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COMPUTERIZED QUALITY CONTROL TECHNIQUES

G. D. Halverson, A. R. Camarata, R. L. Hand
F. W. Spraktes, J. M. Baldwin

Allied Chemical Corporation
550 Second Street
Idaho Falls, Idaho 83401

MASTER

ABSTRACT

Examination of historical semi-controlled data related to a current laboratory environment provides a powerful quality control tool by yielding information for such items as: (1) mathematical correction of unidentified systematic deviations, (2) estimation of confidence in lab results, and (3) detection of correctable trends. The object of this presentation is to discuss some concepts of a computerized quality control system. Topics include: (1) data flow and control, (2) use of statistics and graphics in data examination, (3) aspects of human interaction with a computerized system, and (4) the advantages and possible disadvantages of using the described system.

INTRODUCTION

Quality control apparently has many subjective definitions and modes of application depending upon needs and expectations of control system users and designers. Our application of quality control is directed toward maintenance and improvement of the quality of results of varied laboratory analyses at a nuclear fuel reprocessing plant.

Through the use of statistics and computers, our system provides more "windows" for data observation than do the usual passive control information systems. Computerization allows laboratory and quality control personnel to actively interact with the system. The system provides and applies near real-time quality control information for some operation aspects while actively directing, controlling, and standardizing other aspects.

The system algorithms are essentially based upon statistical examination of laboratory analyses of chemical standards of known content. The statistical analysis attempts to quantify the relationship of reported values to known content values. Thereby, statistics is used to expose systematic biases, trends, random errors, and other important information for scrutiny of laboratory methods. By assuming that samples of unknown content are analyzed under conditions similar to analysis of standards, some information may be applied to results of analyses on those samples for correction of unidentified systematic deviations. Our system also provides capability to detect or identify certain correctable lab malfunctions or inaccuracies such as instrument or analyst performance degradation.

This paper discusses some of the theoretical and practical considerations evaluated in developing our quality control system. This paper also introduces some details of our computerized system design.

DISCLAIMER

DATA FLOW

Data may be categorized into two principle flow cycles (see figure 1). One category of data centers upon customer samples. The other category centers upon quality control functions.

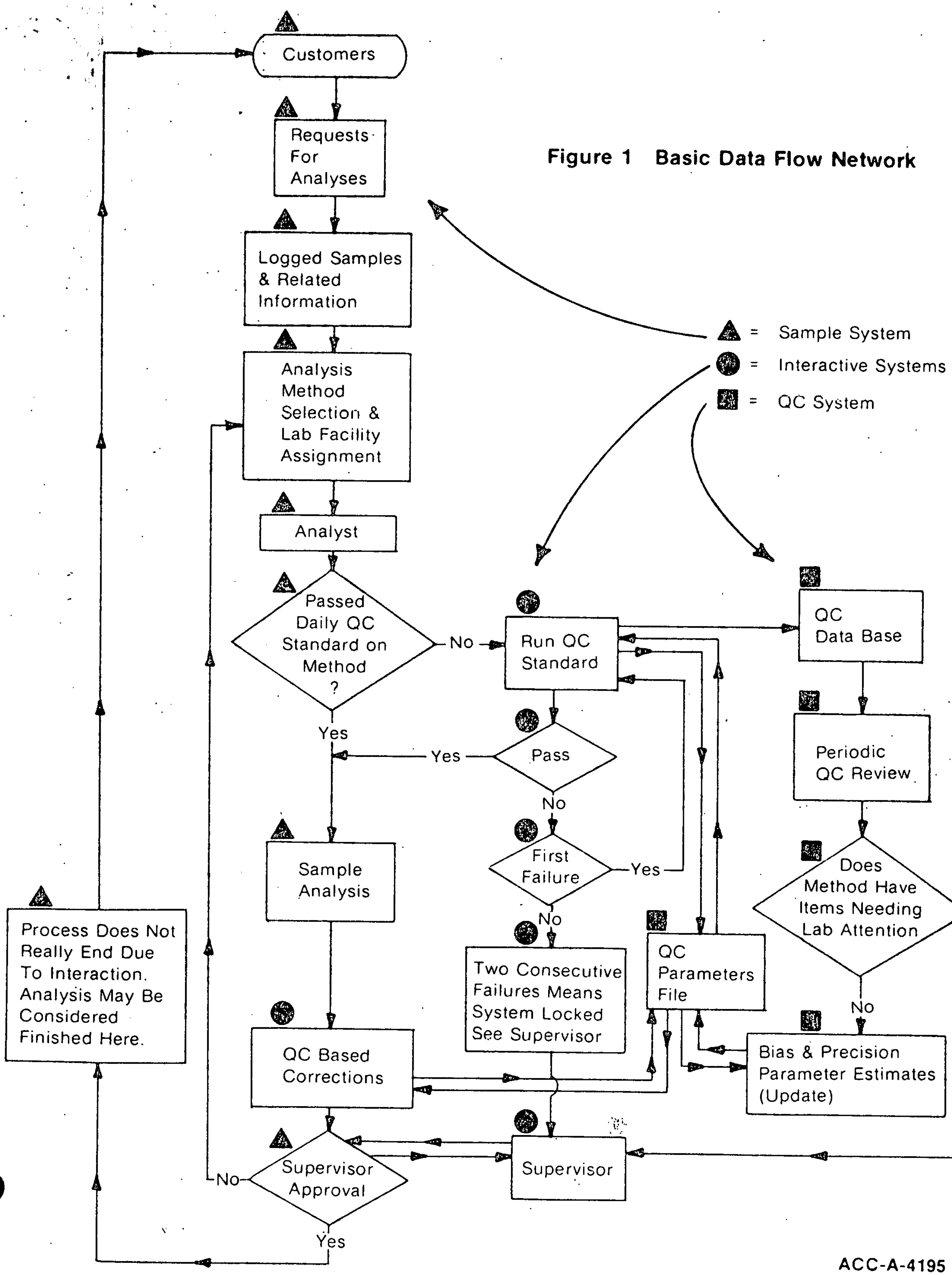
The data flow originates when a customer has need for analyses. The customer usually submits one or more samples requiring one or more methods of analysis on each sample. After customer information is recorded and samples have been logged in, a supervisor assigns analysis tasks to analysts. Analysts then examine samples and report results back to a supervisor who either requests further sample examination or transmits results back to the customer.

Within this basic framework a massive amount of information must be assembled in an orderly manner and be kept ready for immediate access. This information includes, but is not limited to, requestor identification, sample identification, accounting information, analyses requested, methods used, raw analytical data, and results. A variety of routine and nonstandard reports summarizing all these aspects of up to 30,000 individual laboratory/customer transactions per year must be prepared. In addition, the data base must be modifiable with sufficient ease to allow fairly extensive human feedback while still retaining confidence in data integrity.

Serving these data management needs in an efficient manner on a high volume basis required automation. We have automated our data management with a multiterminal minicomputer system and thereby provided the vehicle for the automation of our second data flow path, the quality control system. This phase is also a complex human interactive computer system. The functions of the quality control system are primarily resident within a desktop computer. However, some data collected by the minicomputer are used in the desktop computer and some data generated by the quality control computer are used by the minicomputer (see figure 2). The focus of this paper is upon the quality control system, which involves both computers and both data categories. However, we will omit a significant amount of the description of the minicomputer system, as its operation and function is very rigidly defined vis-a'-vis the flow of quality control data. Most of the manipulative and interpretive capabilities of the quality control system described herein are resident in the desktop computer and are directly available to only a few, hopefully detached, chemists and statisticians.

The basic unit of the quality control system is a laboratory analysis method. The basic mode of quality evaluation is examination of historical semi-controlled data relating to a current laboratory environment. The first step, then, is to collect a set of data about a particular method being applied in its normal environment under a limited number of controlled conditions. The primary control in this situation is the analysis of standards of known content. These standards are created under a strict environment of tolerances so that contents of standards may be considered fixed and known and so that deviations between reported values and standard values are primarily if not totally, attributable to causes other than standards creation. Another control for the quality control data set collection is to encourage the regular evaluation of standards in the normal operating environment. This is easily accomplished since the laboratory computer requires, with few exceptional situations, that analysts desiring to apply a method of analysis must successfully apply that method to a Q.C. standard at least once prior to that day's sample entries.

