An HEU Confirmatory Measurement Instrument

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MASTER

CONTENTS

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by

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ABSTRACT

Precise measurements of an attribute of the special nuclear material (SNM) in a shipping container can confirm that the SNM was not tampered with during shipping. These measurements do not require a calibration, but they should be based on an attribute unique to each sample. This report describes a new instrument, based on a vertical stack of low-resolution detectors, designed to record such a unique attribute: gamma-rays at 185.7 keV, 1001 keV, and 2614 keV. It collects data for 200 s from sealed shipping containers (208-L barrels). These measurements distinguish the issue of material control (has any material been diverted?) from the issue of measurement control (is there a bias between the shipper's and the receiver's measurements of the same item?). This is an important distinction because material control problems require prompt corrective measures, while measurement control problems are never solved (they just get smaller). This instrument satisfies the regulatory requirements for material control in shipper-receiver transfers. In-plant measurement data are presented to illustrate the stability of the instrument and its ability to distinguish between similar items.

INTRODUCTION

Routine operations in the DOE complex involve the transfer of special nuclear materials (SNM) between facilities in a relatively attractive, pure form. These highly visible transfers of nuclear material in various chemical forms and standardized shipping containers are viewed as unique activities in the physical security analysis and are safeguarded by many layers of security. In addition to the physical protection during the transfer, verification of a transfer's success has traditionally relied on accountability measurements, even in the international community.[1] This reliance is not always the best choice in a security analysis that takes a broader view, including the entire DOE complex.[2,3]

The three objectives of safeguarding SNM in transit are:

(1)   to protect the general public from the misuse of SNM,
(2)   to guard the health and safety of personnel employed at facilities that process and handle SNM, and
(3)   to protect the financial interests of the entities that operate the facilities that handle and process SNM.

The first two objectives can be adequately addressed by precise measurements of the SNM in an item, while the third objective requires accurate measurements of the quantity of SNM in the item. The third objective is sometimes compromised to achieve the first two, which typically have higher priority.

Traditionally the transfer of material between facilities has combined the issues of measurement control, how much was transferred? and material control, did it all get there? Material control can be accomplished with precise measurements of the SNM, while measurement control requires accurate and precise measurements of the SNM. These issues must be clearly separated[2] to better identify the causes of shipper/receiver (S/R) differences. MEASUREMENT CONTROL addresses measurement biases between two techniques or results, and is concerned with calibration and measurement accuracy. This important topic is not our subject. MATERIAL CONTROL addresses the integrity of the SNM shipment, that is, whether the same item was subject to both measurement techniques. Was any SNM lost or diverted or did it all arrive at the receiver's facility?

If material control cannot be satisfied, then measurement control is not an issue, and the missing material must be located as soon as possible. Such a response should upset regular facility operations. However, if material control has been demonstrated, measurement control becomes the important issue, and the response-time requirements relax significantly. The documentation, including the determination of the quantity of SNM transferred, is part of the normal facility procedures.

Material control measurements must:

• be hard to fool,
• precisely indicate that the item has not changed,
• be based on a signal unique to the SNM of interest, and
• be faster and cheaper than traditional accounting.

If they are not, traditional accounting procedures that yield more information should be used. Material control measurements are NOT intended to replace the accountability measurement,[3] but rather to supplement it.[2] It is preferable to measure the entire contents of the container. A common error is sampling a heterogeneous item, and assuming the sample is from a homogeneous item. Extrapolating quantitative results from this biased sample to the item leads to "hard-to-find" errors in the accounting system. More quantitative definitions of these desired attributes are the following:

• a measurement precision of 1% (1 σ) on a unique attribute of the SNM,
• short count times (200 s) with a minimum of sample preparation,

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- minimal cost for capital equipment and operators, and
- a measurement that is hard to fool with a bogus item.

Several facilities considered measuring only the 185.7-keV gamma rays from ^{235}U, because of their high count rate, but they do not penetrate well through large amounts of uranium.[*,3,4,5,6] The measured result does not meet the requirements specified above. It can be fooled, at least theoretically, by substituting a container with a core of low enriched uranium (LEU) or lead with a skin of highly enrichred uranium (HEU) around it, for a container full of HEU. The authors believe that this substitution is difficult to accomplish in a secure facility with an assortment of administrative rules and production expectations. It is very difficult to gain access to SNM and equipment for a sufficient time to perpetrate this diversion without alerting other workers.

Nevertheless, the enhancement from measuring a single gamma ray to measuring two additional gamma rays and the mass of the shipping container does satisfy the specified measurement objectives.[7] The 1001-keV gamma ray is from a daughter of ^{238}U (half-life of 24.1 d, implying equilibrium in 4 months or less after the daughter is separated). It has a reasonable count rate for large quantities (>1 kg U) and correlates well with mass. The 2614-keV gamma ray is from the thorium decay chain (from the use of reactor returns in the enrichment plant feed). It has an unpredictable count rate, and acts like an independent spike or tag, uniquely identifying each item. We also measured the mass of the shipping container. This attribute is not unique to the SNM, but constrains the diversion options and is required by the facility.

This combination of four measured quantities provides a fingerprint for the shipment that is unique, even between similar items, and hard to imitate with substitutions. There is NO need to calibrate; it merely introduces an additional source of measurement uncertainty. The mass result from calibrating to convert gamma signals from counts/second to grams of SNM, does not make the safeguards more effective, but it can confuse the accountability system. It can cloud the primary issue (material control) with the secondary issue (measurement control) and effectively sidetrack resources from their most efficient use.

MEASUREMENT GEOMETRY

These HEU S/R measurements will be performed on sealed shipping containers. Preserving the original tamper indication will be usefu' 'f measurement biases result when the material is opened and analyzed before being used in the facility. In addition, there is less chance of contamination or spillage if the outside containers are not opened. Finally, considerably fewer people and facilities will be required to safely handle the items.

The outer container is usually a 208-L barrel. The inside geometry of these barrels can vary widely, but general criticality safety nearly always requires each barrel to have a 5- or 6-in. diam. pipe in its center. The pipe is surrounded by low Z, low density, packing material (which may be somewhat hydrogenous) and holds two 2-L or 4-L bottles, which are inside plastic bags. The contents of the bottles are pure HEU in the form of oxide, metal, or uranyl nitrate. The samples are left in their shipping containers with the tamper indicating devices (TIDs) intact. This minimizes handling time for sample preparation, reduces the possibility of spreading contamination by keeping double containment, and preserves the TID until the accountability measurement is performed.

