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蓝光诱导的 α-硅氧基卡宾与 1,3-二酮 C—H 键的形式上插入反应

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摘要 报道了一种在蓝光诱导下, 酰基硅转化为 α-硅氧基卡宾, 进而与 1,3-二酮的烯醇式互变异构体发生的形式上的 C—H 键插入反应. 该方法使用简便易得、相对稳定的酰基硅作为卡宾前体, 反应条件温和, 无需使用任何过渡金属催化剂. 此外, 反应可以在克级规模下高收率地得到插入产物, 体现了反应的实用性及其在合成上的应用潜力.
关键词 光诱导; α-硅氧基卡宾; 1,3-二酮; C—H 键插入反应

Blue Light-Induced Formal Insertion Reaction of α-Siloxy Carbene into C—H Bond of 1,3-Diketones

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Abstract A blue light-induced formal insertion reaction of α-siloxy carbene into the C—H bond of 1,3-diketones has been reported. Under the irradiation of blue light, acylsilane converts to α-siloxy carbene, which then undergoes formal C—H bond insertion reaction with the enol form of 1,3-diketone. This method uses readily available and relative stable acylsilane as carbene precursor, which features a simple and metal-free approach under mild conditions. Moreover, the synthetic potential of this protocol has been demonstrated by performing the reaction on a gram scale with comparable high yield.
Keywords photoinduction; α-siloxy carbene; 1,3-diketone; C—H bond insertion reaction

1 Introduction

Acylsilanes are readily available reagents with a silyl group connected to a carbonyl group, which have been used in a variety of reactions as a relative stable synthetic building block.[1] For example, the electron-withdrawing ability of the carbonyl unit leads to the α-H acidic and enables the formation of an enol form intermediate.[2] On the other hand, under heating or irradiation with light, siloxy carbene can be generated smoothly through a 1,2-silyl transfer process. The pioneering work in this area can be traced back to 1967, when Brook and Duff[3] reported that 1,2-silyl transfer occurred under irradiation with UV-light, resulting the generation of α-siloxy carbenes, which underwent an O—H bond insertion reaction. With the development of visible light-induced reactions,[4] it has been found that acylsilanes can also form α-siloxy carbenes under the irradiation of visible light, which offers the opportunity to exploration of novel carbene chemistry under mild conditions.[5] The generally accepted mechanism for the generation of α-siloxy carbene is via the excited triplet state of acylsilane that derived from the excited singlet state through intersystem crossing (ISC) process, followed by a 1,2-Brook rearrangement, leading to the key triplet state α-siloxy carbene intermediate. Then, the triplet α-siloxy carbene led to the singlet state carbene via ISC.[6] Omitting the process of ISC can simplify the mechanism to the schematic diagram as shown in Scheme 1a. Like other carbene species, singlet α-siloxy carbene undergoes typical carbene transfer reactions under corresponding conditions, such as O—H bond insertion,[3,7] N—H bond insertion,[8] B—H bond insertion,[9] P—H bond insertion,[10] cyclization reaction,[11] and others,[12] which provides a practical and efficient method for the construction of silicon-containing molecules with structural diversity.

1,3-Diketones are one of the commonly used materials in synthetic chemistry.[13] In recent years, the reaction between 1,3-diketones and carbene intermediates has attracted in-

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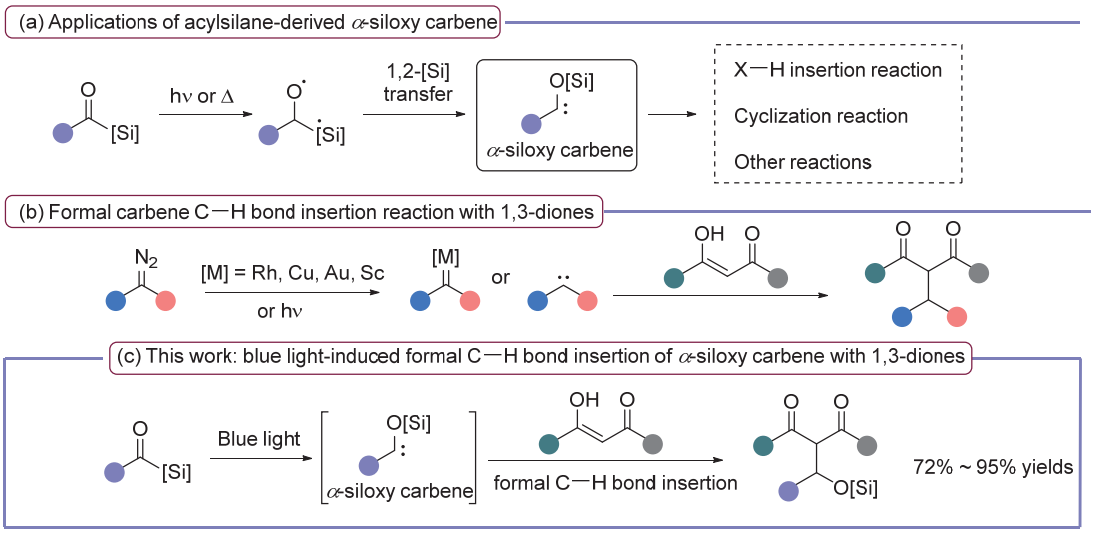


Scheme 1 Generation and application of α-siloxy carbene & formal carbene C—H bond insertion

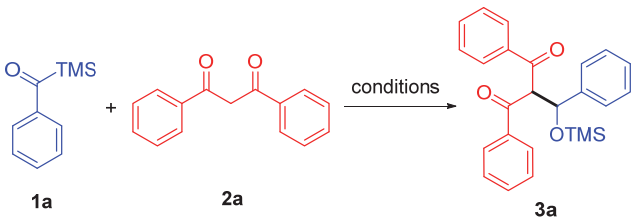
creasing attention, which directly forms C—C bond via C—H bond insertion reactions (Scheme 1, b). Due to the electron-withdrawing property of the bicarbonyl group, the C—H bond of the enol formula of 1,3-diketones is prone to transition metal-catalyzed carbene insertion reactions, such as Rh,[14] Cu,[15] Au[16] or Sc[17] catalysis, resulting the C-alkylation products. On the other hand, free carbene intermediates generated from diazo compounds under visible light irradiation[18] can lead to various transformations, including the C—H bond insertion reaction with 1,3-diketones.[19] These methods provide alternative approaches for the construction of alkylated 1,3-diketones under mild conditions.

