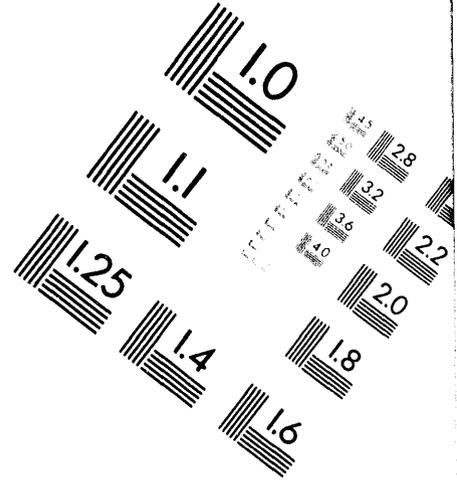
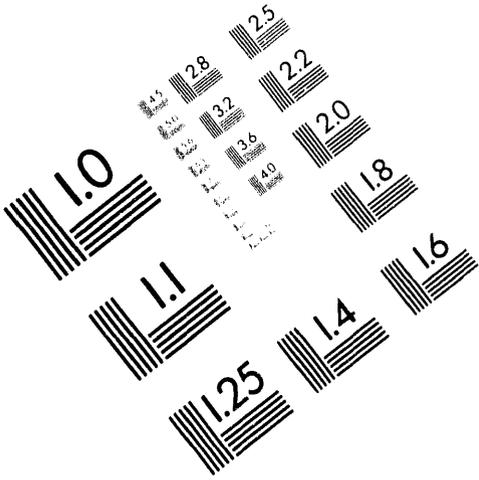




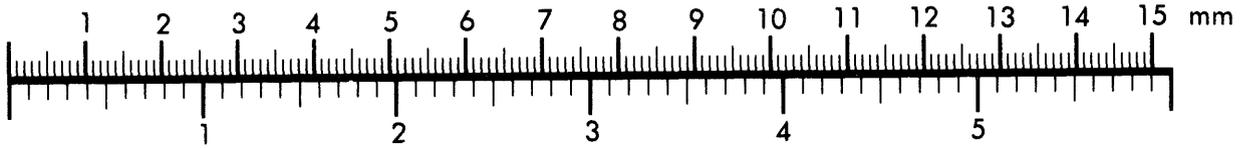
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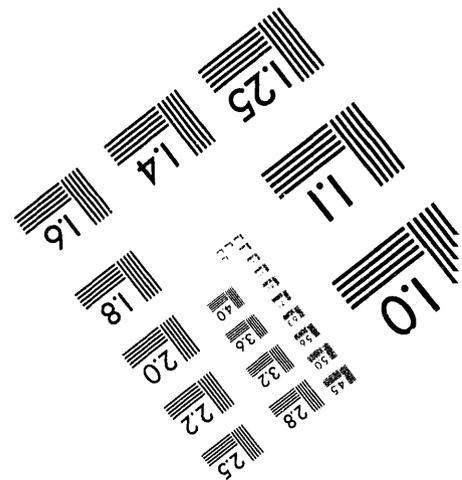
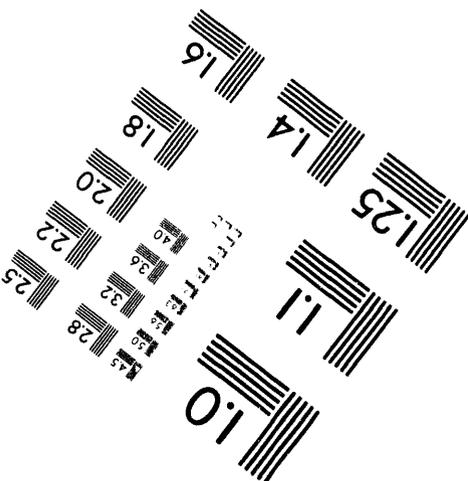
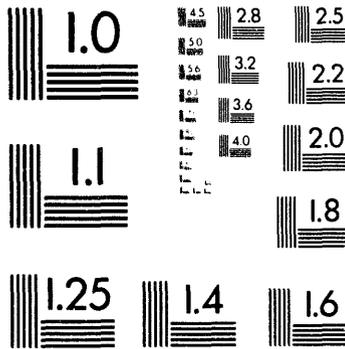
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**MEASUREMENT CONTROL DESIGN AND  
PERFORMANCE ASSESSMENT IN THE  
INTEGRAL FAST REACTOR FUEL CYCLE\***

by

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# MEASUREMENT CONTROL DESIGN AND PERFORMANCE ASSESSMENT IN THE INTEGRAL FAST REACTOR FUEL CYCLE

