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Synthesis of novel tricyclononenes containing alkoxysilyl groups

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1. Materials

Triethanolamine and vinyltrichlorosilane were purchased from Sigma-Aldrich and they were used as received without a preliminary purification. *n*-Hexane was distilled over Na in the argon atmosphere. Methanol was distilled over Mg activated by the trace of iodine. Quadricyclane was synthesized as reported previously.[1]

2. Methods of characterization

NMR spectra were recorded on a Bruker Avance DRX-500 spectrometer at 500.13 MHz (^{1}H NMR) and 125.76 MHz (^{13}C NMR). Each sample was dissolved in a solvent ($CDCl_3$) up to a concentration of 10%. Mass spectra were recorded on a Thermo Focus DSQ II (ionization energy 70 eV, source temperature 230°C). Differential scanning calorimetry (DSC) was performed on a Mettler TA4000 system at a heating rate 20°C/min.

X-ray analysis of 3.

Table S1 Crystal data and structure refinement for **3**.

Identification code	**3**	
Empirical formula	C15 H23 N O3 Si	
Formula weight	293.43	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 6.8473(5) Å	α= 90°.
	b = 16.3825(12) Å	β= 102.171(2)°.
	c = 13.2215(10) Å	γ = 90°.
Volume	1449.80(19) $Å^3$	
Z	4	
Density (calculated)	1.344 Mg/m^3	
Absorption coefficient	0.169 mm^{-1}	
F(000)	632	
Crystal size	0.350 x 0.150 x 0.150 mm^3	
Theta range for data collection	2.007 to 26.995°.	
Index ranges	-8<=h<=8, -20<=k<=20, -16<=l<=16	
Reflections collected	14848	
Independent reflections	3161 [R(int) = 0.0732]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8622 and 0.7615	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3161 / 0 / 181	
Goodness-of-fit on F^2	1.059	
Final R indices [I>2sigma(I)]	R1 = 0.0767, wR2 = 0.1787	
R indices (all data)	R1 = 0.1005, wR2 = 0.1936	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.141 and -0.342 e. $Å^{-3}$	

Table S2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Si(1)	1311(1)	3292(1)	1441(1)	12(1)
N(1)	-884(4)	2716(2)	219(2)	14(1)
O(1)	2932(4)	2848(2)	828(2)	19(1)
O(2)	195(4)	2721(2)	2199(2)	17(1)
O(3)	165(4)	4141(2)	941(2)	19(1)
C(1)	2421(6)	4407(3)	3187(3)	27(1)
C(2)	3215(5)	3789(2)	2491(3)	16(1)
C(3)	4478(5)	4472(2)	2110(3)	15(1)
C(4)	6751(6)	4513(3)	2607(3)	29(1)
C(5)	7352(6)	5370(3)	2288(4)	33(1)
C(6)	6532(7)	5912(3)	2789(4)	35(1)
C(7)	5420(6)	5443(2)	3511(3)	25(1)
C(8)	3587(6)	5109(2)	2761(3)	26(1)
C(9)	6783(6)	4709(3)	3727(3)	29(1)
C(10)	2338(6)	2444(2)	-133(3)	22(1)
C(11)	278(6)	2088(3)	-226(3)	28(1)
C(12)	-1313(6)	2141(2)	1814(3)	22(1)
C(13)	-2368(6)	2373(3)	729(3)	26(1)
C(14)	-1604(5)	4145(2)	157(3)	20(1)
C(15)	-1607(6)	3380(2)	-513(3)	27(1)

Table S3 Bond lengths [Å] and angles [°] for **3**.

Si(1)-O(3)	1.664(3)	O(1)-C(10)	1.415(4)
Si(1)-O(2)	1.669(2)	O(2)-C(12)	1.416(4)
Si(1)-O(1)	1.672(3)	O(3)-C(14)	1.418(4)
Si(1)-C(2)	1.878(3)	C(1)-C(2)	1.542(5)
Si(1)-N(1)	2.176(3)	C(1)-C(8)	1.570(6)
N(1)-C(13)	1.447(5)	C(1)-H(1A)	0.9900
N(1)-C(15)	1.469(5)	C(1)-H(1B)	0.9900
N(1)-C(11)	1.495(5)	C(2)-C(3)	1.562(5)

