

Electronic Supplementary Information

Elusive 2*H*-1,2-Oxasiletes Through Reactions of an Isolable Dialkylsilylene with Diazocarbonyl Compounds

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1. Crystal structure determination

Single crystals of **2**, **3**, **8** and **10** suitable for X-ray analysis were obtained by the recrystallization from hexane. The X-ray diffraction data were collected on a Bruker Smart Apex CCD diffractometer with graphite monochromated Mo-K α radiation ($\lambda = 0.71073$ Å) using the ω - 2θ scan mode. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares methods using SHELX-2000.^{S1} All the structural data are deposited with CCDC. The reference numbers are: 1056197(**2**), 1056194(**3**), 1063129 (**8**), and 1405300(**10**).

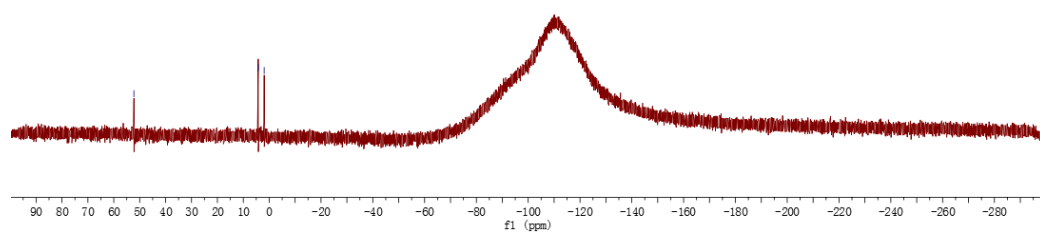
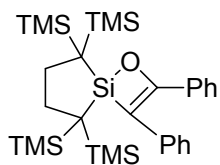
Table S1. Crystal and Refinement Data for **2**, **3**, **8**, and **11**.

Parameters	2	3	8	10
Empirical formula	C ₃₀ H ₅₀ OSi ₅	C ₂₆ H ₅₀ O ₂ Si ₅	C ₂₇ H ₅₀ N ₂ O ₃ Si ₅	C ₂₀ H ₅₀ N ₂ Si ₆
Formula weight	567.15	535.11	591.14	487.16
Crystal system, Space group	Triclinic, P-1	Orthorhombic, Pbca	Triclinic, P-1	Monoclinic, P2(1)/c
<i>a</i> [Å]	11.587(9)	9.661(10)	9.7059(11)	14.925(12)
<i>b</i> [Å]	12.882(10)	18.921(19)	12.3889(14)	11.801(8)
<i>c</i> [Å]	13.076(10)	35.70(4)	14.2286(16)	18.571(14)
α [deg]	89.350(9)	90	93.485(2)	90
β [deg]	65.871(9)	90	91.210(2)	110.765(16)
γ [deg]	74.849(9)	90	97.443(2)	90
<i>V</i> [Å ³]	1709(2)	6526(12)	1692.6(3)	3058(4)
<i>Z</i> , <i>D</i> _{calcd} [g cm ⁻³]	2, 1.102	8, 1.089	2, 1.160	4, 1.058
μ [mm ⁻¹]	0.229	0.238	0.240	0.283
<i>F</i> (000)	616	2336	640	1072
Reflections collected	8633	38614	20688	19805
Independent Reflections	5879	7451	7686	6967
<i>R</i> (int)	0.0153	0.1437	0.0277	0.0389
Data/restraints/parameters	5879 / 0 / 337	7451 / 0 / 311	7686 / 0 / 347	6967 / 0 / 268
final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	0.0407	0.0578	0.0418	0.0451
<i>R</i> _{<i>I</i>}				
<i>R</i> indices (all data) <i>wR</i> ₂	0.1422	0.1735	0.1423	0.1340

1zf-dw-2211-7
29si 1zf-dw-2211-7

-52.24

4.22
1.94



^{29}Si NMR spectrum of **2** in CDCl_3 .

