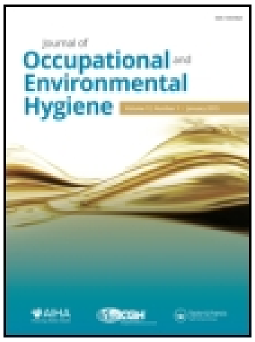

Journal of Occupational and Environmental Hygiene

ISSN: 1545-9624 (Print) 1545-9632 (Online) Journal homepage: https://www.tandfonline.com/loi/uoeh20

Direct Reading Particle Counters: Calibration Verification and Multiple Instrument Agreement via Bump Testing

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To cite this article: John Jankovic, Tracy L. Zontek, Burton R. Ogle & Scott Hollenbeck (2015) Direct Reading Particle Counters: Calibration Verification and Multiple Instrument Agreement via Bump Testing, Journal of Occupational and Environmental Hygiene, 12:7, D116-D122, DOI: 10.1080/15459624.2015.1009990

To link to this article: https://doi.org/10.1080/15459624.2015.1009990

Journal of Occupational and Environmental Hygiene, 12: D116–D122
ISSN: 1545-9624 print / 1545-9632 online

DOI: 10.1080/15459624.2015.1009990

Analytical Performance Issues

Direct Reading Particle Counters: Calibration Verification and Multiple Instrument Agreement via Bump Testing

The calibration records of two direct reading instruments designated as condensation particle counters were examined to determine the number of times they were found to be out of tolerance at annual manufacturer's recalibration. Both instruments were found to be out of tolerance more times than within tolerance. And, it was concluded that annual calibration alone was insufficient to provide operational confidence in an instrument's response. Therefore, a method based on subsequent agreement with data gathered from a newly calibrated instrument was developed to confirm operational readiness between annual calibrations, hereafter referred to as bump testing. The method consists of measuring source particles produced by a gas grille spark igniter in a gallon-size jar. Sampling from this chamber with a newly calibrated instrument to determine the calibrated response over the particle concentration range of interest serves as a reference. Agreement between this reference response and subsequent responses at later dates implies that the instrument is performing as it was at the time of calibration. Side-by-side sampling allows the level of agreement between two or more instruments to be determined. This is useful when simultaneously collected data are compared for differences, i.e., background with process aerosol concentrations.

A reference set of data was obtained using the spark igniter. The generation system was found to be reproducible and suitable to form the basis of calibration verification. The bump test is simple enough to be performed periodically throughout the calibration year or prior to field monitoring.

Keywords air monitoring, calibration, condensation particle counter, nanoscale material

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Color versions of one or more of the figures in the article can be found online at www.tandfonline.com/uoeh.

INTRODUCTION

It is common competent professional practice, as well as a frequent requirement for compliance employee exposure assessments, to calibrate industrial hygiene sampling equipment often before and after sampling.(1) In the expanding field of nanoscale materials, there is increased need to accurately characterize aerosol number and size; instrumentation that measures in the nanoscale is evolving and demonstrates its own limitations.(2) Frequent calibration is not generally feasible for direct reading particle count devices, and the manufacturer's calibration record is relied upon. Instrument manufacturers frequently recommend annual calibration for their sampling/analytical equipment, as does the Maintenance Schedule in the Operation and Service Manual for the Model 3007 condensation particle counter (CPC) manufactured by TSI (Shoreview, MN, 1930035, Rev. D, February 2004)(3), the instruments used for this work. As determined by the manufacturer, a passing tolerance is within 95% to 105% in each of five concentrations ranging from approximately 100 to 10,000 particles /cm^3, as specified on TSI's calibration certificates.

TABLE I. Calibration History of Two CPCs

	2006	2007	2008	2009	2010	2011	2012	2013	2014
CPC #1	Pass	Fail		Fail		Fail	Pass	Fail	Fail
Mean% of Std.[A]	98	21	No report available	120	No report available	16066	100	93	88
CPC #2	Pass		Fail	Pass		Pass	Fail	Fail	Fail
Mean% of Std.[A]	99	No report available	22	98	No report available	103	0	106	106

[A] This represents the average percent difference between the instrument and each standard calibration concentration as reported by TSI.

