

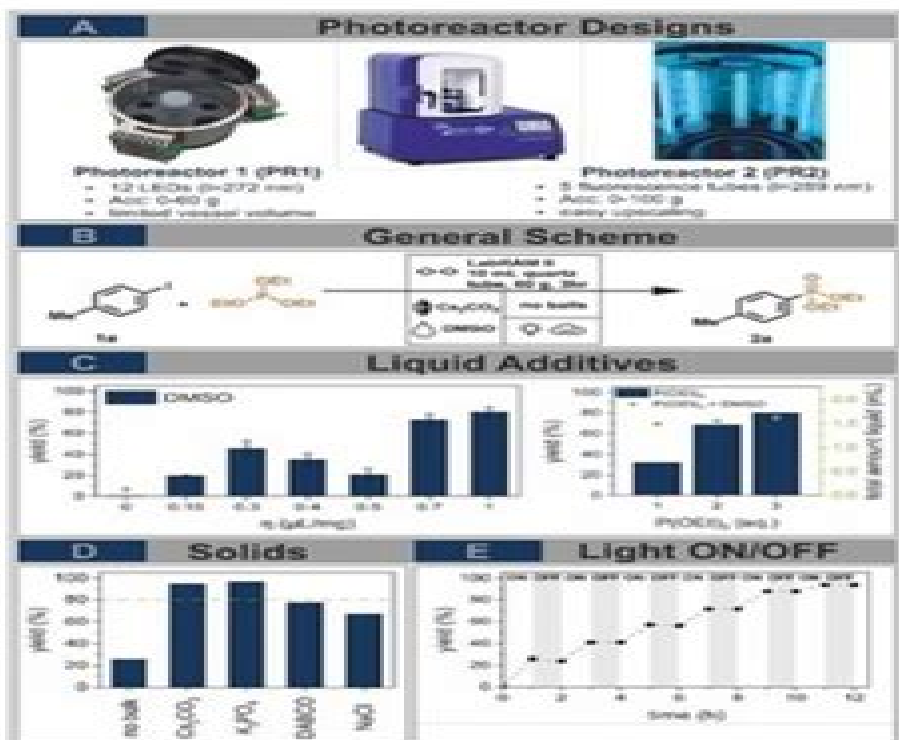
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**Abstract:** Herein, we developed the first photochemical reaction set-up under solid-state conditions using a resonant acoustic mixer (RAM), a novel mechanochemical methodology that eliminates the need for milling media. This innovation outperforms previous approaches in the ball mill under avoidance of milling balls and their related disadvantages. Further, it enables the facile performance and up-scaling of photochemical reactions up to a factor of ten. We investigate the influences of liquid and solid additives on the rheology and performance of two model reactions and demonstrate and compare the efficiency and applicability of two custom-designed photoreactors.

## Introduction

Light is one of the most environment-friendly activation modes, being both renewable and waste-free. However, conventional photochemical reactions suffer from sustainability issues, as they are often performed in highly diluted solutions due to the poor penetration depth of photons which results in substantial waste production.<sup>[1,2]</sup> Owing to the global incentives towards environmentally friendly synthesis alternatives, research on solvent-free pathways is increasing.<sup>[3-6]</sup> Nonetheless, typical photochemical approaches in the solid-state are performed on glass plates suffering from poor diffusion and requiring small sample batches. Mechanochemistry is an alternative solid-state synthesis strategy capable of overcoming these disadvantages. Mechanochemical synthesis describes the performance of chemical reactions under the impact of mechanical energy avoiding bulk solvents,<sup>[3-6]</sup> providing greener and more efficient reactions,<sup>[7-12]</sup> and also opening the pathways towards otherwise inaccessible products.<sup>[13,14]</sup> Next to the limited amount of publications on combining photochemistry and ball milling,<sup>[15-22]</sup> our working group has recently addressed the challenge by designing translucent quartz glass milling vials, opening up the opportunities for photochemical reactions with higher energy UVC light.<sup>[23-26]</sup> This innovation has overcome a major hurdle that enables efficient photochemical organic transformations without the use of metal catalysts and bulk solvents.<sup>[17,24]</sup> While these milling vessels are suitable for use with soft milling media made from polymers, quartz glass is not resistant to harder materials. In mechanochemistry, the usage of ball mills often



