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Dianion Cyclization Strategy for the Synthesis of Macrosilaheterocycles

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ABSTRACT: *A practical and efficient method for the preparation of silaheterocycles is described. The key step involves the initial formation of symmetrical chiral ditopic ligand, N,N′-1,2-cyclohexylenebis(salicylideneimine) followed by sequential deprotonation with NaH to form dianion intermediate, which reacts with diorganodichlorosilanes to furnish dibenzodioxadiazasilamacrocycles. The products were characterized by satisfactory elemental analyses and spectral (IR, ^{1}H, ^{13}C, ^{29}Si NMR, and Mass) studies.* © 2008 Wiley Periodicals, Inc. Heteroatom Chem 19:455–460, 2008; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20460

INTRODUCTION

A Schiff base (or Schiff's base) named after Hugo Schiff [1] is an organic compound in which the nitrogen atom of an amino group is doubly bonded to a carbon atom. The formation of the Schiff base is an important step in many biochemical reactions and is one of the oldest reactions in chemistry [1]. Schiff bases have been widely used as a protective group of amino functionalities in organic synthesis [2,3] and as chelating ligands in the field of coordination chemistry [4,5]. There has been considerable interest in some Schiff bases derived from salicylaldehyde and substituted salicylaldehyde because they show thermochromism and photochromism in a solid state [6,7]. Schiff bases are valuable synthons for the preparation of macroheterocycles [8,9]. The vigorous development in the chemistry of macroheterocyclic compounds has been observed in recent years [10]. Furthermore, it has been investigated that the macroheterocycles bind not only metal cations but also anions and even neutral organic molecules, forming complexes of the "host–guest" types [11]. More recent applications of salen ligands include synthesis of metal containing liquid crystalline polymers [12], antiviral agents [13], asymmetric catalysts [14], and reactions of epoxidation, epoxide ring opening, and aziridination [15,16]. In recent years, symmetric and asymmetric transition metal complexes of the salen-type chiral Schiff bases have been developed and are used as ligands/catalysts in many reactions such as epoxidation [17], asymmetric synthesis [18], asymmetric sulfoxidation [19], asymmetric silylcyanation [20], and many other applications [21–23].

To the best of our knowledge, there are no reports on the synthesis of 13-membered silaheterocycles, containing five heteroatoms (N, O, and Si). Our aim is to broaden the range of useful silaheterocycles, which can provide an easy access to synthetic intermediates and therapeutic agents. With this background, the goal of the present article is to provide cyclization reactions of remote dianion via cyclosilylation. A heteroatom-based remote dianion has become increasingly a popular strategic tool for the synthetic planning [24–28]. A remarkable feature of this reaction is the construction of oxygen–silicon bonds via a tandem process. In our

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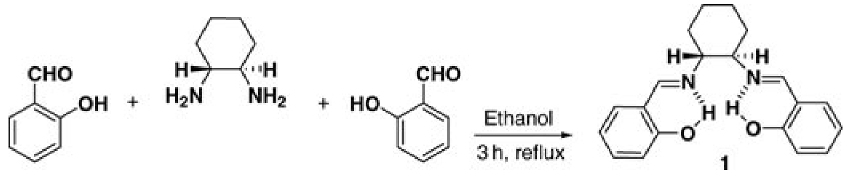
continuing studies on the synthesis of new heterocyclic systems [29–32], using efficient intermolecular cyclization reactions via a dianion intermediate, we herein developed a modular approach to a new class of structurally diverse dibenzodioxadiazasilatridecines in good yields. The compounds may be used in chemical vapor deposition and deoxygenation reactions in organic transformations. Their derivatives may be of use in pharmaceutical chemistry.