A review of the calibration records for two CPCs over a 9-year period is presented in Table I. From a review of data it is easily concluded that instruments go out of calibration over the course of a year; however, it is not discernible when this happens. The presumption is that loss of calibration occurs at some point after return from the manufacturer and not as a result of shipping. With a few exceptions, such as CPC #2 in calibration year 2012 in which the instrument failed to produce a count, the loss of accuracy could be gradual and difficult to discern during the course of regular use. Although typically the instrument operator is unable to perform a factory-type calibration of the instrument, a field-type operational check, here referred to as a bump test, may be accomplished at any time and may suffice under certain conditions of instrument use to confirm the calibration state of the instrument. Additionally, bump test capability facilitates troubleshooting a perceived "high/low count" problem when unexpected results are encountered.

When two or more instruments are used simultaneously to monitor an environment and conclusions based on the differences between the measured concentrations are to be made, actual concentration values may be of less importance than instrument agreement. This is particularly valid for nanoscale materials, where no clear regulatory limits exist due to the plethora of materials and morphologies being developed. A side-by-side agreement test is useful to verify the presumption that two calibrated instruments will produce the same concentration measurement when measuring the same aerosol. If a difference is found, the agreement test results can be used provide a correction. Once agreement is determined, differences in instrument responses are then attributable to differences in the environment alone.

Both cases mentioned above can be addressed by the ability to measure the concentration of a reference aerosol. Ambient air may serve for a single concentration agreement test. However, due to spatial and temporal variability,(4) the ambient air is unsatisfactory for checking concentration measurement consistency via a bump test.

METHODS

Electrical Arc Generation of the Reference Aerosol

The arc from a simple gas grill igniter operating on a 1.5 V dry cell battery which charges a capacitor is capable of generating a reference aerosol. When the capacitor discharges, an electrical arc is produced at the electrical connectors (Figure 1 left). This arc is capable of thermally ablating its electrical connector material producing aerosol particles in the nano-scale sizes at concentrations approaching 10^6 particles /cc. The igniter button is depressed using a small clamp (Figure 1, right) to provide continuous arcing at a frequency related to the voltage applied. The metal aerosol formed is initially believed to be quantum dot size, but rapidly coalesces into individual spherical and agglomerate particulate with diameters up to several hundred nanometers (Figure 2).

The arcing frequency is a function of the battery voltage. As the voltage drops under load, the arcing frequency decreases over a period of time. The number of particles generated also decreases as the arcing frequency decreases. Arcing will occur for hours on a single battery; however, arc rate is not constant. Assuming a bump test duration of 30 minutes, using a new battery results in a larger change in the arcing rate than a battery with more than 2 hours but less than about 5 hours of use to operate in the linear portion of the curves in Figure 3. A constant power source in place of a battery reduces the particle generation variability but adds to the materials needed for the test system. The arcing frequency must be determined and matched when establishing the instrument's bump test data. And this same arcing frequency must be utilized when subsequently bump testing an instrument. The easiest way to achieve the same arcing frequency is to always start with a fresh battery and allow it to operate for 2 hours before conducting the bump test. This was the method used for the data presented here.

To determine the sparking rate, count the number of sparks in 10 seconds and multiply the count by 6 to give the sparking frequency in #/min. The easiest way to do this is to tap a pencil while moving across a piece of paper at the cadence of the spark rate. Then count the dots and complete the calculation as stated above.

Collecting the Reference Data for a Calibrated CPC

The process of generating a reference aerosol requires a gallon glass jar or similar enclosure as a generation chamber (Figure 4). The lid is drilled to make two openings sized to accept hose barbs for the tubing diameter of choice; one for

FIGURE 1. Gas grille igniter

filtered make-up air, and one for a sample outlet connected to the CPC. The sampling outlet line is connected to the inlet of a flow rate meter (1 to 50 cc/min typical). The sampling line from the flow rate meter outlet is provided with a metering valve to set the particle containing flow rate before being led into a "Y" fitting. One leg is then attached to the CPC. The other leg is attached to a zero filter and left open to room air. The flow in this make-up air leg is the difference between the particle laden flow rate and the total flow rate of the CPC. The advantage to this set up is that small dilution metering is possible which provides for generating particle concentrations below 10,000 p/cc. The disadvantage is that particle-laden air is passed through the flow rate meter which may at some point require cleaning.

