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CRI TM 120 - Standard Laboratory Test Procedure For Evaluating Cleaning Effectiveness of Commercial Carpet Cleaning Chemicals, Cleaning Equipment or Complete Cleaning Systems

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Standard Test Method for Using X-Ray Fluorescence Techniques for Evaluating Cleaning Effectiveness of Commercial Carpet Cleaning Chemicals, Cleaning Equipment, or Complete Cleaning Systems

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. The test method may be used to evaluate the effectiveness of a cleaning event, an event in a process of several events, or an entire cleaning process.

1.2 *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 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 D 6540 Standard Test Method for Accelerated Soiling of Pile Yarn Floor Covering

3. Terminology

3.1 *Definitions:*

3.1.1 *cleaning ability, wet, n* – the potential of a cleaning event or complete cleaning process to remove soil from a surface.

3.1.2 *Cleaning system, n* - A functional combination of one or more pieces of cleaning equipment, used with or without a chemical or additive, and a defined set of instructions describing how the equipment and the chemical or additive shall be employed.

3.1.3 *model, n* – the designation of a group of cleaning units having identical mechanical and electrical design with only cosmetic or nonfunctional differences.

3.1.4 *population, n* – the total of all units of a particular model cleaning unit being tested.

3.1.5 *sample, n* – a group of cleaning units taken from a large collection of cleaning units of one particular model which serves to provide information that may be used as a basis for making a decision concerning the larger collection.

3.1.6 *test run, n* – the definitive procedure that produces a singular measured result.

3.1.7 *unit, n* – a single cleaning unit of the model being tested.

3.1.8 *wet stroke, n* – a single pass across the test carpet in one direction with the application of the cleaning solution.

3.1.9 *dry stroke, n* – a single pass across the test carpet in one direction without the application of any cleaning solution.

3.1.10 *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

3.1.11 *X-Ray tube or tube, n* - A device that produces radiation in the X-ray energy range by accelerating electrons from a cathode to an anode.

3.1.12 *tube settings, n* – The voltage and current at which the tube is operated. The voltage is the potential difference between the cathode and anode and the current is the measure of the electrons flowing between the cathode and anode.

3.1.13 *harding (the tube or source), v* – The practice of filtering out lower energy photons coming from the tube, frequently done by placing a metal such as aluminum between the tube and the sample.

3.1.14 *instrument geometry, n* - The geometrical positions and or angles between the X-ray source, sample, and detector.

3.1.15 *sample volume, n* – The volume created by the intersection of the source X-ray beam and the field of view of the detector.

4. Summary of Test Method

4.1 This test method uses a standard set of compounds containing suitable elements for XRF detection. These compounds are called out 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 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 specified cleaning event or 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 cleaning performance of various cleaning chemicals. The generated data will be used to compare the performance between various cleaning chemicals. The generated data may be useful to chemical manufacturers in product development. The generated data will be used by the carpet industry to evaluate and approve cleaning chemicals.

5.2 The test method will be used to generate data that can quantify the carpet cleaning performance of various cleaning equipment. The generated data will be used to compare the performance between various cleaning equipment. The generated data may be useful to equipment manufacturers in product development. The generated data will be used by the carpet industry to evaluate and approve cleaning equipment.

5.3 The test method will be used to generate data that can quantify the carpet cleaning performance of various cleaning systems. The generated data will be used to compare the performance between various cleaning systems. The generated data may be useful to cleaning professionals in cleaning system development. The generated data will be used by the carpet industry to evaluate and approve cleaning systems.

5.4 This test method is not based on home cleaning tests. There is no established correlation between field and laboratory results.

6. Apparatus

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.

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 ± 1 %, and rated frequency having a wave form that is essentially sinusoidal with 3 % maximum harmonic distortion for the duration of the test.

6.3 Conveyor with minimum bed length of 2.7 m (9 ft) and width of 0.9 m (3 ft) equipped with a rigid metal plate affixed to the conveyor, which a carpet template and carpet test specimen may be attached. These minimum dimensions are satisfactory for the suggested sample size. Variations in sample size may dictate variations in the size of the conveyor bed length. The conveyor must be capable of maintaining specified test speeds between 1 meter per second and 0.6 meters per minute in both forward and reverse directions. The conveyor must be equipped with brackets to hold the test equipment stationary during testing, in a manner that approximates the loading of the equipment in normal operation.