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## ABSTRACT

The Integral Fast Reactor (IFR) --consisting of a metal fueled and liquid metal cooled reactor together with an attendant fuel cycle facility (FCF) -- is currently undergoing a phased demonstration of the closed fuel cycle at Argonne National Laboratory. The recycle technology is pyrometallurgical based with incomplete fission product separation and all transuranics following plutonium for recycle. The equipment operates in batch mode at 500 to 1300°C. The materials are highly radioactive and pyrophoric, thus the FCF requires remote operation. Central to the material control and accounting system for the FCF are the balances for mass measurements. The remote operation of the balances limits direct adjustment. The radiation environment requires that removal and replacement of the balances be minimized. The uniqueness of the facility precludes historical data for design and performance assessment. To assure efficient operation of the facility, the design of the measurement control system has called for procedures which assess the performance of the balances in great detail and will support capabilities for the correction of systematic changes in the performance of the balances through software.

## INTRODUCTION

In the Fuel Cycle Facility (FCF) associated with the Integral Fast Reactor (IFR) system[1] the measurement of mass is the most common and basic physical measurement, and is central to the material accounting system. The instrument for determining the mass of an item is an electronic balance. Of interest are its calibration, that is an estimate of the true mass on the balance and the associated uncertainty in the estimate, and the control of that calibration, that is tests of whether the current state of the balance conforms to the state at calibration.

Although an electronic balance for measuring mass is a common instrument in laboratories and industrial facilities, its application in the FCF requires some special considerations. The balances will operate in a high radiation environment with changing temperatures and pressures. This requires that some electronic components, which could be adversely affected by such an environment, be separated from the balance and placed outside of the cell. This is non-standard practice for commercial balances, and its consequences on the performance of the balances will be addressed. The balances will be remotely calibrated and operated, whether this introduces any significant operator bias will be investigated.

In addition, the facility is a new design and, therefore, at best, very little operational or historical data exists for anticipating performance problems which may develop during actual operation. For this reason, the procedures for calibration and control are more elaborate than for other installations at ANL-W, and will assure a clear characterization of anomalous behavior. If it transpires that a balance has stable and predictable anomalous characteristics, the detailed analysis of these characteristics may allow correction for this without immediate removal of the balance and interruption of operation.

## GENERAL MODEL OF A LINEAR BALANCE

Because incell installation of the balances requires tampering with the electronics of the balances and the effects of irradiation and temperature are unknown, there is no assurance that the manufacturer's calibration procedures and parameters remain valid. To overcome this we return to the model for a simple linear balance shown in Fig. 1.

To make a measurement of a mass, we need a relationship between the mass placed on the balance ( $M$ ) and the reading given by the balance ( $W$ ), as shown in Fig. 1. Since we do not know a priori that the reading ( $W$ ) given by the balance corresponds exactly to the mass ( $M$ ) on the balance, for modern balances we assume in general a linear relationship.

The linearity of FCF balances, over the range of operational measurements for material control and accountability, is a necessary condition. DOE in order 5633.3A requires that each balance must be "checked ... for linearity on each day that the scale or balance is used for accountability purposes".[2] Thus, the certification that the balance conforms to a linear relationship over the range of application is a critical element of calibration.

