Conference abstract PO-09

Enantioseparation with Capillary Electrophoresis (CE) as Evidence of Chirality Retention during Kolbe Electrolysis of Amino Acids

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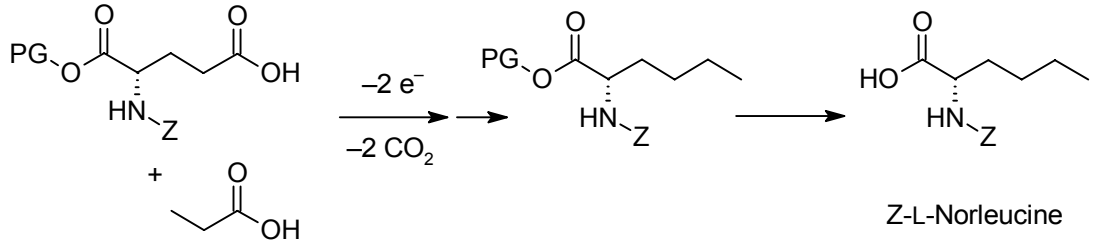
The radical Kolbe electrolysis can be applied to synthesize non-proteinogenic amino acids starting from L-glutamic acid (or L-aspartic acid, respectively). Using suitable protecting groups for both amino and carboxy functionalities, the retention of chirality at the α-carbon atom can be expected.

Z-L-Norleucine was prepared by cross reaction of *Z*-L-glutamic acid and propionic acid. Then, the carboxylic acid protecting group was removed to obtain a charge carrier function which is necessary for a separation via CE.



Electrochemical synthesis of Z-L-norleucine via Kolbe electrolysis

β-Hydroxypropyl-cyclodextrin was used as chiral additive in the mobile phase to obtain chiral selectivity [1]. The composition of the mobile phase and other operation parameters were varied to enhance peak resolution until a baseline separation of the two enantiomers was achieved. 50 mM Boraxbuffer (pH 10,5) with 10 mM β-hydroxypropyl-cyclodextrin and 5% methanol at 20 °C and 20 kV impressed voltage turned out to be optimal convenient.

Z-L-Norleucine, obtained by Kolbe electrolysis as described above, was compared to L-norleucine and D,L-norleucine which are commercially available. Racemization could not be observed. In a further experiment, the Kolbe product was treated with aq. NaOH for about 24 hours to force racemization. In that case, *Z*-D-norleucine could also be detected.

[1] Issaq HJ. A decade in capillary electrophoresis. Electrophoresis. 2000; 21: 1921–1939. doi:10.1002/1522-2683(20000601)21:10<1921::AID-ELPS1921>3.0.CO;2-Y

[2] Salami M, Jira T, Otto HH. Capillary electrophoretic separation of enantiomers of amino acids and amino acid derivates using crown ether and cyclodextrin. Pharmazie. 2005; 60: 181–185. PMid:15801669