
Article

RP-HPLC Analysis of Phenolic Lignin Monomers with DAD and Coulometric Array Detection

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Abstract

An high performance liquid chromatography (HPLC) method, applying diode array (DAD) and coulometric array detection simultaneously, was developed to determine monomeric phenolic compounds in lignin hydrolysates. To allow for additional mass spectrometric detection, a formic acid gradient was designed as an alternative to the non-volatile phosphate buffer gradient. Within a total run time of 28 min, 10 out of 14 analytes were baseline separated by the formic acid gradient, and 8 compounds were completely resolved by the phosphate gradient. The dependence of the coulometric detection on cell potential, potential step width, eluent pH and eluent composition was comprehensively tested. Detection limits ≤ 20 $\mu\text{g/L}$ were achieved by DAD detection for nine analytes and by coulometric array detection for all analytes, demonstrating the superior sensitivity of electrochemical detection. DAD offered better linearity and reproducibility. Inter-day precision of peak heights spanned from 0.26 to 2.00% (formic acid gradient) and from 0.75 to 2.85% (phosphate buffer gradient) operating the DAD detector, but it exceeded 10% for several compounds applying the coulometric array detector. The simultaneous operation of both detectors offered an enhanced certainty of substance identification. The appropriateness of the method was confirmed by the analysis of various hydrolysates of lignin-containing materials.
