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EVALUATION OF PROCESS INVENTORY UNCERTAINTIES

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ABSTRACT

This paper discusses the determination of some of the process inventory uncertainties in the Fast Flux Test Facility (FFTF) process line at the Los Alamos Scientific Laboratory (LASL) Plutonium Processing Facility (TA-55). A brief description of the FFTF process is given, along with a more detailed look at the peroxide precipitation and re-dissolution (PR) process. Emphasis is placed on the identification of the product and sidestreams from the unit processes, as they have application to the accountability measurements. The method of measurement of each of the product and sidestreams and their associated uncertainties are discussed. Some typical data for the PR process are presented, along with a discussion of the data. The data presented are based on our operating experience, and data on file in the TA-55 Nuclear Material Accountability System (PF/LASS).

INTRODUCTION

The Los Alamos Scientific Laboratory has a program requiring the conversion of plutonium metal into reactor grade plutonium dioxide being carried out at TA-55. The major portion of the product from this process line is shipped to another contractor's facility for the production of fuel rods for the FFTF.

Figure 1 is a material flow diagram for the FFTF process at TA-55 showing the unit processes with feed, product, and sidestreams for each of the associated steps. The nondestructive assay (NDA) measurement methods used to determine the amount of material which crosses unit process boundaries are also identified. The FFTF process is a series of unit processes of the type shown in Figure 2, and whenever material crosses a unit process boundary it is moved at a measured value. These measured values are entered into PF/LASS by means of on-line terminals located throughout the process area of TA-55.

This work was performed under the auspices of the U. S. Department of Energy.

AUDIT TRAILS

We have developed a scheme for tracing the movement of materials through the TA-55 facility so as to be able to present an audit trail that serves several useful purposes. One of the purposes of this audit trail is to provide data to assist in the evaluation of process uncertainties. The scheme we use is an expanded and refined version of the technique described in reference 1. Through the use of this technique we are able to identify the sidestreams and product lots associated with a given batch of material as it flows through the FFTF process line.

One of the sidestreams associated with each of the unit process areas is a sidestream labelled MIPxx, where the "xx" is the unit process or receipt area designator. The term MIP is an acronym for Material In Process, and is the summation of the unmeasured sidestreams (primarily, and ideally, the process holdup). Using the data provided by our tracing scheme, we are able to present data associated with individual lots and for cumulative data from a unit process. This data can be presented in several different ways, some of which will be discussed below to show several ways this information can be used in the evaluation of process uncertainties.

Figure 3 is an example of an audit trail for a batch of material as it passed through the FFTF process and shows the relative complexity of this process, which is one of our more straight forward processes. This figure is essentially the same as Figure 1, except that the mass, instrument code, and details have been inserted for each of the material flow paths, and there are no scrap values assigned. It is readily apparent that even when the uncertainty of each of these measured values is known, or can be reasonably estimated, calculation of the uncertainty in the material balance for any lot is a very complicated and cumbersome calculation requiring sophisticated statistical techniques. The calculation is further complicated by the fact that in several of the unit processes, the input and output are both measured using the same instrument with different chemical and nuclear properties and with an unknown statistical distribution. Figure 4 is an example of the measurements associated with one lot through the PR process.

SEQUENTIAL PROCESS LOT DATA

Figures 5 through 8 are plots of accumulated MIP's associated with 24 lots as they passed through four of FFTF unit processes. Figure 9 is the summation of the data in Figures 5 through 8. A look at these five sets of data show that:

1. For the oxide dissolution (OD) process the accumulated MIP behaves much as one would expect, in that it builds up to some level and then tends to level off, and is indicative of classical process holdup.
2. For the PR process there appears to be a consistent MIP "loss". This trend could be the result of numerous causes; such as measurement bias, sampling error, holdup, unmeasured sidestreams, etc.
3. For the oxalate precipitation (OY) process there is a MIP "loss" for the first eight batches and then a MIP "gain" for the remainder of the process.
4. For the hydrocalcination (HC) process there is a consistent MIP "gain".
5. The cumulative MIP "loss" for these 24 batches is almost completely dominated by the "loss" in the OD process. A result which at first glance may be very difficult to understand.

As noted above the cumulative values for the process are very closely related to the OD process, and as one would expect the trend is a slightly positive slope. These two observations indicate that the apparent losses and gains in the other three processes may be related in some way as indicated in (2) above. In fact following a cleanout for inventory a six months comparison for these four processes indicated an ~320 gram gain with an average throughput of ~196 kilograms, or 0.17% processing gain. When there is a significant processing loss, or gain, in a unit process followed by a gain or loss in succeeding processes, it is indicative of a problem of some nature in your measurement or accountability scheme.

When the phenomena described above was first observed at TA-55, the first assumption made by the processing personnel was that there was an obvious bias in the measurement instrument used in the PR process. While this is not a bad assumption, it turned out not to be the case. Investigation by several persons and groups, independently determined that the problem was indeed associated with the measurement of the solutions into and out of the PR process. However, it was conclusively shown that the NDA instrument in use, a solution assay instrument (SAI), was measuring plutonium values that were consistent with values determined by conventional wet chemistry techniques. The investigation did lead to an evaluation of the steps in the operation and it was discovered that there was a sampling problem associated with the measurement

that was consistent with the observed results. It is anticipated that the application of schemes such as the one being discussed in this paper, and others being pursued by the LASL, will lead to an early detection of these types of problems by making use of accountability data coupled with process information.

