

Electronic Supplementary Information

Photosensitized addition of isopropanol to furanones in a 365 nm UV-LED microchip

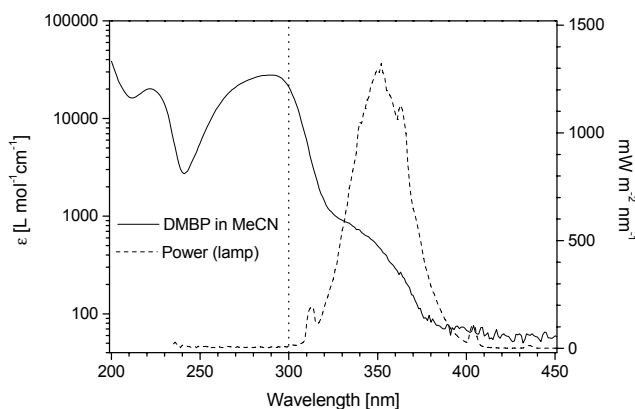
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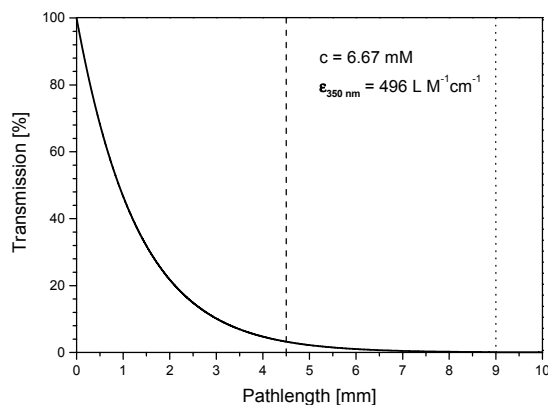
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Comparison of UV-spectrum of DMBP with emission spectrum of UVA (RPR-3500Å) lamp:



Light-penetration profile for Pyrex test tube:



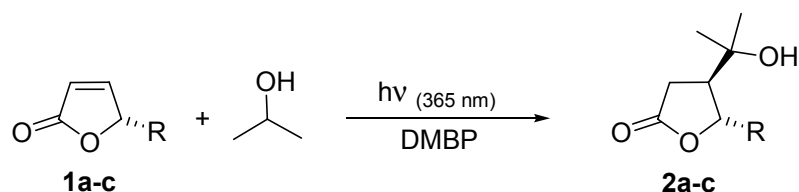
The vertical lines represent the effective pathlength (4.5 mm; dashed) and the inner diameter of Pyrex test tube (9 mm; dotted).

General procedure for irradiation in the LED-microchip:

A solution of the furanone (0.1 mmol) and DMBP (0.02 mmol) in isopropanol (3 mL) was purged with argon and loaded into a syringe pump. The reaction mixture was pumped through a microchip (Micronit Microfluidics FC_R150.676.2; channel width 150 μm , depth 150 μm , internal volume 13 μl) while irradiated by 6×365 nm high power LEDs (Seoul Optodevice, P8D236, 6×75 mW). After evaporation of the solvent, the conversion rate was determined by $^1\text{H-NMR}$ spectroscopy of the crude product. The signal integration for proton in the β -position of **1a-c** was compared to the signal integration for the acetal proton of **2a-c**.

General procedure for irradiation under batch conditions:

A solution of the furanone (0.5 mmol) and DMBP (0.1 mmol) in isopropanol (15 mL) was filled in a Pyrex glass tube (inner diameter: 9 mm) and purged with argon. The tube was stoppered and the reaction mixture was irradiated for 5 minutes at 350 ± 25 nm (Rayonet RPR-100, equipped with $16 \times \text{RPR-3500}\text{\AA}$ lamps). After evaporation of the solvent, the conversion rate was determined by $^1\text{H-NMR}$ spectroscopy of the crude product (see above). For characterization purposes, products were isolated and purified by column chromatography with silica gel.



