1 Dissolution for Total Elemental Analysis

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1-1 INTRODUCTION

Elemental analysis of soils and rocks necessitates their decomposition into soluble forms by acid digestion or fusion with various fluxes. Historically, elemental analysis was developed for the analysis of rocks and minerals (Washington, 1930). The earlier two procedures for bringing the elements of the sample into solution are (i) fusion in Na₂CO₃ and (ii) digestion with HF and either HClO₄ or H₂SO₄ (Groves, 1951; Hillebrand et al., 1953; Jackson, 1958; Bennett & Hawley, 1965; Maxwell, 1968; Jeffery, 1970). Both of these procedures are known to present difficulties in the subsequent measurements of elemental composition by spectrometry (Bowman & Willis, 1967). An advantageous single HF-decomposition atomic absorption technique has been successfully developed for 50- to 100-mg samples of rocks, sediments, and soils by the use of a closed vessel and H₃BO₃ to conserve Si.

1-2 PRINCIPLES

1-2.1 Digestion with Hydrofluoric Acid in Closed Vessel

A specially designed Teflon (duPont) decomposition vessel with a sealed cover allows the rapid decomposition of silicates in HF without volatilization losses of SiF₄ gas (Bernas, 1968). The method is adapted for the determination of 18 elements (Buckley & Cranston, 1971). An improved procedure that better prevents loss of SiF₄ (gas), employs a 250-ml polypropylene bottle and saturated H₃BO₃ solution (Sridhar & Jackson, 1974; Jackson, 1974). The tightly capped, leakproof polypropylene bottle prevents the volatilization losses of the SiF₄ gas that is decomposed by H₃BO₃ (see below). Careful execution of this latter step results in the quantitative recovery of the elements of interest.

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