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The measuring instrument is four low resolution detectors in a vertical stack.[8] The acronym is SRCS (shipper receiver confirmatory system). Figure 1 is a cross section of the detectors in their collimators viewing the three possible shipping containers (30-, 55-, or 110-gal. drums). Each 3 x 3 NaI detector has 6% resolution at 662 keV, is in a 4-in.-thick lead cylindrical collimator recessed 3 in. into the 4-in.-diam collimator center hole. The front face of each detector is 27 in. from the barrel center. The lowest detector's axis is 6 in. from the bottom of the barrel. The detectors are spaced 20 in. apart vertically. The detector geometry and spacing were chosen to give a uniform response over the sample height as discussed in the results section.

The shipping container is rotated during measurement to minimize the (unexpected) effects of off-center inner containers. If used in a warehouse, vault, or other high background environment in an operating facility, this instrument can be surrounded by an additional 2 ft of concrete to attenuate gamma rays from other samples. Both the 4-in. lead and the 24-in. concrete shielding attenuate the 2614-keV gamma ray by approximately 2 orders of magnitude. The top detector, which looks over the top of the shorter barrels, gives an online check of background levels. This system can be easily automated for a large number of measurements.

Y-12 modified an area in its SNM warehouse to house this instrument. A photograph of the measurement room appears in Fig. 2. This physically separate, enclosed area has heating, cooling, and humidity controls to help stabilize the instrument's response. The measurement area is enclosed in a 2-ft-thick concrete wall to shield the instrument from background radiation (1001 and 2614 keV) from other containers in the area. A conveyor system improved the throughput capability for these measurements. The conveyor has built-in turntables, so that the response from unsymmetrical drums can be averaged during the analysis. The turntables include a clamping and centering device. The conveyor has its own programmable logic controller, allowing it to run either manually or automatically. In automatic mode, the drum is weighed and then moved to a turntable, centered, rotated during the measurement, and then offloaded onto an accumulator. The conveyor can handle items up to 72 in. high, up to 600 lb each, with diameters ranging from 18 to 24 in. With this drum handling capability, Y-12 can measure a large number of drums with minimal impact on other warehouse operations.

DATA REDUCTION AND ANALYSIS

We determine the net peak area for all gamma rays with the standard three regions of interest (ROI) formulas. The NaI(Tl) detectors in the instrument are notorious for electronic drifts and sensitivity to small temperature variations. Consequently these detectors are stabilized by a peak from an [241]Am seed. Whereas the peaks from gamma rays are identified by their energies, the peak from the [241]Am seed is used and labelled as a pulser. This peak is also used for normalization to correct for count-rate-related losses. The measurement control built into the instrument uses a [133]Ba source, not SNM. We choose not to require large amounts of SNM for the instrument's measurement control because of the expense and difficulties associated with handling kilogram quantities. This barium ROI also crudely checks to ensure no plutonium is in a uranium sample during regular measurements. The energy equivalents of the ROIs are listed in Table I. Each detector has the same ROIs except for the pulser.

110 GAL

55 GAL

30 GAL

HEU MATERIAL

LEAD SHIELDING

NaI DETECTOR

DRUM ROTATOR

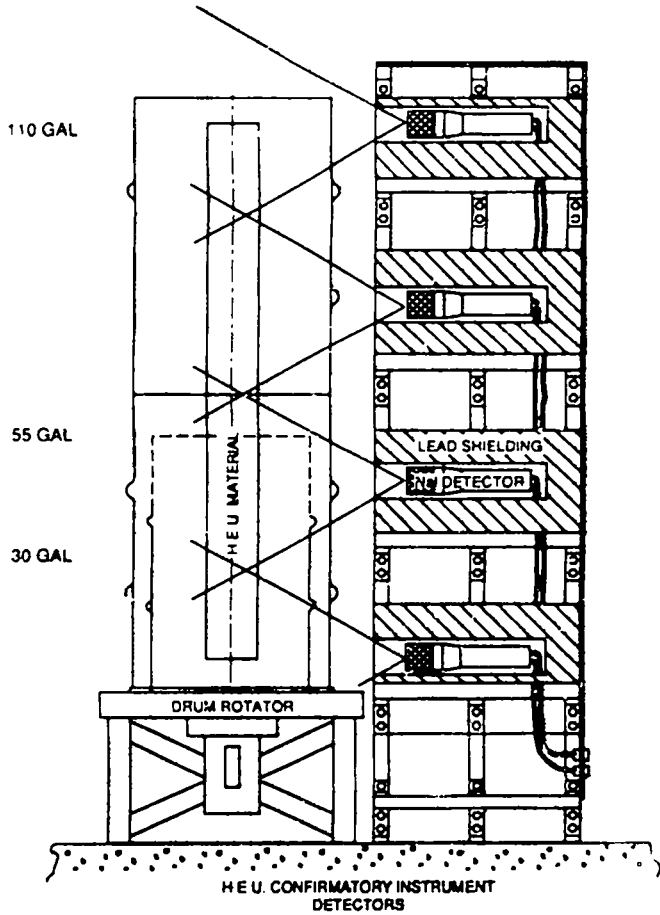H E U. CONFIRMATORY INSTRUMENT
DETECTORS

Fig. 1. Cross-sectional view of the SRCS instrument geometry showing four low resolution detectors. The detector fields of view and the three possible shipping container sizes are indicated. Shorter containers may only require three detectors.