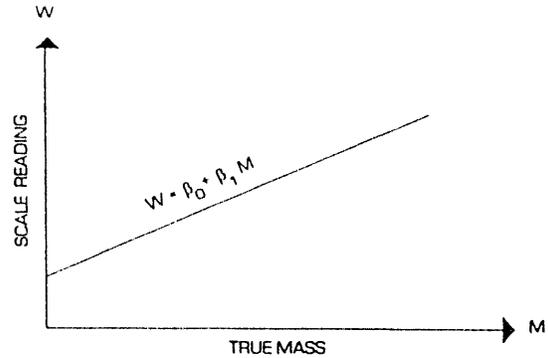


Fig. 1. General Model of a Linear Balance

In the context of Fig. 1, if we knew the parameters  $\beta_0$  and  $\beta_1$  exactly, which characterize a particular balance, we could determine the true mass of the object on the balance by

$$M = \frac{W - \beta_0}{\beta_1} \quad (1)$$

This relationship[3] is central to the issues of

1. Calibration
2. Linearity checks
3. Estimation of the measurement error.

Since the transformation  $W \rightarrow M$ , as given by Eq. 1, is dependent on the parameters  $\beta_0$  and  $\beta_1$ , the above issues will depend on our knowledge of these parameters. Thus, preparation of the balance for the first measurement of an unknown mass revolves around determining  $\beta_0$  and  $\beta_1$ , and their uncertainties.

## CALIBRATION

In principle, calibration of a measurement instrument results in estimates of the bias, the variance in the bias estimate (the systematic-error variance), and the random-error variance.[4] These estimates are based

on repeated measurements of standard masses on the balance. The choice of standard masses is often such that they are equally spaced over the range of application of the balance. The exact number is determined during preliminary evaluations based on a confidence coefficient[5], and on a linear fit test to assure the validity of the linear model. The sequence of measurements of the mass of the standards is randomized to eliminate biases with respect to the selection process.

The parameters  $\beta_0$  and  $\beta_1$  are estimated by regressing the balance reading on the mass of the standard masses. This procedure gives estimates  $\hat{\beta}_0$ , and  $\hat{\beta}_1$ , so that

$$w = \hat{\beta}_0 + \hat{\beta}_1 S, \quad (2)$$

and we also obtain estimates  $\text{Var}(\hat{\beta}_0)$ ,  $\text{Var}(\hat{\beta}_1)$ ,  $\text{Cov}(\hat{\beta}_0, \hat{\beta}_1)$ .

These are used to make inferences about the true mass via (1).

#### **ANALYSIS OF CALIBRATION MEASUREMENTS OF IN-CELL BALANCE 400A**

Mass measurements of three standard masses (10 kg, 20 kg, and 30 kg) were made in-cell on balance 400A. The masses were selected in random order and the operators were requested to simulate, as much as possible, the conditions which would exist during operation. That is, all weighings of standards are assumed to duplicate routine use of the balance with respect to load, temperature, humidity, vibration, loading procedure (such as positioning) and so forth. This is especially critical during the testing period, where primary consideration needs to be given to making certain that the effects of all process variables are taken into account, so that the error limit is representative of the mass measurement process.

It is desirable, due to the remote operation of the in-cell balances, to keep the number of measurements to a minimum. A total of forty measurements was tentatively deemed adequate and resulted in a 98% confidence coefficient.[5] That is, this sample size insures a 98% chance of the sample standard deviation  $s$  being less than  $1.24 \sigma$ , where  $\sigma$  is the population standard deviation. Testing results and future considerations may alter this choice.

The results of the forty measurements of randomly selected standard masses were analyzed with SAS[6], which is the analysis software for all mass measurements in FCF.

The regression procedure of the balance readings  $W$  on the standard masses  $S$  resulted in an estimate of the intercept  $\beta_0$  of 2.571 g, and slope  $\beta_1$  of 0.9998. The p-value under the null hypothesis  $\beta_0 = 0$  is 0.0065, and therefore the estimate is statistically significant. The p-value under the null hypothesis  $\beta_1 = 1$  is 0.0004, and thus also statistically significant. Correction of the balance reading is, therefore, warranted.