All compounds (**2a-c**) are literature known and have been fully characterized earlier:

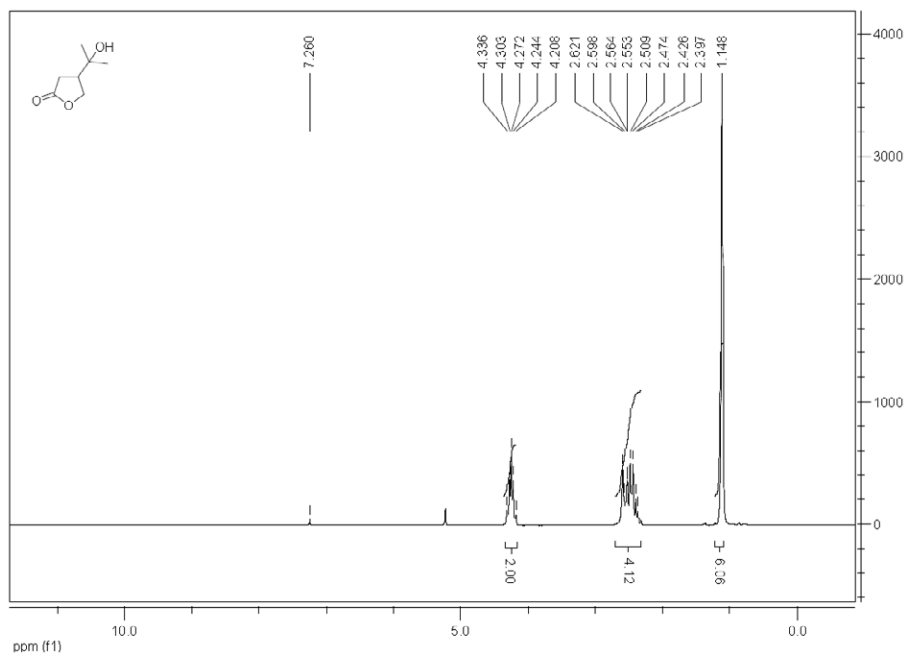
2a: K. Ohga and T. Matsuo, *J. Org. Chem.*, 1974, **39**, 106.

2b and **2c**: N. Hoffmann, *Tetrahedron: Asym.*, 1994, **5**, 879.

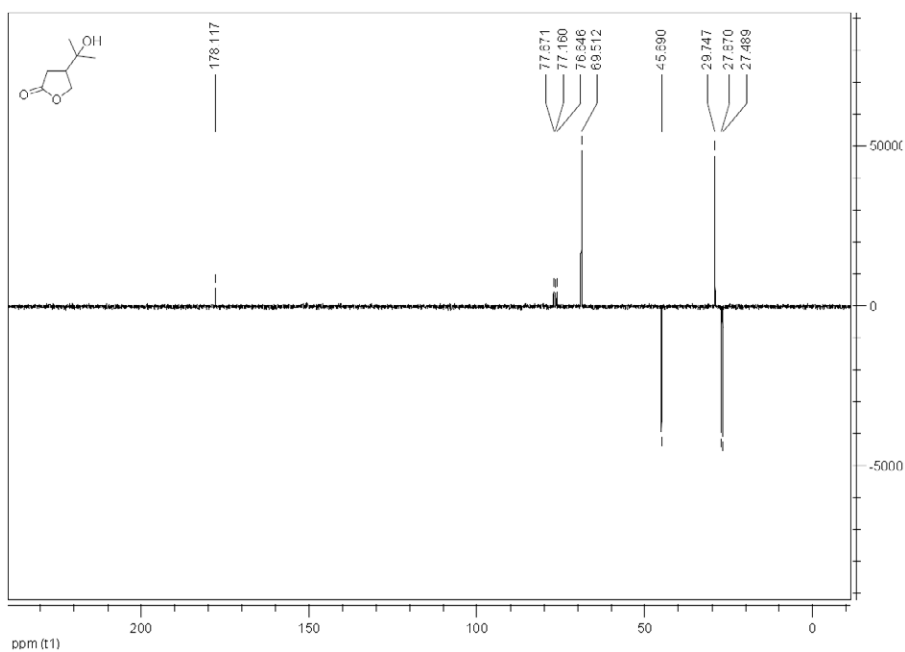
2a: R=H (yellow liquid).

