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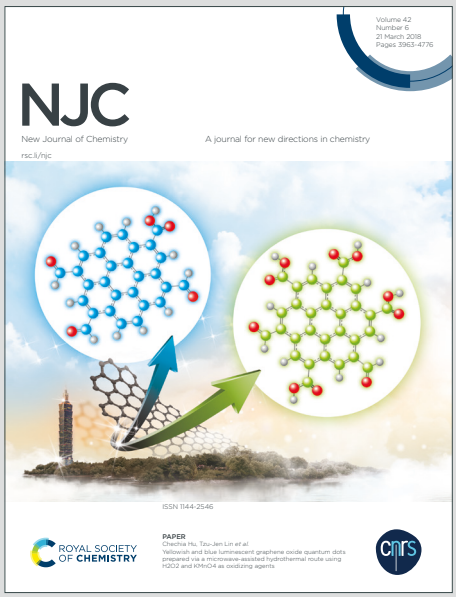

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Ammonia-Free and One-Pot Synthesis of Di-Chloro Silicon Phthalocyanine and Naphthalocyanine

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ABSTARCT

Silicon phthalocyanines (SiPcs) and silicon naphthalocyanines (SiNcs) are promising and versatile organic materials with applications spanning across organic electronics, photocatalysis, and photodynamic therapy. Specifically, the availability for modification of periphery sites and axial positions enables tailoring for solubility, bandgap tuning, or even biological targeting. The dichlorinated species (Cl_2-SiPc and Cl_2-SiNc) are particularly attractive due to the facile substitution of the axial chloride groups. However, conventional syntheses rely on a two-step protocol that requires the use of an ammonia gas tank at high temperatures. Here, we report a safer, one-pot synthesis of Cl_2-SiPc and Cl_2-SiNc directly from unsubstituted phthalonitrile and 2,3-dicyanonaphthalene, respectively. By adapting the alternative route proposed by Lessard *et al.* which uses lithium bis(trimethylsilyl) amide (LiHMDS) in place of ammonia gas, we achieved up to a 45% macrocyclic conversion to-date. Overall, the adapted ammonia-free approach provides a synthetically relevant and sustainable platform for the preparation and functionalization of silicon phthalocyanines and naphthalocyanines. We also studied the microwave process and confirmed also that the silicon phthalocyanines can be formed via the microwave.

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INTRODUCTION

Porphyrin-derived macrocycles, including phthalocyanines (Pcs) and their more conjugated family of naphthalocyanines (Ncs), have been incorporated into a diverse set of applications due to their tunable electronic and photophysical properties. These properties can be finely adjusted through peripheral and axial substitutions, as well as by varying the ring size and metal center of trivalent and tetravalent metal-based Pcs (MPcs) and Ncs (MNcs). To expand, silicon phthalocyanines (R_2-SiPc) and silicon naphthalocyanines (R_2-SiNc), represent a particular well-studied class, demonstrating significance in organic photovoltaics (OPVs), organic thin film transistors (OTFTs), photodynamic therapy (PDT), and even photocatalysis.[1–4] The 4+ oxidation state of silicon provides two axial substituents (R_2), which is most commonly chloride during the first step of forming a R_2-SiPc/R_2-SiNc. Resulting in a hexacoordinated complex, the geometry provides stability towards the Si-N bonds, and opens doors for derivatizations through bulky, polar, or even biological handle substitutions.[5,6] Moreover, axial chloride groups are synthetically attractive as the groups can be readily displaced by a variety of nucleophiles (e.g. phenols, amines, alcohols), leading to diverse libraries of symmetrical or non-symmetrical derivatives.

