

Synthesis of the C19 Methyl Ether of Aspercyclide A via Germyl-Stille Macrocyclisation and ELISA Evaluation of Both Enantiomers Following Optical Resolution

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Supporting Information — X-Ray Crystallography

Crystal data for 27: C₃₂H₂₃F₁₇Ge, *M* = 803.09, triclinic, *P* $\bar{1}$ (no. 2), *a* = 6.09459(14), *b* = 7.56496(17), *c* = 33.7307(8) Å, α = 84.8926(19), β = 87.4794(19), γ = 88.9974(18)°, *V* = 1547.35(6) Å³, *Z* = 2, *D*_c = 1.724 g cm⁻³, μ (Cu-K α) = 2.527 mm⁻¹, *T* = 173 K, colourless needles, Oxford Diffraction Xcalibur PX Ultra diffractometer; 5920 independent measured reflections (*R*_{int} = 0.0312), *F*² refinement, *R*₁(obs) = 0.0517, *wR*₂(all) = 0.1415, 4961 independent observed absorption-corrected reflections [*|F*_o| > 4 σ (*|F*_o)], 2 θ _{max} = 143°, 494 parameters. CCDC 814148.

The terminal –C₃F₇ section of the –C₈F₁₇ chain in the structure of **27** was found to be disordered. Three orientations were identified of *ca.* 38, 31 and 31% occupancy, their geometries optimised, and all of the atoms refined isotropically. The Ge–H proton was located from a ΔF map and refined freely subject to a Ge–H distance constraint of 1.60 Å.

Figures

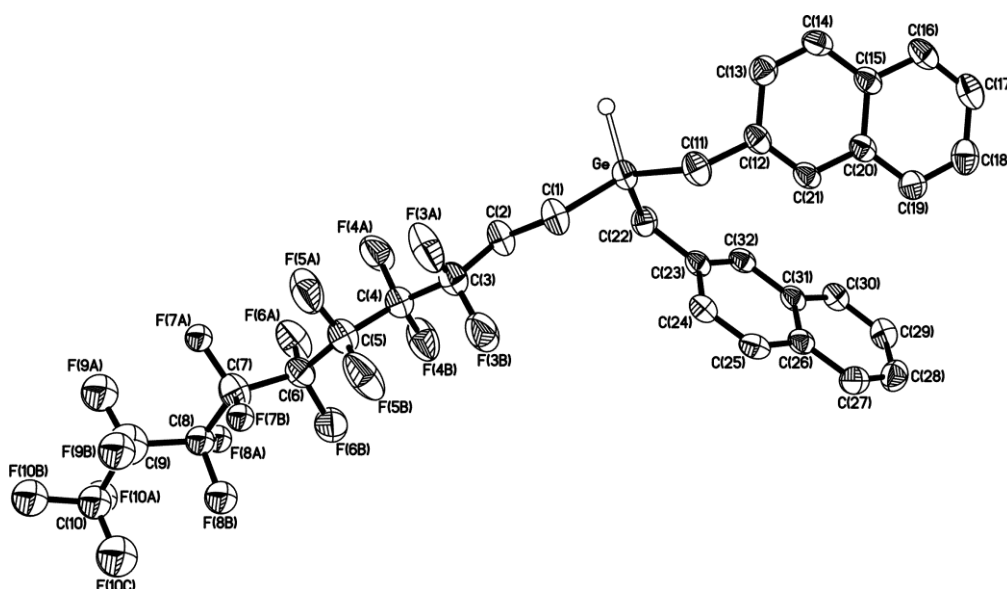


Fig. S1 The molecular structure of **27** (50% probability ellipsoids).