C(2)-H(2A)	1.0000	O(1)-Si(1)-C(2)	96.79(14)
C(3)-C(8)	1.557(5)	O(3)-Si(1)-N(1)	82.73(12)
C(3)-C(4)	1.558(5)	O(2)-Si(1)-N(1)	82.57(11)
C(3)-H(3A)	1.0000	O(1)-Si(1)-N(1)	82.94(12)
C(4)-C(9)	1.511(6)	C(2)-Si(1)-N(1)	179.71(15)
C(4)-C(5)	1.546(6)	C(13)-N(1)-C(15)	115.0(3)
C(4)-H(4A)	1.0000	C(13)-N(1)-C(11)	113.6(3)
C(5)-C(6)	1.304(6)	C(15)-N(1)-C(11)	112.5(3)
C(5)-H(5A)	0.9500	C(13)-N(1)-Si(1)	105.4(2)
C(6)-C(7)	1.545(6)	C(15)-N(1)-Si(1)	104.6(2)
C(6)-H(6A)	0.9500	C(11)-N(1)-Si(1)	104.4(2)
C(7)-C(9)	1.512(5)	C(10)-O(1)-Si(1)	122.9(2)
C(7)-C(8)	1.527(5)	C(12)-O(2)-Si(1)	123.5(2)
C(7)-H(7A)	1.0000	C(14)-O(3)-Si(1)	123.5(2)
C(8)-H(8A)	1.0000	C(2)-C(1)-C(8)	89.9(3)
C(9)-H(9A)	0.9900	C(2)-C(1)-H(1A)	113.7
C(9)-H(9B)	0.9900	C(8)-C(1)-H(1A)	113.7
C(10)-C(11)	1.507(5)	C(2)-C(1)-H(1B)	113.7
C(10)-H(10A)	0.9900	C(8)-C(1)-H(1B)	113.7
C(10)-H(10B)	0.9900	H(1A)-C(1)-H(1B)	110.9
C(11)-H(11A)	0.9900	C(1)-C(2)-C(3)	90.6(3)
C(11)-H(11B)	0.9900	C(1)-C(2)-Si(1)	116.7(3)
C(12)-C(13)	1.512(5)	C(3)-C(2)-Si(1)	114.7(2)
C(12)-H(12A)	0.9900	C(1)-C(2)-H(2A)	111.1
C(12)-H(12B)	0.9900	C(3)-C(2)-H(2A)	111.1
C(13)-H(13A)	0.9900	Si(1)-C(2)-H(2A)	111.1
C(13)-H(13B)	0.9900	C(8)-C(3)-C(4)	102.2(3)
C(14)-C(15)	1.534(5)	C(8)-C(3)-C(2)	89.6(3)
C(14)-H(14A)	0.9900	C(4)-C(3)-C(2)	117.8(3)
C(14)-H(14B)	0.9900	C(8)-C(3)-H(3A)	114.6
C(15)-H(15A)	0.9900	C(4)-C(3)-H(3A)	114.6
C(15)-H(15B)	0.9900	C(2)-C(3)-H(3A)	114.6
O(3)-Si(1)-O(2)	118.03(13)	C(9)-C(4)-C(5)	97.2(3)
O(3)-Si(1)-O(1)	118.51(14)	C(9)-C(4)-C(3)	103.3(3)
O(2)-Si(1)-O(1)	118.76(14)	C(5)-C(4)-C(3)	102.9(3)
O(3)-Si(1)-C(2)	97.32(14)	C(9)-C(4)-H(4A)	116.9
O(2)-Si(1)-C(2)	97.65(14)	C(5)-C(4)-H(4A)	116.9