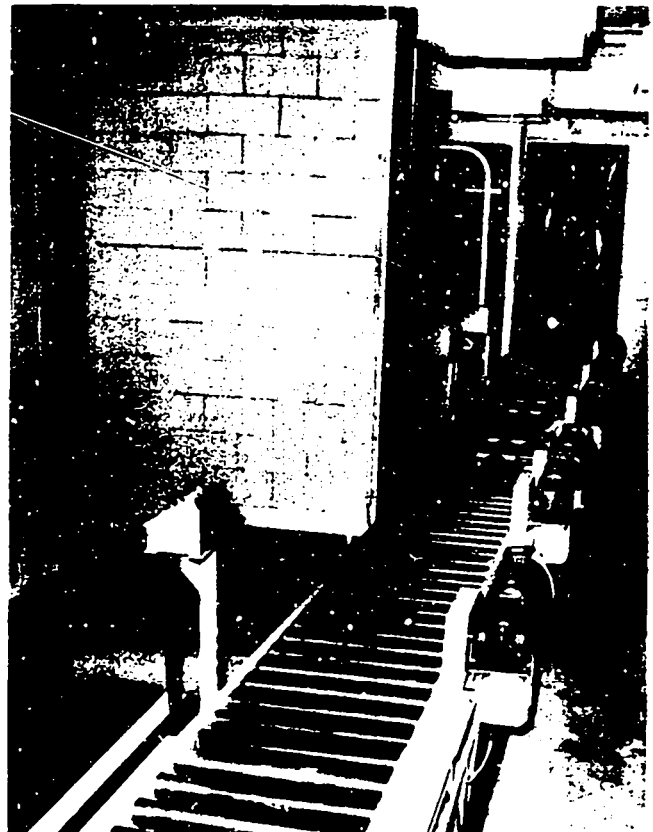Fig. 2. A photograph of the measurement room at Y-12, showing three sample rotation positions inside the concrete walls.

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Table I: Energy equivalents for ROIs			
Peak Energy (keV)	Left Bkgd (keV)	Peak (keV)	Right Bkgd (keV)
185.7	121-134	160-210	280-300
350	280-300	306-485	490-510
1001	857-890	924-1102	1136-1169
Am seed as needed/typical values are:			
1750	1304-1371	1646-1918	2046-2113
2614	2372-2439	2456-2792	2960-3027

INTERNAL MEASUREMENT CONTROL

This instrument has several built-in measurement control functions intended to help the user determine if it is functioning correctly. It stores the results of a background (no sample) run. (This is typically performed daily.) Background results are checked, but not subtracted from each run, for better precision. The results of this background run are checked to be sure that the pulser peak net area is large enough to ensure that counting precision is adequate, and that the other peaks are sufficiently small. We also verify that the seed peak has reasonable centroid and full width at half maximum (FWHM) values. All measurements check at least one peak for an appropriate centroid and FWHM. Most measurements check two peaks.

The second internal measurement control function is a bias check. The user can measure a known sample and see if the results are consistent with the historical value of the sample. Barium-133 sources can be used to perform this check without involving SNM. The precision and reproducibility of a barium source measurement are adequate for measurement control. We recommend that this measurement control check be done at least weekly.

The third internal measurement control function is a precision check. The user can measure any sample 5 or 15 times. The results for each peak are then individually averaged, a standard deviation is calculated and compared to the predicted standard deviation, and a mean square successive difference test is computed to check for randomness of the data. Then the individual peak standard deviations are combined to do a chi-square test on the entire set. This test should be done at least monthly to check for unexpected sources of uncertainty or trends in the response of the instrument.

In combination with the internal measurement control checks, the resolution and centroid checks in each data acquisition assure consistent instrument performance, demonstrate the adequacy of systematic error predictions, and alert the operator if the instrument malfunctions with a low false alarm rate.[9]

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THE SHIPPER/RECEIVER ANALYSIS

All net peak areas are first normalized by the net peak area of the americium seed. The normalized net peak areas $A(i)$ will be the reported results in the units of counts/second. Two sets of results obtained from two instruments $A(i)$ and $B(i)$ will be compared in three ways for statistical consistency. The first comparison will be between the 16 pairs of results individually. The uncertainties in the two results will be compared to make sure they are similar. Then the uncertainties will be combined to form a predicted sigma $\sigma(i)$. Then the difference between the two results will be computed in units of σ:

$$n(i) = \frac{A(i) - B(i)}{\sigma(i)}$$

(1)

The $n(i)$ will be compared to two thresholds, a warning threshold that indicates the data might be inconsistent and an action threshold that indicates the two results disagree. The second comparison will compute a chi-square N from the 16 results:

$$N = \sum_{i=1}^{16} [n(i)]^2$$

(2)

N will also be compared to warning and action limits. The third comparison will compute a chi-square from the m peaks, which have a net area that is statistically significant. First the $A(i)$ will be compared to the $\sigma(i)$. If this ratio exceeds 3.0, the peak will be included in this analysis. The chi-square from the m peaks $N(m)$ will be computed:

$$N(m) = \sum_{i=1}^{m} [n(i)]^2$$

(3)

$N(m)$ will then be compared to both a warning and an action limit. These three analyses are performed on-line by the SRCS instrument at the receiving end.

It is clear that the false alarm rate for this instrument must be very low, if it is to be of use to plant operators. Past experience has shown that the details of this technique will not be understood or even be of interest to the facility operator. A few false alarms a year will rapidly cause the plant operators to respond as if they expect a false alarm rather than a true diversion. This instrument cannot do much to correct mislabeled samples or results that are interchanged between two samples on a short term basis. However, if this behavior is currently a problem, this instrumentation should direct attention to solving this problem in a timely fashion. The action limits can be chosen to minimize the false alarm rate. This does not limit the effectiveness of this technique as much as a cursory overview might suggest. If the three gamma technique is considered in the context of the rest of the system's safeguards, it will be used to rapidly detect large diversions. If some person penetrates the system of barriers, seals, and other protective measures, they will probably tamper with a small number of items in some gross fashion (for example take the entire item). That type

of tampering does not require a high precision, highly accurate measurement to detect it. If a trickle diversion scenario is chosen, the length of time the system is penetrated is much longer. The greater length of time required for a successful diversion will be more susceptible to detection by other means (such as input accountability measurements or physical security). In addition, the use of the 2614-keV emission, whose rate is unpredictable, allows some discernment between items of nearly identical uranium mass and enrichment.

