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## 3*H*-Phenothiazin-3-one: a photocatalyst for the mild oxidation of boronic acids under green LED irradiation

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3*H*-Phenothiazin-3-one is a small organic molecule with established biological properties, yet its potential as an organic photocatalyst remains largely unexplored. Herein, we report a green and efficient aerobic oxidation of boronic acids employing 3*H*-phenothiazin-3-one as the photocatalyst. The transformation proceeds under mild conditions, using green LED irradiation and atmospheric oxygen as the terminal oxidant. A broad range of boronic acids were successfully converted to the corresponding hydroxy derivatives, showcasing the excellent functional-group tolerance and synthetic utility of the developed photochemical protocol. Preliminary mechanistic investigations were undertaken to elucidate the reaction pathway and to identify the reactive oxygen species involved.

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### Introduction

Boronic acids and esters are versatile intermediates in organic synthesis, capable of undergoing a wide range of functional group transformations. Among these, their conversion into phenols or alcohols is particularly valuable, providing a platform for further functionalization in pharmaceuticals, agrochemicals and materials science.<sup>1,2</sup> In recent decades, sustainable and environmentally friendly methodologies have attracted significant attention.<sup>3</sup> Visible light photochemistry using low-energy irradiation sources, such as LED lamps or sunlight, offers a green and mild alternative for promoting chemical reactions.<sup>3</sup> This approach, recognized for more than a century,<sup>4</sup> has gained renewed interest over the last two decades,<sup>5</sup> due to its ability to perform transformations under milder and safer conditions, a feature highly appreciated in the pharmaceutical industry.

The direct conversion of boronic acids or esters into hydroxy compounds is well-studied.<sup>6</sup> Several photochemical protocols for the aerobic oxidation of boronic acids have been reported, using various photocatalysts. Jorgensen and co-workers employed a ruthenium-based catalyst in DMF (Scheme 1A).<sup>7</sup> From a pharmaceutical industry perspective, the

use of DMF and prolonged reaction time limits the practicality of this approach and raises environmental concerns, due to DMF's toxicity. In 2013, Scaiano and co-workers investigated methylene blue as an organic photocatalyst for the same transformation (Scheme 1B).<sup>8</sup> Compared to the ruthenium complex [Ru(bpy)<sub>3</sub>Cl<sub>2</sub>]<sub>6</sub>H<sub>2</sub>O, methylene blue offered superior mechanistic performance, although the substrate scope remained limited. Similarly, Zhang and co-workers utilized Rose Bengal as the photocatalyst (Scheme 1C),<sup>9</sup> but this protocol required high catalyst loading (5 mol%). Our group has focused on developing eco-friendly, aerobic photochemical protocols.<sup>10</sup> Using CFL lamps as the irradiation source, we identified 2,2-dimethoxyphenylacetophenone as a photoinitiator with *N,N'*-diisopropylethylamine (DIPEA) (7.5 equiv.) as sacrificial donor (Scheme 1D).<sup>11</sup> Replacing 2,2-dimethoxyphenylacetophenone with anthraquinone allowed a 10-fold reduction in DIPEA loading (Scheme 1D),<sup>12</sup> and further studies led to a catalyst-free UV-light protocol (370 nm) combined with a low amount of DIPEA under mild reaction conditions, further expanding the substrate scope.<sup>13</sup>

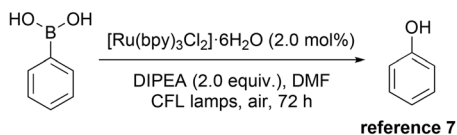
Despite these advances, milder reaction conditions using lower energy irradiation sources, such as green LEDs remain largely unexplored.<sup>14</sup> A literature example of green LED-mediated oxidation of boronic acids employed a Zr-based MOF as the photocatalyst. Building on our prior studies of quinonimines as photocatalysts in aerobic oxidation,<sup>15</sup> we investigated their application to the aerobic oxidation of boronic acids to the corresponding hydroxy derivatives. Quinonimines exhibit multiple biological and antioxidant activities,<sup>16</sup> and molecules