Conventional synthetic routes to Cl_2-SiPc and Cl_2-SiNc involve multistep protocols that rely on ammonia gas. For SiPc, phthalonitrile is first reacted with ammonia gas and sodium methoxide in methanol. After collection of this process the 1,3-diiminoisoindoline (DII/DI3) intermediate if formed, the cyclotetramerization with silicon tetrachloride ($SiCl_4$) in dry quinoline occurs in separate pot.[5] And it is known that $SiCl_4$ does not form Cl_2-SiPc with phthalonitrile, however it is known that Cl_2-SiPc can be formed via DII/DI3 and this may be due to DII/DI3 having higher Lewis basicity. Similarly, Cl_2-SiNc is typically prepared by conversion of 2,3-dicyanonapthalene to the 1,3-diiminobenz[f]isoindoline intermediate under ammonia gas, with subsequent reaction

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with silicon tetrachloride in tri-*n*butylamine and tetraline solvent.[7] While the described protocols can afford conversions of up to 70% (SiPc) and 51% (SiNc),[7–9] they suffer from two key limitations: (i) a lack of commercial availability of DII/DI3 derivatives due to the complication of peripheral substitution strategies; and (ii) the use of constant bubbling ammonia gas requiring hazardous conditions.

To expand, ammonia gas presents substantial safety and practical challenges. Due to its exothermic and corrosive properties, ammonia gas may cause severe burns to the eyes, skin, and may even cause irreversible damage to moistened membranes within the respiratory tract.[10] In addition, it is also capable of forming explosive mixtures and may pose flammability risks at higher concentrations in the presence of an ignition source.[10] Beyond its hazards and risks, ammonia gas overall requires a specialized unit, which complicates the setup of the reaction and may limit scalability.

In this way, due to the safety and practical concerns surrounding ammonia gas as a reagent, we have developed and optimized a one-pot, ammonia-free protocol for the synthesis of Cl_2-SiPc and Cl_2-SiNc, adapted from the method of Lessard *et al*.[11] Notably, while the conventional ammonia-based protocols report modest yields, our work achieves up to a 45% conversion in macrocyclic formation using highly inactivated phthalonitrile and dicyanonapthalene precursors, which serves as a robust and efficient starting point for peripheral functionalizations tailored to specific electronic, biomedical, and catalytic applications.

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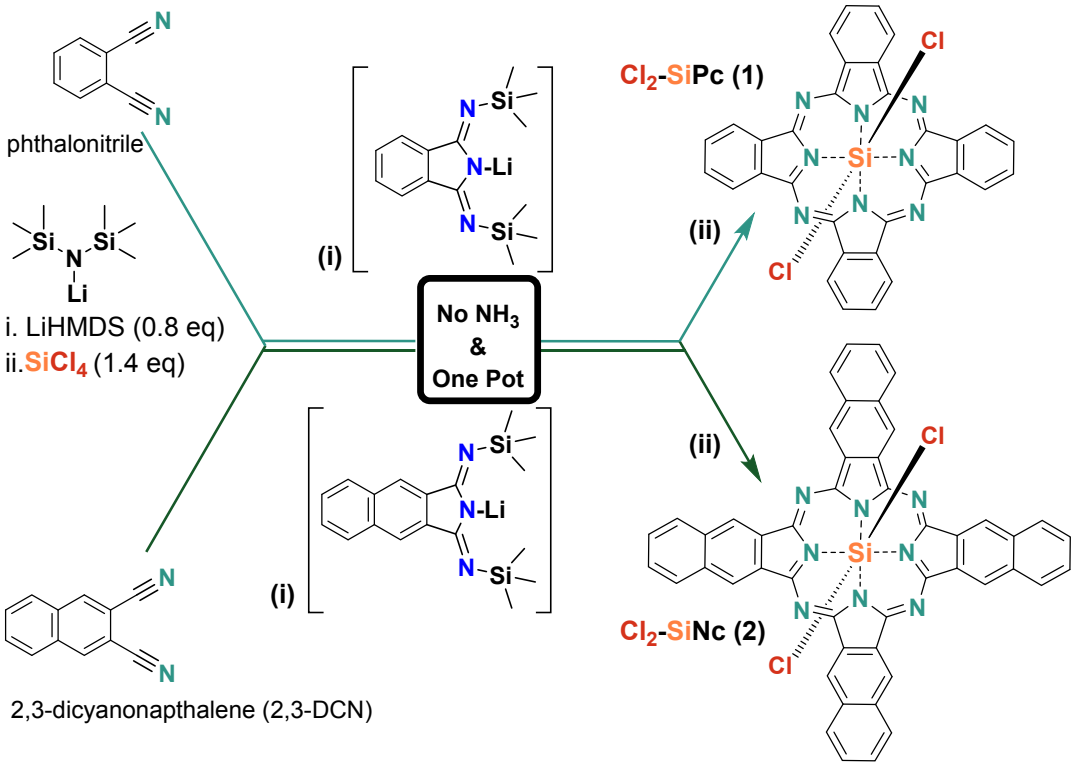

