

Supporting Information

for

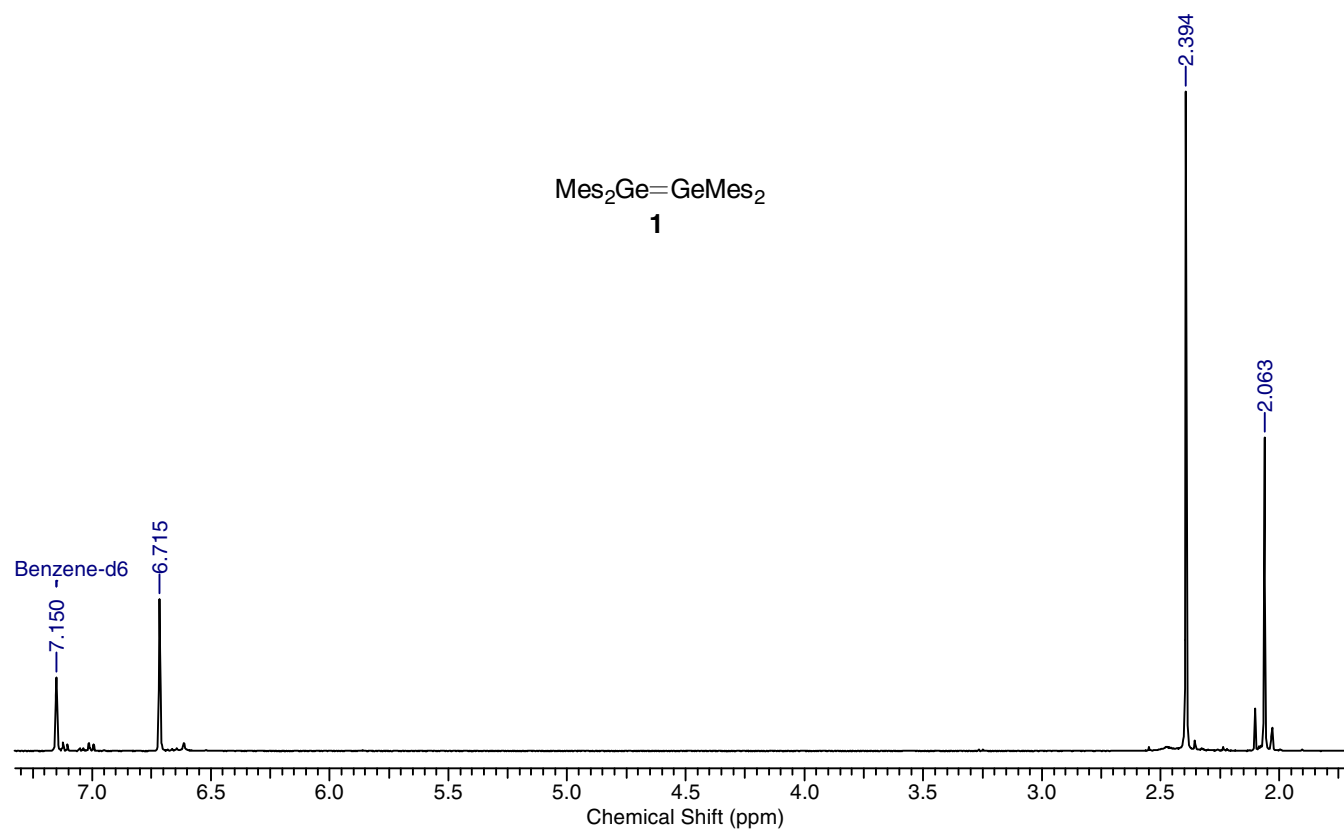
On the Synthesis, Structure, and Reactivity of Tetramesityldigermene

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Fig. S1 ^1H NMR spectrum of tetramesityldigermene (**1**).



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Fig. S2 ^1H NMR spectrum of **8a**.

