## Test Procedure for

# QUALITATIVE AND SEMI-QUANTITATIVE ANALYSIS OF CRYSTALLINE MATERIAL BY X-RAY DIFFRACTION

## TxDOT Designation: Tex-896-B

***Effective Date: August 1999***

## 1. SCOPE

1.1 Use this method to obtain X-ray diffraction patterns from materials possessing some degree of crystallinity and in the qualitative and quantitative analysis of those materials.

1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

## 2. APPARATUS

2.1 *X-ray diffraction apparatus,* with necessary high-voltage generator and data measuring system, capable of scanning the range of interests for the material being examined and recording the X-ray diffraction pattern within this range on a strip chart recorder. The unit must be equipped with an X-ray tube having a copper target, beryllium window, rated for at least 3000 watt, 60 KV operation, and equipped with a scintillation detector and focusing monochromator with a graphite crystal serving as a band filter.

**Note 1**—Philips Electronic Instruments Model 170 - 138 - 01 Wide Range Goniometer with vertical diffraction kit, DMS 31 IC Data Measuring System, XRG 3000 Generator, Focusing Monochromator Model 3-202-GV, and broad focus, copper target X-ray diffraction tube.

2.2 *Stiff, Kraft paper tabs,* approximately 34.9 × 76.2 mm (1-3/8 × 3 in.) punched 6.4 mm (0.25 in.) from one end with a 6.4 mm (0.25 in.) hole. Kraft paper is preferred because it is commonly free of kaolin, which might contribute to the diffraction pattern.

2.3 *Miniature bar coater,* 51 mm (2 in.). Brass screws, 8-32 or 6-32, with the heads cut off, are suitable for this purpose.

2.4 *Glass slides,* approximately 38.1 × 38.1 mm (1.5 × 1.5 in.)

2.5 *Aluminum mask,* 38.1 × 38.1 × 1.6 mm (1.5 × 1.5 × 1/16 in.) with a rectangular milled opening 20 × 15 mm (4/5 × 3/5 in.) whose center is located 12.5 mm (0.5 in.) from one side and centered on the mask.

## 3. DISCUSSION

3.1 This test uses the principle of constructive reinforcement (diffraction) of monochromatic X-rays by the sets of electron-rich parallel planes existing in any crystalline material. Diffraction maxima occur at a set of angles (between X-ray source and sample and between sample and detector) determined by Bragg's Law and the lattice parameters of the crystalline material. The X-ray wavelength employed in practice is often copper K radiation of 1.54 D, but certain elements (e.g., iron) fluoresce at this wavelength, causing an interference. In such cases, substitute an X-ray tube generating iron K or other wavelength, or use a pulse height analyzer or crystal monochromator or both.

3.2 The diffraction maxima occurring at the various Bragg angles of constructive reinforcement appear on the strip chart as peaks as the goniometer scans the selected range of angles. The strip chart is thus a plot of X-ray intensity versus angle. Each crystalline substance has a characteristic set of Bragg angles with a relative intensity (or peak height) associated with each such angle. After correcting for certain matrix effect and particle size effects, the size of a particular peak representing one component of a mixture varies with the percent of that component in the mixture. Interpret the strip chart to determine what crystalline components are present (qualitative analysis) and in approximately what relative quantities (semi-quantitative analysis).

## 4. PREPARING SAMPLES

4.1 *Thick Film Forming Mixtures*—for mixtures that form films sufficiently thick, uniform, and opaque to X-rays when dip-coated and which dry properly at such film thickness*:*

4.1.1 Dip the stiff paper tab 63.5 mm (2.5 in.) into the well-mixed sample, withdraw, and suspend vertically by the punched hole until dry.

4.1.2 Cut 19.1 mm (0.75 in.) from each end of the tab.

**Note 2**—The center section must be coated uniformly with the mixture film.

4.1.3 Place the center section, backed with a glass slide, into the specimen holder of the goniometer, making certain the paper is flat against the slide.