Scheme 1. Synthetic routes in the preparation of Cl_2-SiPc (1) and Cl_2-SiNc (2) without ammonia gas (i.) LiHMDS, tetraline, room temperature, 24 h; and (ii.) $SiCl_4$, 200˚C, 24 h. (b) The intermediate structure (i) is proposed based on the process established.[11]

RESULTS AND DISCUSSION

Unlike adjacent protocols that benefit from electron-withdrawing substituents such as peripheral fluorines to facilitate macrocyclization, this study demonstrates that unsubstituted phthalonitrile can be efficiently converted into a phthalocyanine core. Furthermore, this approach was successfully extended to the naphthalene core, enabling the macrocyclization of a more conjugated aromatic system under identical reaction conditions. As shown in **Scheme 1**, phthalonitrile and 2,3-dicyanonapthalene (2,3-DCN) are employed with LiHMDS and silicon tetrachloride to yield

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the axially chlorinated silicon phthalocyanine (**1**) (crude: 35%) and silicon naphthalocyanine (**2**) (crude: 45%). Despite the similarities in both procedures, compounds **1** and **2** are highly distinct in electronic structure and photophysical properties. Phthalocyanines and porphyrin cores alike, exhibit distinct colours in the solid, pigment state. The deep, dark blue solid illustrated in **Figure 1** resembles the characteristic Cl_2-SiPc colour, whereas the deeper green solid resembles the characteristic Cl_2-SiNc colour. As anticipated, the increase in conjugation in the naphthalocyanine framework results in a bathochromic shift of absorption, correlating with a reduced HOMO-LUMO energy gap, and thus, a colour shift towards the red region.

Figure 1. Solid state colour of macrocyclic products: Cl_2-SiPc (left) Cl_2-SiNc (right).

Following the work-up and isolation of compound **1**, the structure was assessed using MALDI-TOF mass spectrometry. The spectra obtained was assessed as is, without further modifications. The key, parent ion peak that was observed at 575.0 m/z represented the cationic fragment of mono-chlorinated SiPc $[SiPc\text{-}Cl]^+$ (**Fig.S1**). The isotopic distribution of the mono-chlorinated compound can also be observed (**Fig.S2**). After additional sample workup, the fragment pattern decreased and a dominant peak at 609.67 m/z was detected, consistent with the molecular species (**Fig.S3**). Compound **2** was additionally analyzed by mass spectrometry post-work-up. The key,

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parent ion peak that was observed was at 775.1 m/z, represented the cationic fragment of mono-chlorinated SiNc $[SiNc\text{-}Cl]^+$ (**Fig.S4**). Following the additional work-up, the spectrum was substantially cleaned up, leading to a monoisotopic peak of 932.01 m/z (**Fig.S5**). In addition, the splitting pattern was chlorine driven and broadened by carbon and silicon isotopes (**Fig.S6**). It is known in the MS space, due to the MS process the Si-Cl bond is likely 'broken' thermodynamic wise and that is why of the MS outcomes. To expand from structural analysis via mass spectrometry, both compounds were subjected to UV-Visible spectroscopy.