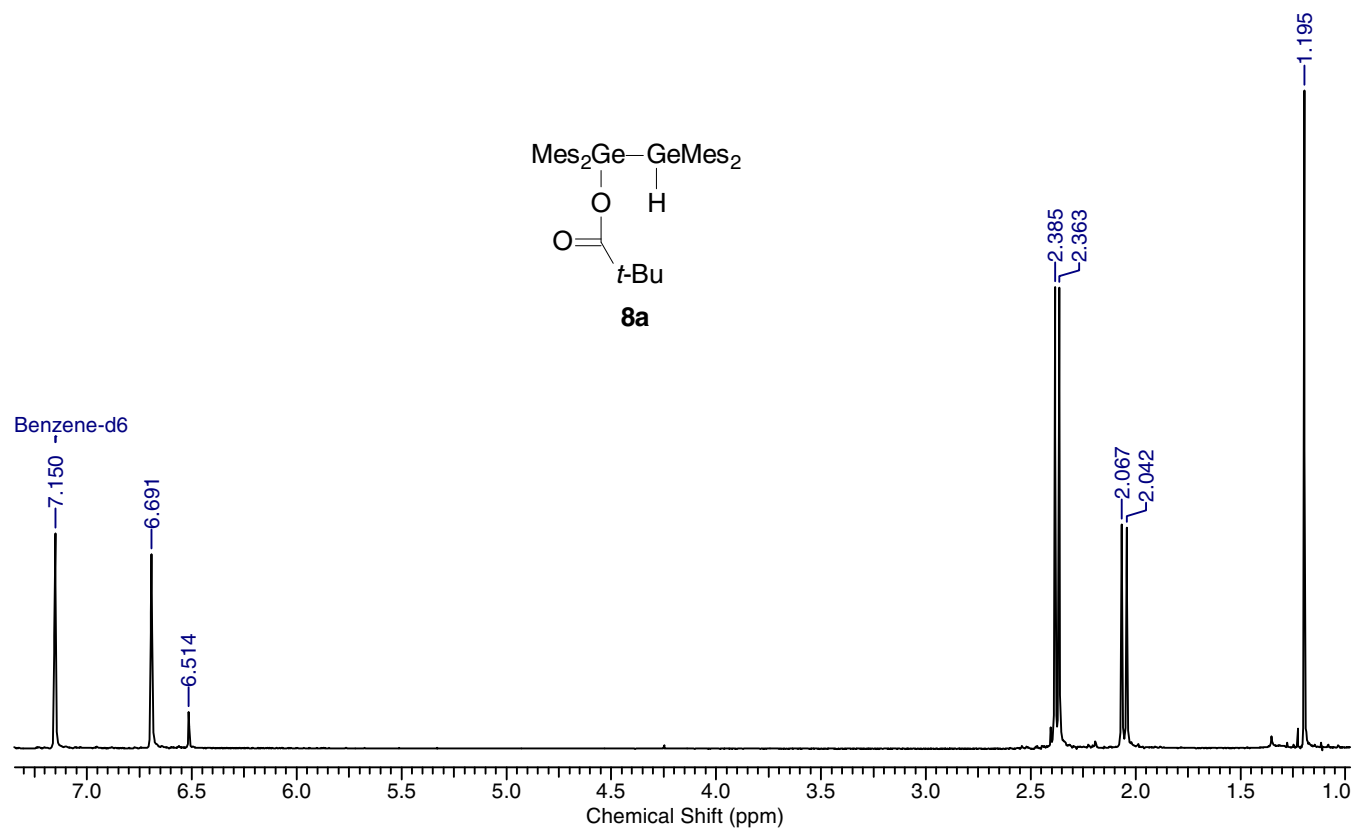


Fig. S3 ^1H NMR spectrum of **8b**.

