Review of laser flash photolysis of organic molecules (2015–2018)

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DOI: 10.1039/9781788016520-00070

This chapter serves as a review of the literature concerning the use of laser flash photolysis (LFP) in research reported between the years 2015 and 2018. The first half of the chapter primarily focuses on fundamental studies to characterize reactive intermediates and elucidate their reaction mechanisms, with special attention to a new LFP technique that allows transient absorption to be obtained in crystals. The detection and identification of excited states and their reactivity is also addressed. The second half of the chapter focuses on the use of LFP to understand and influence the development of photochemistry applications such as synthesis, photocages, polymerization, and photocatalysis. Finally, we briefly mention research that uses LFP but is outside the main focus of this chapter, such as the analysis of biological systems and environmental studies.

1 Introduction

In 1967, the pioneering development of flash photolysis by Norrish and Porter in the 1950s and 1960s was awarded a Nobel Prize.[1] Since this time, the study of the transient absorption produced by reactions with light has captivated spectroscopists. While the original flash photolysis systems allowed for the detection of intermediates on the microsecond and millisecond timescales using UV–Vis spectroscopy techniques, a need for progressively shorter timescales was immediately realized. The major hurdle in detection on shorter timescales was not detector technology, but rather a lack of short-duration light pulses. Thankfully, in the year proceeding Norrish and Porter's Nobel Prize, Lindqvist combined the use of the recently invented pulsed N_2 laser with traditional flash photolysis detection during his study of the triplet state of acridine.[2] The introduction of the short-duration light pulses produced by laser emission opened the door for investigations on nanosecond—and shorter—timescales, and thus the laser flash photolysis (LFP) technique was born. A general instrumental schematic can be seen in Fig. 1.

Further groundbreaking developments in femtochemistry made ultrafast spectroscopy a reality, resulting in the 1999 Nobel Prize in Chemistry being awarded to Zewail for his pioneering work in the field. Thus, LFP can be used to investigate physical and chemical processes over a wide range of timescales, from milliseconds to femtoseconds. For more technical reviews of LFP systems, the reader is directed to excellent book chapters by Scaiano, Gauduel, and Schepp and Cozens.[1,3,4]

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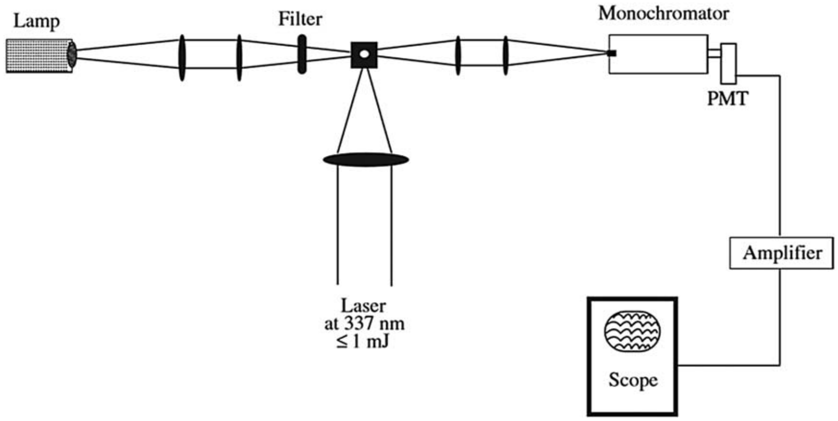


Fig. 1 Schematic representation of a basic LFP apparatus. Adapted from ref. 1 with permission from John Wiley and Sons, Copyright © 2004 John Wiley & Sons, Inc.

Herein, the current state of LFP in the years 2015–2018 is described. Many of the topics studied by the pioneers of flash photolysis, such as the singlet and triplet excited states of organic compounds and reactive intermediates, are still heavily investigated today. This chapter is primarily focused on the photochemical reactions of organic compounds as they relate to the detection and characterization of excited states and reactive intermediates, such as carbenes, nitrenes and nitrenium ions, radicals and diradicals, and cations. LFP studies that have contributed to the development of photochemical applications, such as photocages and polymerization, are also addressed. Additionally, special attention is given to the developing field of monitoring reactions in crystals using nanocrystalline suspensions, a technique analogous to classic organic solution spectroscopy. Finally, some consideration is given to the reactions and photophysics of inorganic compounds, especially to topics relating to the emerging field of photocatalysis. It should be highlighted that advances in theoretical calculations have made the identification of excited states and transient intermediates easier by allowing for comparison between experimental and calculated UV–Vis spectra. Finally, it should be empathized that the commercial availability of turnkey nanosecond LFP apparatus has made LFP more accessible to a broader spectrum of scientists.

2 Fundamental research

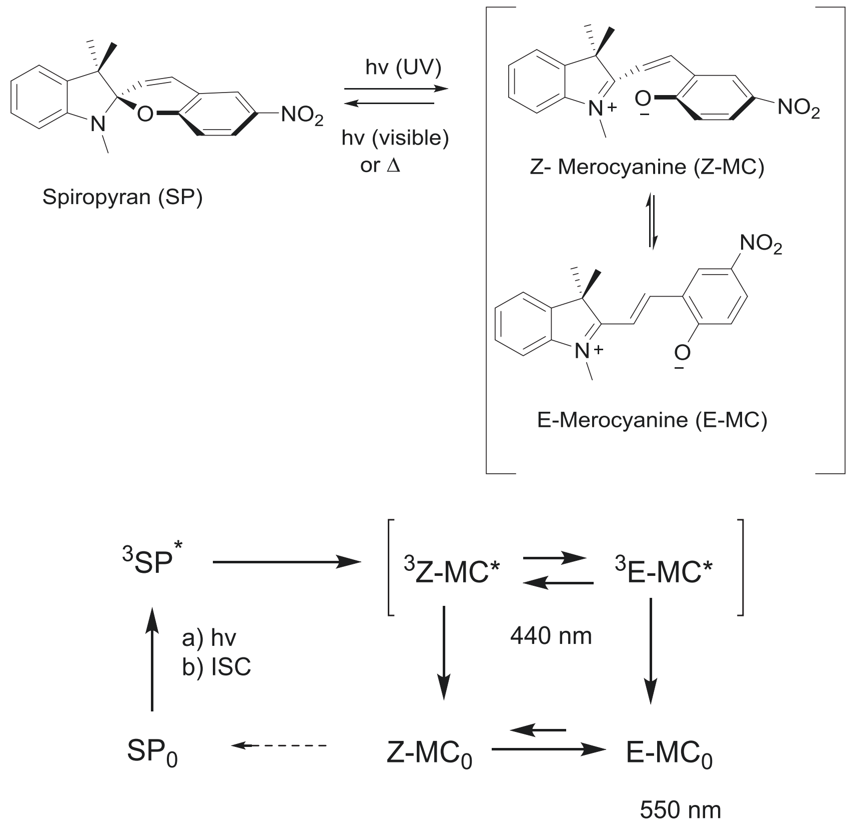
Like Lindqvist, who studied the triplet states of acridine during his first LFP study, many researchers are still interested in the fundamental reactivity of many compounds upon photoexcitation. In this pursuit, LFP is an excellent tool for probing how the molecular environment, solvation, the excitation wavelength, and the presence of other molecules can affect reactivity. The following sections focus on this fundamental research as it pertains to reactive intermediate and excited state reactivity. Special focus has been given to research using nanocrystalline suspensions, which has allowed researchers to gain unprecedented insight into the reactivity within crystals.

2.1 Nanocrystalline suspensions

The photochemistry of crystalline materials has long been an attractive tool for synthetic chemists, largely due to the enhanced selectivity caused by restriction in the crystal lattice.[5–7] Additionally, the reduced solvent use in these solid-state reactions is beneficial for green chemistry principles to combat the problem of excess solvent waste.[8,9] However, for years, the advancement of photochemistry in solids has been hindered by the lack of techniques for mechanistic analysis. As many bulk solids exhibit reflectance issues in LFP, the understanding of solid-state reactions has typically relied on structure–reactivity correlations, such as those obtained by Schmidt in the 1960s for *trans*-cinnamic acids.[10] Fortunately, in recent years, work by Garcia-Garibay and his collaborators has offered a clever solution by adapting a reprecipitation method first described by Kasai *et al.*[11] to suspend nanocrystals in a solvent. This approach allows for spectroscopic analysis, as long as the average size of the crystals is smaller than the irradiation wavelength.[12–14] This section will describe progress made using this nanocrystalline suspension technique in conjunction with LFP.

Using this nanocrystalline suspension technique, Garcia-Garibay and co-workers investigated the solid-state and solution mechanism of 6-nitroindolinospiropyran, as shown in Scheme 1.[15] First, the solution mechanism was determined to clarify inconsistencies in previous reports. Next, investigation of the nanocrystalline mechanism revealed a broad, featureless spectrum that did not resolve on the nanosecond timescale, which was different from the behavior in solution and previously reported spectra of bulk microcrystalline powders. The quantum-theory-supported rationale used to explain this phenomenon indicated that the nanocrystals sufficiently slowed the rotation necessary for transformation between the *Z*- and *E*-merocyanine intermediates on their excited states. It was postulated that this slowing of the rotation allowed for the generation of several high-energy intermediates along the rotation pathway that in turn result in the observation of a broad spectrum. In particular, this paper highlighted how the use of nanocrystalline suspensions allows for more in-depth solid-state studies than previous bulk crystal systems.

The Gudmundsdottir group also investigated how crystal lattices can control the photoreactvity to yield different products in the solid state than in solution, with LFP used to elucidate the solid-state reaction mechanisms.[16,17] When α-azido-*p*-methoxy-acetophenone was studied in solution using LFP, the obtained transient was assigned mainly to a long-lived triplet alkylnitrene. However, in nanocrystalline suspensions, the observed transient was assigned to α-cleavage to form a benzoyl radical. Thus, the crystal lattice stabilized the configuration of the triplet ketone, which undergoes α-cleavage, over the triplet configuration of the ketone, which yields a denitrogenation reaction in solution. Another paper from



Scheme 1

the same group reported that crystal packing of a sterically hindered isobutyrophenone azide in a high-energy conformation altered the reactivity from β-cleavage and the release of hydrazoic acid in solution to γ-cleavage in the solid state.[17] The crystal packing of the molecule was shown to involve a higher energy conformer than the lowest energy conformer identified by calculations. Thus, this paper highlighted how crystals can provide access to higher energy pathways that are inaccessible from solution-based reactions.

On the theme of comparing solution and solid-state reactions using nanocrystalline suspensions, Garcia-Garibay and co-workers published a few papers showing that the rate-limiting step of reactions could change depending on whether the reaction was performed in solution or in the solid state. For example, as shown in Fig. 2, tetraarylacetones underwent decarbonylation in crystals whereas α-cleavage was observed in solution. In nanocrystalline suspensions, a biexponential decay was observed, with the longest lifetime assigned to the rate-limiting step of the decarbonylation reaction, namely intersystem crossing to form the final product.[18] Similarly, LFP investigations of the reaction of Δ2-1,2,3-triazolines to form aziridines revealed the generation of a triplet nitrene by the release of nitrogen from the triazoline in both solution and nanocrystals. However, the latter nitrene had a lifetime that was more than

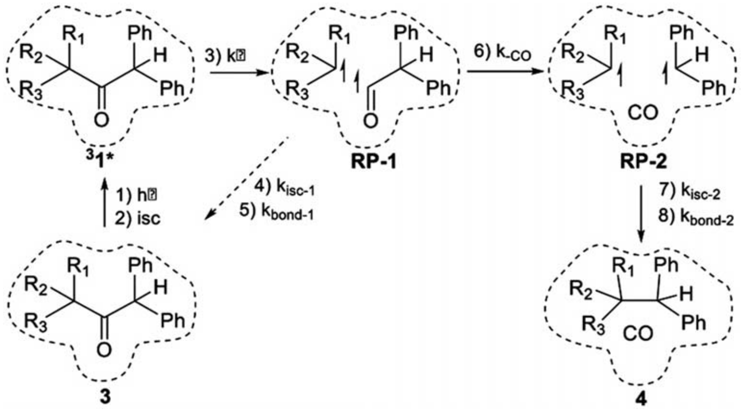


Fig. 2 CO release during solid-state photolysis of tetraarylacetones. Reproduced from ref. 18 with permission from American Chemical Society, Copyright 2017.

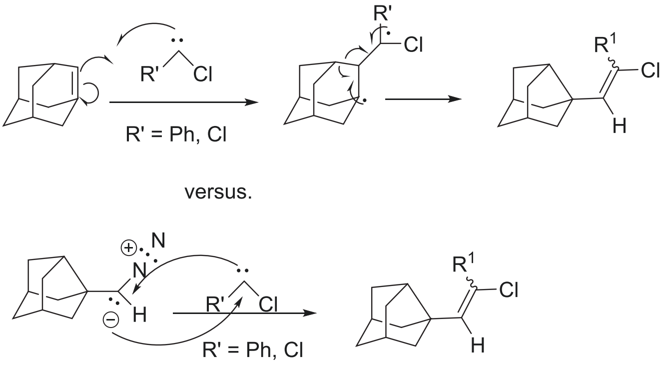
three times longer than that in solution.[19] An analogous finding was obtained for a set of α-phenyl-substituted carbonyls that release carbon monoxide.[20] Again, the rate-limiting step in solids was observed to be the intersystem crossing of the triplet biradical pair to form the product. The Garcia-Garibay research group also probed the electronic effects of tolyl-acetophenones in solution and solids.[21] Based on LFP studies, they determined that hydrogen transfer in these compounds took up to six times longer in crystals than in solution. Lastly, a study on nitrogen gas release from a series of biphenyl azides in solution and nanocrystals revealed that electron-withdrawing and electron-donating effects played a minor role in the kinetics of the photoreaction.[22]

2.2 Reactive intermediates

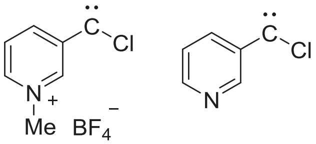
2.2.1 Carbenes, nitrenes, and nitrenium ions. Reactive intermediates such are carbenes, nitrenes, and nitrenium ions are of fundamental interest to chemists. Not only do carbenes and nitrenes have interesting electronic properties owing to their uncharged, monovalent, and radical nature, but they also have emerged as synthetically useful intermediates that allow for easy access to complex molecular architectures.

