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Elucidation of the stoichiometry of Fe(III) and Fe(II) complexes formed with 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (5-Br-PADAP). Application to Fe(III) and Fe(II) Electrochemical Determination at Trace Level in Natural Water

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This work provides some new information on the complexes formed between Fe(III) and Fe(II) with 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (5-Br-PADAP). The electrochemical behavior of both complexes was studied and their stoichiometry, which was uncertain till now, was definitely clarified as a 1:1 metal:ligand ratio on the basis of both electrochemical and spectrophotometric data. The stability constants β for both complexes were also determined and found to be in the range 10^5 and 10^4 for Fe(III)/ and Fe(II)/5-Br-PADAP complex, respectively. Using 5-Br-PADAP as a complexing agent, the determination of Fe at trace level in water was achieved by adsorptive differential pulse voltammetry in the range 1 – 10 μM . Under optimized conditions, a sensitivity of 0.0376 and 0.0063 $\mu\text{A } \mu\text{M}^{-1}$ was found for Fe(III) and Fe(II), respectively. Tests on natural water from Canal du Midi were performed, and allowed values of 2.3 μM for Fe(III) and 1.7 μM for Fe(II) to be obtained. The corresponding value for total Fe was slightly lower than that found with ICP-OES measurements (ca. 4.0 vs. 4.5 μM , respectively), suggesting that 5-Br-PADAP is not a complexing agent strong enough to catch Fe cations in the complexes formed with organic matter nor to complex quantitatively free Fe(III) or Fe(II) species.

Keywords: Complex stoichiometry; 5-Br-PADAP complexation; iron speciation; electrochemical sensor; natural water samples

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