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FULL PAPER

Determination of sulfur in soda-lime silicate glass by inductively coupled plasma atomic emission spectroscopy following separation using an alumina column

Yoshitaka SAIJO[1,3,†], Yuichi SUZUKI[2], Ryoji AKIYAMA[1] and Kiyotaka MIURA[3]

[1]Innovative Technology Laboratories, AGC Inc., 1150 Hazawa-cho, Kanagawa-ku, Yokohama 221–8755, Japan
[2]Electronics Company, AGC Inc., 1–5–1 Marunouchi, Chiyoda-ku, Tokyo 100–8405, Japan
[3]Department of Material Chemistry, Graduate School of Engineering, Kyoto University, Kyoto 615–8510, Japan

We developed a simple, rapid, and accurate method to determine the quantity of sulfur in soda-lime silicate glass using inductively coupled plasma atomic emission spectroscopy (ICP-AES) and sulfur separation using an alumina column. This method requires a smaller number of samples and takes less time for the analysis than conventional methods. The samples of glass were decomposed using hydrofluoric acid, perchloric acid, and an oxidizing agent. For the amber glass samples, the oxidizing agent used was potassium permanganate. The decomposed solution was diluted with perchloric acid. The solution was passed through an alumina column to enable sulfur adsorption on the column. To desorb sulfur from the column, diluted ammonia was passed through it after rinsing it with diluted perchloric acid. ICP-AES was used to determine the quantity of sulfur in the ammonia eluent. The method was validated using three certified glass samples. The sulfur quantities determined by this method were within the certified values. The relative standard deviation of the determined values was less than 2 %.

Key-words : Glass, Sulfur, Separation, Determination, Amber

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1. Introduction

Soda-lime silicate glass is the most widely used glass in the world and is the leader in the world's glass market. The industrial production of glass involves batch preparation, melting, fining, refining, forming, annealing, and post processes.[1)] To obtain a high-quality glass product, the number of bubbles remaining in the final product should be reduced. Fining and refining are, therefore, important in the industrial production of glass. The fining agents reduce the bubbles in the glass. One of the commonly used fining agents in the production of soda-lime silicate glass is sodium sulfate (Na_2SO_4). The melting,[2)–5)] and fining and refining[6)–18)] reactions of soda-lime silicate glass were investigated. In oxidized melts, the main sulfate fining mechanism is thermal sulfate decomposition. It typically occurs between 1430 and 1480 °C[8),18)] as follows:

$$Na_2SO_4 \rightarrow Na_2O + SO_2 + 1/2O_2$$

In reduced melts, which contain cokes, CO, and organic contaminants, some sulfates react to produce sulfides as follows:

$$2C + Na_2SO_4 \rightarrow 2CO_2 + Na_2S$$
$$4CO + Na_2SO_4 \rightarrow 4CO_2 + Na_2S$$
$$4C + Na_2SO_4 \rightarrow 4CO + Na_2S$$

The sulfide formation typically occurs between 700 and 800 °C,[8),18)] and the formed sulfides react with the remaining sulfates. Fining typically occurs between 1000 and 1350 °C as follows:

$$S^{2-} + 3SO_4^{2-} \rightarrow 4SO_2 + 4O^{2-}$$

During annealing, which follows fining, fining gases, such as O_2 and SO_2, are chemically absorbed into the melt, and the process is called refining. Fining and refining reduce the number of bubbles in the final product.

Determining the quantity of sulfur in the final product is important to understand the fining and refining reactions. Sulfur is present in glass as sulfate and sulfide.[19),20)] The total quantity of sulfur, consisting of sulfate and sulfide, also has to be determined, because sulfides can remain in a final product, such as amber glass.[21)–24)]

Several methods are already available to determine the quantity of sulfur in glass. These methods can be categorized into two types. One type decomposes the glass samples to determine the sulfur quantity in the decomposed solutions. An example of this type is the gravimetric

† Corresponding author: Y. Saijo; E-mail: yoshitaka.saijo@agc.com