

Determination of Bromides, Chlorides and Fluorides in Case of Their Joint Presence in Ion-Conducting Electrolyte

Authors : V. Golubeva, O. Vakhnina, I. Konopkina, N. Gerasimova, N. Taturina, K. Zhogova

Abstract : To improve chemical current sources, the ion-conducting electrolytes based on Li halides (LiCl-KCl, LiCl-LiBr-KBr, LiCl-LiBr-LiF) are developed. It is necessary to have chemical analytical methods for determination of halides to control the electrolytes technology. The methods of classical analytical chemistry are of interest, as they are characterized by high accuracy. Using these methods is a difficult task because halides have similar chemical properties. The objective of this work is to develop a titrimetric method for determining the content of bromides, chlorides, and fluorides in their joint presence in an ion-conducting electrolyte. In accordance with the developed method of analysis to determine fluorides, electrolyte sample is dissolved in diluted HCl acid; fluorides are titrated by $\text{La}(\text{NO}_3)_3$ solution with potentiometric indication of equivalence point, fluoride ion-selective electrode is used as sensor. Chlorides and bromides do not form a hardly soluble compound with La and do not interfere in result of analysis. To determine the bromides, the sample is dissolved in a diluted H_2SO_4 acid. The bromides are oxidized with a solution of KIO_3 to Br_2 , which is removed from the reaction zone by boiling. Excess of KIO_3 is titrated by iodometric method. The content of bromides is calculated from the amount of KIO_3 spent on Br_2 oxidation. Chlorides and fluorides are not oxidized by KIO_3 and do not interfere in result of analysis. To determine the chlorides, the sample is dissolved in diluted HNO_3 acid and the total content of chlorides and bromides is determined by method of visual mercurimetric titration with diphenylcarbazone indicator. Fluorides do not form a hardly soluble compound with mercury and do not interfere with determination. The content of chlorides is calculated taking into account the content of bromides in the sample of electrolyte. The validation of the developed analytical method was evaluated by analyzing internal reference material with known chlorides, bromides and fluorides content. The analytical method allows to determine chlorides, bromides and fluorides in case of their joint presence in ion-conducting electrolyte within the range and with relative total error (δ): for bromides from 60.0 to 65.0 %, $\delta = \pm 2.1$ %; for chlorides from 8.0 to 15.0 %, $\delta = \pm 3.6$ %; for fluorides from 5.0 to 8.0%, ± 1.5 %. The analytical method allows to analyze electrolytes and mixtures that contain chlorides, bromides, fluorides of alkali metals and their mixtures (K, Na, Li).

Keywords : bromides, chlorides, fluorides, ion-conducting electrolyte

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