RESULTS

Figure 3 shows the vertical response of this instrument. A small (500 g) LEU sample was moved vertically in 2-in. increments. The 1001-keV response is plotted as a function of the vertical position. The signal from each detector is shown, as is the sum of the responses from the four detectors. The variation in the summed response is consistent with counting statistics. A variation of 1 in. in either the vertical or horizontal positioning of typical shipping containers (containing at least 1 kg of uranium) affects the response by less than 1%.[10]

With a specification that the gamma energy equivalent of the americium seed be 1.7 ± 0.2 MeV, the response from various 3-in. by 3-in. NaI(Tl) detectors is identical. We are investigating the possibility of using short photomultiplier tubes. These tubes provide marginally poorer resolution, but allow the lead shields to be 24% lighter (400 kg instead of 526 kg for each detector).

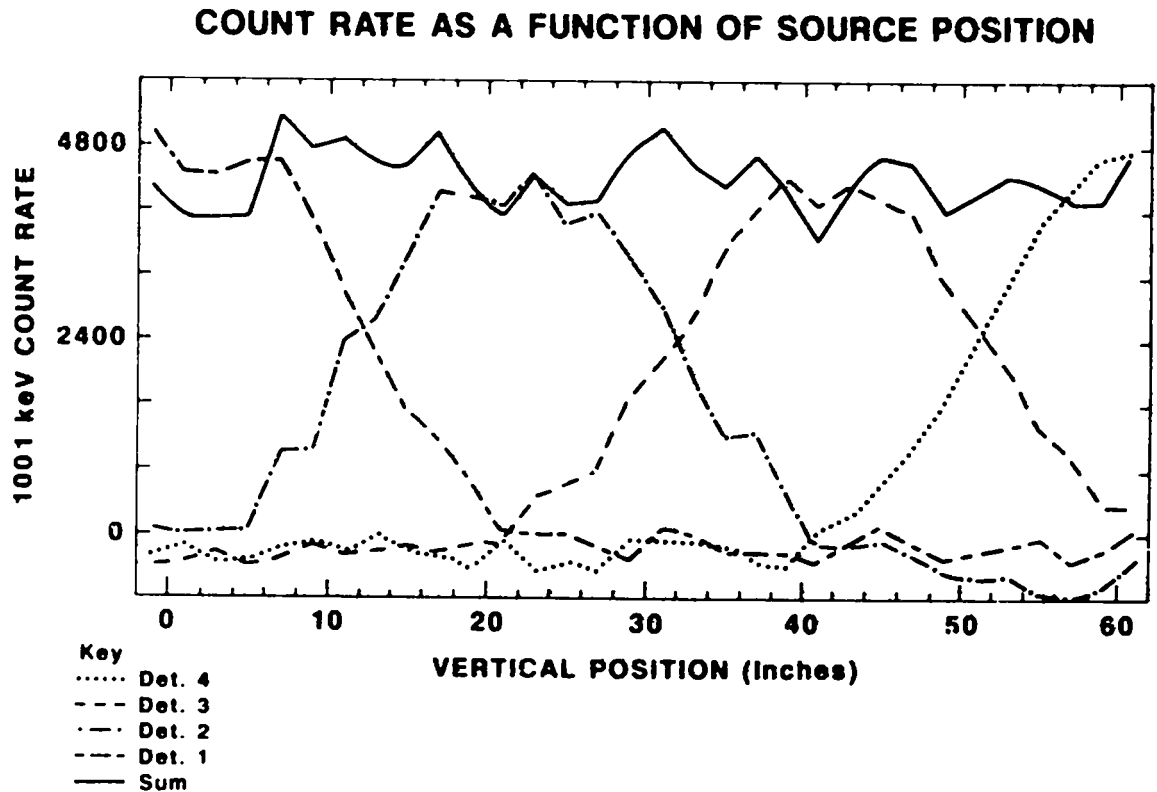## COUNT RATE AS A FUNCTION OF SOURCE POSITION

Fig. 3. Detector count rate for the 1001-keV gamma ray vs sample height. Each detector's response is indicated as well as the sum of the four responses. The sample was a 6-cm-high depleted uranium metal disk. The fluctuations in the summed response are consistent with the precision estimated from counting statistics (1 σ = 340 counts).

Los Alamos evaluated the short term repeatability of the system before it was delivered to Y-12.[10] We performed several tests consisting of 15 cycles of 1000-s counts on assorted samples: a more stringent test than the typical 200-s assay time. The counting statistics reliably estimated the actual standard deviation of the 15 cycles and a mean square successive difference test did not indicate any trends; however, a Shapiro-Wilk test indicated that nearly every run was probably not normal. We suspect that the Shapiro-Wilk test may be too sensitive for our purposes, and that it is reasonable to estimate our measurement uncertainties assuming normally distributed random variables.

After the instrument was delivered to Y-12, its response to actual shipment barrels was evaluated to verify that it could make confirmatory measurements between shipper and receiver, and that the measurement results could be used for inventory verification.[6]

The Y-12 evaluation used 20 test specimens, randomly selected from 4 distinct chemical forms, and randomly divided into 2 groups. The test specimens were blocked by weeks and replicated three times daily and twice weekly for eight weeks. Variation in the ^{235}U mass was not a consideration in selecting the containers, however some items had similar ^{238}U masses. These items were in 208-L barrels or smaller shipping containers, consequently only the bottom two detectors viewed the SNM.

Daily measurement control runs used four barium sources. Data for each detector were collected using a barium source uniquely assigned to that detector (Fig. 4). All detectors have an americium seed for stabilization and rate loss normalization. The barium measurement control displayed a trend similar to that displayed by the 186-keV uranium data.

A temperature problem occurred during the first two weeks of the experiment; we adjusted the equipment and continued the experiment for two extra weeks. The data from the first two weeks of the experiment were thus compromised. They are presented in the plots, but were excluded from the analysis below.