C(3)-C(4)-H(4A)	116.9	N(1)-C(11)-C(10)	105.6(3)
C(6)-C(5)-C(4)	108.2(4)	N(1)-C(11)-H(11A)	110.6
C(6)-C(5)-H(5A)	125.9	C(10)-C(11)-H(11A)	110.6
C(4)-C(5)-H(5A)	125.9	N(1)-C(11)-H(11B)	110.6
C(5)-C(6)-C(7)	107.2(4)	C(10)-C(11)-H(11B)	110.6
C(5)-C(6)-H(6A)	126.4	H(11A)-C(11)-H(11B)	108.8
C(7)-C(6)-H(6A)	126.4	O(2)-C(12)-C(13)	109.8(3)
C(9)-C(7)-C(8)	103.2(3)	O(2)-C(12)-H(12A)	109.7
C(9)-C(7)-C(6)	98.5(3)	C(13)-C(12)-H(12A)	109.7
C(8)-C(7)-C(6)	102.9(3)	O(2)-C(12)-H(12B)	109.7
C(9)-C(7)-H(7A)	116.6	C(13)-C(12)-H(12B)	109.7
C(8)-C(7)-H(7A)	116.6	H(12A)-C(12)-H(12B)	108.2
C(6)-C(7)-H(7A)	116.6	N(1)-C(13)-C(12)	107.3(3)
C(7)-C(8)-C(3)	103.5(3)	N(1)-C(13)-H(13A)	110.3
C(7)-C(8)-C(1)	116.1(3)	C(12)-C(13)-H(13A)	110.3
C(3)-C(8)-C(1)	89.8(3)	N(1)-C(13)-H(13B)	110.3
C(7)-C(8)-H(8A)	114.7	C(12)-C(13)-H(13B)	110.3
C(3)-C(8)-H(8A)	114.7	H(13A)-C(13)-H(13B)	108.5
C(1)-C(8)-H(8A)	114.7	O(3)-C(14)-C(15)	108.4(3)
C(4)-C(9)-C(7)	95.9(3)	O(3)-C(14)-H(14A)	110.0
C(4)-C(9)-H(9A)	112.6	C(15)-C(14)-H(14A)	110.0
C(7)-C(9)-H(9A)	112.6	O(3)-C(14)-H(14B)	110.0
C(4)-C(9)-H(9B)	112.6	C(15)-C(14)-H(14B)	110.0
C(7)-C(9)-H(9B)	112.6	H(14A)-C(14)-H(14B)	108.4
H(9A)-C(9)-H(9B)	110.1	N(1)-C(15)-C(14)	105.4(3)
O(1)-C(10)-C(11)	110.0(3)	N(1)-C(15)-H(15A)	110.7
O(1)-C(10)-H(10A)	109.7	C(14)-C(15)-H(15A)	110.7
C(11)-C(10)-H(10A)	109.7	N(1)-C(15)-H(15B)	110.7
O(1)-C(10)-H(10B)	109.7	C(14)-C(15)-H(15B)	110.7
C(11)-C(10)-H(10B)	109.7	H(15A)-C(15)-H(15B)	108.8
H(10A)-C(10)-H(10B)	108.2		

Table S4 Anisotropic displacement parameters ($Å^2 x 10^3$) for **3**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Si(1)	11(1)	13(1)	10(1)	0(1)	2(1)	-2(1)
N(1)	14(1)	17(1)	12(1)	0(1)	3(1)	-2(1)
O(1)	14(1)	28(1)	17(1)	-9(1)	4(1)	-2(1)
O(2)	19(1)	22(1)	10(1)	1(1)	1(1)	-6(1)
O(3)	19(1)	14(1)	20(1)	3(1)	-3(1)	-3(1)
C(1)	22(2)	35(2)	24(2)	-1(2)	5(2)	-1(2)
C(2)	16(2)	18(2)	13(2)	-1(1)	2(1)	-5(1)
C(3)	15(2)	15(2)	15(2)	-2(1)	5(1)	-3(1)
C(4)	20(2)	36(2)	28(2)	-5(2)	2(2)	7(2)
C(5)	27(2)	30(2)	44(3)	-1(2)	13(2)	-12(2)
C(6)	31(2)	22(2)	50(3)	-1(2)	6(2)	-6(2)
C(7)	23(2)	29(2)	22(2)	-9(2)	0(2)	2(2)
C(8)	27(2)	22(2)	24(2)	-1(2)	-1(2)	8(2)
C(9)	22(2)	26(2)	32(2)	-6(2)	-11(2)	2(2)
C(10)	21(2)	28(2)	18(2)	-10(2)	4(2)	0(2)
C(11)	26(2)	32(2)	27(2)	-10(2)	7(2)	-3(2)
C(12)	22(2)	23(2)	21(2)	5(2)	4(2)	-10(2)
C(13)	25(2)	32(2)	23(2)	-2(2)	6(2)	-13(2)
C(14)	16(2)	22(2)	20(2)	6(2)	0(2)	1(1)
C(15)	27(2)	28(2)	24(2)	4(2)	-1(2)	-3(2)

Table S5 Hydrogen coordinates ($x 10^4$) and isotropic displacement parameters ($Å^2 x 10^3$) for **3**.

	x	y	z	U(eq)
H(1A)	2894	4303	3936	32
H(1B)	952	4475	3010	32
H(2A)	4094	3372	2914	19
H(3A)	4142	4571	1346	18
H(4A)	7592	4044	2469	34
H(5A)	8176	5482	1810	39
H(6A)	6605	6488	2722	42

H(7A)	5168	5747	4127	30
H(8A)	2720	5533	2346	31
H(9A)	8136	4851	4121	35
H(9B)	6211	4265	4082	35
H(10A)	3298	2003	-187	27
H(10B)	2335	2835	-705	27
H(11A)	-346	1981	-960	34
H(11B)	341	1570	165	34
H(12A)	-2288	2122	2270	26
H(12B)	-708	1593	1808	26
H(13A)	-2988	1885	350	32
H(13B)	-3430	2778	752	32
H(14A)	-1648	4641	-274	24
H(14B)	-2792	4144	473	24
H(15A)	-2971	3261	-910	33
H(15B)	-711	3454	-1004	33

Table S6 Torsion angles [°] for **3**.