In any evaluation of a measurement instrument, the estimation of the bias is a critical element. The equality of the bias with respect to the different standards is an indication of the performance of the instrument. The latter can be estimated through an analysis of variance of the standard minus the balance reading with the mass of the standard as the fixed effect. For standards  $S = 10000$  g and  $S = 20000$  g the p-value for the null hypothesis of mean bias equal to zero are 0.22 and 0.76 respectively, and, thus, support the null hypothesis. On the other hand, the p-value for  $S = 30000$  g is 0.0002, which indicates the null hypothesis to be highly unlikely.

The effectiveness of a correction, that is, taking the estimates of  $\beta_0$  and  $\beta_1$  into account, is illustrated by an analysis of variance of the residuals. The p-value of 0.8648 strongly supports the null hypothesis

that the estimated linear model explains the variation of balance reading with regard to a mass placed on the balance.

A regression of the bias on the observation number indicates a slight drift of -0.049 gm/observation. This drift is not removed by correcting the measurements with  $\hat{\beta}_0$  and  $\hat{\beta}_1$ . A test of the hypothesis Mean Drift = 0, however, results in a p-value of 0.183. This result, therefore, is sufficiently likely at the 5% level of confidence that we can accept the null hypothesis.

Since bias correction of measurements has not yet been adopted by FCF operations\*, the only statistics pertinent for the measurement uncertainty analysis are the random error variance and the systematic error variance. To this end we propose the following procedure for calculating these statistics.[7] This approach is dependent on the assumption that measurement errors are independent of the standard both with respect to bias and the error variance. This is not the case with respect to some of the balances. This in itself may not be sufficient to preclude this approach to estimation. However, caution in its application is warranted.

To calculate the error variances we form the following quantities. Let  $S_k$  be the mass of the k-th standard and  $W_{ik}$  the i-th measurement of the k-th standard. The k-th standard mass is estimated by the average  $\bar{W}_k$  where

$$\bar{W}_k = \frac{1}{m_k} \sum_{i=1}^{m_k} W_{ik}, \quad (4)$$

---

\*"plus ça change plus c'est les mêmes choses" John L. Jaech in his 1973 text Ref. 4 warned "no laboratory or operations manager reacts favorably to the idea of making very small bias corrections."

and the k-th variance  $s_k^2$  where

$$s_k^2 = \frac{1}{m_k - 1} \sum_{i=1}^{m_k} (W_{ik} - \bar{W}_k)^2, \quad (5)$$

where  $m_k$  is the number of measurements of the k-th standard. The bias in the estimate of the mass of the k-th standard is computed as

$$\theta_k = (\bar{W}_k - S_k), \quad (6)$$

We can then compute a weighted bias  $\theta$  and a weighted estimate of the variance  $s^2$  for K standards as

$$\theta = \frac{1}{n} \sum_{k=1}^K m_k \theta_k \quad (7)$$

and

$$s^2 = \frac{1}{n - K} \sum_{k=1}^K (m_k - 1) s_k^2, \quad (8)$$

where  $n = \sum_{k=1}^K m_k$  is the total number of observations. Then our estimate of the random error variance is  $s^2$ . With regard to the systematic error variance, we use  $\theta^2$  since no bias correction is applied. However, if

$$\theta^2 < \frac{1}{n^2} \sum_{k=1}^K m_k^2 \sigma_k^2 + s^2/n \quad (9)$$

the right hand side of the above inequality is used as the systematic error variance. The  $\sigma_k$  is the assigned uncertainty to the k-th standard.

## EFFECT OF INTERNAL CALIBRATION

The balances proposed for the Fuel Cycle Facility (FCF), and their operational

use, introduce an issue which is unique to FCF operation. Namely, the balances are designed to be operated with an internal calibration. This internal calibration is to be performed before each measurement, and for which the balance must be empty. During FCF operation, however, many weighings are expected to be performed under situation where the internal calibration will not be possible. The natural question to ask is, what is the difference between a measurement for which an internal calibration was performed and one for which it was not. That is, can we assign a bias and an error estimate to measurements made under this condition.