Figure 1 Basic Data Flow Network



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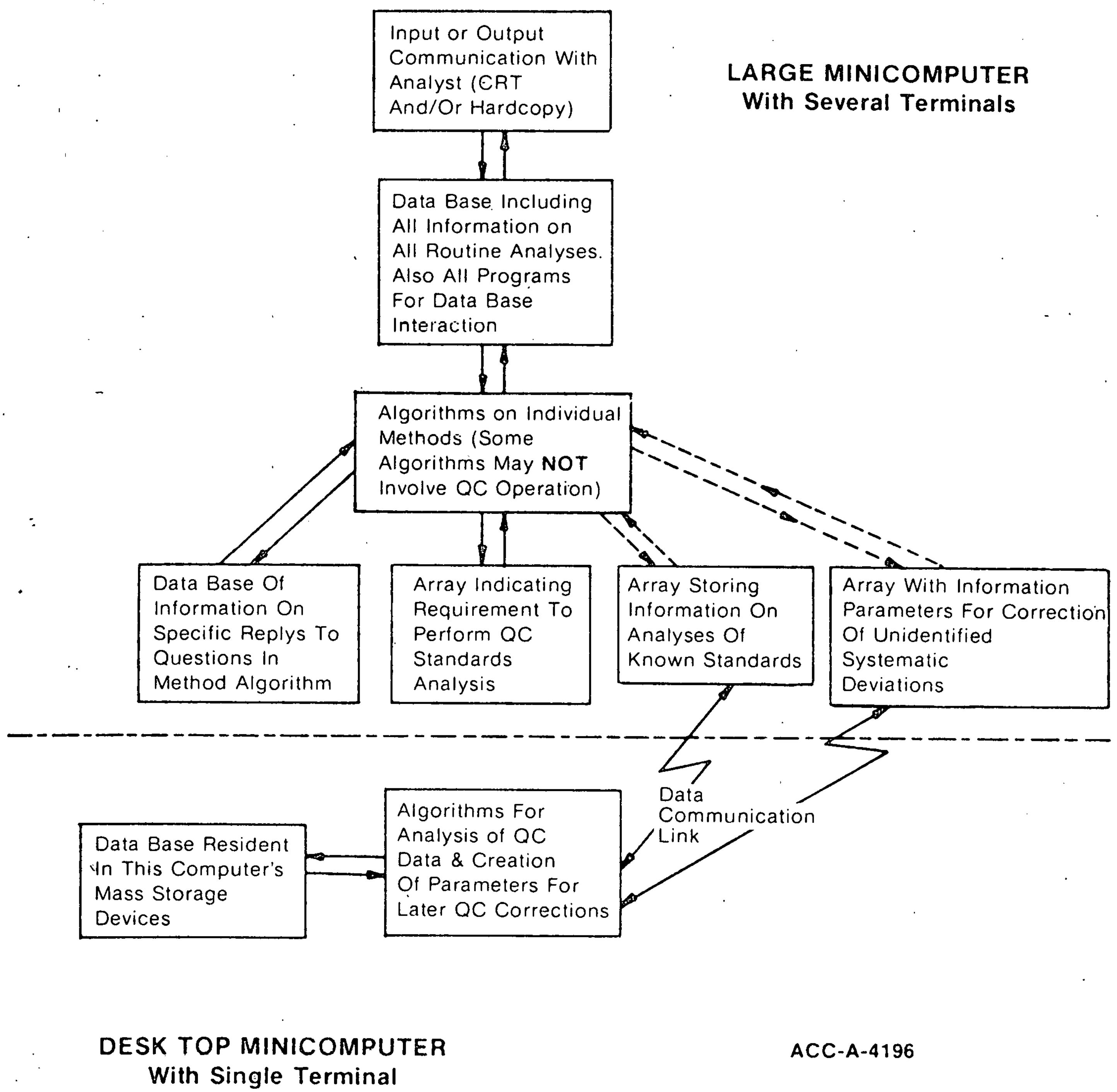


Figure 2 Hardware/Software Configuration

The laboratory computer may control the requirement for standard analysis since it provides calculations, data storage, and other services essential to routine laboratory operations. Computerization provides yet another advantage over manual operation since standard content values are not disclosed to analysts. Routine calculations are internal to the minicomputer software and the analyst identifies the standard by code. Thus, standard values may theoretically be as unknown by an analyst as sample content values and the quality control data base is generated under an environment similar to sample analyses.

Periodically, or as often as desired, the quality control data base which was collected in the laboratory computer is passed to a desktop computer. The separation of computers allows certain data bases and software to be secured and allows extensive data base examination under a controlled environment without delayed operation of the laboratory computer. After data examination and testing (described with greater detail in the next section) has yielded estimates for precision parameters and parameters to correct for unidentified systematic deviations or bias, the parameter estimates are transferred to the laboratory computer.

The parameter estimates provided to the laboratory computer support additional quality control functions. When parameter estimates are applied to results of analyses on samples of unknown content, effects of systematic deviations are mathematically nullified. The parameter estimates are also used to create intervals which have a high probability of including the true value of the sample content. It should be noted, however, that sample results are modified only for operations of the lab studied by quality control and do not include modifications necessary due to other operations such as difficulties in customer sampling.

When results of analyses on standards are submitted, the parameter estimates are used to bias correct and compare results to a "reasonable" interval about the true standard content. Thus, the laboratory computer may use quality control information to block certain analysts from using certain methods until a supervisor is aware of questionable operations. The cause for a questionable result may have been analyst technique, instrument performance, or some other factor. The computer simply allows better control by terminating abnormal daily input until investigations and/or corrections may be performed. The computers can then assist in locating problem sources.

This section has only briefly described the data flow associated with quality control as the data are processed from analyst to storage and from results of data examination to laboratory computer system. Subsequent sections will develop and detail desktop computer data examination techniques.

DATA EXAMINATION

The desktop computer system provides descriptive and probabilistic examination of data that are electronically collected through the previously mentioned laboratory computer. Since the laboratory computer may not encompass all data sources, however, the desktop computer also permits manual Q.C. data base entry. The data collected from either source are stored in a data file which includes information such as date and time of analysis, analyst initials, method number, instrument identification, reported result, true content value, and estimates of bias and precision based upon minicomputer stored parameters.

Data in the desktop computer are examined under a concept that standards are developed on or about a single level or under a concept that standards are developed to span a range of levels. The single level standards deal with methods which always result in approximately one value. The single level standards, then, are created with a very small range varied only so that the exact answer is unknown to the analyst at the time of analysis. Multiple level methods deal with ranges of values which may even cover several orders of magnitude.

In our system, single level methods are examined by assuming one of two models. The "absolute" model is a simple additive model where the corrected estimate is obtained by subtracting a bias estimated by the average reported value minus its respective standard value. The "relative" model is a simple multiplicative model where the corrected estimate is obtained by multiplying the reported value by an estimated quantity based upon the average deviation divided by its standard value. Both models are apparently well known in the nuclear industry[1].

The parameter estimates may be based upon the entire method Q.C. data base or any subset thereof. The computer allows the user to define a data subset, process that set through a series of tests, and then end or define a new data subset. This creates a kind of cycling process whereby a user may select the best set of estimates to describe recent method activity. Also, this cycling permits more detailed scrutiny of possible trends or abnormal occurrences.

Each cycle provides a selection of descriptive and statistical tests for the data. Summary data is provided first. This summary details such information as: (1) method name, (2) bias to be applied, (3) application formula, (4) number of values used in estimations, (5) systematic standard deviation, (6) random standard deviation, and (7) standard deviation of any individual corrected result. The summary is followed by a tabular listing. The listing itemizes an identifying index, the reported result, the standard content value, the corrected result based upon summary values, the deviation between the bias corrected result and the standard content, and the deviation expressed in units of standard deviation of the bias corrected result. The tabular listing provides an elementary view of data for location and identification of method performance deviations.

Since the above mentioned listing may be long or otherwise evasive or inconclusive, several statistical tests have been provided to give additional insight. One test compares deviations as a dependent variable to index as an independent variable through a linear least squares regression. Then, testing the regression slope[2] by comparison to a computer generated critical value of the Student's T-distribution[3], the system generates a statement concerning the significance of a linear trend for data in the entered order.

A more complex trend such as cyclic data may not be detected by the linear regression but is easily determined by a different test dealing with mean square successive differences[4 5] known as the von Neumann test. (this test is quite similar to a test for autocorrelation known as the Dubin-Waston Test[2].)

Another trend test is a test for independence of deviations. The validity of an assumption of independence is quite essential for most commonly used statements of inference. The test for independence currently incorporated in the system is a relatively weak test based upon the hypergeometric distribution and is known as the runs test[6].