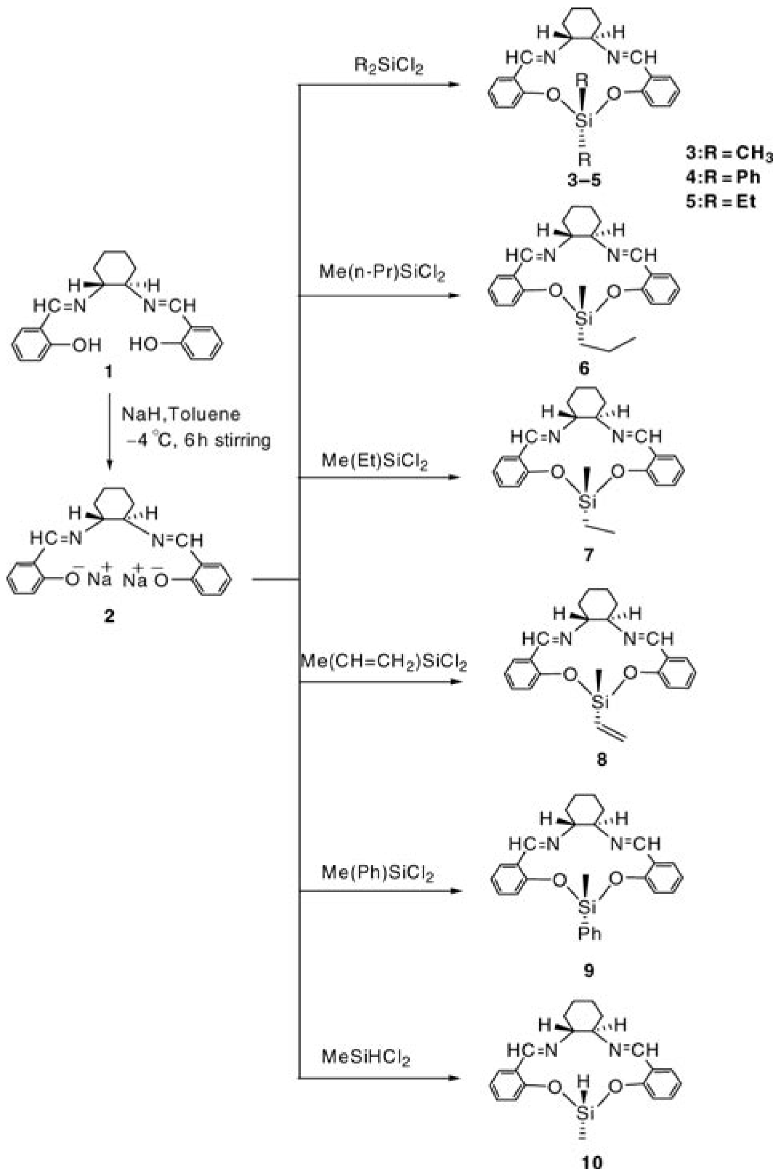
RESULTS AND DISCUSSION

The reaction of 1 equivalent of diaminocyclohexane with 2 equivalents of salicylaldehyde in ethanol under reflux for 3 h according to the literature method [21b] afforded the corresponding symmetrical salen Schiff base ligand, *N,N′*-1,2-cyclohexylenebis-(salicylideneimine) **1**. The structure of ligand (**1**) was established on the basis of its spectral and analytical data. The IR spectrum shows the lack of N—H stretching bands in 3150–3450 cm^{-1} and the presence of strong C=N stretching at 1630 cm^{-1}. In its ^{1}H NMR spectrum, a singlet at 13.29 ppm exhibits for two phenolic hydroxyl groups that are D_2O exchangeable. The downfield signal shows that there is intramolecular hydrogen bonding between the azomethine nitrogen and OH group ortho to it, forming a six-membered ring. Two azomethine protons of the CH=N group manifests at 8.25 ppm as a sharp singlet. A multiplet in the range 6.75–7.25 ppm for eight aromatic protons, a multiplet in the range 3.26–3.32 ppm for two aliphatic CH protons, and a multiplet in the range 1.95–1.43 ppm for eight aliphatic CH_2 protons have been observed. Two identical salicylaldehyde moieties on both sides of the diamine in the ligand make the same electronic and steric contributions.



