Technical Information:

Date:	Nov 2006
Subject:	Chlorine Dioxide
Topic :	Generation Of Small Volumes Of Chlorine Dioxide For Labs

Preliminary remarks

The process described here below is designed according to the Chlorine Dioxide generation taking place in BelloZon plants. As in laboratories, unlike the plant conditions, the process takes place in a **pressureless** way and Chlorine Dioxide as a gas is quite volatile despite its good solubility in water, the process has to take place in a fumehood. Chlorine Dioxide is toxic (threshold limit value: 0.1 ppm) and explosive starting from a certain partial pressure. According to some literature, also aqueous solutions disintegrate spontaneously at a concentration above ~ 28 g/l. The concentration of the reaction solution being generated in this case is approx 20 g/l.

Generation

In an open and, if possible, dark coloured glass vessel the volume of which should not exceed 20 ml, 5 ml of at least 9% Hydrochloric Acid is added. Next, 5 ml 7.5% Sodium Chlorite solution is added quickly while swivelling it from a pipette, and the mixture is put apart for some 15 minutes, covered with e.g watchglass, base board etc. In the meantime, a 11 volumetric flask is filled with 700 ml distilled water, and after the reaction time having elapsed, the 10 ml mixture is added to the distilled water. After having filled up the flask to the marking, we have a stock solution which is some 180 mg/1 and can be stored in a refrigerator over a longer period due to its excess acid (approx pH 2). Should this excess acid affect the subsequent tests, the solution can be purified by transferring the gas into a second washing bottle by means of air using a vacuum pump. It is true, however, that the losses are considerable and the stability of the neutral solution is not very high.

<u>Analysis</u>

We recommend to check the concentration of the stock solution regularly. For this, iodometric titration by thiosulfate is well suited. As during the generation no chlorine is formed, the Chlorine Dioxide concentration is directly proportional to the thiosulfate consumption measured at pH 7.



Example:

50 ml stock solution + 10 ml Phosphate buffer pH 7 + 2 g Potassium Iodide Indicator: starch solution (to be added at the end of the titration) Consumption: 1.33 ml 0.1 N Sodium Thiosulphate Calculation: 1.33 ml x 6.75 / volume 0.05 l = 180 mg/l (= ppm)

In the water treated by Chlorine Dioxide, the ClO_2 -concentration is most easily determined according to the DPD method by means of reagent DPD No 1, and the result ("Free Chlorine") is calculated into PPM Chlorine Dioxide by multiplying it with factor 1.9. If the DT1 photometer is used, the Chlorine Dioxide value can be measured directly by selecting ClO_2 measurement parameter.