Australian Standard®

Methods of test for supplementary cementitious materials for use with portland cement

Method 7: Determination of sulfide sulfur content

PREFACE

This Standard was prepared by the Standards Australia Committee on Supplementary Cementitious Materials for use with Portland Cement.

METHOD

1 SCOPE This Standard sets out the reference method for determination of the sulfide sulfur content of supplementary cementitious materials.

NOTE: This method assumes the absence of sulfites, thiosulfates and any other sulfur compounds intermediate between sulfides and sulfates.

WARNING: OBSERVE SAFE PROCEDURES FOR DILUTING CONCENTRATED ACIDS AND ALKALIS AND WHERE TOXIC GASES ARE GENERATED.

2 PRINCIPLE The sulfide content of a sample is released as hydrogen sulfide by reaction with hydrochloric acid. The gas emitted is quantitatively absorbed in an ammoniacal zinc sulfate solution and titrated with standard iodate solution to the iodine-starch complex end point.

3 REAGENTS

3.1 General All reagents shall be of analytical reagent grade and free from impurity levels which will significantly interfere with the determination of sulfide sulfur by this method.

Distilled or demineralized water shall be used throughout the analysis.

3.2 Solutions The following solutions are required:

(a) Ammoniacal zinc sulfate solution—dissolve 50 g of zinc sulfate heptahydrate ($ZnSO_4.7H_2O$) in 150 mL of water. Add 350 mL concentrated ammonia solution (ϱ_{20} 0.880kg/L) and stir well.

Allow to stand for at least 24 h and filter the solution.

NOTE: Alternatively a solution of ammoniacal cadmium chloride may be used, prepared in the same way as the ammoniacal zinc sulfate solution but using 15 g of cadmium chloride. (Zinc sulfate solution is preferred because zinc sulfate is more soluble in concentrated ammonia solution. Cadmium chloride may be used when there is doubt about the presence of traces of sulfide sulfur. The appearance of yellow cadmium sulfide clearly indicates its presence).

(b) Hydrochloric acid (500 mL/L)—prepare from concentrated hydrochloric acid (ϱ_{20} 1.180 kg/L).

(c) Hydrochloric acid (250 mL/L)—prepare from concentrated hydrochloric acid (ϱ_{20} 1.180 kg/L).

(d) Potassium iodate solution—(1 mL $\equiv 4.809 \times 10^{-4}$ g sulfide).

After drying approximately 2 g of potassium iodate (KIO_3) crystals to constant mass at 180°C, weigh between 1.069 g and 1.071 g into a beaker and add 12 g of potassium iodide (KI). Dissolve in a minimum of water and quantitatively transfer to a 1 L volumetric flask. Make up to the mark and mix thoroughly.

NOTE: This solution is normally stable for up to 12 months when stored out of direct sunlight at below 20°C. If solution is older than this, or if there is any doubt as to the accuracy of its concentration, it should be replaced with a fresh solution.