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Investigation of high-resolution absorption spectra of diatomic sulfides of group 14 elements in graphite furnace and the comparison of their performance for sulfur determination



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ABSTRACT

For the purpose of finding suitable molecules applicable to sulfur determination and to compare their analytical sensitivity systematically, high-resolution overview molecular absorption spectra of sulfides of group 14 elements produced in a graphite furnace were investigated. To that end a modular simultaneous echelle spectrograph (MOSES) was used, which allows recording sub-ranges of spectra out of a total wavelength range from 190 nm to 735 nm. The combined overview spectra show a complex structure with many vibrational bands, each of them consisting of a multitude of sharp rotational lines. The absorption of rotational lines of SiS (282.910 nm), GeS (295.209 nm), SnS (271.578 nm), and PbS (335.085 nm) has been analyzed for optimizing the particular experimental conditions regarding to the sulfur determination. Using the commercial CS AAS instrument contrAA 600 under optimized conditions such as the temperature program, the modification of the platform with Zr and the use of chemical modifiers, the achieved characteristic masses for sulfur are 12 ng (CS), 15.7 ng (SiS), 9.4 ng (GeS), 20 ng (SnS), and 220 ng (PbS). The first four sulfides provide an analytical sensitivity at roughly the same level, but the GeS molecule seems to be the best one with respect to analytical sensitivity, and flexibility in molecular formation control. The PbS molecule provides the lowest analytical sensitivity, and together with its low bond strength it is not recommended for sulfur determination.

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1. Introduction

Carbon, silicon, germanium, tin, and lead belong to the group 14 elements in the periodic system with four valence electrons. Diatomic sulfides with these elements, including six electrons from the sulfur atoms, then possess a total of ten valence electrons. According to the quantum chemical principle, in such systems both π and σ molecular orbitals are fully occupied, and thus they show the highest stability of all diatomic molecules [1]. The theory can be confirmed by the high bond strengths of diatomic sulfides in this group, namely 713 (CS), 623 (SiS), 551 (GeS), 464 (SnS), and 346 (PbS) kJ mol⁻¹ [2], and consequently they are much more stable than sulfides of elements in the neighbor groups of the periodic system. The group 14 diatomic sulfides are subject of various interesting research. For example, the stable CS and SiS molecules are present inside the dark clouds in the galaxy [3,4], and investigation of their spectra provides information about the interstellar medium. The

* Corresponding author. *E-mail address:* huang@isas.de (M.D. Huang). GeS and SnS molecules are promising photovoltaic materials for thin film solar cells [5,6].

Impelled by the restricted accessibility of atomic absorption, the determination of sulfur via its molecular absorption is a recent and attractive application of diatomic sulfides in analytical chemistry [7]. High stability sulfides involving group 14 elements are favored here; they can easily be formed in gaseous phase, which generally results in high sensitivity. Besides, fewer matrix interferences would be encountered, because in this case side reactions, which destroy stable target molecules, can hardly happen.

Among the sulfides including group 14 elements, CS exhibits the highest bond strength, but it is not a stable molecule in chemical sense, as it reacts readily with oxygen [8]. Therefore, only emission spectra of CS were discovered in 1911 by Strutt and Fowler [9] and were investigated later by Martin [10] and Jevons [11]. Following the observation and investigation of an overview CS absorption spectrum with high spectral resolution by Huang et al. [7], analytical application became feasible [7,12]. Recent review articles of Butcher [13] and Resano et al. [14] about high-resolution continuum source molecular



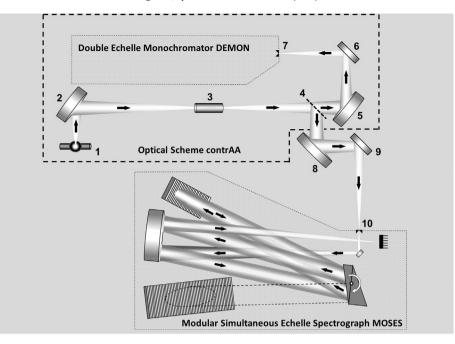


Fig. 1. Optical scheme of the set-up for the recording of molecular absorption spectra. 1 – Xe arc continuum source; 2, 5, 8 – elliptical mirrors; 3 – atomizer; 4 – beam splitter; 6, 9 – plane mirrors; 7 – DEMON entrance slit; 10 – MOSES entrance slit.

absorption spectrometry include the state of knowledge regarding the determination of S via CS and SnS molecules.

compound shows the second highest bond strength of the sulfide molecules, analytical application has not been reported to date.

The emission and absorption spectrum of SiS was first observed and investigated by Barrow and Jevons in 1938 [15]. Although this

The feasibility of sulfur determination via molecular absorption of GeS [16] and SnS [17] has been explored using line-AAS

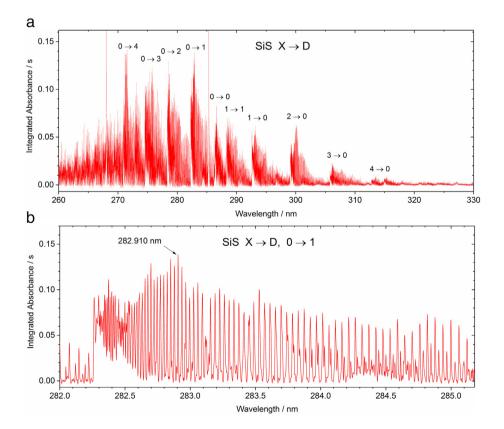


Fig. 2. a) Overview absorption spectrum of SiS using 5 µL 0.5% Si (sodium silicate) standard solution and 10 µL 500 mg L⁻¹ S (ammonium sulfate) standard solution in the graphite furnace, recorded with the MOSES spectrograph. Conspicuous band heads are marked by the corresponding vibrational quantum numbers. Additional Na atomic lines appear at 268.043 nm and 285.29 nm. b) Detail of the SiS absorption spectrum of panel (a) with marking of the rotational line selected for S determination.

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