

Standard Test Method for Evaluation of Solid Particulate Removal Effectiveness Using X-Ray Fluorescence Techniques for Evaluating Cleaning Effectiveness of Residential/Commercial and Central Vacuum Cleaners

1. Scope

- 1.1 This test method covers the use of X-Ray Fluorescence (XRF) to measure the average percent removed by weight of a set of compounds used to soil test carpet.
- 1.2 This test method provides a laboratory test for determining the relative solid particulate removal effectiveness of residential and/or commercial upright, canister, central, and combination vacuum cleaners when tested under specific conditions.
- 1.3 This test method applies to solid “dry soil” particulate (*XRF soil*) removal from carpets, not the removal of common surface litter and debris.
- 1.4 This standard may involve hazardous materials, operations, and equipment. It 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 and health practices and determine the applicability of regulatory limitations prior to use. This test utilizes a small X-Ray generating machine, which is regulated by State and Federal Laws. See Section 8.

2. Referenced Documents

2.1 ASTM Standards:

ASTM D6540
Standard Test Method for Accelerated Soiling of Pile Yarn Floor Covering

3. Terminology

- 3.1 *model, n* – the designation of a group of cleaning units having identical mechanical and electrical design with only cosmetic or nonfunctional differences.
- 3.2 *test run, n* – the definitive procedure that produces a singular measured result.

- 3.3 *unit, n* – a single cleaning unit of the model being tested.
- 3.4 *X-ray fluorescence, n* – An emission spectrographic technique that detects chemical elements by ionizing the constituent atoms and recording the characteristic energy signatures of photons given off by the elements
- 3.5 *pass, n* – A linear vacuum cleaner movement in one direction. (The out and back motion of manual vacuuming involves two passes.)
- 3.6 *XRF soil, n* – the combination of five particulate compounds designed to provide a consistent dry soiling media for use in the CRI SOA/GL Vacuum Cleaner Program (see 7.1, Table 1)

4. Summary of Test Method

- 4.1 This test method uses a standard set of particulate compounds (*XRF soil*) containing suitable elements for XRF detection. These compounds are described in Section 7.1. The compounds are first individually applied to nylon pellets, which are used to transfer the compounds to a specified area of a sample carpet following ASTM D6540. XRF is used to verify the starting concentration of each compound. The soiled carpet samples are then subjected to a specific vacuuming process, followed by XRF scans to determine the amount of each compound remaining in the carpet.

5. Significance and Use

- 5.1 The test method will be used to generate data that can quantify the carpet vacuum performance of various vacuum cleaning equipment.
- 5.2 This test method will provide an indication of the capability of a specific vacuum cleaner to remove solid “dry soil” particulate (*XRF soil*) from carpet. The amount of particulate removed in the laboratory practice will differ from that in residential and/or commercial installations due to variations in carpet styles, soil and other solid particulate composition, the vacuuming process employed by individual operators and other factors.
- 5.3 In order to provide a uniform basis for measuring the performance in section 1.1, specific test carpets and specific test solid particulate (*XRF soil*) are employed in this practice.
- 5.4 There is no established correlation between field and laboratory results. However, the relative differences between equipment tested under this specific test method provide a means for performance comparisons.

6. Apparatus

- 6.1 X-ray Fluorescence Instrumentation (See Annex A.6.1)
- 6.2 Voltage-Regulator System (See Annex A.6.2)
- 6.3 Reciprocating Conveyor or equivalent (See Annex A.6.3)
- 6.4 XRF Instrument Stand or equivalent (See Annex A.6.4)
- 6.5 Non-metallic Grid Riser or equivalent (See Annex A.6.5)
- 6.6 XRF sample cup. (See Annex A.6.6)
- 6.7 Weight Scales (See Annex A.6.7)
- 6.8 Carpet Pile Comb with plastic teeth. (See Annex A.6.8)
- 6.9 Control Vacuum Cleaner (See Annex A.6.9)
- 6.10 Drum Tumbler/Ball Mill (See Annex A.6.10)
- 6.11 Carpet Sample Rack (See Annex A.6.11)
- 6.12 Jar Mill and Bell Jar (See Annex A.6.12)
- 6.13 Chrome Alloy Ball Bearings (See Annex A.6.13)
- 6.14 Vacuum Gauge (See Annex A.6.14)
- 6.15 Tachometer scaled in feet/second. (See Annex A.6.15)
- 6.16 USA Standard Test Sieve No.16 and Standard 8" Round Layer Cake Pan (See Annex A.6.16)
- 6.17 Temperature Controlled Air Circulating Oven (See Annex A.6.17)

7. Reagents and Materials

- 7.1 Soiling compounds suitable for XRF detection range (*XRF soil*). (See Annex A.7.1)

| | |
|---------------------|--------------------------------|
| Iron Oxide | Fe ₃ O ₄ |
| Zinc Oxide | ZnO |
| Strontium Carbonate | SrCO ₃ |
| Yttrium Oxide | Y ₂ O ₃ |
| Zirconium Boride | ZrB ₂ |

7.2 Test carpet

Table 2 – Test Carpet Specifications (See Annex A.7.2)

| Residential Cut Pile | | Loop Pile | | Commercial Cut Pile | |
|-----------------------------|---------------------|-----------------------|---------------------|----------------------------|---------------------|
| Weight | 25 oz/sq yd ± 7% | Weight | 30 oz/sq yd ± 7% | Weight | 30 oz/sq yd ± 7% |
| Color | * | Color | * | Color | * |
| Pile Height | 0.470 inch | Pile Height | 0.123 inch | Pile Height | 0.281 inch |
| Gauge | 1/8 | Gauge | 1/8 | Gauge | 1/10 |
| Stitches/Inch | 8 | Stitches/Inch | 8 | Stitches/Inch | 9 |
| Yarn | Nylon | Yarn | SD Nylon | Yarn | Nylon |
| Fluorochemical | No | Fluorochemical | No | Fluorochemical | No |

7.3 Test Carpet Template (See Annex A.7.3)

7.4 Polyamide polymer pellets (Annex A.7.4)

7.5 Isopropyl alcohol (see Annex A.7.5)

8. Hazards

8.1 The XRF unit as described in this standard typically operates at less than 50 kV and 15 μ A, and produces X-rays at much lower intensities than a typical medical X-ray machine; however, the unit and its use will still be covered by state and federal regulations. Federal regulations usually apply to the manufacturer, while state regulations apply to the user. State regulations vary and are usually handled by either the Department of Health or Radiation Safety. It is a common requirement for each unit to be registered annually with the state. All users must be trained in both operation and general radiation safety. The XRF instrument produces low levels of radiation such that radiation monitoring is generally not required. The manufacturer of the unit shall provide the necessary training and guidance for complying with applicable regulations.