Inspired by above advances and as our ongoing interest in the catalytic carbene transformations with different precursors, we herein report a blue light-induced C—H bond insertion reaction of α-siloxy carbene that derived from acylsilane. To the best of our knowledge, this is the only example of C—H bond insertion reaction using acylsilane as the carbene precursor, which features a simple and metal-free approach for the direct access to the alkylated 1,3-diketones under mild conditions (Scheme 1, c). Moreover, the synthetic potential of this protocol has been demonstrated by performing the reaction on a gram scale with comparable high yield.

2 Results and discussion

The initial investigation using phenyl(trimethylsilyl)-methanone (**1a**) and 1,3-diphenylpropane-1,3-dione (**2a**) as model substrates in 1,2-dichloroethane (DCE) under 427 nm blue LED at room temperature for 4 h afforded 76% yield of the expected product **3a** (Table 1, Entry 1). Subsequently, the effect of the light source was investigated (Entries 2～5), however, no improved result has been obtained in term of yield. It's worth mentioning that the target product was not obtained when the reaction was conducted in the absence of light, implying that the reaction was driven by visible light. Next, varying the ratios of the two substrates was studied to determine the best ratio for this reaction, and higher yield was recorded with the ratio 1.5∶1 of **1a**∶**2a** (Entries 6 and 7). An evaluation of different solvents, such as dichloromethane (DCM), $CHCl_3$, hexane, xylene, toluene, PhCl, tetrahydrofuran (THF), and *N*,*N*-di-

Table 1 Optimization of the reaction conditions[a]



1a **2a** **3a**

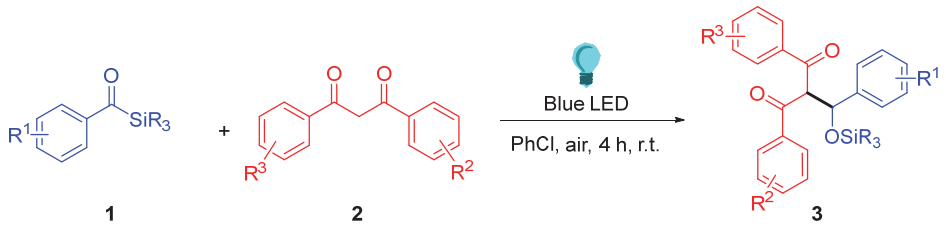
Entry	Light source	**1a**∶**2a**	Solvent	Yield[b]/%
1	10 W, 427 nm	1∶1	DCE	76
2	Dark	1∶1	DCE	0
3	10 W, 390 nm	1∶1	DCE	43
4	10 W, 450 nm	1∶1	DCE	52
5	10 W, 470 nm	1∶1	DCE	47
6	10 W, 427 nm	1.5∶1	DCE	86
7	10 W, 427 nm	2∶1	DCE	86
8	10 W, 427 nm	1.5∶1	DCM	83
9	10 W, 427 nm	1.5∶1	$CHCl_3$	78
10	10 W, 427 nm	1.5∶1	Hexane	53
11	10 W, 427 nm	1.5∶1	Xylene	61
12	10 W, 427 nm	1.5∶1	Toluene	50
13	10 W, 427 nm	1.5∶1	PhCl	94
14	10 W, 427 nm	1.5∶1	THF	23
15	10 W, 427 nm	1.5∶1	DMF	15
16[c]	10 W, 427 nm	1.5∶1	PhCl	93
17[c,d]	10 W, 427 nm	1.5∶1	PhCl	92
18[c,e]	10 W, 427 nm	1.5∶1	PhCl	81

