

Electrostatic microprobe for determining charge domains on surfaces

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(Received 12 June 2015; accepted 22 October 2015; published online 11 November 2015)

An electrostatic microprobe was developed to measure charge on wipes and various test surfaces. The device is constructed on an optical microscope platform utilizing a computer controlled XY stage. Test surfaces can be optically imaged to identify microscopic features that can be correlated to the measured charge domain maps. The ultimate goal is to quantify charge on wipe cloths to determine the influence of electrostatic forces on wipe sampling efficiency. We found that certain wipe materials do not extensively charge while others accumulate charge by making contact with other surfaces (through the triboelectric effect). Charge domains are found to be nonuniform. [http://dx.doi.org/10.1063/1.4935168]

INTRODUCTION

We have developed an electrostatic microprobe (ESMP) on an optical microscope platform that permits collection of optical images and electrostatic potential maps on the same test surface. The motivation for this development relates to forensic wipe sampling. Particles are collected by a contact wiping (or swiping) process that entails rubbing a cloth or wipe across a surface. One possible mechanism for particle collection is by electrostatic attraction between the wipe surface and the particles. Some wipes can be charged by the rubbing process through the triboelectric phenomena. Triboelectric charging occurs when two surfaces touch or are rubbed against each other. To test the hypothesis that charged wipes have some effect on particle collection, we needed a method to gauge the level of charge on these nonuniform surfaces and the spatial distribution found on a typical wipe when it is used in the wiping mode. We demonstrate the utility of the instrument for determining charge distributions on a number of wipe materials and particle sets. Many of the wipe materials and the surfaces that are swiped are made of polymers. There has been considerable interest in electrical charge generated on polymer surfaces by triboelectric methods. A recent review by Williams (2012a) gives an excellent overview of the topic. Contact electrification is a complex process often described as electron transfer, but also potentially including bond cleavage, chemical changes and in some cases material transfer from each surface (Williams, 2012b and Burgo et al., 2012). Chemical specific measurements by Raman and xray photoelectron spectroscopy support the material transfer concept. Especially interesting is the discovery that polymer surfaces charge in a way such that inhomogeneous domains that look much like a checkerboard of different polarities because the charge is unable to rapidly migrate or diffuse (Baytekin et al., 2011). Charge was found to be in higher surface densities than previously believed with charges being separated by only a few 10's of nm on the surface.

Other methods for charge mapping include atomic force microscopy/electrostatic force microscopy (AFM/EFM) and the Kelvin probe. The utility of the EFM is limited to very small (nano-scale) spatial extent measurements. In fact previous researchers (Mazumder et al., 2001) failed to map charge on collections of 1 μ m-50 μ m particles or macro scale objects. The Kelvin probe, a macroscopic device used to measure work functions on conductors (metals) (Baiki et al., 1988 and Baiki and Estrup, 1998) may have slightly finer spatial resolution than our probe but is over 10 times more expensive and may not be as useful to map large rough surface areas. A Kelvin probe and an atomic force microprobe were used to study the effects of humidity on charge retention and charge patterns on polyethylene polymer surfaces with spatial resolution of $5 \text{ mm} \times 5 \text{ mm}$ (Burgo *et al.*, 2012). The Kelvin probe has no optical imaging capabilities. A homemade electrostatic probe (based on a capacitance Qprobe design) was developed to measure charge transferred by desorption electrospray ionization mass spectrometry (Foord, 1969; Takuma et al., 1998; and Gao et al., 2010). An ultrahigh impedance based probe sensor has been made that has a reported spatial resolution of approximately 5 μ m and the capability to charge map a single finger print (Watson et al., 2011). This sensor is not based on the same principle as the NIST probe. The NIST design offers the capability to measure the electrical potential at an array of distances, albeit sacrificing some resolution. Many wipe materials have uneven, textured surfaces which do not lend themselves to contact or close probe to surface measurement. The NIST electrostatic probe provides a method to scan or survey wipes or other test surfaces with moderate spatial resolution and at the same time obtain an optical micrograph of the test surface. The capability to obtain a real optical image of the test object to correlate with the electrical potential values from the identical field-by-field location is believed to be unique to this instrument and is often very important for characterizing the sample.

