

## DETERMINATION OF SODIUM HYPOCHLORITE BY FLOW INJECTION ANALYSIS

Zhou Zeng-Nan, Cheng Jian, Fan Xiao-Qin

Department of Chemistry, Textile University of China  
1882 Yan-An Rd. (west), Shanghai, 200051, PRC

### SUMMARY

The results of determination of available chlorine in NaOCl in the textile industries using flow injection analysis is discussed in this paper. NaBr and HOAc are used as carriers. The absorbance is monitored at 420 nm. The procedure is simple, fast and can be carried out easily. The consumption of the reagents is very low. To be compared with the iodometric titration method, the relative errors of FIA is less than  $\pm 2.1\%$ . The results show high accuracy and good reproducibility.

In comparison with the routine quantitative analytical method, flow injection analysis (FIA) has an advantage of not only a high degree of accuracy and good reproducibility, but also smaller injected sample volume, more simple and rapid analytical procedures and less amount of reagent consumed. Especially, the versatility and simplicity of the FIA systems possibly leads to a significant change in attitude towards automation in the analytical laboratory. Now FIA has developed into a well-established analytical procedure and a very useful method for determination (1,2).

We applied the recently developed FIA method for determination of available chlorine in NaOCl.

### EXPERIMENTAL SECTION

**Reagents and solutions:** NaBr, KI,  $\text{Na}_2\text{S}_2\text{O}_3$  were of analytical reagent grade from commercial source and were used after dissolving in distilled water. HOAc, NaOCl and other chemicals were of chemical pure grade. Distilled water was used throughout.

**Apparatus:** A FIA-21 (Orient Instrument and Apparatus Co.) was used in this study. The detector used was a UV-721 Spectrophotometer with a flow cell (The Third Analytical Instrument Manufactory). The absorbance was monitored at different wavelengths, in order to obtain the maximum absorbance under the approximate wavelength for different "colored" forms.

**Procedure:** Calibration curves were obtained by adding with pipets NaOCl solutions (2.5, 5.0, 7.5, 10.0, 12.5, 15.0 mL) into six 250 mL volumetric flasks. Dilute to the mark, shake well, keep away from strong lights as much as a possible. Inject each solution through pump into FIA instruments (Fig. 1). Pump samples into FIA systems directly.

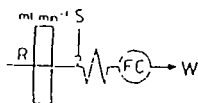


Fig. 1: The sample (S) is injected into the reagent carrier (R) which is continuously added through a pump. After passing through the reaction coil (45 cm x 0.5 cm) and further carried through the flow cell (FC) for spectrophotometric measurement. R: 8g NaBr in 1L 0.17 mmol HOAc; Flow rate: 1.0 mL/min. S: Sample volume 27  $\mu$ l, Detector wavelength 420 nm.

## RESULTS AND DISCUSSION

The calibration curve (Fig. 2) was prepared for use with conventional iodometric titration method. The results show good linearity. To be compared with the iodometric method, the relative errors of FIA is less than  $\pm 2.1$  %. Reproducibility testings were performed and high reproducibility was observed. For  $n=10$ , variation coefficient is  $5.4 \times 10^{-3}$ .

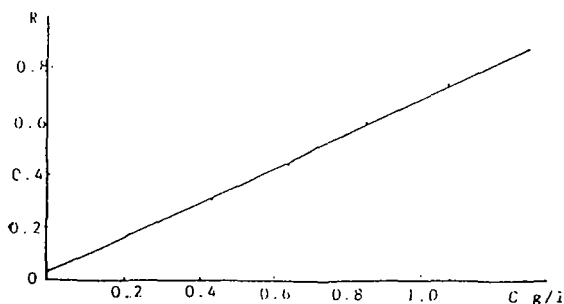


Fig. 2: The calibration curves of available chloride in NaOCl. The linearity equation is  $X = 1/B \times Y - A$ ,  $1/B$  is the slope of this curve. Here  $B = 1.49$ ,  $A = -8.8 \times 10^{-2}$ , correlation coefficient is 0.9999

**Peak shapes and Measurements:** A typical peak shape was observed in this system. The peak heights vary with NaOCl concentrations. Also, as the NaBr concentration was increased, the peak height was increased. However, at concentrations of NaBr between 2.0 to 4.0 g/L, the peak is splitted. It is because the content of NaBr is too low to react with NaOCl. Response is over the detection range, if above 10.0 g/L. Here, the concentration of 8.0 g/L NaBr was chosen.

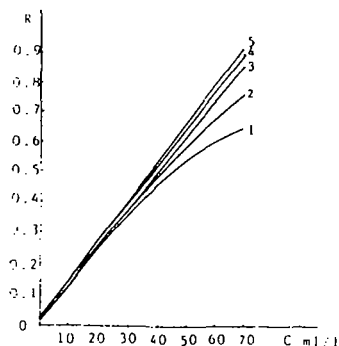


Fig. 3: Effect of NaBr concentrations on responses. 1,2,3,4 and 5 represent 2.4, 4.0, 6.0, 8.0, 10.0 (g/L) NaBr, respectively.

**Effect of Carrier Acidity:** In this system the effect of the carrier concentration was investigated. The response was increased as the acidity of the carrier was increased. The peak was split when HOAc concentration was lower than 0.14 mol/L. Especially at the higher sample concentration. Fig. 4 shows peak shape of 0.14 mol/L HOAc.



Fig. 4: Effect of HOAc concentrations on peak shape. 1. represents 1.2 g/L NaOCl, 2. represents 1.07 g/L NaOCl.

HOAc concentration above 0.26 mol/L,  $\text{Cl}_2$  is formed and interferes in the detector. In our system, the optimum concentration of HOAc is 0.17 mol/L.

**Effect of injected sample volume and length of reaction coil:** The response was increased as the injected sample volume was increased. Considering the upper limit, 27  $\mu\text{l}$  is suitable. As the reaction coil length was increased from 45 cm to 90 cm, the response was decreased. This is clearly due to the fact that in FIA the sample zone is dispersed as well as the length. Therefore, dispersion was increased as the length was increased. Thus, 45 cm was selected as the length of reaction coil.

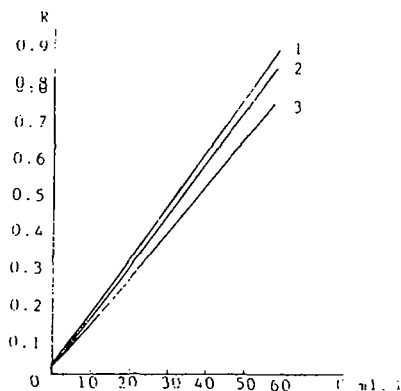


Fig. 5. Effect of reaction coil length on responses. 1,2,3 represents 45 cm, 65 cm and 90 cm, resp.

## CONCLUSIONS

A new method of determination of available chlorine in NaOCl in the textile industry has been developed. In this system the sample could be directly pumped with a pump and continuously collected and analyzed by FIA apparatus. The good accuracy was obtained as in the conventional titration method. Moreover, the consumption of the sample and chemicals were very small. If further investigations are performed, the technique could be applied in on-line continuous monitoring.

## REFERENCES

- (1) J.Ruzicka, E.H. Hansen *Anal. Chim. Acta*, **78**, 145 (1975)
- (2) Z.N. Zhou et al. *J. China Textile University*, **16**, 75 (1990)