First, determine that the CPC is operating at the same flow rate as the manufacturer specifies. Prior to attaching the CPC and air filter, place a battery into the igniter and use a small clamp to depress the igniter button to start the igniter arching. Next determine the arc rate in terms of the number of arcs per minute. Lower the igniter into the jar, close the lid, and connect the CPC /filter to the "Y" inlet. Adjust sample flow to a low value, e.g., 5 cc/min. The CPC is then operated in survey mode until the CPC concentration stabilizes at which point logging is started (one second averaging time is recommended). After about 1 minute the logging is stopped. The sample flow is increased and the process above is repeated until the concentration range of interest is observed.

Alternately, in lieu of data logging, the TSI CPC can be operated directly from a computer via the Aerosol Instrument Manager software provided with the instrument (TSI, Shoreview, MN). Three to five data points are usually sufficient to cover the working range. A data point consists of a minimum of 1 minute at the respective concentration level before adjusting the flow rate to produce the next concentration level. Repeat the entire process for at least two more cycles. Construct a plot of each particle concentration mean and confidence interval (Microsoft Excel [Microsoft, Redmond, WA] confidence.norm 95%) as a function of the sample airflow rate (dilution factors) for the data which become the bump test reference. At any time a single cycle following the above process can be completed and compared with the reference data. Agreement can be decided based on inspection of the results or with simple statistical tests; agreement meaning that it is reasonable to conclude that the instrument is performing as it was when the reference data were obtained.

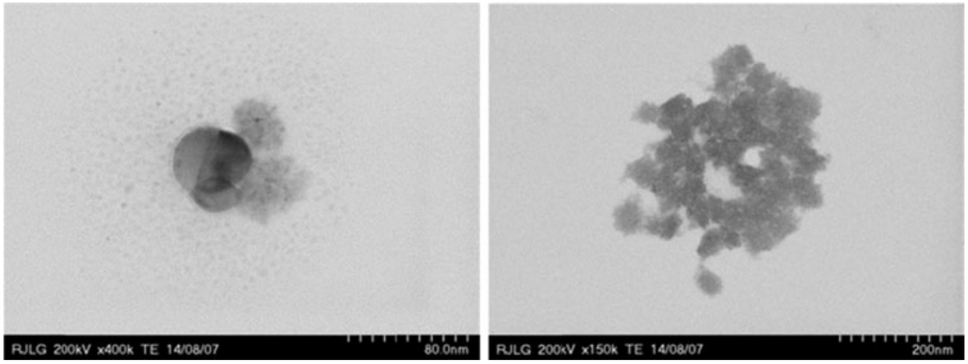

FIGURE 2. Arc generated aerosol characteristics (Image by RJ Lee Group

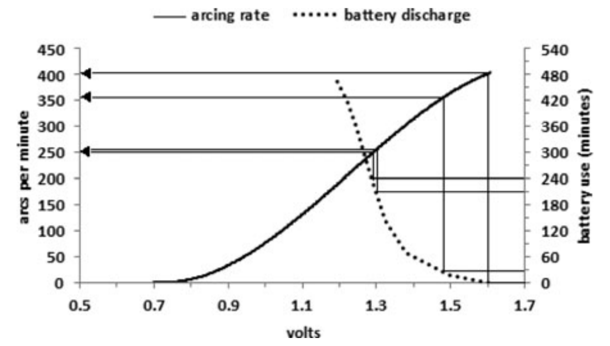

FIGURE 3. Voltage and arc frequency decline with time of operation

Determining Agreement Between Two CPCs

The agreement between two CPCs can be easily determined using the generating system outlined above. The flow rate meter is removed, the needle valve is removed or fully opened, and the zero filter is replaced with a second CPC (Figure 5).

Start the igniter and place in the jar as before. Allow the concentration in the jar to build up. Then open the jar and stop the igniter, replace the lid, connect the sample line to the two CPCs, and begin logging the particle concentration decrease over time. Download the logged data from both instruments and plot concentration either as a function of time for each instrument, or CPC 1 concentration versus CPC 2 concentration (matched times). Determine agreement by inspection, for example, the current data measurements falls within the confidence intervals for the reference data. Other simple statistical tests may also be employed.