Eluent: ethyl acetate/petroleum ether: 2.5/7.5

^1H NMR (250 MHz, CDCl_3): δ =4.29 (dd, J =12.4, 8.2Hz, 1H), 4.25 (dd, J =9.2, 10.8Hz, 1H), 2.34-2.62 (m, 4H), 1.14 (s, 6H) ppm.



^{13}C NMR (62 MHz, CDCl_3): δ =178.11, 69.51, 69.51, 45.69, 29.74, 27.87, 27.48 ppm.

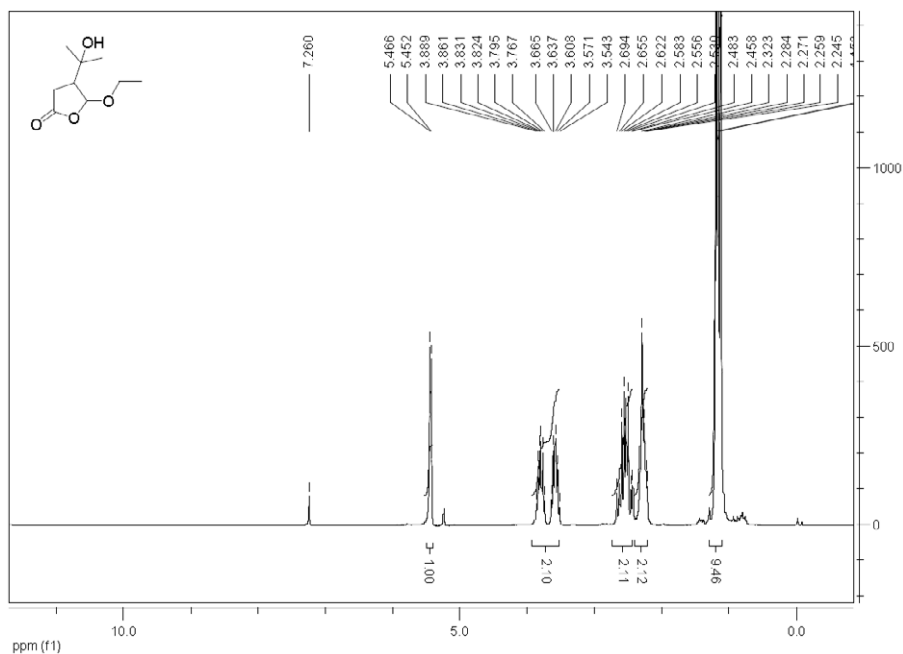


IR (film): ν =3455, 2975, 2932, 1772, 1372, 1182, 1022 cm^{-1} .

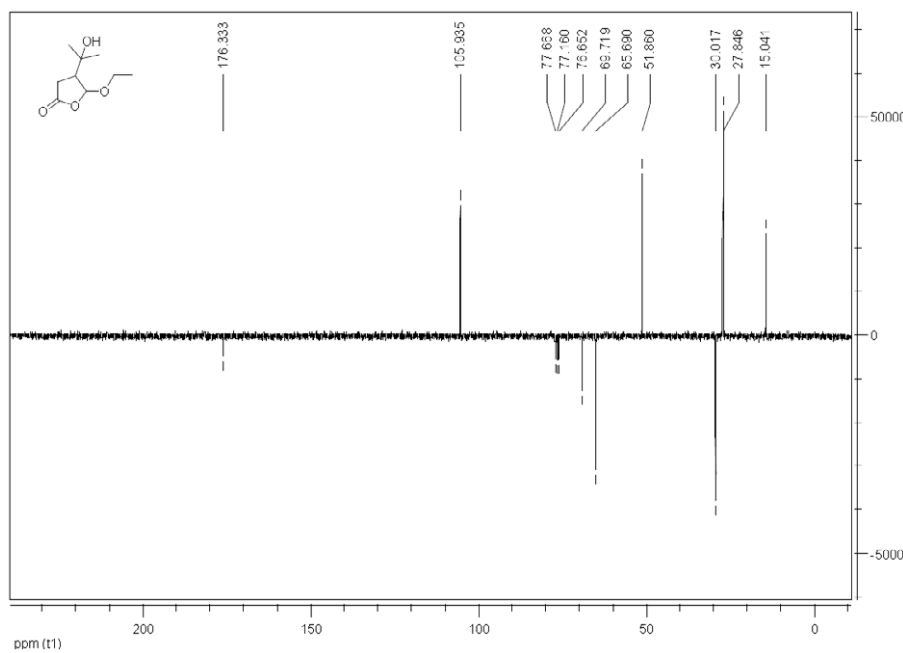
2b: R = OEt (yellow liquid).