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INSTRUMENT DESCRIPTION

A simple, relatively inexpensive ESMP is constructed by coupling an isoprobe electrostatic voltmeter (model 244A made by Monroe Electronics, Lyndonville, NY) (Instrument Manual) and an optical microscope equipped with an automated sample stage (PRIOR Scientific, Rockland, MA). These are commercial off-the-shelf components. The stage control and voltmeter values are controlled and read by Image-Pro software (Media Cybernetics, Inc., Rockville, MD). A diagram of the microprobe is shown in Figure 1. The sample is mounted by suspending it across monofilament polymer threads or by affixing the sample to a glass microscope slide and mounting the slide in the normal manner on the microscope stage. The electrostatic microprobe sensor is located below the sample surface (approximately 1 mm away) with the sensor facing upward (Stranick, 2009). The sensor opening is circular and 510 μ m in diameter as shown in Figure 1. The automated stage translates the sample across and over the stationary sensor; the range of translation, the sample boundaries, and the area interrogated are controlled by the analyst through the automated stage. The sensor works by setting up a condition of parallel plate capacitance. The electrostatic voltmeter seeks to establish a field free condition by equalizing the charge on the probe (isoprobe) to match the charge on the surface under investigation. The applied voltage to the probe to accomplish this condition equals the potential on the surface relative to electrical ground. Consequently, the probe voltage becomes the parameter used to quantify the static charge on the sample surface. This capacitance probe's working principle is different from the capacitance probes mentioned in the Introduction; these are Q-probes. The meter is interfaced and read through a Stage Pro macro



FIG. 1. Schematic of the electrostatic microprobe. Sample mounted on an automated microscope stage. An optical microscope fitted with digital camera can view the sample from above and the electrical potential can be determined from the electrostatic (ES) probe located below the sample. (Media Cybernetics, Inc., Rockville, MD) such that for each location on the sample, the voltage is divided by 1000 and the XY positions are recorded. Readings at each location can be accumulated and averaged up to 1000 readings if so desired; averaging reduces the variance in the sensor. The ESMP has a voltage range of -3400 V to +3400 V. Since the probe is at elevated DC potentials, the probe is electrically insulated and held in place by an XYZ stage mount made of Teflon. The high voltage does not pose as a health risk being a low current DC source. The probe mount ensures that the sensor is held at a constant, known distance from a flat sample surface. The optical axis through the microscope objective is aligned with the electrostatic probe sensor by viewing the sensor opening in the microscope field of view and adjusting the XY position of the electrostatic sensor. The optical microscope is used to image the same spatial domain of the surface where relative charge is determined. The sensed area on the sample for the 510 μ m probe is a circular spot approximately 500 μ m-1000 μ m in diameter, increasing in diameter with increasing sample surface to sensor distance. There are other Monroe sensors that can be used with larger area to cover more area with less spatial resolution. Also, sensors with smaller cross section could be made by affixing a metallic cover with a smaller orifice diameter. For purposes of description, we will focus on the smallest probe (510 μ m).

An example of optical and electrostatic charge imagings is presented in Figure 2 for a small Teflon rectangle mounted on a glass slide and rubbed by a cloth. The Teflon negatively charges upon contact with most surfaces and the charge is retained by the polymer, the glass being a good insulator. Much as the optical image is composed of individual images (fieldof-views visible in the image) tiled together by the software, the electric potential (voltage) is determined from each fieldof-view. The potential reading represents the overlap of the sensor area and the field of view presented to the sensor by the XY stage. Electrical potential readings are stitched together to form a color coded image using SigmaPlot software (San Jose, CA; http://www.sigmaplot.com). Since the optical image is from the back side of the Teflon square and electrical potential representation is from the opposite side, the potential image directly overlays the optical image. The image shows that the charge distribution on the Teflon is not uniform but



FIG. 2. Optical image on the left of a 3 mm×4 mm Teflon BYTAC square formed by tiling individual optical images (small rectangles). The right frame shows the corresponding electrical potential representation for the Teflon square formed by combining individual voltage measurements from each optical field-of-view. The electrical potentials are represented by the color scale.



