

Химия және химиялық
технология факультеті



Факультет химии и
химической технологии

БАҒДАРЛАМА

Студенттер мен жас ғалымдардың

«ФАРАБИ ӘЛЕМІ»

атты халықаралық ғылыми конференциясы

Алматы, Қазақстан, 2016 жыл, 11-14 сәуір



ПРОГРАММА

Международная научная конференция студентов и молодых ученых

«ФАРАБИ ӘЛЕМІ»

Алматы, Казахстан, 11-14 апреля 2016 года



PROGRAM

International Scientific Conference of Students and Young Scientists

«FARABI ALEMI»

Almaty, Kazakhstan, April 11-14, 2016

**PROBLEMS OF SPME GC-MS QUANTIFICATION OF BENZENE,
TOLUENE, ETHYLBENZENE AND XYLENE IN SOILS**

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The main problems affecting measurement accuracy and precision here are losses of analytes during sample preparation and matrix effect. About 60% loss of VOCs could take place due to sample preparation including such steps as sampling, storage, weighing and preparing the calibration samples. Thus the method for BTEX determination in soil should provide matrix controlling calibration with minimal analyte-loss factors while meeting the requirements of green analytical chemistry. The problem of known SPME GS-MS methods of BTEX determination is that mostly they do not consider analyte loss in sample preparation steps including introduction of standard solutions into the sample, addition of organic solvents, water, etc. and multiple extraction, since BTEX are lost from the extraction system after septa was pierced due to volatilization and adsorption on septa's silicon.

The loss of BTEX from extraction system caused by volatilization out of pierced-seal vial was studied. Two types of sealing material were compared: conventional silicon and Viton septa. Solid phase microextraction from vials containing standard solutions was held each 3 and 10 h and obtained results were compared. Change in peaks areas in time for two types of septa was measured. Viton septa have higher density and provide better isolation of extraction system even after piercing the septa compared to silicon septa. Experiments on multiple BTEX extraction from same vials with different storage times and use of different sealing materials showed that about 5-10 % of analytes an hour are lost to side processes when using conventional silicon septum after their first piercing at room temperature. 10 and 40% loss of benzene in 10 h was measured from vials sealed with Viton and silicon septa, respectively. This side processes of analyte loss cause inaccurate measurement of concentration in sample from pierced-seal vials. The problem can be solved by optimizing sampling and sample preparation steps of soil analysis. For BTEX determination in soil sampling protocol when only needed soil amount to be introduced into vial without any further modification and separately a bigger sample amount to be taken for preparing calibration for this sample matrix was proposed.

Another limitation of SPME-GC-MS determination of BTEX caused by matrix effect is considerably long equilibration of standards and different agitation techniques and approaches required for different matrix types. Equilibration of standards in sample is the speed- and calibration accuracy- limiting step of soil analysis. During the method development, optimal conditions (temperature, addition of water, agitation) for faster standard equilibration were chosen.

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