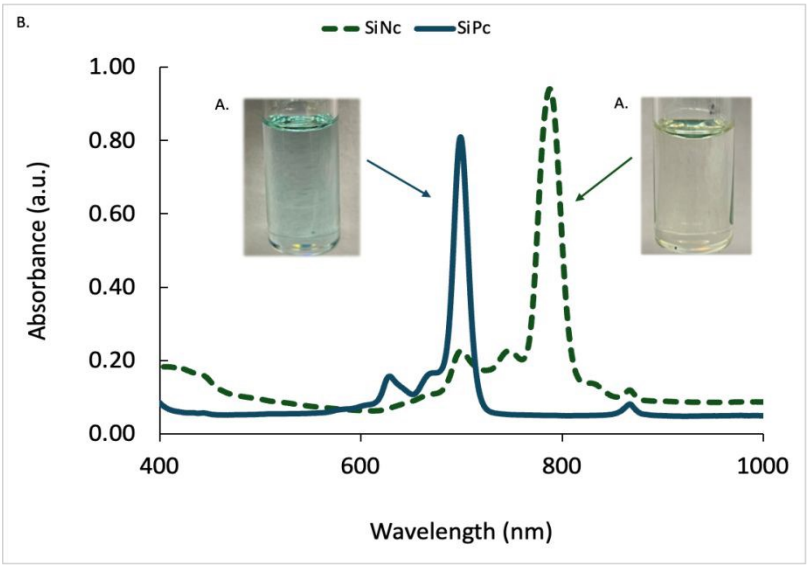

Figure 2. A. Colour of Cl_2-SiPc (left) and Cl_2-SiNc (right) dissolved in 1,2-dichlorobenzene solution for photophysical characterization. **B.** Normalized absorption spectra of Cl_2-SiPc (dotted) Cl_2-SiNc (bolded) in 1,2-dichlorobenzene solution.

Two solutions with concentrations of 20 ng/µL were prepared by dissolving compound **1** (Cl_2-SiPc) and compound **2** (Cl_2-SiNc) in 1,2-dichlorobenzene. **Figure 2A** displays the distinct colour change into teal and light green for compound **1** and **2** dissolution. As a result, the UV absorption of compound **1** revealed a λ_{max} of 699 nm while the absorption for compound **2** revealed a red-

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shifted λ_{max} of 788 nm (**Figure 2B.**). The absorption maxima are consistent with previously reported values of 694 nm for Cl_2-SiPc dissolved in chloroform and 770 nm for Cl_2-SiNc dissolved in dimethylformamide.[1,12] Altogether, the photophysical data supports the identity and purity of the synthesized macrocycles. Other side, due to the lack of solubility, at least a little bit can be diluted in 1,2-dichlorobenzene to enabled the UV absorption data to acquire, but not enough mass can be dissolved, then the ^{1}H-NMR data could not be acquired. But others in the space know that once the axial substituent is away from -Cl to something else then the SiPc and SiNc can have more solubility and other analytics can be taken on.

This work adapts the ammonia-free macrocyclization protocol from Lessard *et al.*[11] which studied fluorinated silicon phthalocyanines (F_2-F_x-SiPc, X=4, 8, 16), which employed lithium bis(trimethylsilyl) amide etherate (LiHMDS·Et_2O) in place of ammonia gas.[11] By extending this one-pot methodology to unsubstituted dichloride SiPc and SiNc with LiHMDS, this project demonstrates that peripheral fluorination is not essential for successful conversion. To expand, our conditions afforded conversions of 35% (Cl_2-SiPc) and 45% (Cl_2-SiNc) at the 250 mg scale, which exceeded the 21-43% range reported by Lessard *et al.* at the 5 g scale.[11] Although the reported yields remain modest, the 2-fold increase highlights the synthetic relevance of the ammonia-free methodology towards dichlorinated SiPcs and SiNcs.