CUMULATIVE DATA FOR PR PROCESS

Let us now examine another facet of the same problem from a slightly different perspective using more data from the accountability system. Figure 10 is a plot of the cumulative MIP for the PR process from startup and continuing for the first six months of operation. Process lots are identified on Figure 10 by circles, and sidestreams (process cleanup) are identified with *'s. It will be observed that as discussed earlier there is the expected buildup, and as cleanout and scrap are removed from the process line and measured, the trends in the slope of the cumulative MIP are as expected. However, while the slope is of the generally expected shape it is much steeper than desired or expected, indicating "losses" in excess of those anticipated by processing history and experience. At batch # 86 cleanup for inventory was complete and the amount of holdup in the box would have an expectation value of nearly zero. From Figure 10 it can be seen that the cumulative value is ~940 grams. The throughput for this same period was ~64 kilograms yielding a 1.47% process loss. The average MIP for these lots is $2.4 \pm 6.0\%$ of the throughput. Since a value of "zero" at cleanout is expected, a value of 940 grams would be considered extremely high. Examination of the companion process data, as stated earlier, reveals that this "loss" is offset by a "gain" in subsequent processes (namely OY and HC). When calculated by "conventional" techniques the 2-sigma value for the uncertainty in the holdup is of the order of 230 grams. Under the isolated circumstances of one piece of information this situation would call for an investigation into the "loss" since it is 8-sigma above the "zero" value. The investigation that was conducted and discussed earlier was started before the data presented here were all available.

The problem that remains is clearly indicated by the data that are presented in Figure 10, that is, what do you do with the cleanout value of 940 grams? Since the value could be assumed to be acceptable based on other related data, the real problem is in what does one do with this kind of a value in an isolated instance? Several methods of calculating the uncertainties associated with nuclear materials control have been proposed over the last several years. Most of them focus their attention on the area under the statistical distribution

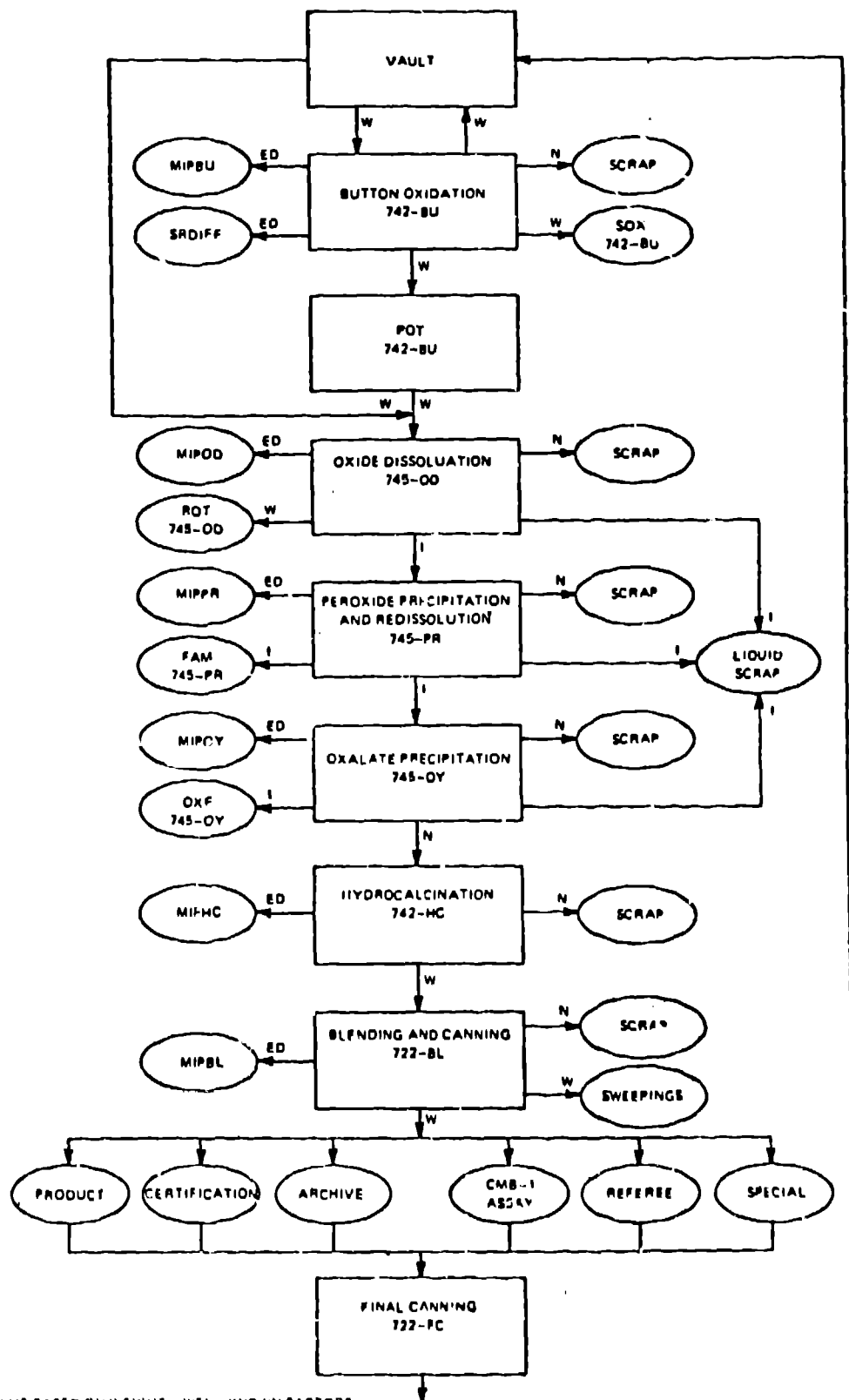
curves that are considered to be normally distributed. In a recent study at the LASL by Johnson and Tietjen², they have shown that the difference of two slightly non-normal, but not detectable from normal, distributions can generate some interesting values. For example, they show that the number of process uncertainty investigations would increase from 5 to 7.5% and from 1.0 to 1.9% for the 95 and 99% confidence levels respectively, for the case they discuss. When one focuses on the area under the curve this difference is only 2.5 to .9% greater than the expected value, but when one looks at the area under the tails of the curve it represents an increase of 50 and 90% respective for the 95 and 99% levels of interest. Anyone associated with performing, or funding, process uncertainty investigations can readily appreciate the significance of this effect.