LFP was used by Merrer and co-workers to investigate carbene reactivity with anti-Bredt behavior using two different bridgehead carbenes.[23] The photolysis of noradamantyldiazirine and adamantyldiazirine resulted in the formation and subsequent trapping of adamantene and homoadamantene by chlorocarbenes. Based on LFP experiments, quantum mechanical calculations, and monitoring by NMR and UV–Vis spectroscopy, the reactions between the carbenes likely proceeded by the two pathways shown in Scheme 2.

In addition to the structurally fascinating bridgehead alkenes, carbenes with electronic properties of interest have been investigated. In two separate papers, the Moss laboratory investigated the reactivity[24] and equilibria with trimethoxybenzene[25] of *N*-methyl-3-pyridinium chlorocarbene and 2-pyridinylchlorocarbene (Scheme 3). Analysis of the Stern–Volmer



Scheme 2

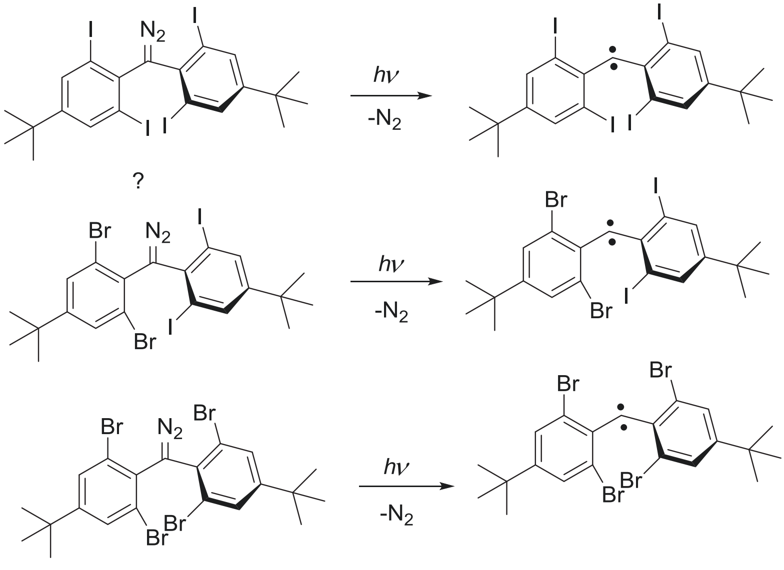


Scheme 3

plot generated by LFP of *N*-methyl-3-pyridinium chlorocarbene and its addition to various alkenes gave the most negative thermodynamic parameters and activation energy observed to date. This behavior was attributed to the highly electrophilic nature of the carbene induced by the cationic nature of the methylated pyridinium ring. The Moss laboratory also used *N*-methyl-3-pyridinium chlorocarbene to investigate the equilibrium between the carbenic form and Zwitterionic form caused by chlorine atom addition to the carbene as well as the equilibria with trimethoxybenzene. Again, the thermodynamic constants obtained from LFP studies showed that *N*-methyl-3-pyridinium chlorocarbene had a more negative enthalpy, entropy, and free energy than pyridinyl chlorocarbene and phenylchlorocarbene, with rate constants enhanced by up to 1100 times compared with those of phenylchlorocarbene. These findings again demonstrate how the strong cationic nature of the carbene influences its electrophilicity.

Harai *et al.* used the substitution and electronic properties of diphenyl carbenes to develop an ''unusually persistent'' carbene that had a half-life of up to 18 min by adding 4-iodine substituents *ortho* to the carbene.[26] In contrast, *ortho*-bromo substituents yielded a 16 s half-life, and a 1 : 1 mixture of *ortho*-bromo and *ortho*-iodo substituents resulted in a 24 s half-life (Scheme 4). The researchers attributed the long half-life associated with *ortho*-iodo substitution to the large van der Waal's shell of iodine, which prevents the triplet carbene from reacting.

Substitution was also used by the Richard research group to develop 5-halogenosalicylic acids as precursors to triplet carbenes.[27] Prior to this study, the literature on 4-halophenols had given inconclusive evidence for the formation of triplets during the dehalogenation pathway to form benzoquinones. The addition of a salicylic acid moiety as a spectroscopic

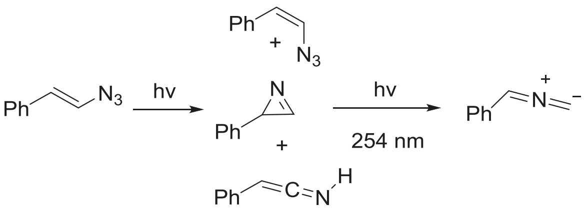


Scheme 4

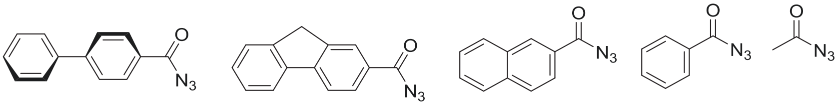
tracer that could be easily monitored by LFP was chosen because salicylic acids had been previously shown to produce long-lived triplets in organic or aqueous solution. This work provided evidence for the involvement of triplet carbenes in the dehalogenation pathway of 4-halophenols to benzoquinones.

Nitrenes have also been an active area of study in the Gudmundsdottir laboratory, which has investigated several vinylnitrene structures. The researchers reported the direct detection of triplet vinylnitrenes in solution and at cryogenic temperatures by using a ring architecture to slow the intersystem crossing from the triplet vinylnitrene to azirine.[28] Using the constraint provided by ring architecture, the researchers were also able to create and characterize a vinylnitrene that was sufficiently long-lived in solution to dimerize, forming a new dinitrogen heterocycle with synthetic appeal.[29] Adding to the wealth of vinylnitrene chemistry, the Gudmundsdottir laboratory showed that while triplet vinylnitrenes could not be observed during the photolysis of vinylbenzene azide, photolysis of the corresponding nonconjugated azirine at wavelengths above 300 nm yielded the triplet vinylnitrene through triplet sensitization of the benzene moiety.[30] However, when the azirine moiety, which absorbs below 300 nm, was selectively irradiated, the observed transient spectra were assigned to the ylide shown in Scheme 5. A similar phenomenon was observed for 3-methyl-2-phenyl-2*H*-azirine, which exhibited singlet reactivity to form an ylide when irradiated at wavelengths below 300 nm, but yielded triplet vinylnitrene when irradiated above 300 nm, as confirmed using LFP.[31]

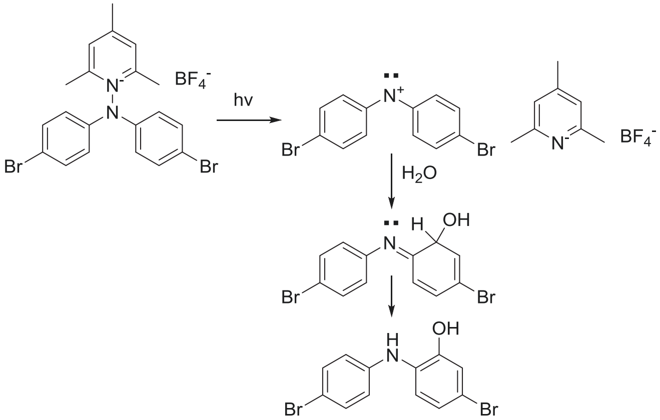
Other nitrene chemistry reported by the Poole research group was concerned with determining whether dimerizations of aryl nitrenes occurred *via* nitrene–nitrene collisions or nitrene–azide collisions.[32]



Scheme 5



Scheme 6



Scheme 7

Based on their LFP studies, the researchers believe that it is most likely that aryl azides react by nitrene–nitrene collisions.

Peng investigated why the spectroscopic lifetimes of 4-biphenyl carbonyl azides (<5 ps) are shorter than those of similar phenylcarbonyl azides, which have lifetimes of tens to hundreds of picoseconds (Scheme 6).[33] Rotation about the single bond of biphenyl was investigated using a constrained analog of 4-biphenyl carbonyl azide, but the dynamics barely changed. Instead, computational theory was used to explain the reversed solvent polarity of the π(AR)π* state of 4-biphenyl carbonyl azide, which allows for ultrafast relaxation.

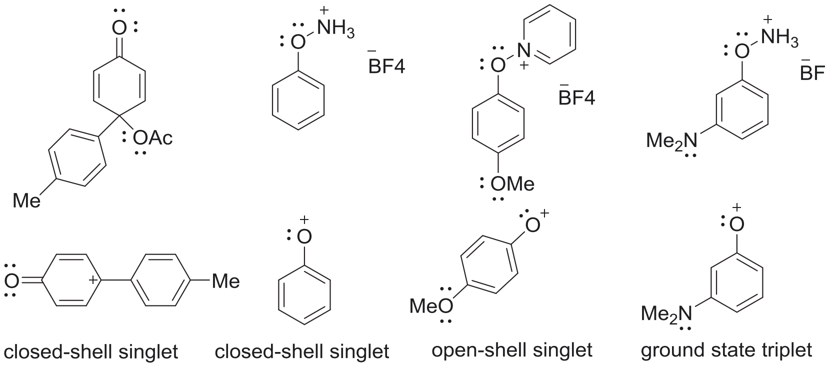
Another important class of reactive intermediates is nitrenium ions. Phillips and co-workers used nano- and femtosecond LFP along with nanosecond time-resolved Raman spectroscopy to elucidate the reaction pathway for the formation of a nitrenium ion, as displayed in Scheme 7.[34] They demonstrated that the nitrenium ion formed within 4 ps in acetonitrile. Furthermore, they performed spectroscopic investigations of the reaction of nitrenium ions with water.

2.2.2 Oxenium ions.

LFP and electron paramagnetic resonance (EPR) spectroscopy have been used successfully to detect and characterize singlet and triplet oxenium ions. One of the major challenges in studying oxenium ion chemistry is the development of suitable precursors for these intermediates (Scheme 8). Winter and his co-worker reported that protonated hydroxylamine tetrafluoroborate salts can be used as oxenium ion precursors.[35] Furthermore, a triplet oxenium ion was detected and characterized directly by Philips and collaborators for the first time using an array of techniques including transient absorption, both on nanosecond and femtosecond timescales, time-resolved resonance Raman spectroscopy, and EPR spectroscopy.[36] Investigations of 4-methoxyphenoxypyridinium tetrafluoroborate as a photoprecursor confirmed the existence of an open-shell singlet phenyloxenium ion.[37] Interestingly, this open-shell singlet oxenium ion was found to be more electrophilic than its closed-shell configuration, as reflected by their lifetimes.

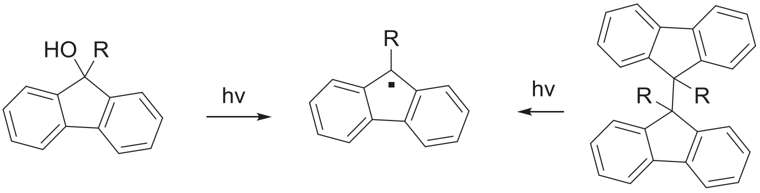
2.2.3 Radicals centered on carbon, nitrogen, oxygen, and sulfur.

Carbon-, nitrogen-, oxygen-, and sulfur-based mono- and biradicals are another group of important intermediates that can be identified directly using LFP.[38–47] Duffy *et al.* compared the photochemistry of fluorenols and bifluorenyl derivatives and found that they all yielded the same photoproducts. Thus, they proposed that these compounds cleave to form the corresponding fluorenyl radicals (Scheme 9).[38]

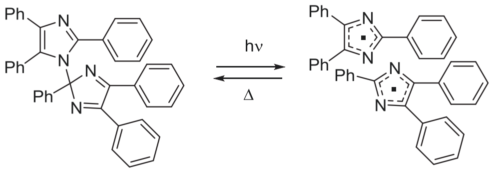


Precursors and the corresponding oxenium ions generated upon photolysis

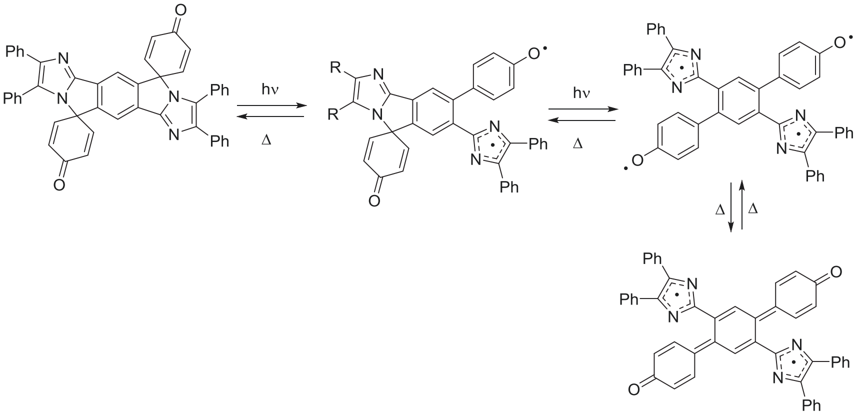
Scheme 8



Scheme 9



Scheme 10



Scheme 11

Using LFP, they verified that the fluorenol and bifluorenyl derivatives underwent C–C and C–O cleavage, respectively, to yield the corresponding fluorenyl radicals.

