

IODOMETRIC METHOD FOR CCA SOLUTION ANALYSIS

by

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Background

Staccioli and Tamburini (1982) have reported a method of analyzing chromated-copper-arsenate (CCA) solutions using a titrametric method during which the pH of the test solution is carefully controlled. The method involved addition of ethylene diamine tetra-acetic acid (EDTA) to the CCA solution followed by acidifying it with concentrated hydrochloric acid. Potassium iodide was added and the liberated iodine titrated with sodium thiosulphate to determine the chromium. The pH of the solution is modified when the end-point for the first titration is reached and the titration continued to determine the copper. The process is repeated for the arsenic.

When the procedure was investigated at Forintek, it was not possible to produce reliable and consistent results when the method described by Staccioli and Tamburini was followed. It was therefore studied and modifications made to the procedure, mainly affecting the pH's of the solutions used in the titration steps. Following modification, the method was tested extensively and found to produce excellent reproducible analyses on both industrial as well as specially prepared laboratory CCA solutions.

Initial investigations using buffer solutions, while useful, were found to be unnecessary. The elimination of the surfactant from the titration was found to have no effect.

Method

Take 10 ml of 0.1 M EDTA, 10 ml of 10% KI and 20 ml of petroleum ether (30-60) and adjust the pH of the mixture to 2.00 through the addition of concentrated hydrochloric acid. The resulting solution is titrated with 0.1 M sodium thiosulphate until the organic layer turns from violet to colorless. The aqueous layer turns from brown to green as the thiosulphate is added. When the end-point is reached the aqueous layer turns blue-green. Wait 30 seconds to ensure that the violet color does not return. Add concentrated hydrochloric acid until the pH is 0.65-0.67. The bottom layer will be brown-green while the upper layer will be violet. A fine white precipitate (CuI) will appear. Titrate again to a colorless end-point in the organic layer. Add 60 ml of concentrated hydrochloric acid and repeat the titration a third time.

Reference

Staccioli, G. and U. Tamborini. 1982. Iodometric analysis of copper-chrome-arsenic wood preservative solutions. Material U. Organismen. 17(4):199-204.