8.2 Chemical compounds may pose a health risk; therefore, refer to the Materials Safety Data Sheets (MSDS) for the selected compounds.

9. Sampling, Test Specimens, and Test Units

9.1 Only one test vacuum unit is required for this test method.

9.2 A minimum of three (3) carpet samples of the loop pile test carpet and three (3) samples of the appropriate cut pile carpet (based on the vacuum's intended use) shall be tested for each unit. Prepare carpet samples in accordance with ASTM D 6540, except as noted here:

- 9.2.1 The carpet sample size shall be 263 mm X 1003 mm (10 ³/₈" X 39 ¹/₂") or equivalent based on test apparatus. (See Annex A.9.2.1)
 - 9.2.2 Soiling shall be done with "XRF soil" as found in the five Suitable XRF Soiling Compounds in Table 1. (See Annex A.7.2)
 - 9.2.3 Soil transfer shall be facilitated by using polyamide polymer pellets. (See Annex A.7.4, and A.10.1)
 - 9.2.4 Identify all test samples with identification numbers.
- 9.3 Vacuum Cleaner Conditioning
- 9.3.1 Preconditioning a new test vacuum cleaner by energizing the vacuum cleaner in a stationary position at the rated voltage \pm 1% and rated frequency with filters in place for one (1) hour.
 - 9.3.2 Preconditioning New Rotating Agitator Type Test Vacuum Cleaner in a stationary position by energizing the vacuum cleaner for one (1) hour with the agitator bristles not engaged on any surface.
 - 9.3.3 Preconditioning a New Straight-Air Canister Test Vacuum Cleaner by energizing the vacuum cleaner in a stationary position for one (1) hour with a wide-open air inlet with hose attached.

10. Data Collection Software and XRF Unit Setup and Verification

- 10.1 The supporting computer software will collect a spectrum from the XRF unit and save the spectrum as a function of Energy-versus-Counts. The Operation of the XRF Unit and software must be verified on the day the data is collected. (See Annex A.10.1)

11. Conditioning

- 11.1 Test Room – Maintain the test room in which all conditioning and testing is performed at 21 ± 3 °C (70 ± 5 °F) and 50 ± 5 percent relative humidity. Store carpet samples in a horizontal, non-ventilated sample rack. Do not stack carpet samples directly against each other.
- 11.2 All components involved in the test shall remain and be exposed in the controlled environment for at least 16 hours prior to the start of the test.
- 11.3 The test carpets can be prepared and stored in test room up to 72 hours in advance of testing.

12. Procedure

12.1 Test Carpet Preparation

12.1.1 Vacuum each test specimen with the control vacuum cleaner using four passes at a rate of 0.55 meters (1.8 ft.) per second, prior to the application of the soiling media.

12.2 Test Carpet Soiling Procedure

12.2.1 Mount the carpet sample in the drum of the drum tumbler apparatus with the drum in a horizontal position and the seam at the top with pile lay opposite the rotation direction of the drum. (See Annex A.6.10)

12.2.2 Prepare compound coated nylon pellets for each of the five soiling compounds listed in Section 7.1, per ASTM D 6540. There will be five different sets of pellets, one for each of the five soiling compounds. (See Annex A.7.1)

12.2.3 Weigh-out $1193 \text{ g} \pm 2 \text{ g}$ of the chrome alloy steel balls.

12.2.4 Weigh out $50 \text{ g} \pm 0.1 \text{ g}$ of pellets for each of the five compound coated pellets. The total weight of coated nylon pellets shall be $250 \text{ g} \pm 0.5 \text{ g}$.

12.2.5 With the drum horizontal to the axis of rotation and the carpet seam at the top, spread the chrome alloy steel balls uniformly across the width of the mounted carpet sample.

12.2.6 Spread the compound coated pellets over the steel balls such that they are uniformly distributed across the width of the carpet sample in the order they are listed in Table 1.

12.2.7 Seal the drum and rotate for 30 minutes at $35 \text{ RPM} \pm 2 \text{ RPM}$.

12.2.8 Carefully remove, by hand (do not vacuum), the steel balls and all of the used compound coated pellets. Remove the carpet sample from the steel drum being careful not to dislodge the XRF soil. Place carpet samples in a horizontal, non-ventilated sample rack. Do not stack carpet samples against each other.

12.2.9 Comb the sample against the pile lay one time (See 6.8) with a carpet pile comb to stand the pile erect before scanning.

Note: The XRF peak intensity drops as the inverse square of the distance from the fiber to the detector; hence, it is important that the samples be combed before they are scanned to maintain a constant position. Short loop pile carpets do not need to be combed; however, it is essential that pile orientation and its distance relative to the sensor be maintained.

12.2.10 Perform the XRF scan procedure on the soiled carpet samples.

12.3 XRF Scan Procedure

12.3.1 The XRF scan shall be performed before and after the cleaning of the test carpet (See Annex A.12.3)

Note: Sample carpet supplier normally pretests sample carpet for this test method to eliminate XRF soil elements from the test carpet. (See Annex A.12.3)

12.3.2 Perform all scans of the sample using the XRF unit and support software. To scan the sample, place the XRF unit in its stand at the set height so that the carpet does not touch the unit. Place the carpet sample on its non-metallic riser on the conveyor and move it to an appropriate start point to start the automated XRF Scan Pattern. Use non-metallic shims below the non-metallic riser for each carpet type to assure the static height of the XRF head is consistently positioned above the carpet sample pile at the correct focal length determined for each sample type. (See Annex A.12.3)

12.3.3 The objective is to scan the largest practical area to a depth of the carpet fiber column above the carpet backing, which is representative of the soiling and/or cleaning. The scan pattern shall be defined to cover the largest practical area of the sample in three minutes based on the specified XRF scan speed and XRF scan width (See Annex A.12.3)

Note: When soiled as described above, the samples should have starting concentrations in the ranges recommended in Table 3. Specific starting concentrations may vary with the exact carpet used and batch of compounds used. For a given set of tests, the starting concentrations for each compound should not vary by more than 20% within a given sample.

