Nitrogen, Kjeldahl

For water and wastewater

Peroxide Digestion Method

Introduction

The test for Kjeldahl nitrogen, also referred to as crude protein, is used to determine ammonia and organic nitrogen present in a sample. Only small fractions of nitrite and nitrate nitrogen are included in the test. A preliminary digestion is used to oxidize carbon compounds to carbon dioxide, and to convert organic forms of any nitrogen present (amino acids, proteins, peptides) to ammonia. The traditional digestion uses sulfuric acid and various combinations of metallic catalysts and salts. Digestion of at least 2 hours is followed by addition of sodium hydroxide to the digest and then distillation of the ammonia into a boric acid or buffer solution. Ammonia in the distillate is measured with back titration or nesslerization. This procedure requires several hours for reagent preparation, digestion, distillation and final measurement.

The Hach Digesdahl Digestion Apparatus and the Peroxide Digestion Method allow completion of the Kjeldahl test within 15 minutes or less, depending on the nature of the sample. First, the sample is charred in concentrated sulfuric acid. Fifty percent hydrogen peroxide is fed into the reaction mixture, where it oxidizes organic carbonaceous matter and converts organic nitrogen into ammonium bisulfate. For example, the reaction with glycine, a simple amino acid is:

 $NH_2CH_2COOH + 2H_2O_2 + H_2SO_4$ Ç $NH_4HSO_4 + CO_2 + 2H_2O$ Glycine

Digestion apparatus

The Digesdahl digestion apparatus includes a fractionating column. Hydrogen peroxide, added slowly, trickles into the reaction mixture in the flask below. The temperature of the reaction is maintained near the boiling point of sulfuric acid (300 °C, 572 °F). Vapors from the reaction rise to the column where SO₂ and water vapors are drawn off by an aspirator. Hydrogen peroxide vapors condense in the column and return to the reaction mixture.

It should be noted that no metal catalysts or salts are used in digestion. The digest is suitable for nesslerization for final measurement without an intermediate distillation step. The digest is also suitable for mineral analysis of Ca, Mg, Mn, K, P and Zn. See *Nitrogen, Ammonia* for more information about the Nessler method.