Designation: F3419 – 22

Standard Test Method for Mineral Characterization of Equine Surface Materials by X-Ray Diffraction (XRD) Techniques[1]

This standard is issued under the fixed designation F3419; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 X-Ray diffraction (XRD) is a tool for identifying minerals, such as quartz and feldspar, and types of clay present in bulk samples of equine surfaces. Determining the mineralogy of a given bulk sample provides insight into surface properties, such as abrasion resistance by comparing the relative differences of hardness of the various mineral fractions such as quartz or feldspar or the plasticity differences in clay minerals such as smectite or kaolinite. XRD techniques are qualitative in nature and only semi-quantitative.

1.2 Particle size distribution analyses methods including hydrometer tests to determine proportions of sand, silt, and clay fractions based upon particle size but are not able to distinguish particles by shape or mineralogy of materials. In addition to a qualitative detection of minerals present in a sample, XRD methods are also semi-quantitative and also yield important data on the relative proportion of particular minerals present.

1.3 XRD techniques are generally semi-quantitative in nature. Even so, such semiquantitative data is useful in determining relative proportions of each mineral type. This method is also semi-qualitative in nature as it is geared for the determination or mineral groups. For example, it will determine the relative amount of alkali feldspars (such as K-feldspar or Nafeldspar) from Plagioclase-feldspar but not necessarily if the Plagioclase-feldspar is albite or anorthite nor whether the K-feldspar is orthoclase of microcline. Likewise, it will differentiate smectite from mica from kaolinite but not whether the smectite is montmorillonite or saponite. More precise determination of mineral species by XRD is possible but involves more advanced preparation and treatment methods than what is within the scope of this standard.

1.4 The XRD method herein primarily makes use of "Glass Slide Method" but may be subject to modification depending on the user's needs.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D75 Practice for Sampling Aggregates

F3401 Test Method for Wax Binder Removal from Equestrian Synthetic Track Surfaces

3. Terminology

3.1 *Definitions:*

3.1.1 *clay fraction, n*—a less than 4 µm Equivalent Spherical Diameter (ESD) fraction of the sample.

3.1.1.1 *Discussion*—Clay size in many classification systems is the <2 µm fraction but for the purposes of this method the < 4 µm size is used. Also, the clay fraction does not necessarily mean clay minerals (phyllosilicates) but rather it is a size term and, as such, this size fraction includes non-clay minerals (quartz, plagioclase, etc.). This size fraction is used because it also typically contains abundant clay minerals.

3.1.2 *petrography, n*—branch of petrology that focuses on the detailed description and classification of minerals, rocks, sands, and soils.

3.1.3 *solid solution, n*—a homogeneous solid phase capable of existing throughout a range of chemical composition.

3.1.4 *X-ray diffraction (XRD), n*—a rapid analytical technique primarily used for phase identification of a crystalline material and for determining unit cell dimensions.

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[1] This test method is under the jurisdiction of ASTM Committee F08 on Sports Equipment, Playing Surfaces, and Facilities and is the direct responsibility of Subcommittee F08.28 on Equestrian Surfaces.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.1.4.1 *Discussion*—XRD involves the scattering of X-rays by the regularly spaced atoms of a crystalline mineral which is useful for identification of the mineral and information about the structure of the crystal. In the preparation process, the mineral material to be analyzed is finely ground, homogenized, and from which the average bulk composition is then determined.

4. Summary of Test Method

4.1 Identification of the constituents of a sample is usually a necessary step towards recognition of the properties that may be expected to influence the behavior of the material in its intended use, but identification is not an end in itself. The value of any petrographic examination will depend to a large extent on the representativeness of the samples examined, the completeness and accuracy of the information provided to the petrographer concerning the source and proposed use of the material, and the petrographer's ability to correlate these data with the findings of the examination.

5. Significance and Use

5.1 Petrographic examinations are made for the following purposes:

5.1.1 To determine the mineralogy of the material that may be observed by petrographic methods (in this method, by use of XRD) and that may have a bearing on the performance of the material in its intended use.