Note: By averaging over 3 samples, uncertainty arising from starting variations will be reduced. The percent removed will be reported instead of the amount removed because of possible variations in starting concentrations. Additional information on method precision is given in Annex A.13.1.

Table 3 – Required Starting Concentrations For XRF Soil for Vacuums

| Compound | Residential Cut Pile | Loop Pile | Commercial Cut Pile |
|--------------------------------|-----------------------------|-----------------------------|-----------------------------|
| Fe ₃ O ₄ | 0.62 to 0.98 grams/sq meter | 0.37 to 0.88 grams/sq meter | 0.39 to 0.80 grams/sq meter |
| ZnO | 0.37 to 0.70 grams/sq meter | 0.30 to 0.78 grams/sq meter | 0.79 to 1.24 grams/sq meter |
| SrCO ₃ | 0.30 to 0.68 grams/sq meter | 0.25 to 0.55 grams/sq meter | 0.62 to 0.93 grams/sq meter |
| Y ₂ O ₃ | 0.56 to 0.88 grams/sq meter | 0.33 to 0.80 grams/sq meter | 1.09 to 1.54 grams/sq meter |
| ZrB ₂ | 0.88 to 1.27 grams/sq meter | 0.64 to 1.20 grams/sq meter | 1.45 to 1.90 grams/sq meter |

12.4 Vacuum Cleaning Procedure

12.4.1 Place the soiled carpet sample in an appropriate carpet template (See 7.3) of the same style of carpet so that a larger carpet sample is presented to the test vacuum cleaning equipment, and the vacuuming process does not encounter a height difference at any edge of the sample. Place soiled sample and template on conveyor with pile lay, if present, toward the vacuum.

- 12.4.2 Mount the test vacuum cleaning unit in place on the conveyor with the handle in an inclined operating position at a vertical handle height of 888 mm (31 ½”) above the test material and adjust the vacuum following the manufacturer directions for any adjustable settings. All adjustable settings shall be recorded.
- 12.4.3 Initiate reciprocating movement of conveyor at 1.8 feet (0.55 meters) per second, making four shift passes. All direction changes shall be done on template surface, not on test sample surface. (See 4 Shift Pass Diagram, Annex A.12.4).
- 12.4.4 After vacuuming, the carpet samples are to be stored in a horizontal, non-ventilated sample rack in standard test conditions. After a minimum of 16 hours to allow sample reconditioning, and not more than 72 hours, repeat the XRF scan procedure to establish the ending concentrations of soiling compounds in the vacuumed test carpet. (See 11.1)

13. Calculation or Interpretation of Results

- 13.1 Three soiled carpet samples are used for each test and the results are averaged. The reported result is the cumulative average percent removed of all soiling compounds, with the exclusion of zinc oxide. The average removed of each compound should also be reported because each compound has distinct properties and can indicate more detail than the average.

The concentration for each compound is calculated from the XRF scan, using a calibration that is dependent on the XRF instrument's sensitivity to the element in the selected compound. Each compound, due to its natural affinity to the carpet, soils at a different rate. Therefore, even though the same amount of each compound was applied to the carpet, the starting concentrations of each compound are not the same. The cumulative average percent removed is an average across compounds. (See Annex A.13.1)

Note: Five compounds are included in the standard XRF soil composition (see 7.1). Vacuums are not designed to remove sticky contaminants like Zinc Oxide (ZnO), but sticky contaminants can be present on carpet and can have an adverse effect on overall soil removal when vacuuming. Although the amount of Zinc Oxide removed is not included in the calculated percentage for total soil removal by test vacuum cleaners its presence in XRF soil is indicative of typical carpet soiling in its effect on the total percent removed by vacuuming of the other four “dry” XRF soil compounds.

14. Report

- 14.1 Product test number, chain of custody number (CRI number), test date, test method conducted, vacuum name, model number, manufacturer, serial number, bag type, filter types, and vacuum settings.
- 14.2 Complete carpet test material description, including carpet style and other specifications from Table 2. (see 6.0)
- 14.3 Calculated percentage removed for each compound, excluding zinc oxide, and the average percentage removed of all compounds combined, excluding zinc oxide.

Note: These reporting differences are designed to provide CRI only with the data necessary to make a pass/fail determination for the vacuum being tested under the test method and to protect proprietary data of individual vacuum manufacturers.

- 14.4 Any modification or deviation from the standard test method shall be noted in the test report.

15. Precision and Bias

- 15.1 Precision – No inter-laboratory tests have been performed; therefore, no precision statement regarding the repeatability and reproducibility of this test method are available at this time.
- 15.2 Bias – No bias has been established for this practice.

16. Keywords

- 16.1 Vacuum; Cleaning; Soil Removal; Carpet

ANNEX A (Mandatory Information)

A.5.0 Significance and Use

- A.5.1 NOTE: The generated data may be useful to equipment manufacturers in product development. Selected generated data will be used by the CRI to evaluate and approve vacuum cleaning equipment and to rate equipment tested to standards established under the voluntary SOA/GL Vacuum Cleaner Program.

A.6.0 Apparatus Details

- A.6.1 **X-Ray Fluorescence Instrumentation** capable of detecting elemental concentrations of the selected compounds through XRF spectral analysis. Related computer software providing support for XRF analysis of selected soiling compounds.

Note: Handheld TRACeR XRF, with software capable of quantifying soil removal from floor coverings, supplied by Bruker Industries has been found to be a suitable instrument for this purpose. The TRACeR XRF and required software is covered by a patent

TRACeR XRF (handheld)
Bruker Industries
800-466-5323

- A.6.2 **Voltage-Regulator System**, to control the input voltage to the cleaning equipment. The regulator system shall be capable of maintaining the equipment's rated voltage \pm 1 %, and rated frequency having a wave form that is essentially sinusoidal with 3 % maximum harmonic distortion for the duration of the test.