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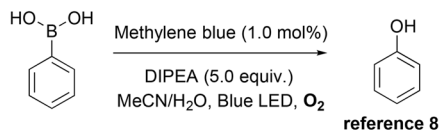
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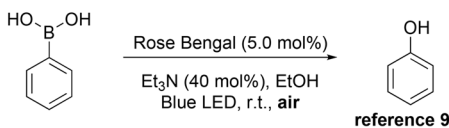
## A. Ruthenium-mediated photochemical aerobic oxidation



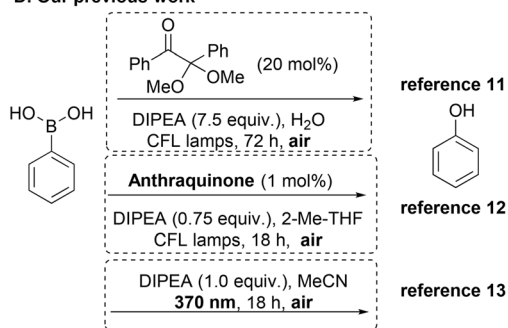
## B. Photochemical aerobic oxidation utilising methylene blue



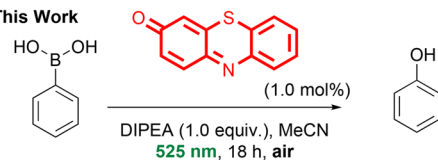
## C. Photochemical aerobic oxidation utilising rose bengal



## D. Our previous work



## E. This Work



**Scheme 1** Previous photochemical aerobic oxidation protocols for boronic acids and this work.

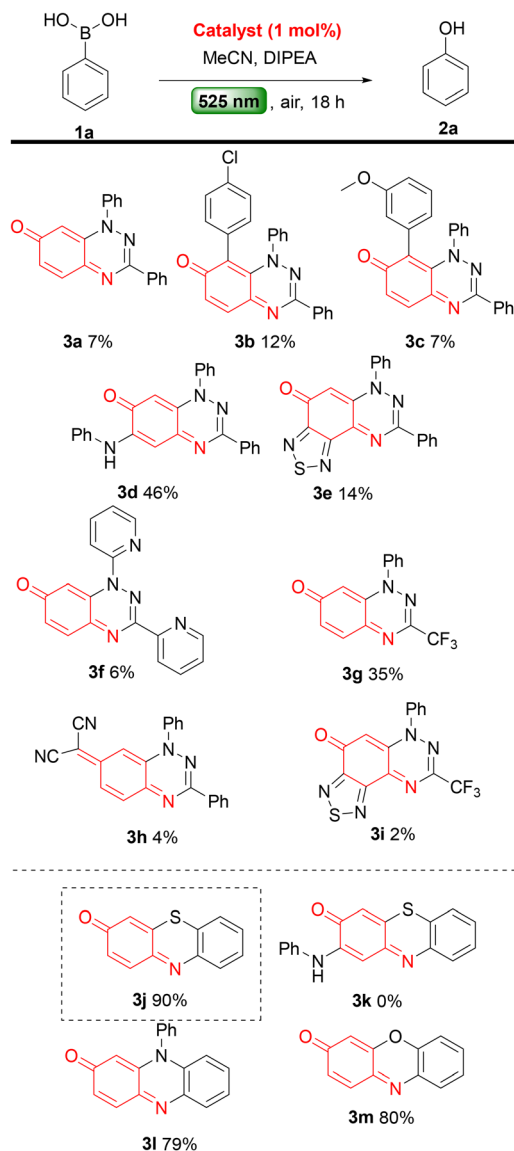
such as 3*H*-phenothiazin-3-one have demonstrated diverse biological effects, including antiparasitic activity,<sup>17</sup> enzyme inhibition (*e.g.*, succinoxidase, cytochrome oxidase, and cytochromes P450), and psychopharmacological effects.<sup>17–20</sup> While phenothiazine derivatives, such as methylene blue<sup>21</sup> or methylene violet,<sup>22</sup> have been widely employed as photocatalysts, the structurally simpler 3*H*-phenothiazin-3-one remains underexplored in photochemical applications. Previous studies from the Koutentis group, described the synthesis of the quinonimines investigated in this work.<sup>16a,23,24</sup> Building on these studies and our earlier investigations of the effect of irradiation wavelength on the aerobic oxidation process of boronic acids,<sup>13</sup> we developed a mild, green and sustainable aerobic photooxidation protocol that converts boronic acids to hydroxy derivatives using 3*H*-phenothiazin-3-one as the photocatalyst under green light irradiation (Scheme 1E).