FIG. 3. Left image is an electrical potential map of a copper plate held at 500 V DC by a laboratory high voltage power supply. Right image shows the plot of the voltage applied to the copper strip vs the ESMP mean measured voltage. The uncertainties are presented in Table I.

concentrated in the lower edge (intense blue and black colors corresponding to -150 V to -300 V).

MICROPROBE CHARACTERIZATION

The ESMP was characterized to verify the sensor accuracy, precision, sensitivity, and the spatial resolution. The sensor accuracy was obtained by determining the sensor response to a copper plate held at a known voltage. A 5 cm \times 20 cm copper strip supported by the polymer strings was attached to a variable high voltage power supply. A typical scan is shown as a 3-dimensional plot in Figure 3 for the case that the copper strip was held at +500 V. Similar scans were obtained at -500 V and at other voltages. The measured mean (501 V) had a relative standard deviation (RSD) for this scan (n = 385, over area of 14 mm \times 16 mm) of 1.7%. Each stage field of view was 868 μ m × 651 μ m and the ES sensor collected information from a spot of approximately 510 μ m in diameter from the central portion of each field. The voltage readings are not averaged values, but single individual voltage readings for each position on the map. A plot showing the correlation between the applied voltage to copper strip and the measured voltage is shown in Figure 3. Table I contains the data. It is clear that the RSD for low voltages was higher when the power supply used. The values obtained for the power supply at 0 V had large variation. To verify that the variation was in the power supply, we used a 1.5 V D-cell battery as the voltage source and found the RSD was reduced to 0.5%as shown in Table I. The variance found for measurements with the ES probe stationary (measuring one location on the copper surface) and with the ES probe scanning were the same,

TABLE I. Data used for the plot appearing in Figure 3.

Standard

deviation (V)

RSD

(%)

Mean measured

voltage (V)

suggesting that the major source of variance in the voltage determination arises from fluctuations in the power supply.

As mentioned, we treat the sensor as one half of a parallel plate capacitor (see Figure 1). The spatial resolution of this sensor was on the order of the individual optically scanned fields obtained at low magnification. For a parallel plate capacitor, the area sensed is dependent upon the distance between the two plates. The expressions for a simple parallel plate capacitor are

$$C = Q/V, \tag{1}$$

$$C = \varepsilon A/d, \tag{2}$$

from which the charge is

$$Q = \varepsilon AV/d, \tag{3}$$

where C is the capacitance, Q the electrical charge, V the voltage, ε the electrical constant, A the area, and d is the distance between the plates. Based on normal methods of estimating spatial resolution, the area sampled should be the area of the detector when the detector is a distance from the surface equal to a characteristic diameter of the sensor. In this case, at a distance of 0.51 mm, the sample area should be a circle with a diameter of 510 μ m (Vosteen, 1984 and Vosteen, private communication, 2008). There is a well known edge effect for parallel plate capacitors where the field lines do not remain normal to the surfaces. This edge effect increases the sample area, A, by an estimated amount proportional to adding a length of 3d/8 to the radius (Feynman et al., 1977). Table II shows the estimated effective diameter (D_e) of the electric field as a function of the sensor diameter ($D_0 = 510 \ \mu m$) and the distance between the probe and the surface

$$D_e = D_o + 2(\frac{3d}{8}).$$
 (4)

TABLE II. Estimated effective diameter as a function of distance between the probe and the surface.

| 500 | 501 | 86 | 17 | 385 | the probe and the surface. | |
|---------|-------------|------------|-----------|------------|----------------------------|----------------------------------|
| -500 | -495 | 6.7 | 1.3 | 358 | Distance (d) (μ m) | Effective diameter, $D_e(\mu m)$ |
| 0 50 | 2.9 54.5 | 7.3 7.2 | 250 13 | 385 385 | 510 | 892 |
| -50 | -44 | 6.6 | 15 | 385 325 | 1000 2000 | 1260 2010 |
| 1.5 | 1.0 | 0.008 | 0.5 | 325 | 3000 | 2760 |

Number of

values (n)

^aA battery source connected into the interface.

Applied

voltage (V)

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FIG. 4. Horizontal scans for the sensor distance indicated in the plot. Each line represents the mean value from 5 individual scans. The uncertainties are based on the standard deviations determined for the scans at each distance across the surface and the assumption that the edge is ± 1 field of view from one selected. Effectively, this becomes $2 \times 434 \ \mu m$ (one field from each side of the Teflon rectangle). An optical micrograph of the Teflon rectangle that is 3.89 mm across the base in the direction of the electrostatic scan is shown as an inset.