In our first attempt, we applied the identical conditions outlined by Lessard *et al.* which resulted only ~ 20% conversion for both SiPc and SiNc at the 250 mg scale, indicating room for optimization. Subsequently, alterations including reduction in solvent volume, reaction time, controlled heating during reagent additions, as well as the work-up procedure, resulted in improved conversions. Altogether, our observations suggested that higher product concentration coupled with prolonged reaction time, facilitated intermediate formation at room temperature and

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subsequent macrocyclization with silicon tetrachloride, altogether effective for both phthalonitrile and 2,3-dicyanonaphthalene systems. Additionally, the following results open doors to even greater sustainable optimization via microwave chemistry.

Microwave-assisted synthesis has been recently reported as a convenient and quick alternative to longer and rigorously heated benchtop protocols.[13] Specifically, our group has recently demonstrated that microwave irradiation reduced reaction times significantly from 3 to 24 hours to under 36 minutes, in the formation of Cl-BsubPcs and Cl-BsubNcs.[14] In this paper, we have applied our previous microwave-assisted approach to the synthesis of Cl_2-SiPc, under the same conditions as the optimized benchtop procedure (See Supporting Information, heating cycle in **Fig.S6**.). As a preliminary result, the yield obtained (~8 mg, 3%) used the microwave protocol of first irradiating the reaction mixture at 600 W for one cycle, followed by 5 more cycles at 500 W. This was significantly lower than the benchtop procedure since some of the reaction mixture's solvent exited out of the pressure vessel through its bushing during irradiation, therefore, we also studied Cl_2-SiPc microwave synthesis at 400 W cycles (**Fig.S7**). At this power, we estimate its conversion to be roughly 45-60% and no solvent exited occurred. This was done by comparing its relative phthalonitrile absorbance after microwave-assisted synthesis of Cl_2-SiPc to its initial phthalonitrile concentration (**Fig.S8**). Due to Cl_2-SiPc's lack of solubility, we could only estimate the reaction's conversion using HPLC-PDA by observing phthalonitrile that has been consumed. However, at our current status, we only did Cl_2-SiPc, which has no solubility, therefore for future studies, we might be able to measure the reaction's true conversion by phenoxylating the Cl_2-SiPc's axial substituent, which then improves its solubility.[15] This would allow us to then monitor both the reaction's phthalonitrile and the SiPc consumption using HPLC-PDA. While further optimization parameters remain to be explored, microwave irradiation has shown its capability in

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achieving conversion for silicon-based Pcs and Ncs, and most importantly, at a significantly lower heating cycle of 1 hour in comparison to 24 hours.

Outside of the microwave, regarding the past studies of F/fluoro electron withdrawing substitutions and the process setup,[11] given this outcome of non-substitutions was good to see the SiPc and SiNc can be formed without electron withdrawing intermediates. Therefore, for upcoming, we will study other substitutions such as -CR_x, -NR_x, -OR_x, -Cl, nitro, etc., as the SiPcs and SiNcs have applications. We will also try to crystallize the intermediate(s) after their reaction with LiHMDS, to acquire XRD data to confirm the proposed structure(s) (**Scheme 1 i**).

EXPERIMENTAL

General

Phthalonitrile (99%) was purchased from TCI America. Lithium bis(trimethylsilyl) amide (LiHMDS (97%), silicon tetrachloride ($SiCl_4$, 99%), 1,2,3,4-tetrahydronaphthalene (tetraline, anhydrous, 99%), and 1,2-dichlorobenzene (99%) were purchased from Sigma-Aldrich. Dicyanonapthalene was prepared as outlined in literature and recrystalized using acetone.