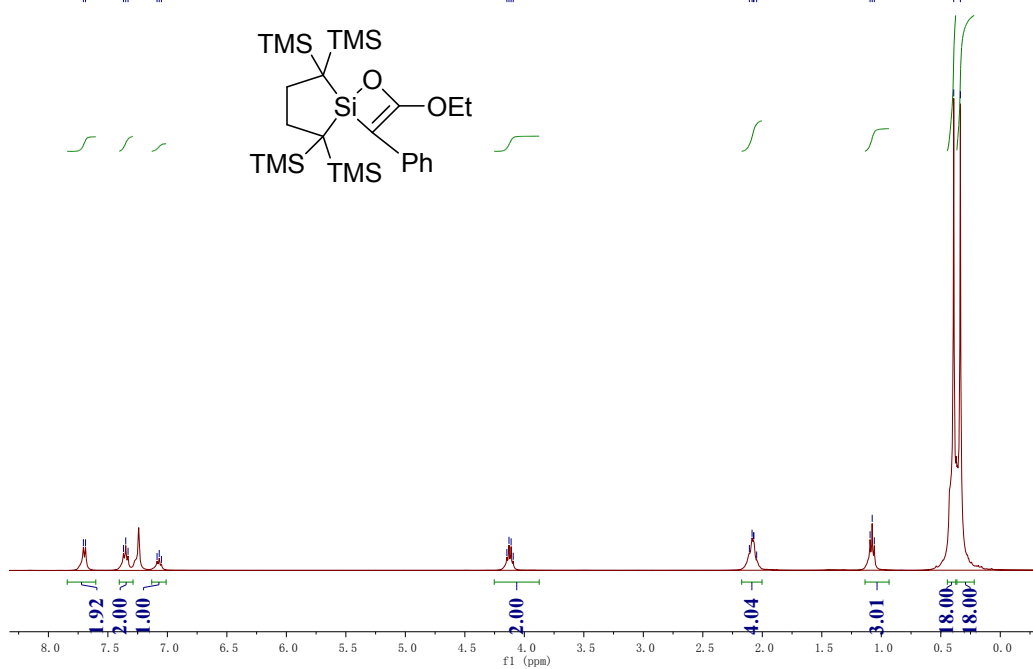
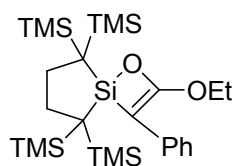
LZF-71
LZF-71
7.39
7.37
7.35
7.33
7.09
7.07
7.05

