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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.^[1,2] Owing to the global incentives towards environmentally friendly synthesis alternatives, research on solvent-free pathways is increasing.^[3-6] 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,^[5-8] providing greener and more efficient reactions,^[7-12] and also opening the pathways towards otherwise inaccessible products.^[13,14] Next to the limited amount of publications on combining photochemistry and ball milling,^[15-22] 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.^[23-26] This innovation has overcome a major hurdle that enables efficient photochemical organic transformations without the use of metal catalysts and bulk solvents.^[17,24] 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

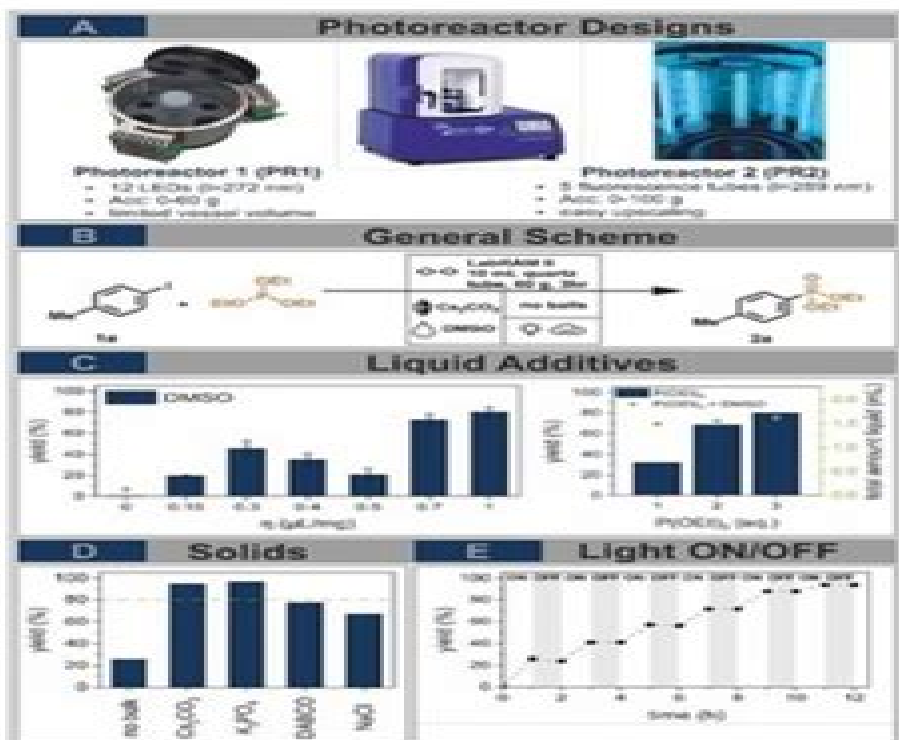


Figure 1. A: Photoreactor designs for the resonant acoustic mixer (RAM, middle), right (PR1) based on LED arrays, left (PR2) based on low-pressure mercury fluorescence tubes. B: General scheme of model reaction, photochemical phosphonation reaction of 4-iodotoluene (1a) with triethyl phosphite, reaction conditions if not stated otherwise: 0.38 mmol 1a, 3 eq. $\text{Pr}(\text{OEt})_3$, 2 eq. Ca_3CO_3 , 1 g 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 $\text{Pr}(\text{OEt})_3$ 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 (1a) under optimized conditions using 3 eq. $\text{Pr}(\text{OEt})_3$, 1.2 g K_2PO_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.

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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

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Molecular Inclusion and Molecular Recognition R. Gerdil, 1987 **Zeitschrift Für Kristallographie**, 1979 Doklady Akademii [a] nauk SSSR., 1987 **Subject Catalog** Library of Congress, 1976 Solid State Reactions Hermann Schmalzried, 1981 **Structure and Dynamics in Chemistry** Per Ahlberg, Lars-Olof Sundelöf, 1978 **Subject Catalog, 1976** Library of Congress, 1976 A to Z of Physicists, Updated Edition Darryl Leiter, 2019-11-01 A to Z of Physicists Updated Edition focuses not only on the lives and personalities of those profiled but also on their research and contributions to the field A fascinating and important element of this work is the attention paid to the obstacles that minority physicists had to overcome to reach their personal and professional goals Through incidents quotations and photographs the entries portray something of the human face which is often lost in books on science and scientists A to Z of Physicists Updated Edition features more than 150 entries and 51 black and white photographs Culturally inclusive and spanning the whole range of physicists from ancient times to the present day this is an ideal resource for students and general readers interested in the history of physics or the significant aspects of the personal and professional lives of important physicists People covered include Archimedes ca 285 212 BCE Homi Jehangir Bhabha 1909 1966 Pavel Alekseyevich Cherenkov 1904 1990 Marie Curie 1867 1934 George Gamow 1904 1968 Tsung Dao Lee 1926 present Lise Meitner 1878 1968 Yuval Ne eman 1925 2006 Johannes Stark 1874 1957 Nikola Tesla 1856 1943 Alessandro Volta 1745 1827 Hideki Yukawa 1907 1981 **Books in Print**, 1994 *Canadian Chemical Processing*, 1977

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