Data for detectors one and two at each net peak were plotted against the date interval (a number representing the week of the experiment). The data appeared to show a trend in the pulser and the 186-keV net peak area, but not in the 1001-keV and the 2614-keV net peak areas. Further
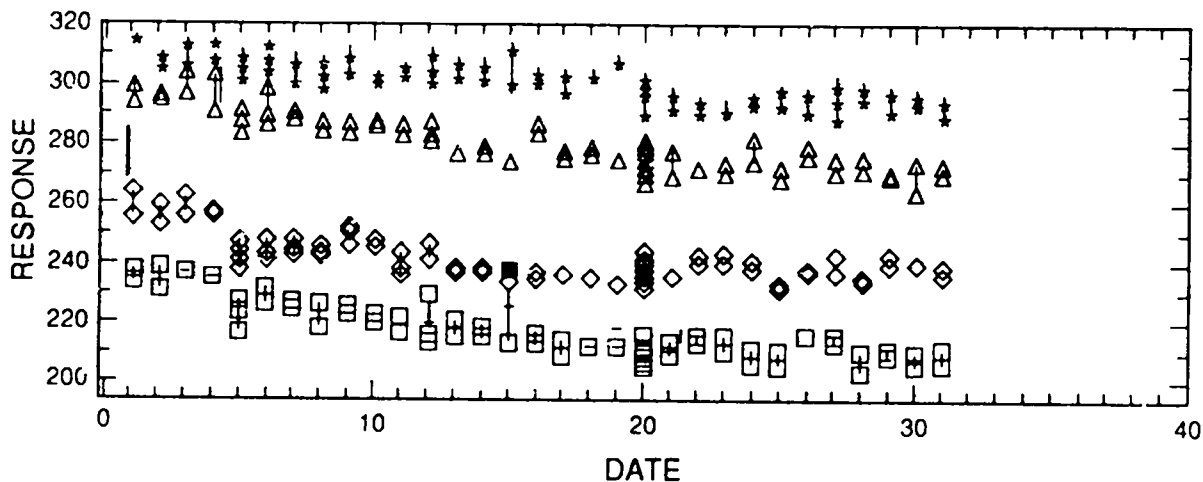

Fig. 4. Plot of variation of barium source response for each detector as a function of time. These data span 10 weeks.

investigation using Shewhart charts confirmed the observed trend. The net peak area of the americium seed is expected to behave in nonnormal patterns, such as trends, but the effect on the other net peak areas should be nullified by normalization by the pulser peak net area. The Kolmogorov-Smirnov test was used to verify that the pulser and 186 keV net peak areas were behaving as a nonnormal distribution. Data from the 2614-keV and 1001-keV net peak areas were normal for most of the 20 samples. The 186-keV peak has much better counting statistics than either the 1001- or the 2614-keV peak.

A second experiment was begun eight months after the first. The air ventilation system had been adjusted because it was felt that the trending might have been caused by temperature variations, but the equipment had not been physically modified. Three of the previously used samples were selected, but not at random, and two replications were taken daily for two weeks. Notable exceptions in this test were that two samples were run only once instead of being replicated and another sample was run three times.

Before the beginning of the second experiment, 30 measurements on the barium sources were taken in 1 day. The data from this exercise proved to be nonnormal, but compatible with the prior distribution. All detectors were performing at different levels, but with the same variation in this short time frame. These data form the large bar at point 20 on the Barium Standard Plot (Fig. 4).

The data for the first and second experiments were analyzed separately, then together, when it was determined that the variances and means were not significantly different for the two groups of data. The single exception to this is discussed below. Plots presenting the results of the two experiments must be interpreted carefully because of the time factor: a week in the first experiment data is the same as a day in the second experiment. Figure 5 shows a representative example of the data from the two experiments. Note the nonlinear horizontal scale. The squares span 18 weeks; the dots span 2 weeks.
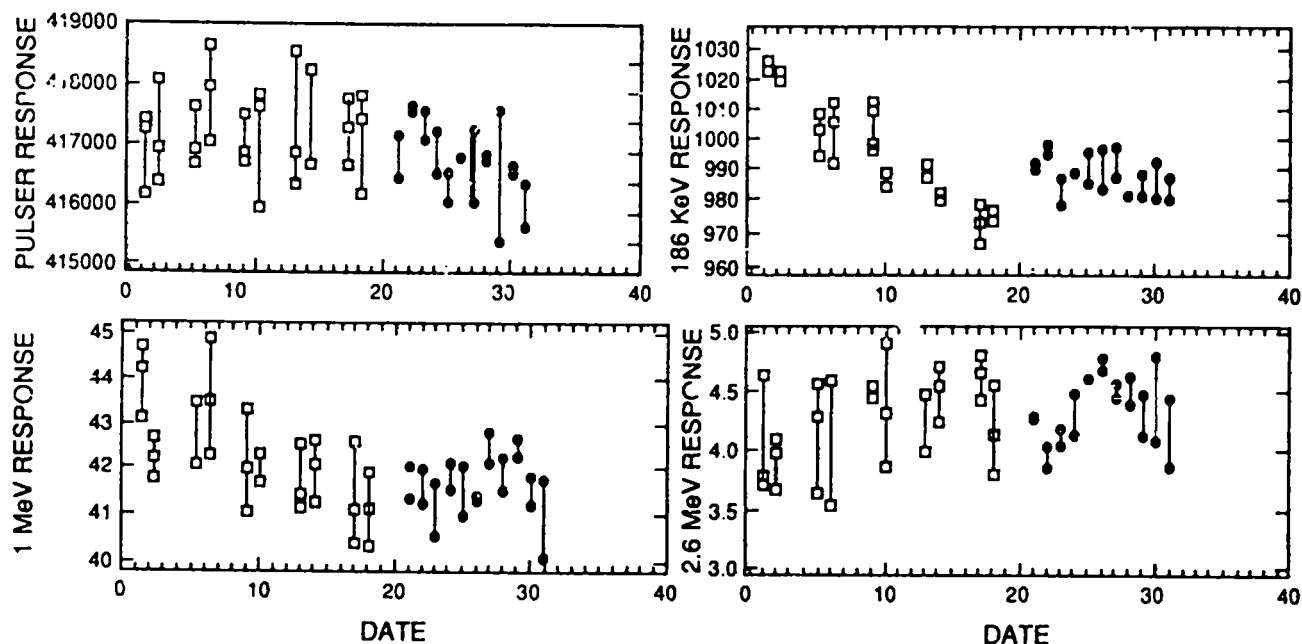

Fig. 5. Plot of sample 10 for the 3 peaks of interest and the americium seed peak for the 10 week initial data acquisition (squares) and the 2 week acquisition period 8 months later (dots).