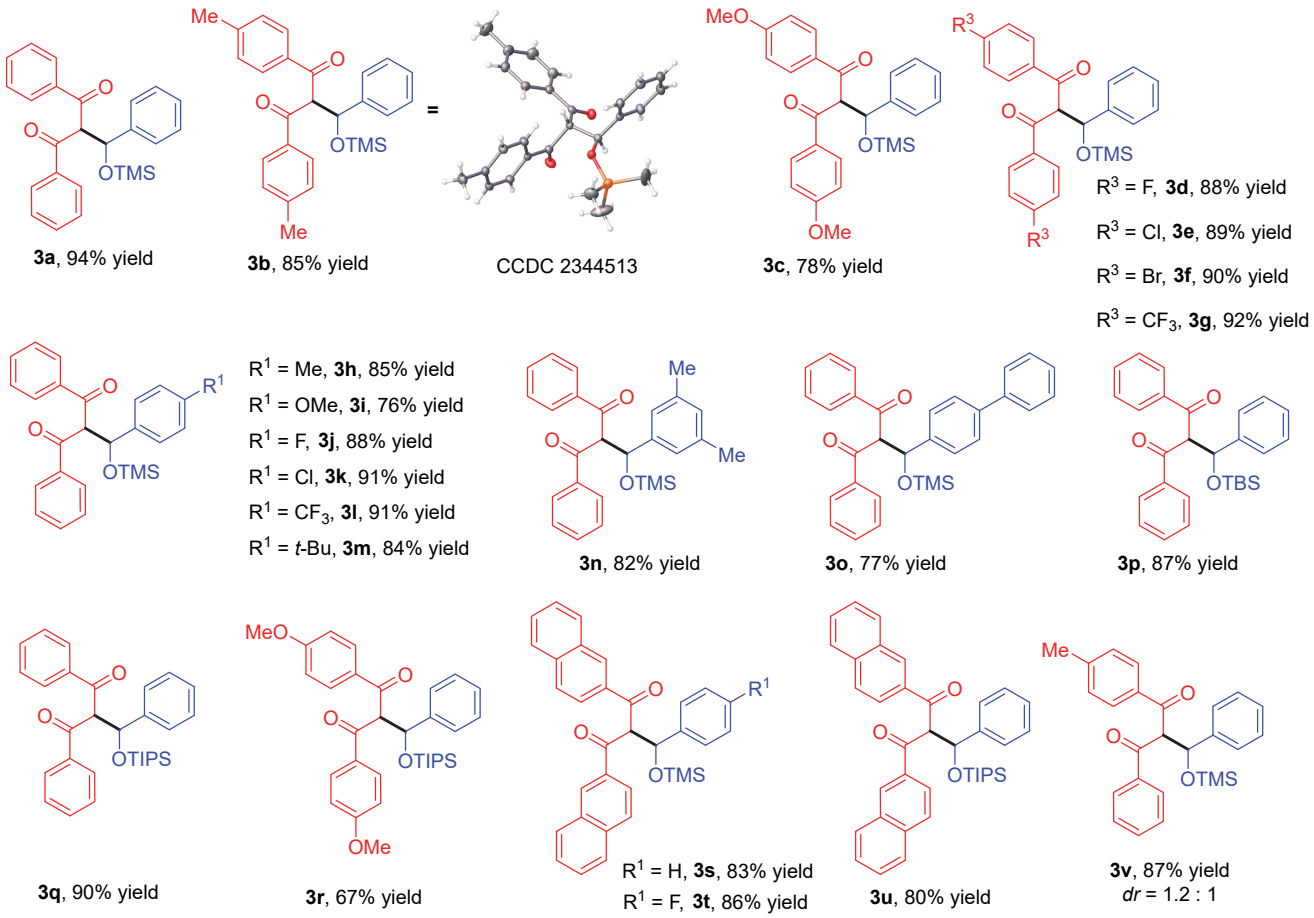
[a] Unless otherwise noted, the reactions were carried out with acylsilane **1a** (26.7 mg, 0.15 mmol), 1,3-diketone **2a** (22.4 mg, 0.1 mmol) in indicated solvent (1.0 mL) under the light irradiation (1×10 W, 427 nm) and argon atmosphere at 25 ℃ for 4 h. [b] Isolated yields of **3a**. [c] Under the air. [d] Running for 6 h. [e] Running for 3 h.

methyl formamide (DMF), revealed that the reaction could be carried out in most of these tested solvents (Entries 8～15), and PhCl proved to be the most efficient one, affording the product **3a** in 94% yield (Entry 13). Additionally, the reaction was not affected at all without the protection of argon atmosphere, producing **3a** in comparable high yield under the air atmosphere (Entry 16). Whiles, the yields dropped when the reaction time was shortened (Entries 17 and 18).

With the obtained optimal reaction conditions, the substrate generality of this transformation was investigated and the results are summarized in Table 2. The protocol showed good tolerance for substituted 1,3-diphenylpropane-1,3-diones **2** bearing electron-neutral (Me), electron-donating (OMe) and electron-withdrawing (F, Cl, Br and CF_3) groups. All the reactions proceeded smoothly to afford the desired products in good yields (**3b**～**3g**, 78%～92% yields). The structure of **3b** was determined by X-ray crystallography analysis. The reaction could be successfully applied to substrates bearing different substituents on the *para* position of phenyl(trimethylsilyl)methanone (**1a**). For example, derivatives containing electron-neutral (Me), electron-donating (OMe), electron-withdrawing (CF_3) and halogen (F, Cl) groups, could effectively generate products **3h**～**3l** in 76%～91% yields. For substrates **1** containing a steric bulky tertiary butyl, 3,5-dimethyl or additional phenyl substituent on the *para*-position of the benzene ring, the reaction all performed well with **2a**, generating corresponding target products **3m**～**3o** 77%～84% yield under current conditions. The *tert*-butyl silyl (TBS) and triiso-

Table 2 Substrate scope for the formal C—H insertion[a]

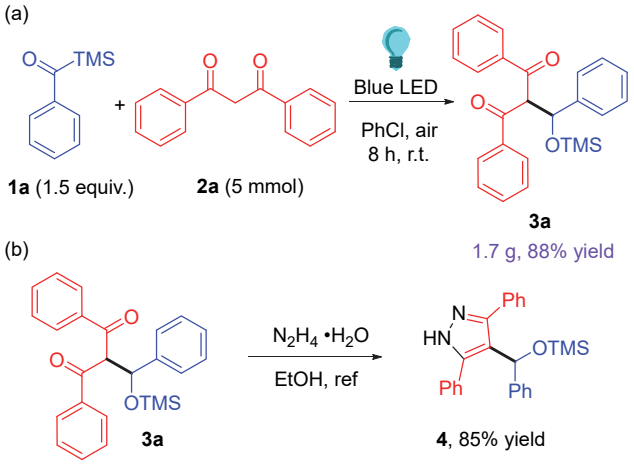




[a] Reaction conditions: acylsilanes **1** (0.15 mmol), 1,3-diketones **2** (0.1 mmol) in PhCl (1.0 mL) under the light irradiation (1×10 W, 427 nm) and air atmosphere at 25 ℃ for 4 h; The yields are given in isolated yields; TMS=trimethylsilyl.

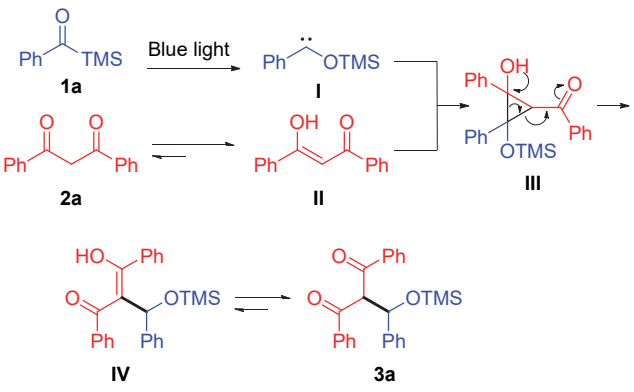
propylsilyl (TIPS) substituted analogues, (*tert*-butyldimethylsilyl)(phenyl)methanone (**1p**) and phenyl(triisopropylsilyl)methanone (**1q**), also proved suitable for this reaction and afforded the desired products **3p** and **3q** in 87% and 90% yields, respectively. In addition, the target products **3s**～**3u** derived from 2-naphthyl 1,3-diketone could be obtained in ＞80% yields. 1,3-Diketones with asymmetric structure were also suitable for this reaction, providing product **3v** in 87% yield with1.2∶1 *dr*.