Another statistical test is a test for the validity of the assumption that residuals may be described by a normal probability distribution. Besides being useful in inference statements, this test is quite helpful in detection of multi-population situations such as one analyst performing differently from others when all analysts have data throughout the data base. The normality test currently in use is known as the Chi-Square goodness-of-fit test. The validity of this test has been addressed by many authors[7 8 9] and is apparently heavily dependent upon proper and non-subjective categorization of standardized residuals. Utilizing a number of these considerations the computer generates properly placed category cell divisions, classifies data, generates a statistic, and compares the resultant statistic to a critical value of the Chi-Square probability distribution.

Following these tests, is a graphic plot of standardized residuals vs index of order. The plot provides a visual representation useful in confirmation of model aptness (see figure 3 for an example). After the plot, a table lists standards based upon the defined data base set. Each standard has its own bias and precision which may be compared to other standards if more than one standard is present in the set. The table is useful for identification of standards which may be causing erroneous or misleading method performance determinations. The tests used for pairwise comparison of bias and precision of standards involve statistics of the F-distribution, T-distribution, and F-distribution with modified degrees of freedom[10] (when variability of standards may not be assumed equal).

The final test performed in each cycle of the single level method is called the moving mark test. This test searches for significant levels of change in bias or precision by comparing values before a point to values after a point as that point is moved consecutively through the data set (see figure 4).

The multiple level methods define bias and precision over a range of values through the use of weighted regressions. Bias is described by inversion of a polynomial least squares regression of reported results on standard values. The above regression utilizes data transformed by dividing each value by the sample standard deviation at that level. Thus, large values do not carry more significance than small values when parameters estimates are developed by minimizing squared differences. Precision is then an error propagated combination of regression results and level variability. When applying the above information to sample data, however, the standard deviation of a level is usually not known. Thus, the standard deviation must be developed through a weighted regression. Again, the weights are a way of modifying the data so that large values do not retain all the significance in parameter estimation. Our system generates level variability by using one of several polynomial regressions using standard deviation or ln(std. dev.) as a dependent variable and value of known standard or ln(value of known) as the independent variable.

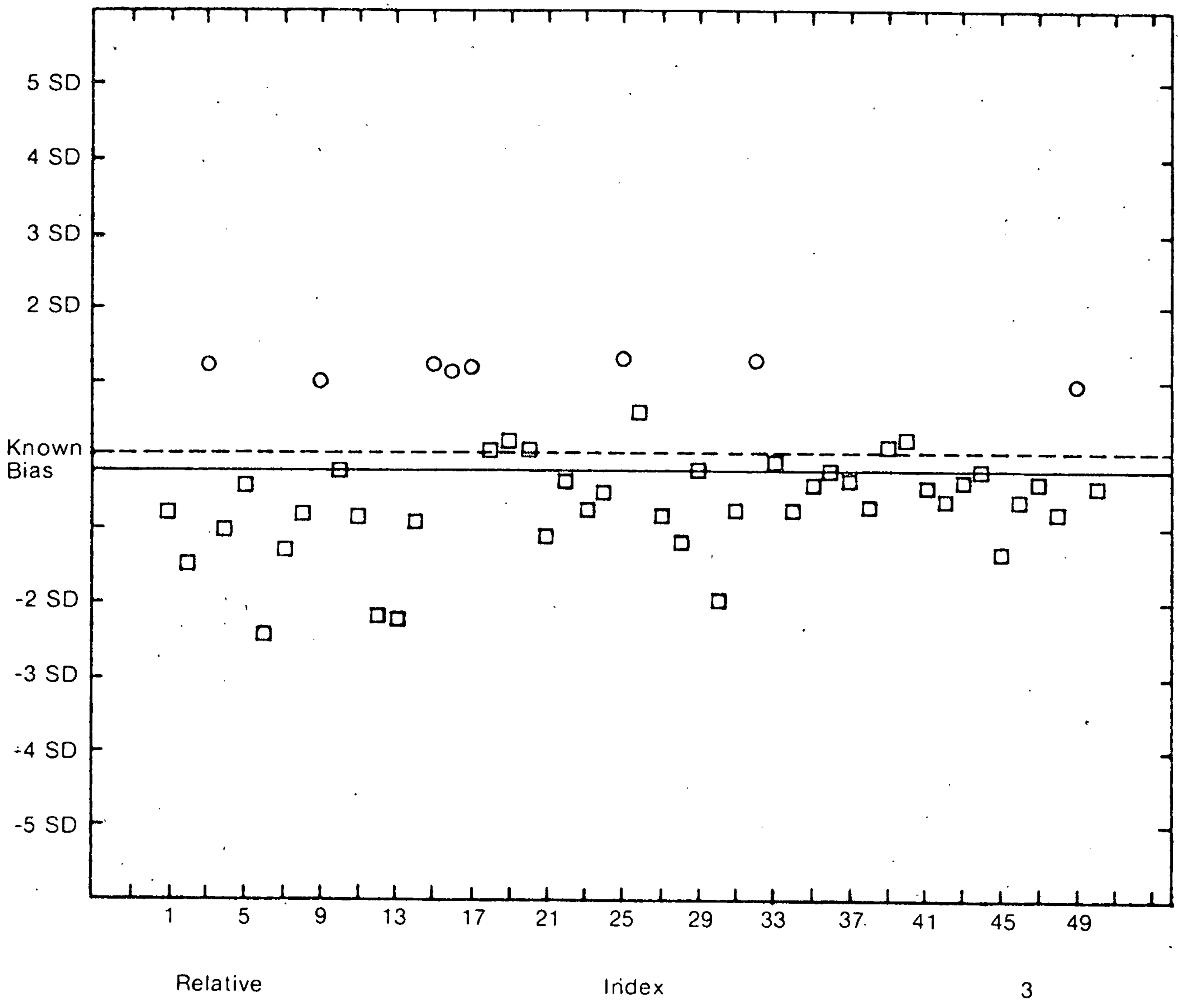
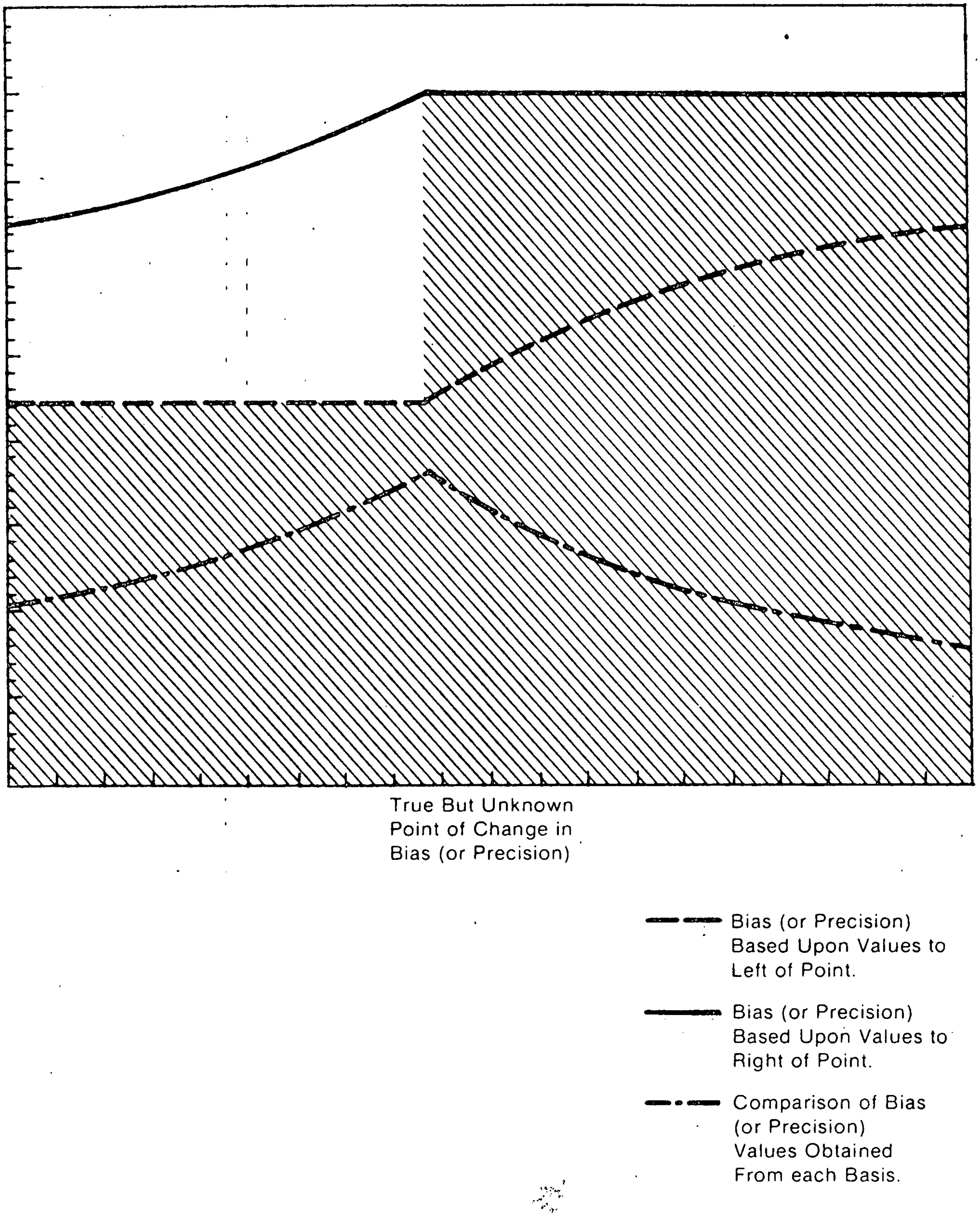


