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Reversed micellar mediated chemiluminescence determination of antimony(III,V) using rhodamine B following on-line extraction

○ <u>Tomoyuki Yamamoto</u>¹, Shoji Ishizaka¹, and Terufumi Fujiwara¹ Grad. Sc. Sci., Hiroshima Univ.

The differential determination of antimony(III,V) in environmental analysis is important. There are several methods for the Sb determination, but each has some drawbacks such as sensitivity, selectivity or simplicity. In order to overcome their defects, we have developed a flow method for determination of Sb(V) based on a reversed micellar mediated chemiluminescence (RMM-CL) detection following an off-line solvent extraction using rhodamine B (RB) ¹⁾. In this study, the flow method was applied to an on-line extraction-CL procedure for a speciation analysis of Sb(III,V), where a reaction process using the Ce(IV) oxidant was added for oxidizing Sb(III) to Sb(V).

The system of the combination of extraction procedures with the RMM-CL detection using RB for the indirect Sb determination is shown in Fig.1. In the solvent extraction process, Sb(V) chloro-complex anion, [SbCl₆], was extracted from a HCl solution into toluene via ion-pair formation with the protonated RBH⁺ ion. Upon mixing the extract with a reversed micellar reagent solution containing cerium(IV), uptake of the ion-pair by reversed micelles occurred easily, followed by an oxidation reaction of RB with Ce(IV) in the CL process. The proposed method was successfully applied to a mixture of Sb(III) and Sb(V), where total antimony, Sb(III)+Sb(V), was measured using Ce(IV) as an oxidant to oxidize Sb(III) to Sb(V) prior to extraction, Sb(V) was determined directly without the use of an oxidant, and Sb(III) was calculated by difference (Fig.2).

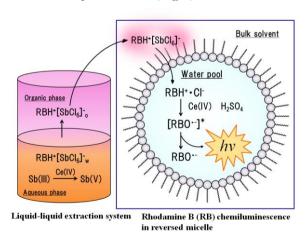


Fig.1 A system based on a RMM-CL detection following a solvent extraction for determination of Sb ¹⁾.

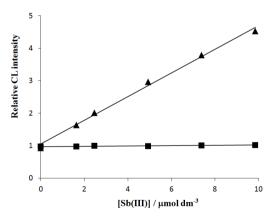


Fig. 2 Variation of the CL intensity with the concentration of Sb(III) in mixed sample solutions of Sb(III) and Sb(V) in the absence (\blacksquare) and presence(\blacktriangle) of Ce(IV). [Sb(V)] = 2.5 μ mol dm⁻³

1) T. Yamamoto, Y. Tsunemine, F. Hayakawa, T. H. A. Hasanin, Y. Okamoto, S. Ishizaka, and T. Fujiwara, *Anal. Sci.*, **2013**, 29,73.