Abe and collaborators elegantly designed biradical complexes that are photochromic (Scheme 10).[39] In more detail, the photolysis of hexarylbiimidazaole results in the formation of imidazolyl radicals. These biradicals thermally revert to their precursors and the decay rates depend on the molecular structure. These studies were extended to study the secondary photochemistry of similar biradicals to form a quinoidal species, as shown in Scheme 11.[40] The thermodynamic parameters for the equilibrium between the biradical and quinoidal form were investigated using LFP.[41]

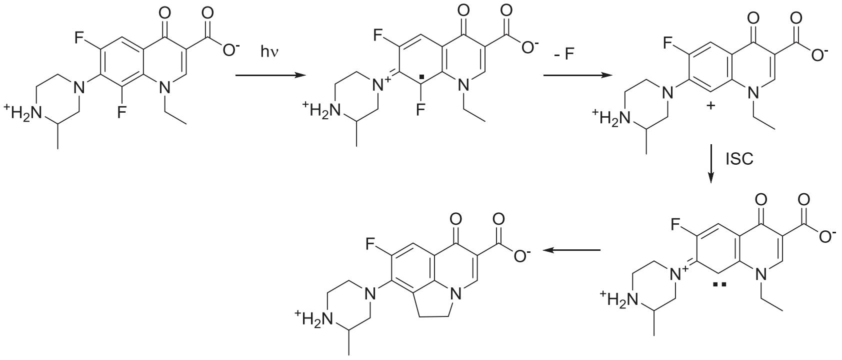
Thiyl radicals can also be formed by irradiating an appropriate precursor. For example, Chatgiliaoglu *et al.* investigated the reaction of an alkanethiyl radical with methyl linoleate.[42] With the aid of LFP studies and calculations, they concluded that thiyl radical addition to vinyl bonds is more efficient than H atom abstraction from bisallylic compounds. Furthermore, LFP studied also verified that aryl thiyl radicals ($ArS^{\bullet}$) are formed by homolytic fragmentation of diaryl sulfides.[43]

2.2.4 Cations centered on carbon, nitrogen, phosphorus, and sulfur atoms. As another group of reactive intermediates, carbocations have been investigated extensively for more than a century.[48] Because carbocations can be formed photochemically, they can also be detected directly by LFP of appropriate precursors.

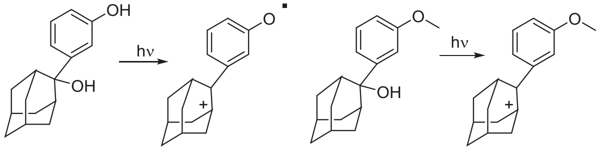
Fluoroquinolones are used as antibiotics but they can produce phototoxic side effects. Therefore, Phillips and co-workers studied the photochemistry of lomefloxacin (Scheme 12), a fluoroquinolone derivative, using LFP and transient resonance Raman spectroscopy.[49] They identified that upon irradiation, lomefloxacin forms a singlet excited state that undergoes defluorination to form a singlet cation. Intersystem crossing of the cation yields a triplet carbene that decays by cyclization.

Another example of photochemical cation formation comes from Basarić and co-workers, who synthesized benzylic alcohol derivatives and compared their thermal acid-catalyzed and photochemical solvolytic reactivities (Scheme 13).[50] The acid-catalyzed reactivity resulted in products that could be attributed to the formation of the corresponding carbocations. As an example, LFP of benzylic alcohol derivatives in polar protic solvents resulted in the formation of long-lived zwitterions that did not react with oxygen. In comparison, LFP of benzylic ether derivatives resulted in the corresponding carbocations.

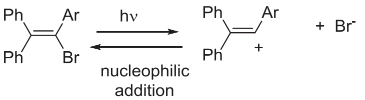
Mayr and collaborators used LFP to form and characterize vinylcations from the corresponding vinyl bromides precursors (Scheme 14).[51]



Scheme 12



Scheme 13



Scheme 14

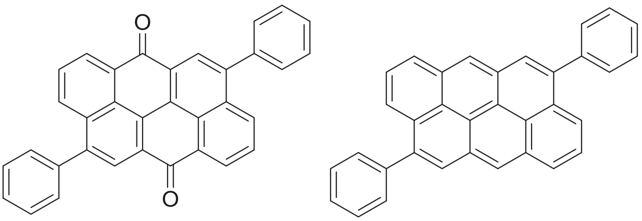
Determination of the kinetics for reaction of these vinylcations with nucleophiles showed that they react very slowly. It was theorized the intrinsic barrier for sp^2 to sp rehybridization of the vinylcations made them inefficient electrophiles.

Furthermore, electron transfer reactions can also be used to form radical cations that are centered on nitrogen, sulfur, and phosphorus atoms. These cations have been detected and characterized using LFP.[52–56] For example, Bonesi *et al.* revealed that the photochemical oxidization of tris(*p*-bromophenyl)amine results in the formation of the corresponding radical cation, which is centered on the nitrogen atom.[54] Triarylphosphine radical cations have also been detected directly using LFP, and their reactivity towards oxygen has been reported.[52] Similarly, the characterization of sulfur-centered radical cations has been achieved by LFP of the appropriate precursors.[52,55]

2.3 Excited states

2.3.1 Characterization of singlet and triplet excited states. The reactive intermediates discussed above that have been detected directly with LFP are formed photochemically from an excited state of their precursors. However, the excited state of the precursor is not always sufficiently long-lived to be detected using nanosecond LFP. As the lifetimes of triplet excited states typically range from 10^{-7} s to approximately 1 s,[57] they are well within the timescale limits of nanosecond LFP apparatus. In contrast, the majority of singlet excited states are shorter-lived and their observation requires much faster time resolution. A wealth of information about the lifetimes and reactivities of various triplet excited states has been obtained by detecting their transient UV–Vis absorption and monitoring their decay in the presence of quenchers.

The triplet excited state of various carbonyl and aromatic compounds have been detected and characterized using LFP.[57–64] For example, Stewart *et al.* used LFP to study the effect of substituents on the photophysical properties of anthanthrene and anthanthrone derivatives (Scheme 15).[61] Their study revealed that increasing the number of substituents red shifted the absorption and emission features in both the anthanthrene and anthanthrone series. For the investigated molecules, it was found that the singlet excited state decayed by both radiative decay and intersystem crossing. The anthanthrone derivatives were found to have quantum yields greater than 0.8 for intersystem crossing, whereas



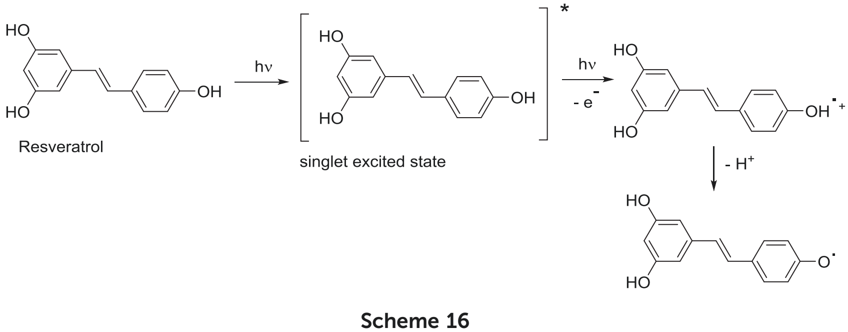
Scheme 15

the anthanthrene derivatives showed comparable rates for intersystem crossing and radiative decay.

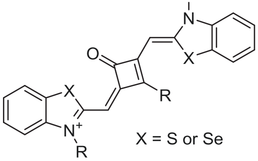
Although singlet excited states are shorted lived than triplet excited state, they can also be characterized using LFP. The photochemical properties of resveratrol were studied by Dzeba and co-workers to better understand its role as an antioxidant (Scheme 16).[65] They used ultrafast LFP of resveratrol to reveal the formation of its short-lived singlet excited state. Biphotonic LFP of resveratrol resulted in the formation of its radical cation, which undergoes efficient deprotonation to form the corresponding phenoxyl radical. Another interesting report on the detection of singlet excited states comes from the Arce and Crespo-Hernandez research teams, who studied the photochemistry of 1,6-dinitropyrene to elucidate it photodegradation process, as nitropolycyclic aromatic derivatives are mutagenic and carcinogenic pollutants in the environment.[66] With the aid of ultrafast and nanosecond LFP, they determined that the singlet excited state decays by photodissociation to form a nitropyrenoxy radical in competition with intersystem crossing to the triplet excited state. At lower excitation wavelengths, the photodissociation process becomes more prominent.

Another important class of photoactive compounds is dyes,[67,68] especially those used in solar cells. Conceição *et al.* prepared and investigated the photochemistry of benzothiazole- and benzoselenazole-derived aminosquarylium dyes for solar cells (Scheme 17).[67] They characterized the triplet excited states of these dyes using LFP and triplet sensitizers.

The triplet excited state of thiophenol was identified using LFP, and this characterization was further supported by quenching studies.[69] In addition, LFP of thiophenol revealed the formation of the PhS•



Scheme 16



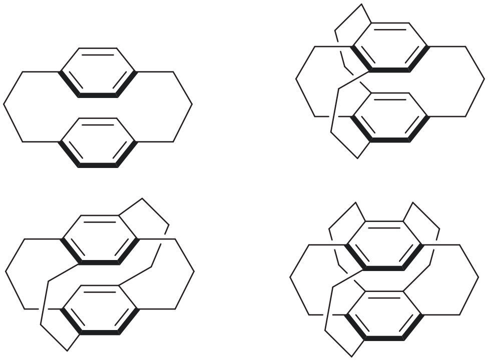
Scheme 17

radical, which is presumably formed from the singlet excited state of thiophenol.

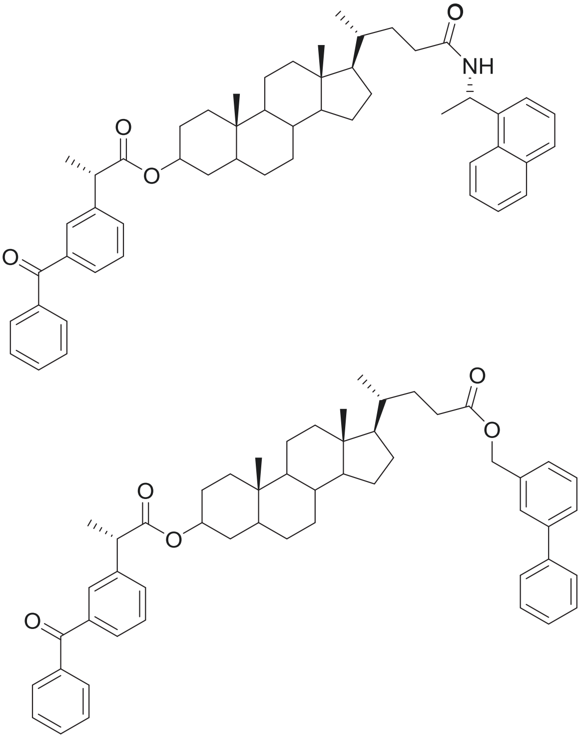
It should also be highlighted that LFP can be used to probe how the excited states of organic compounds are affected by the media in which they are dissolved or dispersed.[70–74] For example, the photophysical behavior of Eosin Y and Phloxine B in microcrystalline cellulose particles was investigated by Litman *et al.*[70] They used diffuse reflectance LFP to obtain the quantum yields for formation of the triplet excited states of these compounds at different concentrations, which provided an improved understand of how molecular crowding at higher concentrations affects the photophysical behavior. Similarly, the photophysics and photochemistry of a thionine dye has been investigated in reverse micelles.[71] LFP was used to characterize the triplet excited state of the thionine dye in the micelles and its quenching by aliphatic amines.

2.3.2 Identification of triplet exciplexes and excimers and triplet energy transfer in rigid systems. Excimers and exciplexes are complexes formed between an excited state of one molecule and the ground state of another. In excimers, the two molecules are the same and in exciplexes, they are different. LFP has been used to address the formation of excimers and exciplexes. Yamaji *et al.*[75] investigated formation of triplet excimers of [3_n]cyclophanes, which consisted of two benzene rings tethered together by oliogmethylene chains (Scheme 18). Most of the cyclophanes showed phosphorescence at cryogenic temperatures owing to excimer formation, whereas LFP of these compounds at ambient temperature allowed the direct detection of the triplet excimers, even for those that did not show phosphorescence.

