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STUDY ON BISPHENOL A ANALYSIS FROM WATER SOURCES BY DERIVATIZATION WITH TRIFLUOROACETIC ANHYDRIDE AND DETERMINATION BY GC-MS

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Introduction

Bisphenol A, (BPA), is largely used in the production of epoxy resins and polycarbonate plastics as additive and antioxidant. Approximately one million tons of BPA were used only in EU annually. From municipal wastewater and industrial discharge, large quantity of BPA is introduced into the aquatic environment. For determination of bisphenol A in environmental water most frequently used methods are based on liquid chromatography and gas chromatography. As BPA is a phenolic compound, its volatility is relatively low, resulting in poor sensitivity and peak tailing using gas chromatography. To improve analytical performances derivatization is necessary. Stehmann and Schröder previously studied derivatization of BPA with trifluoroacetic anhydride (TFAA) and characterized the formed derivative by mass-spectra, infrared-spectra and the results of the NMR spectroscopy, but no analytical method for BPA using derivatization with TFAA was developed.

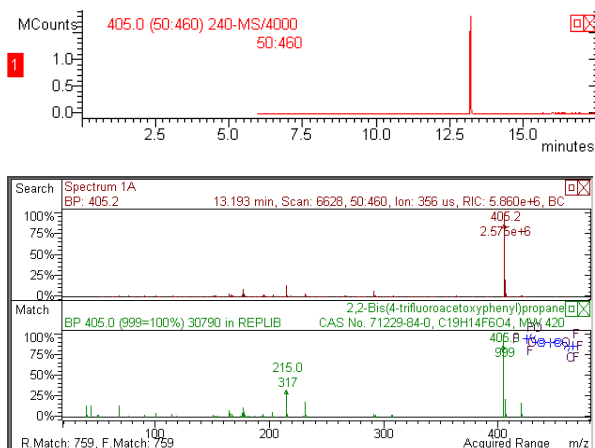
Materials and methods

GC-MS analysis was performed on an Agilent 7890A Gas Chromatograph equipped with an Agilent 240 MS ion trap mass spectrometer. Chromatographic separation was performed on a TG-5MS metallic column, length: 60m, diameter: 250 µm, film thickness: 0.25µm (Thermo Scientific), with helium 99.9999% purity as carrier. SPE extraction was performed on a CHROMABOND SPE extractor for up to 12 cartridges (Macherey-Nagel, Germany) attached to a vacuum pump. Strata C18-U with 1g C18 stationary phase (Phenomenex) were used for extraction. Bisphenol A (99% GC purity), Trifluoroacetic anhydride (≥99%), toluene (for GC-MS) and methanol (HPLC grade) were used.

Results and conclusions

Tests for identification of the derivatization product were performed using 1mL of BPA 1mg/L solution in toluene over which 50µL TFA was added. Mixture was heated at 50°C for 2h. Gas chromatographic conditions: injection of 1 µL sample, split injection 5:1, inlet temperature: 280°C, heating program: 50 ° C (1 min); 20 ° C / min to 280 ° C (5 min) (total: 17.5 min). The MS spectrum was recorded in the 50-460

amu range. Di(trifluoroacetyl)-Bisphenol A was identified using NIST 11 spectral database with a retention time 13.193 min.



Effect of derivatization time/temperature and injector temperature was studied, optimal results being obtained for 2 hour derivatization at 70°C and injection at 250°C. Using more than 20 µL TFAA for derivatization bring no improvement for sensitivity of method. Addition of pyridine as acid remover bring no major improvement.

Calibration for 10-1000 ug BPA/L was performed (Linear fit, Ignore origin, RSD: 2.135%, R²: 0.9990, SN for 1ug/L: 654, LOQ= 15.3ng/L).

Method was used for evaluation of BPA degradation under photocatalytic condition, using direct extraction of aqueous samples with toluen, at pH=2.

SPE extraction of BPA was evaluated using cartiges with 1g C18 stationary phase. 50 mL of various syntetic solutions with corrected pH (phosphate buffer, pH 7) were extracted with 5mL/min rate. As toluen give poor recovery rates (2.55-6.17%) for BPA, methanol recovery, nitrogen drying and reconstruction of extract with 1 mL toluen was used, with recovery rates up to 91%. Solid phase extraction of bisphenol A folowed by derivatization with trifluoroacetic anhydride and GC-MS analysis is an efficient solution for BPA analysis from various aqueous matrices.

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