Figure 3 Residual Plot Using Single Level Concept (Relative Model-Analyst AAA Emphasized)

Figure 4 Moving Mark Concept



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The multiple level methods cycle much as the single level methods to permit data set selection and identification of abnormal occurrences. In each cycle a standard deviation curve is developed and a bias curve is defined. Each curve may be plotted with data to visually inspect the fit. Also, each cycle provides some of the types of tests previously described. The tests include a test for the validity of the normality assumption, a runs test for independence, the von Neumann test for trends, and residuals plot. The multiple level methods also include a form of stepwise regression to optimize model selection and tests for such things as significance of bias[11] or significance of the relationship of result and standard value.

SUMMARY

Fundamentally, our quality control system is a collection of plots and statistics provided for scrutiny of a semi-controlled data base. Complexities are introduced by allowing the system to be interactive. In the interactive mode, results must be concise and conclusive. This means great detail must be included to provide not only statistics but probability distributions resulting in inference leading to conclusions. This provides some insight into system creation difficulties. Problems include choice of presentations, algorithms and program flow.

By allowing interaction, users obtain greater information through their control. The increased user power, however, can easily lead to system abuse. Training, then becomes essential for proper implementation of this type of control.

This presentation has only provided some description of the main parts of our system. Other features include special abilities to compare individually defined data base subsets, plots of a history of parameters (see figure 5), printed reports and lists, manual or computer communicated data base modification abilities, and others. The numerous and often involved details have been omitted due to presentation length.

Future development of our system will certainly include an improved selection of parametric statistics, non-parametric statistics and computer algorithms. The system may well be improved through study of considerations such as long term cyclic patterns or more detailed method specialization. The already experienced returns on our quality control system coupled with future possibilities assure the strength and potential in this design.

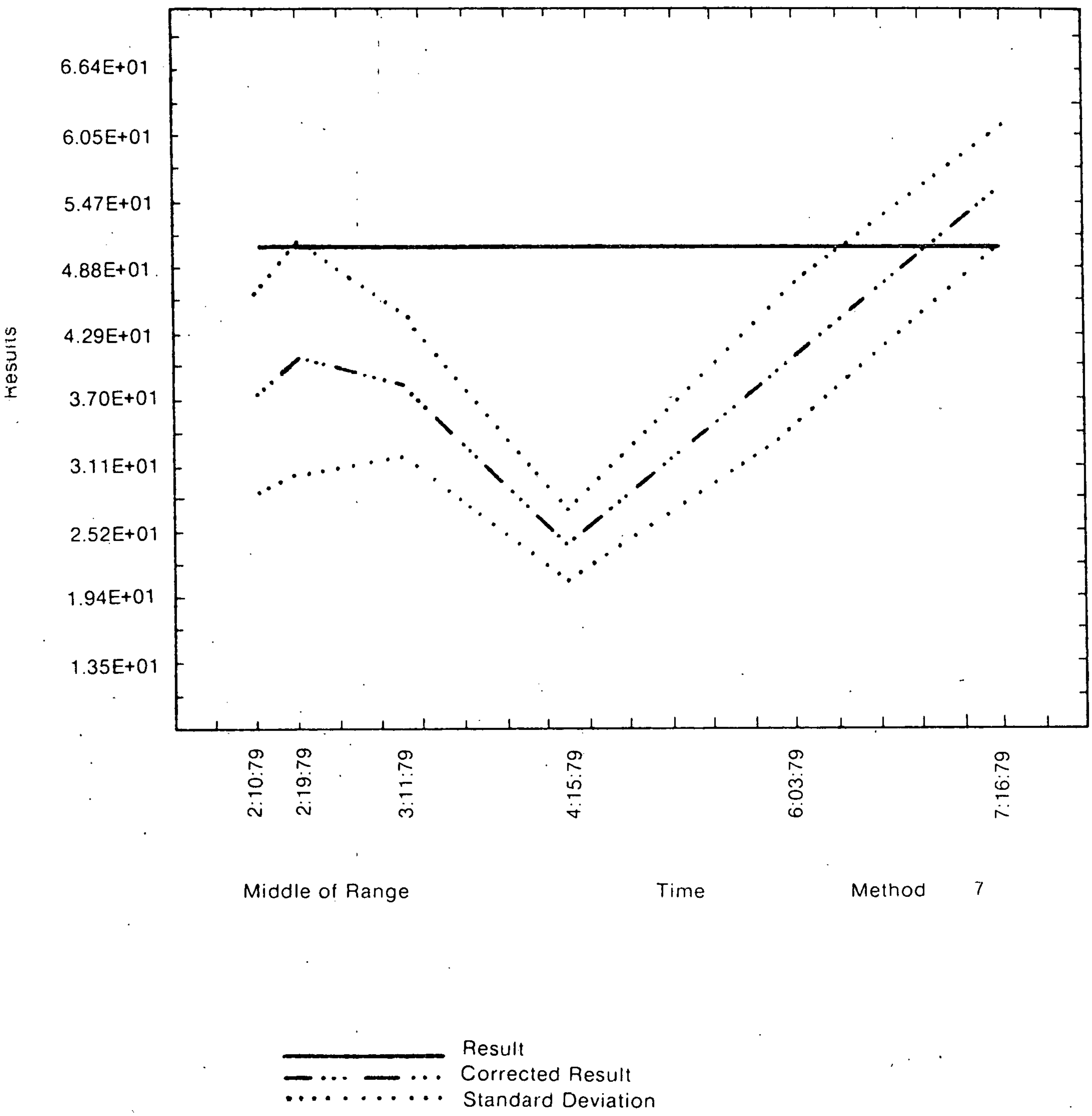


Figure 5 Simulated Historical Plot

REFERENCES

1 John L. Jaech, Statistical Methods In Nuclear Material Control, TID-26298, Technical Information Center, Office of Information Services, U. S. Atomic Energy Commission, 1973.

2 John Neter and William Wasserman, Applied Linear Statistical Models, Richard D. Irwin, Inc., Homewood, Illinois 60430, 1974.

3 Milton Abramowitz and Irene A. Stegun, Handbook Of Mathematical Functions, Dover Publications, Inc., New York, New York, 1972.

4 John von Neumann, R. H. Kent, H. R. Bellinson and B. I. Hart, "The Mean Square Successive Difference", Annals Of Mathematical Statistics, V12, pp 153-162, 1941.

5 John von Newmann, "Distribution Of The Ratio Of The Mean Square Successive Difference To The Variance", Annals Of Mathematical Statistics, V12, pp 367-395, 1941.

6 William C. Guenther, "Some Remarks On The Runs Test And The Use Of The Hypergeometric", Research Paper #198, S-1977-556, Dept. of Statistics, University of Wyoming, Laramie, Wyoming, 1977.

7 Robert G. D. Steel and James H. Torrie, Principles and Procedures of Statistics, McGraw-Hill Book Company, Inc., New York, 1960.

