl in all structures reported.

General NMR data for the 8-phenylmenthyl unit: ^1H NMR (500 MHz) δ 7.3-7.2 (m, 4H) 7.15-7.05 (m, 1H), 2.0-0.8 (m, 9H), 1.29 ± 0.05 (s, 3H), 1.21 ± 0.05 (s, 3H), 0.87 ± 0.05 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz) δ 151.7 ± 0.2 , 128.0 ± 0.1 , 125.4 ± 0.1 , 125.1 ± 0.1 , 74.5 ± 0.4 , 50.3 ± 0.2 , 41.7 ± 0.1 , 39.7 ± 0.2 , 34.5 ± 0.1 , 31.2 ± 0.1 , 28.1 ± 0.2 , 26.5 ± 0.1 , 24.7 ± 0.2 , 21.6 ± 0.1 .

The NMR signals for H1 (see figure below) vary more strongly depending on the exact structure of the compound, and are reported separately for each compound, together with other NMR data.

nOe NMR spectra for compounds **21** and **24**

Compounds **21** and **24**: The positive nOe between H-2 and H-5 in **21**, and the lack of a corresponding nOe in **24**, support the indicated assignments.

21

24

nOe NMR spectrum for compound 33

Compound **33**: The positive nOe between H-2 and H-4, and the lack of nOe between H-6 and H-2 as well as between H-6 and H-4, support the indicated relative configuration.

nOe NMR spectrum for compound **36a**

Compound **36a**: The positive nOe between H-2 and H-6, and the lack of nOe between H-5 and H-2 as well as between H-5 and H-6, support the indicated relative configuration.

nOe NMR spectrum for compound 40

Compound **40**: The positive nOe between H-2 and H-4, and the lack of nOe between H-6 and H-2 as well as between H-6 and H-4, support the indicated relative configuration.