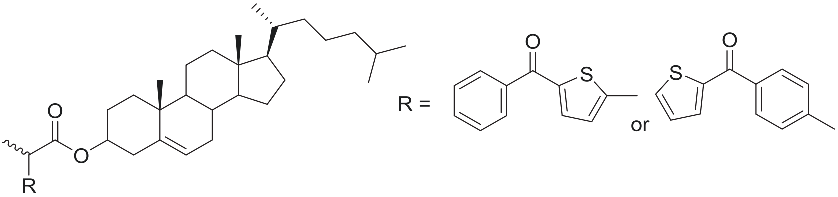
Exciplex formation has also been studied in rigid systems by Miranda and co-workers,[76] who investigated triplet exciplexes by tethering triplet donor and acceptor molecules onto a rigid framework (Scheme 19). In more detail, they linked benzophenone and naphthalene or biphenyl through a bile acid. LFP of this system allowed measurement of triplet energy transfer between the donor and the acceptor chromophores and estimation of the contribution of through-bond triplet exciplex formation.



Scheme 18



Scheme 19



Scheme 20

Furthermore, using a donor system tethered to a rigid steroidal scaffold, Miranda and co-workers studied triplet energy transfer between two chromophores to evaluate steric shielding *versus* σ–π orbital interactions of the donor with the scaffold (Scheme 20).[77] LFP studies demonstrated that the rate constants for intermolecular triplet energy transfer to triplet acceptor molecules were affected by the σ–π orbitals of the donors.

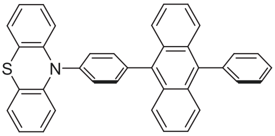
2.3.3 Electron transfer reactions of excited states. Upon electronic excitation of a molecule, its redox properties changes; therefore, an excited state that encounters an electron donor or acceptor can undergo electron transfer to form an ion pair. Electron transfer reactions are important owing to their various applications in photocatalysis and solar-powered water splitting. Furthermore, numerous biological processes involve electron transfer reactions, including photosynthesis and DNA UV damage repair. Electron transfer reactions can occur from both singlet and triplet excited states.

Within the field of charge transfer reactions, a new research area is emerging that is focused on characterizing the unique photophysical properties and reactivity of the excited state of carbocations.[78,79] These studies, which require ultrafast spectroscopy techniques, have potential for use in the development of solar energy harvesting materials. Furthermore, Lu *et al.* used ultrafast transient spectroscopy to study intramolecular electron transfer from the excited states of perylenediimide and naphthalene diimide radical anions.[80,81] The excited state properties of C_{70} radical anions and the nature of their electron transfer processes have also been investigated.[82]

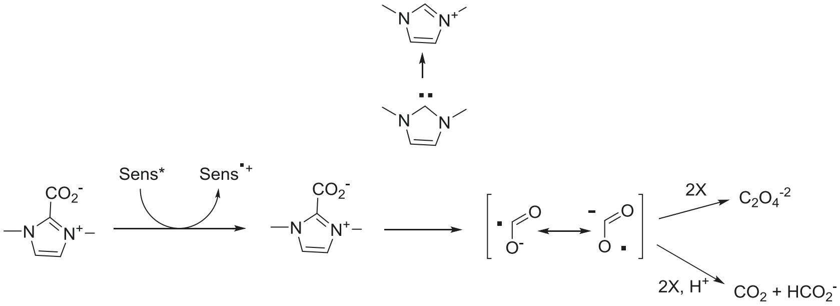
Electron transfer reactions have also been reported for systems in which the donor and the acceptor are linked together.[83–85] Hou and co-workers studied electron transfer in phenothiazine–anthracene dyads (Scheme 21) to explore spin–orbit charge recombination intersystem crossing as a function of their conformation.[85]

Bimolecular electron transfer reactions have also been reported for various donor–acceptor systems.[86–90] For example, Falvey and co-workers carried out the photoreduction of CO_2 using 1,3-dimethylimidazolium-2-carboxylate, as shown in Scheme 22.[84] Aoki *et al.* investigated photoinduced electron transfer (PET) from the singlet excited states of porphyrins to hexaazatriphenylene charge-transfer complexes using ultrafast absorption spectroscopy.[87]

The effect of the media has been explored for various charge-transfer systems.[91–96] For example, PET between the triplet excited state of thioxanthene-9-one and diphenylamine as a function of different solvents was investigated using LFP.[91] Another example comes from Altamirano *et al.*,



Scheme 21



Scheme 22

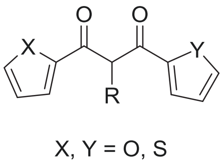
who studied the photoionization of (4-(1-pyrenyl)butyl)trimethylammonium bromide and 1-pyrene sulfonic acid in reverse micelles.[92]

Additionally, through triplet sensitization, some organic catalysts have been used to produce singlet oxygen species upon photolysis.[97–99] Singlet oxygen sensitizers in biological systems are ruled by the triplet excited state population and their lifetimes. In studies aimed at investigating photosensitization for biological applications, LFP has been used to obtain insight into the mechanism of photocytotoxicity of various compounds by clarifying the kinetics of the triplet-excited state. Using LFP, the photoinduced apoptosis mechanism of a new series of pyrrolo[3′,2′:6,7]cyclohepta[1,2-*d*]pyrimidin-2-amines was attributed to a long-lived triplet excited state and a significant singlet oxygen production ability.[99]

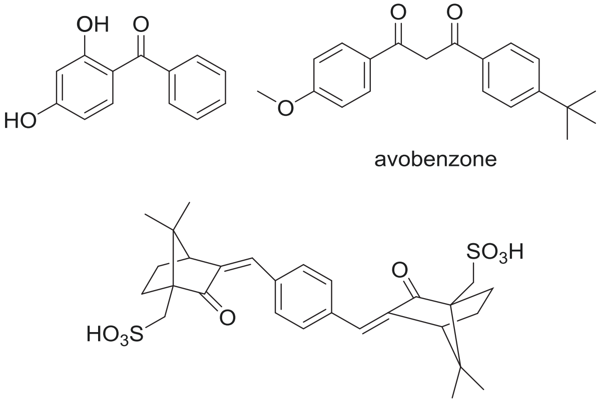
2.3.4 Excited state intramolecular proton transfer (ESIPT), phototautomerization processes, and hydrogen transfer reactions. ESIPT processes generally involve the transfer of an acidic proton to a basic site within a molecule. Typically, the proton is transferred between two heteroatoms to form a phototautomer, which reforms the starting material thermally. ESIPT processes are excellent for deactivating excited states without forming photoproducts; therefore, molecules that undergo efficient ESIPT processes can be used as sunscreens and photostabilizers. Furthermore, ESIPT is of potential use in applications such as sensing, bioimaging, and molecular switching. Because ESIPT generally does not yield any isolatable photoproducts, LFP is an ideal technique for identifying this process.

There is general interest in the keto–enol tautomerization of 1,3-diketone derivatives owing to their potential applications as sunscreens. For example, Suwa and Yamaji investigated the keto–enol tautomerization of 1,3-diketones bearing furan and thiophene moieties (Scheme 23).[100] These 1,3-diketones underwent enolization upon irradiation and the enols reformed the keto tautomers thermally. LFP of the 1,3-diketones revealed that the enol tautomers are formed from their triplet excited states. However, 1,3-diketones without alkyl substituents also yielded enol tautomers from their singlet excited states. Yamaji *et al.* further extended these investigations to study 1,3-diketone derivatives with halogen substituents.[101] LFP studies revealed that the 1,3-diketones with α-bromo substituents underwent debromination from the singlet excited state, whereas those with α-chloro substituents underwent α-cleavage from the singlet excited state.

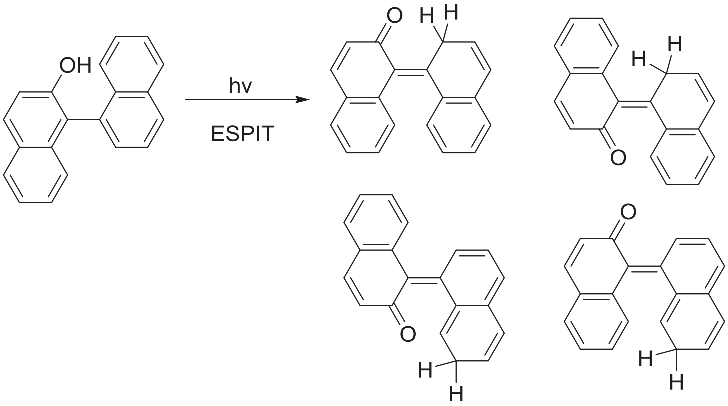
Abid *et al.* compared the photochemistry of the compounds displayed in Scheme 24 to explore their feasibility as sunscreens.[102] They used LFP to



Scheme 23



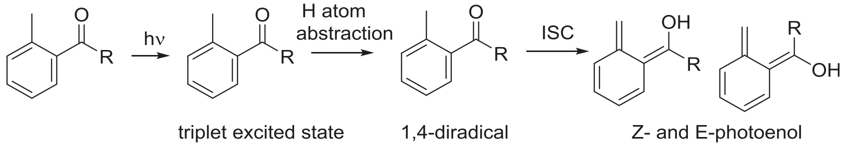
Scheme 24



Scheme 25

explore the ability of these compounds to undergo phototautomerization. The picosecond dynamics of avobenzone aided in the elucidation of the phototautomerization process in solution.[103] Furthermore, using LFP, Sarkar *et al.* showed that simple *o*-hydroxyacetophenone derivatives also undergo phototautomerization in both their triplet and singlet excited states.[104] Basarić and his research team also studied ESIPT in several different systems.[105–108] In thorough LFP studies, they were able to characterize several quinone methide derivatives and identify their reactivity. Arai and co-workers studied the effect of extended conjugation on the photochemistry of 2-hydroxychalcone derivatives and their ability to undergo ESIPT.[109] Freccero and co-workers used LFP to establish that 4- and 5-arylethynyl-substituted quaternary ammonium salts undergo phototautomerization upon irradiation.[110]

ESIPT from phenolic OH to the carbon atoms of aromatic rings has been reported by Lukeman *et al.*[111] LFP of hydroxy polycyclic aromatic derivatives (Scheme 25) allowed direct detection and characterization of the resulting quinone methide intermediates. Interestingly, some of



Scheme 26

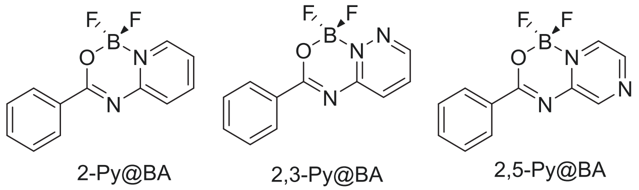
these quinone methides reacted further to form photoproducts through photocyclization or photoaddition to the solvent.

Das *et al.* investigated the photochemistry of an *o*-methylacetophone derivative (Scheme 26)[112] that does not yield any photoproducts in argon-saturated solution. However, LFP verified that this compound undergoes intramolecular H-atom abstraction to form the corresponding *E*- and *Z*-photoenols. Several other researchers have also investigated photo-induced H-atom transfer reactions using nanosecond LFP.[113–116]

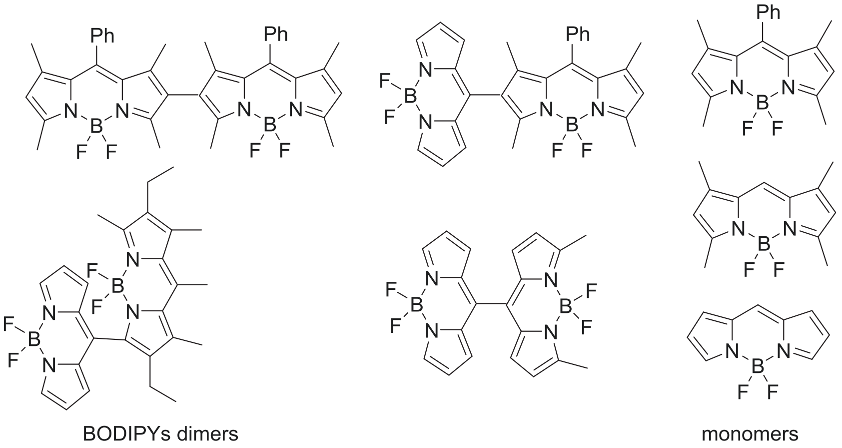
2.4 Boron

Difluoroboron compounds are known to exhibit very strong fluorescence, making them of interest for use in organic light-emitting diodes (LEDs) or as fluorescent probes for use in medicine. Yamaji *et al.* published a study aimed at creating strong solid-state fluorescent probes that emit blue light.[117] Among the studied compounds (Scheme 27), 2-Py@BA showed the best solid-state quantum yield of fluorescence (0.66), whereas the other two compounds showed quantum yields of approximately 0.16. The researchers used LFP to explain this difference, as 2,3-Py@BA and 2,5-Py@BA both showed triplet formation, whereas 2-Py@BA did not owing to its more efficient internal conversion ability.

