

Release of aromatic amines from building sealants

Preparation of a process for analytical quantification
as a basis for the assessment under the
General Building Inspectorate Approval

Final report on DIBt research project

P 52-5-20.85.1

Summary

Two-component polyurethane systems (polyurethane = PUR) consisting of a polyol and a diisocyanate component are often used for waterproofing building structures. The two components are injected into a blending chamber using an applicator and mixed together. They do not react until on the site of operation. During the application of the waterproofing agent, organic amine compounds might be released into the ground water. The aim of the project (DIBt grant agreement number: P 52-5-20.85.1) was to prepare a process for the analytical quantification as a basis for the assessment of PUR injection resins as part of a general building inspectorate approval. In the first phase of the project, the release of 16 amines in 2 PUR injection resins was investigated and also the hydrolysis rate of 6 isocyanate derivatives. The present report contains the results of the second project phase. The focus in Phase II lay on the extension of the process to cover additional amines and to optimize the instrumental analytics and enrichment. The analytical process (ultra-high performance chromatography coupled with tandem mass spectrometry, UPLC-MS-MS) was extended to include 26 amines.

Two chromatographic methods, an isocratic and a gradient method were developed and compared with one another. Due to the strong matrix effects it is essential to pre-clean eluates from the inverse elution column (IEC). The solid phase extraction (SPE) was developed using ultra-pure water and IEC eluates. In the SPE with reversed phases (RP-SPE), water-soluble amines are not satisfactorily extracted from the water sample. In contrast, the solid phase extraction with a cation exchanger (XC-SPE) proved to be basically suitable for the accumulation of amines. At the same time, however, the XC-SPE resulted in an accumulation in the matrix containing the amine group. As some of the amines of interest could not be, or could only be unsatisfactorily recorded by the RP-SE there is no simple way of developing a robust, verifiable analysis process which will record simultaneously all 23 stable amines of interest. 4.4'-Methylenedianiline (4.4'-MDA) and 2.4'-MDA can be analyzed without enrichment because of the highly sensitive analysis process and high rate of selectivity of the mass transfers. An MDA-optimized chromatographic method can reduce the analysis period to 10 minutes so that classical standard addition trials focusing on MDA are economically more advantageous than purifying IEC eluates with the help of SPE with reverse phases.