To show the synthetic potential of this method, gram-scale reaction was conducted with **1a** and **2a** under standard conditions. To our delight, the desired product **3a** was isolated in 88% (1.57 g) yield (Scheme 2, a). Besides, we have successfully synthesized heterocyclic compound **4** containing siloxy fragment based on the condensation reaction of the 1,3-bicarbonyl unit of product **3a** with hydrazine hydrate (Scheme 2, b). In addition, we also attempted asymmetric catalysis, but unfortunately, only moderate enantioselectivity was observed due to the strong background reaction (see Table S1 in Supporting Information for details).



Scheme 2 Gram-scale reaction and synthetic transformation

A plausible reaction mechanism for the C—H bond insertion reaction has been proposed in Scheme 3 according to the reported literature. Initially, direct excitation of acylsilane **1a** by visible light generates an excited state, which then produces siloxy carbene **I** through a 1,2-silyl transfer process.[6] Subsequently, cyclopropanation of **I** with enol form species **II** of 1,3-diketone leads to the intermediate **III**. Finally, ring opening of this cyclopropane structure forms intermediate **IV**,[19-20] followed by keto-enol tautomerization, giving the target product **3a**.



Scheme 3 Proposed reaction mechanism

3 Conclusions

In summary, a blue light-induced formal insertion reaction of *in situ* formed *α*-siloxy carbene into the C—H bond of the enol forms of 1,3-diketones has been developed. A variety of siloxy-substituted 1,3-diketones have been obtained in good to high yields under mild and metal-free conditions. Gram-scale synthesis and synthetic transformation demonstrated the potential practicality of this method.

4 Experimental section

4.1 Instruments and reagents

^{1}H NMR, ^{13}C NMR and ^{19}F NMR were performed by Bruker 500 MHz/Avance III or Bruker 400 MHz/Avance III nuclear magnetic resonance spectrometer. The internal standard is tetramethylsilane (TMS), and the solvent is deuterated chloroform ($CDCl_3$). ESI high-resolution mass spectrometry was determined by an Agilent ultra-high performance liquid chromatography. The melting point was determined by a microscope melting point analyzer (X4) from Beijing Optical Instrument Factory. Blue light source proposed by a Kessil lamp (10 W, 427 nm). The other instruments used are the German IKA C-MAG HS7 magnetic stirrer, German IKA RV 8 rotary evaporator, and 10 mL Synthware screw sample bottle (high type).

All reagents are commercially available analytical reagents (AR). The starting materials for the synthesis of various substrates **1** and **2** are all purchased from commercially available sources (Shanghai Energy Pharmaceutical Chemistry Co., Ltd or Shanghai Bide Pharmatech Co., Ltd.). Column chromatography silica gel (200～300 mesh) and thin layer silica gel plate (TLC) purchased from Yantai Jiangyou Silicone Development Co., Ltd. The developing agents are industrial grade petroleum ether (PE) and ethyl acetate (EA).

4.2 General procedure for the synthesis of products 3

To a 25 mL round-bottom flask, acylsilanes **1** (0.15 mmol), 1,3-diketones **2** (0.1 mmol) and PhCl (2.0 mL) were added sequentially. Then, the mixture was stirred at room temperature under the blue light irradiation (1×10 W, 427 nm) and air atmosphere for 4 h. After the reaction was completed, the reaction mixture was purified directly by column chromatography on silica gel (eluent: PE/EA, *V*∶*V*=30∶1 to 10∶1) without any additional treatment to give pure products **3** in good to high yields.

1,3-Diphenyl-2-(phenyl((trimethylsilyl)oxy)methyl)pro-

pane-1,3-dione (**3a**): Colourless oil, 37.8 mg, 94% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.26 (d, J=7.7 Hz, 2H), 7.87 (d, J=7.8 Hz, 2H), 7.79～7.73 (m, 1H), 7.67 (d, J=7.8 Hz, 4H), 7.57 (d, J=7.7 Hz, 1H), 7.42 (q, J=7.0 Hz, 4H), 7.36～7.30 (m, 1H), 6.03～5.91 (m, 2H), 0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.3, 193.2, 142.8, 138.0, 136.8, 133.30, 133.27, 129.2, 128.7, 128.6, 128.5, 128.3, 127.9, 127.2, 75.8, 65.9, −0.2; HRMS (TOF MS ESI$^+$) calculated for $C_{25}H_{26}NaO_3Si$ $[M+Na]^+$ 425.1543, found 425.1550.

2-(Phenyl((trimethylsilyl)oxy)methyl)-1,3-di-*p*-tolylpropane-1,3-dione (**3b**): Yellow solid, 36.6 mg, 85% yield. m.p. 192.5～193.6 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.16 (d, J=8.3 Hz, 2H), 7.77 (d, J=8.3 Hz, 2H), 7.67 (dd, J=8.2, 1.3 Hz, 2H), 7.45～7.37 (m, 4H), 7.34～7.28 (m, 1H), 7.20 (d, J=8.0 Hz, 2H), 5.99～5.90 (m, 2H), 2.57 (s, 3H), 2.42 (s, 3H), 0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 193.8, 192.8, 144.1, 144.0, 142.9, 135.5, 134.3, 129.4, 129.28, 129.26, 128.6, 128.2, 127.8, 127.2, 75.7, 21.8, 21.6, −0.2; HRMS (TOF MS ESI$^+$) calcd for $C_{27}H_{30}NaO_3Si$ $[M+Na]^+$ 453.1856, found 453.1857.