4.15
4.13
4.11
4.09

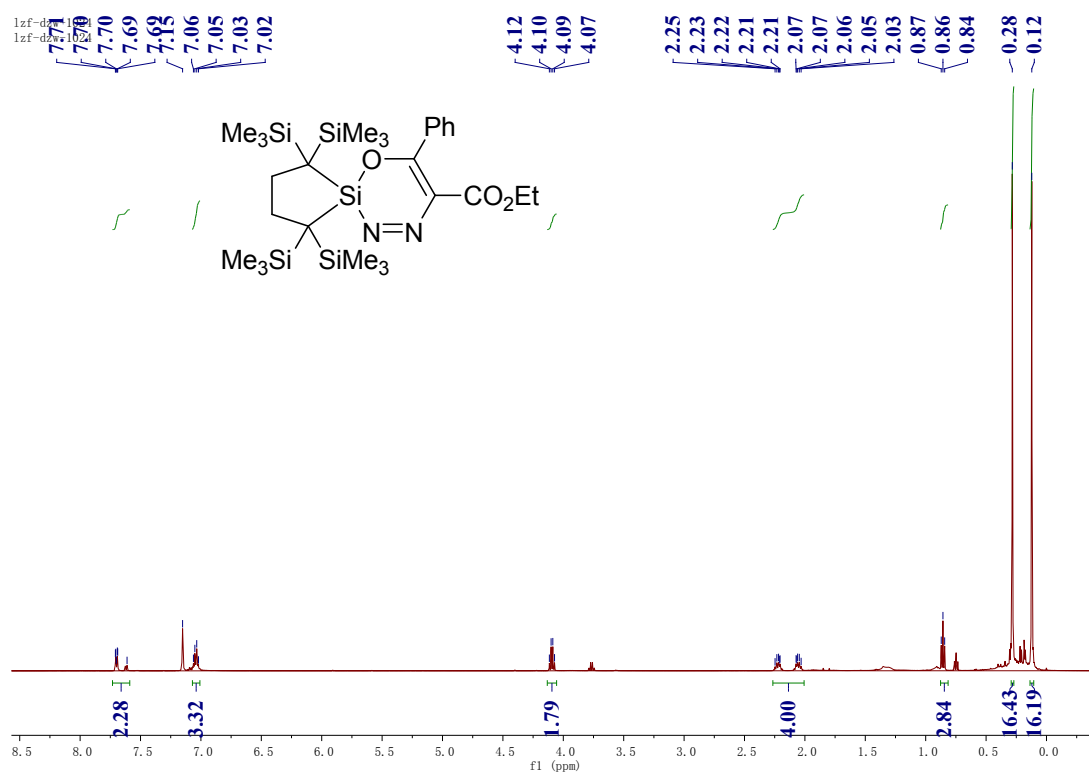
2.11
2.09
2.07
2.05

1.09
1.08
1.06

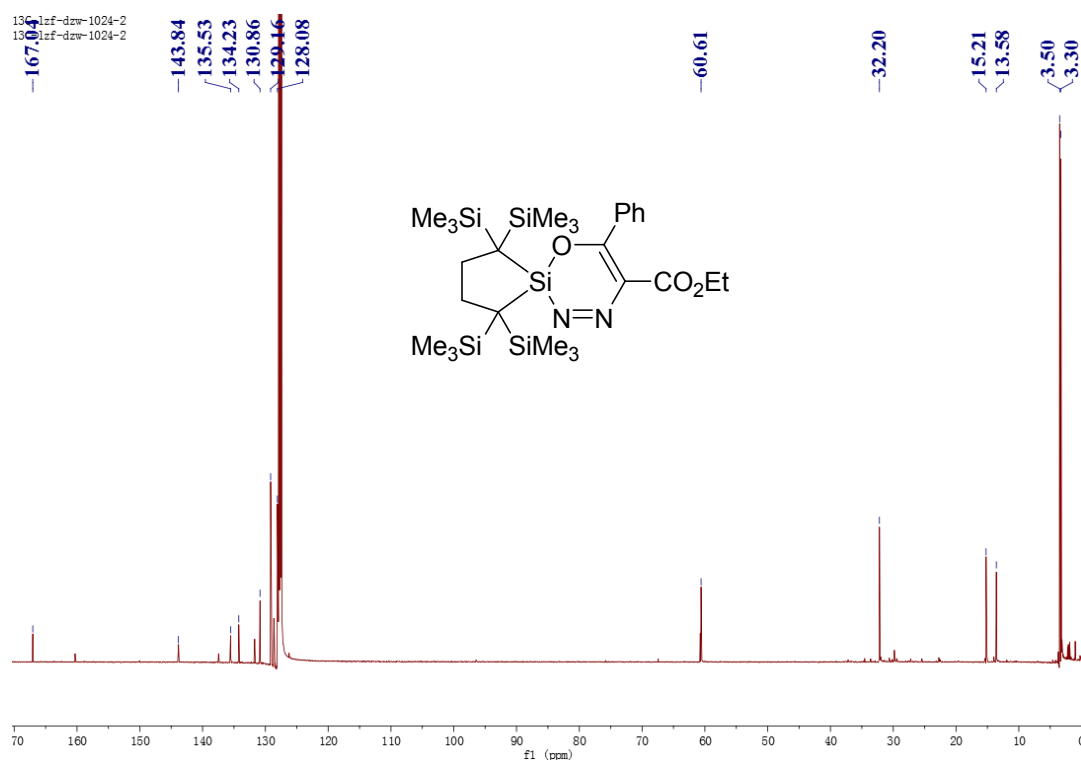
0.39
0.34



^1H NMR spectrum of **3** in CDCl_3 .



