

Photolysis of NSAIDs. II. Online LC-MS Determination

of Photodegradants from Carprofen

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Abstract

Carprofen in a 10% aqueous ethanol solution at a concentration of 0.10 mg/mL was subjected to photo-irradiation under sunlight for 24 hr. Four photodegradants were separated and subsequently followed an online determination of their quasi molecular ions using an LC-MS method. The HPLC consisted an Inertsil 5 ODS-80A (2.1 mm i.d. x 150 mm) column, and the mobile phase was initially CH₃CN: NH₄OAc (20 mM in D.I. H₂O) = 43: 57 (v/v). After 14 min, CH₃CN: NH₄OAc (20 mM in D.I. H₂O) was changed to 54: 46 (v/v). The UV detector was set at 260 nm. The parameters of LC-MS for mass determination were optimized with an API electron spray interface with negative mode of polarity (ESI⁻). The chemical structures of the degradants were elucidated based on the m/z of the quasi molecular ions. Two degradants were found to proceed via an initial dechlorination with C-Cl cleavage. Dechlorination was observed to be competing with decarboxylation, i.e. either reaction is in accord with our previously reported result with a first-order photodecomposition kinetics of carprofen.

Key words: photolysis, carprofen, NSAIDs, LC-MS, dechlorination