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Weather-Related Variability of Calorimeter Performance in a Poorly-Controlled Environment

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Project Hanford Management Contractor for the
U.S. Department of Energy under Contract DE-AC06-96RL13200

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ABSTRACT

Four Antech airbath calorimeters at the Hanford site were studied for three summers and two winters in a location not well-shielded from outside temperature changes. All calorimeters showed significant increases in variability of standard measurements during hot weather. The increased variability is postulated to be due to a low setting of the Peltier cold face temperature, which doesn't allow the instrument to drain heat fast enough in a hot environment. A higher setting of the Peltier cold face might lead to better performance in environments subjected to a broad range of temperatures.

INTRODUCTION

Calorimetry is used at many Department of Energy (DOE) sites to measure the plutonium content of special nuclear material (SNM). Although the counting time is long, the uncertainty is very low, especially at higher wattages, thus making calorimetry a desirable method for measuring SNM. Because calorimetry measures emitted heat, the stability of the environment around the calorimeter is important. At many cleanup sites, it is not feasible to keep the instruments in a laboratory setting, where temperature (and other factors) can be easily controlled.

Antech calorimeters were used at DOE's Rocky Flats site to complete the Residues and PuSPS projects. The instruments were also used at the Plutonium Finishing Plant (PFP) at the Hanford site to complete the Residues project. At both sites, the instruments were forced to perform in environments without adequate temperature control. At PFP, the calorimeters were qualified and the measurement control limits calculated during the winter months of 2003. When the hot summer months arrived, significantly more variability was seen on all the instruments, which caused numerous non-conformance reports (NCRs) to be generated, and caused recalculation of the control limits. When the cooler weather arrived, the variability subsided. However, another problem surfaced. The mean of the cool weather quality control (QC) standards was slightly different than the mean calculated using the wildly fluctuating summer data. This caused more NCRs to be generated because of "trends" (defined in the WIPP-WAC as eight points in a row above or below the mean). Because the Residues program was in full swing and approaching a milestone, there was not time to explore the origin of the "trends", but the data was deemed acceptable because there were no QC failures and the precision of the standard measurements was very good.

The summer of 2004 again showed an increase in variability that again generated NCRs. When the cooler fall weather came, the lab personnel began joking about the "winter trend" that was to be expected. True to expectations, the lower variability was seen on all the instruments, causing some trend NCRs to be generated. The generation of so many NCRs related to QC standard measurements caught the attention of the WIPP-appointed quality assurance (QA) oversight. Justification had to be made for the multiple trends in the winter and the frequent QC failures in the summer, which resulted in a large amount of paperwork being generated.

This paper shares the data gathered at PFP as well as a possible explanation for the behavior of the instruments. It is hoped that if this phenomenon is observed at other sites, this paper may help eliminate a good deal of extra paperwork either by leading to changes in the instrument settings which may eliminate the problem, or by acting as a reference for data generators when this behavior occurs.

EXPERIMENTAL DETAILS

Instruments and Standards:

Four Antech calorimeters were used in this study: one AR series (AR5), two P series (P13 and P14), and one Q series (Q1). The AR and P series measure heat flow across nickel windings around the measurement cavity, while the Q series uses thermopile technology.

The standards used for QC measurement were electrical standards, which are considered NIST-traceable through the digital volt meter and resistor plate calibration. AR5, P13, and Q1 used the standards at a setting of 0.25 watts. P14 used the standard at a setting of 0.3 watts. QC measurements are reported as raw measured power with no bias correction applied.

Temperature and Weather:

All four instruments were located in the same room, which had inadequate air conditioning and a south-facing outside wall. The air conditioning in the room consisted of two small units on the south wall, only one of which usually worked at any given time. Disposable temperature recorders designed for postal use were used sporadically to measure temperature in the room, mainly during the summer months. Outdoor temperatures referred to in this paper are from the Hanford Weather Station, located a few miles east of the PFP.

Temperatures at the Hanford site vary from 0 degrees Fahrenheit (F) or lower during the winter to well over 100 degrees F during the summer. The room temperature, measured in early winter (2003), spring, summer, and fall of 2004, ranged from 65 F to 84 F, and showed a gradient across the room at any given time of as much as 6 F (taken from a data set of two thermometers on opposite sides of the room monitoring continuously for one month during the early winter).

Selection of Parameters:

The selection of dates to define "summer" and "winter" is highly dependent on individual judgment. As a baseline for this study, the summer was nominally defined as starting the first day of the month during which at least five days reached an outside temperature above 80 F and ending the first day of the month in which there were not at least five days above 80 F. Table 1 shows the temperature distribution on site for the summers that were studied.