CONCLUSION

In light of the specific cases discussed above, it is our conviction that conventional error propagation and/or the use of percent of throughput are not very useful in several aspects of nuclear materials accountability and safeguards at TA-55 and other non-routine process areas at the LASL. It is our intent at the LASL to perform a timely and technical evaluation of all process uncertainties, and to respond to this evaluation in the appropriate manner. The extremes of the response would range from no action to the complete shut down of a process and performing a very comprehensive process loss investigation and evaluation. Briefly, the current thinking is that a valid approach is to simulate by an appropriate technique the errors associated with a series of measurements and assign a probability to the observed value. The response would then be dependent upon parameters such as the probability calculated, amount of process uncertainty, attractiveness of material in question, etc. It is anticipated that the results of some preliminary studies of this approach will be completed shortly and reported in Nuclear Materials Management.

REFERENCES

1. R. G. Bearse et al, "Computer Assisted Audit Trails on the Los Alamos DYMAC System", LA-UR-80-899, Submitted to Nuclear Materials Management, 1980.
2. Private communication from M. Johnson & G. Tietjen to M. M. Thorpe, "Simulation Study of Propagation of Error Behavior", May 21, 1980.



W - SNM VALUE BASED ON WEIGHT & WELL KNOWN FACTORS
 ED - ESTIMATED VALUE BASED ON BY DIFFERENCE MEASUREMENT
 N - SNM VALUE MEASURED WITH THERMAL NEUTRON COINCIDENCE COUNTER
 I - SOLUTION ASSAY INSTRUMENT

Fig. 1 Flow diagram of the Production of FTF-Grade Oxide Process.

