Determination of Chlorine in Cardboard Gasket after Pyrohydrolysis Sample Preparation

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Abstract

A pyrohydrolysis system was developed for cardboard gasket sample preparation with the aim of determining chlorine.

Introdução

Cardboard gaskets are used as seals, especially in combustion machines, nuclear reactors, and chemical plants. They usually consist of compressed graphitized asbestos, binders, such as elastomers, and several other materials. When cardboard gaskets are used in combustion machines and nuclear reactors, the refrigerant liquid that is in contact with the cardboard can leach halogens present as contaminants. These elements, especially chlorine, are corrosive. Therefore, in order to prevent corrosion of the metallic parts of the equipments, halogens must be accurately controlled in the cardboard gaskets. One of the major difficulties facing the determination chlorine in solid samples is the sample preparation step. Analyte loss and contamination of the sample are the main drawbacks. Besides, the methods are time consuming. Pyrohydrolysis have been used successfully for preparation of different samples where halogens were accurately determined. Pyrohydrolysis is a relatively simple sample preparation method and, until now, it has not been applied to cardboard gaskets. In view of the difficulties associated with cardboard gasket sample preparation when chlorine determination is required, we propose the use of pyrohydrolysis.

Resultados e Discussão

To implement the pyrohydrolysis, a new system was constructed where modifications in the reactor design and water steam condenser were done. A device for introducing the sample into the reactor was also constructed in order to provide better performance of the whole system. Conditions that affect the pyrohydrolysis method were studied and the respective precision and accuracy were validated. Chloride was determined by ion chromatography with a chromatograph equipped with a conductivity detector and a chemical and CO_2 suppression unit. The calibration curve of Cl^- was prepared in 0.1 mol L^{-1} sodium carbonate/sodium bicarbonate (pH = 7.00) buffer solution which was also used as mobile phase for Cl^- determination. Good precision and accuracy were observed when the reactor temperature, reaction time, sample amount, water and gas flow rates were set to 1000 ^oC, 7.5 min, 300 mg, 1.0 mL min^{-1}, and 0.2 L min^{-1}, respectively. It was observed that an accelerator and absorbing solution were not necessary if the temperature of the condensing system was near 0 ^oC. The limit of detection (LOD) achieved was 1.5 µg g^{-1} Cl, for 300 mg of sample and a final volume of 15 mL. The RSD was lower than 10% for five replicates of the sample. Certified reference material (CRM - low density polyethylene, ERM 680K, European Reference Materials), results from samples decomposed by microwave-induced combustion and analyte recovery were used to check the accuracy. The results are in good agreement. Two commercial samples of cardboard gasket were analyzed. These samples were washed with acetone and high purity water, and cut into small pieces or ground with a cryogenic mill. The results were 293 ± 5 and 281 ± 31 µg g^{-1} Cl^-. These values are bellow the maximum concentration allowed (450 µg g^{-1}) for halogens in cardboard gasket used in machines and nuclear reactors.

Conclusões

The developed pyrohydrolysis system is suitable for the preparation of cardboard gasket samples when Cl determination is required. The method can be implemented relatively easily and chemical reagents are not necessary. The LOD is suitable and the results obtained using pyrohydrolysis agreed well with those of CRM and those obtained by an independent method, thereby ensuring the accuracy of the results.

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