

Abstract

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Title of Diploma Thesis: Determination of ferric ions in the SIA system with chemiluminescence detection

Catalytic influence of ferric ions on the oxidation of luminol using hydrogen peroxide in alkaline environment has been observed through chemiluminescence detection. The sequence of sample and reagent aspiration, their volumes, mixing flow rate and flow rate through the detector have been optimized. The influence of mixing, various ferric compounds, and the presence of ferrous ions on the intensity of chemiluminescence have also been tested. The resulting optimization was an aspiration sequence of 50 μl of $10^{-3} \text{ mol}\cdot\text{l}^{-1}$ H_2O_2 , 50 μl of $10^{-3} \text{ mol}\cdot\text{l}^{-1}$ $\text{K}_3[\text{Fe}(\text{CN})_6]$, 50 μl of $10^{-3} \text{ mol}\cdot\text{l}^{-1}$ luminol. Solution aspiration speed was $100 \mu\text{l}\cdot\text{s}^{-1}$, flow rate through detector was $70 \mu\text{l}\cdot\text{s}^{-1}$. Calibration dependence of chemiluminescence intensity on ferric ion concentration at low concentrations ($10^{-7} - 10^{-9} \text{ mol}\cdot\text{l}^{-1}$) was non-linear. Linear area of calibration for Fe^{3+} ions at standard conditions has not been found with correlation coefficient high enough, therefore polynomial calibration dependence of second degree with correlation coefficient of $R^2 = 0.9785$ has been used. Detection limit of $7.48\cdot 10^{-10} \text{ mol}\cdot\text{l}^{-1}$ Fe^{3+} ions has been calculated and the value of quantification limit is $1.26\cdot 10^{-9} \text{ mol}\cdot\text{l}^{-1}$ Fe^{3+} ions. For the concentration of $2\cdot 10^{-9} \text{ mol}\cdot\text{l}^{-1}$ Fe^{3+} ions at standard conditions, repeatability is evaluated as relative standard deviation of 3.79 %. For the concentration of $4\cdot 10^{-9} \text{ mol}\cdot\text{l}^{-1}$ Fe^{3+} ions at standard conditions, relative standard deviation is 6.28 %.