To study the question of the effect of the internal calibration on the overall calibration a series of mass measurements were made of a 5 kg standard on a Mettler K-series balance. First a sequence of 25 measurements of a 5 kg standard were made with an internal calibration before each measurement, then a sequence of 25 measurements of a 5 kg standard without an internal calibration before each measurement. The first measurement in this latter series was preceded by a measurement with an internal calibration. Qualitatively, the dispersion of the measurements without an internal calibration is much greater than that of the measurements which were preceded by an internal calibration. For these two samples, the bias also appears to be somewhat greater in the case of no internal calibration.

In addition, 50 measurements of a 5 kg standard were made where internal calibrations before each measurement have been performed at random with a probability of an internal calibration of 0.5. In this case the dispersion lies between that of the series with internal calibration and that without. Thus, the internal calibration appears, at least qualitatively, to influence the measurements without internal calibration. If we separate the measurements into two sets—one consisting of those with internal calibration

the other of those without internal calibration, qualitatively, we can say that the sequential random interspersions of noninternally calibrated measurements appears to have had little effect on the dispersion of the internally calibrated measurements. Looking at it from the other side, however, the sequential random interspersions of internally calibrated measurements into a sequence of noninternally calibrated measurements reduces the dispersion in the noninternally calibrated measurements.

To assure that the sequential data have no systematic time behavior, and can, therefore, be used as cross section data, the autocorrelation functions for the sequences were calculated. Qualitatively the autocorrelations for the internally calibrated series appear random. This is confirmed by the p-value for the autocorrelation check for white noise. On the other hand, the series with noninternally calibrated measurements shows a statistically significant correlation at lag 1 and a distinct nonrandom pattern at higher lags. This is confirmed by the very small p-value. The nonrandom pattern in the autocorrelation function suggests that the series is nonstationary. This is supported by the fact that, if we difference the data, the autocorrelation function indicates that the resultant series is white noise with a p-value of 0.782. It is likely that there is a probabilistic drift in the mean. Such a short term probabilistic systematic error will be reflected in a larger variance in the long term.

These results support, perhaps not conclusively at this point, that during the calibration of the balance we can intersperse, at random, the measurement of the standards, with an internal calibration with some fixed probability  $p$ . This will result in a calibration curve and a predictive interval which we can apply to the operation of the balance in the FCF.

The linearity assumption can be tested through the standard goodness-of-fit test. The minimum requirement is that there be at

least two measurements for each standard weight, which will be satisfied to high probability by the calibration data.

This test compares the variation of the data about the regression line to that of the mean behavior of the random variable  $W$  at each standard weight  $s_j$ . For balance 400A, our paradigm, the p-value for the null hypothesis (that the balance is linear) is 0.592.

## ROUTINE CALIBRATION AND LINEARITY CHECKS

The procedure described above tells us that the assumed model is appropriate in describing the population of responses of the balance to measurements of unknown masses, and gives us the estimate of the variance which we associate with the estimated mass. The balance is out of calibration, if the new sample of responses is statistically different from that on which the calibration was based.

Thus, a calibration check can be based on periodic random measurements of the same standard weights used in the calibration. If the observed value falls within the predictive interval at some level of confidence, we can conclude, with that level of confidence, that the population of responses has not changed. This is shown graphically in the following figure.

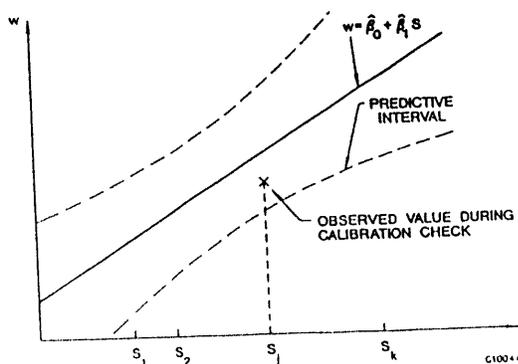


Fig. 2. Predictive Interval

We note the predictive interval is non-linear, and most narrow at  $\bar{S}$ , the grand mean of the sample data (where the information is most dense), and get progressively wider away from that mean. This is because the interval reflects probability--the farther from the sample data the less confident we are about a model estimated with that data.