## Results and discussion

Previously, we had examined the effect of irradiation wavelength on the oxidation of boronic acid derivatives.<sup>13</sup> Although that study provided a photocatalyst-free protocol, it required UV light (370 nm), which limits functional-group tolerance and promotes side reactions. To address these limitations, we sought to develop a milder photochemical method using green LED irradiation at a wavelength not absorbed by the substrate. As the transformation does not proceed in the absence of a photocatalyst,<sup>13</sup> our effort focused on identifying a catalyst capable of promoting the reaction efficiently under these conditions. Optimisation commenced with phenylboronic acid (**1a**) as the model substrate and a small library of in-house quinonimines (**3**, 1 mol%), including benzotriazinones, phenazinones, phenoxazinones and phenothiazinones to identify the most active photocatalyst (Scheme 2) (see SI, Sect. S2). Reactions were carried out in acetonitrile with DIPEA as the reductant under 525 nm irradiation for 18 h. Quinonimine **3a** delivered phenol **2a** in low yield (7%), and modifications to the aromatic core (**3b** and **3c**) offered no improvement. Notably, introduction of an anilino substituent at the  $\alpha$ -position of the quinonimine moiety (**3d**) significantly enhanced the reaction efficiency, affording phenol **2a** in 46% yield. Decorating the benzotriazinone scaffold with heteroaryl or trifluoromethyl groups led to poor yields (**3e–3i**), while derivative **3h**, afforded **2a** in only 4%. These unsatisfactory results prompted exploration of phenazine, phenothiazine, and phenoxazine derivatives. Phenothiazine **3j**, *N*-phenylphenazinone **3l** and phenoxazinone **3m** proved highly effective affording phenol **2a** in 90%, 79%, and 80% yields, respectively (Scheme 2). Considering the beneficial effect of an anilino substituent on the benzotriazinone scaffold (**3a** vs. **3d**, 7%  $\rightarrow$  46%), we next evaluated 2-phenylaminophenothiazinone **3k**. In this case, the anilino substituent had a detrimental effect, and no product was observed, likely due to poor solubility in the reaction mixture.

Upon identifying phenothiazinone **3j** as the most efficient photocatalyst, we investigated solvent effects (Table 1) (see SI, Sect. S2). Polar aprotic solvents, such as dichloromethane, chloroform or ethyl acetate, afforded moderate yields (entries 2–4). Polar protic solvents (EtOH, *i*-PrOH) gave variable results, with *i*-PrOH completely suppressing reactivity (entries 5 and 6), while ethereal solvents (dioxane, THF) afforded moderate yields (entries 7 and 8). Reactions in non-polar solvents were sluggish (petroleum ether, cyclohexane; entries 9 and 10), likely due to poor solubility of the polar substrate and catalyst. Aromatic solvents, particularly toluene and xylene, performed best among the alternatives (entries 11–13), whereas benzene gave only moderate yield. The highly polar aprotic solvent dimethylsulfoxide (DMSO) completely suppressed reactivity (entry 14), likely due to quenching of the photocatalyst excited state or interference with electron-transfer processes. Although DMF and DMAc showed excellent performance by <sup>1</sup>H NMR analysis, they were not pursued, due to their poor green credentials (entries 15 and 16). Water





**Scheme 2** Quinonimine-based photocatalysts for the aerobic oxidation of phenylboronic acid (**1a**) under green LED irradiation (525 nm, 40 W).

was also evaluated as a greener alternative, but low solubility of the organic substrate and catalyst resulted in only trace product (entry 17).