Eluent: ethyl acetate/petroleum ether: 7/3.

^1H NMR (250 MHz, CDCl_3): δ =5.46 (d, J =3.3Hz 1H), 3.82 (dq, J =10.6, 7.1Hz, 1H), 3.66 (dq, J =11.0, 7.1Hz, 1H), 2.63 (dd, J =18.1, 9.8Hz, 1H), 2.50 (dd, J =18.1, 6.1Hz, 1H), 2.20 (m, 1H), 1.22 (s, 3H), 1.19 (t, J =7.0Hz, 3H), 1.15 (s, 3H) ppm.



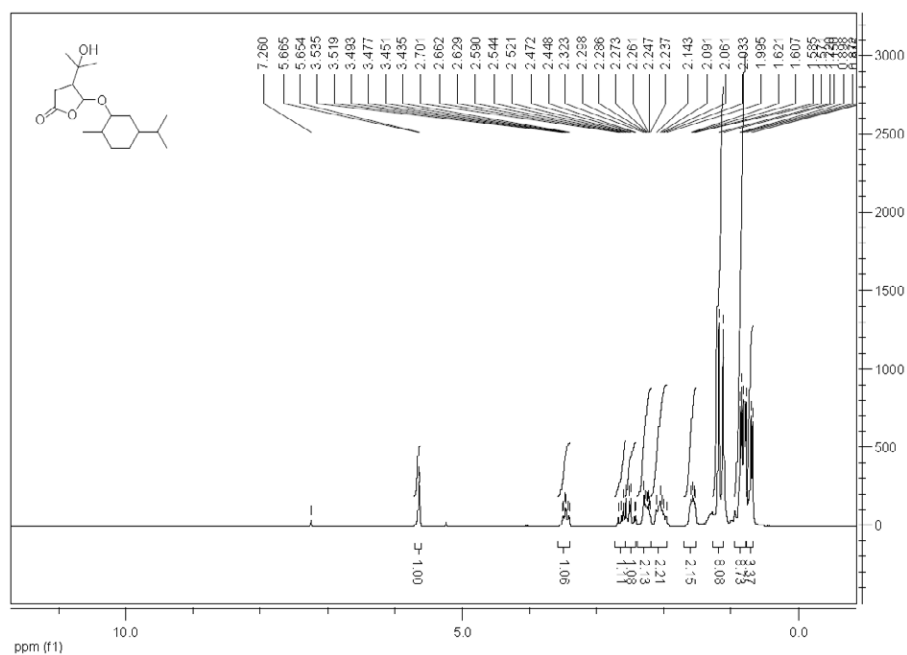
^{13}C NMR (62 MHz, CDCl_3): δ =176.33, 105.93, 69.71, 65.69, 51.86, 30.01, 27.84, 27.84, 15.04 ppm.



2c: R = Omenth (colorless crystals).

Eluent: ethyl acetate/petroleum ether: 1.5/8.5.

^1H NMR (250 MHz, CDCl_3): δ =5.65 (d, J =2.8Hz, 1H), 3.48 (dt, J =10.5, 3.9Hz, 1H), 2.64(dd, J =18.1, 6.1Hz, 1H), 2.49 (dd, J =18.1, 5.9Hz), 2.23-2.32 (m, 2H), 1.99-2.14 (m, 2H), 1.57-1.62 (m, 2H), 1.22 (s, 3H), 1.15 (s, 3H), 0.88 (d, J =6Hz, 3H), 0.83 (d, J =6.8Hz, 3H), 0.72 (d, J =6.8Hz, 3H) ppm.



^{13}C NMR (62 MHz, CDCl_3): δ =176.48, 102.32, 77.32, 69.92, 51.82, 47.81, 39.82, 34.32, 31.39, 30.15, 27.80, 27.77, 25.38, 23.08, 22.30, 20.94, 15.69 ppm.

