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Energy-dispersive X-ray spectrometry combined with directly suspended droplet microextraction for determination of dissolved silicate in surface water *via* silicomolybdenum blue complex

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Abstract

Energy-dispersive X-ray fluorescence spectrometry (EDXRF) is a well-established analytical technique successfully applied with good precision and accuracy to determination of many elements. However, in the case of elements of low atomic number, such as silicon, direct determination is hampered due to low fluorescence yield and relatively low energy easily absorbed by sample matrix. An indirect method for determining surface water silicate is thus proposed. The method is based on silicate determination *via* molybdenum present in silicomolybdenum blue complex. Determination follows directly suspended droplet microextraction. Optimum conditions for both microextraction and EDXRF measurement were studied. A good ratio of silicon to molybdenum (1 to 41) and a sensitive K_{α} line of molybdenum make it possible to determine low concentrations of silicate. Under optimized conditions, good linearity, up to $3 \mu\text{g mL}^{-1}$ ($r=0.9990$), and good detection limit (6 ng mL^{-1}) were achieved. The total RSD for the EDXRF determination of silicate following DSDME was 6.7%. Taking into account all steps preceding the determination and the uncertainty of XRF measurements, the proposed method can be recognized as precise. The enrichment factor was 140. The developed method was used to determine dissolved silicate content in surface water samples. The accuracy and repeatability of the proposed procedure were checked by standard addition method and compared to the results obtained using ICP-OES technique. The recovery (92.2–96%) was satisfactory and indicates usefulness of the developed procedure.

Keywords: silicate, silicomolybdenum blue complex, energy dispersive X-ray fluorescence spectrometry, water samples

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