

## SHORT COMMUNICATIONS

## A New Spectrophotometric Method for the Determination of Tungsten with Stilbazogall I

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A new spectrophotometric method for the determination of a trace of tungsten has been developed. It is based upon the formation of a red tungsten(VI) complex of Stilbazogall I, stilbene-4, 4' - bis [(1-azo)-2, 3, 4-trihydroxybenzene] - 2, 2' - disulfonic acid (I), which was synthesized by Cherkosov et al.<sup>1)</sup> and which has recently been reported to be a reagent for the determination of zirconium.

The absorption spectra of the complex at various pH values are shown in Fig. 1, from which it may be seen that tungsten(VI) forms more than one type of complex. In detailed experiments, two types of complexes were found. One of them was a 1 : 1 metal-to-reagent complex formed at about pH 2, while the other was a 1 : 2 complex

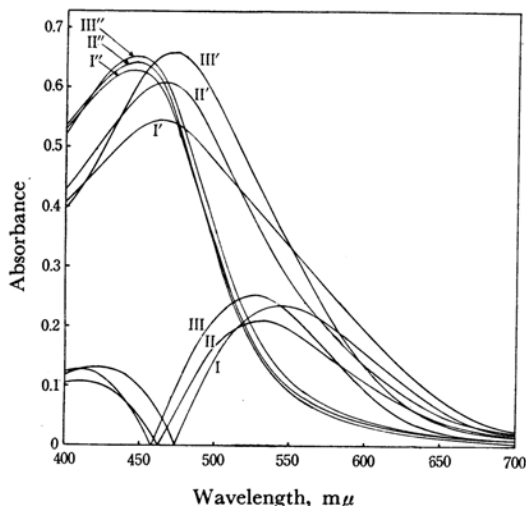
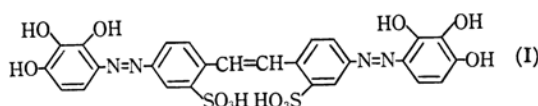


Fig. 1. Absorption spectra of W(VI) - Stilbazogall I complex.

W(VI):  $1 \times 10^{-5}$ ; Stilbazogall I:  $2 \times 10^{-5}$  mol./l.  
pH: I, I', II'', 2.2; II, II', II'', 3.5; III, III' III'', 4.8.

Reference: I, II, III, reagent blank  
I', II', III', I'', II'', III'', water  
I'', II'', III'' stilbazogall I alone

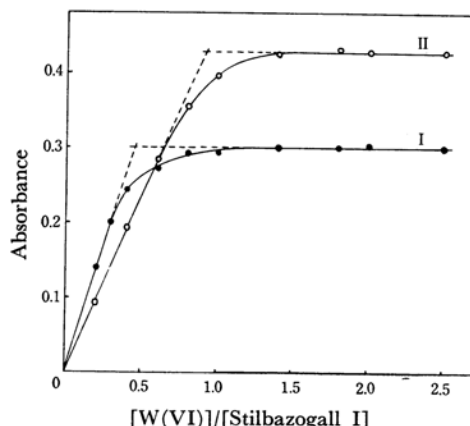


Fig. 2. Determination of mole ratio of W(VI) - Stilbazogall I complex by the mole ratio method.  
Stilbazogall I:  $2 \times 10^{-5}$  mol./l.

I: pH 4.9, 520 mμ II: pH 2.2, 550 mμ

formed at about pH 5. The composition of the complex was found to change continuously from 1 : 1 to 1 : 2 with an increase in the pH value. Figure 2 shows the results obtained by the mole ratio method as an example. A linear relationship was found between the concentration of tungsten(VI) and the absorbance. The molar extinction coefficients of 1 : 1 and 1 : 2 complexes were about  $2.1 \times 10^4$  and  $3.7 \times 10^4$ , and the sensitivities were  $8.7 \times 10^{-3}$  and  $4.9 \times 10^{-3}$   $\mu\text{g. W/cm}^2$  respectively, corresponding to  $\log(I_0/I) = 0.001$ . The proposed procedure for the determination is as follows. To a sample solution containing tungsten(VI) in a 25-ml. volumetric flask, 5.0 ml. of a Stilbazogall I solution ( $10^{-4}$  mol./l.) and 5 ml. of a sodium acetate-acetic acid buffer solution (pH=4.9) are added. The solution is then diluted to the mark with distilled water. The absorbance of the solution is measured at 520 mμ against a reagent blank solution between 5 and 15 min. after the addition of the reagent solution. Some metals, such as aluminum(III) copper(II), molybdenum(VI), titanium(IV), and zirconium(IV), interfere under the conditions proposed.

The details of this study will be reported elsewhere.

1) A. I. Cherkosov and Yu. V. Pushinov, *Zhur. Anal. Khim.*, **20**, 665 (1965).