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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Contents

X-ray chrystallography

X-ray crystal structures were determined from data collected with a Nonius KappaCCD area detector diffractometer, using graphite monochromatized MoK_{α}.[1] Solution of the structures were made by direct methods[2] and refinements on F^2.[3] 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.

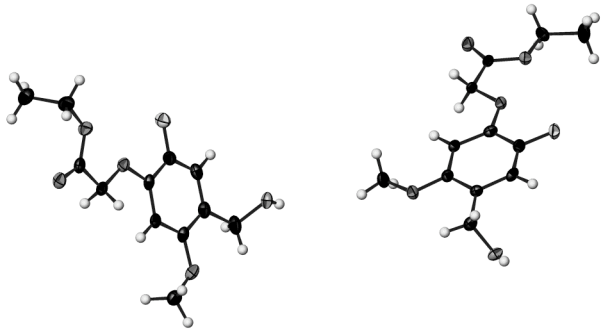

Figure 1. Single crystal X-ray structure of compound **11** (two crystallographical non-identical molecules), with displacement ellipsoids of non-hydrogen atoms drawn at the 50% probability level and hydrogen atoms of arbitrary size (ATOMS[4]).

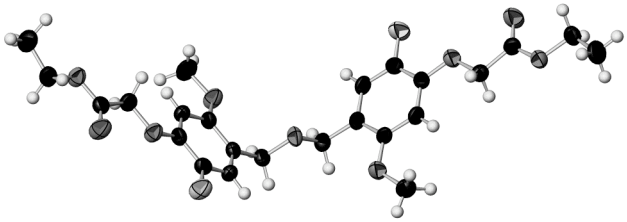

Figure 2. Single crystal X-ray structure of compound **13** with displacement ellipsoids of non-hydrogen atoms drawn at the 50% probability level and hydrogen atoms of arbitrary size (ATOMS[4]). The compound has imposed twofold symmetry with the central oxygen on the twofold axis.

Table 1. Crystal data for **11** and **13**.

Identification code	**11**	**13**
Empirical formula	C12 H15 F O5	C24 H28 F2 O9
Formula weight	258.245	498.477
Temperature of data collection	100K	298K
Wavelength	0.71073 Å	0.71073 Å
Crystal system	Triclinic	Orthorhombic
Space group	P-1	P b c n
Unit cell dimensions	a = 4.7854(6) b = 11.316(2) c = 22.705(3) α = 80.348(8) β = 86.525(10) γ = 86.318(7)	a = 13.7696(5) b = 8.3551(2) c = 20.4951(6)
Volume	1208.0(3)	2357.89(12)

Gel-phase ^{19}F NMR spectra for substance 18, 21, 23, 26 and 27.

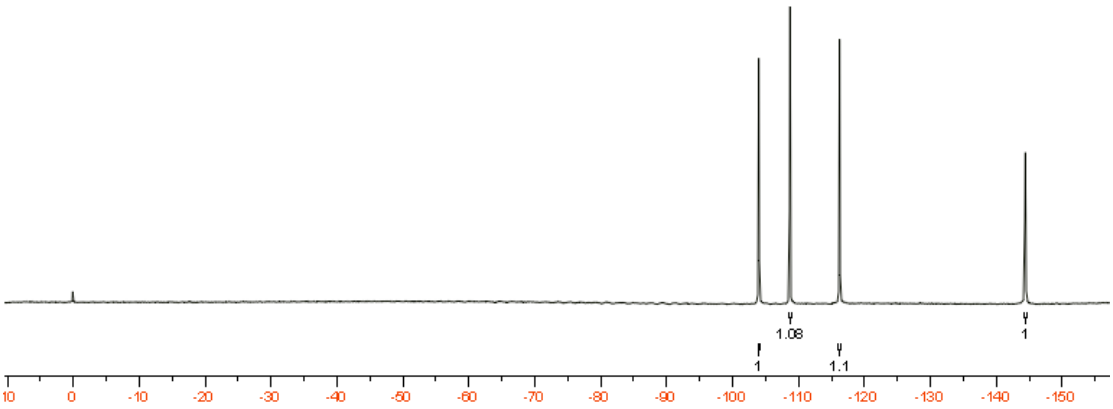

Figure 3. *Gel-phase ^{19}F NMR spectrum for* ***18***.

