Supporting information for

Synthesis and evaluation of 2-(2-fluoro-4-hydroxymethyl-5-methoxy-phenoxy) acetic acid as linker in solid-phase synthesis monitored by $^{19}$F gel-phase NMR spectroscopy

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X-ray crystallography

X-ray crystal structures were determined from data collected with a Nonius KappaCCD area detector diffractometer, using graphite monochromatized MoKα. Solution of the structures were made by direct methods and refinements on F2. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were isotropically refined on calculated positions (riding model).

Crystallization of 11 and 13

From a different batch both 11 and the dimer 13 were purified by preparative LC/MS. Crystallisation from heptane, for 11, or EtOH, for 13, gave thin needles. X-ray diffraction on the crystals showed the expected structures.

Figure 1. Single crystal X-ray structure of compound 11 (two crystallographical non-identical molecules), with displacement ellipsoids of non-hydrogen atoms drawn at the 50% probability level and hydrogen atoms of arbitrary size (ATOMS).

Figure 2. Single crystal X-ray structure of compound 13 with displacement ellipsoids of non-hydrogen atoms drawn at the 50% probability level and hydrogen atoms of arbitrary size (ATOMS). The compound has imposed twofold symmetry with the central oxygen on the twofold axis.
Table 1. Crystal data for 11 and 13.

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<th>13</th>
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Gel-phase $^{19}$F NMR spectra for substance 18, 21, 23, 26 and 27.

**Figure 3.** Gel-phase $^{19}$F NMR spectrum for 18.

**Figure 4.** Gel-phase $^{19}$F NMR spectrum for 21.
Figure 5. Gel-phase $^{19}$F NMR spectrum for 23.

Figure 6. Gel-phase $^{19}$F NMR spectrum for 26.
Figure 7. Gel-phase $^{19}$F NMR spectrum for 27.

References