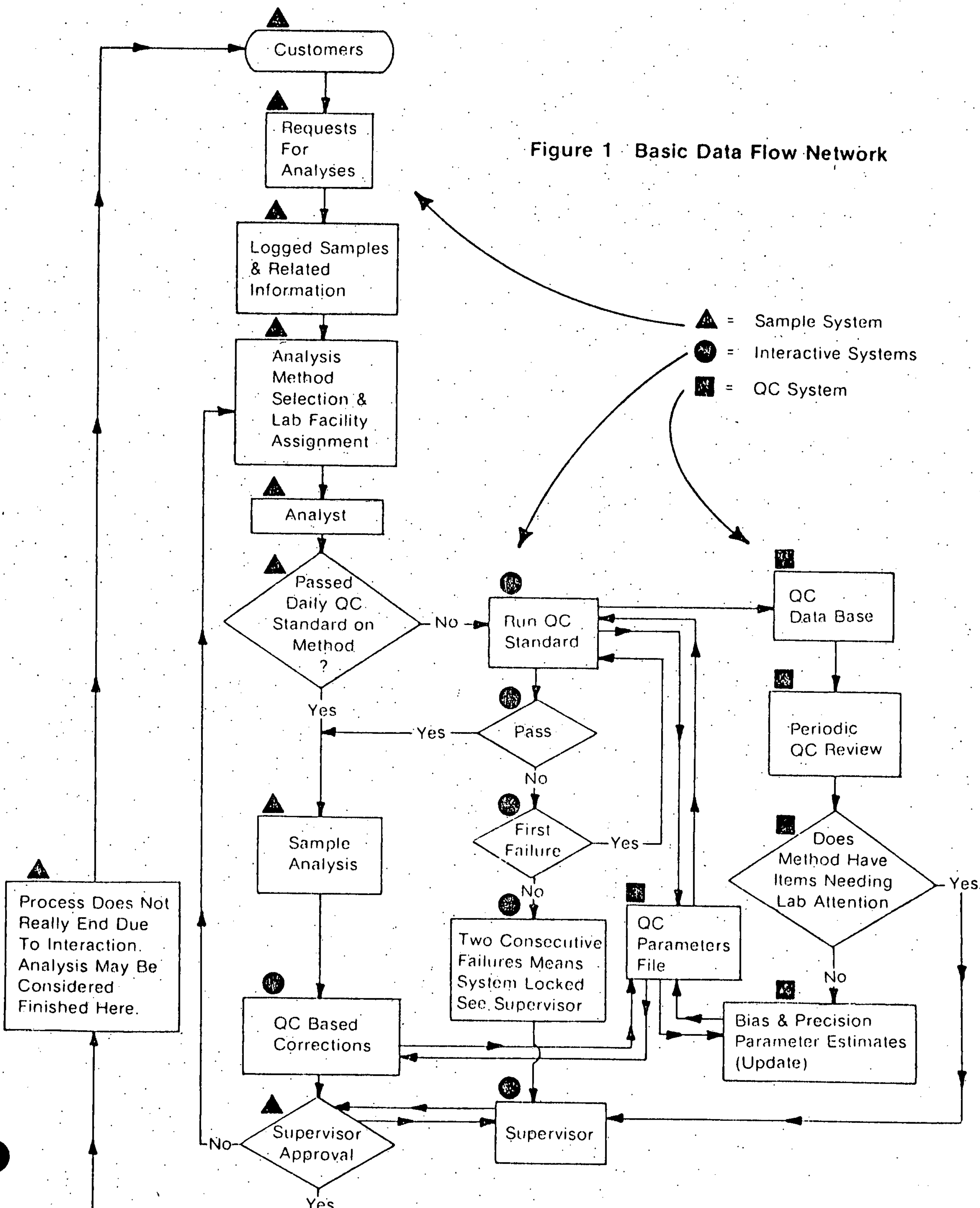
8 Jean Dickinson Gibbons, Nonparametric Methods For Quantitative Analysis, Holt, Rinehart and Winston, New York, 1976.

9 E. L. Lehmann, Nonparametrics: Statistical Methods Based On Ranks, Holden-Day, Inc., San Francisco, 1975.

10 Mary Gibbon Natrella, Experimental Statistics, National Bureau of Standards Handbook 91, 1963.

11 Franklin A. Graybill, Theory And Application Of The Linear Model, Duxbury Press, North Scituate, Massachussetts, 1976.

Figure 1 Basic Data Flow Network



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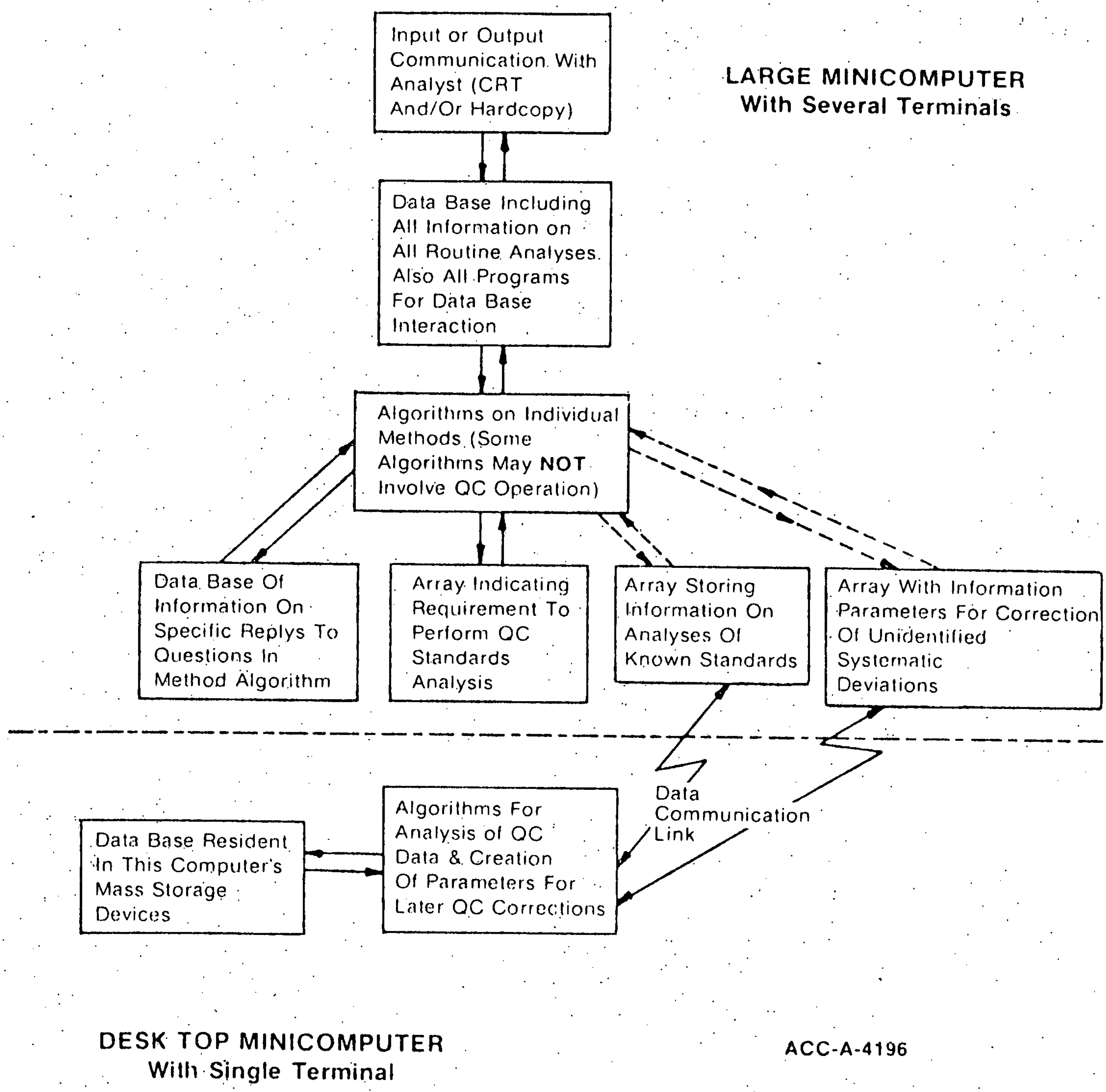