The data for Q1 and P14 (both located on the south wall) seem to indicate an earlier onset of summer behavior. The south wall is an outside wall, and is presumably the main source of heat from the outside. The air conditioning units are located on the south wall, but it should be noted that these units were not consistently in service. An earlier date of April 1 for the start of the summer 2004 was also calculated for these instruments, as the data seemed to indicate that the heat began to take effect earlier.

Neither air conditioning units were operational during the summer of 2004. However, they were both repaired on August 18, 2004. The measured room temperature dropped almost 10 degrees after the air conditioning repair, although the outside temperature concurrently dropped. Q1 shows an onset of the "winter" behavior soon after this date, even though there were 15 days above 80 F during the month of September.

The air conditioner above Q1 was functioning throughout the summer of 2005, while the one above P14 was not functional until July 28, 2005. The entry in the lab notebook for July 6, 2005 is typical of room conditions for the summer of 2005: "Room is very warm. Large temperature differential across room. AR5 and SGSAS very warm. Q1 cool." Q1 shows almost no change in variability during the summer of 2005, while the other three calorimeters show a significant change.

Data:

The data for all the instruments is shown in Table 2 and Figures 1-4. The tabulated data is in three sections. The top section gives the average standard deviation of the QC measurements for the nominal baseline summer and winter dates. The bottom sections show the average standard deviation using adjusted parameters. The middle section contains adjusted values for P14 and AR5 based on the exclusion of outlier points believed to be out because of unrelated issues. The adjusted parameters for the bottom section are the dates. The start date of summer 2004 on Q1 and P14, is adjusted to April 1 instead of May 1, and the adjusted end date is August 20, 2004, corresponding to the repair date for the air conditioners. P14 data in the adjusted dates section also has the outliers removed. The data that is considered the best selection of parameters is shown in bold.

Figures 1-4 graphically show all the data used in the study. The seasonal changes are obvious even by the most cursory look. P14 had a couple of unrelated problems during 2004, which may have skewed the appearance of the data a bit. Q1 appears to have only a tiny rise in variability during the summer of 2005, although the data only goes through July 30, 2005 due to a hard drive failure, which put Q1 out of commission for some time.

Start and Finish Dates:

Table 3 shows the beginning and ending dates of the study for each instrument. The dates chosen to begin the study correspond to the instruments first coming online at PFP. AR5 came online first, and a significant amount of "winter" data was gathered in 2003. Q1 was the last qualified, so the beginning of the study is well into the "summer" behavior.

The ending dates for the study are also somewhat widespread. Starting in 2004, the room began to experience serious power spikes which the UPS units did not successfully filter. These power spikes crippled many of the instruments in the room, some for long periods of time. Approximately 10 hard drives were destroyed in a two-year period, as well as monitors, printers, relay boards, and other equipment. The data for Q1 ends on July 30, 2005 due to a series of power spikes that destroyed a good deal of its hardware. P14 data ends on September 9, 2005, and AR5 on April 29, 2006 for the same reason. P13 data through July 28, 2006 is shown on the graph, with visible gaps from February 7, 2004-April 3, 2004 and August 19, 2005-October 7, 2005 due to hard drive failures. AR5 also has a gap from September 8, 2004-October 8, 2004 and P14 has one from June 19, 2004-August 2, 2004. When available, the data is shown into 2006 to give more evidence to the repeatability of the phenomenon over several years.

DISCUSSION

Magnitude of the Ratios

The variability in the summer data ranges from two to three times higher than in the winter. Q1 and P14 have the lowest overall difference. Because the air conditioner over Q1 was functioning properly during the summer of 2005, the summer variation that year was much smaller than in previous years. It is probable that the overall ratio would have been higher if the air conditioner had been in the same state as in 2004. P14 experienced a string of unrelated electrical problems during 2004 that may have slightly affected the ratios, although the effect is not quantifiable. The fact that the temperature effect is still visible through electronic and other instrument problems caused by power spikes indicates that it is a real effect and even suggests that its sole source is not the internal workings of the instruments themselves.

Cause of the Increased Variability

Airbath calorimeters do not directly measure the power of an item over a baseline. Instead, a constant high wattage (called the base power) is maintained at all times. When a heat-generating item is placed in the measurement cavity, the instrument has to input less power to keep the base power because the item is generating part of the necessary power. The difference between the power supplied by the instrument with and without the item is the wattage of the item. The base powers for the AR and P series calorimeters are set at approximately 12 watts and for Q1 is set at approximately 15 watts.