1,3-Bis(4-methoxyphenyl)-2-(phenyl((trimethylsilyl)oxy)methyl)propane-1,3-dione (**3c**): Yellow oil, 36.1 mg, 78% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.41～8.33 (m, 2H), 8.02～7.94 (m, 2H), 7.77 (d, J=7.0 Hz, 2H), 7.55～7.49 (m, 2H), 7.46～7.40 (m, 1H), 7.26～7.21 (m, 2H), 7.05～6.98 (m, 2H), 6.06～5.94 (m, 2H), 4.16 (s, 3H), 4.03 (s, 3H), 0.11 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 192.8, 191.8, 163.6, 163.6, 143.1, 131.5, 131.1, 130.9, 129.9, 128.2, 127.8, 127.2, 113.9, 113.8, 75.6, 65.6, 55.6, 55.5, −0.1; HRMS (TOF MS ESI$^+$) calcd for $C_{27}H_{30}NaO_5Si$ $[M+Na]^+$ 485.1755, found 485.1758.

1,3-Bis(4-fluorophenyl)-2-(phenyl((trimethylsilyl)oxy)methyl)propane-1,3-dione (**3d**): Yellow oil, 38.6 mg, 88% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.32～8.26 (m, 2H), 7.92～7.85 (m, 2H), 7.67～7.61 (m, 2H), 7.45～7.39 (m, 2H), 7.37～7.31 (m, 3H), 7.14～7.07 (m, 2H), 5.95～5.83 (m, 2H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 192.6, 191.5, 167.2, 167.1, 164.7, 164.6, 142.5, 134.3, 134.2, 133.1, 133.0, 131.84, 131.75, 131.2, 131.1, 128.4, 128.1, 127.1, 116.1, 116.0, 115.9, 115.8, 75.7, 66.0, −0.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −104.29, −104.58; HRMS (TOF MS ESI$^+$) calcd for $C_{25}H_{24}NaF_2O_3Si$ $[M+Na]^+$ 461.1355, found 461.1357.

1,3-Dis(4-chlorophenyl)-2-(phenyl((trimethylsilyl)oxy)methyl)propane-1,3-dione (**3e**): Yellow oil, 42.0 mg, 89% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.18 (d, J=8.7 Hz, 2H), 7.77 (d, J=8.6 Hz, 2H), 7.67～7.61 (m, 4H), 7.44～7.38 (m, 4H), 7.35 (dd, J=8.4, 6.2 Hz, 1H), 5.87 (q, J=9.4 Hz, 2H), 0.00 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 192.9, 191.8, 142.4, 140.04, 140.00, 136.1, 134.9, 130.5, 129.8, 129.2, 129.17, 129.15, 128.4, 128.1, 127.1, 75.7, 65.9, −0.2; HRMS (TOF MS ESI$^+$) calcd for $C_{25}H_{24}NaCl_2O_3Si$ $[M+Na]^+$ 493.0764, found 493.0764.

1,3-Bis(4-bromophenyl)-2-(phenyl((trimethylsilyl)oxy)methyl)propane-1,3-dione (**3f**): Yellow solid, 50.4 mg, 90% yield. m.p. 197.0～197.6 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.09 (d, J=8.6 Hz, 2H), 7.80 (d, J=8.5 Hz, 2H), 7.68 (d, J=8.6 Hz, 2H), 7.63 (d, J=7.2 Hz, 2H), 7.56 (d, J=8.6 Hz, 2H), 7.44～7.39 (m, 2H), 7.37～7.31 (m, 1H), 5.91～5.80 (m, 2H), 0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 193.1, 191.9, 142.4, 136.5, 135.3, 132.2, 132.14, 132.07, 132.04, 132.01, 130.6, 129.9, 128.84, 128.80, 128.78, 128.4, 128.2, 127.1, 75.7, 65.9, −0.2; HRMS (TOF MS ESI$^+$) calcd for $C_{25}H_{24}NaBr_2O_3Si$ $[M+Na]^+$ 582.9734, found 582.9735.

2-(Phenyl((trimethylsilyl)oxy)methyl)-1,3-bis(4-(trifluoromethyl)phenyl)propane-1,3-dione (**3g**): Yellow solid, 49.5 mg, 92% yield. m.p. 183.0～183.8 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.35 (d, J=8.1 Hz, 2H), 7.97～7.87 (m, 4H), 7.67 (dd, J=17.3, 7.5 Hz, 4H), 7.42 (t, J=7.4 Hz, 2H), 7.38～7.31 (m, 1H), 6.00～5.89 (m, 2H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 193.2, 192.0, 142.2, 140.5, 139.1, 134.92, 133.85, 134.7, 134.6, 129.4, 128.7, 128.6, 128.4, 127.1, 126.0, 125.92, 125.86, 125.8, 124.7, 124.5, 122.6, 122.4, 75.8, 66.4, −0.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −63.18, −63.36; HRMS (TOF MS ESI$^+$) calcd for $C_{27}H_{24}F_6NaO_3Si$ $[M+Na]^+$ 561.1292, found 561.1293.

1,3-Diphenyl-2-(*p*-tolyl((trimethylsilyl)oxy)methyl)propane-1,3-dione (**3h**). Yellow oil, 35.4 mg, 85% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.31～8.25 (m, 2H), 7.89 (dd, J=8.4, 1.3 Hz, 2H), 7.78～7.73 (m, 1H), 7.69～7.63 (m, 2H), 7.57 (t, J=7.5 Hz, 3H), 7.45～7.40 (m, 2H), 7.23 (d, J=7.9 Hz, 2H), 6.01 (d, J=9.5 Hz, 1H), 5.92 (d, J=9.4 Hz, 1H), 2.42 (s, 3H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.4, 193.2, 139.8, 138.0, 137.5, 136.7, 133.2, 129.1, 128.9, 128.7, 128.6, 128.5, 127.1, 75.7, 65.9, 21.2, −0.2; HRMS (TOF MS ESI$^+$) calcd for $C_{26}H_{28}NaO_3Si$ $[M+Na]^+$ 439.1700, found 439.1700.