We next investigated the effect of the additive on the reaction outcome (Table 2). Having established DIPEA as the base, we first examined the effect of lowering its equivalents (see SI, Sect. S2). Decreasing the amount of DIPEA led to a gradual decline in yield, with 0.75 and 0.5 equivalents affording 63% and 50% of phenol **2a**, respectively (entries 2 and 3). Use of primary amines, such as propylamine, were ineffective (5% yield, entry 4), while secondary amines, including morpholine or *N*-methylmorpholine, afforded poor yields (18% and 27%, entries 6 and 7). Tertiary amines, such as triethylamine, provided a moderate yield (76%, entry 5). The biomimetic reducing agent Hantzsch ester was also tested as a hydrogen

**Table 1** Solvent screening for the photochemical aerobic oxidation of **1a**

Entry	Solvent	<b>2a</b> <sup>a</sup> (%)
1	MeCN	100 (90)
2	CH <sub>2</sub> Cl <sub>2</sub>	57
3	CHCl <sub>3</sub>	29
4	EtOAc	60
5	EtOH	65
6	<i>i</i> -PrOH	0
7	Dioxane	55
8	THF	32
9	Pet. Ether (40–60 °C)	7
10	Cyclohexane	14
11	Benzene	65
12	Toluene	87 (58)
13	Xylene	84 (79)
14	DMSO	0
15	DMF	90 (61)
16	DMAc	82
17	H <sub>2</sub> O	2
18	H <sub>2</sub> O–MeCN (1 : 1)	85 (80)

<sup>a</sup> The reaction was performed with phenylboronic acid (**1a**) (24 mg, 0.20 mmol), DIPEA (26 mg, 0.20 mmol) and catalyst **3j** (1.0 mol%) in solvent (1 mL), under green LED (Kessil PR160L, 525 nm, 40 W) irradiation for 18 h. Conversion was determined by <sup>1</sup>H NMR. Yield of isolated product after column chromatography in parenthesis.

donor, affording phenol **2a** in 46% yield (entry 8). Finally, inorganic base K<sub>2</sub>CO<sub>3</sub> proved completely ineffective, with no product observed (entry 9).

**Table 2** Additive screening for the photochemical aerobic oxidation of **1a**

Entry	Additive (equiv.)	<b>2a</b> <sup>a</sup> (%)
1 <sup>b</sup>	DIPEA (1)	90
2	DIPEA (0.75)	63
3	DIPEA (0.5)	50
4	Propylamine (1)	5
5	Et <sub>3</sub> N (1)	76
6	Morpholine (1)	18
7	<i>N</i> -Methylmorpholine (1)	27
8	Hantzsch ester (1)	46
9	K <sub>2</sub> CO <sub>3</sub> (1)	0

<sup>a</sup> The reaction was performed with phenylboronic acid (**1a**) (24 mg, 0.20 mmol) and catalyst **3j** (1 mol%) in MeCN (1 mL), under green LED (Kessil PR160L, 525 nm, 40 W) irradiation for 18 h. <sup>b</sup> Yield of isolated product after column chromatography.