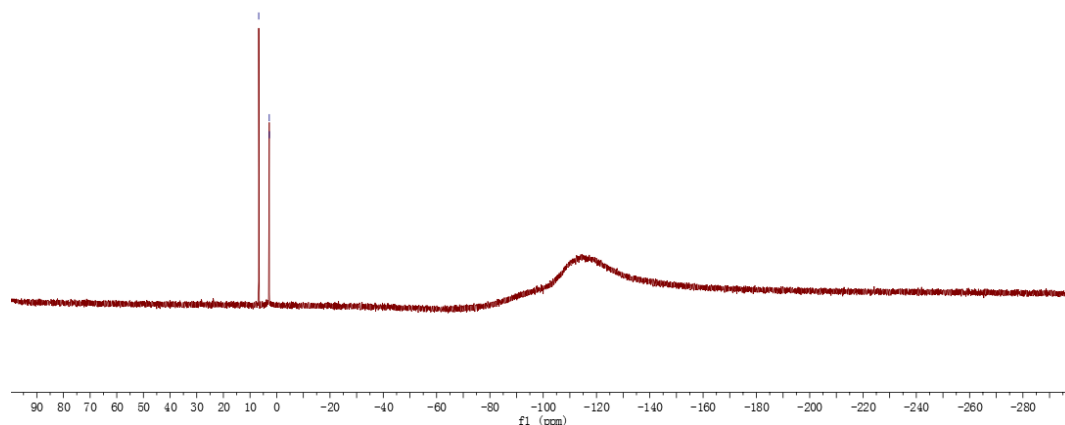
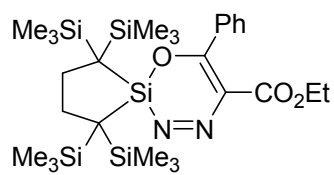
¹H NMR spectrum of **8** in C₆D₆.



¹³C NMR spectrum of **8** in C₆D₆.

29si lzf-dzw-1024
29si lzf-dzw-1024

6.74
2.78
2.75

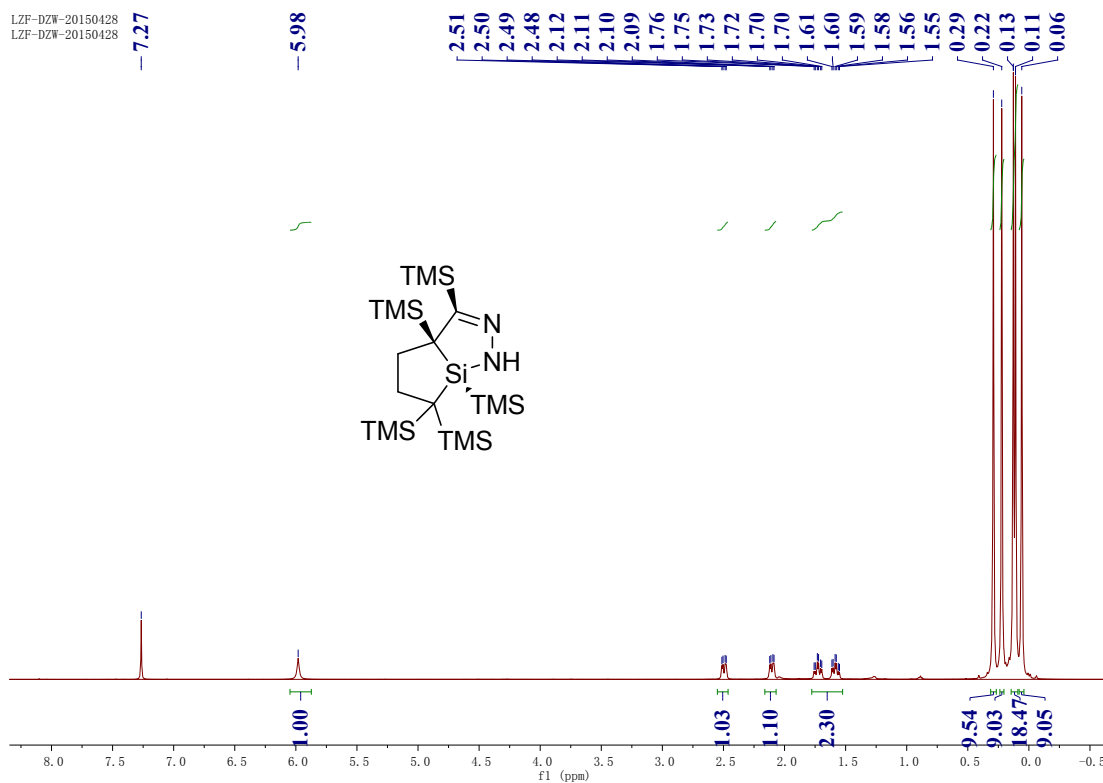
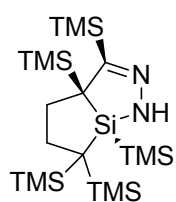