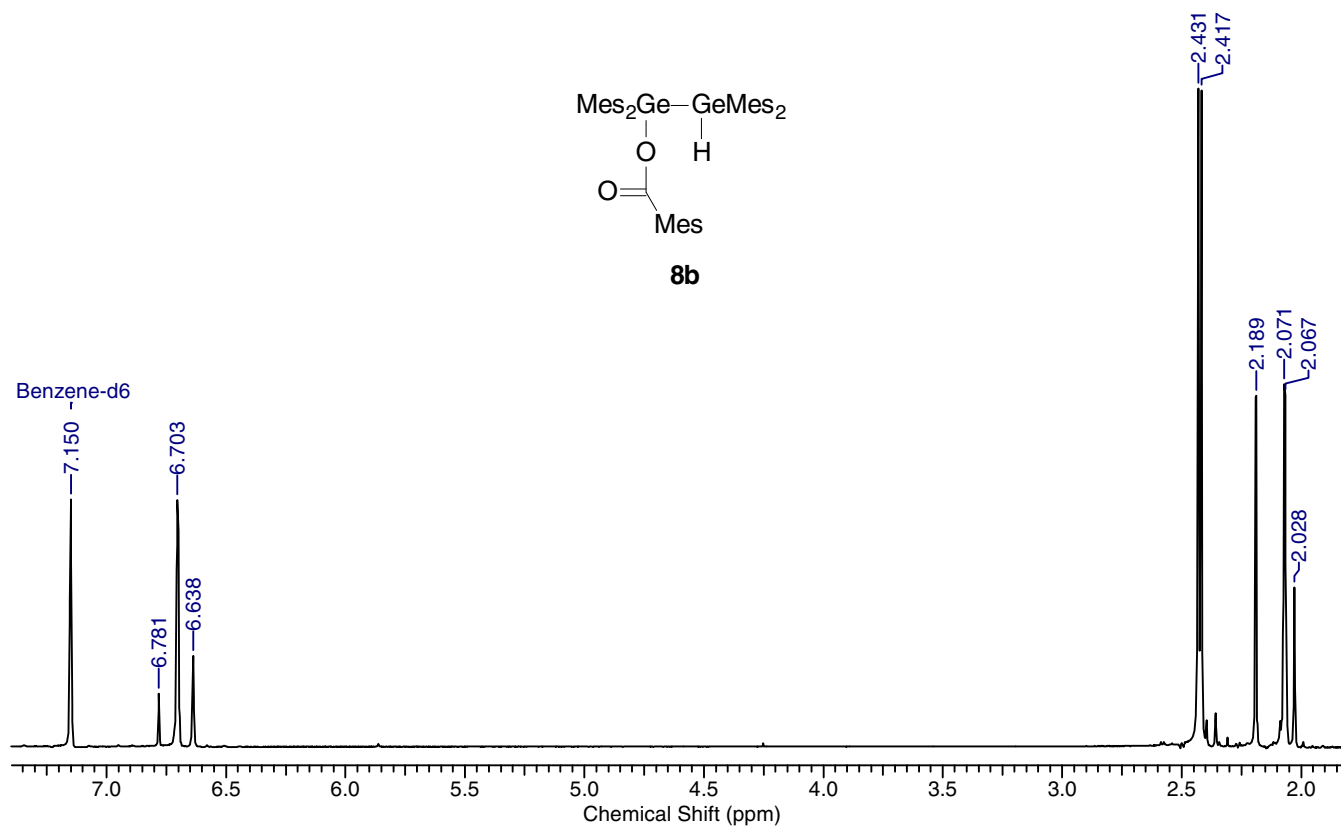


Fig. S4 Expansion of the ^1H NMR spectrum of **8b**.