If one assumes that the generation system has not changed it follows that an instrument losing accuracy produces a sample set in which both the measured concentration, as well as the variance, becomes larger or smaller than that of the reference data set. The concentration plot against the reference can be used to assess the former while a two-tailed F-Test for differences in the two variances can be used to assess the later. Since returning an instrument for calibration prematurely is costly and if degraded performance in concentration measurements do not entail serious health concerns from its continued use, a stringent α level of 0.01 should be considered, thereby minimizing the likelihood of concluding that a difference exists when in fact there is none.

RESULTS

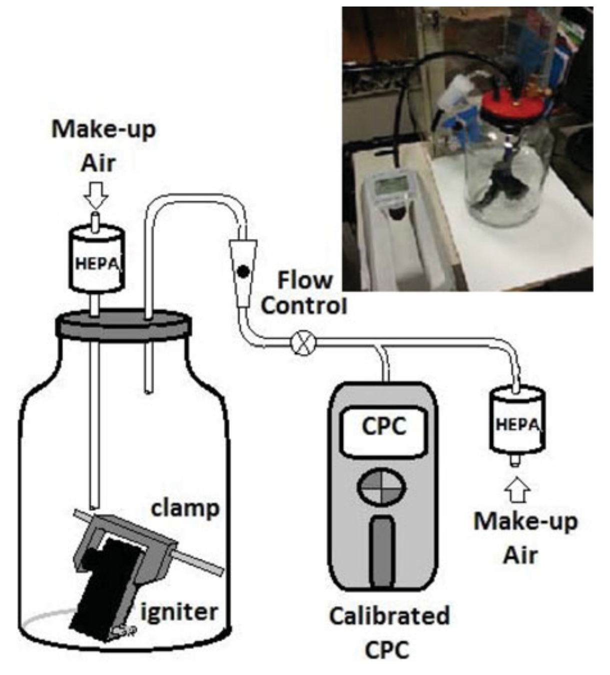

FIGURE 4. Bump test apparatus

Condensation particle counter flow rate(s) should be verified prior to collection of bump test reference data, conducting a bump test, or conducting an instrument agreement test. TSI's Operation and Service Manual indicates a flow rate of 700 cm^3/min nominal. The CPCs used in this study were within those tolerance limits (CPC #1 flow rate at calibration 0.76 lpm, current flow rate 0.76 lpm; CPC #2 flow rate at calibration 0.72 lpm, current flow rate 0.72 lpm).

CPC # 1 Reference Data

Bump test reference data for CPC #1 were collected immediately following the instrument's return from factory calibration. The procedure followed was as stipulated in the Methods section above. Sixteen complete cycles were logged at various battery voltages and corresponding spark frequencies from 360 to 180 per min. The results are summarized in Table II and Figure 6.

Examining the values in Table II for each concentration range gives some idea of the large concentration variation when battery voltage is not kept constant. This results in wider confidence intervals, i.e., less sensitivity of the bump test to detect an instrument change.

CPC #1 Bump Test

A bump test for CPC #1 was conducted on August 22, 2014. The bump test data set was plotted with the mean and confidence levels at the various concentration levels of the reference data set found in Table III to provide for graphical inspection of the data (Figure 6).

Not surprisingly the plot of the bump test data in Figure 6 departed from the reference data mean with some readings lower and others somewhat higher, but within the confidence limits for each reference concentration mean (Figure 6).

Next, sampling results are examined for error due to post-calibration instrument changes in reference to a set of concentrations obtained with the newly calibrated instrument.