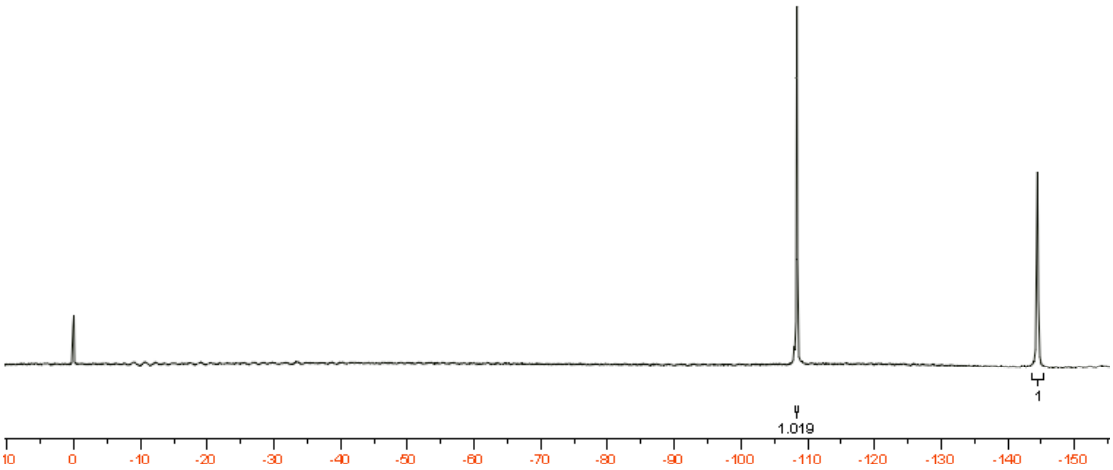

Figure 4. *Gel-phase ^{19}F NMR spectrum for* ***21***.

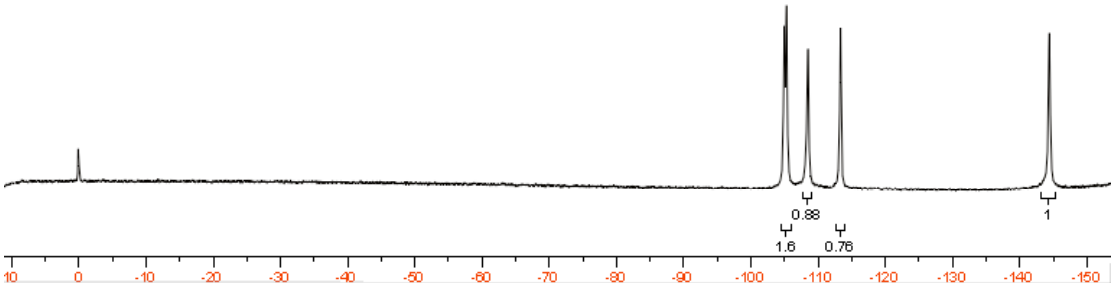

Figure 5. *Gel-phase ^{19}F NMR spectrum for* ***23***.

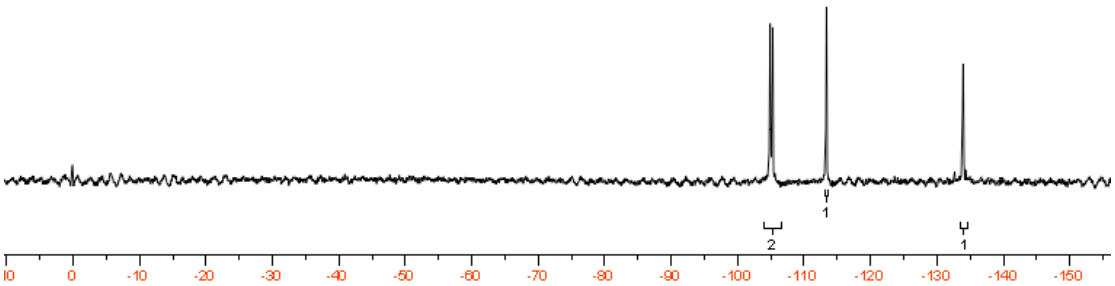

Figure 6. *Gel-phase ^{19}F NMR spectrum for* ***26***.

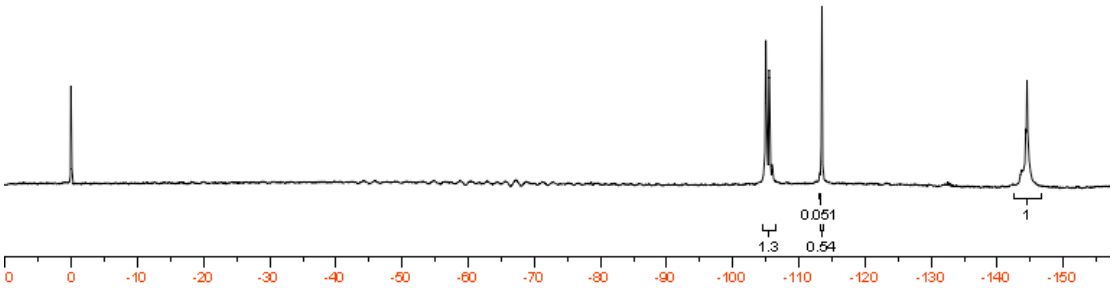

***Figure** 7. Gel-phase ^{19}F NMR spectrum for **27**.*

References

1. Nonius., *COLLECT*, (1999) Nonius BV, Delft, The Netherlands.
2. A. Altomare, M. C. Burla, M. Camalli, G. L. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori and R. Spagna, *J. Of Appl. Cryst.*, 1999, **32**, 115-119.
3. G. M. Sheldrick, *SHELXL97*, (1997) University of Göttingen, Göttingen, Germany.
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