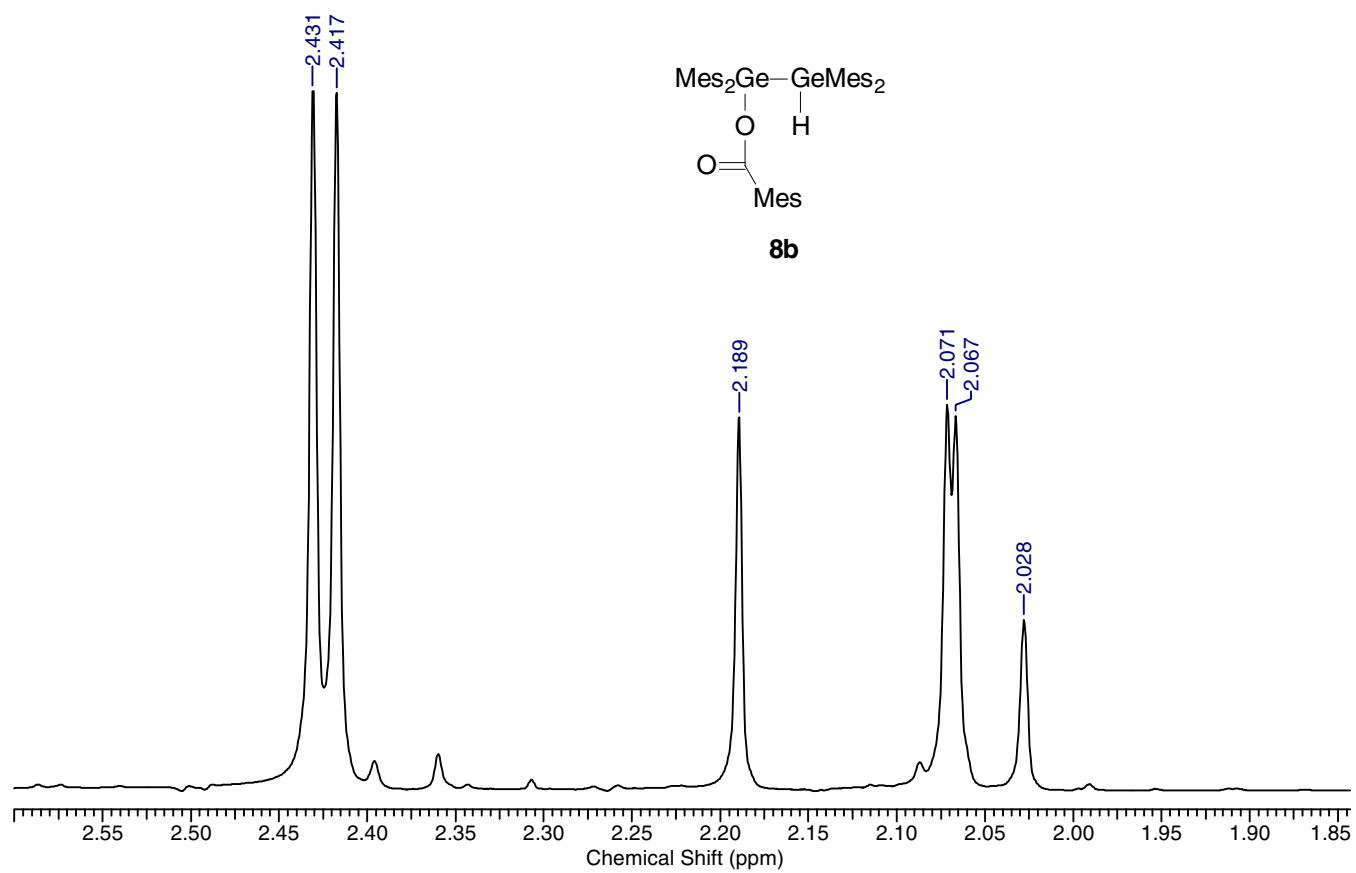


Fig. S5 ^1H NMR spectrum of **8c**.