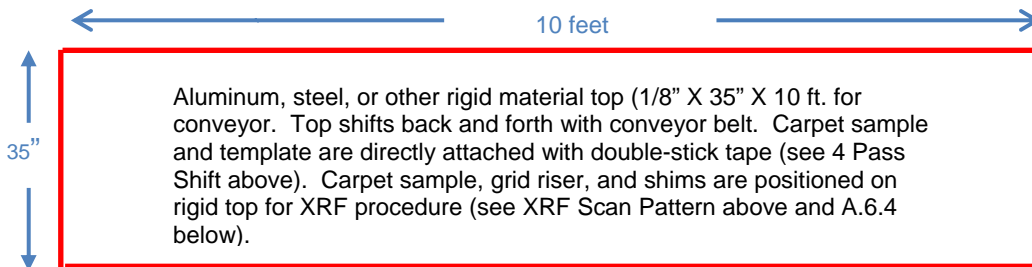
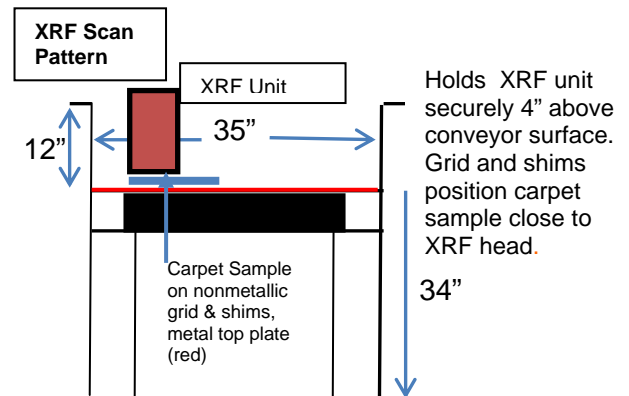
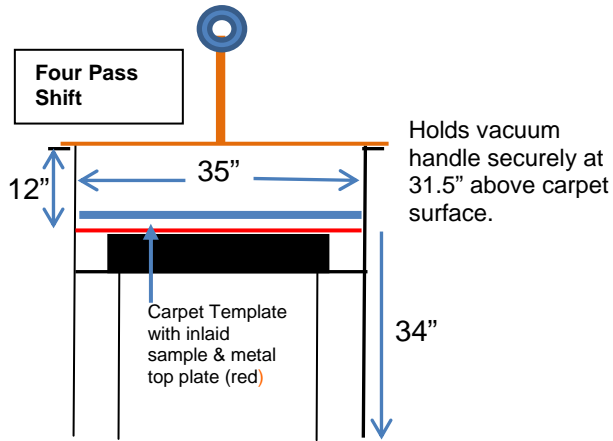
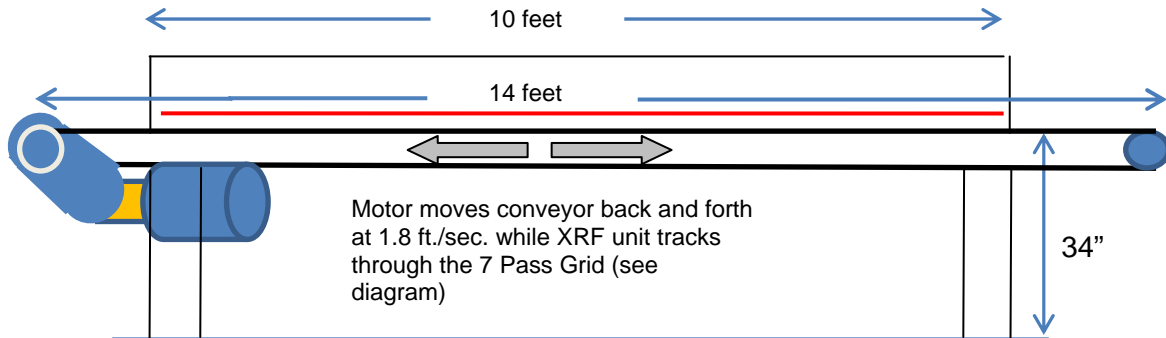
Power Conditioner
Allied Electronics
800-433-5700

- A.6.3 **Reciprocating Conveyor** (or equivalent) 1) to control the speed and path of the test vacuum movements during XRF soil removal from the carpet sample (see 4 Pass Shift in B.12.4) and 2) to control the speed and path of the XRF unit to measure XRF soil removal from the carpet sample (see XRF Scan Pattern in B.12.3).

Note: A specific conveyor with an adequate bed length of 4.3 m (14 ft) and width of 0.915 m (3 ft) has been adapted to be sufficient to the task. The reciprocating conveyor is capable of maintaining specified test speeds between 1 meter (3.3 ft.) per second and 0.55 meters (1.8 feet) per second with automatic forward and reverse direction changes for a specified number of vacuum passes. A 35" X 10 ft. rigid 1/8" aluminum or steel plate is positioned on top of the conveyor and a specific sample carpet size (10 3/8" X 39 1/2") and specific sample carpet template (35" X 10 ft. with an opening centered for the carpet sample to fit into) are temporarily adhered with double-stick tape. Each vacuum pass is started and ended on the carpet template surface to assure the sample is fully exposed to the test vacuum. The conveyor is equipped with brackets or attachment points to hold the test vacuum and XRF test equipment stationary during testing per the diagrams below.