The introduction of diorganosilylene group is normally achieved by the reaction of diorganodichlorosilane with a proper substrate in the presence of a base to prepare heterocyclic compounds containing a silicon atom. Our synthesis involves the initial formation of dianion (**2**) from a sequential deprotonation of the phenolic OH groups of ligand **1** by sodium hydride in dry toluene. The remote dianion (**2**) thus generated attacks on a variety of diorganodichlorosilanes leading to the formation of compounds from **3** to **10** (Scheme 1). The beauty of reaction resides in the in situ formation of remote dianion and further cyclization, so that the multistep reaction sequence is synchronized in a simple one pot. TLC of all compounds confirmed their purity. The disappearance of absorption band and signals, corresponding to the —OH group in both IR and ^{1}H NMR spectra, and appearance of new band, in the region 1048–840 cm^{-1}, may be assigned to Si—O bond [33], suggest the cyclic structure of prepared compounds. The IR spectra exhibit C=N absorptions in the range 1600–1630 cm^{-1} and Si—C absorption in the range 1265–1285 cm^{-1}. No lowering of ν(C=N) [34] is a further indication of noncoordination of C=N to the silicon atom exhibiting tetracoordinated state, which is further supported by ^{29}Si NMR chemical shifts.^{1}H NMR spectra display a singlet at 8.25 ppm for azomethine proton, a multiplet in the range 6.75–7.25 ppm for eight aromatic protons, a multiplet in the range 3.36–3.26 ppm for two aliphatic C—H protons, a multiplet in the range 1.97–1.41 ppm for eight aliphatic CH_2 protons, and singlets of silicon methyl in the range 0.11–0.70 ppm. The signals for the vinyl and *n*-propyl proton were visible in the expected regions. The alkyl groups attached to silicon displayed single resonance for chemically equivalent protons and carbons. In addition, the ^{13}C NMR spectra supported the assigned structures indicated in Scheme 1. The upfield shifts of the ^{29}Si NMR signal of the compounds indicate the presence of tetracoordinate [35] silicon in all the compounds.

Because the R group is bound directly to the ^{29}Si nucleus, it is not surprising that the value of δ depends primarily on the nature of this R group. When R = phenyl, the chemical shift is consistently more negative (by about 25 ppm) than that R = alkyl. Although the alkyl moiety has a greater electron-pushing capacity (σ donation) than the aryl, the delocalized π system in the phenyl-substituted compound allows for dπ–pπ interaction to dominate the overall shielding of the ^{29}Si nucleus [36]. Mass spectroscopic data of the compounds established their monomeric nature. The newly synthesized



SCHEME 1

silaheterocyclic rings gain rigidity due to the presence of two benzene rings in their skeleton.

$SiCl_4$ and $HSiCl_3$ both were used as trapping reagents, but in both cases the TLC shows many close spots that are not separable with column chromatography. It seems that in both cases monomer, dimer, and some polymeric materials are formed. Still the work is in progress to isolate the products. All products from **3** to **10** are amorphous solids. We did not try to develop crystals. We welcome the valuable suggestion, which will be taken care in future.

EXPERIMENTAL

Chemicals were obtained from Sigma-Aldrich, Merck, Fluka, and Lancaster, and are used as such without further purification. All solvents (AR or extra pure grade) used for spectroscopic and other physical studies were further purified by the literature methods [37]. All operations were performed under nitrogen atmosphere using standard glassware. IR spectra were recorded as KBr disks and in Nujol mull on JASCO FT/IR-5300 spectrophotometer. Melting points were determined using a calibrated thermometer by Remi digital melting point apparatus and are uncorrected. Elemental analysis was determined with a Vario-EL analyzer. NMR (^{1}H, ^{13}C, and ^{29}Si) spectra were recorded on a JEOL AL 300FT NMR spectrometer. All chemical shifts were reported in parts per million relative to TMS as an internal standard in $CDCl_3$. Mass spectra were recorded at 70 eV ionizing voltage on a JEOL SX-102 (FAB).