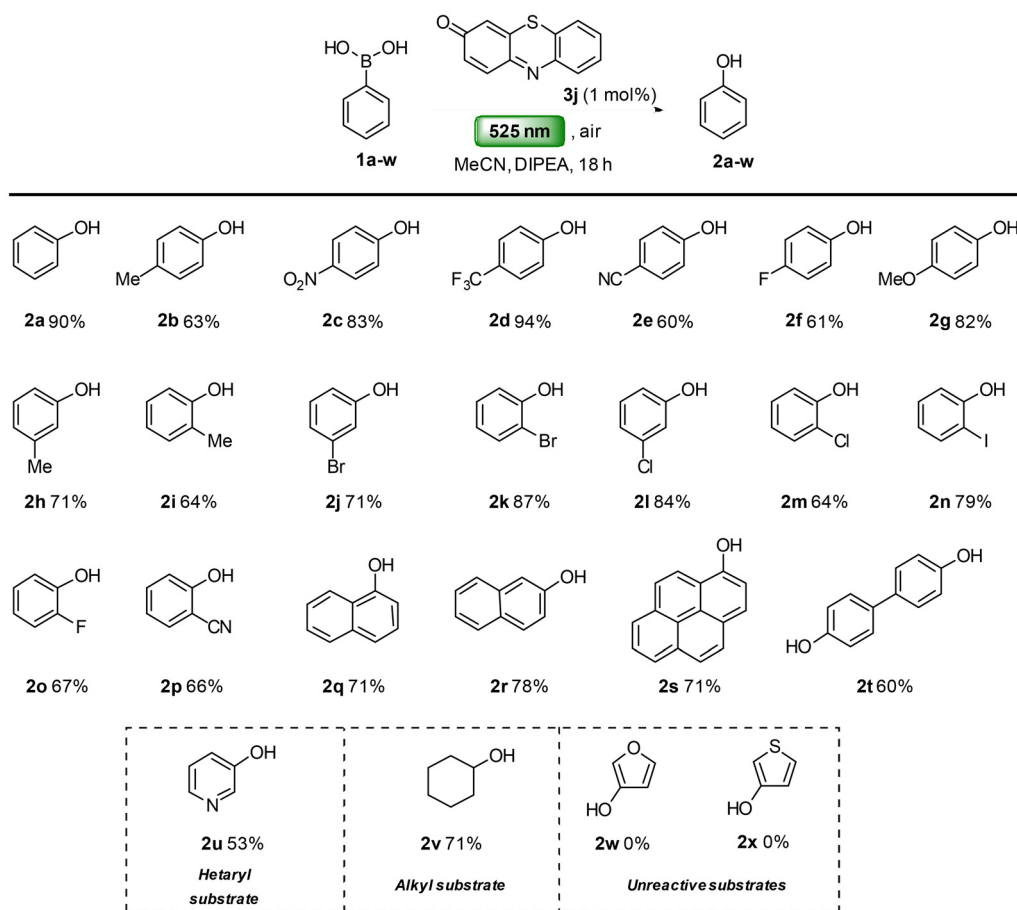


Having established the optimal reaction conditions, we next turned our attention to the substrate scope and functional group tolerance of the photooxidative protocol. A variety of boronic acids were subjected to the optimised conditions (Scheme 3). The reaction tolerates both electron-withdrawing and electron-releasing substituents at *ortho*-, *meta*- and *para*-positions of the phenyl boronic acid, affording the corresponding phenols in good to excellent yields (**2a–p**), indicating only a subtle electronic influence on the reaction efficiency. Minor variations in yields may be attributed to the possible sublimation during their isolation. The position of the substituent on the aryl ring did not impede the reaction, although slight differences in yield were observed (**2h–r**). Notably, in all cases, functional groups including methyl (**2b**, **2h**, **2i**), nitro (**2c**), trifluoromethyl (**2d**), cyano (**2e**, **2p**), halogens (**2f**, **2j–o**), and methoxy (**2g**) remained intact under the reaction conditions. Fused arylboronic acids were also well tolerated, giving the corresponding phenols in good to excellent yields (**2q–s**). More challenging substrates were then examined. 1,1'-Diphenyl-4,4'-bisboronic acid (**1t**), was converted to the corresponding 1,1'-diphenol (**2t**) in 60% yield. Heteroarylboronic acids (**2w–x**) were also tested: pyridin-3-ylboronic acid (**1u**) gave pyridin-3-ol (**2u**) in 53% yield without any N-oxidised side product. In contrast, fur-3-ylboronic acid (**1w**) and thien-3-ylboro-

nic acid (**1x**) produced complex mixtures that were not further investigated. Finally, an alkylboronic acid, cyclohexylboronic acid (**1v**) gave cyclohexanol (**2v**) in 71% yield, with no overoxidized products, such as cyclohexanone, observed.

