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BOOK OF ABSTRACTS

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PY_20: DETERMINATION OF TRANSFORMATION PRODUCTS OF UNSYMMETRICAL DIMETHYLHYDRAZINE IN WATER USING VACUUM-ASSISTED HSSPME

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Vacuum-assisted headspace solid-phase microextraction (Vac-HSSPME) method proposed by Psillakis et al. [1] in 2012 was applied for determination of transformation products (TPs) of unsymmetrical dimethylhydrazine (UDMH) in water using GC-MS. The list of studied analytes included: (1) pyrazine; (2) 1-methyl-*1H*-pyrazole; (3) *N*-nitrosodimethylamine; (4) *N,N*-dimethylformamide; (5) 1-methyl-*1H*-1,2,4-triazole; (6) 1-formyl-2,2-dimethylhydrazine; (7) 1-methyl-*1H*-imidazole; (8) formamide and (9) 1H-pyrazole, which have low Henry's law constants ($3 \cdot 10^{-10}$ – $8 \cdot 10^{-5}$ atm·m^3/mol). Experiments were conducted using modified Mininert valves [2], which provided efficient seal of the vial and stable vacuum. Vac-HSSPME allowed up to 10 times greater responses of analytes compared to atmospheric pressure HSSPME and proportionally lower detection limits. Effects of extraction temperature (30, 40, 50 and 70°) and time (10, 20, 30 and 40 min) on analytes' responses and their relative standard deviations (RSDs) were investigated. Increase of extraction effectiveness with increase of both temperature and time of Vac-HSSPME was observed for most of analytes, except (6) and (8), probably because of their very high polarities and low stability of (6). For most analytes, equilibrium was reached after 30 min of extraction at 40 and 50°C, and after 20 min at 70°C, which is significantly faster than earlier observed values for atmospheric pressure HSSPME (no equilibrium reached after 60 min) [3]. Vac-HSSPME for 30 min at 50°C provided the best combination of responses and RSDs for most analytes. Calibration plots with wide linearity range of 0.1-100 µg/L with coefficients of determination (r^2) in the range of 0.990-0.999 were obtained for analytes (1)-(3), and 0.3-500 µg/L with r^2 in the range of 0.985-0.996 for analytes (4)-(5), (7) and (9). The developed method is recommended for determination of traces of TPs of UDMH in water samples.

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References:

[1] E. Psillakis, E. Yiantzi, L. Sanchez-Prado, N. Kalogerakis, Anal. Chim. Acta. 742 (2012).

[2] M.J. Trujillo-Rodriguez, V. Pino, E. Psillakis, J. Anderson, et al., Anal. Chim. Acta 962 (2017).

[3] N.V. Bakaikina, B. Kenessov, N.V. Ul'yanovskii, D.S. Kosyakov, et al., Chromatographia (2017), In Press