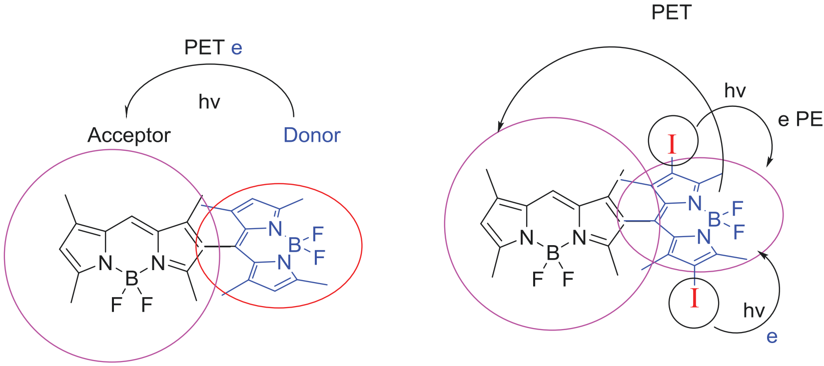
Difluoroboron compounds can also exhibit tunable reactivity. BODIPY complexes, a class of difluoroboron compounds with a chemical backbone of boron-dipyrromethene, have been targeted as heavy-atom- and metal-free organic triplet photosensitizers for use in the generation of singlet oxygen for medicinal applications. Zhang and collaborators have been especially productive in this field over the last three years. LFP was used to identify the absorption of the triplet excited state of the BODIPY groups and the effects of substitution at the *meso*-position with anthryl and pyrenyl substituents,[118] substitution at the *meso*-position with electron-withdrawing phenyl rings,[119] and substitution with naphthyl substituents.[120] The effect of the linkage position in BODIPY dimers was also evaluated by synthesizing dimers with monomers in different positions (Scheme 28).[121] Importantly, the dimers were able to form singlet oxygen, whereas the monomers were not, showing that dimerization is crucial for triplet formation. These investigations of substitution effects on triplet formation showed that BODIPY complexes have highly tunable triplet formation and ground-state absorption properties, making them intriguing fully organic photocatalysts for medicinal or synthetic applications. Zhang and collaborators also investigated the mechanism of triplet formation by BODIPY dimers. Using LFP, they were able to determine that the triplet is formed by PET from one monomer of the



Scheme 27



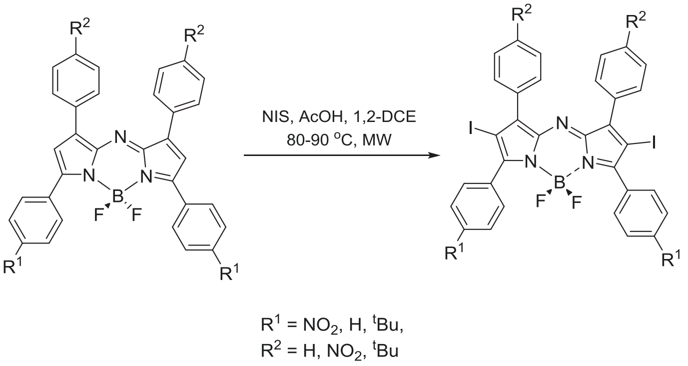
Scheme 28



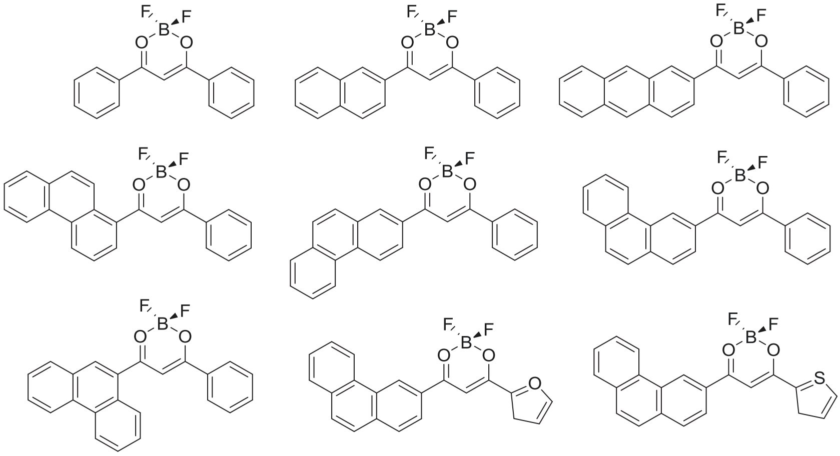
Scheme 29

dimer to the other monomer, followed by radical recombination, resulting in the formation of the triplet state (Scheme 29).[120,121] The proposed mechanism was supported by electrochemistry experiments, quantum mechanical calculations, and solvent effect studies.

Adding to the work on BODIPY complexes, Gut *et al.* created a series of aza-BODIPY complexes with very high singlet oxygen formation quantum yields (Scheme 30).[122] The reactivity of these compounds was investigated using both femto- and nanosecond LFP, with which the researchers were



Scheme 30

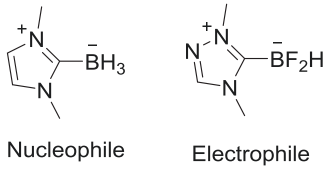


Scheme 31

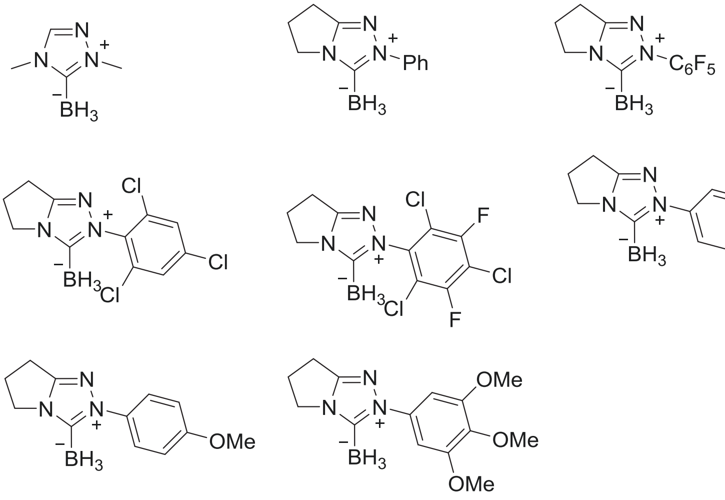
able to observe the formation and decay of the singlet and triplet excited states of the BODIPY complexes, respectively. Obłoza *et al.* also used hexaiodio-subphthalocyanine to obtain water-soluble photocatalysts with high triplet quantum yields that are capable of forming singlet oxygen with quantum yields of up to 0.90.[123] Femto- and nanosecond LFP were used to investigate the singlet and triplet excited states of these molecules, along with phosphorescence and electrochemical techniques.

As relatives of BODIPY compounds, difluoroboronated β-diketones are known to exhibit strong fluorescence in both solution and in the solid state. To examine the factors that influence these fluorescence properties, Mamiya *et al.* synthesized a series difluoroboronated β-diketones substituted with phenanthrene and other arenes (Scheme 31).[124] Nanosecond LFP was used to investigate the deactivation pathway of the singlet excited state of these compounds, and triplet excited states were observed for all but one compound. The researchers concluded that intersystem crossing competes with internal conversion for the deactivation of the singlet excited state. Adding to the work on efficient internal conversion, difluoroboronated β-diketones molecules bearing a ferrocene moiety were shown to be very photostable in comparison to the diketone without difluoroboron owing to high internal conversion.[125]

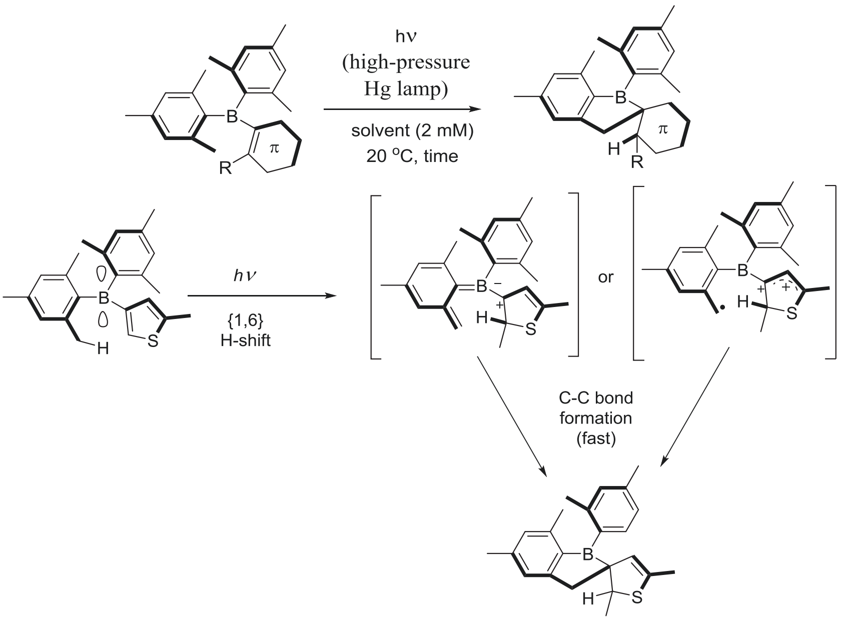
Difluoroboronated compounds can also be used to generate boryl radicals, which are typically nucleophilic and have found applications in synthesis and polymer chemistry. The Lacôte research group showed that by introducing boron difluoro groups to *N*-heterocyclic carbene compounds, the radical character could be tuned to become electrophilic rather than nucleophilic (Scheme 32).[126] Nanosecond LFP revealed the tunable reactivity of these compounds, as the difluoro radical added more efficiently to electron-rich alkenes, whereas a traditional borane derivative, a known nucleophile, added more efficiently to electron-poor alkenes, despite the kinetics of radical formation and bond dissociation energies being similar. Continuing their work on NHC-substituted boron compounds, the researchers also synthesized a series of NHC-boranes with different electron-donating and electron-withdrawing substituents to determine the effect of electronics on their behavior as radical polymerization coinitiators (Scheme 33).[127] Overall, the boron radicals were still nucleophilic and LFP showed that the addition rate of the boryl radicals to methyl acrylate was correlated to their electronegativity.



Scheme 32



Scheme 33



Scheme 34

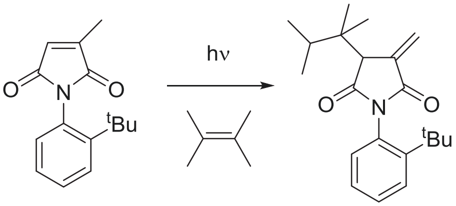
Finally, the Yamaguchi group reported a novel photochemical reaction type for boron compounds that was analogous to the Nazarov cyclization.[128] LFP was used to investigate the reaction mechanism, which is believed to involve a [1,6]-sigmatropic hydrogen shift. Transient spectra were not observed in DCM or toluene but were observed in ACN, suggesting that the intermediate had partial ionic character. The thermodynamic constants extracted from the LFP measurements and quantum mechanical calculations indicated that the intermediate following the [1,6] hydrogen shift was likely a zwitterionic form of the intermediate as shown in Scheme 34. Based on these findings, the researchers suggested that boron atoms could function as analogs of carbocations in pericyclic reactions, which could be of significant synthetic appeal.

3 Photochemistry applications

LFP is a powerful tool that can help influence the use of photochemistry for various applications ranging from the unlocking of photocages to photocatalysis and solar cell design. This technique provides insights into the mechanisms of the underlying reactions involved in these applications. By understanding the forces that drive the applications, molecules can be tuned to best serve their proposed applications. The following sections provide an introduction to how researchers use LFP to better understand their systems for applications.

3.1 Photochemical synthesis

Because light has been identified as a traceless reagent, there is a new focus on utilizing photochemistry for sustainable synthesis, with natural



Scheme 35

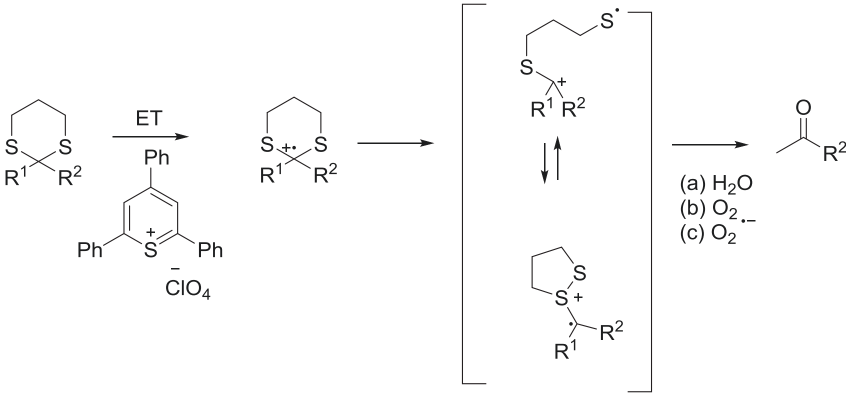
sunlight or energy-efficient LEDs as light sources to drive the reactions. The increased use of photochemistry in synthesis has in turn led to more mechanistic studies of photochemically driven reactions.[129–132] An elegant example comes from Sivaguru and co-workers, who used visible light to drive a photo-ene reaction at ambient temperature (Scheme 35).[129] They verified the mechanism of the photo-ene reaction using LFP. Understanding the reaction mechanisms of photoreactions makes it easier to use them in synthetic applications.

3.2 Photocages

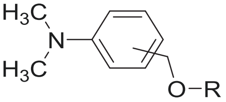
Photocages or photoremovable protecting groups are typically used as a method of protecting/deprotecting a functional group during chemical synthesis or releasing of a specific compound or intermediate for household goods or biological applications. As an example of the former, the Peñéñory group investigated the transformation of dithianes into carbonyl groups and determined the mechanism or the source of oxygen in the reaction.[133] LFP studies revealed that the deprotection process consists of step-wise single electron transfer from the thiapyrylium to the dithiane, followed by unimolecular fragmentation to form a radical cation, and subsequent reaction with a superoxide anion to form the resulting carbonyl. This mechanism, as shown in Scheme 36, was supported by steady-state UV–Vis absorption, which showed the necessity of oxygen, and computational studies.

