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METHODOLOGY AND PRINCIPLE OF X-ray Diffractometer (XRD)

X-ray diffraction Analysis for Mineral Identification

- Powdered samples were pelletized and sieved to 0.074mm. these were later taken in an aluminum alloy grid (35mm x 50mm) on a flat glass plate and covered with a paper. Wearing hand gloves, the samples were compacted by gently pressing them with the hand.
- Each sample was run through the Rigaku D/Max-lllC X-ray diffractometer developed by the Rigaku Int. Corp. Tokyo, Japan and set to produce diffractions at scanning rate of 2 °/min in the 2 to 50° at room temperature with a CuKa radiation set at 40kV and 20mA. The diffraction data (d value and relative intensity) obtained was compared to that of the standard data of minerals from the mineral powder diffraction file, ICDD which contained and includes the standard data of more than 3000 minerals.
  Similar diffraction data means the same minerals to standard minerals which exist in the soil sample.

PRINCIPLE OF DIFFRACTION

The XRD analysis is based on passing X-ray beam through a clay sample. The X-ray idenfies the structural layers which is dependent on the d-spacing of the clay minerals. The d-spacing is the exact spacing of the staking of the crystal lattices which indicates the arrangement of the atoms in a mineral. The X-ray on passing through the clay samples gives peaks that is typical of each type of diffracted along a group of planes and the way they are diffracted is characteristics of the arrangement of the atoms within the mineral.

DIFFRACTOMETER TECHNIQUE

- The less than 2 micron clay fraction was grouped and the powder was pressed into an aluminium sample holder and rim through a wide angle Phillips P.W. 1011 goriometer connected to a PM 8220 recorder.
- The scanning was done from 2' to 40' under the following instrumental setting conditions.
- Nickel filtered Fek and radiation
- Recording/scanning rate = 1' 20cm/min.
- Time = constant 4.
- Range = 4 x 10 C.P.S
- Voltage is 28kv/12mH
- The interpretation of the diffractograms was done by using the reference conversion table to 20 to d-values for the Fek alpha radiation to the JCPDC manual (1972)