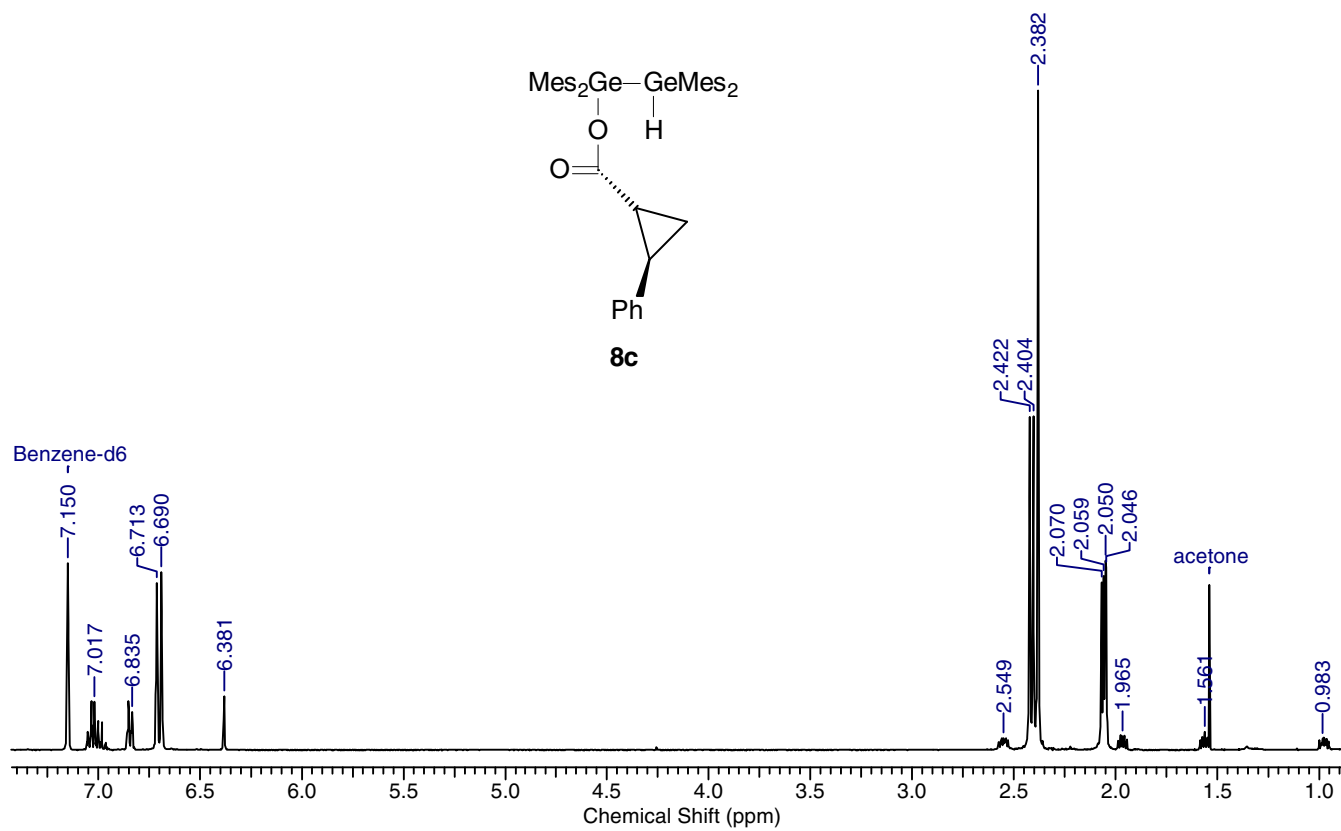


Fig. S6 Expansion of the ^1H NMR spectrum of **8c**.

