

## CALCITE IN THE SEMI-ARID SOILS OF PAKISTAN I. COMPARISON OF QUANTIFICATION METHODS

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### ABSTRACT

Calcite quantification made by the HCl and H<sub>2</sub>SO<sub>4</sub> methods, using Merck pure calcium carbonate precipitated, indicated that both the methods have serious discrepancies at low levels of carbonate content. In the calcimeter, rise in the mercury level, with each increment of carbonate, was linear with no variation among the replicates. In acetic acid method the relationship of pH of solution in equilibrium with varying amount of CaCO<sub>3</sub> was linear with log[(mg CaCO<sub>3</sub>)/(500-CaCO<sub>3</sub>)]. Soil carbonates determined from the standard curve by pH developed by acetic acid were compared with those obtained by the HCl, H<sub>2</sub>SO<sub>4</sub>, and calcimeter methods. Generally the methods compared well with the published values. Although, the four methods yielded comparable values for carbonate contents, the acid neutralization methods, HCl, H<sub>2</sub>SO<sub>4</sub>, and acetic acid, showed slightly higher values than that of calcimeter method.

### INTRODUCTION

Accurate quantification of carbonate is important to understand chemical and physical properties of arid zone soils. Inorganic carbonates occur in soils as calcite (CaCO<sub>3</sub>) and dolomite (CaCO<sub>3</sub>.MgCO<sub>3</sub>) calcite being the dominant phase (Doner and Lyvnn. 1989). Hereafter,

calcite and dolomite in soil (Peterson et al., 1966; Nelson, 1982). The most commonly used procedures involve dissolution of solid carbonates with acid and either quantification of CO<sub>2</sub> evolved or measurement of acid consumed during the neutralization reaction. The evolved CO<sub>2</sub> is measured volumetrically, gravimetrically, manometrically or by infrared spectroscopy or gas chromatography. Acid consumed is measured by addition of excess acid and back titration of unreacted acid with a standard base.

The acid neutralization procedures (U.S. Salinity Laboratory Staff, 1954; Cottenie, 1980; Bundy and Bremner, 1972) are probably the most widely used methods due to their simplicity. However, many potential sources of error exist in acid neutralization methods. Less crystalline clay and Fe oxide may mask titrimetric end point and/or consume protons from the solution by reaction with structural hydro-oxides (Loeppert et al., 1984). Another widely used procedure is quantitative volumetric measurement of CO<sub>2</sub> with Chittike apparatus following addition of excess HCl. Rabenhorst 1988) proposed a method which utilizes dry combustion and gravimetric determination of evolved CO<sub>2</sub> for the determination of both organic and inorganic C in soil samples. The pressure-calcimeter, which measures increase in pressure due to evolved CO<sub>2</sub> as a result of acid-carbonate reaction at constant temperature

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