The calibration data is used to calculate the predictive interval for the calibration. This interval is given by

$$w_j \pm t_{\alpha/2, k-2} s_j, \quad (14)$$

where  $w_j$  is the predicted value for the  $j$ -th standard weight based on the calibration curve, and  $s_j^2$  is the estimate for the variance in the future observation.

The predictive interval lends itself to statistical control use.[8] The basic idea of a predictive interval is to choose a range in the distribution of  $W$ , the balance readings, wherein most of the observations will fall, and to declare that the next observation will fall in this range. If it falls outside this range there is an indication that a change in the performance of the balance has occurred, and it may be necessary to look for an assignable cause.

## MEASUREMENT CONTROL OF IN-CELL BALANCE 400A

The predictive interval is used to signal the possibility that a shift has occurred in one or more of the population parameters, which describe the relationship of the instrument reading to the true mass. Thus, an instrument reading of a standard mass outside the predictive interval warrants a search for a possible cause for this shift and its probability.

In general, the out-of-control behavior of the balance manifests itself as an increase in the dispersion of the instrument readings

about the mean, or a shift in the bias, or both. No one test can give a definitive answer as to which of these possibilities are the cause of the out-of-control behavior. The mass balances are an integral part of FCF operation, thus cessation of operation to recalibrate the balance, or remove and replace it, can incur considerable cost.

Control charts give a holistic view of the performance of the balance. If sufficient calibration checks have been accumulated before there is an indication of out-of-control balance performance, a direct test of calibration can be made.

Since the calibration check measurements form an independent sample, they will have their own regression line. We can then test whether the two regression lines, the original calibration and the one based on the current sample, are the same. That is, we test the hypothesis

$$H_0 : \beta_{01} \text{ and } \beta_{02} \text{ and } \beta_{11} \text{ and } \beta_{12}$$

$$H_A : \text{Either } \beta_{01} \neq \beta_{02} \text{ or } \beta_{11} \neq \beta_{12} \text{ or both.}$$

If the two regression lines are the same, both the intercept and slope terms must be equal. If the regression lines are not the same, they must differ with respect to either the intercept or the slope or with respect to both.

To minimize cost of unnecessary removals of balances, a number of different tests, such as control charts, for example, are used to gain confidence that a true problem exists and give an indication as to the source. The existence of readily available modern computer software for statistical analysis,[6] allows quick and extensive testing of hypothesis with regard to the population parameters.

A measurement control procedure described previously was implemented during the initial startup and testing of the FCF. Over that period 45 calibration check mea-

surements were made on in-cell balance 400A. The measurements span a period of six months. Over this period no measurement exceeded the  $3\sigma$  control limits, and only on two occasions, at measurements 28 and 30, was the  $2\sigma$  control limit exceeded. A Stewhart control chart for the bias and a moving range chart for these data, generated with the SAS/QC procedures, show that the mean bias and the variance appear to differ from those established at calibration for measurements with this instrument.

The V-mask parameters for the CUSUM chart were set to detect a change in the mean bias of  $0.5\sigma$  with a Type I error probability of 0.05 and a Type II error probability of 0.2. For no change in the mean bias, at these parameters, the average run length (ARL) is 209, while with a deviation of  $0.5\sigma$  the ARL is 24.

SAS/QC performs a number of runs tests,[9] whose purpose is to detect a particular nonrandom pattern in the points plotted on the control chart. In the series of calibration check measurements, the first indication of a possible problem would have occurred at the 9th measurement. The information which we would have had at that point would not have indicated a problem. The Shapiro-Wilk statistic would have had a p-value of 0.81 indicating normality. Visually the data indicate a downward shift in bias and a variance smaller than expected. The cusum chart, however, does not indicate an out-of-control condition, for the V-mask is not crossed by the sequence of cumulative sums. This is consistent with the parameters of the V-mask, which correspond to an average run length of 24 measurements for an out-of-control condition, while we have only nine at this point. In addition to this, the test comparing the regression parameters has a p-value of 0.257 which suggests the parameters are based on samples drawn from the same population.