Perpetual Machines
706-226-1883

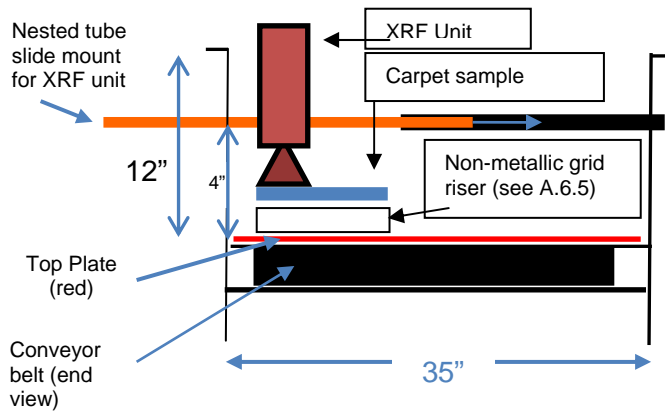
Reciprocating Conveyor and Attachments



A.6.4 **XRF Instrument Stand** to maintain the XRF instrument at a fixed height above the carpet sample mounted on the conveyor (or equivalent), while allowing the XRF unit to traverse from an indexed starting position to 7 additional scan positions (see diagram below and Annex B.12.3)

Instrument Stand for TRACeR XRF (handheld)
 Bruker
 800-466-5323

XRF Instrument Stand Setup for Reciprocating Conveyor



Holds XRF unit securely 4" above conveyor surface. Grid and shims (see A.6.5) are used to position carpet sample close to XRF head per XRF Scan Procedure.

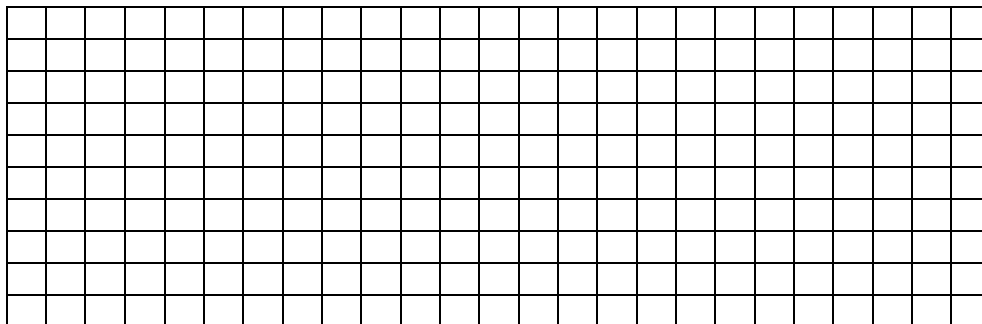
XRF Unit moves left to right from a set starting position making 7 additional indexed scans the length and width of sample as conveyor reciprocates beneath. (see Annex B.12.4)

A.6.5 **Non-Metallic Grid Riser** to hold carpet test sample 6.4 mm (1/4") or more above the metallic conveyor table top plate. Non-metallic shims may be used for leveling and additional height adjustment below the grid riser. See setup in diagram above and grid riser diagram below. See XRF Scan Pattern for sample positioning. (A.12.3)

Rigid Non-Metallic Grid Riser (1/2" thickness with 1/2" X 1/2" openings)

(As in a flush-mounted fluorescent light fixture diffuser)

Lowes Home Center
 (Locally available)



Note: A light diffuser from a flush-mounted fluorescent light fixture (approximately 1/2" thickness and 24" W X 48" L) has been shown to be effective. The standard diffuser is cut-down to match the carpet sample size.

Note: The non-metallic grid riser may be cut to the same dimensions as the carpet samples. If carpet samples do not lay flat against the riser, a weighted non-metallic open picture frame may be used to weight the perimeter of the sample against the riser. The XRF scan pattern should be designed so that use of a frame does not interfere with the scan pattern.

A.6.6 **XRF Sample Cup** for measuring-out the polyamide pellets.

Note: A 100 ml polyethylene beaker is satisfactory.

A.6.7 **Weight Scales – 1)** a scale having a capability of weighing specimens up to 100 grams with an accuracy of 0.01 grams, and 2) a scale having a minimum 2000 gram capacity and an accuracy of 0.1 grams.

Precision Balance
Mettler Toledo
770-476-8500

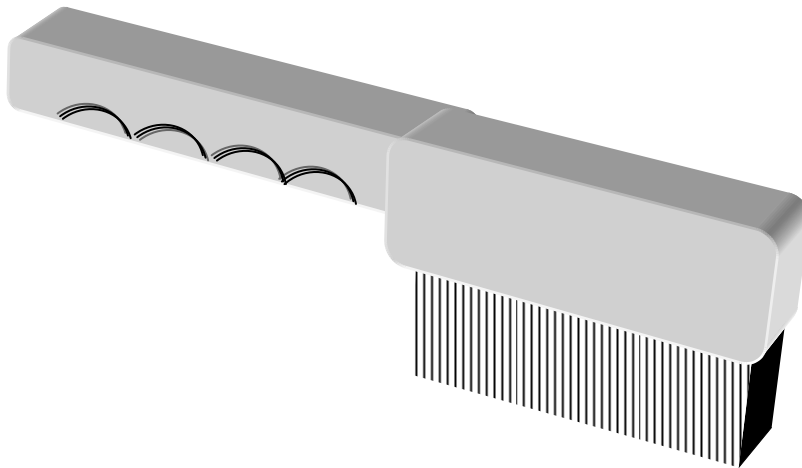
A.6.8 **Carpet Pile Comb** with plastic bristles per diagram below.

Note: The teeth in the representative comb are made of a flexible synthetic polymer with a round crosssection and a diameter of roughly 2 mm. They are 34 mm long and mounted in a plastic base with two parallel rows of teeth spaced 1 cm apart. The individual teeth in each row are spaced 5 mm apart. The molded handle gives the overall comb and handle a total length of approximately 30.5 cm and allows the comb to be used with one hand.

Note: All available apparatus may not be suitable for this application. Apparatus considered for use in this application shall be checked for the ability to groom the tips of the carpet fibers to a uniform, smooth appearance. A Handi Groom™ has been found suitable for this purpose.

Handi Groom
800-334-8418

Comb Pattern – 34 teeth X 2 rows = 68 teeth



A.6.9 **Control Vacuum Cleaner** –Agitator equipped upright vacuum with a minimum SOA/GL Bronze level certification.

