



Sulfur determination in coal using molecular absorption in graphite filter vaporizer

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ABSTRACT

The vaporization of sulfur containing samples in graphite vaporizers for atomic absorption spectrometry is accompanied by modification of sulfur by carbon and, respectively, appearance at high temperature of structured molecular absorption in 200–210 nm wavelength range. It has been proposed to employ the spectrum for direct determination of sulfur in coal; soundness of the suggestion is evaluated by analysis of coal slurry using low resolution CCD spectrometer with continuum light source coupled to platform or filter furnace vaporizers. For coal in platform furnace losses of the analyte at low temperature and strong spectral background from the coal matrix hinder the determination. Both negative effects are significantly reduced in filter furnace, in which sample vapor efficiently interacts with carbon when transferred through the heated graphite filter. The method is verified by analysis of coals with sulfur content within 0.13–1.5% (m/m) range. The use of coal certified reference material for sulfur analyte addition to coal slurry permitted determination with random error 5–12%. Absolute and relative detection limits for sulfur in coal are 0.16 μg and 0.02 mass%, respectively.

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1. Introduction

Sulfur is one of the relevant elements in coals. Due to the different compositions of coals, sulfur content can range from 0.1 up to 6% m/m. Furthermore, sulfur compounds in coals can be listed in three distinct groups:

- Organic sulfur, i.e. sulfur bound in carbon structures. These species show a limited thermal stability.
- Inorganic sulfur, i.e. sulfur bound in inorganic compounds (sulfides, sulfates, etc.). These species show a thermal stability related to the specific compound.
- Pyritic sulfur, i.e. sulfur as FeS_2 . This species show relative chemical stability but limited thermal stability.

Sulfur content is usually determined to evaluate the emissions of SO_2 in the atmosphere after coal combustion. Sulfur is also determined in coal ash as a by-product in coal combustion.

Various test methods have been developed by standardization bodies. In particular, ASTM International (American Standards for Testing and Materials) developed the test method D 4239, first published in 1983 and reapproved in 2002 [1]. In this method, the coal sample is burned in a high temperature furnace (minimum operat-

ing temperature 1350 °C). Sulfur oxides formed during combustion are collected and then determined via acid–base titration or via measurement of infrared absorption. The latter determination is commonly employed in automated instruments. The reproducibility of infrared method is about 0.13% (m/m) at 1.5% (m/m) sulfur content. Both methods are capable to overcome the problems associated with the different sulfur species in coal, as all sulfur species are oxidized and detected. However, the precision of both test methods becomes less satisfactory when the sulfur content in the samples is lower than 0.5–1.0%.

An alternative test method should provide better precision but at the same time should overcome the problems related to the different thermal stabilities of sulfur species. Furthermore, a new method should guarantee correct sample introduction for a variety of coals with different compositions of organic and inorganic constituents, short analysis time and simple calibration. Hence, it is reasonable to evaluate other spectroscopic techniques for sulfur determination in coal. The simplest approach could be coal slurry analysis using the technique of electrothermal atomic absorption spectrometry (ET AAS). Several problems can be envisaged on this approach.

One problem is related to the detection of sulfur. Direct determination of atomic sulfur using atomic lines in far-ultraviolet region (180.7, 182.0 and 182.6 nm) would require special vacuum or inert gas purged instrumentation [2]. Alternatively, CS bands could be used for detection of sulfur. This idea was applied, first, by Tittarelli and Lavorato in the determination of sulfur in oil [3]:

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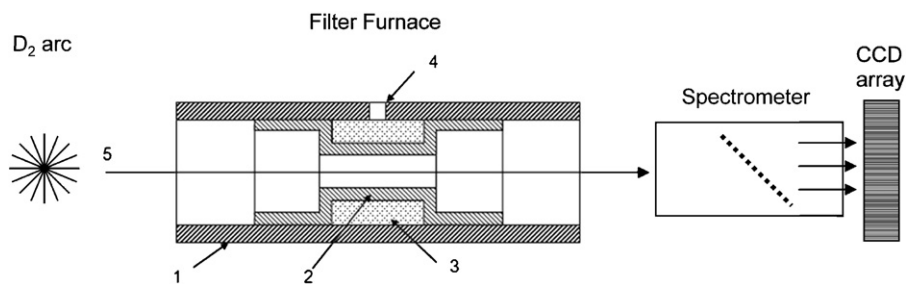


Fig. 1. Experimental setup. 1: pyrocoated graphite tube, 2: graphite filter, 3: ring cavity (empty for the slurries or filled with carbon fiber for liquid samples), 4: sampling port, and 5: light beam.