4.2 *Semi-Transparent Film Forming Mixtures*—for mixtures that do not dry well when dip-coated, or that form films semi-transparent to the X-ray so that the cellulose and kaolin of the paper might contribute to the X-ray pattern:

4.2.1 Coat one surface of a 38.1 × 38.1 mm (1.5 × 1.5 in.) glass slide with the well-mixed sample using a miniature bar coater to produce a thick, uniform film.

4.2.2 Allow to dry with the slide resting horizontally.

4.2.3 Place the slide in the specimen holder of the goniometer.

4.3 *Satisfactory Film Forming Mixtures*—for mixtures that form a satisfactory film, opaque to X-rays, by simply pooling a quantity of the material on a glass slide and drying:

4.3.1 Apply an appropriate amount of the mixture to one surface of a 38.1 × 38.1 mm (1.5 × 1.5 in.) glass slide to produce a uniform film.

4.3.2 Allow to dry with the slide resting horizontally.

4.3.3 Place the slide in the specimen holder of the goniometer.

4.4 *Powdered and Packed Materials*—for materials satisfactorily ground to a dry, uniform powder and packed to give a surface thick and uniform enough to be opaque to x-rays:

4.4.1 Place an aluminum mask, referenced under Section 2, on a 38.1 × 38.1 mm (1.5 × 1.5 in.) glass slide.

4.4.2 Place an appropriate amount of material for analysis in the opening of the aluminum mask.

4.4.3 Pack the material in the opening of the aluminum mask using another glass slide.

4.4.4 Remove the excess material with the edge of the glass slide, then place the slide flat on the surface of the aluminum mask creating a sample "sandwich" between the two glass slides.

4.4.5 Reverse the sample pack and remove the front slide carefully.

4.4.6 Place the pack, with the packed surface exposed, in the specimen holder of the goniometer.

4.5 *Crystalline Materials*—for crystalline materials, cut or cloven (sliced) to present a smooth, planar surface:

4.5.1 Prepare the material to present a smooth, planar surface of appropriate size.

4.5.2 Place the slide in the specimen holder of the goniometer.

## 5. DATA COLLECTION

5.1 *Qualitative Analysis:*

5.1.1 Operate the equipment according to the manufacturer's instructions to produce the desired diffraction patterns.

5.1.2 Permit the goniometer to scan over an angular range corresponding to a "D" spacing range of approximately 12.5–1.75 D.

5.1.3 If suspecting the presence of expanded clay minerals, it may be necessary to search the very low angles corresponding to "D" spacing up to 25 D or more to detect the strong peaks.

5.2 *Quantitative Analysis:*

5.2.1 After identification of the crystalline matter in the sample, prepare standards containing known quantities of each.

5.2.2 Determine the diffraction pattern of these standards in the same manner as that of the diffraction pattern of the unknown.

5.2.3 Locate one or more well-resolved peaks, without known interference from probable ingredients (in the literature or from charts run on individual raw materials) to represent each crystalline ingredient of the material. Any peak used for estimating quantity of an ingredient must be at least 20% of full scale above the background. It is not necessary that all peaks remain on chart. Scan peaks that go off chart individually at a less sensitive setting after completing the main scan. It is usually possible, however, to adjust the sensitivity setting so that most peaks remain on the chart. Compared peaks must be from the same equipment, using the same diffractometer setting.

## 6. DETERMINATIONS

6.1 *Qualitative:*

6.1.1 Compare patterns according to ASTM D 934, paragraph 9.1.

6.1.2 Compare interplanar spacings and relative intensities determined from the diffraction patterns with those of published data until a match is found

6.2 *Quantitative:*

6.2.1 Select suitable and similar diffraction lines and compare the intensities produced by the sample with the intensities produced by standards with known quantities. (When attaining a match of similar intensities, estimate quantity of materials in unknown sample.)