^{29}Si NMR spectrum of **8** in C_6D_6 .

LZF-DZW-20150428
LZF-DZW-20150428

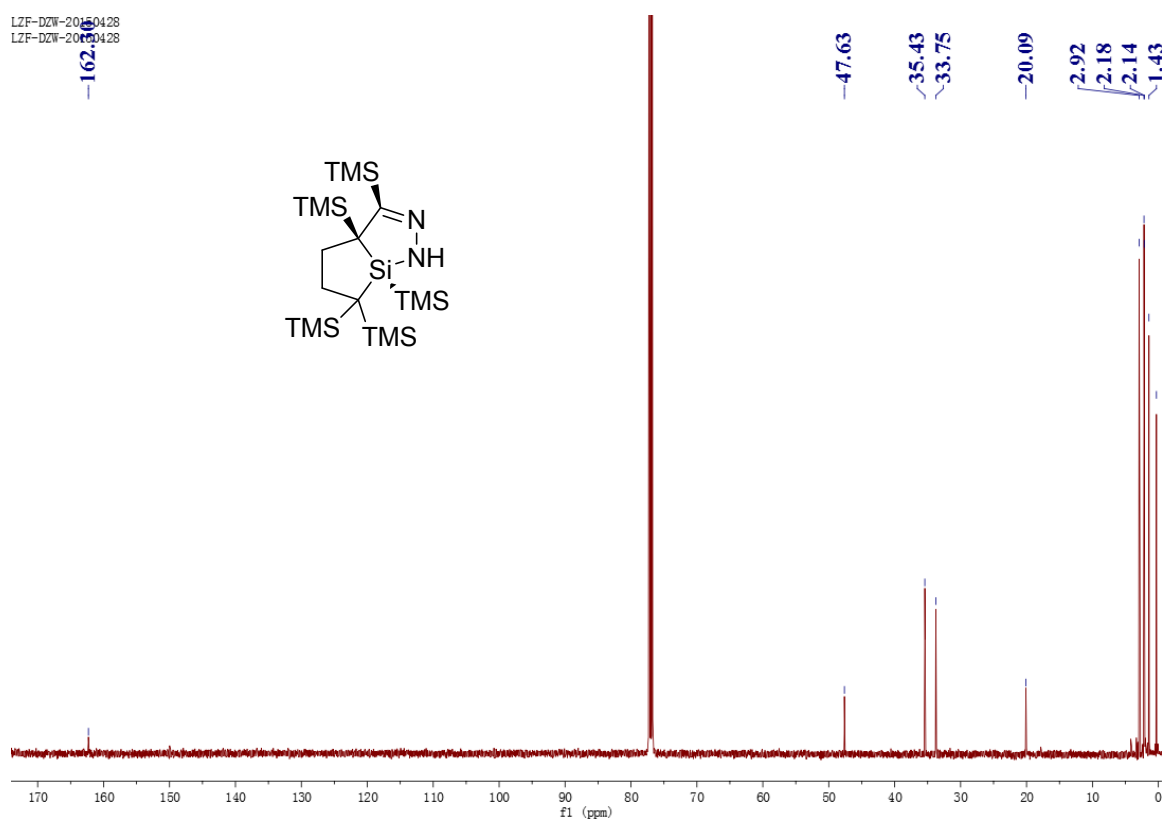
7.27

5.98



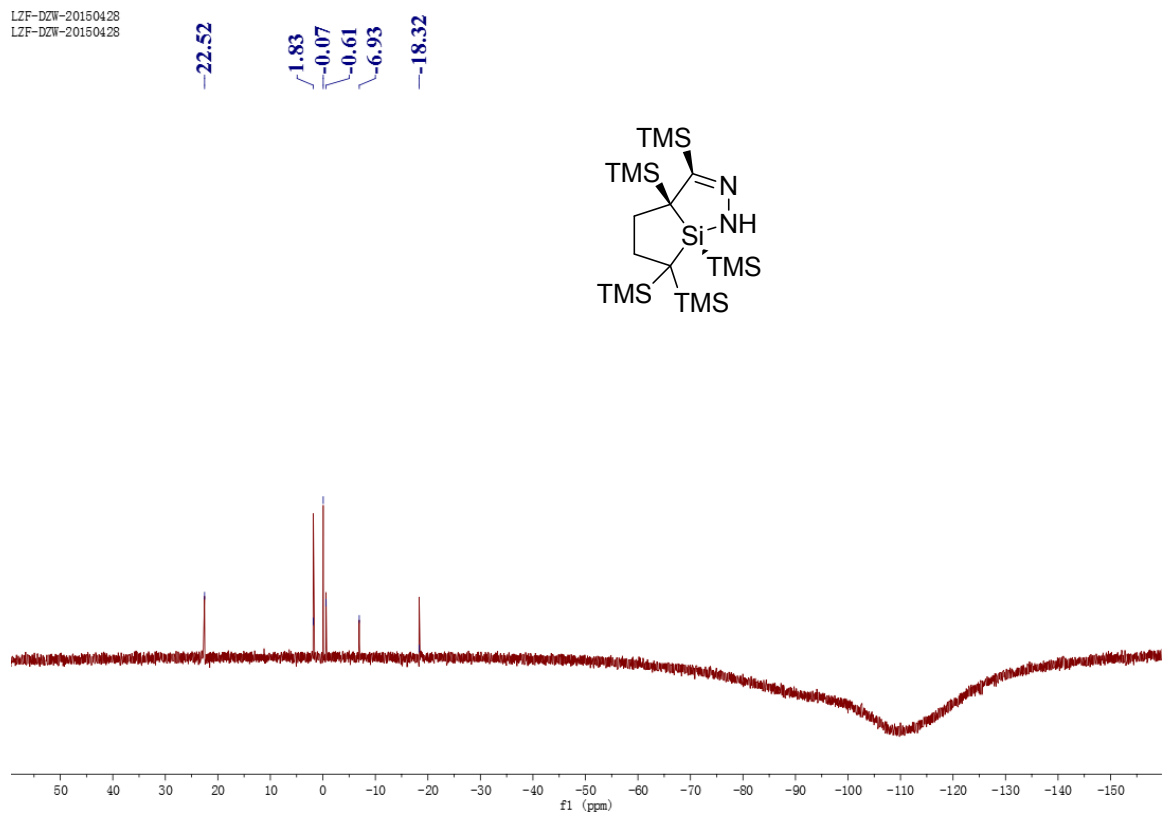
^1H NMR spectrum of **10** in CDCl_3 .

LZF-DZW-20150428
LZF-DZW-20150428



¹³C NMR spectrum of **10** in CDCl₃.

LZF-DZW-20150428
LZF-DZW-20150428



²⁹Si NMR spectrum of **10** in CDCl₃.

3. References

- (S1) (a) C. L. Picou, E. D. Stevens, M. Shah, J. H. Boyer, *Acta Crystallogr. Sect. C* 1990, **46**, 1148. (b) SMART, SAINT, SADABS and SHELXTL, Bruker AXS Inc., Madison, 2000.