

ANALYSIS OF CHLORINE IN HIGHLY TURBID OR COLORED WATERS

Problem

Analyzing chlorine in highly turbid or colored waters is difficult due to the selection of a proper sample blank that accurately compensates for the residual sample turbidity and color that remains under the reaction conditions of the developed DPD Chlorine test. This is especially difficult in produced and flowback water samples. The visual DPD titrametric test was not considered due to the potential interference of high color backgrounds. Alternative analysis technologies, such as amperometric titration, have proven inadequate.

Solution

The background color and turbidity level in samples often changes under the reaction conditions of the chlorine test method being used. This makes the background “Zero” difficult to determine. The volatility of chlorine eliminates filtration to remove turbidity or the use of other chemical means to remove the background color.

Ideally, a best solution is to create same or nearly identical reaction conditions in the sample blank to compensate for background effects in the developed sample. Dissolution/formation of turbidity is duplicated and pH effects are captured. The background “zero” is made by removing the DPD color with excess FAS.

Procedure for determining chlorine in turbid or colored samples:

1. Collect a chlorinated water sample
2. Add DPD Chlorine Free or Total Reagent to the sample; allow color to react for specified time.
3. Read color on chlorine calibration curve vs. DI water.
4. Remove sample cell and add 1-2 drops of (FAS) Ferrous Ammonium Sulfate Solution, 0.125 N or other similar reducing agent, to discharge the developed pink color.
5. Read the sample cell again. A negative absorbance will be obtained proportional to the amount of chlorine present.
6. *Alternatively*, split the developed sample in step 2 into two sample cells. Add the FAS to one cell, swirl to mix.
7. Use the FAS sample to set ZERO.
8. Read the remaining sample cell to determine the amount of chlorine present.

Experimental

Sample: Eagle Ford Shale black flowback water sample. 100 ml sample size spiked with 8 ml of ~ 1000 mg/L chlorine solution. Let stand 30 minutes. This was repeated using a Laredo, TX shale flowback water sample.

- Add 1 pp of DPD Total Rgt (1406499) to 25 ml of sample.
- Wait 3 minutes. Split into two 10 mL portions in 1" sample cells.
- Add 1 drop of Ferrous Ammonium Sulfate (FAS), 0.125 N, PN2054849, to one sample cell. Swirl to mix. Pink color was discharged leaving a yellow background with some turbidity.
- Zero the instrument using this FAS treated sample split.
- Read the prepared sample.

Used Program 87 DR 3800 and 1" glass cells. All values read vs. DI water.

Sample	DPD (No FAS)	DPD (with FAS)	Orig Sample	Orig with FAS
Eagle Ford	3.84	0.79	0.88	0.88
Laredo	2.38	0.36	0.40	0.40

Conclusion

Samples had less visual turbidity in DPD FAS blank than did original sample. This had been reported by MI-SWACO & MIOX test site. No change occurred on original sample when FAS was added to it. Results look very encouraging and technically feasible.

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