Similarly, the Basarić group investigated using hydroxymethylaniline photocages (Scheme 37) to release alcohols and carboxylic acids.[134] Their experiments on nine different aniline compounds showed that while *para*-substituted hydroxymethylaniline compounds were poor photocages, *meta*- and *ortho*-substituted analogs efficiently released alcohols and carboxylic acids, respectively. LFP studies, supported by fluorescence measurements, showed that *meta*-substituted derivatives likely undergo heterolysis through an excited state that results in the formation of excited state carbocations. In contrast, the *ortho*-substituted derivatives were thought to undergo heterolysis through a conical intersection to subsequently form ground state carbocation intermediates.

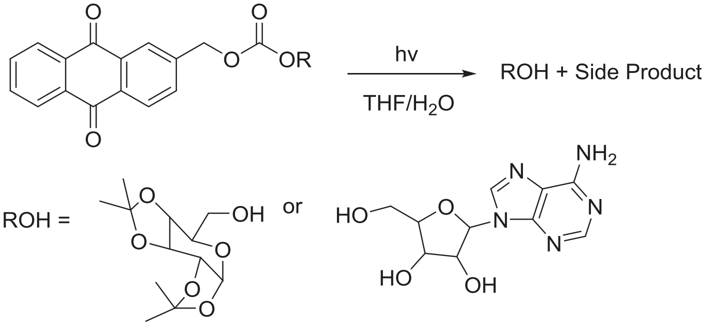
The Phillips research group investigated systems that release alcohols[135] and hydroxy radicals[397] using transient absorption spectroscopy as well as time-resolved Raman spectroscopy and computational methods (Scheme 38). The release of galactose and adenosine was triggered from anthraquinone methoxycarbonyls. Nanosecond LFP of acetonitrile,



Scheme 36



Scheme 37



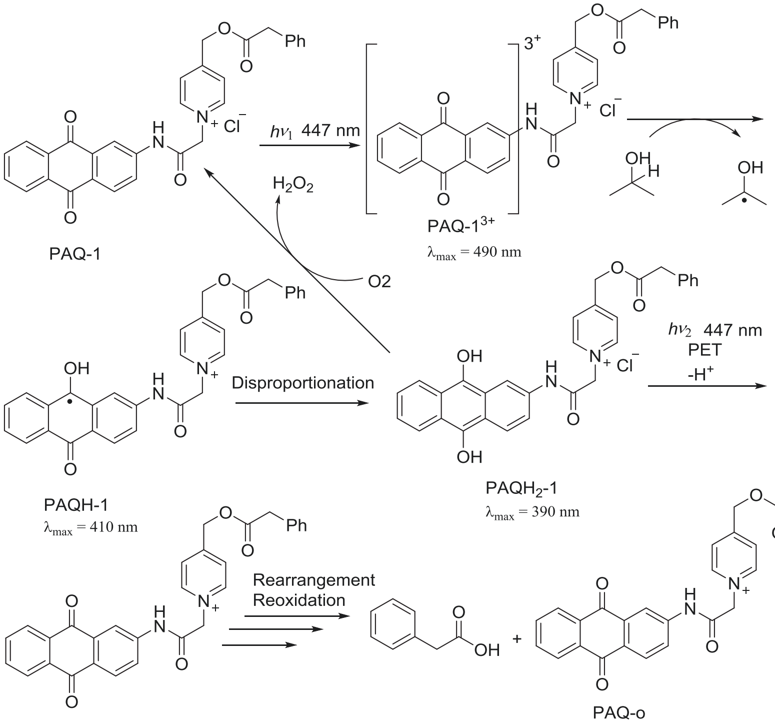
Scheme 38

isopropyl alcohol, and THF solutions was used to identify several different intermediates that formed upon irradiating anthraquinone methoxycarbonyls. Subsequent femtosecond transient spectroscopy allowed determination of the photorelease mechanism. By changing the solvent properties, the researchers were able to obtain experimental evidence for all the intermediates in their proposed mechanism.

The Phillips research group investigated the release of hydroxy radicals from blebbistatin by blue light using transient absorption spectroscopy, time-resolved Raman spectroscopy, and computational studies.[397] The researchers also showed that this phototoxic release of hydroxyl radicals was achievable by two-photon excitation. Takeshita and Hara also capitalized on the strength and flexibility provided by two-photon absorption to show that *trans*-azobenzene moieties encapsulated in β-cyclodextrin cages can be expelled by activating *trans*-to-*cis* isomerization of the photocage.[136] Continuing this research on two-photon photorelease, the

Falvey group constructed a step-wise two-photon absorbing dyad that released a carboxylate anion (Scheme 39).[137] The dyad consisted of an anthraquinone (AQ) that was designed to absorb a photon and transfer an electron to an *N*-alkylpicolinium (NAP) moiety, causing the release of the carboxylate anion. As planned, the AQ and NAP dyad was observed to release the carboxylate anion, but LFP analysis ruled out direct energy transfer from the AQ to the NAP moiety. Instead, the LFP experiments suggested that the reaction proceeds *via* abstraction of a proton from the solvent by the AQ to form a semiquinone, which rearranges to form a $PAQH_2$ diol that can absorb another photon to release the carboxylate anion following the loss of a proton. This proposed mechanism is displayed in Scheme 39.

Lhiaubet-Vallet and co-workers sought to create a dyad that would release a UV-A filter to protect the nonsteroidal anti-inflammatory drug diclofenac from a photoreaction that forms a carbazole, which can cause skin damage by radical formation.[138] Avobenzone was chosen as the UV-A filter because it had previously been used in cosmetic applications for that purpose. The coupled avobenzone and diclofenac compound was shown to release two compounds upon irradiation in diethylene glycol. The release of avobenzone from the dyad was shown to protect the diclofenac moiety, but it did not completely inhibit the carbazole forming reaction. LFP studies revealed evidence for the triplet excited state of the dyad molecule, which is thought to be the source of the release.



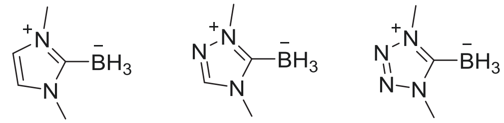
Scheme 39

3.3 Polymerization

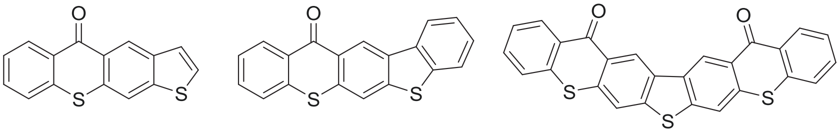
Choosing the appropriate photoinitiator for a polymerization reaction can be influenced by the dynamics of the produced excited states. The choice of photoinitiator can be influenced by whether the photoinitiator forms a singlet or triplet excited state or by whether the photoinitiation is the result of energy or electron transfer. Thus, LFP has been a valuable tool for polymer chemists to observe excited photoinitiators and their interactions with monomers.

Between 2015 and 2018, Lalevée and collaborators published an impressive 24 papers involving the LFP of photoinitiating or polymer-based compounds. LFP was used to investigate photoinitiators that resulted in neutral radicals,[139–141] to observe radical cations,[142,143] or to provide evidence for singlet reactivity, as indicated by the absence of absorption or the observation of weak absorption.[144–157] These papers describe both organic and inorganic photoinitiators, many of which absorb light in the visible region, allowing for more sustainable polymerization by LEDs. Capitalizing on the use of borane derivatives, the researchers were able to achieve radical initiated polymerization using visible light irradiation.[141] They also used LFP to observe the cations produced by photoacid release from a naphthalimide–iodonium complex, which was found to promote cationic and radical polymerization under LED irradiation.[142] LFP was used to investigate the differences in reactivity among the three boryl radicals produced by photolysis (Scheme 40). Dihydroxyanthraquinones are an example of initiators capable of promoting both cationic and radical poylmerizations using blue LEDs.[154] When LFP was performed on these compounds, a triplet excited state was not observed, which the researchers attributed to the singlet reactivity of these compounds. Iron complexes were also used for both cationic and radical polymerizations from the singlet surface under LED irradiation, as verified by LFP and time-resolved fluorescence spectroscopy.[156]

Initiators for radical polymerization have also been heavily studied during this time frame.[158–171] In many studies, quenching of the observed radical intermediates was investigated using either radical polymerization monomers or known radical quenchers. Thioxanthones were among the most thoroughly studied of the various initiators. For example, the groups of Arsu and Jockusch synthesized a few thioxanthone–benzothiophene systems (Scheme 41) and investigated their reactivity.[166] The triplet excited states of these compounds were observed in LFP studies and quenching studies with pyrene were used to elucidate their energy transfer abilities. Another study undertaken in the Gescheidt group investigated bis(acyl)phosphane oxide (BAPO) compounds and their ability to induce radical polymerization.[163] In this



Scheme 40



Scheme 41

case, LFP was used to observe the formation of a phosphorus-based radical and to perform quenching studies with various monomers with different electronic characters. These quenching studies revealed that the phosphanoyl radicals were preferentially added to electron-rich monomers.

Several groups have also studied ionic polymerization. Most of the studied ionic photoinitiators were cationic,[172–176] with only one reaction being induced by an anionic photoinitiator.[177] Additionally, a few photoinitiators could act as a radical or cationic initiator, making them valuable photoinitiators owing to their dual functionality.[178–182] One type of cationic photoinitiator is phenothiazine, which promotes a radical reaction by donating an electron to form a radical cation intermediate. A report from the Matyjaszewski group used LFP to show that the excited state of a phenothiazine compound was a strong reductant capable of donating an electron (Fig. 3).[173] An example of anionic polymerization using ketoprofen was published by the Wan group.[177] Using LFP, the carbanion form of ketoprofen was investigated in both pristine solution and in the presence of two methacrylate monomers, which exhibited quenching effects. The mechanism of this anionic polymerization is depictured in Fig. 4. As dual-action photoinitiators, squarine and squarylium dyes can be used to initiate radical or cationic polymerizations depending on their environment. LFP was used to confirm this behavior for a squarine dye using different photoinitiators with different electronic properties, as shown in Scheme 42.[180] The excited state of the squarine dye, the radical anion form of the dye, and the cationic form of the dye were observed in LFP transient spectra by changing the co-initiator used in the experiment.

3.4 Macromolecules

In addition to studying the formation mechanisms of polymers and other macromolecules and the factors that affect such processes, the properties of these larger molecules can also be investigated. For example, LFP was used to compare the behavior of a polymer bearing zinc porphyrin pendants with that of the corresponding monomer (Scheme 43).[183] The researchers found that the polymers could more effectively quench their singlet excited states through excimer formation by the zinc porphyrin pendants, resulting in a lower triplet excited state yield than that obtained with the monomer. Another paper on a series of zinc porphyrin pendant polymers made the observation that singlet–singlet annihilation was more prevalent in polymers with a greater extent of stacked structures.[184] It was also observed that accumulated triplet excited states on the zinc pendants could also act as quenchers.

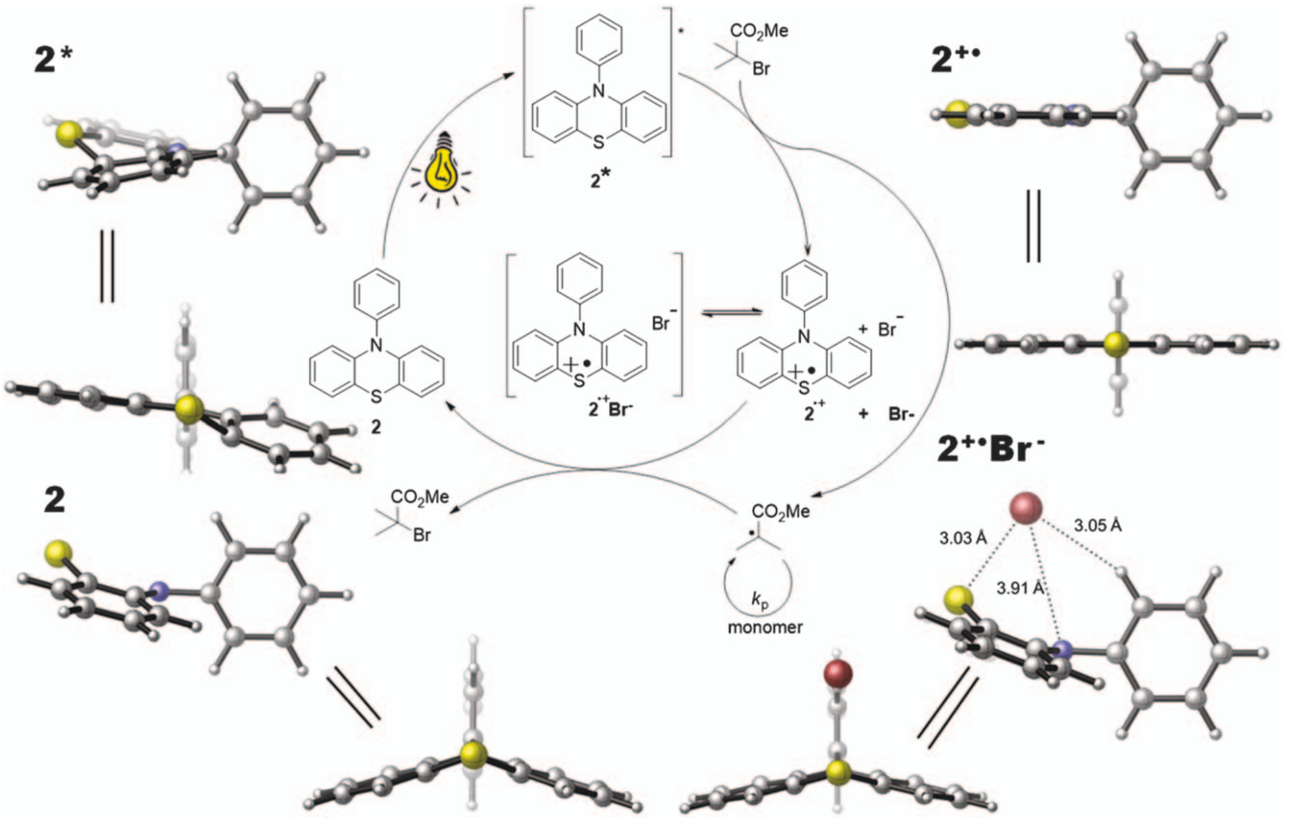


Fig. 3 Mechanism of phenothiazine reactivity. Reproduced from ref. 173 with permission from American Chemical Society, Copyright 2016.