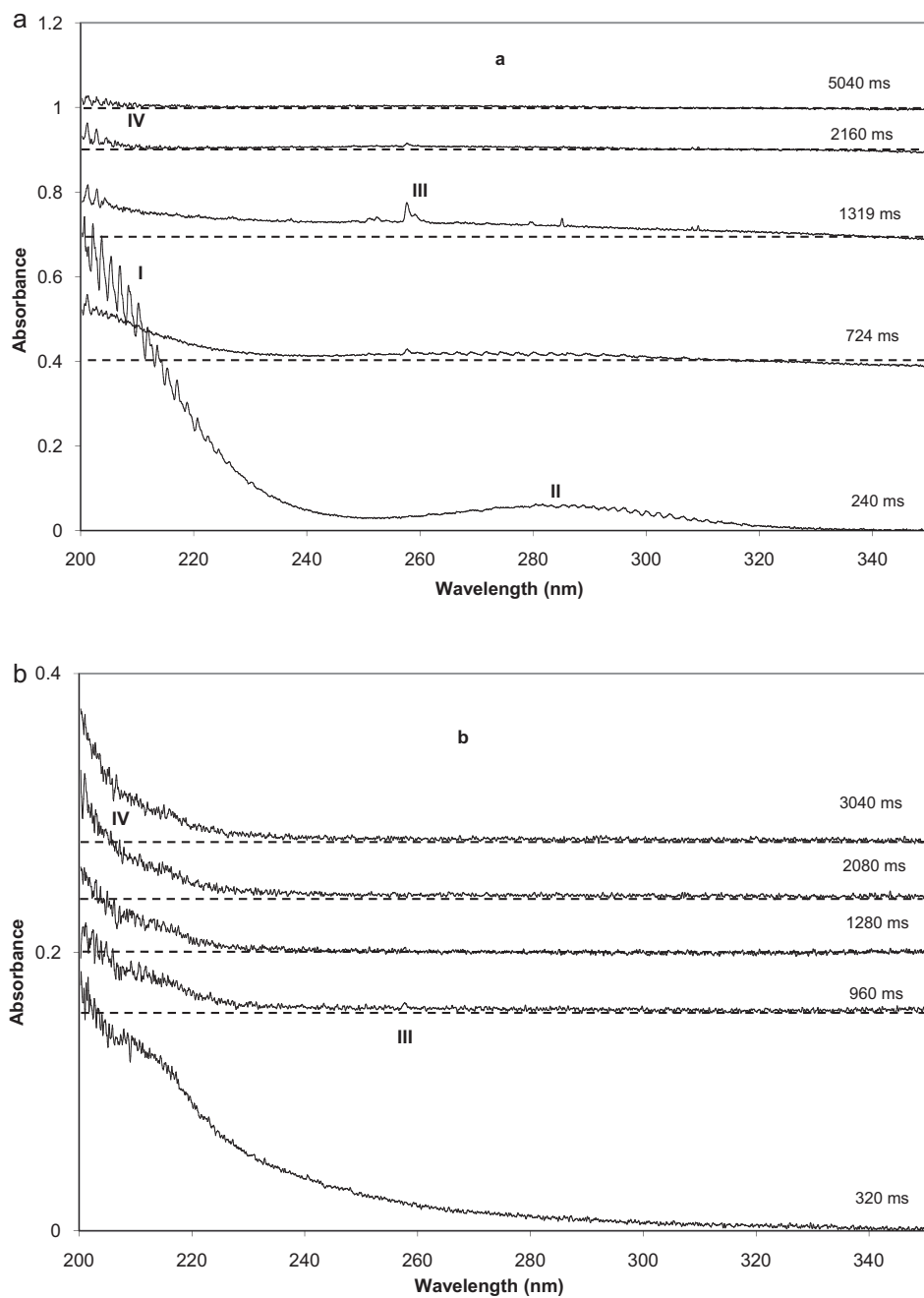


Fig. 2. Absorption spectra acquired during the vaporization of 20 μg sulfur injected in platform furnace as solutions in water (a) and hexane (b). Dashed lines indicate zero absorbance for respective spectrum.

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