



Free Chlorine Standard

Voluette® Cat. No. 14268

Preparation

These standards were prepared by generating and dissolving chlorine gas in slightly alkaline, high purity water of zero chlorine demand. The standard is packaged into clean ampules and sealed under argon. After preparation, ampules are assayed and stored between 2° - 8° C prior to shipment.

Standardization

From a practical standpoint, it is impossible to prepare an accurate chlorine standard by direct weighing chlorine added to water. It is necessary to prepare the chlorine standard and then determine the actual chlorine content.

Analyses are performed on 10 ampules taken at random from the production lot. The analyses are performed spectrophotometrically, using a method calibrated versus titration with standard sodium thiosulfate. The sodium thiosulfate is standardized against NIST's SRM 83d, primary standard arsenic trioxide.

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Storage and Life Expectancy

Many types of chlorine solutions were investigated for stability. It was found that pure chlorine in pure water was the most stable, although it does decay at a rate dependent upon temperature.

Storage Temperature

Rate of Loss

3°C (40° F, refrigerated)

No measurable loss in 18 months

10°C (50°F)

0.009% loss/day

25°C (room temp.)

0.05% loss/day

100°C (boiling temp.)

18.7% loss/day

It is recommended that the chlorine ampules be stored under refrigeration between 35° - 45° F, (2° - 10° C), immediately upon receipt.

Minimize exposure to sunlight by keeping unopen ampules in its closed box. When stored under these conditions, the expected stability at the stated concentration is at least 18 months.

Procedure

1. Prepare a chlorine determination by adding DPD to a 25-mL water sample.
2. Measure the color and read the mg/L free or combined chlorine. Record the value.
3. Snap open an ampule of chlorine standard and, with the use of the TenSette Pipet, add 0.1 mL of standard to the sample. Swirl to mix.

4. Read the mg/L chlorine and record.
5. Calculate the concentration of chlorine added to the sample:

$$\frac{\text{mL of standard added}}{\text{mL of standard added} + \text{mL of sample}} \times \text{label Value of Voluette (mg/L)}$$

For example:

The label shows the average free chlorine concentration of the ampules to be 65.5 ± 0.3 mg/L.

Using 0.1 mL standard added and 25mL sample, the concentrations of free chlorine added to the sample is calculated according to the equation in step 5:

$$\frac{0.1 \text{ mL}}{0.1 \text{ mL} + 25 \text{ mL}} \times 65.5 \text{ mg/L} = 0.26 \text{ mg/L chlorine}$$

Therefore, the addition of 0.1 mL ampule standard should increase the chlorine content by 0.26 mg/L (Step 4 - Step 2). If less is recovered, begin a systematic check to locate and correct the problem. See the general booklet, *Standard Additions for Analytical Quality Control*.

Notes

The use of the Free Chlorine Standards for standard additions performs entirely satisfactorily when there is a chlorine residual already in the sample and the standard additions are made to the sample after the chlorine reagent (DPD) is already in the sample. The system will work

quite well even if the sample has a small chlorine demand because the free chlorine will react with the chlorine reagent to produce the chlorine color before it is consumed by the chlorine demand in the sample.

It will not work if there is a chlorine demand and the standard addition is made to the sample before the chlorine reagent is added to the sample. In this case some or most of the chlorine from the standard addition will be consumed before the chlorine reagent is added and the color is developed.

There is a very slow loss of chlorine from the ampules after they are opened. This is negligible if the ampule is used immediately after being opened. The rate of loss is about 5% per hour from an opened ampule even when there is gentle air movement in the room.

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