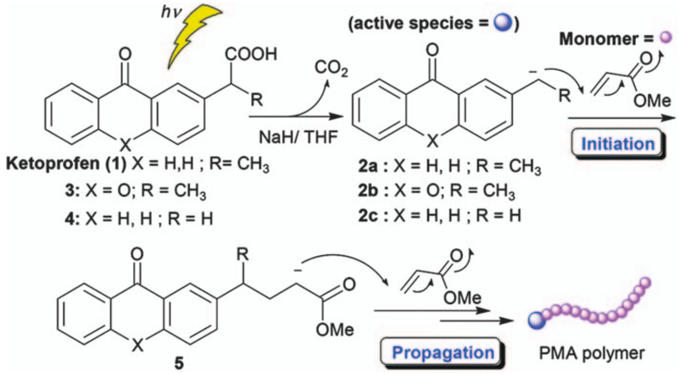
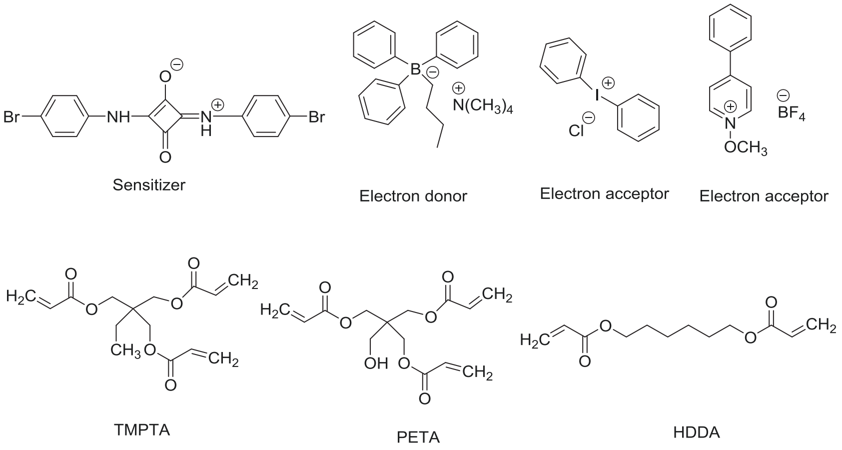
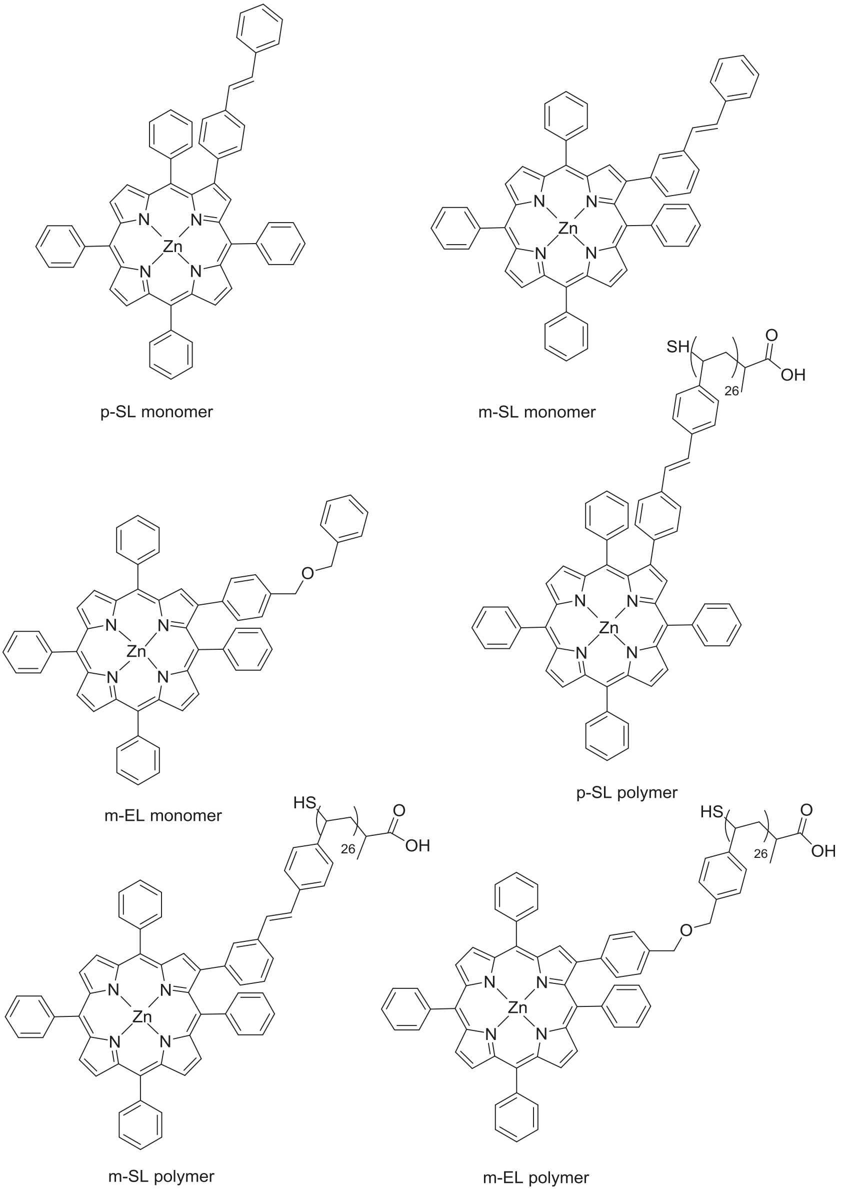


Fig. 4 Anionic photopolymerization with ketoprofen. Reproduced from ref. 177 with permission from the Royal Society of Chemistry.



Scheme 42

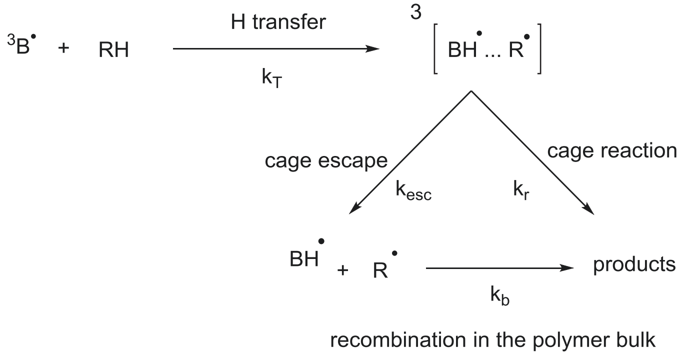
The interactions of small molecules within polymers and macromolecules are also of importance. Constrained environments such as films can affect the lifetimes and decay pathways of molecules trapped inside. Ketyl radicals produced by excitation of benzophenone and subsequent hydrogen abstraction were studied inside polymers[185] and thin films[186–188] to elucidate how constrained media affect the decay lifetimes. When benzophenone ketyl radicals were formed by hydrogen abstraction from a polymer fragment inside a polymer cage, the resulting radical pairs could decay quickly by geminate recombination. Alternatively, the ketyl radical could escape the cage and decay elsewhere in the bulk polymer. LFP was used to characterize these time-separated processes, which are depicted schematically in Scheme 44. Further studies investigated the effect of elongating the polymer films, which showed slower bulk recombination by ketyl radicals that escaped the polymer cage. Additionally, benzophenone triplet excited state lifetimes were measured in nanogel environments.[189] The nanogel environment (Fig. 5) was



Scheme 43

shown to increase the lifetime of the benzophenone and also be chemically tuned to initiate the polymerization of acrylates by donating a radical. Other LFP experiments in thin films were aimed at investigating the confirmation effects of photoswitchable *N*-salicylidene anilines[190] and the use of Rose Bengal dyes as fluorescent traps.[191]

The photoinitiating behavior of polymers and macromolecules has also been of interest to many researchers. Polyamidoamine (PAMAM) dendrimers are popular owing to their wide availability, water solubility, and ease of functionalization. Using LFP transient and quenching



Scheme 44

studies, PAMAM dendrimers were shown to be a reliable radical generator and act as an electron donor to several dye molecules, examples of which are shown in Fig. 6.[192–195] Interestingly, in these systems, the electron donating ability increases with dendrimer size. Energy transfer from a triplet polymer species to a singlet fullerene derivative has also been investigated by LFP (Fig. 7).[196] Additionally, LFP studies showed fullerenes to be useful as cationic photoinitiators that can help to improve the spatial resolution of laser writing.[197] Although direct energy transfer was favorable, the researchers could not rule out electron transfer followed by extremely fast back electron transfer in the geminate pairs as an energy transfer mechanism. C_{60} conjugates (Fig. 7) were also observed to be useful in photosensitizing and singlet oxygen generation.[198]

PET from macromolecules such as polymerized carbon dots (Fig. 8)[199] and graphene quantum dots[200] was investigated using LFP to elucidate the electron transfer mechanisms. In addition, PET in supramolecular systems was studied by Gopidas and co-workers by dissolving equimolar amounts of β-cyclodextrin (β-CD), pyrene-linked adamantane (PYAD), and *tert*-butylpyromellitic diimide (PMDI) in aqueous solution, which self-assembled to form the supramolecular donor–acceptor system PYAD–β-CD–PMDI.[201]

3.5 Photocatalysts

While most of the work described in this chapter is primarily focused on organic compounds, compounds containing metals and metalloids, which are used as catalysts, are also very important. This section highlights some studies that have used LFP to investigate such catalysts for use in water splitting, triplet sensitization, solar cell cocatalysis, radical scavenging, and gas binding. Additionally, special consideration has been paid to ruthenium-based catalysts, as they are the most commonly investigated photocatalysts.

With the worldwide rise in energy demands and the emphasis on reducing the use of fossil fuels, the generation of H_2 gas from water as a renewable source of power has become the subject of intensified

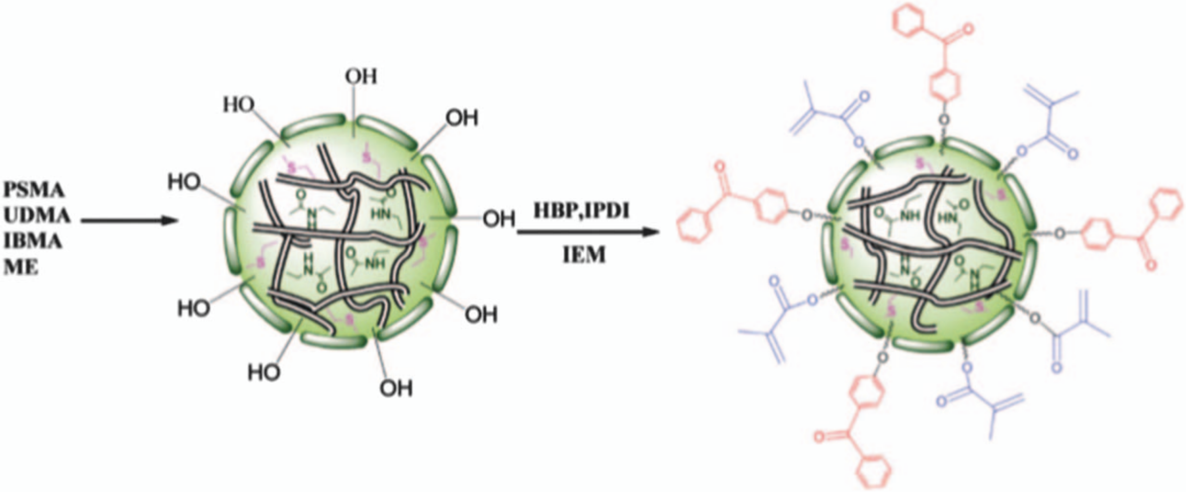


Fig. 5 Schematic of the nanogel system. Reproduced from ref. 189 with permission from the Royal Society of Chemistry.