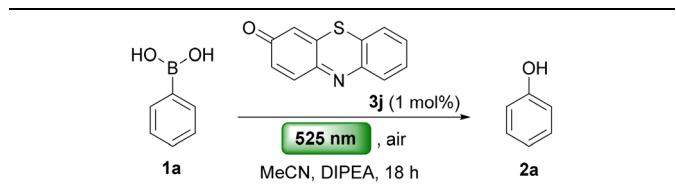
With phenothiazinone **3j** established as an efficient photocatalyst for the aerobic photochemical oxidation of boronic acids, we next investigated the reaction mechanism. The involvement of radical intermediates was probed by conducting the reaction in the presence of TEMPO or BHT as radical scavengers (see SI, Table S4). In both cases, formation of the desired product was significantly suppressed, consistent with a radical pathway. Additional control experiments further demonstrated the necessity of all reaction components (Table 3).

UV-Vis studies were conducted to probe the possible formation of an electron donor-acceptor (EDA) complex (see SI, Sect. S5), as the pivotal role of EDA complexes in photochemical reactions is well established.<sup>25</sup> Mixing phenylboronic acid (**1a**) with DIPEA did not produce a bathochromic shift in the green-light region, and the addition of phenothiazinone **3j** did not induce further spectral changes (SI, Fig. S6). However, upon irradiation of phenothiazinone **3j** at 525 nm in the presence of DIPEA, a significant change in the UV-Vis spectrum was observed, suggesting that the excited catalyst interacts with



**Scheme 3** Substrate scope for the photochemical aerobic oxidation of boronic acids.



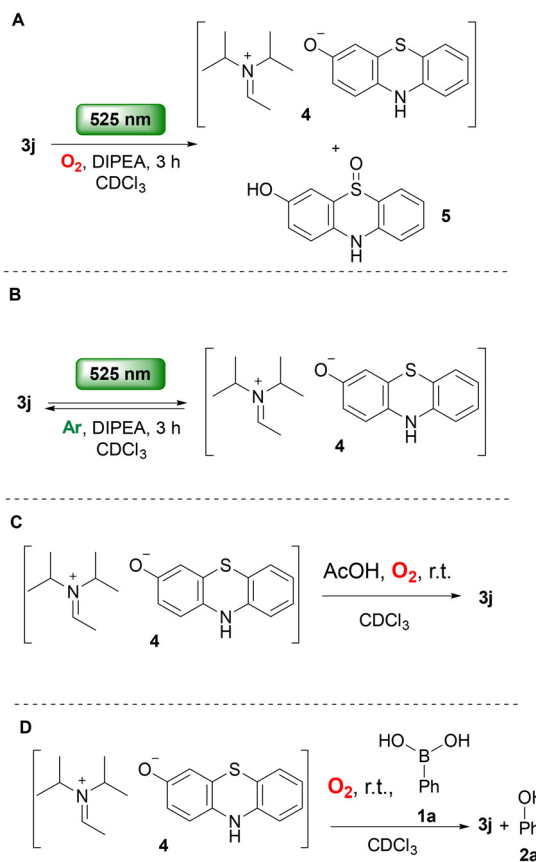
**Table 3** Control experiments for the aerobic photochemical aerobic oxidation of **1a**

Entry	3j	DIPEA	525 nm	O <sub>2</sub>	Yield <sup>a</sup>
1	–	+	+	+	0
2	+	–	+	+	0
3	+	+	–	+	0
4	+	+	+	–	0

<sup>a</sup>The reaction was performed with phenylboronic acid (**1a**) (24 mg, 0.20 mmol) and catalyst **3j** (1.0 mol%) in MeCN (1 mL), under green LED (Kessil PR160L, 525 nm, 40 W) irradiation for 18 h.

DIPEA (SI, Fig. S5). Prior to irradiation, the mixture of catalyst **3j** and DIPEA showed absorption bands at 367 and 500 nm, corresponding to the catalyst. After irradiation at 525 nm for 120 s, the original bands disappeared, and a single band at 323 nm appeared, characteristic of phenothiazines (SI, Fig. S5).<sup>26</sup>

