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**Automated procedure for determination of ammonia in concrete with headspace single drop micro-extraction by stepwise injection spectrophotometric analysis**

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Abstract

A novel automatic stepwise injection headspace single-drop micro-extraction system is proposed as a versatile approach for automated determination of volatile compounds. The system application is demonstrated for ammonia determination in concrete samples. An ammonia gas was produced from ammonium ions and extracted on-line into 5L 0.1 M H3PO4 to eliminate the interference effect of concrete species on the ammonia stepwise injection spectrophotometric determination. The linear range was 0.1 to 1 mg kg-1 with LOD 30 μg kg-1. The sample throughput was 4 h-1. This system has been successfully applied for the determination of ammonia in concretes.

*Keywords:* Headspace single-drop micro-extraction; Stepwise injection analysis; Ammonia; Concrete.

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References

[1] R.P. Pohanish, Elsevier, Sittig's Handbook of Toxic and Hazardous Chemicals and Carcinogens, USA, 2012.

[2] Z. Bai, Y. Dong, Z. Wang, T. Zhu, Emission of ammonia from indoor concrete wall and assessment of human exposure, Environ. Int. 32 (2006) 303-311.

[3] N.T. Crosby, Determination of ammonia by the Nessler method in waters containing hydrazine, Analyst 93 (1968) 406-408.

[4] W.T. Bolleter, C.J. Bushman, P.W. Tidwell, Spectrophotometric determination of ammonia as indophenol, Anal. Chem. 33 (1961) 592–594.

[5] J.K. Fawcett, J.E. Scott, A rapid and precise method for the determination of urea, J. Clin. Path. 13 (1960) 156-159.

[6] A.J. Kempers, C.J. Kok, Re-examination of the determination of ammonium as the indophenol blue complex using salicylate, Anal. Chim. Acta 221 (1989) 147-155.

[7] A. Aminot, D.S. Kirkwood, R. Kerouel, Determination of ammonia in seawater by the indophenol-blue method, Marine Chemistry 56 (1997) 59–75.

[8] N.M. Tzollas, G.A. Zachariadis, A.N. Anthemidis, J.A. Stratis, A new approach to indophenol blue method for determination of ammonium in geothermal waters with high mineral content, Intern. J. Environ. Anal. Chem. 90 (2010) 115–126.

[9] J.P. Hamalainen, J.L. Tummavuori, M.J. Aho, Determination of NH3 in pyrolysis gases by ammonia selective electrode, Talanta 40 (1993) 1575-1581.

[10] F. Valentini, V. Biagiotti, C. Lete, G. Palleschi, J. Wang, The electrochemical detection of ammonia in drinking water based on multi-walled carbon nano-tube/copper nanoparticle composite paste electrodes, Sens. Actuators B 128 (2007) 326–333.

[11] D.M. Coulson, Electrolytic conductivity detector for gas chromatography, J. Chromatogr. Sci. 4 (1966) 285-287.

[12] N. Kashihira, K. Makino, K. Kirita, Y. Watanabe, Chemiluminescent nitrogen detector gas chromatography and its application to measurement of atmospheric ammonia and amines, J. Chrom. A. 239 (1982) 617-624.

[13] B. Pranaityte, S. Jermak, E. Naujalis, A. Padarauskas, Capillary electrophoretic determination of ammonia using headspace single-drop microextraction, Microchem. J. 86 (2007) 48-52.

[14] W. Beck, H. Engelhardt, Capillary electrophoresis of organic and inorganic cations with indirect UV detection, Chromatogr. 33 (1992) 313-316.

[15] A. Padarauskas, V. Paliulionyte, B. Pranaityte, Single-run capillary electrophoretic determination of inorganic nitrogen species in rainwater, Anal. Chem. 73 (2001) 267-271.

[16] L. Wang, T.J. Cardwell, R.W. Cattrall, M.D. Luque de Castro, S.D. Kolev, Determination of ammonia in beers by pervaporation flow injection analysis and spectrophotometric detection, Talanta 60 (2003) 1269-1275.