This type of measurement is very precise, but is very dependent on minimizing fluctuations in the base power. The wattage is calculated using the last measured base power value as the nominal base power. Since each measurement can take up to 17 hours, if the actual base power fluctuates while measuring an item, the item measurement will be "off" by the amount of fluctuation in the base power during the measurement (or since the last base power measurement). This may sound like an unreliable way to make a measurement, but in reality the instruments are designed to keep the base power very stable.

A Peltier cooling unit located at the bottom of the instrument is used to drain heat created by the constant wattage out of the calorimeter and into the surrounding environment. The normal laboratory set point for the cold face of this unit is 11.5 Celsius (C), (52.7 degrees F) although a range of temperatures is acceptable for proper functioning. It is this Peltier cold face setting that may be responsible for the hot weather behavior of the calorimeters.

When going back through the run data for some measurements, it was found that often the voltage going to the Peltier cold face was at its maximum for most of the run time, yet the reported cold face temperature was not constant. This suggests that although the instrument was working at its maximum capacity to drain heat out into the environment, it was not able to do so successfully, thus raising the temperature of the acting heat sink (the cold face). This, in turn, could potentially cause the measured base power to fluctuate through a measurement because heat is being "generated" by a third source: the environment, as experienced through the Peltier unit.

Effect on Data

The effect of temperature on the standards will obviously be an equal effect on the accountability measurements. It will increase the uncertainty on the measured values. At higher wattages, the gamma portion of the calorimetric assay measurement contributes significantly more uncertainty than the wattage measurement. However, the effect could become significant at lower wattages.

At PFP, a total measurement uncertainty (TMU) has been developed for calorimetric assay, which incorporates an 8 milliwatt (mW) uncertainty for base power fluctuation. It also includes a 2.5mW uncertainty for wattage fluctuation during the measurement. These and a few other uncertainties combine to give approximately a 10mW (2-sigma) uncertainty on the wattage measurement. The highest observed standard deviation (1-sigma) in this study is 3.4mW, which would lead to a 2-sigma value of about 7mW. Thus, the observed temperature effect, while increasing the uncertainty, is still well within the TMU reported with the measured values.

NCRs

One of the major problems for the PFP program caused by this phenomenon was the generation of a large number of non-conformance reports (NCRs). When the summer data was incorporated into the control limits, the large uncertainty usually led to a calculated mean that was either slightly above or below the true mean of the data. This caused the winter data, which was tightly grouped, to all be on one side of the mean or the other. Because the WIPP-WAC defines a "trend" as eight points above or below the mean, trend NCRs had to be written which were included in all winter data packages. It was recommended by our QA oversight to use only the winter data to calculate the control limits, thus centering in on the correct mean. However, the small uncertainty on the standards during the winter months tightened the control limits to the point that the large variability in the summer would lead to most of the standards being outside acceptable limits. This would require NCRs for violation of measurement control limits. Even when the winter and summer data were combined (which is how the control limits were calculated), the winter data tightened the limits enough to require several NCRs throughout the summer.

Is There a Way Out?

Of course the obvious answer is to maintain a constant laboratory temperature around 65-75 F. However, as many scientists working at closure sites realize, this can be an impossible task! Old buildings in the process of being torn down offer only slightly better working environments than the outdoors.

Installing additional air conditioners near the instruments may also be helpful. Q1 showed a marked improvement in the summer of 2005, while the other instruments continued to show a large variability throughout that summer. This is presumably because the air conditioning unit next to it was repaired prior to the onset of hot weather, and continued working throughout the summer.

When altering the environment is not an option, there is a potential modification on the calorimeter itself which may offer a solution. The Antech calorimeters allow qualified users to manually adjust many of the set points. The Peltier cold face can be set up to about 12.5 C and still function adequately. PFP has not yet implemented this adjustment because of the software requirements of our customers. However, in the future it may be possible to adjust this setting and monitor the performance over the course of a year.

CONCLUSION

Increased variability (and consequently uncertainty) is caused when the environment surrounding an Antech calorimeter is too hot for the Peltier cooler to efficiently dump heat into. This can be a significant problem at sites where the environment cannot be controlled.

The problem might be helped, or even eliminated, by either providing a stable cooler environment or by raising the set point of the Peltier cold face from 11.5 C to 12 C or even 12.5 C (54.5 F). If neither of these solutions is possible (such as was the case at PFP), it is the goal of this paper to serve as a reference when analyzing and reporting trends and/or QC failures that can be attributed to changing temperatures.