CRI SOA/GL Approved Vacuum Cleaner
See CRI website: [http://: www.carpet-rug.org](http://www.carpet-rug.org)

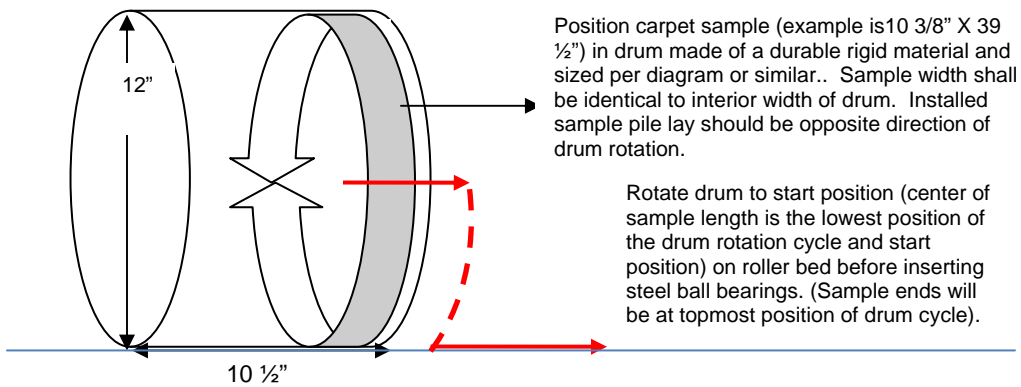
A.6.10 **Drum Tumbler/Ball Mill** for soiling the carpet sample.

Note: Sample size noted herein is designed for use with a 26.7 cm (10 ½”) drum tumbler external width and a 23.6 cm 10 3/8” internal width per the diagram below. Drum tumbler sound enclosures and roller beds will commonly accommodate this and other drum sizes.

Drum Tumbler
Dalton Metal Fabricators
706-226-7194

Sound Enclosure/Roller Bed
US Stoneware
Fax 330-426-1859

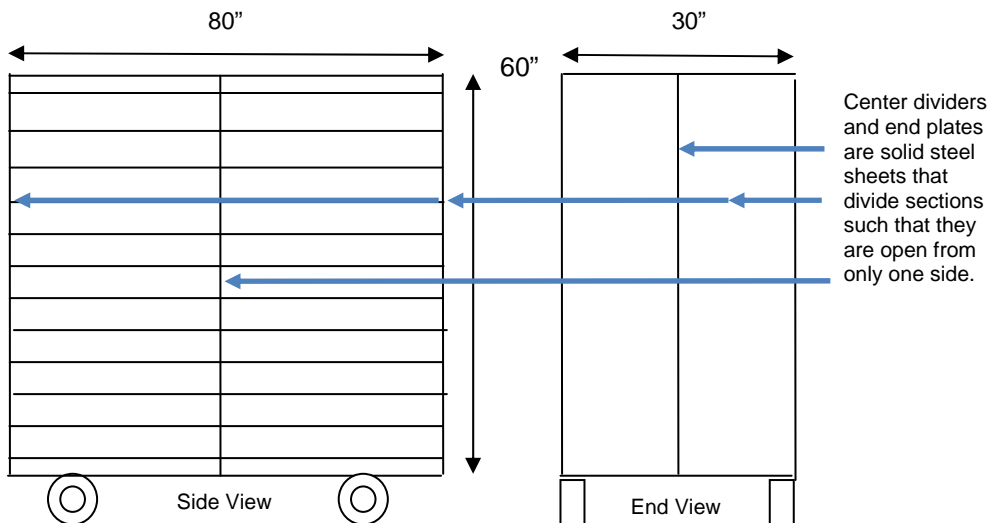
Drum Tumbler and Position of Carpet Test Sample



A.6.11 **Sample Rack (or equivalent)** - horizontal , non-ventilated per diagram below or adequate to keep entire sample flat .

Professional Testing Laboratory
706-226-3283

Sample Rack for 10 3/8" X 39 1/2" Sample Size

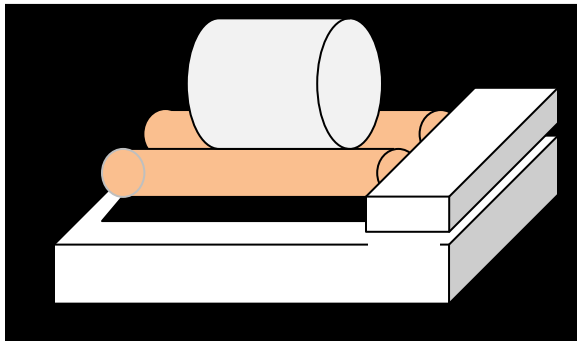


A.6.12 **Jar Mill and Bell Jar** capable of mixing an XRF soil component and nylon 6 pellets in sufficient quantity to meet the sample size requirements. A 263 mm X 1003 mm (10 3/8" X 39 1/2") carpet test sample size is the basis of the diagram below.

Note: The jar mill and bell jar are used in soiling the nylon 6 pellets for each of the five XRF soil types in sufficient quantities. The apparatus is a smaller scale version of the Drum Tumbler/Ball Mill in A.6.10. The example in the diagram uses a one gallon glass jar and appropriately sized roller bed to mix nylon 6 pellets and XRF soil. The Drum Tumbler/Ball Mill in A.6.10 may be used for this purpose if pre-processing larger quantities of soiled nylon pellets.

Jar Mill
US Stoneware
Fax 330-426-1859

Ball Jar (1 gallon glass)
Fisher Scientific
800-766-7000



A.6.13 **Chrome Alloy Ball Bearings** 9.5 mm (.375 diameter) to facilitate soiling of carpet sample in the Drum Tumbler/Ball Mill.

Note: After removing steel ball bearings by hand at the completion of sample soiling, place balls in a sieve and immerse completely in a pan of isoprophyl alcohol to remove XRF soil. Dry steel balls in an air dryer to prevent corrosion.

Industrial Bearing
706-278-8130

A.6.14 **Vacuum Gauge** capable of achieving measurements of 1-120 inches of water column.