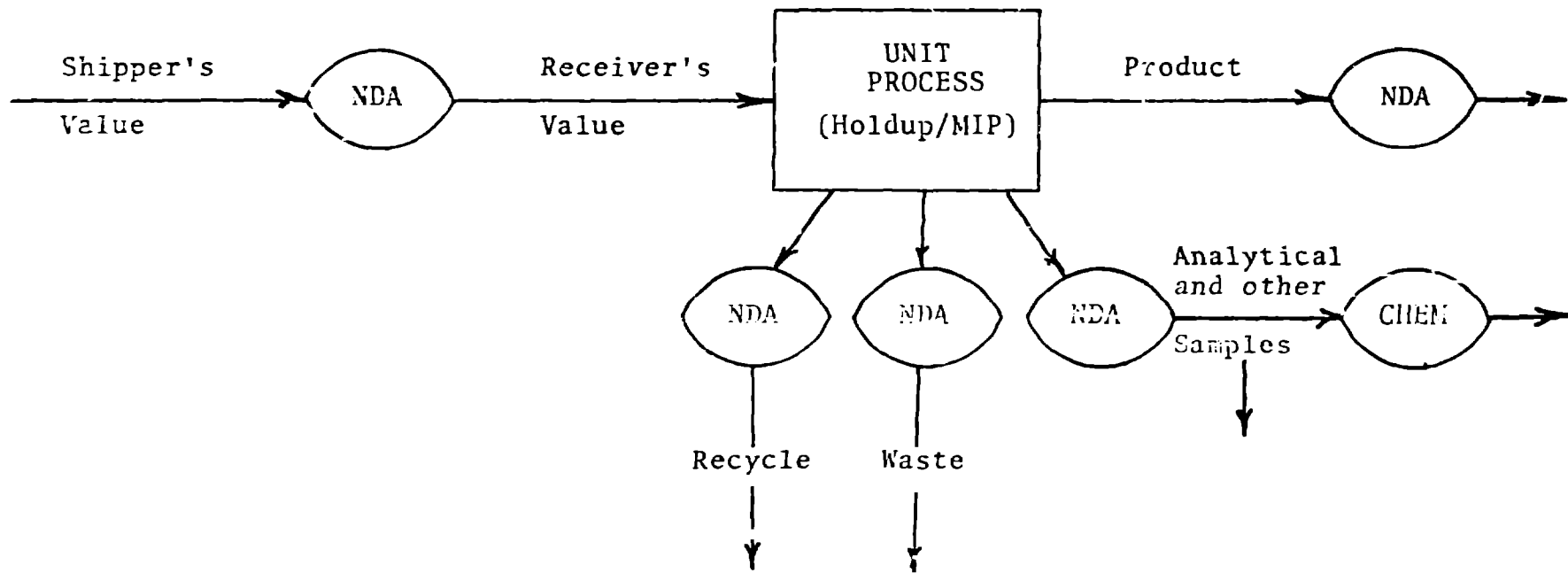


Figure 2 Schematic of a Unit Process

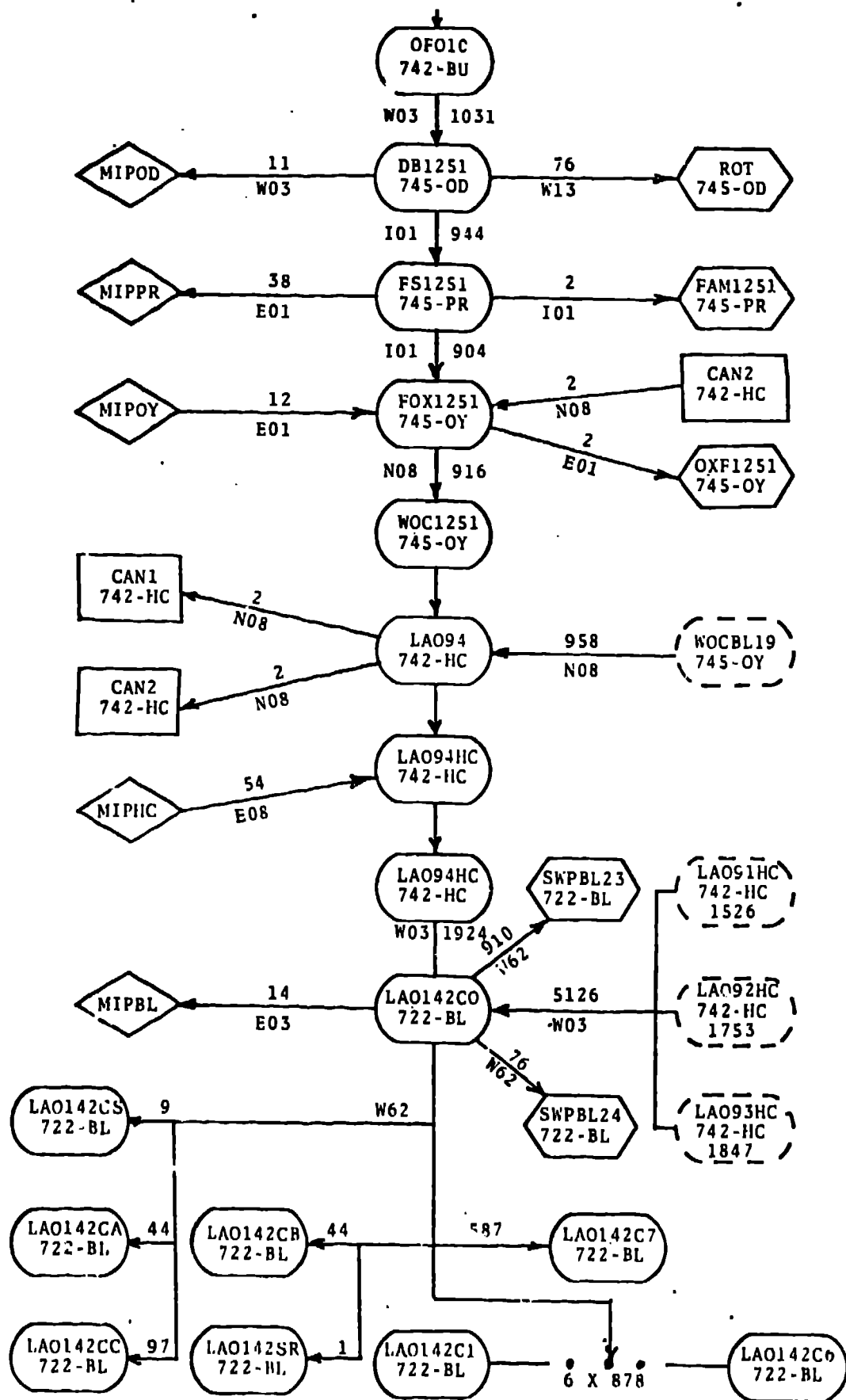
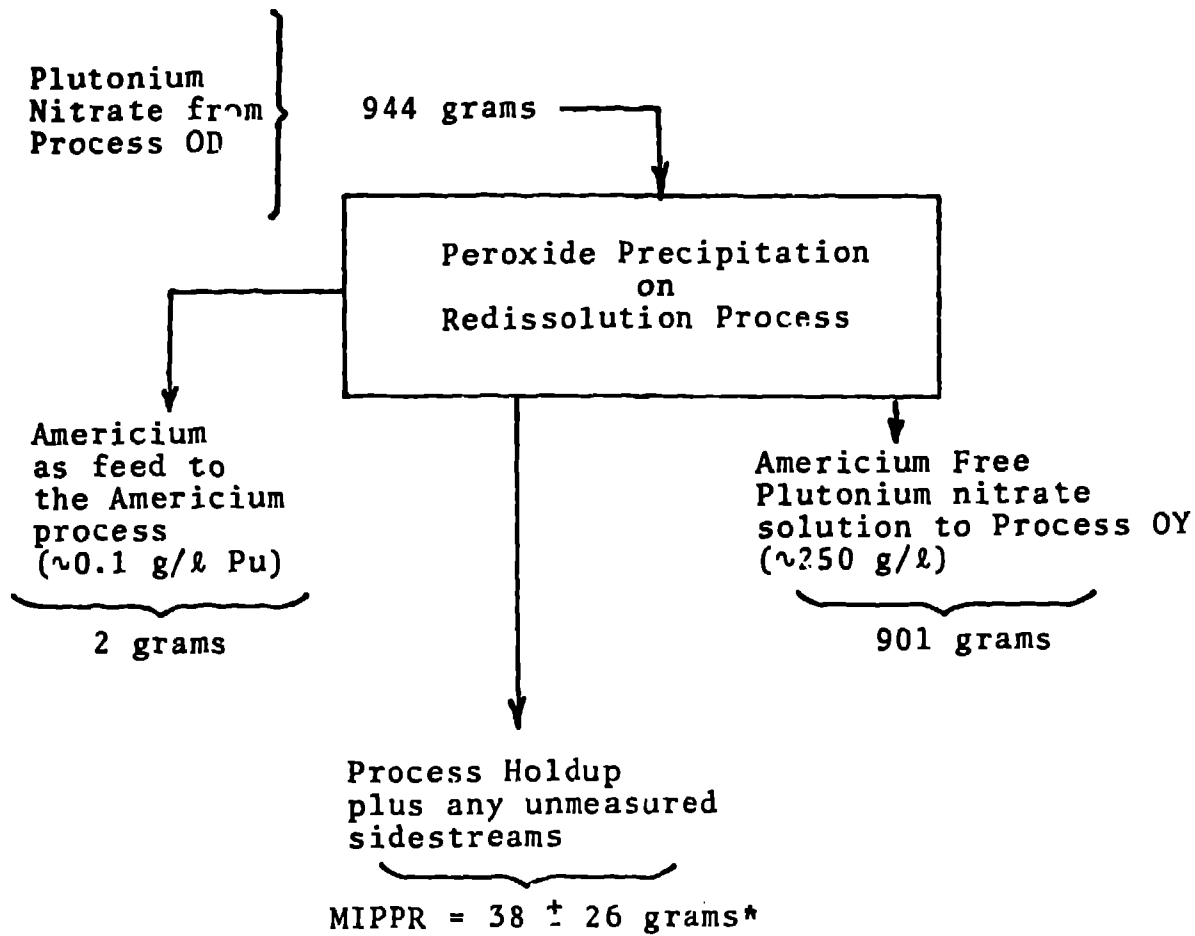


Figure 3
Audit Trail Example



* Estimated process uncertainty and assumes each measurement is normally distributed and independent. Assumptions we knew were not necessarily correct.