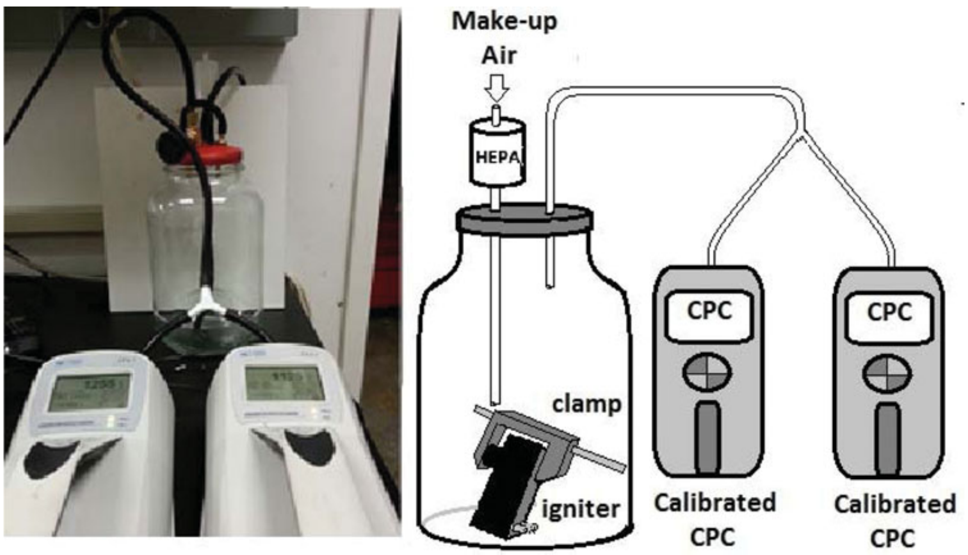

FIGURE 5.. Two CPC agreement test

Subsequent sampling of the same aerosol parameters provided a bump test data set. Table III presents the results of an F-Test for two sample variances. The conclusion based on the bump test examination was that the instrument was performing as it was upon return from factory calibration, differences in the readings being attributed to chance variation (F > F-crit, $p = 0.36$).

CPC #1 and CPC #2 Agreement Test

The aerosol collected in the bump test generation chamber (jar) was simultaneously measured by CPC #1 and CPC #2

TABLE II. Descriptive Statistics for CPC #1 Reference Data

cc/m	5	10	15	21	35	50
Mean	179	477	1153	1927	3590	6173
Standard Error	18	51	124	184	397	567
Standard Deviation	74	203	494	735	1590	2270
Sample Variance	5417	41376	244053	539686	2527248	5152423
Count[A]	16	16	16	16	16	16
Confidence Norm (95.0%)	39	108	263	391	847	1210
Relative Standard Dev	2.4	2.4	2.3	2.6	2.3	2.7

[A] A count of 16 refers to 16 separate measurements at each concentration level.

TABLE III. F-Test Two Sample for Variances

	Bump Test	Ref data
Mean	2099.3333	2249.85417
Variance	3729773.5	5184451.19
Observations	6	6
df	5	5
F	0.7194153	
P(F< = f) one-tail	0.3633203	
F Critical one-tail	0.0911825	