Table 1. Summer Temperatures on the Hanford Site (2003-2006)

HANFORD TEMPS (from weather station, Fahrenheit)			
Month/year	Days>80/90/100	Total days>80	Ave temp
May-03	6/2/0	8	74
June-03	14/11/1	26	88
July-03	5/14/12	31	97
August-03	7/20/3	30	92
September-03	10/6/3	19	84
October-03	10/0/0	10	71
November-03	0/0/0	0	48
April-04	3/0/0	3	70
May-04	7/0/0	7	74
June-04	7/9/3	19	85
July-04	5/16/9	30	95
August-04	5/11/10	26	92
September-04	15/0/0	15	79
October-04	3/0/0	3	67
November-04	0/0/0	0	50
April-05	1/0/0	1	68
May-05	4/5/0	9	78
June-05	14/5/1	20	83
July-05	10/13/8	31	94
August-05	8/15/7	30	93
September-05	9/3/0	12	79
October-05	0/0/0	0	66
April-06	2/0/0	2	65
May-06	2/4/2	8	77
June-06	14/3/3	20	84
July-06	5/14/12	31	97

Table 2. Variability Data: Standard deviations of QC Wattages (Watts)

Section 1: Nominal Dates											
Calorimeter	winter 2003	summer 2003	winter 2003/4	summer 2004	winter 2004/5	summer 2005	winter 2005/6	summer 2006	Summer Ave	Winter Ave	S/W ratio
AR5	0.0007	0.0016	0.0006	0.0031	0.0007	0.0017	0.0005	n/a	0.0021	0.0006	3.331
P13	**n/a**	**0.0025**	**0.0009**	**0.0034**	**0.0013**	**0.0026**	**0.0007**	**0.0015**	**0.0028**	**0.0011**	**2.697**
P14	0.0013	0.0019	0.0019	0.0019	0.0009	0.0026	n/a	n/a	0.0021	0.0013	1.5907
Q1	n/a	0.0032	0.0014	0.0014	0.0004	0.001	n/a		0.0019	0.0009	2.0736
Section 2: Outliers Removed											
AR5	**0.0007**	**0.0016**	**0.0006**	**0.0023**	**0.0007**	**0.0017**	**0.0005**	**n/a**	**0.0019**	**0.0006**	**2.92**
P14	0.0013	0.0019	0.0013	0.0019	0.0009	0.0026	n/a	n/a	0.0021	0.0011	1.9518
Section 3: Altered Dates (outliers removed)											
P14 early summer	0.0013	0.0019	0.001	0.0019	0.0009	0.0026	n/a	n/a	0.0021	0.001	2.0766
P14 early winter	0.0013	0.0019	0.0013	0.0021	0.001	0.0026	n/a	n/a	0.0022	0.0012	1.9037
P14 early both	**0.0013**	**0.0019**	**0.001**	**0.002**	**0.001**	**0.0026**	**n/a**	**n/a**	**0.0022**	**0.0011**	**2.065**
Q1 early summer	n/a	0.0032	0.0011	0.0015	0.0004	0.001	n/a	n/a	0.0019	0.0008	2.4973
Q1 early winter	n/a	0.0032	0.0014	0.0015	0.0004	0.001	n/a	n/a	0.0019	0.0009	2.111
Q1 early both	**n/a**	**0.0032**	**0.0011**	**0.0016**	**0.0004**	**0.001**	**n/a**	**n/a**	**0.0019**	**0.0008**	**2.5248**

Table 3. Beginning and Ending Dates and Other Information

Calorimeter	Start date	End date	Out of service dates	outliers
AR5	2/15/2003	4/29/2006	9/8/04-10/8/04, 3/10/06-4/8/06	8/6/04, 9/1/04, 9/2/04
P13	5/17/2003	7/28/2006	2/7/04-4/3/04, 8/19/05-10/7/05	none
P14	4/6/2003	9/9/2005	6/19/04-8/2/04	4/14/2004
Q1	5/25/2003	7/30/2005	none	none