The spatial resolution as a function of probe or sensor distance was tested using a Teflon rectangle (homemade electret) attached to a glass microscope slide. A micrograph of this piece is shown in Figure 4. The optical micrograph was composed of tiled individual optical images that were 434 μ m × 320 μ m each. For the electrical field measurements, the same field of view size was used. The mosaic has 9 fields of view across the horizontal direction and 14 fields vertical as shown in Fig. 4. The Teflon surface was charged negatively by rubbing on a cloth surface. The width of the Teflon rectangle was scanned from right to left. Distance between the Teflon and the sensor was set using the vertical optical mount adjust and Si wafer spacers, the thickness of the spacers was determined by a calibrated micrometer. We would expect as the distance to sample increases, the sampled area increases and the resolution decays. We recognize that even at a probe diameter of 510 μ m the sensor over-samples for these fields of view, i.e., there are probe-sampled areas overlapping with respect to the optical fields because the electrical interaction area is larger as discussed above. Results of the scans at 1.1 mm, 2.3 mm, and 3.4 mm heights are shown in Figure 4. Each scan line presented in the plot is composed of an average of 5 separate line scans. The scan trace starts by displaying the charge level of the glass slide (near V = 0), encounters the edge of Teflon, scans across the Teflon which is negatively charged, traversing over the other Teflon edge and finally encountering the other glass slide surface. At all distances, the charged Teflon is mapped by the scan. It is obvious that the scan at 1.1 mm distance more closely represents the charged object which is 3.89 mm wide. The voltage magnitudes are different because of different charge loads on the Teflon surface since the charging mechanism is not well controlled as in the copper plate electrode case described above. Table III presents the calculated Teflon widths based on the sensor diameter corrected for the distance from the sample and the assumption TABLE III. Measured average scan width at full width half maximum of the 3.89 mm wide Teflon surface for three heights above the sample surface. The measured scan width is given with the standard error in the mean (n = 5).

| Distance above surface (mm) | Sensor effective diameter (mm) | Measured scan width (mm) |
|--------------------------------|-----------------------------------|-----------------------------|
| 1.1 | 1.3 | 4.1 (0.3) |
| 2.3 | 2.2 | 4.6 (0.6) |
| 3.4 | 3.1 | 5.25 (0.4) |

that when the corrected sensor cross section leaves the edge of the Teflon, the voltage falls to the background glass slide voltage.

Measurements made from full width half max indicate object width values in the range of 4.13 mm-5.25 mm (with ascending widths as a function of height) which agrees well with actual width of 3.89 mm. Half max values correspond physically to the sensor being one half over the Teflon and one half over the glass surface.

IMAGING TRIBOELECTRIC CHARGING

Most of the charging on nonconductive materials was brought about by rubbing two surfaces together, inducing the triboelectric effect. When two different materials are brought into contact (order 0.4 nm separation) there is usually a charge transfer such that there will be a net negative charge on one surface and a positive charge on the other. The mechanism is related to the different chemical potentials of the material driving electrons to transfer from one surface, leaving positively charged holes behind. This phenomenon has been observed for centuries and is known as the triboelectric effect. An example of a triboelectric series containing polymer materials is listed below in Table IV (Lowell and Rose-Innes, 1980). The series gives one a general way to determine how a material will charge when it comes in contact with another material. In general, the polarity of the charge conveyed depends on where the two materials reside in the series. From the order in the list, Teflon will charge negatively if rubbed against virtually any material and glass will be charged

TABLE IV. Triboelectric series.

| Wool (most positive) |
|------------------------|
| Nylon |
| Glass |
| Viscose |
| Cotton |
| Silk |
| Acetate rayon |
| Polyvinyl alcohol |
| Dacron |
| Orlon |
| PVC |
| Dynel |
| Polystyrene |
| Polyethylene |
| Teflon (most negative) |



FIG. 5. Charge distributions of two materials brought in contact. Electrical potential is represented in terms of volts measured by the electrostatic voltmeter. The clear vinyl (left) is charged negatively while the glass fiber wipe (right) is charged positively.

positively if rubbed against the materials below it in the series and negatively if brought in contact with materials above glass. Park *et al.* (2008) and Mazumder *et al.* (2001) conducted

studies related to the practical application of material separation utilizing tribo-charging and electrostatic extraction.