Figure 2 Hardware/Software Configuration

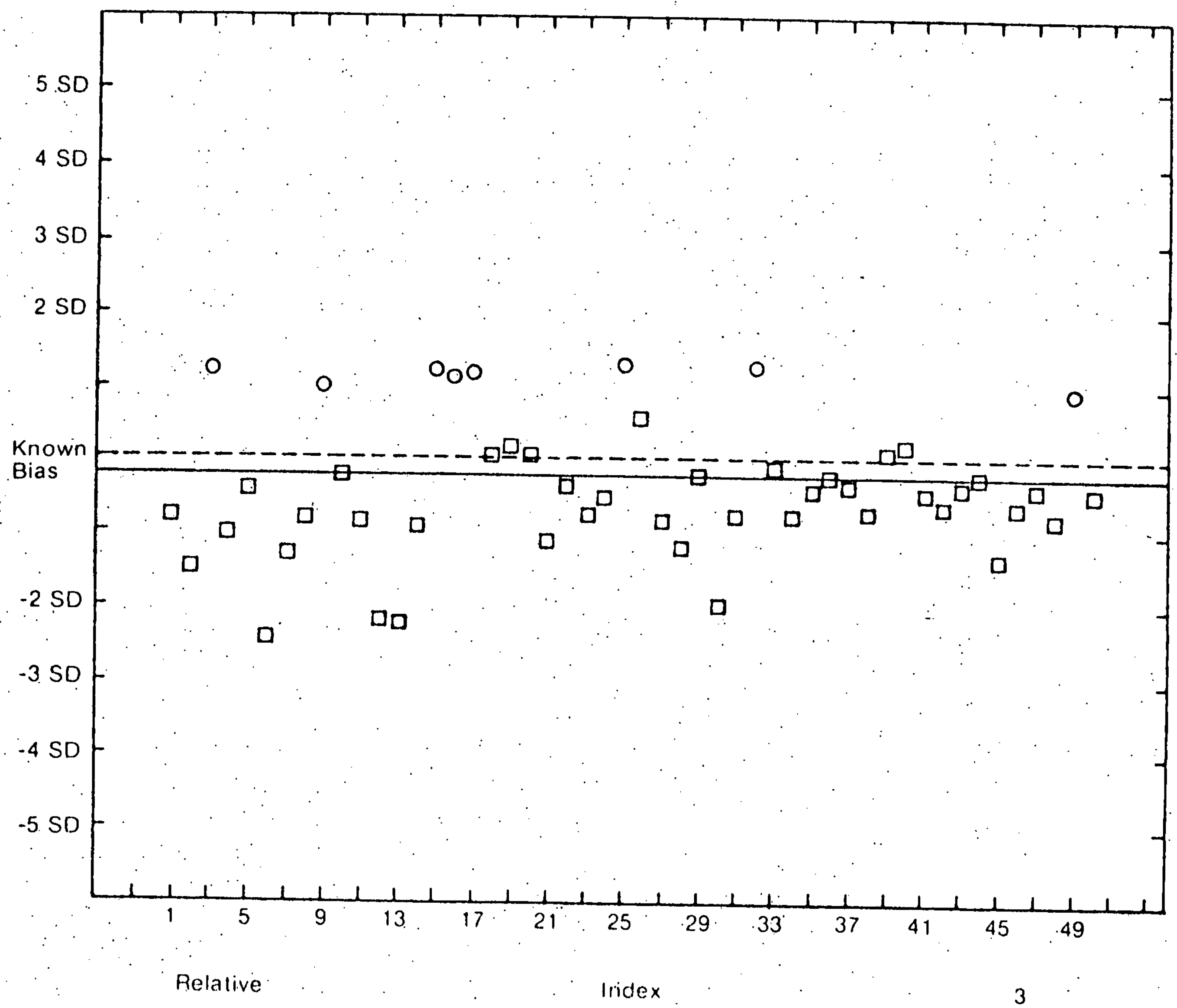
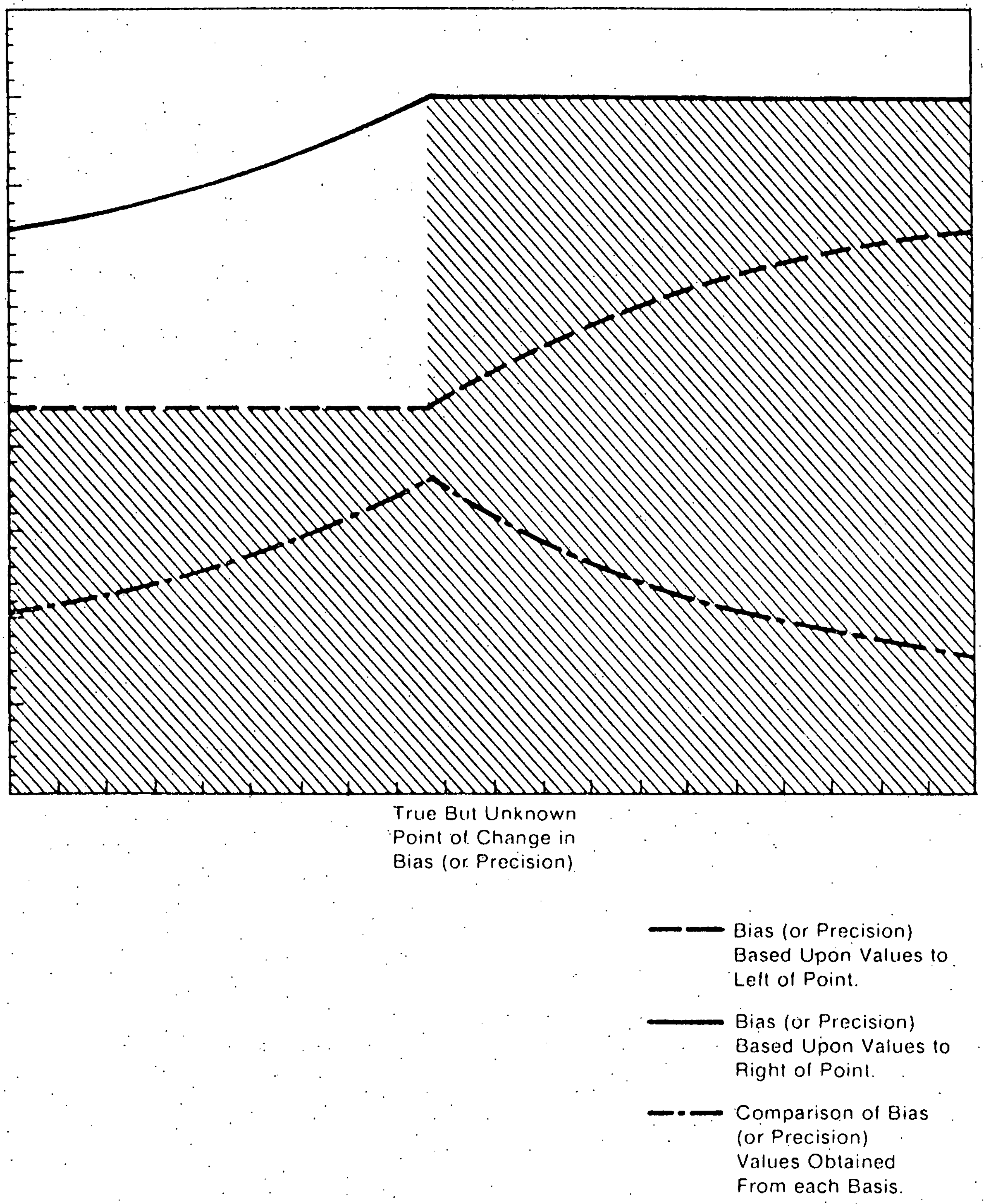


Figure 3 Residual Plot Using Single Level Concept
(Relative Model-Analyst AAA Emphasized)

Figure 4 Moving Mark Concept



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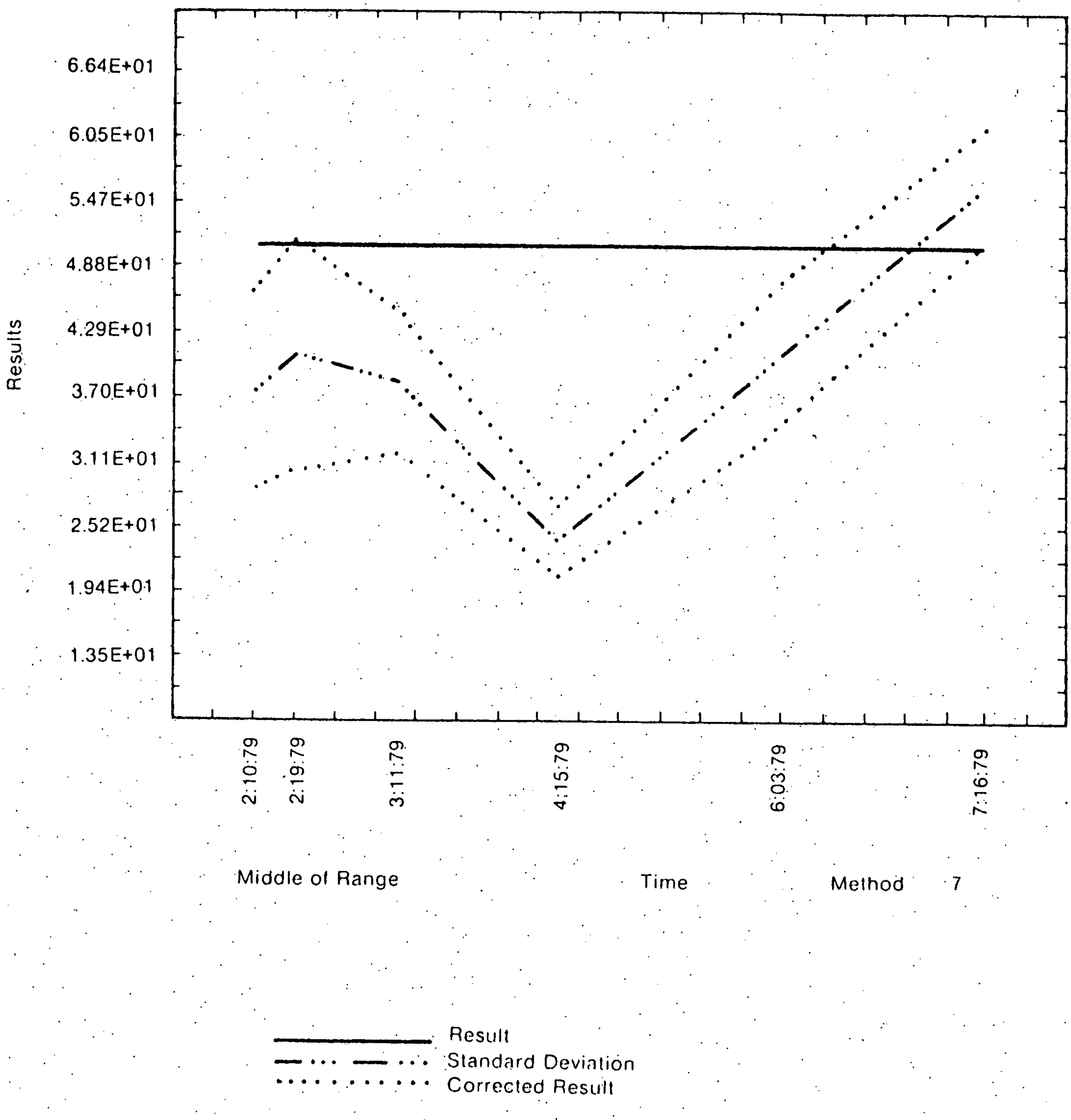