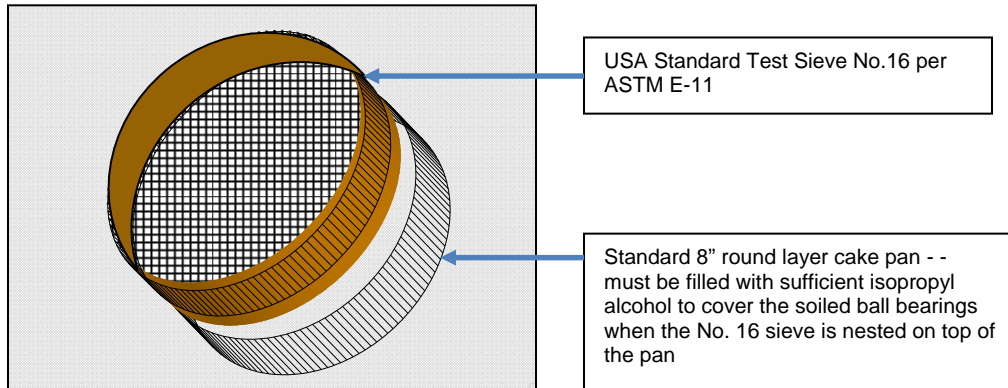
Marsh Bellofram
304-387-1200

A.6.15 **Tachometer** used to calibrate reciprocating conveyor (or equivalent) cycle speed in feet/second.

Grainger Industries
Contact Info

A.6.16 **USA Standard Test Sieve No.16 and Standard 8” Round Layer Cake Pan** used to clean the chrome alloy ball bearings with isoprophyl alcohol after testing.

Note: Use sufficient isopropyl alcohol (see A.7.5) to completely cover the ball bearings when the sieve is nested in the layer cake pan. Dip sieve repeatedly into the alcohol until the XRF soil is removed from the ball bearings. Dry ball bearings in a temperature controlled air circulating oven (see A.6.17)



A.6.17 **Temperature Controlled Air Circulating Oven** sufficient to dry ball bearings after cleaning with isopropyl alcohol to prevent corrosion.

Fisher Scientific
800-555-1212

A.7.0 Reagent and Material Details

A.7.1 **Soiling Compounds** suitable for XRF detection range (*XRF soil*).

| | |
|---------------------|--------------------------------|
| Iron Oxide | Fe ₃ O ₄ |
| Zinc Oxide* | ZnO |
| Strontium Carbonate | SrCO ₃ |
| Yttrium Oxide | Y ₂ O ₃ |
| Zirconium Boride | ZrB ₂ |

*Note: All listed compounds are included in the standard XRF soil composition used for solutions, cleaning equipment and cleaning system testing under CRI SOA/GL Carpet Cleaning Programs. Vacuums are not designed to remove sticky contaminants like Zinc Oxide (ZnO), but sticky contaminants can be present on carpet and can have an adverse affect on overall soil removal. Although the amount of Zinc oxide removed is not included in the calculated percentage for cleaning efficacy of vacuum cleaners its presence in XRF soil is indicative of typical carpet soiling in its effect on the total percent removed by vacuuming of the other four “dry” XRF soil compounds.

XRF Soil Compounds available from:

Alfa Aesar 978-521-6300
CERAC 414-289-9800

A.7.4 **Polyamide Polymer Pellets** used to transfer XRF soil to the test carpet sample mounted in the drum tumbler. (See 12.2.2)

BASF Nylon 6 Resin Pellets
Extrusion Services, Inc.
706-220-0386

A.7.5 **Isopropyl Alcohol** used to clean steel ball bearings after carpet sample soiling is completed..

(Locally available)

Note: An 8" diameter #16 USA Standard Test Sieve is sufficient to assist in cleaning the soiled steel ball bearings. Use an 8" round cake pan to hold enough isopropyl alcohol to cover the soiled ball bearings when the sieve and pan are nested. Dip the sieve and ball bearings repeatedly in alcohol until clean, then dry the steel ball bearings in a temperature controlled air circulating oven.

See diagram in A.6.16

A.9.0 Sampling, Test Specimens, and Test Units

A.9.2.1 **Carpet Samples and Templates** per diagram below or equivalent.

See diagram in A.7.3

Note: Carpet sample and template sizes are expressed in width X (by) length and orientation to the parent carpet roll is important. Width corresponds to manufactured carpet roll width (usually 12 ft.). Length corresponds to manufactured carpet roll length (highly variable) Carpet sample and template cutting accuracy can affect pile lay orientation during testing and can affect multiple sample comparisons if samples are not cut identically

Note: Test carpet sample size may be varied to accommodate limitations imposed by other similar test apparatus. Care must be taken to adjust all other factors dependent on the test carpet sample size, including all apparatus, reagents and materials, procedures, and XRF testing parameters.

A.10.0 Data Collection Software and XRF Unit Setup and Verification

A.10.1 **XRF Verification Software** – the XRF unit used has a test “puck” available to use in verifying that the calibration of the unit has not been compromised. Use the test puck to verify operating conditions before each day’s XRF scans at minimum.

Bruker
800-466-5323

XRF Setup and Calibrations

It is necessary to create a calibration curve for the concentration of each compound applied to the pellets. This in turn will be used to create the carpet calibration. The pellet concentrations are used to determine the amount of compound transferred during soiling.

Calibration of compound concentrations on the pellets: For each compound, mix a batch of compound coated nylon pellets at a minimum of three different concentrations, for example 0, 3, 6, and 9 grams per 1000 grams of pellets. Scan the pellets by placing the pellets in an XRF sample cup and sealing it with Mylar or other suitable window material.

Record the sample cup type and window material as future scans will need to be done the same way. Pellet scans are best done with the XRF unit in its stand. Each sample should be scanned a minimum of five times rearranging the pellets between scans. Create the pellet calibrations for each compound and configure the Real Time Analysis (RTA) program for the pellets.

Calibration of concentration of compounds on carpet samples: For each compound and each concentration of pellets use 50 grams of compound coated pellets to soil a calibration carpet sample. Weights other than 50g may be used especially if 6g/1000g pellets is too high a concentration to allow for sufficient adhesion of the compound to the pellets for consistent soiling. After soiling each calibration sample collect as many of the used pellets as reasonably possible and scan them using the RTA computer program that was set up for the pellets. Rearrange the pellets in the sample cup during tests. Care should be taken to minimize the transfer of compound to surfaces other than the carpet.