Synthesis of N,N′-1,2-Cyclohexylenebis(salicylideneimine) (**1**). To a solution of 1,2-diaminocyclohexane (140 mg, 1.23 mmol) in absolute ethanol (1 mL), salicylaldehyde (300 mg, 2.46 mmol) was added. The resulting mixture was then refluxed for 3 h. After cooling to room temperature, water (5 mL) was added and the mixture was stirred for 30 min. Upon the removal of the solvent and cooling, a yellow precipitate formed was collected and recrystallized from ethanol to give a symmetrical chiral ligand as yellow needles (277 mg, 70.5%), mp 74°C. Anal. Calcd for $C_{20}H_{22}N_2O_2$: C, 74.51; H, 6.88; N, 8.69. Found: C, 74.94; H, 7.22; N, 8.36. IR (KBr): 3060, 2932, 2858, 1630 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 13.29$ (s, 2H, OH), 8.25 (s, 2H, CH=N), 7.25–6.75 (m, 8H, ArH), 3.32–3.26 (m, 2H, CH), 1.95–1.43 (m, 8H, CH_2). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 164.70$, 160.97, 132.14, 131.47, 118.67, 118.58, 116.77, 72.65, 33.11, 24.18. MS: $m/z = 322$.

Synthesis of Dibenzodioxadiazasilatridecine (**3**): Ligand (322 mg, 1 mmol) dissolved in toluene (55 mL) was added dropwise to stirred suspension of NaH (48 mg, 2 mmol) in dry toluene (5 mL) with constant stirring at −4°C for 6 h in an inert atmosphere. Dichlorodimethylsilane (129 mg, 1 mmol) in dry toluene (5 mL) was added dropwise with constant stirring to get the yellowish solution of dianion, generated in situ. The reaction mixture was stirred at room temperature for an additional 4 h. Completion of the reaction was confirmed by TLC. The reaction mixture was evaporated with the help of a rotary evaporator, and the residue was subjected to column chromatography (diethyl ether) to give yellow solid (257 mg, 68%), mp 88°C. Anal. Calcd for $C_{22}H_{26}N_2O_2Si$: C, 69.80; H, 6.92; N, 7.40. Found: C, 69.98; H, 6.64; N, 7.23. IR (Nujol): 2852, 1630, 1278, 1041 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 8.25$ (s, 2H, CH=N), 7.25–6.76 (m, 8H, ArH), 3.36–3.27 (m, 2H, CH), 1.97–1.43 (m, 8H, CH_2), 0.14 (s, 6H, $SiCH_3$). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 164.70$, 160.97, 132.14, 131.47, 118.67, 118.58, 116.77, 72.65, 33.11, 24.18, 1.00. ^{29}Si NMR (59.60 MHz, $CDCl_3$): $\delta = -52.26$. MS: $m/z = 378$.

All other silaheterocycles (**4–10**) were synthesized analogously as mentioned above. The analytical and spectral data for the compounds from **4** to **10** are listed below:

4: Following the general experimental procedure for **3** with 322 mg (1 mmol) of *N,N′*-1,2-cyclohexylenebis(salicylideneimine), 48 mg (2 mmol) of sodium hydride and 253 mg (1 mmol) of dichlorodiphenylsilane afforded yellow solid (276 mg, 55%), mp 87°C. Anal. Calcd for $C_{32}H_{30}N_2O_2Si$: C, 76.46; H, 6.01; N, 5.57. Found: C, 76.82; H, 6.36; N, 5.82. IR (Nujol): 2856, 1630, 1280, 1047 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 8.25$ (s, 2H, CH=N), 7.50–6.50 (m, 18H, ArH), 3.31–3.27 (m, 2H, CH), 1.95–1.43 (m, 8H, CH_2). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 163.66$, 159.92, 133.31, 131.10, 130.42, 126.66, 117.60, 117.54, 115.72, 71.56, 32.04, 23.12. ^{29}Si NMR (59.60 MHz, $CDCl_3$): $\delta = -56.24$. MS: $m/z = 502$.