5.1.2 To determine the relative amounts of the constituents of the sample which is essential for proper evaluation of the sample when the constituents may differ significantly in properties that have a bearing on the performance of the material in its intended use.

5.1.3 This method helps to evaluate mineral aggregate sources for suitability as a material to be used for construction, renovation, or modification of equine surfaces. The information gathered will allow for the comparison of the composition of new mineral sources with samples of other mineral aggregate from one or more sources, for which test data or performance records are available.

5.2 This method may be used by a petrographer employed directly by those for whom the examination is made. The employer should tell the petrographer, in as much detail as necessary, the purposes and objectives of the examination, the kind of information needed, and the extent of examination desired. Pertinent background information, including results of prior testing, should be made available. The petrographer's advice and judgment should be sought regarding the extent of the examination.

5.3 This method may form the basis for establishing arrangements between a purchaser of consulting petrographic service and the petrographer. In such a case, the purchaser and the consultant should together determine the kind, extent, and objectives of the examination and analyses to be made and should record their agreement in writing. The agreement may stipulate specific determinations to be made, observations to be reported, funds to be obligated, or a combination of these or other conditions.

6. Interferences

6.1 Mineral standards used to determine calibration factors are often different from the actual minerals analyzed.

6.2 With this method, the data always sums to 100 %. This means that the percentages reported for each mineral are dependent upon the percentages reported for the other minerals. If one mineral is under-estimated, the others will be overestimated. Also, if one or more minerals are present, but not detected, then the percentages of the minerals that are detected will be overestimated.

6.3 Detection limits for XRD are on the order of one to five weight percent. The detection limits differ for each mineral species.

6.4 Certain types of materials may be difficult to test. Minerals such as feldspars that undergo solid solution are especially problematic. Clay minerals are problematic for this same reason. Clay minerals also have a wide range of crystallinities (poorly crystallized to well crystallized), which may compound this problem.

6.5 XRD methods can quantify crystalline material only. Organic non-crystalline or other non-crystalline material, or both, in large concentrations can be detected, but not quantified. Therefore, any organic or non-crystalline material, or both, is not included in the accompanying results.

6.6 Any or all the above factors may affect the estimated weight percentages. Data are formatted as weight percent but are calculated as weight fractions. Therefore, slight rounding errors may be observed in the formatted data.

7. Apparatus

7.1 *Convection Oven*—A device capable of heating material and holding a temperature of 110°C (230°F) ± 1°C for a minimum of a 16-h period.

7.2 *Mortar and Pestle*—A device capable of disaggregating a sample into powdered form.

7.3 *Micronizing Mill*—A device used to pulverize sample material into a refined powder.

7.4 *Sonic Probe*—A device used to promote disaggregation of particles held in solution.

7.5 *XRD*—An automated powder diffractometer equipped with a copper X-ray source (40 kV, 30 mA) and scintillation X-Ray Detector capable of scanning over an angular range of 2 to 60° two theta (2θ) at a scan rate of one degree per minute.

8. Reagents and Materials

8.1 Varies with the material tested.

9. Sampling, Test Specimens, and Test Units

9.1 Samples for petrographic examination should be taken by or under the direction of an individual familiar with the requirements for random sampling of aggregates. This may include using the sampling protocol from Practice D75. Information on the exact location from which the sample was taken and other pertinent data should be submitted with the sample to allow for any necessary changes in preparation techniques to provide the most accurate results. The amount of material actually studied in the petrographic examination will be determined by the nature of the examination to be made and the nature of the material to be examined, as discussed below.

NOTE 1—Depending on the materials tested, the procedure may be subject to modification provided it is performed in accordance with any applicable ASTM standard or equivalent international standards and reported appropriately.[3]

10. Procedure

10.1 Depending on the materials tested, the procedure may be subject to modification provided it is performed in accordance with any applicable ASTM standard or equivalent international standards and reported appropriately. XRD samples when loaded into their glass slide mounts must be smooth, flat, long enough, thick enough, and should be mineralogically homogenous throughout their depth of thickness.[4] If the samples are from a synthetic surface (containing a wax-oil based binder), using Test Method F3401 or similar wax extraction method must be performed (stripping using solvents).