2-((4-Methoxyphenyl)((trimethylsilyl)oxy)methyl)-1,3-diphenylpropane-1,3-dione (**3i**): Yellow oil, 32.9 mg, 76% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.31～8.26 (m, 2H), 7.93～7.87 (m, 2H), 7.79～7.74 (m, 1H), 7.67 (tt, J=6.6, 1.4 Hz, 2H), 7.63～7.56 (m, 3H), 7.47～7.42 (m, 2H), 7.00～6.92 (m, 2H), 6.00 (d, J=9.5 Hz, 1H), 5.90 (d, J=9.5 Hz, 1H), 3.91 (s, 3H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.4, 193.2, 159.2, 138.0, 136.7, 135.0, 133.3, 133.2, 129.1, 128.7, 128.6, 128.5, 128.4, 113.6, 75.5, 66.0, 55.2, −0.2; HRMS (TOF MS ESI$^+$) calcd for $C_{26}H_{28}NaO_4Si$ $[M+Na]^+$ 455.1649, found 455.1655.

2-((4-Fluorophenyl)((trimethylsilyl)oxy)methyl)-1,3-diphenylpropane-1,3-dione (**3j**): Colourless oil, 37.0 mg, 88% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.30～8.21 (m, 2H), 7.93～7.84 (m, 2H), 7.79～7.74 (m, 1H), 7.66 (ddd, J=8.7, 6.1, 2.1 Hz, 4H), 7.63～7.57 (m, 1H), 7.48～7.43 (m, 2H), 7.10 (t, J=8.7 Hz, 2H), 6.00～5.88 (m, 2H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.1, 193.2, 163.6, 161.2, 138.73, 138.70, 137.9, 136.6, 133.5, 133.4, 132.6, 129.1, 129.0, 128.9, 128.83, 128.77, 128.5, 127.3, 115.3, 115.0, 75.1, 66.0, −0.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −114.50. HRMS (TOF MS ESI$^+$) calcd for $C_{25}H_{25}NaFO_3Si$ $[M+Na]^+$ 443.1449, found 443.1447.

2-((4-Chlorophenyl)((trimethylsilyl)oxy)methyl)-1,3-diphenylpropane-1,3-dione (**3k**): Colourless oil, 39.8 mg,

91% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.26 (d, J=7.2 Hz, 2H), 7.87 (d, J=7.2 Hz, 2H), 7.77 (t, J=7.4 Hz, 1H), 7.68 (d, J=7.6 Hz, 2H), 7.62 (dd, J=13.8, 7.9 Hz, 3H), 7.48～7.38 (m, 4H), 5.93 (d, J=1.6 Hz, 2H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.0, 193.1, 141.5, 137.8, 136.5, 133.6, 133.5, 133.4, 129.2, 128.79, 128.76, 128.7, 128.5, 127.3, 75.1, 65.8, −0.2; HRMS (TOF MS ESI$^+$) calcd for $C_{25}H_{25}NaClO_3Si$ $[M+Na]^+$ 459.1154, found 459.1150.

1,3-Diphenyl-2-((4-(trifluoromethyl)phenyl)((trimethylsilyl)oxy)methyl)propane-1,3-dione (**3l**): Yellow oil, 42.8 mg, 91% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.25 (d, J=7.3 Hz, 2H), 7.88～7.73 (m, 5H), 7.66 (t, J=6.8 Hz, 4H), 7.59 (t, J=7.4 Hz, 1H), 7.43 (t, J=7.7 Hz, 2H), 6.05～5.89 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 193.9, 193.0, 146.9, 137.7, 136.4, 133.6, 133.5, 132.6, 130.2, 130.1, 130.0, 129.2, 128.84, 128.77, 128.5, 127.6, 127.3, 125.33, 125.30, 123.1, 75.0, 65.6, −0.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −62.57; HRMS (TOF MS ESI$^+$) calcd for $C_{26}H_{25}F_3NaO_3Si$ $[M+Na]^+$ 493.1418, found 493.1421.

2-((4-(*tert*-Butyl)phenyl)((trimethylsilyl)oxy)methyl)-1,3-diphenylpropane-1,3-dione (**3m**): Yellow oil, 38.5 mg, 84% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.27～8.21 (m, 2H), 7.87～7.79 (m, 2H), 7.75～7.70 (m, 1H), 7.63 (t, J=7.7 Hz, 2H), 7.58～7.51 (m, 3H), 7.39 (t, J=7.7 Hz, 4H), 5.97 (d, J=9.4 Hz, 1H), 5.90 (d, J=9.4 Hz, 1H), 1.38 (s, 9H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.3, 193.4, 150.7, 139.6, 138.0, 136.7, 133.2, 133.1, 129.1, 128.7, 128.5, 126.7, 125.1, 75.5, 65.8, 34.5, 31.4, −0.2; HRMS (TOF MS ESI$^+$) calcd for $C_{29}H_{34}NaO_3Si$ $[M+Na]^+$ 481.2169, found 481.2172.

2-((3,5-Dimethylphenyl)((trimethylsilyl)oxy)methyl)-1,3-diphenylpropane-1,3-dione (**3n**): Yellow oil, 35.3 mg, 82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29～8.23 (m, 2H), 7.88 (dd, J=8.5, 1.3 Hz, 2H), 7.77～7.71 (m, 1H), 7.67～7.61 (m, 2H), 7.59～7.54 (m, 1H), 7.42 (t, J=7.8 Hz, 2H), 7.24 (s, 2H), 6.93 (s, 1H), 5.98 (d, J=9.4 Hz, 1H), 5.85 (d, J=9.4 Hz, 1H), 2.40 (d, J=0.8 Hz, 6H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.4, 193.2, 142.5, 138.0, 137.6, 136.8, 133.2, 129.5, 129.1, 128.7, 128.5, 125.0, 75.8, 65.8, 21.4, −0.1; HRMS (TOF MS ESI$^+$) calcd for $C_{27}H_{30}NaO_3Si$ $[M+Na]^+$ 453.1856, found 453.1855.