O(3)-Si(1)-O(1)-C(10)	-70.4(3)
O(2)-Si(1)-O(1)-C(10)	84.8(3)
C(2)-Si(1)-O(1)-C(10)	-172.6(3)
N(1)-Si(1)-O(1)-C(10)	7.3(3)
O(3)-Si(1)-O(2)-C(12)	83.1(3)
O(1)-Si(1)-O(2)-C(12)	-72.3(3)
C(2)-Si(1)-O(2)-C(12)	-174.4(3)
N(1)-Si(1)-O(2)-C(12)	5.4(3)
O(2)-Si(1)-O(3)-C(14)	-70.4(3)
O(1)-Si(1)-O(3)-C(14)	85.0(3)
C(2)-Si(1)-O(3)-C(14)	-173.2(3)
N(1)-Si(1)-O(3)-C(14)	7.1(3)
C(8)-C(1)-C(2)-C(3)	3.0(3)
C(8)-C(1)-C(2)-Si(1)	-115.1(3)
O(3)-Si(1)-C(2)-C(1)	50.4(3)
O(2)-Si(1)-C(2)-C(1)	-69.3(3)
O(1)-Si(1)-C(2)-C(1)	170.3(3)
O(3)-Si(1)-C(2)-C(3)	-53.7(3)
O(2)-Si(1)-C(2)-C(3)	-173.4(2)
O(1)-Si(1)-C(2)-C(3)	66.3(3)
C(1)-C(2)-C(3)-C(8)	-3.1(3)
Si(1)-C(2)-C(3)-C(8)	116.8(3)
C(1)-C(2)-C(3)-C(4)	100.5(3)
Si(1)-C(2)-C(3)-C(4)	-139.6(3)
C(8)-C(3)-C(4)-C(9)	32.0(4)
C(2)-C(3)-C(4)-C(9)	-64.0(4)
C(8)-C(3)-C(4)-C(5)	-68.7(4)
C(2)-C(3)-C(4)-C(5)	-164.7(3)
C(9)-C(4)-C(5)-C(6)	-36.0(4)
C(3)-C(4)-C(5)-C(6)	69.5(4)
C(4)-C(5)-C(6)-C(7)	2.7(5)
C(5)-C(6)-C(7)-C(9)	31.7(4)
C(5)-C(6)-C(7)-C(8)	-74.0(4)
C(9)-C(7)-C(8)-C(3)	-34.5(4)
C(6)-C(7)-C(8)-C(3)	67.5(4)
C(9)-C(7)-C(8)-C(1)	62.0(4)
C(6)-C(7)-C(8)-C(1)	164.1(3)

C(4)-C(3)-C(8)-C(7)	1.5(4)
C(2)-C(3)-C(8)-C(7)	119.9(3)
C(4)-C(3)-C(8)-C(1)	-115.4(3)
C(2)-C(3)-C(8)-C(1)	3.0(3)
C(2)-C(1)-C(8)-C(7)	-108.1(3)
C(2)-C(1)-C(8)-C(3)	-3.1(3)
C(5)-C(4)-C(9)-C(7)	52.7(4)
C(3)-C(4)-C(9)-C(7)	-52.4(4)
C(8)-C(7)-C(9)-C(4)	53.6(4)
C(6)-C(7)-C(9)-C(4)	-51.9(4)
Si(1)-O(1)-C(10)-C(11)	-30.0(4)
C(13)-N(1)-C(11)-C(10)	-147.2(3)
C(15)-N(1)-C(11)-C(10)	79.9(4)
Si(1)-N(1)-C(11)-C(10)	-32.9(3)
O(1)-C(10)-C(11)-N(1)	40.3(4)
Si(1)-O(2)-C(12)-C(13)	-25.3(4)
C(15)-N(1)-C(13)-C(12)	-145.3(3)
C(11)-N(1)-C(13)-C(12)	83.0(4)
Si(1)-N(1)-C(13)-C(12)	-30.6(3)
O(2)-C(12)-C(13)-N(1)	36.2(4)
Si(1)-O(3)-C(14)-C(15)	-30.0(4)
C(13)-N(1)-C(15)-C(14)	80.3(4)
C(11)-N(1)-C(15)-C(14)	-147.5(3)
Si(1)-N(1)-C(15)-C(14)	-34.8(3)
O(3)-C(14)-C(15)-N(1)	41.6(4)

Symmetry transformations used to generate equivalent atoms:

Reference

1. C. D. Smith, *Org. Synth.*, 1971, **51**, 133.