6.4 XRF instrument stand to maintain the XRF instrument at a fixed height above the carpet sample when the carpet sample is mounted in position on the conveyor.

6.5 Non-metallic grid riser to hold carpet test sample 6.4 mm (1/4 inch) or more above the metallic conveyor table top. Shims may be used to give additional height adjustment.

6.6 XRF sample cup. A 100 ml polyethylene beaker is satisfactory.

6.7 Weighing scales having a capability of weighing specimens up to 100 grams to an accuracy of 0.01 grams, and 2000 grams to an accuracy of 0.1 grams.

6.8 Carpet pile brush with plastic bristles. 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.
NOTE: A Handi Groom has been found suitable for this purpose.

6.9 Control vacuum cleaner – Straight air type, with no rotating brush and or beating bar. When the carpet tool is replaced by a 5/8 inch orifice, the vacuum suction of the cleaner hose shall be between 35 and 45 inches of water (8.7 and 11.2 kPa).

7. Reagents and Materials

7.1 Soiling compounds suitable for XRF detection range.

Iron Oxide	Fe_3O_4
Zinc Oxide	ZnO
Strontium Carbonate	$SrCO_3$
Yttrium Oxide	Y_2O_3
Zirconium Boride	ZrB_2

Table 1 – Suitable Soiling Compounds

7.2 Control test carpet

CONTROL CUT PILE		CONTROL LOOP PILE	
Weight	25 oz/sq yd, +/- 7%	**Weight**	30 oz/sq yd, +/- 7%
Color	*	**Color**	*
Pile Height	0.470 inch	**Pile Height**	0.123 inch
Gauge	1/8	**Gauge**	1/8
Stitches per inch	8	**Stitches per inch**	8
Yarn	Nylon	**Yarn**	SD Nylon
Fluorochemical	No	**Fluorochemical**	No

Table 2 – Test Carpet Specifications

* Color selection affects XRF detection only when dyes containing elements in the soiling compound have been used to color the yarn. This situation is easily detected by strong peaks in the XRF data. The test method cannot be run on test samples with such dyes.

8. Hazards

8.1 The XRF unit as described in this standard typically operates at less than 50 kV and 15 μA, 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 can provide the necessary training and guidance for complying with applicable regulations.

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

9. Sampling, Test Specimens, and Test Units

9.1 Due to the nature of the equipment intended to be evaluated by this test method, it is not practical to require a population sample greater than one unit. The resulting report will apply only to those samples tested and will not necessarily be indicative of apparently identical or similar products. The number of units tested shall be reported and the report shall state that the testing has not accounted for variations within models, and that this variation may be significant.

9.2 A minimum of three (3) carpet samples shall be tested for each unit. Prepare carpet samples in accordance with ASTM D 6540, except as noted here:

The carpet sample size may be 125 mm x 205 mm (5” x 8”) as specified in ASTM D6540, or a larger size to accommodate larger equipment. The sample size will be dictated by the size of the carpet soiling equipment, and the size of the sample needed to satisfactorily represent the cleaning processes being evaluated. In the absence of any restrictions, the suggested sample size is 254 mm x 1016 mm (10” x 40 “).

If the size is varied, the amount of soiled polyamide polymer pellets called out in section 12.2.4, and the amount of chrome alloy steel ball bearings called out in section 12.2.3, shall be varied proportionately. The soiling media shall be the five soiling compounds listed in Section 7.1.

10. Data Collection Software Setup and Calibration

10.1 The supporting computer software will collect a spectrum from the XRF unit and save the spectrum as a function of Energy-verse-Counts. For these saved values to be useful, the software must be calibrated for the instrument used at the time the data is collected. There is a calibration of the XRF unit, and a second calibration of the measurement system. Once the system is calibrated it can be used by any trained user. A description of the calibration procedures is given in Annex A1.

10.2 The calibration of the XRF unit shall be checked before each series of tests with a control contaminate sample.

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.

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 solution temperature for cleaning equipment employing solution without onboard heating systems shall be held at 21 ± 3 °C (70 ± 5 °F). The solution temperature for cleaning equipment employing solution with onboard heating systems shall be operated with the heating system employed at the intended operating temperatures as specified in manufacturer’s instructions.

11.4 All cleaning equipment with solution dispensing systems shall have the solution system operated for at least one minute prior to the test to fill all hoses and warm up all heating system components. After the warm up period, the solution flow rate shall be measured and reported.