** These values determined prior to the identification of a significant sampling error in this process.

Figure 4 Typical Batch Values for PR Process**

MIPOD FOR SELECTED FFTF LOTS

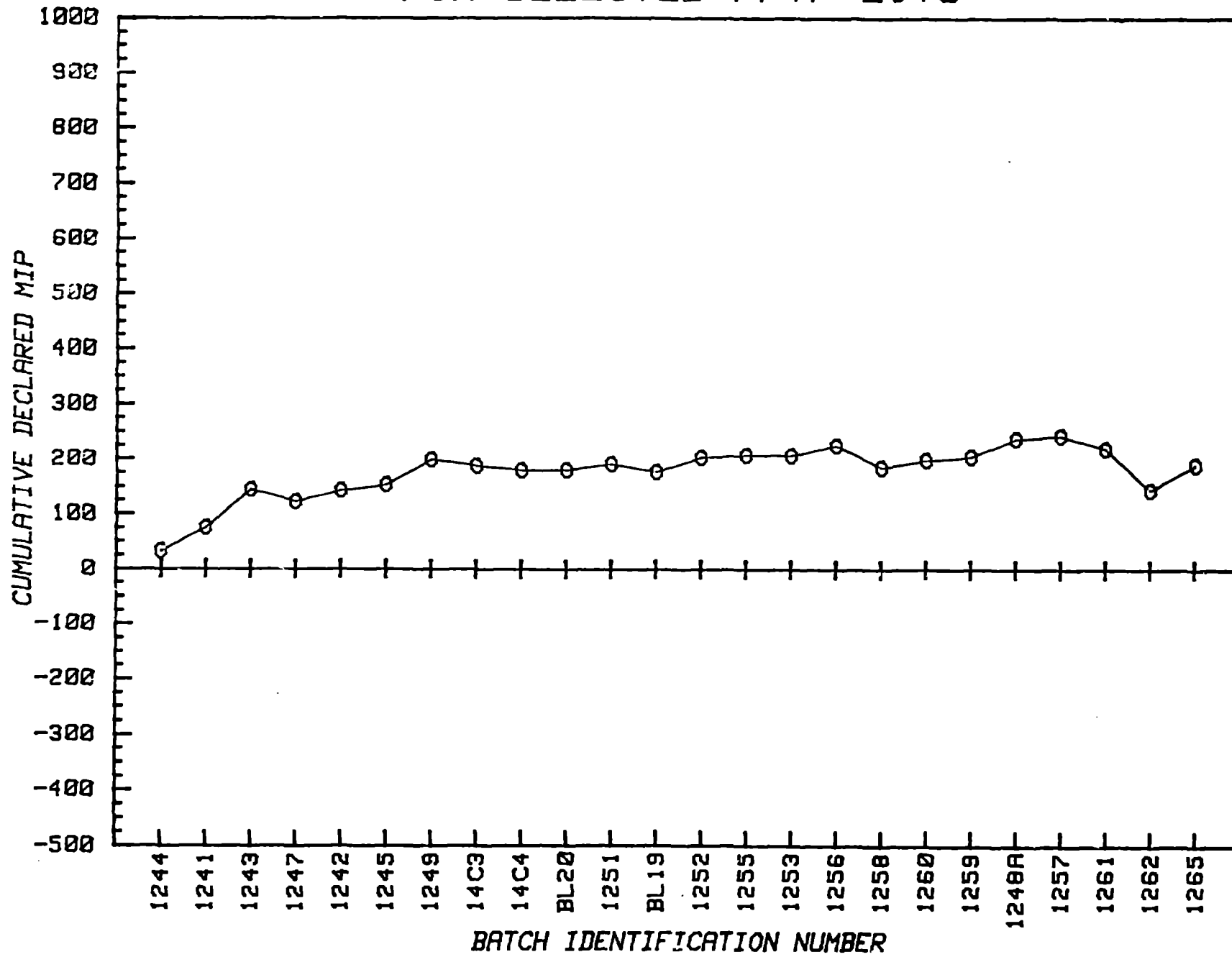


