ON THE COLORIMETRIC DETERMINATION OF BORON

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It has been established that the method suggested by MONNIER, RUSCONI and WENGER cannot lead to reliable results. Namely, the boron content of the solution is only in part responsible for the changes of extinction and they are strongly influenced by the buffercapacity of the sample.

The pH of the medium has a very important role in analytical procedures based on complex formation. On the basis of our former work [1] we made the MONNIER, RUSCONI and WENGER's method for determination of boron [2] the subject of a critical examination.

The principle of this method is that the ferric-sulphosalicylic acid complex is partially decolourized by fluoride ion and the increasing of absorbancy due to the effect of borax solution is measured:

$$2\,Fe^{3+} + 2\,A{-}S^{-n} = 2(Fe\,A{-}S)^{3-n}$$

$$2\,(Fe\,A{-}S)^{3-n} + 12\,F^{1-} = 2\,(FeF_6)^{3-} + 2\,A{-}S^{-n}$$

$$2\,(FeF_6)^{3-} + 3\,B^{3+} = 3\,(BF_4)^{1-} + 2\,Fe^{3+}$$

$$2\,Fe^{3+} + 2\,A{-}S^{-n} = 2\,(Fe\,A{-}S)^{3-n}.$$

The authors mentioned in their paper that "the colour is a function of the pH value, and it is necessary therefore, to work to a definite hydrogen ion concentration. With this object in view we have added phenolphtalein to the borax solutions, followed by hydrochloric acid, 2N, until the indicator is just decolourized." However, it is evident that this procedure cannot eliminate the change of pH during the determination, because the pH of ferric-sulphosalicylic acid prepared according to the instruction is 1,67.

Consequently, it follows that the change of absorbancy can not be attributed to the effect of the boron alone, but the boron and the change of pH are jointly responsible for it.

The effect of pH and concentration of boron on the absorbancy of ferric-sulphosalicylic acid-NaF system was studied. The colorimetric measurements were carried out with Pulfrich photometer, for the pH determinations Radiometer PHM Type 22 instrument was used. First of all we took down — following the authors' instructions — the calibration curve, and the change of pH was determined simultaneously. As from the paper cited it is

not clear that the original calibration curve at what wavelength was taken down, the measurements were carried out with three filters: S_{42}, S_{47} and S_{50}.

Table I contains the result of these experiments.

Table I

ml 0,2 % borax	pH	Absorbancy		
		S_{42}	S_{47}	S_{50}
0	3,50	0,189	0,166	0,149
4	3,73	0,481	0,413	0,355
6	3,88	0,583	0,607	0,442
8	3,90	0,655	0,681	0,520

Further, we carried out analogous experiments for studying the effect of pH. In these experiments instead of 0,2 per cent borax solution 0,2 per cent $NaHCO_3$ solution was applied the pH of which was equal to that of the borax solution. The data are summarized in Table II.

Table II

ml 0,2 % $NaHCO_3$	pH	Absorbancy		
		S_{42}	S_{47}	S_{50}
0	3,50	0,189	0,166	0,149
3	4,00	0,312	0,335	0,241
5	4,78	0,981	1,180	0,780
8	5,89	2,950	2,950	2,340

Comparing the data of the tables it is evident that the boron determination according to MONNIER, RUSCONI and WENGER's method cannot give reliable results because for the change of absorbancy observed the boron content of the solution is partly responsible, and these are significantly influenced by the pH of the final solution which is determined by the buffer capacity of the solution examined. An other inconvenient problem, discernible from the data of tables, under the above conditions the indicator properties of ferric-sulphosalicylic acid complex has to be taken in consideration as well.

Experiments were carried out at constant pH, however, the changes of absorbancy observed were so slight that an advantageous method on the basis of principle suggested by the authors is not to be expected.

References

[1] *Szabó, Z. G., M. T. Beck:* Anal. Chem. **25**, 103 (1953).

[2] *Monnier, D., Y. Rusconi, P. Wenger:* Anal. Chim. Acta **1**, 13 (1947).