5: Following the general experimental procedure for **3** with (322 mg, 1 mmol) of *N,N′*-1,2-cyclohexylenebis(salicylideneimine), 48 mg (2 mmol) of sodium hydride and 157 mg (1 mmol) of dichlorodiethylsilane gave yellowish brown solid (203 mg, 50%), mp 92°C. Anal. Calcd for $C_{24}H_{30}N_2O_2Si$: C, 70.89; H, 7.43; N, 6.88. Found: C, 70.43; H, 7.12; N, 6.35. IR (Nujol): 2858, 1630, 1278, 844 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 8.25$ (s, 2H, CH=N), 7.25–6.76 (m, 8H, ArH), 3.36–3.27 (m, 2H, CH), 1.95–1.47 (m, 8H, CH_2), 0.98 (t, $J = 7.8$ Hz, 6H, $SiCH_3$), 0.61 (q, $J = 7.8$ Hz, 4H, $SiCH_2$). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 164.70$, 160.97, 132.14, 131.47, 118.67, 118.58, 116.77, 72.65, 33.11, 24.18, 16.13, 14.13. ^{29}Si NMR (59.60 MHz, $CDCl_3$): $\delta = -48.46$. MS: $m/z = 406$.

6: Following the general experimental procedure for **3** with 322 mg (1 mmol) of *N,N′*-1,2-cyclohexylenebis(salicylideneimine), 48 mg (2 mmol) of sodium hydride and 157 mg (1 mmol) of dichloromethyl-*n*-propylsilane afforded yellow solid (256 mg, 63%), mp 86°C. Anal. Calcd for $C_{24}H_{30}N_2O_2Si$: C, 70.89; H, 7.43; N, 6.88. Found: C, 70.64; H, 7.23; N, 6.52. IR (Nujol): 2910, 1628, 1280, 981 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 8.22$ (s, 2H, CH=N), 7.23–6.74 (m, 8H, ArH), 3.32–3.23 (m, 2H, CH), 1.92–1.40 (m, 8H, CH_2), 0.85–1.00 (m, 5H, $SiCH_2CH_2CH_3$), 0.57 (t, $J = 8.4$ Hz, 2H, $SiCH_2$), 0.11 (s, 3H, $SiCH_3$). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 165.74$, 165.17, 162.28, 162.01, 133.28, 133.20, 132.50, 132.48, 119.85, 119.68, 119.62, 119.53, 118.06, 117.80, 73.65, 70.47, 34.13, 31.71, 25.21, 23.53, 20.39, 18.92, 17.49. ^{29}Si NMR (59.60 MHz, $CDCl_3$): $\delta = -53.57$. MS: $m/z = 406$.

7: Following the general experimental procedure for **3** with 322 mg (1 mmol) of *N,N′*-1,2-cyclohexylenebis(salicylideneimine), 48 mg (2 mmol) of sodium hydride and 143 mg (1 mmol) of dichloroethylmethylsilane yielded yellowish green solid (227 mg, 58%), mp 80°C. Anal. Calcd for $C_{23}H_{28}N_2O_2Si$: C, 70.37; H, 7.18; N, 7.13. Found: C, 70.12; H, 7.46; N, 7.37. IR (Nujol): 2926, 2854, 1631, 1278, 1045 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): $\delta = 8.25$ (s, 2H, $C\underline{H}{=}N$), 7.25–6.76 (m, 8H, $Ar\underline{H}$), 3.33–3.29 (m, 2H, $C\underline{H}$), 1.96–1.47 (m, 8H, $C\underline{H}_2$), 0.96 (t, $J = 7.8$ Hz, 3H, $SiCH_2C\underline{H}_3$), 0.53 (q, $J = 8.7$ Hz, 2H, $SiC\underline{H}_2$), 0.17 (s, 3H, $SiC\underline{H}_3$). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 164.28$, 163.71, 160.82, 160.53, 131.83, 131.73, 131.05, 118.38, 118.16, 118.08, 116.60, 116.33, 72.18, 68.99, 32.66, 30.24, 23.74, 22.05. ^{29}Si NMR (59.60 MHz, $CDCl_3$): $\delta = -3.56$. MS: $m/z = 392$.