[17] C. Pasquini, W.A. de Oliveira, Monosegmented system for continuous flow analysis. Spectrophotometric determination of chromium (VI), ammonia, and phosphorus, Anal. Chem. 57

(1985) 2575-2579.

[18] M. E. Meyerhoff, Y.M. Fraticelli, Flow injection determination of ammonia-N using polymer membrane electrode-based gas sensing system, Anal. Lett. 14 (1981) 415-432.

[19] V. Bulatov, P.A. Ivasenko, A.L. Moskvin, L.N. Moskvin, Stepwise injection potentiometric determination of ammonium-ions in water, J. Flow Inj. Anal. 26 (2009) 49-52.

[20] R. Kerouel, A. Aminot, Fluorometric determination of ammonia in sea and estuarine waters by direct segmented flow analysis, Mar. Chem. 57 (1997) 265-275.

[21] A. Aminot, R. Kerouel, D. Birot, A flow injection-fluorometric method for the determination of ammonium in fresh and saline waters with a view to in situ analyses, Wat. Res. 35 (2001) 1777-1785.

[22] J.F. van Staden, R.E. Taljaard, Determination of ammonia in water and industrial effluent streams with the indophenol blue method using sequential injection analysis, Anal. Chim. Acta 344 (1997) 281-289.

[23] W.E. Van der Linden, Membrane separation in flow injection analysis, Membrane separation in flow injection analysis, Anal. Chim. Acta 151 (1983) 359-369.

[24] C. Pasquini, L.C. de Faria, Flow-injection determination of ammonia in Kjeldahl digest by gas diffusion and conductometry, Anal. Chim. Acta 193 (1987) 19-27.

[25] J. Gonzalez-Rodrguez, P. Perez-Juan, M.D. Luque de Castro, Method for monitoring urea and ammonia in wine and must by flow injection–pervaporation,Anal. Chim. Acta. 471 (2002) 105-111.

[26] L. Wang, T.J. Cardwell, R.W. Cattrall, M.D. Luque de Castro, S.D. Kolev, Pervaporation flow injection determination of ammonia in the presence of surfactants, Anal. Chim. Acta 416 (2000) 177-184.

[27] J.-H. Wang, E.H. Hansen, Development of an automated sequential injection on-line solvent extraction-back extraction procedure as demonstrated for the determination of cadmium with detection by electrothermal atomic absorption spectrometry, Anal. Chim. Acta 456 (2002) 283- 292.

[28] A.N. Anthemidis, Automatic sequential injection liquid–liquid micro-extraction system for on-line flame atomic absorption spectrometric determination of trace metal in water samples, Talanta 77 (2008) 541-545.

[29] A.N. Anthemidis, I.S.I. Adam, Development of on-line single-drop micro-extraction sequential injection system for electrothermal atomic absorption spectrometric determination of trace metals, Anal. Chim. Acta 632 (2009) 216-220.

[30] S. Liu, P. K. Dasgupta, Liquid droplet. A renewable gas sampling interface, Anal. Chem. 67 (1995) 2042-2049.

[31] A.A. Cardoso, P. K. Dasgupta, Analytical chemistry in a liquid film/droplet, Anal. Chem. 67 (1995) 2562-2566.

[32] H. Liu, P.K. Dasgupta, A renewable liquid droplet as a sampler and a windowless optical cell. An automated sensor for gaseous chlorine, Anal. Chem. 67 (1995) 4221-4228.

[33] M. Sarangapani, C.-T. Yan, H.-K. Shih, Determination of ammonium in aqueous samples using new headspace dynamic in-syringe liquid-phase microextraction with in situ derivitazation coupled with liquid chromatography–fluorescence detection, Anal. Chim. Acta. 754 (2012) 54- 60.

[34] A.V. Bulatov, A.L. Moskvin, L.N. Moskvin, A.V. Mozhuhin, The stepwise injection analysis as a new opportunity for automation of chemical analysis of liquid, gaseous and solidphase samples, J. Flow Inj. Anal. 27 (2010) 13.

[35] A.V. Bulatov, I.I. Timofeeva, A.L. Moskvin, Stepwise injection spectrophotometric determination of carbamides in construction materials, J. Flow Inj. Anal. 30 (2013) 51-54.