Figure 5

MIPPR FOR SELECTED FFTF LOTS

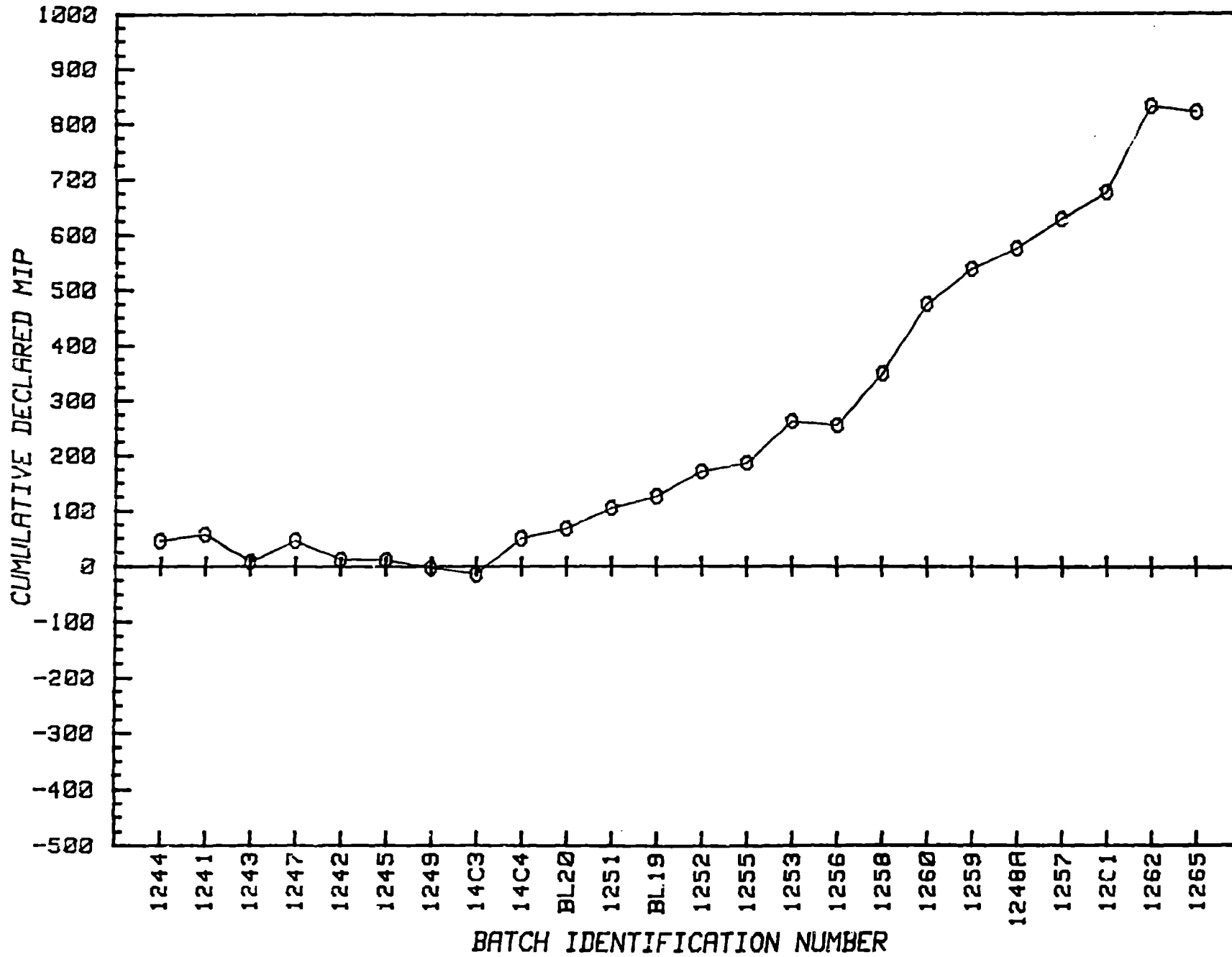


Figure 6

MIPOY FOR SELECTED FFTF LOTS

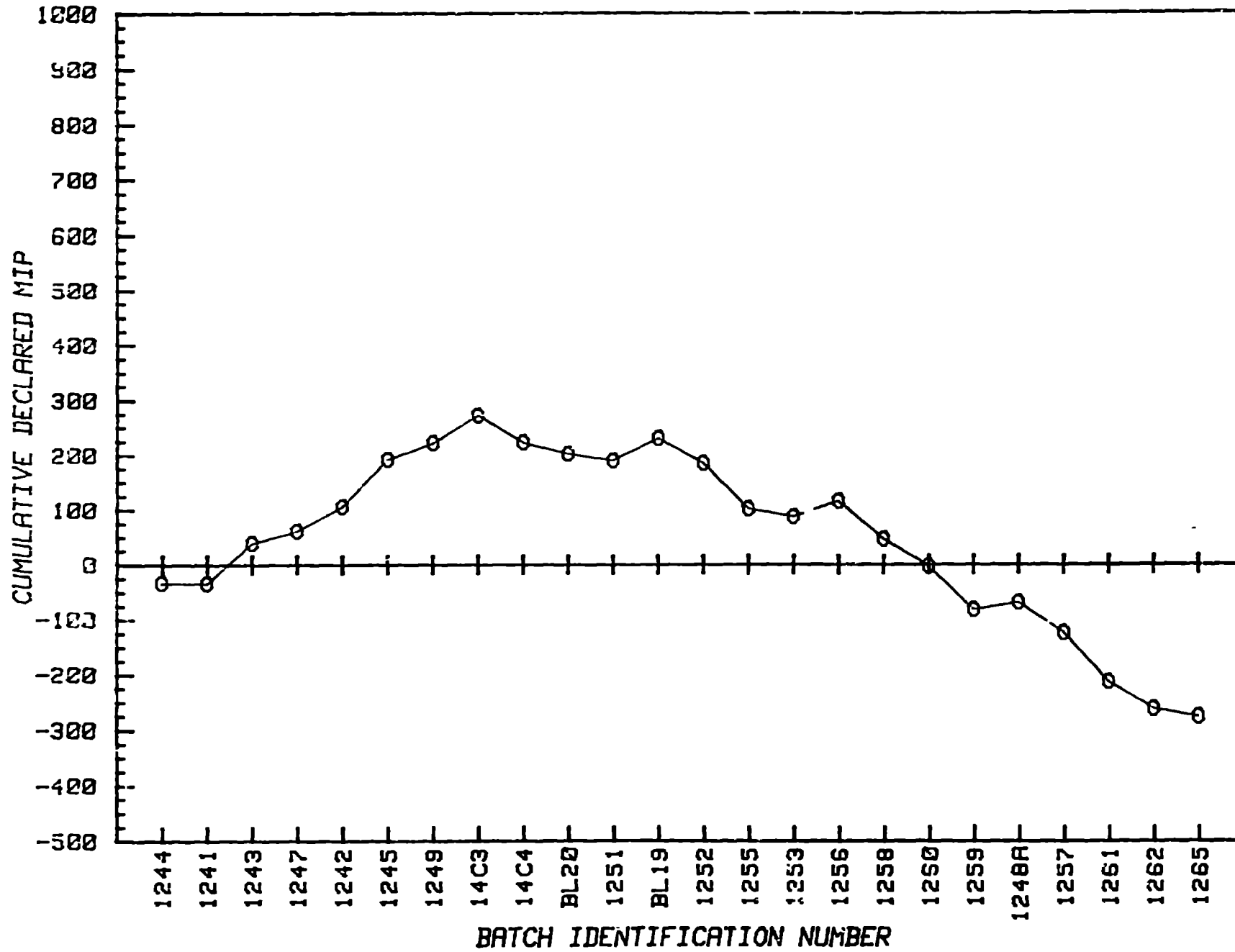
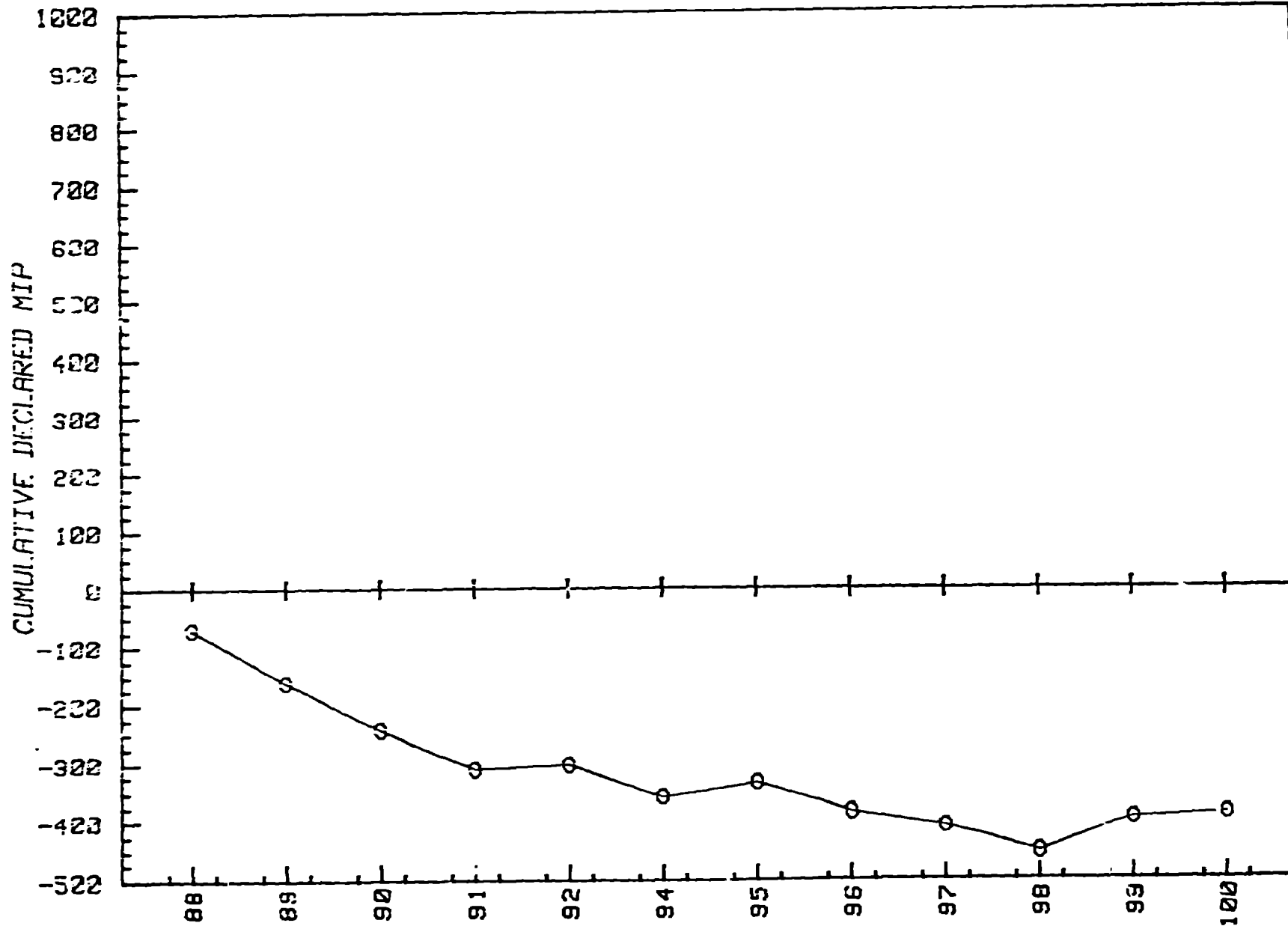