11.5 If the solution tank weight, or chemical receptacle weight, affects the performance of the cleaning equipment, then the test shall be performed with the tank or receptacle full.

11.6 If the recovery tank weight, or other receptacle weight, affects the performance of the cleaning equipment, then the test shall be performed with the tank or receptacle empty.

11.7 The dilution ratio and application rate of any chemical used shall be held constant during the test and shall be reported.

12. Procedure

12.1 Test Carpet Preparation

12.1.1 If sample storage follows good laboratory practices, the test carpets can be prepared up to 72 hours in advance of testing.

12.1.2 Vacuum each test specimen with the control vacuum cleaner using four passes at a rate of 0.55 meters 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 stainless drum per ASTM D 6540.

12.2.2 Prepare compound coated nylon pellets using each of the five soiling compounds listed in Section 7.1, following ASTM D 6540. There will be five different sets of pellets, one for each of the five soiling compounds.

12.2.3 Weigh out the chrome alloy steel balls and place them in a single layer on the carpet, following ASTM D6540. The amount of balls used shall be adjusted as required to achieve the desired soiling level when the size of the test carpet varies from that called out in the standard. For the suggested sample size, 1193 g ± 2 g has been shown to be satisfactory.

12.2.4 Weigh out 50 g ± 0.01 g of each of the five compound coated pellets, following ASTM D6540. This gives a total weight of coated pellets of 250 g, but this amount shall be adjusted as required to achieve the desired soiling level when the size of the test carpet varies from that called out in the standard. For the suggested sample size, 50 g ± 0.01 g of pellets, of each of the five compounds, has been shown to be satisfactory.

12.2.5 Spread the compound coated pellets over the bearings such that they are uniformly distributed along the width of the carpet sample, in the order they are listed in Table 1.

12.2.6 Seal the drum and rotate per ASTM D 6540 for 30 minutes.

12.2.7 Inspect the sample and remove the steel balls and all of the used compound coated pellets.

12.2.8 Comb the sample with a carpet pile brush 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 brushed or combed before they are scanned to maintain a constant position. Short loop pile carpets may not need to be bushed; however, it is essential that pile orientation and its distance relative to the sensor be maintained.

12.3 XRF Scan Procedure

12.3.1 The XRF scan shall be performed before and after the cleaning of the test carpet. For a combination of cleaning steps being evaluated in series, the XRF scan should be repeated as necessary to establish a starting concentration and a final concentration for each step of the process. The scan shall be repeated at each required step following the procedure described in the remainder of this clause.

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 and so that the unit does not have significant sensitivity to the elements in the carpet backing. Place the carpet sample on its nonmetallic riser on the conveyor and move it to a point to start a scan.

12.3.3 The objective is to scan the largest practical area, which is representative of the soiling and/or cleaning. If the time to take the scan is three minutes, then the variation from the inherent statistical nature of XRF will not be the dominate source of variation in the test procedure. Therefore, the scan pattern shall be defined to cover the largest practical area in three minutes.

Depending on the sample size, it will be necessary to balance the speed of the XRF conveyor against the overlap of the scan pattern. For the suggested sample size of 254 mm x 1016 mm (10” x 40”), five evenly spaced, 36-second passes, with a scan speed of 1.3 meters per minute, has been shown to be satisfactory.

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%.

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 is starting concentrations. Additional information on method precision is given in Appendix X1.1.

Compound	Residential Cut Pile	Loop Pile	Commercial Cut Pile
Fe_3O_4	0.62 to 1.02 grams/sq meter	0.91 to 1.16 grams/sq meter	0.39 to 0.64 grams/sq meter
ZnO	0.37 to 0.62 grams/sq meter	0.47 to 0.78 grams/sq meter	0.83 to 1.18 grams/sq meter
$SrCO_3$	0.47 to 0.78 grams/sq meter	0.93 to 1.55 grams/sq meter	0.62 to 0.93 grams/sq meter
Y_2O_3	0.56 to 0.85 grams/sq meter	0.73 to 0.88 grams/sq meter	1.19 to 1.35 grams/sq meter
ZrB_2	0.64 to 1.09 grams/sq meter	1.08 to 1.55 grams/sq meter	1.49 to 1.88 grams/sq meter

Table 3 Recommended Starting Concentrations

12.4 Cleaning Procedure

12.4.1 Place the soiled carpet sample in an appropriate template of the same style of carpet so that a larger carpet sample is presented to the cleaning equipment, and the cleaning process does not encounter a transition at any edge of the sample. Mount the cleaning unit in place and clean the sample following the manufacturer directions for any adjustable settings on the cleaning equipment. All adjustable settings shall be recorded and reported.