**Figure 1.** A: Photoreactor designs for the resonant acoustic mixer (RAM, middle), right (PR1) based on LED arrays, left (PR2) based on low-pressure mercury fluorescence tubes. B: General scheme of model reaction, photochemical phosphonation reaction of 4-iodotoluene (1a) with triethyl phosphite, reaction conditions if not stated otherwise: 0.38 mmol 1a, 3 eq. PhOEt, 2 eq.  $\text{Ca}_3\text{CO}_3$ , 1 g  $\text{SiO}_2$ , and DMSO ( $\eta=0.3$ ) were mixed in a 1.4 mL quartz glass tube at 60 g in the RAM for 3 h under ambient air conditions and constant irradiation of 12 UVC-LEDs ( $\lambda=272$  nm) in PR1. C: Investigation of the influence of liquid additives including, left: amount (n) of DMSO and right equivalents of PhOEt in dependency of the total amount of liquid. D: Influence of solid additives, green line indicates the yield under previously optimized conditions. E: Kinetic investigation of the phosphonation reaction of 4-iodotoluene (1 eq.) under optimized conditions using 3 eq. PhOEt, 1.2 g  $\text{K}_3\text{PO}_4$ , DMSO ( $\eta=1$ ) were mixed under ambient air conditions in the RAM at 60 g for 1 h under irradiation using PR1 followed by 1 h in the dark. The process was repeated until full conversion of the substrate to the product was achieved. Corresponding conversions were determined by  $^1\text{H-NMR}$  spectroscopy by analyzing a small amount of the crude reaction mixture.

# Solid State Photochemistry Monographs In Modern Chemistry 8

**M Woodhall**



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**Journal of Chemical Research**, 1977 Contains papers abstracted in Journal of chemical research Synopses Solid State Photochemistry Gerhard M. J. Schmidt, 1976 **Library of Congress Catalogs** Library of Congress, 1978 **Topics in Stereochemistry, Volume 25** Scott E. Denmark, Jay A.

Siegel, 2006-05-05 Diese Reihe zur Stereochemie wendet sich an fortgeschrittene Studenten und praktisch tätige Naturwissenschaftler in erster Linie organische Chemiker mit Interesse an Fragen der Physikochemie und Molekularphysik Themen sind vielfältige Aspekte der Stereochemie der räumlichen Anordnung organischer und biologischer Moleküle sowie die Auswirkungen der Orientierung auf die Eigenschaften der Verbindungen **Monographic Series** Library of Congress, 1977

**Supramolecular Photochemistry** V. Ramamurthy, Yoshihisa Inoue, 2011-07-07 This is the most updated comprehensive collection of monographs on all aspects of photochemistry and photophysics related to natural and synthetic inorganic organic and biological supramolecular systems Supramolecular Photochemistry Controlling Photochemical Processes addresses reactions in crystals organized assemblies monolayers zeolites clays silica micelles polymers dendrimers organic hosts supramolecular structures organic glass proteins and DNA and applications of photosystems in confined media This landmark publication describes the past present and future of this growing interdisciplinary area Directory of Graduate Research American Chemical Society. Committee on Professional Training, 1977 Faculties publications and doctoral theses in departments or divisions of chemistry chemical engineering biochemistry and pharmaceutical and or medicinal chemistry at universities in the United States and Canada **Photochemistry of Proteins and Nucleic Acids** A. D. McLaren, D.

Shugar, 2014-05-16 Photochemistry of Proteins and Nucleic Acids deals with photobiology The book briefly introduces the

principles of photochemistry with a short discussion on absorption spectra and related absorbing structures as well as on the general nature of photochemical reactions in solutions The following information covers how proteins nucleic acids viruses and their basic structures are chemically and physically affected when exposed to visible radiation and ultraviolet light The book explains that the site of action of the ultraviolet light when applied to proteins in a small active center shows that other parts of the protein are also affected considered as an energy transfer Then the general conclusions pertaining to the physicochemical and chemical changes in nucleic acids through directed radiation doses that are big enough to induce modifications are enumerated The text further explains that the physicochemical modifications which occur when proteins are irradiated can accompany loss in enzyme activity The kinetics of inactivation of dry enzymes is compared to the photochemistry of enzymes in a solution The inactivation of viruses is important as the process of replication can cause some formed viruses to spread in its environment worsening the state of infection Hence the problem which components in a biological system absorb effective radiation is analyzed The book is suitable for microbiologists microchemists and laboratory researchers and scientists dealing with infectious diseases

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