10.2 Thoroughly clean approximately 20 g of sample material for any obvious contaminants such as fiber or wax content by appropriate means.

10.3 Place the prepared sample into a mortar and pestle and gently and thoroughly grind to initially disaggregate the sample material.

10.4 Examine the sample material again for any obvious contaminants and remove them as appropriate and create a split sample.

10.5 Using distilled water, create a small solution with the sample material per the micronizing mills manufacturer's instructions and transfer into the mill and pulverize the sample accordingly.

10.6 Afterwards, place the sample material into a convection oven set at 60°C until all moisture has been thoroughly removed.

NOTE 2—A 16-h period is recommended but not required.

10.7 Disaggregate the material again using the mortar and pestle and place into the petrographer's preferred sample holder to produce bulk sample mounts.

10.8 With the separate split of the sample material, disperse the sample in distilled water using a sonic probe. When relevant, proper pre-treatment will be required for removal of salts and gypsum.

10.9 Fractionize the suspensions with a centrifuge to isolate the clay fractions for a separate clay mount.

10.10 Vacuum deposit the suspensions on a nylon membrane filter to produce oriented clay mineral amounts.

10.11 Attach glass slides to the mounts and expose to ethylene glycol vapor for a minimum of 12 h.

10.12 Using the XRD, analyze the bulk sample over an angular range of 5 to 60° two theta (2θ) at a scan rate of one degree per minute. Upon completion, results should be qualitatively analyzed to determine the clay types present in the samples. Determinations of mixed layer clay ordering and expandability should be done by comparing experimental diffraction data from the glycol-solvated clay mounts with simulated one-dimensional diffraction profiles. More precise determination of phyllosilicates would require mono-saturation of clays.

10.13 Using the XRD, analyze the clay fraction sample over an angular range of 2 to 36° two theta (2θ) at a scan rate of one degree per minute. Upon completion, semiquantitative determinations of the bulk mineral amounts should be completed using Whole Pattern Fitting Rietveld refinement methods on observed data. A diffraction model is then fit to the measured pattern by non-linear least square optimization in which parameters must include background, profile parameters, and lattice constants. For Rietveld refinement, a complete physics simulation should be used in which crystal structures of the phases are required.

11. Report

11.1 Report the following:

11.1.1 Date and test date.

11.1.2 Name of laboratory, company, or individual issuing the report.

11.1.3 Project identification number.

11.1.4 Bulk sample (weight percentage), Phyllosilicate Mineralogy (relative abundance), and Summary Mineralogy (weight percent) data in a tabularized summary.

11.2 Any graphs and charts may also be included to illustrate the results but are not required.

12. Precision and Bias

12.1 *Precision*—Due to the bias associated with the semi-qualitative nature of XRD testing, standard deviation can vary significantly depending on the test sample, preparation method, and operator.

12.2 *Repeatability*—The repeatability values associated with this test method are based on the repeated mounting and analysis of a sample by a single operator. Ten trials were performed to determine the weight percentage magnetite in a sample. The repeatability standard deviation was determined to be 0.324wt%, with a 95 % repeatability standard deviation of 0.907wt%. The average presence of magnetite in the sample was 27.5wt%. Standard deviation and error will vary depending on the type and weight percent of a mineral within a sample, as demonstrated in the reproducibility section.

12.3 *Reproducibility*—The reproducibility of this test method is based on an interlaboratory study conducted in the year 2019. Four laboratories were provided a set of diffraction traces and asked to quantitatively analyze the samples. Their results for each mineral within a sample were averaged, and used to observe relative error with respect to mineral weight

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[3] Burnett, A., "A Quantitative X-Ray Diffraction Technique for Analyzing Sedimentary Rocks and Soils," *Journal of Testing and Evaluation*, Vol. 23, No. 2, 1995.

[4] Moore, D. M. and Reynolds, R. C., Jr., *X-Ray Diffraction and the Identification and Analysis of Clay Minerals*, Oxford University Press, New York, NY, 1997.