

2. 固相抽出-エキシマー蛍光誘導体化 HPLC 法による食品中不揮発性アミン類の分析

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A method for the determination of nonvolatile amines (putrescine, cadaverine, histamine, tyramine and spermidine) in foods by solid-phase extraction and excimer-forming derivatization was investigated. Nonvolatile amines in a solid sample were extracted with 3% trichloroacetic acid, and the amines in a liquid sample were extracted with water. The extract was applied to polymer-based strong cation exchange mini-column, which was then rinsed with phosphate buffer of pH 6.8 and water. Nonvolatile amines were eluted with 100 mmol/L potassium carbonate solution. The solution was mixed with 6 mmol/L 1-pyrenebutyl chloride solution and derivatized. Derivatives of nonvolatile amines were analyzed by LC-FLD, and the identity of the amines was confirmed by LC-MS/MS without derivatization. The limit of detection ($S/N \geq 3$) of nonvolatile amines in all samples was $0.04 \mu\text{g/g}$, and the limit of quantitation ($S/N \geq 10$) was $0.1 \mu\text{g/g}$. Recoveries of nonvolatile amines from fish tissues, miso, shoyu and red wine were in the range of 80.4-111%.