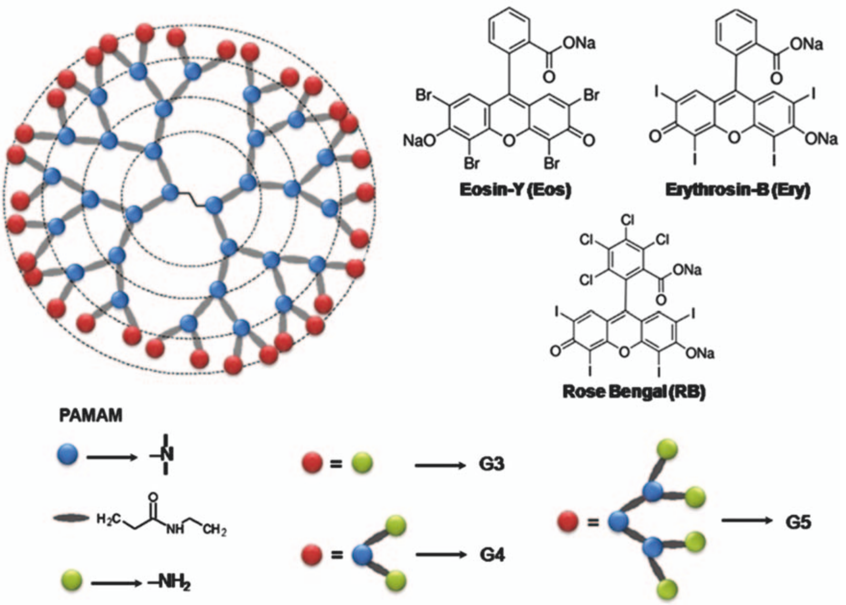


Fig. 6 PAMAM dendrimer and various dyes. Reproduced from ref. 192 with permission from Elsevier, Copyright 2016.

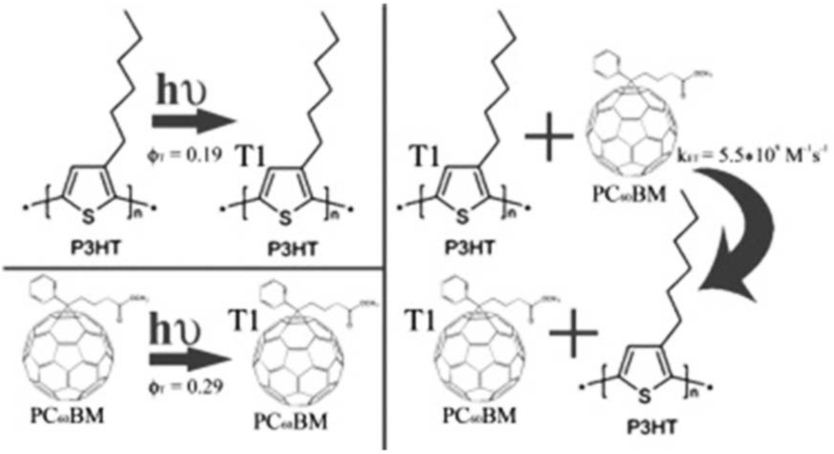


Fig. 7 Polymer to fullerene energy transfer. Reproduced from ref. 196 with permission from Elsevier, Copyright 2015.

research. Much of the research in this area has focused primarily on cobalt complexes,[202–204] TiO_2-based materials,[205,206] and other metal-based systems.[207,208] LFP of these compounds has primarily been used to investigate the electron transfer kinetics and fates of the intermediates involved in the H_2 production cycle. In a different approach to photocatalytic water splitting, one study on these photocatalytic compounds showed that water could also be used as an oxygen source in asymmetric epoxidation. The Nam group used a high-valent manganese

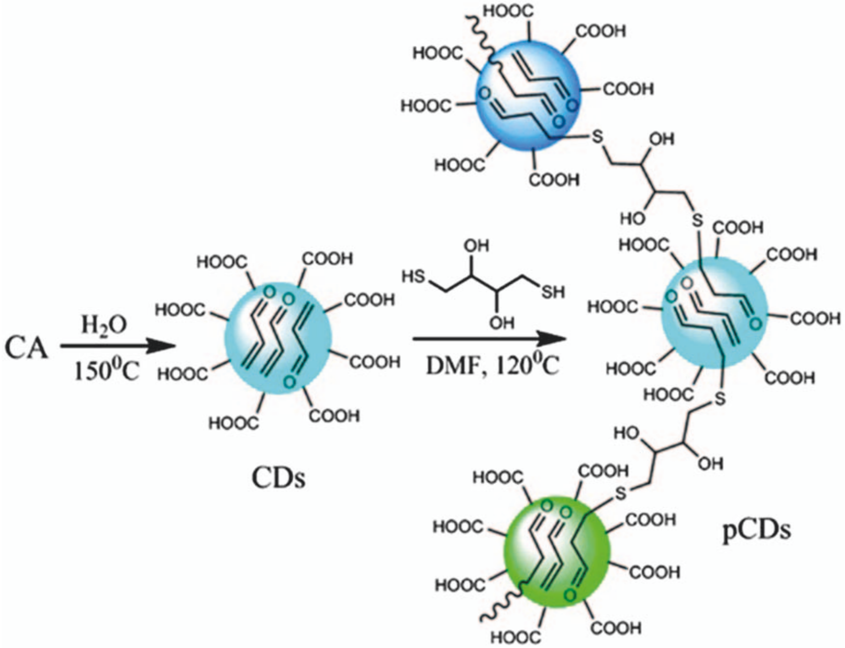


Fig. 8 Polymerized carbon dots. Reproduced from ref. 199 with permission from American Chemical Society, Copyright 2018.

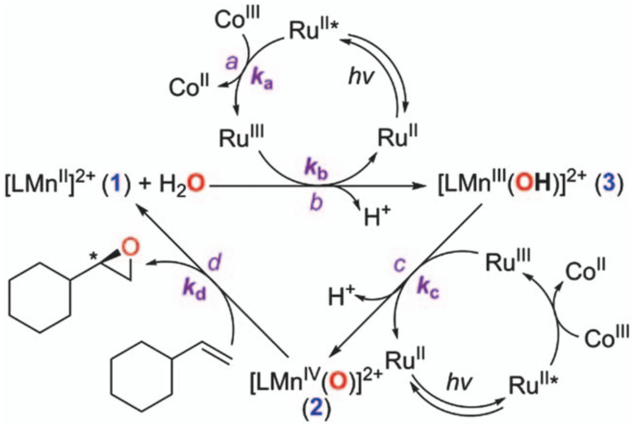


Fig. 9 Mechanistic details for enantioselective epoxidation of terminal olefins mediated by a high-valent manganese catalyst. Reproduced from ref. 209 with permission from American Chemical Society. Copyright 2016.

compound to perform enantioselective epoxidation of terminal olefins using water as an oxygen source.[209] LFP allowed the electron transfer kinetics from Mn(II) to Mn(III) and from Mn(III) to a Ru(III) cocatalyst to be probed during the reaction. The mechanism for this epoxidation process is depicted in Fig. 9.

In the field of renewable energy, much work has also focused on the kinetics of processes related to solar cells, which typically contain

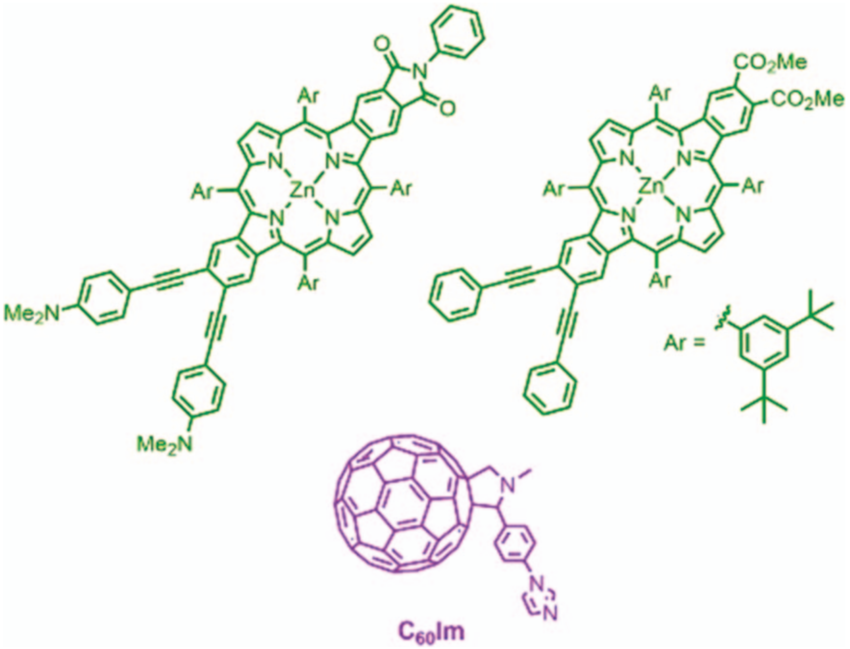


Fig. 10 Zinc porphyrins used to investigate push–pull effects. Reproduce from ref. 219 with permission from Canadian Science Publishing, Copyright 2018.

Ti-based catalysts.[210–217] Mostly, these studies have focused on increasing charge separation or stability, as investigated using LFP methods. Similarly, zinc compounds have been investigated as cocatalysts for solar cells.[218–221] A representative study on this group of compounds from the D'Souza group used LFP to investigate the push–pull effects of zinc porphyrins (Fig. 10).[219] They determined that electron-donating substituents on the porphyrin ring promoted faster charge separation, but the substituents did not significantly change the redox properties of the systems.

In addition to the generation of gases by photocatalytic means, the capture of gases by photocatalysts has also been investigated using LFP. A few iron compounds were investigated for their ability to bind discriminately to O_2 and CO for insights into hemoglobin binding.[222,223] A manganese/copper photocatalytic system was studied for its ability to capture CO_2 and convert it into environmentally benign CO.[224] Further studies were performed on the ability of photocatalysts to transfer oxygen during an oxidation reaction[225] and to use O_2 during a photocatalytic cycle, resulting in the oxygenation of organic compounds.[226]

LFP has been used to investigate the exploitation of the unique electronic properties of metalloids and metals for applications related to ionic liquids,[227,228] reduction/oxidation processes,[97–99,229–236] and radical scavenging,[237–239] as well as the formation of long-lived excited states or radicals for use in sensitization or other applications such as LEDs and charge storage/separation.[240–247] An example of a redox process investigation is a study focused on using a decatungstate photocatalyst to initiate hydrosilylation reactions.[233] A study on the radical scavenging ability of these photocatalysts was conducted by the Skibsted group using a kaempferol moiety bound to a Zn(II) system.[237] LFP was used to investigate the kinetics of radical binding to the Zn-kaempferol system, which was interpreted by comparison with the ionization potentials calculated using theoretical calculations. Additionally, the Machura group investigated rhenium complexes that had intraligand charge transfer characteristics and found that they were capable of forming a triplet excited state, identified by LFP, that was sufficiently long-lived for sensitization purposes.[242]

Among heavy metal complexes, ruthenium complexes are the most extensively studied and used in photocatalysis. Owing to their high redox reactivity in the excited state, these complexes have found applications in uncaging photoremovable protecting groups,[248] photocatalytic access to hydrated electrons,[249–251] incorporation in photoreactive supramolecular self-assemblies[252] and metal–organic frameworks,[253] artificial photosynthesis,[254] photocatalytic water oxidation,[255] and hydrogen evolution.[256] For the diverse Ru complexes used in such applications, LFP has been employed as a tool to study the photoredox reactivity and the redox nature of the excited state. LFP has also been used to study how the bimolecular quenching of two different Ru polypyridine complexes containing H-bond accepting sites by six *p*-substituted phenols switches between concerted electron–proton transfer and proton transfer mechanisms depending on the solvent.[257]

3.6 Theoretical calculations

Dóka and Lente carried out modeling studies to investigate spatial inhomogeneity in solutions during LFP.[258] They concluded that determining molar absorption coefficients from LFP experiments is complex and has potential for large errors.

4 Additional LFP reports outside the scope of this chapter

It is not possible to cover all the fields in which LFP has been used in the last three years. Therefore, plenty of significant high-quality work has been excluded from this chapter. However, we have listed the references for this work below in an attempt to highlight that this chapter is not comprehensive but focused on specific areas of research that use LFP and to provide information for those interested in learning more about the fields outside the scope of the chapter. These fields include radical combination kinetics in inorganic[259–266] and organic compounds,[267–283] compounds related to biological,[284–308] DNA,[309–344] or medicinal applications,[345–359] and compounds with atmospheric[360–373] or aquatic remediation implications.[66,374–396]

5 Conclusion and outlook

We hope the reader has obtained some appreciation of the breadth of research questions that LFP can address. LFP is no longer a technique only used by physical organic chemists studying reactive intermediates or photochemists identifying excited states, but rather a tool that is used by a wide spectrum of researchers in chemistry, physics, and material science. The success of LFP is closely associated with the ability to use theoretical calculations to aid in characterizing the transient spectra. Furthermore, the availability of turnkey nanosecond LFP apparatus has also widened the use of this technique.

Acknowledgements

We are grateful for the generous support from the National Science Foundation for research in the Gudmundsdottir group. DJS acknowledges Doctoral Enhancement, Ann P. Villalobos, Twitchell, and Laws Fellowships, and NA acknowledges a Halshall Fellowship from the Department of Chemistry at the University of Cincinnati.

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