2-([1,1'-Biphenyl]-4-yl((trimethylsilyl)oxy)methyl)-1,3-diphenylpropane-1,3-dione (**3o**): Colourless oil, 36.9 mg, 77% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29～8.23 (m, 2H), 7.88～7.83 (m, 2H), 7.75～7.69 (m, 3H), 7.66～7.60 (m, 6H), 7.55～7.49 (m, 3H), 7.46～7.41 (m, 1H), 7.40～7.34 (m, 2H), 6.04～5.94 (m, 2H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.2, 193.2, 141.9, 140.8, 140.6, 137.9, 136.7, 133.32, 133.30, 129.1, 128.79, 128.75, 128.7, 128.6, 128.5, 127.6, 127.3, 127.1, 127.0, 75.5, 65.8, −0.2; HRMS (TOF MS ESI$^+$) calcd for $C_{31}H_{30}NaO_3Si$ $[M+Na]^+$ 501.1856, found 501.1857.

2-(((*tert*-Butyldimethylsilyl)oxy)(phenyl)methyl)-1,3-diphenylpropane-1,3-dione (**3p**): Yellow oil, 38.7 mg, 87% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.32～8.27 (m, 2H), 7.87～7.81 (m, 2H), 7.78～7.72 (m, 1H), 7.71～7.63 (m, 4H), 7.58～7.52 (m, 1H), 7.41 (td, J=7.7, 7.2, 2.4 Hz, 4H), 7.35～7.29 (m, 1H), 6.04 (d, J=9.4 Hz, 1H), 5.94 (d, J=9.4 Hz, 1H), 0.75 (s, 9H), −0.00 (s, 3H), −0.14 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 192.9, 191.9, 142.0, 136.8, 135.7, 132.4, 132.3, 128.3, 127.8, 127.6, 127.4, 127.2, 126.9, 126.4, 75.2, 64.4, 24.5, 16.9, −5.7, −6.5; HRMS (TOF MS ESI$^+$) calcd for $C_{28}H_{32}NaO_3Si$ $[M+Na]^+$ 467.2013, found 467.2019.

1,3-Diphenyl-2-(phenyl((triisopropylsilyl)oxy)methyl)-propane-1,3-dione (**3q**): Yellow oil, 43.8 mg, 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.18～8.10 (m, 2H), 7.71～7.65 (m, 2H), 7.62～7.56 (m, 1H), 7.55～7.47 (m, 4H), 7.39 (t, J=7.4 Hz, 1H), 7.24 (tt, J=7.5, 3.3 Hz, 4H), 7.18～7.12 (m, 1H), 5.98～5.90 (m, 2H), 0.87～0.75 (m, 21H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 193.8, 192.9, 143.1, 137.8, 136.8, 133.4, 133.2, 129.3, 128.8, 128.5, 128.4, 128.2, 128.0, 127.6, 76.3, 65.7, 17.9, 17.8, 12.5; HRMS (TOF MS ESI$^+$) calcd for $C_{31}H_{38}NaO_3Si$ $[M+Na]^+$ 509.2482, found 509.2478.

1,3-Bis(4-methoxyphenyl)-2-(phenyl((triisopropylsilyl)-oxy)methyl)propane-1,3-dione (**3r**): Yellow oil, 36.6 mg, 67% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.13 (d, J=8.5 Hz, 2H), 7.70 (d, J=8.6 Hz, 2H), 7.53 (d, J=7.5 Hz, 2H), 7.23 (q, J=8.5, 7.5 Hz, 2H), 7.14 (t, J=7.3 Hz, 1H), 6.96 (d, J=8.6 Hz, 2H), 6.72 (d, J=8.5 Hz, 2H), 5.93 (d, J=9.0 Hz, 1H), 5.80 (d, J=9.1 Hz, 1H), 3.88 (s, 3H), 3.74 (s, 3H), 0.87～0.77 (m, 21H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 192.5, 191.6, 163.7, 163.5, 143.4, 131.6, 131.0, 130.8, 129.9, 128.1, 127.8, 127.6, 113.9, 113.7, 76.1, 65.5, 55.6, 55.5, 18.0, 17.9, 12.6; HRMS (TOF MS ESI$^+$) calcd for $C_{33}H_{42}NaO_5Si$ $[M+Na]^+$ 569.2694, found 569.2696.

1,3-Di(naphthalen-2-yl)-2-(phenyl((trimethylsilyl)oxy)-methyl)propane-1,3-dione (**3s**): Yellow solid, 41.7 mg, 83% yield. m.p. 190.7～191.7 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.80 (d, J=1.8 Hz, 1H), 8.38 (d, J=1.8 Hz, 1H), 8.30 (dd, J=8.6, 1.8 Hz, 1H), 8.09～7.99 (m, 3H), 7.93 (dd, J=8.7, 1.8 Hz, 1H), 7.87～7.78 (m, 3H), 7.75～7.49 (m, 6H), 7.42～7.36 (m, 2H), 7.30～7.24 (m, 1H), 6.26 (d, J=9.4 Hz, 1H), 6.06 (d, J=9.3 Hz, 1H), −0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.2, 193.1, 142.8, 135.7, 135.5, 135.4, 134.1, 132.6, 132.3, 131.0, 130.5, 129.9, 129.7, 128.8, 128.7, 128.64, 128.55, 128.3, 128.0, 127.9, 127.7, 127.2, 126.9, 126.8, 124.7, 124.0, 75.9, 66.3, −0.1; HRMS (TOF MS ESI$^+$) calcd for $C_{33}H_{30}NaO_3Si$ $[M+Na]^+$ 525.1857, found 525.1859.