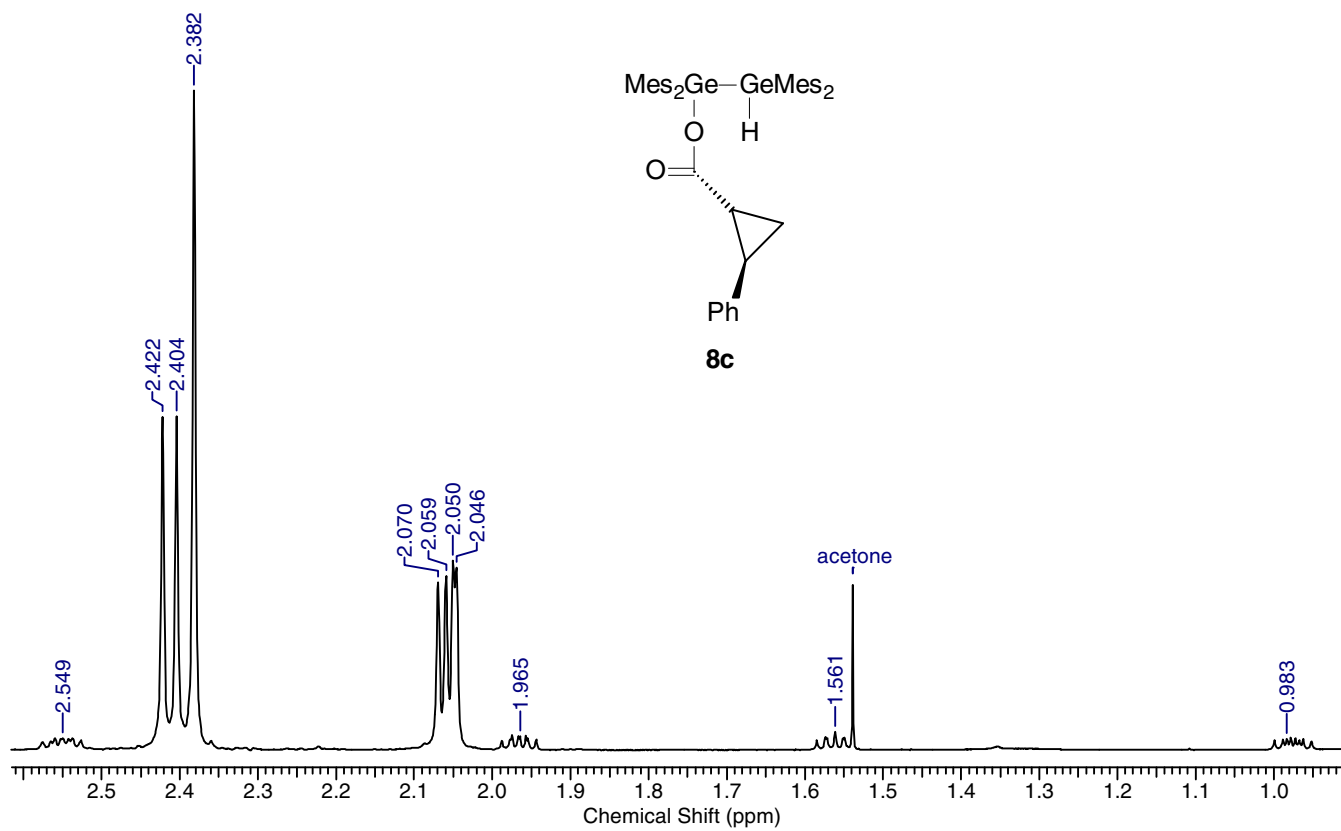
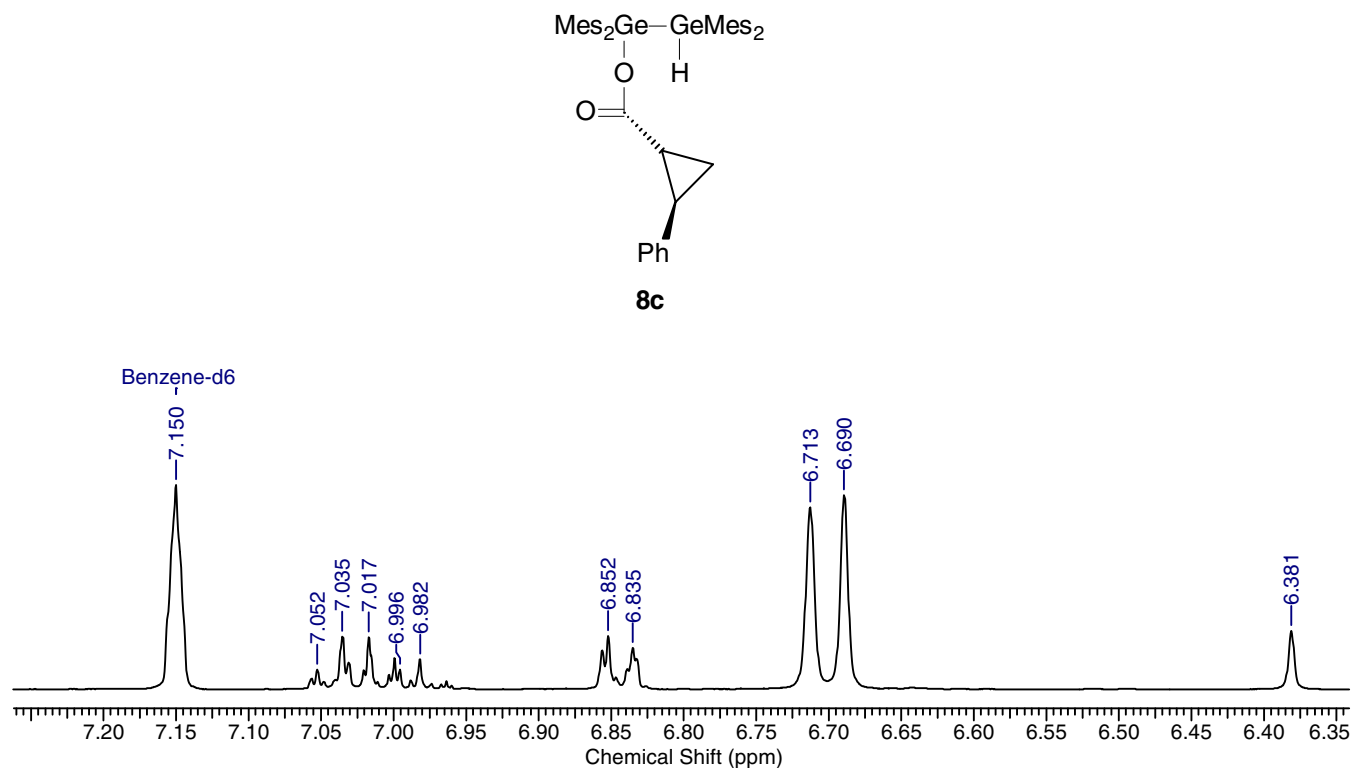


Fig. S7 Expansion of the ^1H NMR spectrum of **8c**.



