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One-pot chlorocarboxylation of toluenes with chlorine dioxide under photoirradiation

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Experimental

Materials: Sodium chlorite (NaClO₂) was commercially obtained from Sigma-Aldrich Co. The other chemicals were also purchased from commercial source and used without purification. ClO₂• solution (50 mM) was prepared by mixing sodium chlorite (NaClO₂, 200 mg) and 35% HCl aq (100 μL) in an aqueous solution (28 mL). The product analyses were performed on a Shimadzu GC-17A gas chromatograph and Shimadzu MS-QP5000 mass spectrometer.

General procedure for the chlorocarboxylation: A solution of acetone- d_6 (1.0 mL) containing 20 ppm toluene was added to aqueous solution (1.0 mL) containing ClO₂• (50 mM) to prepare a two-phase system. The solution was then irradiated with a 60 W LED lamp ($\lambda = 365$ nm, dotAqua model) at room temperature. After photoirradiation for 10 minutes, the corresponding product was identified and quantified by GCMS with that of an authentic sample.

Procedure for the ¹⁸O-label experiments: A solution of acetone- d_6 (1.0 mL) containing 20 ppm toluene was added to H₂O¹⁸ solution (1.0 mL) containing ClO₂• (50 mM) to prepare a two-phase system. The solution was then irradiated with a 60 W LED lamp (λ = 365 nm, dotAqua model) at room temperature under nitrogen atmosphere. After photoirradiation for 10 minutes, the corresponding product was identified and quantified by GC-MS with that of an authentic sample.

Procedure for the chlorocarboxylation of toluene- d_8 : A solution of acetone- d_6 (1.0 mL) containing 20 ppm toluene- d_8 was added to aqueous solution (1.0 mL) containing ClO₂• (50 mM) to prepare a two-phase system. The solution was then irradiated with a 60 W LED lamp ($\lambda = 365$ nm, dotAqua model) at room temperature. After photoirradiation for 10 minutes, the corresponding product was identified and quantified by GC-MS with that of an authentic sample.