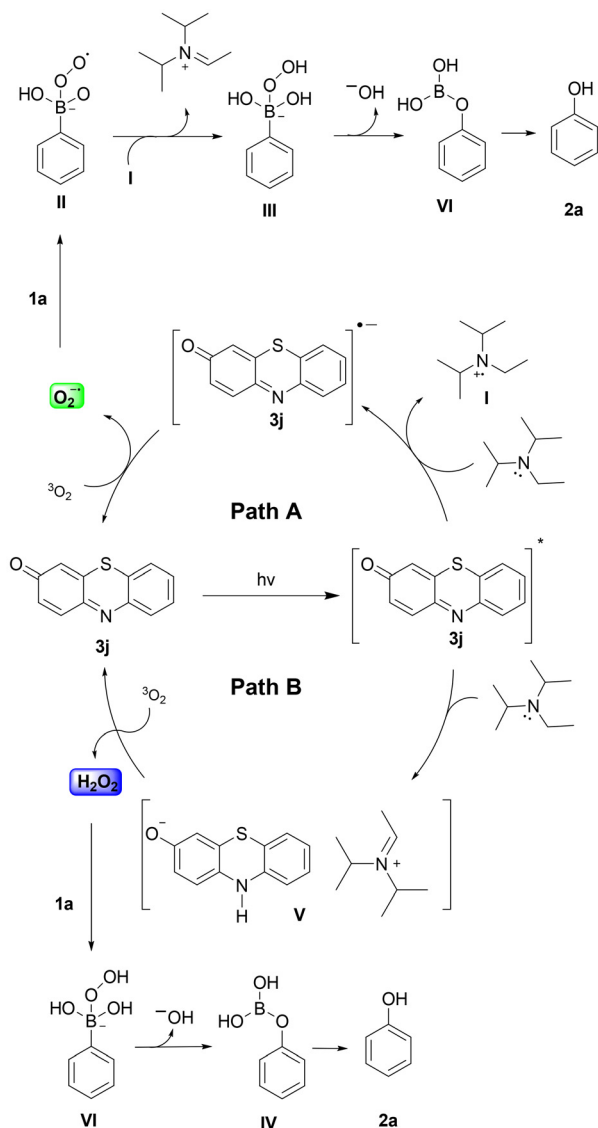
To further probe the role of DIPEA and its interaction with the excited phenothiazinone catalyst **3j**, a series of NMR experiments were conducted (see SI, Sect. S6). Irradiation of catalyst **3j** or DIPEA individually produced no observable changes. However, upon irradiation of a mixture of catalyst **3j** with DIPEA at 525 nm in CDCl<sub>3</sub> for 3 h, consumption of catalyst **3j** was observed, accompanied by the formation of 3-hydroxyphenothiazine salt **4** and 3-hydroxy-phenothiazine sulfoxide **5** (Scheme 4A). The formation of sulfoxide **5** can be rationalized by a photochemical aerobic sulfide oxidation. The aerobic oxidation of salt **4** to the correspondingsulfoxide **5** is well-precedented. Such oxidations of diaryl sulfides typically proceed *via* SET between the sulfide and molecular oxygen, generating superoxide radical anion.<sup>10c,27,28</sup> Therefore, the formation of sulfoxide **5** provides evidence that superoxide anion is generated under our photochemical conditions.<sup>20</sup> When the same irradiation experiment was carried out in the presence of phenylboronic acid (**1a**), no formation of by-product **5** was detected. Further evidence for the generation of superoxide anion came from the H<sub>2</sub>O<sub>2</sub> test (see SI, Sect. S8). H<sub>2</sub>O<sub>2</sub> was detected upon irradiation of phenothiazinone **3j** with DIPEA at 525 nm, whereas irradiation of **3j** in the absence of DIPEA did not produce H<sub>2</sub>O<sub>2</sub>. Additional experiments were conducted to gain insight into the fate of the 3-hydroxyphenothiazine salt **4**. Irradiation of a mixture of phenothiazinone **3j** with DIPEA at 525 nm under an oxygen-free atmosphere (argon) resulted exclusively in the formation of salt **4** (Scheme 4B). When the reaction mixture was subsequently stirred under air and treated with 1.0 equivalent of acetic acid to quench the salt, regeneration of catalyst **3j** was observed (Scheme 4C). Furthermore, when phenylboronic acid (**1a**) was added instead of acetic acid, phenol (**2a**) was formed (Scheme 4D) (see SI, Sect. S6).