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Figure 6 shows the responses of the 20 items in the bottom detector. The 186-keV net peak area is plotted against the 1001-keV net peak area. Different geometrical arrangements in the shipping containers and the four chemical forms cause the data to fall on lines of various slopes. The 20 items are segregated into 9 distinct groups. The trends discussed above appear to have little or no effect on the discrimination capability of this technique. Figure 7 shows similar data from detector 2. The 20 items are segregated into 7 different and distinct groups. The solitary green data point is caused by an out-of-control instrument response.

Scatter plots were generated for all the data, and a cluster analysis was completed. Most test specimens were distinguishable from all other test specimens as shown in the composite detector 1 scatter plot (Fig. 8). Some individuals appear nearly identical, but by eliminating the data compromised by temperature problems and the second experiment data, plots with separated individuals are produced.

There were three major findings:

1. Individuals were unique as demonstrated in the scatter plots. There is a unique signature, and within a short time frame (less than 70 days), each item can be distinguished from the others.
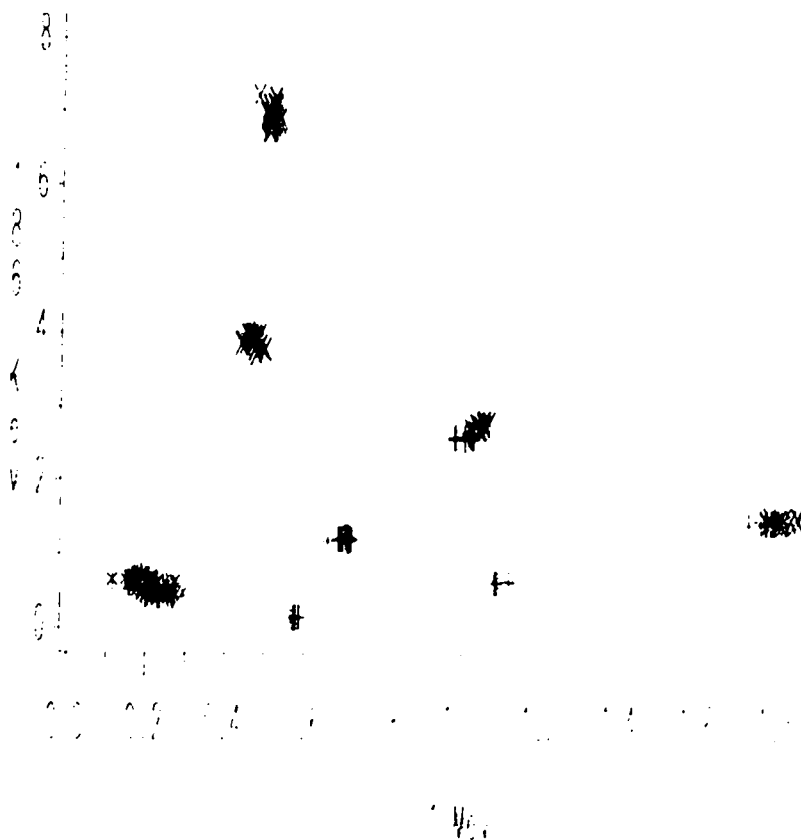
Fig. 6. SRCS test container data for detector 1 indicate the 186-keV vs 1001-keV net peak areas for the 20 items from the lowest detector. Geometrical and enrichment variations cause data to fall on lines of different slope. Nine different sample categories are discriminated.
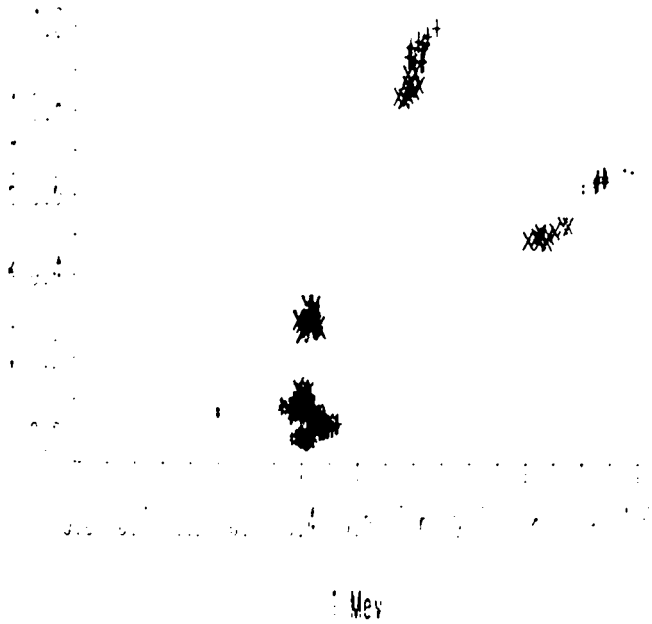
Fig. 7. SRCS test container data from detector 2 indicate the 186-keV vs 1001-keV net peak areas for the 20 items from the second lowest detector. Seven different sample categories are discriminated.
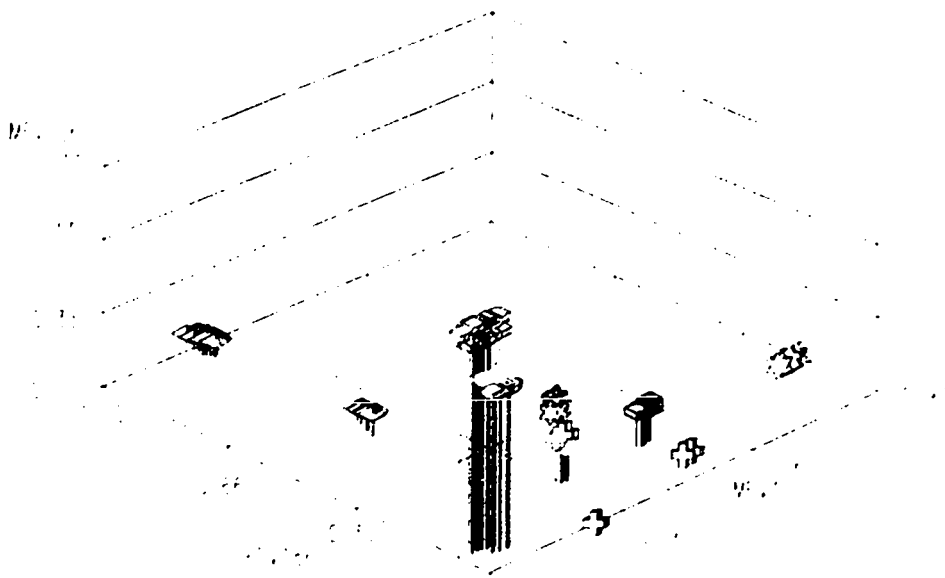

Fig. 8. The group averages from the SRCS test container data for detector 1 yield this three dimensional scatter plot of the three peaks, illustrating the uniqueness of each item. Nineteen sample categories are discriminated vs 20 samples. The trends described in the text are not visible.