12.4.2 In general, the cleaning process will include a vacuuming step, followed by an extraction step, although cleaning processes that involve other steps can be evaluated by this test method.

12.4.3 For cleaning equipment that is being evaluated after vacuuming only, vacuum each test specimen with the control vacuum cleaner using 4 strokes, at 0.55 meter per second. Repeat the XRF scan after vacuuming to establish the starting concentrations of compounds for the extraction step of the cleaning process.

12.4.4 If the vacuum step of the cleaning process is part of a combination of cleaning steps being evaluated in series, then vacuum using the test vacuum. Unless otherwise required by the instructions or limitations for the equipment, or directed by the instructions for the cleaning system being evaluated, vacuuming shall involve 4 passes at a rate of 0.55 meters per second.

All variations shall be recorded and reported. Repeat the XRF scan after vacuuming to establish the ending concentrations for compounds for the vacuuming step, and the starting concentrations for the next step of the cleaning process.

12.4.5 For extraction type equipment, two wet passes followed by two dry passes at 0.3 meters per second should be used, with no intentional dwell time. This shall be varied if required by the instructions for limitations for the equipment, or directed by the instructions for the cleaning system being evaluated. All variations shall be recorded and reported.

The final pass of the vacuum shoe or wand shall be with the lay of the carpet pile. Previous pass directions shall be as dictated by the details of the equipment. If the operation of equipment sets no limitations, the passes shall be made against the pile, with the pile, against the pile, with the pile. The stroke details shall be recorded and reported.

12.4.6 After cleaning, the carpet samples are to be stored horizontally on a non-ventilated drying rack to dry at standard conditions. After a minimum of 16 hours, and not more than 72 hours, repeat the XRF scan to establish the ending concentrations for compounds for this cleaning step, and the starting concentrations for any additional step of the cleaning process.

13. Calculation or Interpretation of Results

13.1 If more than one cleaning step is being evaluated, each step, or combination of steps, in the process shall be treated separately in the averaging and calculations.

13.2 Three soiled carpet samples are used for each test and the results are averaged. The reported result is the cumulative average percent removed of each compound. 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 average percent removed is an average across compounds.

14. Report

14.1 Starting compound concentration for each cleaning step being evaluated, expressed in mass per unit area (grams per square meter) for each compound.

14.2 Amount of compound remaining after cleaning for each cleaning step being evaluated, expressed in mass per unit area (grams per square meter) for each compound.

14.3 Calculated percentage removed for each compound for each cleaning step being evaluated, and the average for all compounds combined.

14.4 Number of carpet samples tested per unit and the number of units tested per population sample, and a statement that the testing has not accounted for variations within models, and that this variation may be significant.

14.5 Carpet sample details, including carpet style, sample size amount of each compound added to the carpet, and any variation in carpet detail from the information given in Table 2.

14.6 Cleaning process details. Details of the steps included in the cleaning process. Details of the equipment, chemicals used, concentrations, number of passes, speed of each pass, process, equipment settings, etc.

15. Precision and Bias

15.1 Precision – No interlaboratory test have been performed; therefore, no precision statements regarding the repeatability and reproducibility of this test methods are available at this time.

15.2 Bias – No justifiable statement can be made on the accuracy of this test method, since there true value of the property cannot be established by an acceptable referee method.

16. Keywords

16.1 Vacuum; Extractor; Cleaning

ANNEX
(Mandatory Information)

A1. Equipment Setup and XRF Calibrations

A1.1 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.

A1.1.1 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.

A1.1.2 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.

A1.1.3 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 grams pellets)/(1000 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.

A1.1.4 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 brush 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 shims, raise the carpet on the riser up until it almost touches the noise 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.

A1.1.5 If there are no elements that can be detected that are known to be in the backing, record the height of the carpet position. If a XRF peak from an element in the backing is detected lower the carpet and repeat the scan. Repeat the process of scanning and checking for elemental XRF peaks from the backing until the peak intensity is negligible relative to the intensity of peaks from the compounds used to soil a 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.

APPENDIX
(Nonmandatory Information)

X1.

X1.1 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 maintain a constant position.

REFERENCES

(1)