ABSTRACT

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Title of diploma thesis: Study of α-bromophenylacetic acid suitability as a model analyte for chiral separations using capillary electrophoresis as a separation technique II

Rizvi and Shamsi (Rizvi S.A.A., Shamsi S. A. Anal. Chem. 2006, 78(19), 7061-7069) reported the employment of α-bromophenylacetic acid (BPAA) dissolved in water/methanol mixture (1:1, v/v) as a model analyte for chiral separations in capillary electrophoresis (CE). However, the stability of BPAA in aqueous methanol is questionable (Kováčová G. 2018. Diploma thesis. Charles University, Faculty of Pharmacy). The aim of this work was to determine the reaction order of the decomposition of BPAA (c = 0.47 mM) in 50% aqueous methanol. The reaction kinetic study was performed by capillary electrophoresis in 50 µm (i.d.) poly(vinyl alcohol)-coated capillary (30 cm/24.5 cm) with UV detection. The BPAA and products of nucleophilic substitution (mandelic acid, α-methoxyphenylacetic acid, and Br^-) were separated in 60 mM formate buffer (pH 3.0) at -30 kV; the detection λ was 200 nm. The first order reaction kinetics was confirmed by linear and non-linear regression, yielding the rate constant $1.52\times10^{-4} \pm 2.76\times10^{-5}$ s^{-1} and $7.89\times10^{-5} \pm 5.02\times10^{-6}$ s^{-1}, respectively. Additionally, the BPAA dissolved in 100% methanol separated in 60 mM formate buffer (pH 3.0) and 50 mM phoshphate buffer (pH 7.5) showed a slower course of degradation. The identity of the degradation products was confirmed by CE coupled to mass spectrometric (MS) detection. The CE-MS separations carried out in 60 mM formate buffer (pH 3.0) and in 60 mM acetate buffer (pH 5.0) confirmed the results obtained by CE with UV detection. Results of this work provide strong evidence of the instability of BPAA in 50% aqueous methanol indicating that it is not suitable as a model analyte for chiral separations.