**Scheme 4** NMR experiments to identify catalyst's **3j** active forms. (A) irradiation of **3j** and DIPEA under oxygen. (B) irradiation of **3j** and DIPEA under argon. (C) **4** under air and AcOH. (D) **4** under air and **1a**.

Cyclic voltammetry (CV) measurements of phenothiazinone **3j** showed a reduction potential of  $E_{\text{red}} = -0.66$  V *vs.* SCE (see SI, Sect. S9). The excited-state reduction potential of catalyst **3j** was estimated at  $E_{\text{red}}^* = 0.80$  V *vs.* SCE, indicating that the excited state is a strong oxidant capable of accepting an electron from DIPEA ( $E_{\text{red}} = 0.64$  V *vs.* SCE).<sup>15</sup> To evaluate possible side pathways, we assessed whether DIPEA could be oxidized by <sup>1</sup>O<sub>2</sub> to generate superoxide. This process is thermodynamically disfavoured: the reduction potential of <sup>1</sup>O<sub>2</sub> ( $E_{\text{red}} = -0.87$  V *vs.* SCE)<sup>27</sup> is more negative than the oxidation potential of DIPEA ( $E_{\text{ox}} = 0.64$  V *vs.* SCE). Consequently, oxidation of DIPEA by <sup>1</sup>O<sub>2</sub>, to form superoxide is not supported.<sup>8</sup> Additionally, the quantum yield ( $\Phi$ ) of the photochemical reaction was determined following the procedure by Yoon and co-workers.<sup>29</sup> The value of  $\Phi = 0.28$  is consistent with a closed catalytic cycle rather than a radical chain mechanism.

Summarizing our findings, a plausible mechanistic pathway is depicted in Scheme 5. Upon irradiation at 525 nm, phenothiazinone **3j** is excited to its singlet state and *via* intersystem crossing is led to its triplet state **3j\***. The excited photocatalyst then interacts with DIPEA *via* a SET event to produce the radical anion of photocatalyst **3j**, which proceeds to be quenched by molecular oxygen and provides superoxide





**Scheme 5** Proposed mechanistic pathway for the photochemical aerobic oxidation of phenylboronic acid (**1a**).

radical anion that oxidizes boronic acid **1a**, leading to intermediate **II** (Scheme 5, Path A top). A HAT event leads to **III**, which upon rearrangement and hydrolysis leads to the desired product. Our mechanistic (NMR and UV-Vis) studies further support an alternative possible interaction between the excited state catalyst **3j\*** and DIPEA, leading to species **V**. Under aerobic conditions, **V** is unstable and undergoes oxidation, generating hydrogen peroxide, which can contribute to the oxidation of boronic acids (Scheme 5, Path B).

## Conclusions

The advent of photochemistry has opened new avenues for accessing unique reactivities. In particular, oxygen-mediated photochemical oxidation of boronic acids has attracted con-

siderable attention. Herein, we report a mild and efficient photochemical protocol for the aerobic oxidation of boronic acids to their hydroxy derivatives, employing 3*H*-phenothiazin-3-one (**3j**) as the photocatalyst. This catalyst proved highly effective under green LED irradiation (525 nm), exhibiting excellent functional group tolerance across a broad range of substituted boronic acids.

## Author contributions

C. G. K. conceived the study, P. L. G., C. T. C., S. K. S. and C. G. K. designed the experiments and analyzed the data. D. F. F. and G. A. Z. performed the photocatalyst's synthesis. C. T. C., S. K. S. and P. L. G. performed the experiments. C. T. C. and P. L. G. prepared the draft of the manuscript. P. A. K. and C. G. K. performed the final editing. The manuscript was written through contributions of all authors.

## Conflicts of interest

There are no conflicts to declare.

## Data availability

The data that support the findings of this study are available in the supplementary information (SI). Supplementary information: experimental data,  $^1\text{H}$  and  $^{13}\text{C}$  NMR data, UV-Vis, optimisation and mechanistic studies. See DOI: <https://doi.org/10.1039/d6ob00133e>.

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