After scanning the used pellets with the RTA program to measure the final concentration left on the pellets, calculate the concentration of the compound on the calibration sample by first determining the grams per 1000 grams pellets difference between the starting concentration and the after-soiling concentration on the pellets. As 50 grams of pellets were used, if one multiplies the difference in grams per 1000 grams pellets by $(50 \text{ grams pellets}) / (1000 \text{ grams pellets})$ or $1/20$ the result should be a satisfactory estimate of the grams of compound on the carpet. Dividing the grams on the carpet by the area of the sample will determine the concentration of the compound on the calibration sample.

A.12.0 Procedure

A.12.3 XRF Scan Procedure

The proper height of the XRF unit above the carpet sample must be determined and maintained before scanning the calibration samples. After brushing a sample carpet with a carpet pile comb to erect the piles and to remove matting, place the carpet sample on the plastic riser under the XRF unit mounted in its stand. Using non-metallic shims, raise the carpet sample and riser up until it almost touches the nose piece of the XRF unit. It is essential that pile orientation and its distance relative to the sensor be maintained. Using PXRF software or its equivalent, collect a spectrum. At this point it is not critical that the carpet be moving on the conveyor. Examine the spectrum for peak intensities of the elements found in the backing.

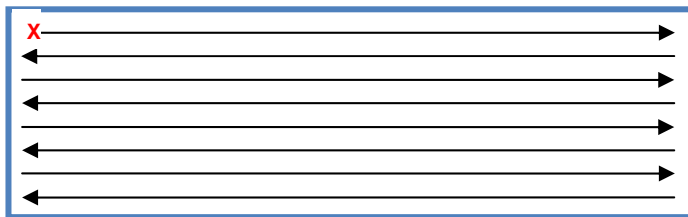
Note: Sample carpet supplier normally pretests sample carpet for this test method to eliminate XRF soil elements from the test carpet backing.

The vertical scan objective is to create a focal length that measures as much of the fiber column of a particular sample type above the backing as possible. If there are no XRF soil elements that can be detected in the backing of an unsoiled sample, the bottom of the fiber column above the sample will be identified as the point at which the XRF elements are detected in the expected range for an XRF soiled sample. Record the height of the carpet position relative to the fixed XRF head height for scanning setup of all samples of that sample type.

If an XRF peak from an element in the backing is detected in an unsoiled sample, lower the carpet and repeat the scan until the XRF elemental peak intensity is negligible relative to the intensity of peaks from the XRF compounds detected in an XRF soiled sample. Now that the proper height has been determined, scan all the calibration samples and create a calibration curve for each element and import to a second copy of the RTA program.

XRF Scan Pattern on the Carpet Sample

The objective of the XRF Scan pattern is to cover as much of the surface area of the carpet sample as possible within the three minute XRF cycle at 1.8 feet (.055 meters) per second. For the roughly 10" X 40" ($\approx 25.4 \text{ cm} \times 101.6 \text{ cm}$) sample presented herein, the XRF scan covers approximately 120 in² (775 cm²) of the approximately 400 in² (2580 cm²) total when 8 identical passes across the sample area alternating from left to right then right to left are scanned, and separated on 1.0 inch (2.54 cm) centers top to bottom as depicted below.

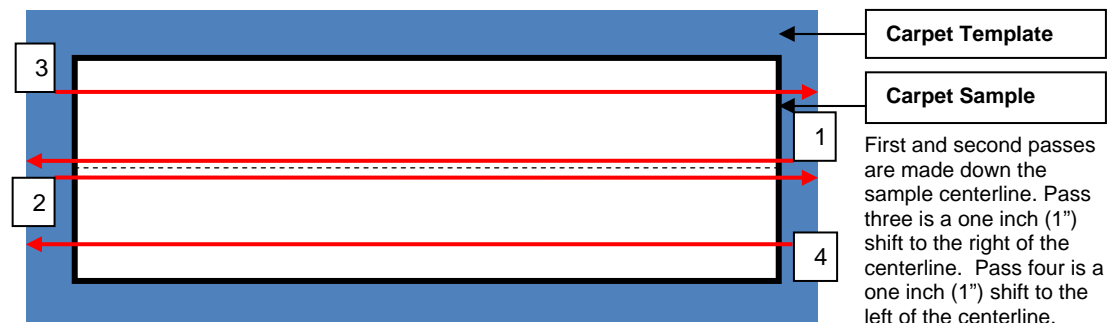


Scan pattern is achieved through L to R motion of conveyor under the XRF head and top to bottom movement of the XRF unit from the top start position X to 7 additional scan paths equidistant to the bottom of the carpet sample.

Note: No carpet template is used when the carpet sample is mounted on the grid riser for the XRF Scan Procedure. The width of the XRF scan beam, is approximately .375 inch ($\approx .95 \text{ cm}$). A non-metallic weighted open picture frame may be fabricated to closely fit over the sample size to hold the carpet sample flat if any sample curl is observed. The XRF scan pattern should be designed so that the frame does not interfere with the automated scan sequence.

A.12.4 Vacuum Cleaning Procedure

Four Pass Shift Vacuum Diagram



First and second passes are made down the sample centerline. Pass three is a one inch (1") shift to the right of the centerline. Pass four is a one inch (1") shift to the left of the centerline.

A.13.0 Calculation or Interpretation of Results

A.13.1 Calculation and Interpretation of XRF Results.

Discussion of XRF Precision

Using an XRF scan time of 3 minutes, an un-cleaned soiled sample produces XRF elemental peak intensities of 1700, 2500, 3000, 6000, and 8000 counts for Fe, ZN, Sr, Y, and Zr respectively on the cut pile carpet. This corresponds to a respective peak intensity precision of 4.9%, 4.0%, 3.7%, 2.6%, and 2.2%. After averaging over 3 scans, the respective precision will drop to 2.8%, 2.3%, 2.1%, 1.5%, and 1.3%. If these peak intensities are in the range of the above intensities, then the variation from the inherent statistical measure of XRF will not be the dominate source of variation on this test procedure. The dominant source of variation is believed to be caused by several factors relating to maintaining the relative distance between the soiled carpet fiber and the XRF unit. The XRF peak intensity drops as the inverse square of the distance from the XRF source element to the detector; hence, it is important that the samples be combed before they are scanned to present a constant position.