Figure 1. AR5 Data

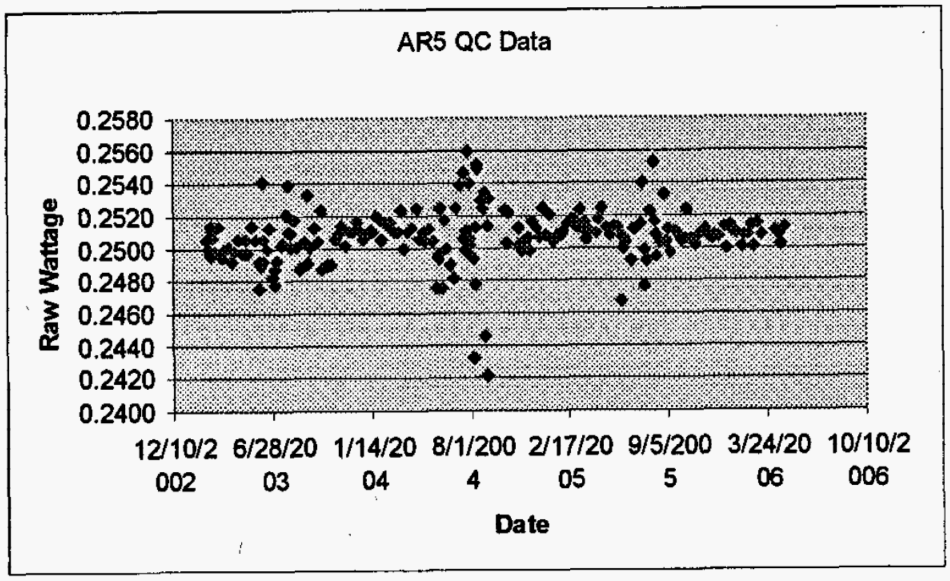

Figure 2. P13 Data

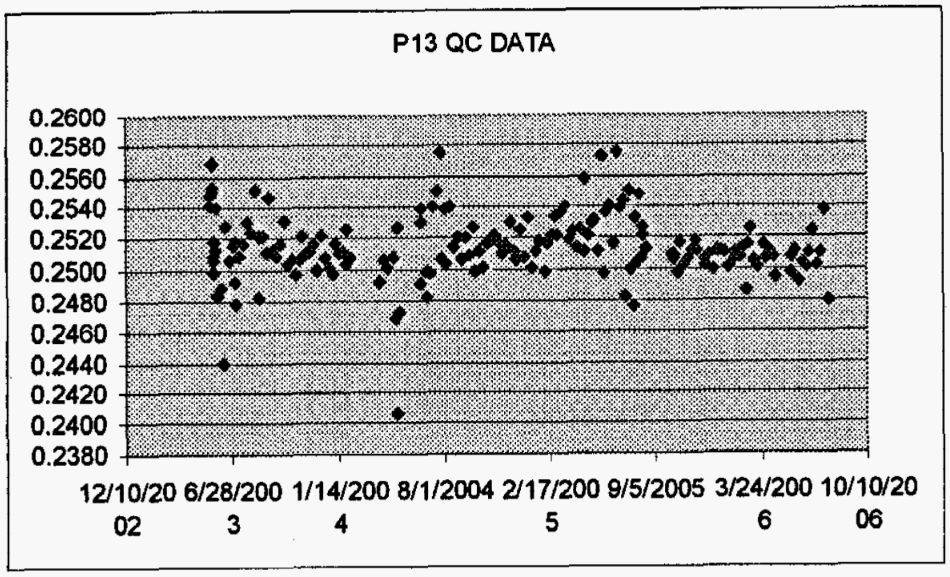

Figure 3. P14 Data

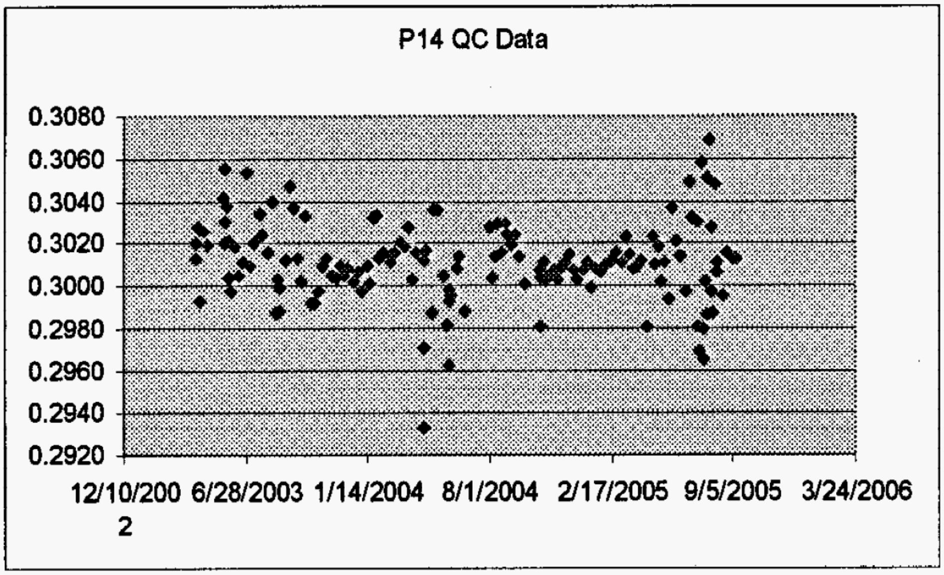

Figure 4. Q1 Data