An interesting demonstration is shown in Figure 5 for a clear vinyl plastic patch brought in contact with a glass fiber wipe material. There was no rubbing, only touching of the two surfaces. When the materials were separated, the charge distributions were determined on both surfaces. As can be seen, the clear vinyl was charged negatively and the glass fiber wipe was charged positively. This was consistent with other observations that glass fiber wipes charge positively when wiped against vinyl. Electrons were transferred from the glass fiber material to the clear vinyl. The magnitudes were not exactly the same, but this may be due to the fact that the charge measurements were made serially and some

neutralization occurred to one surface. Also, the surfaces may be different; the glass fiber wipe was an unwoven fiber material while the vinyl was more planar.

CHARGE LEVELS

What is the elementary charge levels deposited in the swiping process? To estimate the levels, we use Eq. (3) and our experimental values. The calculation does not include the increased sample area (3d/8 term),

$$Q = \varepsilon AV/d.$$
(5)

For $A = \pi (2.5 \times 10^{-4} \text{ m})^2 \approx 2 \times 10^{-7} \text{ m}^2 \approx 2 \times 10^5 \mu \text{m}^2$, d = 0.001 m (1 mm), $\varepsilon = 8.85 \times 10^{-12} \text{ C/Vm}$, V = 1 V, then $Q = 1.7 \times 10^{-15} \text{ C}$ where there are 6.2×10^{18} charges for 1 Coulomb, $Q \approx 11 000$ charges for each 1 V on the sampled wipe area. This translates to about 5.5×10^6 charges/cm².



FIG. 6. Optical micrographs and charge distributions for muslin cloth and Teflon wipe material. The charge images are mirror images of the actual cloth as indicated. The electrical potential maps are artificially colored for the muslin cloth to indicate the level of charge. The muslin is 22.3 mm wide and shows only small charging. The optical image of the Teflon wipe is shown in (a), the blank, uncharged electrical gray level potential map is presented in (b) and charge image for the same wipe is shown in (c).

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MAPPING CHARGE ON WIPES

We have obtained co-located optical micrographs and charge map for three wipe materials: muslin, Teflon wipes, and commercial Swiffer-Dry. Contour maps of the charge distributions are presented for muslin cloth and Teflon wipe materials in Figure 6. The cloths were rubbed in a single wipe mode against a melamine polyester lab counter top surface. The cloth optical images and their corresponding charge images are usually mirror images of each other because of the manner that the images are acquired. Muslin does not charge significantly and the distribution appears uniform at around -20 V while the Teflon wipe produces significant charge and disparate charge domains. The Teflon wipe, a woven material, was attached to a glass microscope slide. First the Teflon strip was optically imaged and then the slide was inverted and electrical potential reads were obtained of the uncharged wipe (shown in 6(b)). Then the Teflon strip was rubbed and imaged again (Fig. 6(c)). The result was that the Teflon became charged by rubbing and the charge image indicates some non-uniformity in the gray level intensity possibly due to the woven nature of the wipe. Figure 7 shows an enhanced

optical micrograph of a section of Swiffer-Dry material and the associated potential map showing the regions of charge. The enhanced optical image shows raised fiber regions which is indicative of the surface for Swiffer-Dry material. The charge map has been inverted such that it overlays the optical image directly (is not presented as the mirror image) and indicates, in general, high charge accumulation where the raised fibers reside. Both the Teflon and Swiffer cases are good examples of relating the optical features of the sample to the potential domains.

CHARGED PARTICLES

Polystyrene latex spheres were negatively charged by rolling them in a styrene Petri dish and then transferring them to a glass slide. The example shown in Figure 8 is a large single particle imaged optically and electro statically. The electrical potential of the sphere was distinctly higher than the glass slide. The charge map was constructed by the use of SigmaPlot (San Jose, CA) software that merges the potential values found for the fields-of-view. Another example was obtained from a



FIG. 7. Optical image of a section of Swiffer-Dry and the associated charge distribution. The charge map was inverted such that it is a direct over lay match to the optical image.