X-Ray Experimental Details

1: Crystals of **1** were grown during the photolysis of a saturated solution of hexamesitylcyclotrigermane (**2**) in THF at $-70\text{ }^{\circ}\text{C}$. A yellow block was coated in heavy paraffin oil, mounted on a nylon loop, and transferred to an Oxford Cryosystems cold stream. Data were collected at low temperature (150(2) K) on an Enraf-Nonius Kappa CCD area detector diffractometer with COLLECT (Nonius B.V., 1997-2002). The unit cell parameters were calculated and refined from the full data set. Crystal cell refinement and data reduction was carried out using HKL2000 DENZO-

SMN.¹ The absorption correction was applied using HKL2000 DENZO-SMN (SCALEPACK). The reflection data were consistent with an orthorhombic space group: Pbcn.

The SHELXTL/PC V6.14 for Windows NT (Sheldrick, G.M., 2001) suite of programs was used to solve the structure by direct methods. Subsequent difference Fourier syntheses allowed the remaining atoms to be located. With a molecule of tetrahydrofuran solvent present, and a Z value of 4, 2-fold symmetry is imposed upon the Ge species, passing through the mid-point of the Ge=Ge bond. A study of a related digermene molecule has been made in the same space group with the same 2-fold symmetry imposed.² Disorder is commonly observed for tetrahydrofuran, and is required in this space group. The tetrahydrofuran molecule was successfully modeled as the δ conformer, disordered by a 2-fold rotation through the mid-point of a C—O bond.³ All non-hydrogen atoms on the digermene were refined with anisotropic thermal parameters. Non-hydrogen atoms in the disordered THF molecule were refined with isotropic thermal parameters. Hydrogen atoms associated with carbon were calculated geometrically and were included as riding on their respective carbon atoms.

The largest residual electron density peak ($2.31\text{e}/\text{\AA}^3$) was associated with the germanium. Full-matrix least squares refinement on F^2 gave $R_1 = 5.65$ for 2σ data and $wR_2 = 17.78$ for all data (GOOF = 1.058). The disordered THF is responsible for the Alert level B in the IUCR checkCIF basic structural check.

8a: Crystals of **8a** were grown by slow evaporation of a concentrated C_6D_6 solution. A colorless block was mounted on a glass fiber and transferred to an Oxford Cryosystems cold stream. Data were collected at low temperature (150(2) K) on an Enraf-Nonius Kappa CCD area detector diffractometer with COLLECT (Nonius B.V., 1997-2002). The unit cell parameters were calculated and refined from the full data set. Crystal cell refinement and data reduction were carried out using HKL2000 DENZO-

SMN.¹ The absorption correction was applied using HKL2000 DENZO-SMN (SCALEPACK). The reflection data were consistent with a monoclinic space group: P2(1)/c.

The SHELXTL/PC V6.14 for Windows NT (Sheldrick, G.M., 2001) suite of programs was used to solve the structure by direct methods. Subsequent difference Fourier syntheses allowed the remaining atoms to be located. There was one distinct molecule in the asymmetric unit. The *t*-Bu group attached to the ester was disordered and was modeled using 6 carbons with 1/2 occupancy. The carbon atoms in the *t*-Bu group were restrained to have similar bond lengths and isotropic thermal parameters. All other non-hydrogen atoms were refined with anisotropic thermal parameters. Hydrogen atoms associated with carbon were calculated geometrically and were included as riding on their respective carbon atoms. The hydrogen atom attached to one of the germanium atoms was located during refinement.

The largest residual electron density peak ($0.896 \text{ e}/\text{\AA}^3$) was associated with the disordered *t*-Bu group. Full-matrix least squares refinement on F^2 gave $R_1 = 5.22$ for 2σ data and $wR_2 = 14.14$ for all data (GOOF = 1.043). The large isotropic thermal parameters for the disordered *t*-Bu group are responsible for the Alert level A in the IUCR checkCIF program.

References

- (1) Otwinowski, Z.; Minor, W. In *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography, Part A*. Carter Jr., C.W. and Sweet, R.M., Ed.; Academic Press: New York, **1997**; pp. 307.
- (2) Snow, J.T.; Murakami, S.; Masamune, S.; Williams, D.J. *Tetrahedron Lett.* **1984**, 25, 1491.
- (3) Luger, P.; Buschmann, J. *Angew. Chem. Int. Ed. Engl.* **1983**, 22, 410.