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2. Statistical analysis of data in sample 20, the 2614-keV net normalized peak area for the second experiment, was from a statistically different population than that of the first experiment. This implies that the time factor can play a distinct part in the 2614-keV readings for certain types of materials. Because the initial data acquisition over 8 weeks did not appear inconsistent, but data 8 months later did, we assume that sample 20 had the largest growth in the ^{208}Tl daughter.

3. The same sort of trend was exhibited in the 2-week evaluation and in the 4-month evaluation, implying that the trend is not yet understood. As can be seen from the plots, the trend is not continued, but is repeated with an offset. Further investigations of the system are planned. The apparent trending does not hamper the use of the SRCS for short-term confirmatory measurements, but understanding its cause could lead to the improvement of confirmatory techniques so that they are of sufficient accuracy and repeatability to be used for inventory verification. (Temperature instability is the primary candidate for investigation.)

Data from the last 8 of the 10 weeks of the first experiment were grouped according to chemical forms and a regression was attempted to correlate the counts from each of the energy levels and the weight to the assay. As expected from the significant trending, the variates, or their reciprocals correlated poorly. However the plot of the mass of ^{235}U vs count rate (Fig. 9) yielded useful information: distinct curves for the various material types. That this is possible for ^{235}U is surprising given the large quantities of material and the large self-absorption. It must be caused by the purity of the uranium and the uniformity of its packaging. The ^{238}U plot is similar and gives a better correlation because of its "better" self-absorption properties. Figure 10 is a plot of the 1001-keV response vs tag mass of ^{238}U for 41 items of uranium oxide. The spread in the data around a straight line is quite small, considering no correction has been made for self-absorption of the 1001-keV gamma ray. Four items did not seem to be consistent with the other items. Subsequent investigation showed that sample labels had been inadvertently interchanged. Figure 11 shows the same data with the correct sample masses. The SRCS should not be used for materials accountability at this time, but for verifying the integrity of the shipment only. Further work is needed before the method is acceptable for inventory verification.

SUMMARY

The SRCS can meet the design objective of confirming an HEU S/R transfer. The instrument is based on attributes of the HEU; it samples the entire shipping container while preserving its TID, discriminates well between similar items, is hard to fool, and has adequate stability. Its large throughput capacity is improved when the shipping containers are handled automatically. It is quite helpful in identifying items with interchanged labels, so that the ever-present human error can be corrected. This technique also has promise for rapid inventory verification based on a correlation between mass and response for similar items, if its long term stability can be improved. It is a cost-effective complement to layered safeguards, which previously relied primarily on physical protection, because it measures attributes unique to uranium.

The SRCS will help separate the issues of material control and measurement control. It is hoped that this will eventually lead to increased attention in the area in which most of us believe S/R differences occur, measurement control. The precision of this measurement is much better than its accuracy. We are accustomed to thinking about a comparison between two results in terms of the accuracies of the two types of measurement. In this case the precision can often allow the
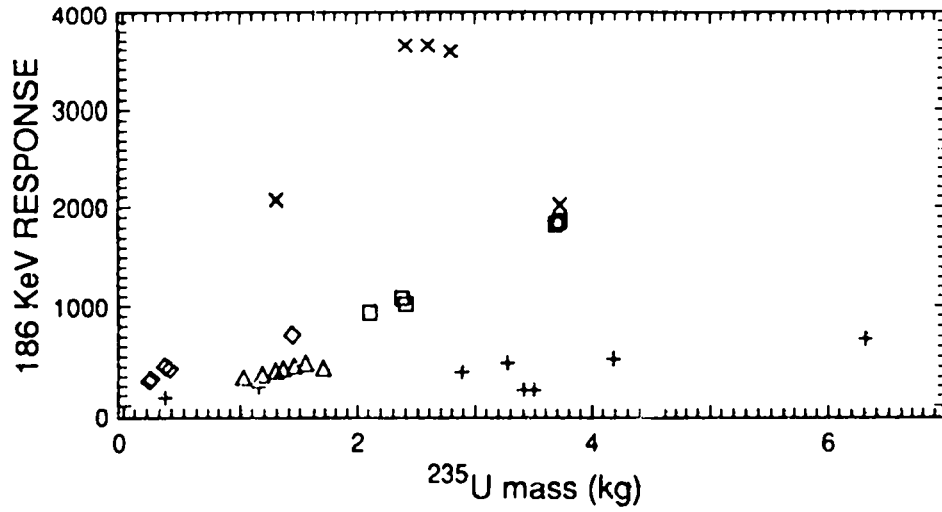
Fig. 9. Plot of ^{235}U mass vs count rate for the 20 items. Various chemical forms seem to fall on various lines.
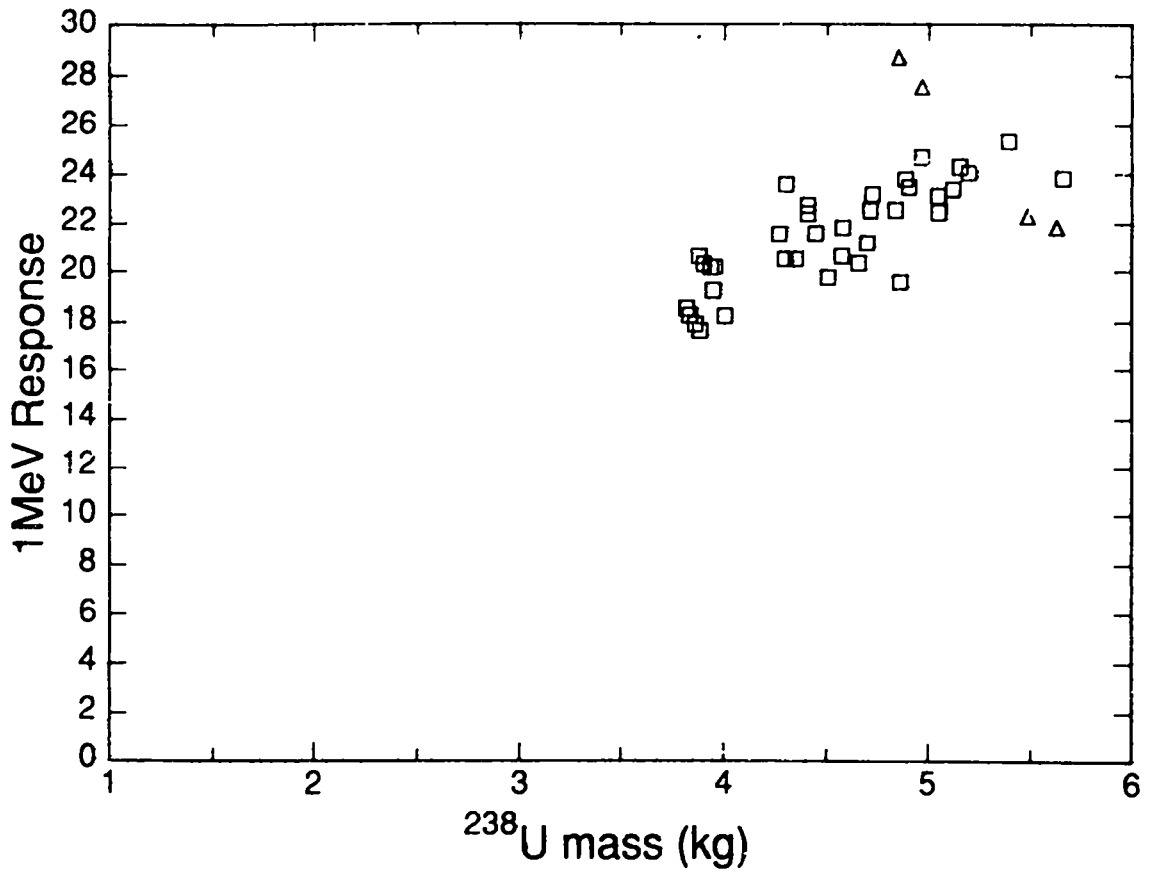