Figure 5 Simulated Historical Plot

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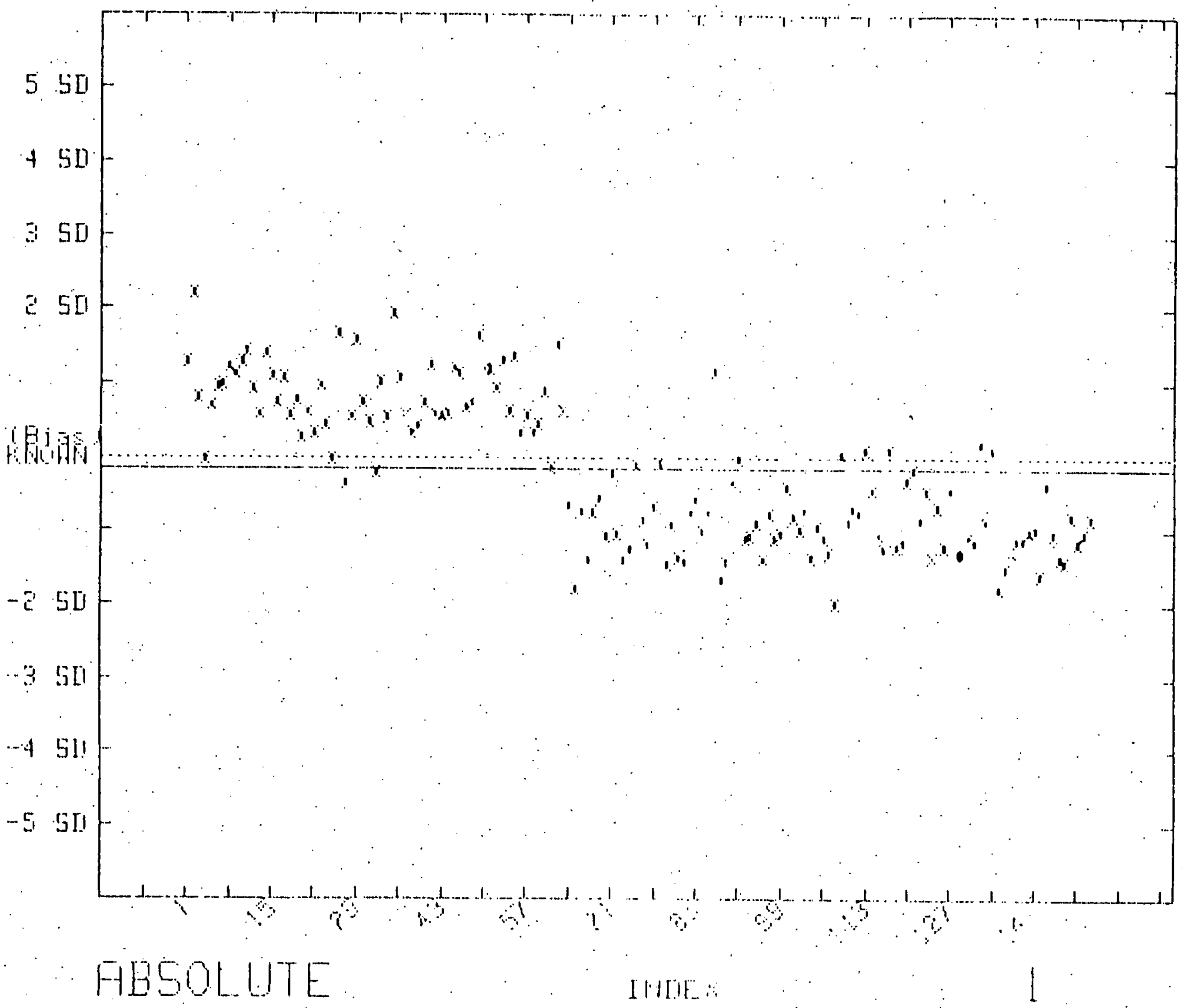
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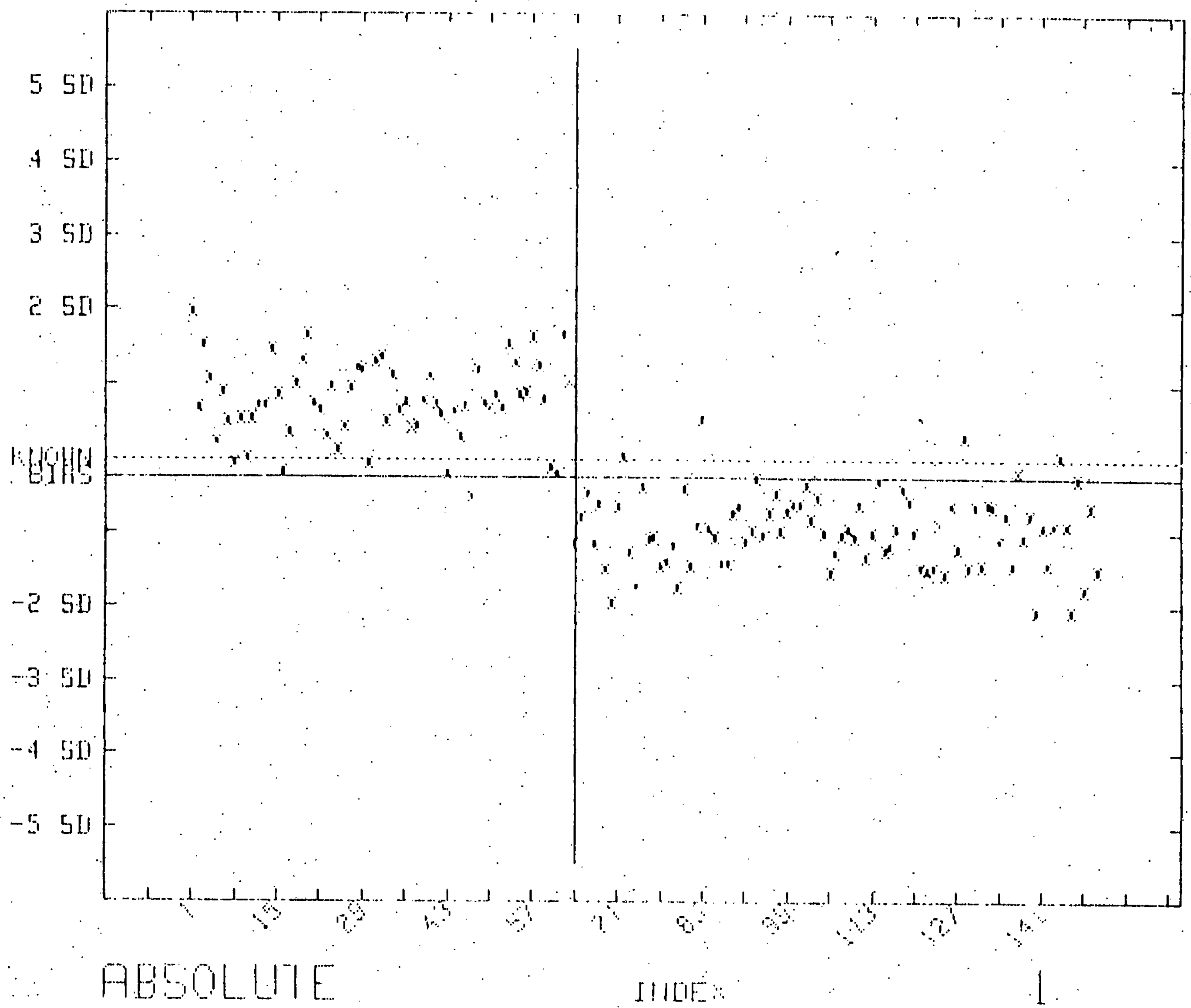
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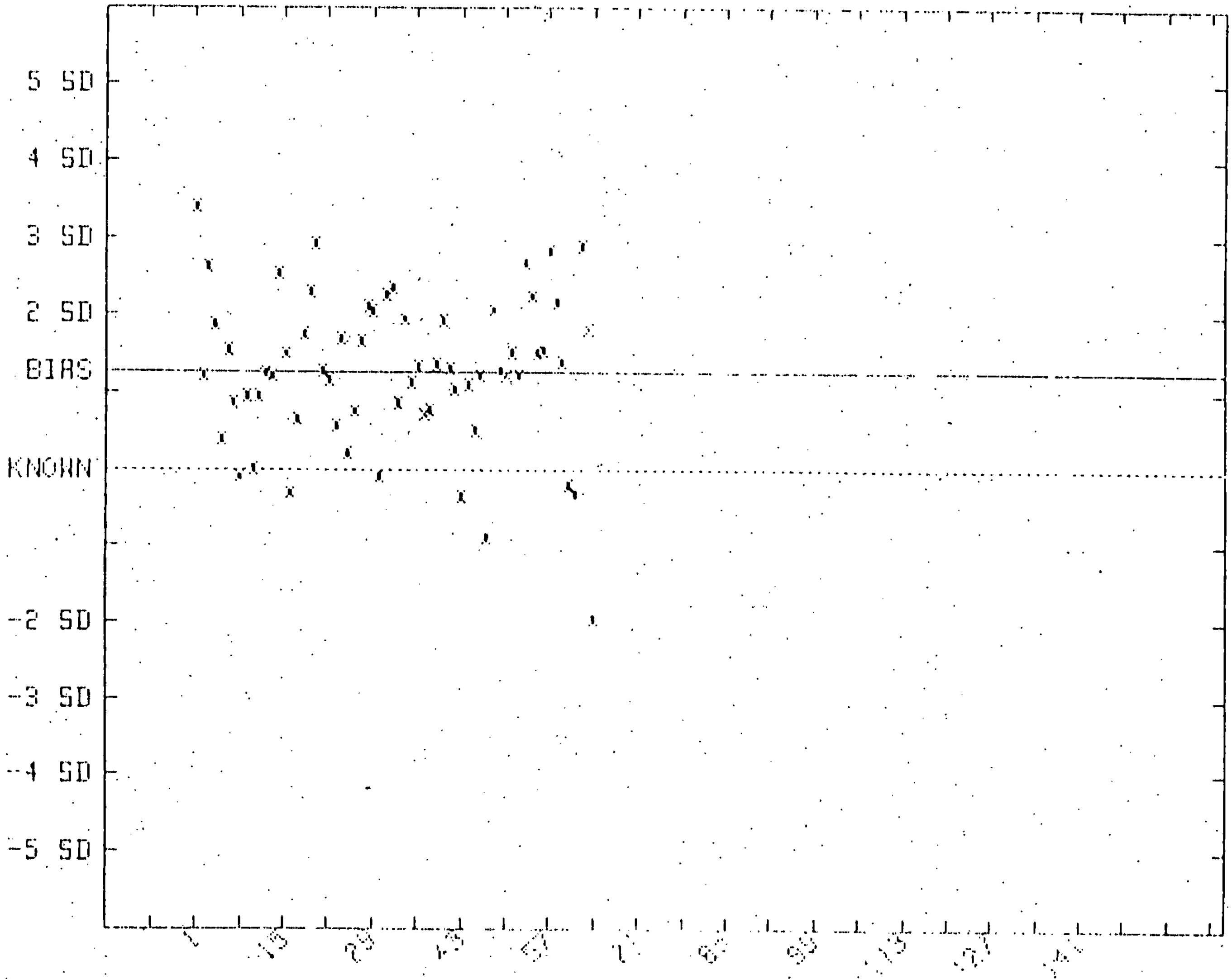