Synthesis of dichloride-silicon phthalocyanine (1)

An oven-dried, 50 mL three-neck round bottom flask was charged with phthalonitrile (250 mg, 1.95 mmol). After purging the flask with argon for 10 minutes, LiHMDS (197 mg, 1.17 mmol) was added under inert atmosphere and the flask was sealed with rubber septa and placed under positive argon pressure.[11] Next, anhydrous tetralin solvent (6 mL) was added via a needle syringe

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transfer and the solution was set to stir rigorously (370 RPM) at room temperature for 24 hours. After that, the flask was connected to a water condenser and continued remaining under positive argon pressure. Silicon tetrachloride (0.31 mL, 2.73 mmol) was slowly added dropwise to the stirring reaction mixture (370 RPM), which was immediately heated to 200˚C. After 24 hours, the deep turquoise solution was allowed to cool to room temperature which was then admixed with hexanes (20 mL) and methanol (50 mL) and dried under vacuum to result in a deep bluish-teal solid (crude: 95 mg (32%)). MS (MALDI) m/z: [M] calcd for $C_{32}H_{16}Cl_2N_8Si$, 610.06; found, 575.0). To clean up the mass spectrum, a small amount of the solid (20 mg) was stirred in hexanes (10 mL) at room temperature overnight. The solvent was removed rotary evaporation and the product was dried under vacuum, and finally re-subjected to mass spectrometry.

Synthesis of dichloride-silicon naphthalocyanine (2)

An oven-dried, 50 mL three-neck round bottom flask was charged with dicyanonapthalene (250 mg, 1.40 mmol). After purging the flask with argon for 10 minutes, LiHMDS (141 mg, 0.84 mmol) was added under inert atmosphere and the flask was sealed with rubber septa and placed under positive argon pressure. Next, anhydrous tetralin solvent (5 mL) was added via a needle syringe transfer and the solution was set to stir rigorously (370 RPM) at room temperature for 24 hours. After that, the flask was connected to a water condenser and continued remaining under positive argon pressure. Silicon tetrachloride (0.23 mL, 1.96 mmol) was slowly added dropwise to the stirring reaction mixture (370 RPM), which was immediately heated to 200˚C. After 24 hours, the deep yellow-brown solution was allowed to cool to room temperature which was then admixed

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with hexanes (20 mL). The solid was further washed with hexanes (20 mL) and methanol (30 mL) and dried under vacuum to result in a deep green solid (crude: 127 mg (45%)). MS (MALDI) m/z: [M] calcd for $C_{48}H_{24}Cl_2N_8Si$, 810.13; found, 775.1). To clean up the mass spectrum, the identical phthalocyanine procedure was applied to the naphthalocyanine.

These reactions were scaled up to the 500 mg scale. See Supporting Information for synthetic protocol and crude yield.

CONCLUSION

All in all, we have adapted a one-pot synthesis of dichlorinated SiPcs and SiNcs using LiHMDS in place of ammonia gas, aiming towards a safer and more sustainable alternative to the traditional protocols. Our method is not only applicable to both inactivated phthalonitrile and 2,3-dicyanonaphthalene precursors but achieves up to a 45% macrocyclic conversion, which is up to a 2-fold increase in comparison to reported yields and via a microwave process, ~45-60% macrocyclic conversion was also achieved. While further optimization towards higher yields and improved purification strategies remains an open direction, this method provides a safe and practical alternative for access to dichlorinated SiPcs and SiNcs, which further downstream, grants strategies towards applications in organic electronics, photocatalysis, and photodynamic therapy.

ABBREVIATIONS

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SiPc, silicon phthalocyanine; SiNc, silicon naphthalocyanine; OPVs, organic photovoltaics; OTFTs, organic thin film transistors; PDT, photodynamic therapy' LIHMDS, lithium bis(trimethylsilyl) amide; DII, 1,3-diiminoisoindoline; MALDI-TOF MS, matrix-assisted laser desorption/ionization time-of-flight mass spectrometry; UV, ultraviolet.

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Data availability statement

The authors confirm that the data supporting the findings of this study are available within the article and its Supplementary Information.