
LETTER

Importance of Temperature on the Starch-Iodine End Point

To the editor:

We wish to point out the importance of the effect of temperature on the accuracy of the iodometric titration, colorimetric, starch-iodine end point. This basic procedure is used to determine chlorine residuals (< 10 mg/L) according to ASTM Tests for Residual Chlorine in Waste Water, Method A—Starch-Iodide Titration Method; (D 1427-68) (newly revised, ASTM Tests for Residual Chlorine in Water [D 1253-76]).

In this standard method, temperature is not mentioned as being an important variable. The results of preliminary tests in our laboratory have confirmed that as the sample solution temperature rises above 20°C, significant error can result because of premature end points. For example, a 0.25 mg/L chlorine residual titrated at 25°C gave an apparent value of 0.16 mg/L; and, for a 0.75 mg/L chlorine residual titrated at 30°C, only 0.50 mg/L was obtained. Indeed, a sample containing an actual chlorine residual of 0.50 mg/L was found not to produce a blue color at slightly over 30°C. Preci-

sion for the titrations was determined to be ± 0.03 mg/L. A ten-fold increase in starch content (0.25% starch in 1% acetic acid; Fisher Scientific, Chicago, IL) did reduce the error somewhat, but an increase in iodide ion had no effect.

A literature search provided surprisingly little information on this subject. Several analytical text books provide information that imply that the starch-iodine end point is temperature dependent, but no quantitative data or references are given. A detailed study to further characterize this phenomenon is currently underway in our laboratory.

It is urgently recommended that an appropriate note or supplement be included in the newly revised ASTM D 1253-76 stating the above findings that when using the starch-iodine titration for determining low levels of residual chlorine the temperature of the sample solution must be kept at or below 20°C.

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