Figure 7

MIPHC FOR SELECTED FFTF LOTS



BATCH IDENTIFICATION NUMBER

Figure 8

CUMULATIVE MIP FOR SELECTED FFTF LOTS

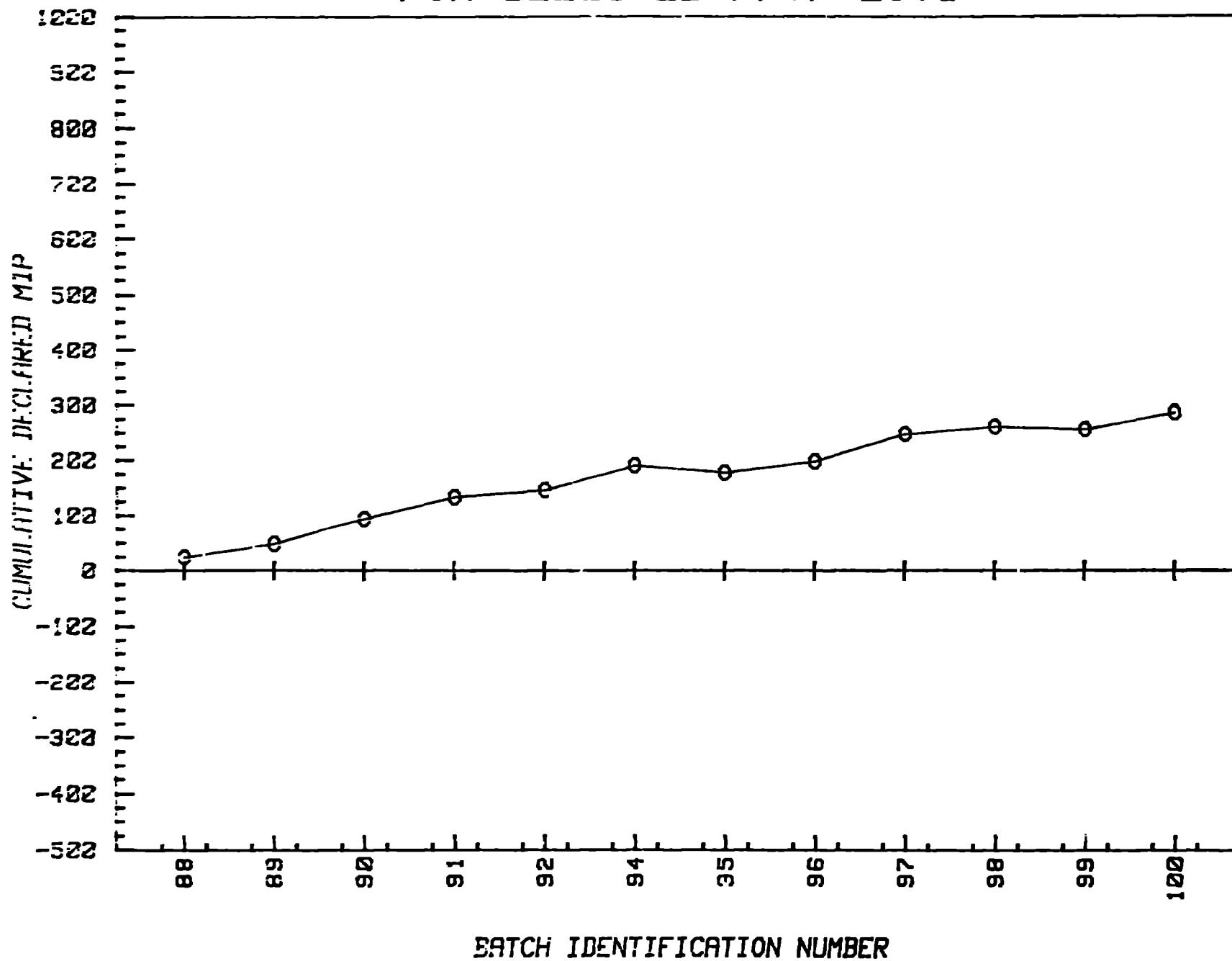
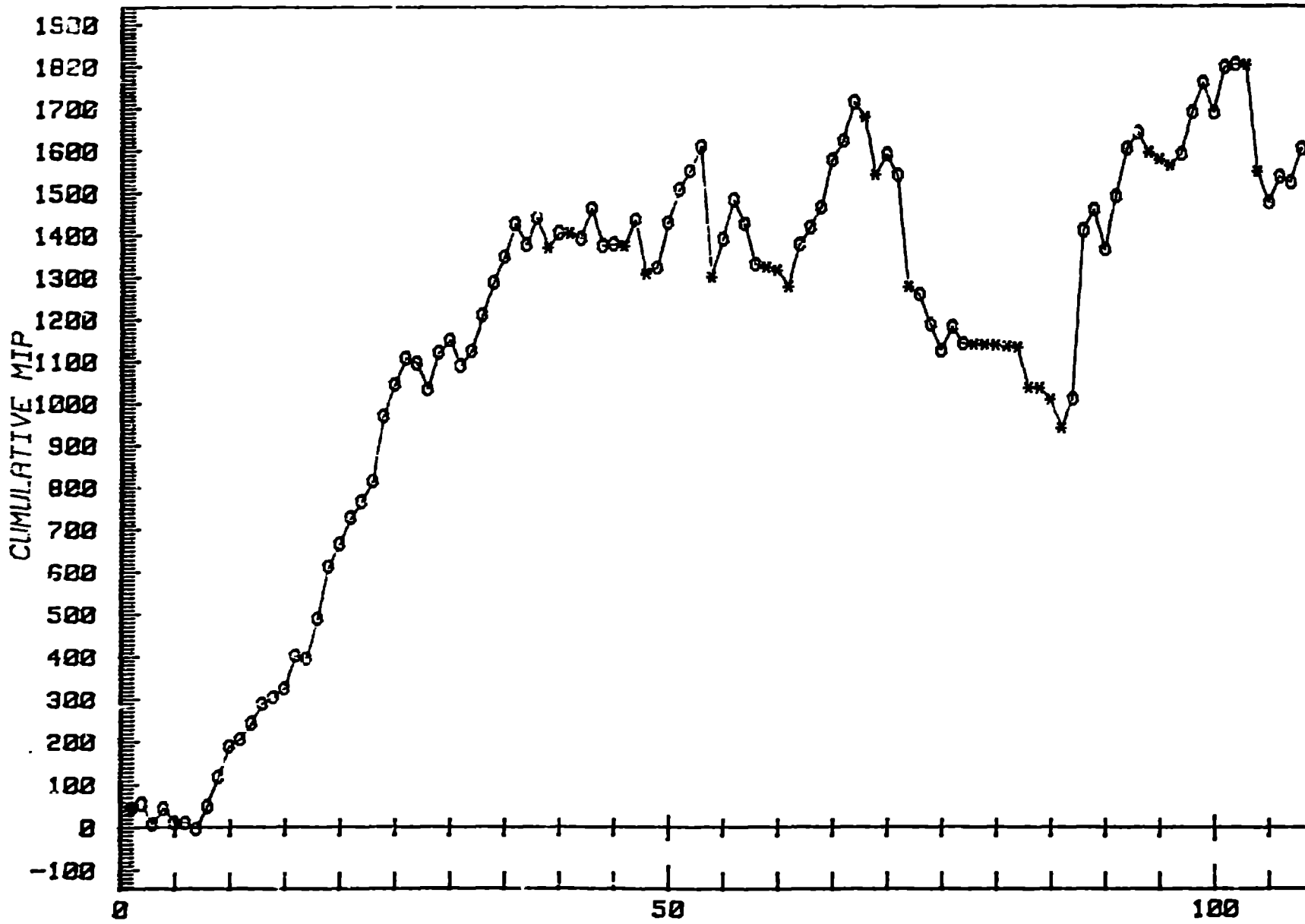


Figure 9

MIP/PR (09/17/79 - 03/15/80)



DECLARED MIP NUMBER

Figure 10