Fig. 10. Plot of ^{238}U mass vs count rate for 41 samples of the same chemical form. Note four anomalous points (designated by Δ) caused by interchanged mass values.
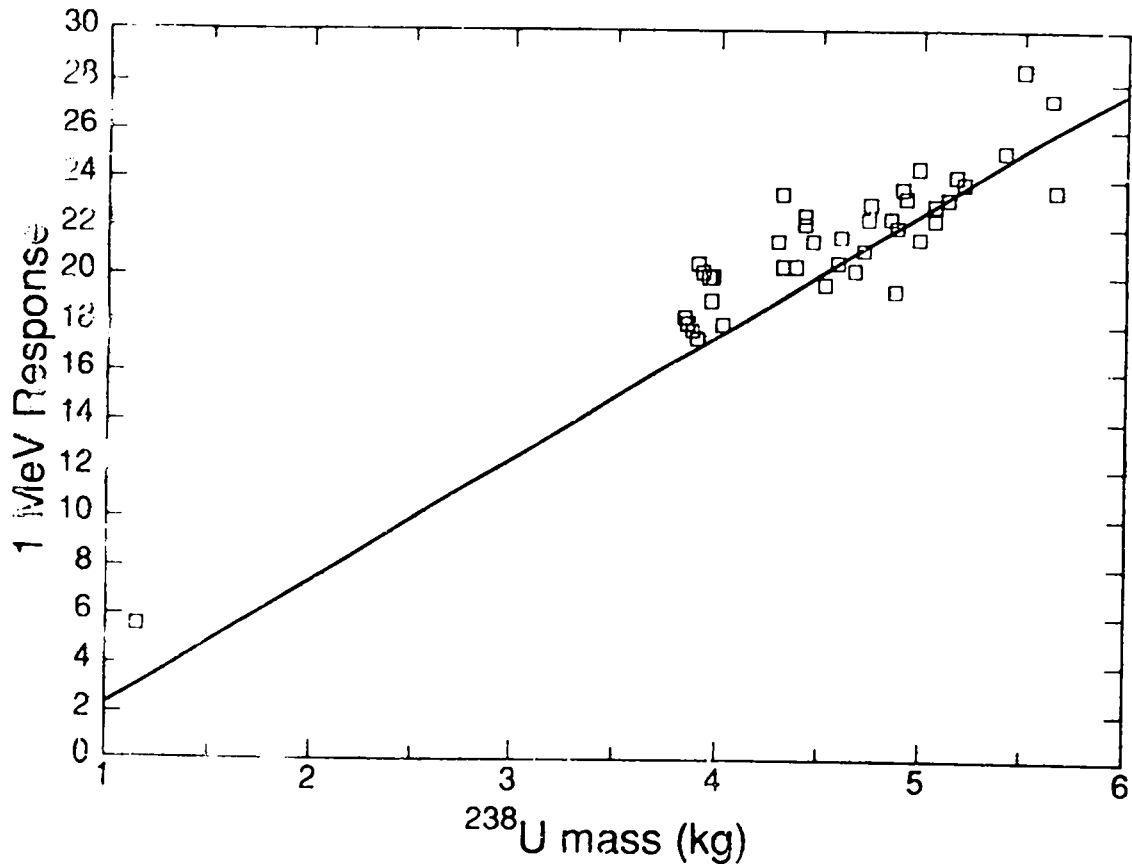
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Fig. 11. Plot of ^{238}U mass vs count rate for 41 samples of the same chemical form. Interchanged mass values have been corrected.

user to distinguish between two similar items in terms of counts/second, while an attempt to calibrate in grams brings in other sources of measurement uncertainty such that the user can only determine that the two items are in fact similar. Despite drifts of 3% to 6% in the results, we easily distinguished pairs of similar items in the Y-12 evaluation. In fact, identical measurements are susceptible to identical calibration errors, which this technique avoids using.

ACKNOWLEDGEMENTS

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