2-((4-Fluorophenyl)((trimethylsilyl)oxy)methyl)-1,3-di(naphthalen-2-yl)propane-1,3-dione (**3t**): Yellow solid, 44.8 mg, 86% yield. m.p. 183.2～184.3 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.68 (d, J=2.2 Hz, 1H), 8.27 (d, J=1.8 Hz, 1H), 8.16 (dd, J=8.6, 1.8 Hz, 1H), 7.98～7.88 (m, 3H), 7.81 (dd, J=8.6, 1.8 Hz, 1H), 7.78～7.67 (m, 3H), 7.65～7.55 (m, 4H), 7.52 (ddd, J=8.2, 7.0, 1.4 Hz, 1H), 7.43 (ddd, J=8.2, 6.9, 1.3 Hz, 1H), 7.00～6.92 (m, 2H), 6.09 (d, J=9.4 Hz, 1H), 5.90 (d, J=9.3 Hz, 1H), −0.14 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.1, 193.1, 163.6, 161.1, 138.8, 138.7, 135.7, 135.6, 135.3, 134.0, 132.6, 132.3,

131.0, 130.6, 129.9, 129.7, 129.2, 129.0, 128.92, 128.88, 128.8, 128.72, 128.70, 127.9, 127.8, 127.02, 126.95, 124.7, 123.9, 115.3, 115.1, 75.2, 66.4, −0.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −114.36. HRMS (TOF MS ESI$^+$) calcd for $C_{33}H_{29}NaFO_3Si$ $[M+H]^+$ 543.1762, found 543.1763.

1,3-Di(naphthalen-2-yl)-2-(phenyl((triisopropylsilyl)-oxy)methyl)propane-1,3-dione (**3u**): Yellow solid, 46.9 mg, 80% yield. m.p. 191.2～192.1 ℃; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.78 (d, J=1.7 Hz, 1H), 8.31 – 8.26 (m, 1H), 8.23 (dd, J=8.6, 1.8 Hz, 1H), 7.99 (d, J=8.2 Hz, 1H), 7.95 (d, J=8.6 Hz, 1H), 7.90 (d, J=8.1 Hz, 1H), 7.81 (dd, J=8.7, 1.8 Hz, 1H), 7.72 (d, J=8.2 Hz, 1H), 7.68 (d, J=7.2 Hz, 4H), 7.62 (t, J=7.5 Hz, 1H), 7.57 (t, J=7.5 Hz, 1H), 7.49 (t, J=7.5 Hz, 1H), 7.41 (t, J=7.5 Hz, 1H), 7.29 (t, J=7.6 Hz, 2H), 7.17 (t, J=7.4 Hz, 1H), 6.26 (d, J=8.9 Hz, 1H), 6.13 (d, J=8.9 Hz, 1H), 0.92～0.80 (m, 21H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 193.9, 192.9, 143.2, 135.7, 135.5, 135.3, 134.1, 132.6, 132.3, 131.2, 130.4, 129.9, 129.6, 128.8, 128.72, 128.68, 128.5, 128.2, 128.0, 127.9, 127.7, 127.6, 127.0, 126.8, 124.8, 124.0, 76.4, 66.4, 18.0, 17.9, 12.6; HRMS (TOF MS ESI$^+$) calcd for $C_{39}H_{42}NaO_3Si$ $[M+H]^+$ 609.2795, found 609.2795.

1-Phenyl-2-(phenyl((trimethylsilyl)oxy)methyl)-3-(*p*-tolyl)propane-1,3-dione (**3v**): Yellow oil, 36.2 mg, 87% yield. A mixture of two isomers (1.2∶1). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.25 and 8.16 (two doubles, J=8.0 Hz, 2H), 7.86 and 7.77 (two doubles, J=8.0 Hz, 2H), 7.74～7.51 (comp, 8H), 7.42 (comp, 8H), 7.35～7.28 (m, 2H), 7.21 (d, J=8.0 Hz, 2H), 5.95 (comp, 4H), 2.59 (s, 3H), 2.44 (s, 2H), −0.00 (two singlets, 17H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.4, 193.8, 193.3, 192.7, 144.2, 144.1, 142.9, 142.8, 138.0, 136.8, 135.5, 134.3, 133.22, 133.19, 129.4, 129.34, 129.31, 129.1, 128.69, 128.65, 128.6, 128.5, 128.3, 127.9, 127.2, 75.8, 75.7, 65.8, 65.7, 21.8, 21.7, −0.18, −0.19; HRMS (TOF MS ESI$^+$) calcd for $C_{26}H_{28}NaO_3Si$ $[M+Na]^+$ 439.1700, found 439.1703.

4.2 General procedure for gram-scale synthesis

To a 100 mL round-bottom flask, acylsilane (**1a**, 7.5 mmol), 1,3-diketone (**2a**, 5.0 mmol) and PhCl (20 mL) were added sequentially. Then, the mixture was stirred at room temperature under the blue light irradiation (1×10 W, 427 nm) and air atmosphere for 8 h. After the reaction was completed, the solvent was evaporated under reduced pressure, and the reaction mixture was purified directly by column chromatography on silica gel (eluent: PE/EA, V∶V=30∶1 to 10∶1) without any additional treatment to give 1.7 g pure product **3a** in 88% yield.

4.3 General procedure for the synthesis of product **4**

To a solution of **3a** (161.0 mg, 0.4 mmol) in 96% ethanol (3 mL), was added aqueous hydrazine (206.0 mg, 6.4 mmol, 80% weight solution). The reaction mixture was refluxed for 10 min. Then, ethyl acetate (40 mL) was added and the organic phase was washed with 1 moll/L HCl (10 mL) and saturated brine (20 mL×3). The organic phase was then dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. The reaction mixture was purified by column chromatography on silica gel (eluent: PE/EA, V∶V 30∶1 to 10∶1) to give pure product **4** in 85% yield.

3,5-Diphenyl-4-(phenyl((trimethylsilyl)oxy)methyl)-1*H*-pyrazole (**4**):[21] Yellow oil, 135.5 mg, 85% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.12～8.09 (m, 1H), 7.93 (dd, J=7.7, 1.9 Hz, 4H), 7.74～7.67 (m, 8H), 7.62 (t, J=7.4 Hz, 2H), 7.56 (t, J=7.2 Hz, 1H), 6.54 (s, 1H), 0.27 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.2, 131.8, 128.92, 128.87, 128.3, 128.2, 127.9, 126.7, 126.4, 125.8, 118.5, 67.8, −0.1; HRMS (TOF MS ESI$^+$) calcd for $C_{25}H_{27}N_2OSi$ $[M+H]^+$ 399.1887, found 399.1886.

Supporting Information General Information, general procedure for the preparation of acylsilane and 1,3-diketones, ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra of products and single-crystal X-ray diffraction. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn.

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