The next signal would occur at measurement 22.

Visually the data continue to show a downward shift in the bias. At this point this is confirmed by the cusum chart. In addition, the p-value for the comparison of regression coefficients is 0.000077. Clearly indicating that the populations are different.

The box plot in Fig. 3 indicates a systematic trend in the bias and therefore the possibility of correcting these measurements via Eq. (1). The efficacy of this correction is given in the box plot in Fig. 4.

### CONCLUSIONS

We have presented a methodology for the statistical evaluation of electronic balance measurement data in FCF for determining control limits, calibration limits, and precision and accuracy levels. This methodology ensures the quality of measurement and measurement control data and provides estimates of uncertainty. FCF startup operational data of in-cell balance 400A was used to demonstrate the statistical techniques to determine total random error, the measurement biases, the systematic error, control limits, and decision points. Major assumptions under which the methodology is applicable have been identified and their validity investigated.

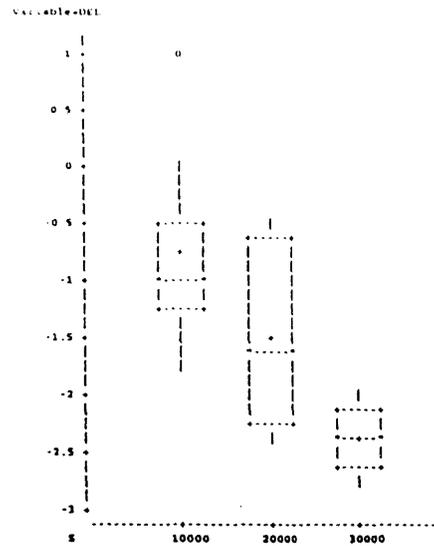


Fig. 3. Box Plots of Uncorrected Bias

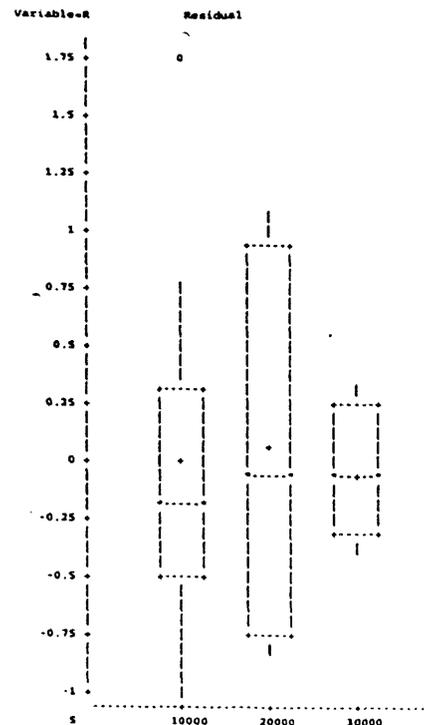


Fig. 4. Box Plots of Residuals

## REFERENCES

1. C. E. Till and Y. I. Chang, "The Integral Fast Reactor Concept," Proc. American Power Conference, 48, Chicago, Illinois (1986).
2. Department of Energy (DOE) Order 5633.3A, Control and Accountability of Nuclear Materials, Chapter II, Section 4.e.1.a. (February 22, 1993).
3. "Statistical Methods for Nuclear Material Management," W. M. Bowen and C. A. Bennett (eds.), NUREG/CR-4604 (December 1988).
4. John J. Jaech, "Statistical Methods in Nuclear Material Control," United States Atomic Energy Commission (1973).
5. ANSI N15.18-1988, American National Standards Institute, Inc.
6. Statistical Analysis System, Version 6, SAS Institute Inc., Cary, NC.
7. IAEA Safeguards Technical Manual IAEA-TECDOC-227 (1980).
8. John Neter and William Wasserman, "Applied Linear Statistical Models," Richard D. Irwin, Inc. (1974).
9. SAS/OC \*Software: Reference, Version 6, First Edition, SAS Institute Inc., Cary, NC.

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