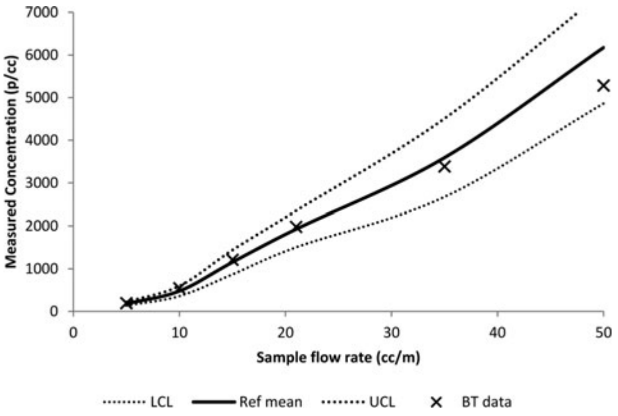

FIGURE 6. Reference mean with confidence limits overlaid with bump test results

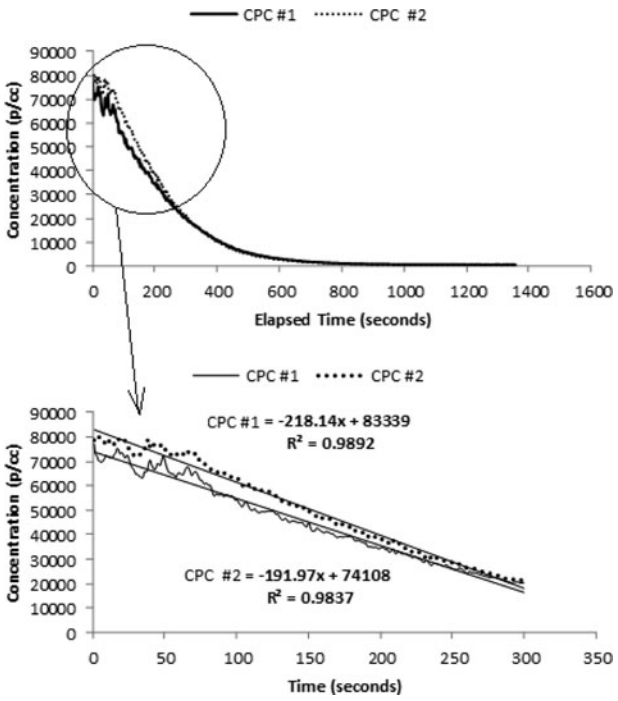

FIGURE 7. Comparison of CPC #1 with CPC #2

following the agreement procedure stipulated in the Methods section. By inspection of Figures 7 and 8 it was concluded that the instruments were in agreement up to about 10,000 p/cc. Above 10,000 p/cc CPC #2 had a high bias, even though both instruments were in calibration and passed bump tests.

A consistent, quantifiable bias does not invalidate the use of the instruments for comparing the concentrations from separate locations temporally. Data from one instrument are corrected using the regression equation from a CPC #1 vs. CPC #2 concentration plot before any comparisons are made.

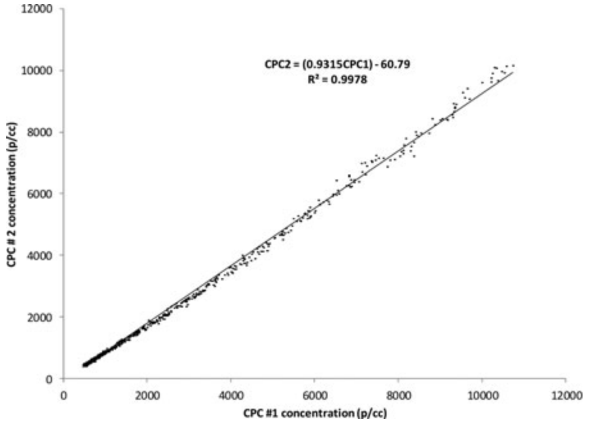

FIGURE 8. CPC #1 regressed on CPC #2 for the 10,000 p/cc and less fraction

DISCUSSION

A pass/fail criterion should be established before conducting a bump test. The bump test itself is not as accurate as the factory calibration primarily because the aerosol generation method is more variable. This alone does not invalidate the test if one is able to decide in advance what practical level of accuracy is required.

Spark generation rate variability could be reduced by replacing the battery in the spark igniter with a controlled source of electrical power. A more constant generation rate would produce tighter limits and increase the ability of the system to detect a change in the instrument. However if an analytical error of +/−25% is adopted, a value sometimes recommended for sampling and analytical methods,(6) the system as described should suffice. Considering the case above, even if the 240 p/cc difference was statistically significant, that difference would not be practically significant should one be interested only in a difference of a thousand or more.

Instrument agreement could be thought of as precision of measurements between two instruments. And, it is tempting to analyze agreement between two instruments measuring the same thing using correlation coefficients.(7) This seems reasonable since two instruments designed to measure the same thing should correlate, and they usually do. However, the correlation coefficient (r) measures the strength of an association, not necessarily the strength of agreement.(8) That is, there can only be perfect agreement if the points lie along the line of equality (slope = 1), but there can be perfect correlation if the points lie along a straight line of any slope regardless of the actual agreement between the values. And a slope other than 1 (or -1) represents a bias.

CONCLUSION

A ready means of testing direct-reading instrument performance should be a requirement prior to conducting airborne concentration measurements. Bump testing, as described here, can provide an indication of continued instrument performance subsequent to factory calibration. The bump test method using a spark-generated aerosol is easily accomplished using readily available materials at a nominal cost. However, any reliable particulate source may be used in a similar fashion as a means of verifying instrument performance.

Two calibrated instruments may differ from each other to some degree. Instrument agreement should be determined and any difference corrected any time the readings of two or more instruments are intended to be compared for differences in concentration. Regression line equations may be used to adjust one instruments bias relative to another for purposes of comparison.

ACKNOWLEDGMENTS

This research was conducted at the Center for Nanophase Materials Sciences, which is sponsored at Oak Ridge National Laboratory (ORNL) by the Scientific User Facilities Division, Office of Basic Energy Sciences, US Department of Energy (DOE).

FUNDING

Additionally, this research was supported by the DOE Higher Education Research Experience (HERE) and was administered by the Oak Ridge Institute for Science and Education (ORISE).

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