8: Following the general experimental procedure for **3** with (322 mg, 1 mmol) of *N,N′*-1,2-cyclohexylenebis(salicylideneimine), 48 mg (2 mmol) of sodium hydride and 141 mg (1 mmol) of dichloromethylvinylsilane gave brown solid (234 mg, 60%), mp 92°C. Anal. Calcd for $C_{23}H_{26}N_2O_2Si$: C, 70.73; H, 6.71; N, 7.17. Found: C, 70.46; H, 6.27; N, 6.89. IR (Nujol): 2920, 1626, 1261, 956 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): $\delta = 8.25$ (s, 2H, $C\underline{H}{=}N$), 7.25–6.78 (m, 8H, $Ar\underline{H}$), 5.77–6.02 (m, 3H, $SiC\underline{H}{=}C\underline{H}_2$), 3.34–3.29 (m, 2H, $C\underline{H}$), 1.91–1.47 (m, 8H, $C\underline{H}_2$), 0.18 (s, 3H, $SiC\underline{H}_3$). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 164.67$, 160.93, 132.12, 131.43, 118.62, 118.55, 116.73, 72.60, 33.07, 29.68, 26.74, 24.15, 1.00. ^{29}Si NMR (59.60 MHz, $CDCl_3$): $\delta = -46.26$. MS: $m/z = 390$.

9: Following the general experimental procedure for **3** with 322 mg (1 mmol) of *N,N′*-1,2-cyclohexylenebis(salicylideneimine), 48 mg (2 mmol) of sodium hydride and 191 mg (1 mmol) of dichloromethylphenylsilane afforded yellowish brown solid (233 mg, 53%), mp 89°C. Anal. Calcd for $C_{27}H_{28}N_2O_2Si$: C, 73.60; H, 6.40; N, 6.35. Found: C, 73.94; H, 6.23; N, 6.03. IR (Nujol): 2924, 2852, 1630, 1278, 840 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): $\delta = 8.25$ (s, 2H, $C\underline{H}{=}N$), 7.75–6.65 (m, 13H, $Ar\underline{H}$), 3.31–3.26 (m, 2H, $C\underline{H}$), 1.95–1.43 (m, 8H, $C\underline{H}_2$), 0.83 (m, 3H, $SiC\underline{H}_3$). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 163.66$, 159.92, 133.31, 131.10, 130.42, 126.66, 117.60, 117.54, 115.72, 71.56, 32.04, 23.12, 14.68. ^{29}Si NMR (59.60 MHz, $CDCl_3$): $\delta = -57.86$. MS: $m/z = 440$.

10: Following the general experimental procedure for **3** with 322 mg (1 mmol) of *N, N′*-1,2-cyclohexylenebis(salicylideneimine), 48 mg (2 mmol) of sodium hydride and 115 mg (1 mmol) of dichloromethylsilane afforded brown solid (233 mg, 64%), mp 90°C. Anal. Calcd for $C_{21}H_{24}N_2O_2Si$: C, 69.19; H, 6.63; N, 7.68. Found: C, 69.47; H, 6.42; N, 7.26. IR (Nujol): 2924, 1630, 1278, 940 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): $\delta = 8.25$ (s, 2H, $C\underline{H}{=}N$), 7.54–6.76 (m, 8H, $Ar\underline{H}$), 4.70 (s, 1H, $Si\underline{H}$), 3.32–3.27 (m, 2H, $C\underline{H}$), 1.90–1.47 (m, 8H, $C\underline{H}_2$), 0.19 (s, 3H, $SiC\underline{H}_3$). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 164.70$, 160.97, 132.14, 131.47, 118.67, 118.58, 116.77, 72.65, 33.11, 24.18, 14.55. ^{29}Si NMR (59.60 MHz, $CDCl_3$): $\delta = -9.26$. MS: $m/z = 364$.

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