FIG. 8. A 1.35 mm diameter polystyrene bead shown in the left optical micrograph (left) had the corresponding electrical potential gradients shown in the contour image (right). The potential scale is indicated in volts.

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FIG. 9. Polystyrene 50 μ m charged microspheres were dusted on the glass slide, concentrated on the left side of the image. Optical (left) and charge (right) images of the same region. Charge image, scaled in volts, shows the charge location.



FIG. 10. Three dimensional plots of charge domains for a Swiffer-Dry cloth where (a) measurements were promptly made after charging and the same measurements were repeated 20 min later (b).

dusting of 50 μ m diameter dry polystyrene spheres that were charged as described above and were dusted on a glass slide. The spheres are concentrated in the left side and lower corner of the optical image (Figure 9) which is also where the elevated charge regions are found. Again, the relation of the physical objects shown in the micrographs and the electrical potentials found by this microprobe are illustrated.

CHARGE DECAY

All our measurements are made in laboratory conditions where the temperature is approximately 20 °C and the relative humidity is about 45%. Objects can lose their charge by conduction, but in the case of dielectrics such as some of the polymers used in these studies, charge neutralization is by charge carriers, like water molecular clusters, found in the air. Our first observation of charge decay was during the process of making a charge map of a Swiffer-Dry material. Since the automated stage can reposition the measurement locations for all practical purposes nearly exactly with respect to our ability to determine charged area, we can remap the charge domains after an initial determination. An example of charge level decay is found in Figure 10 for a swiping cloth



FIG. 11. Plot of charge decay for four wipe materials. From the top down a glass fiber coated wipe (blue), a NOMEX wipe background charge levels (black), Swiffer dry (red), Teflon (green), and NOMEX charged (orange).

that was rescanned. The two 3-dimentional plots contain the same contour information, but the charge level has decreased by about 1000 V for the cloth after approximately 20 min exposure to the laboratory air.

Subsequent to these observations, we determined charge decay rates for various wipe materials in our lab conditions. Plots in Figure 11 show the decay rates for wipes made of Teflon, coated glass fiber, Swiffer-Dry, and NOMEX. It is significant that measureable charge is retained by the wipes after 10 min–20 min. This may be relevant to reuse of the wipes since the analyst may be charging their wipes inadvertently between sampling and possibly changing the collection efficiency.

SUMMARY AND CONCLUSIONS

We have developed an instrument and method to quantify charge on surfaces with spatial resolution on the order of 1000 μ m, enabling us to map high and low charge locations on surfaces. Surfaces, regardless of the surface structure, with dimensions of 6 cm by 10 cm (i.e., fits on the microscope stage) can be measured with this electrostatic microprobe. The electrostatic microprobe was useful for mapping charge domains on smooth or rough surfaces with approximately 1000 μ m spatial resolution. The accuracy associated with measuring electrical potentials was determined and the spatial resolution was assessed. Correlating the optical micrograph and the potential map, a capability of this instrument has metrological value. As a demonstration, we have applied the instrument to wipe materials that are charged by rubbing via the triboelectric effect. Four wipe materials have been characterized. Some wipe materials retain charge for in excess of 5 min at laboratory conditions. This may be relevant to those doing wipe sampling since in the sampling process wipes are used to rub objects to collect particles and in the process most likely charge the wipe material. There is no procedure to electrically ground the wipe before the next sampling effort. A charged surface may have a positive or negative effect on sampling efficiency depending upon the magnitude and polarity of the charged wipe and particles that are being sampled. We have charged particles and observed that their charge can be detected using this electrostatic microprobe. Future work includes further surface charge characterizations, attempting to reduce the spatial resolution and measurements of selected electronegative particles.

ACKNOWLEDGMENTS

The author acknowledges a key design idea provided by Dr. Stephen Stranick, the aid of Matthew Staymates for constructing stage components to hold the electrostatic probe and the sample and valuable discussions with Jennifer Verkouteren. This work was funded by support from the Department of Homeland Security (DHS), Science and Technology Directorate, Explosives Division (EXD).

Commercial equipment, instruments, and materials, or software are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement of these items by the NIST, nor does it imply that they are the best available for the purpose.

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