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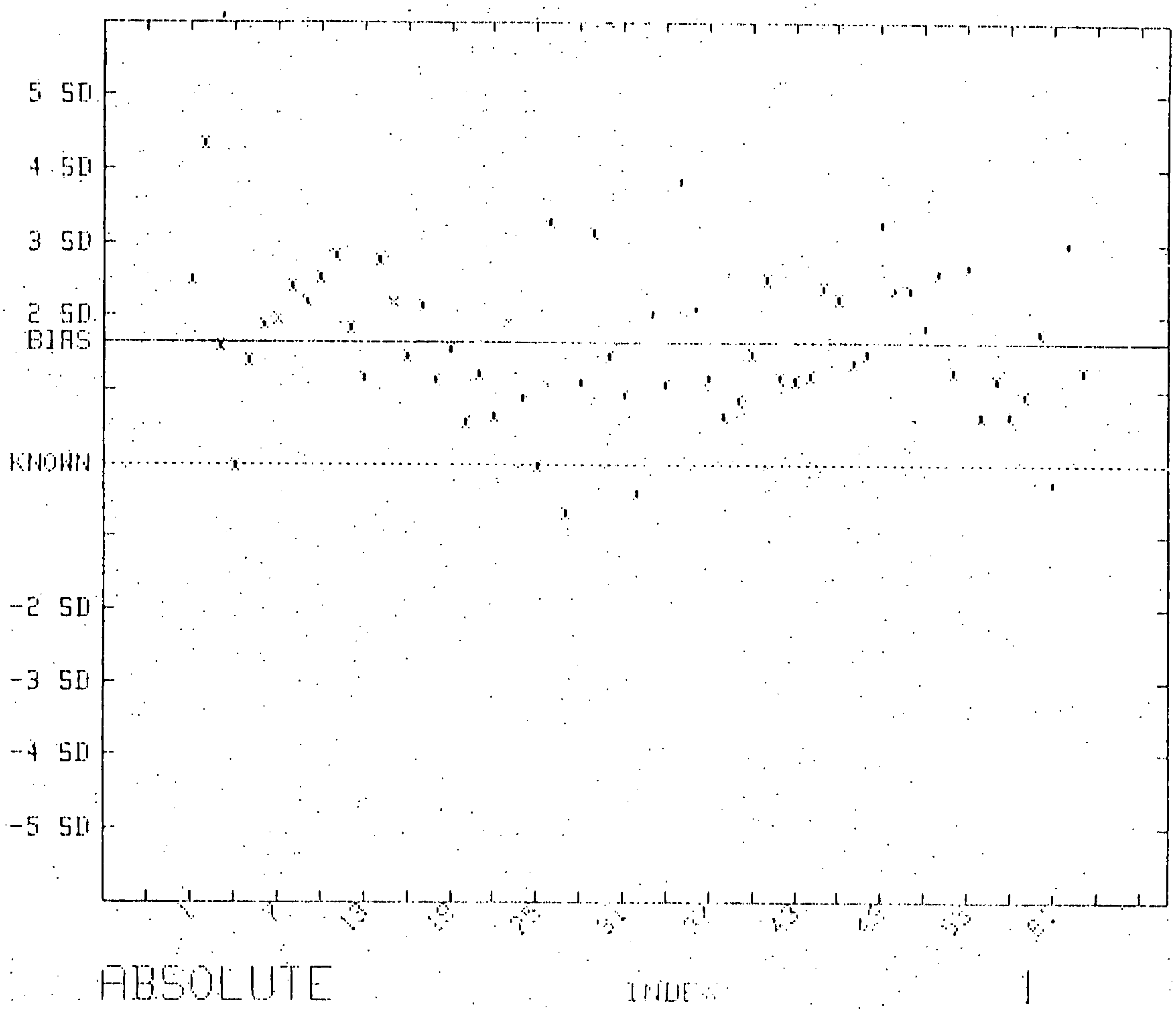

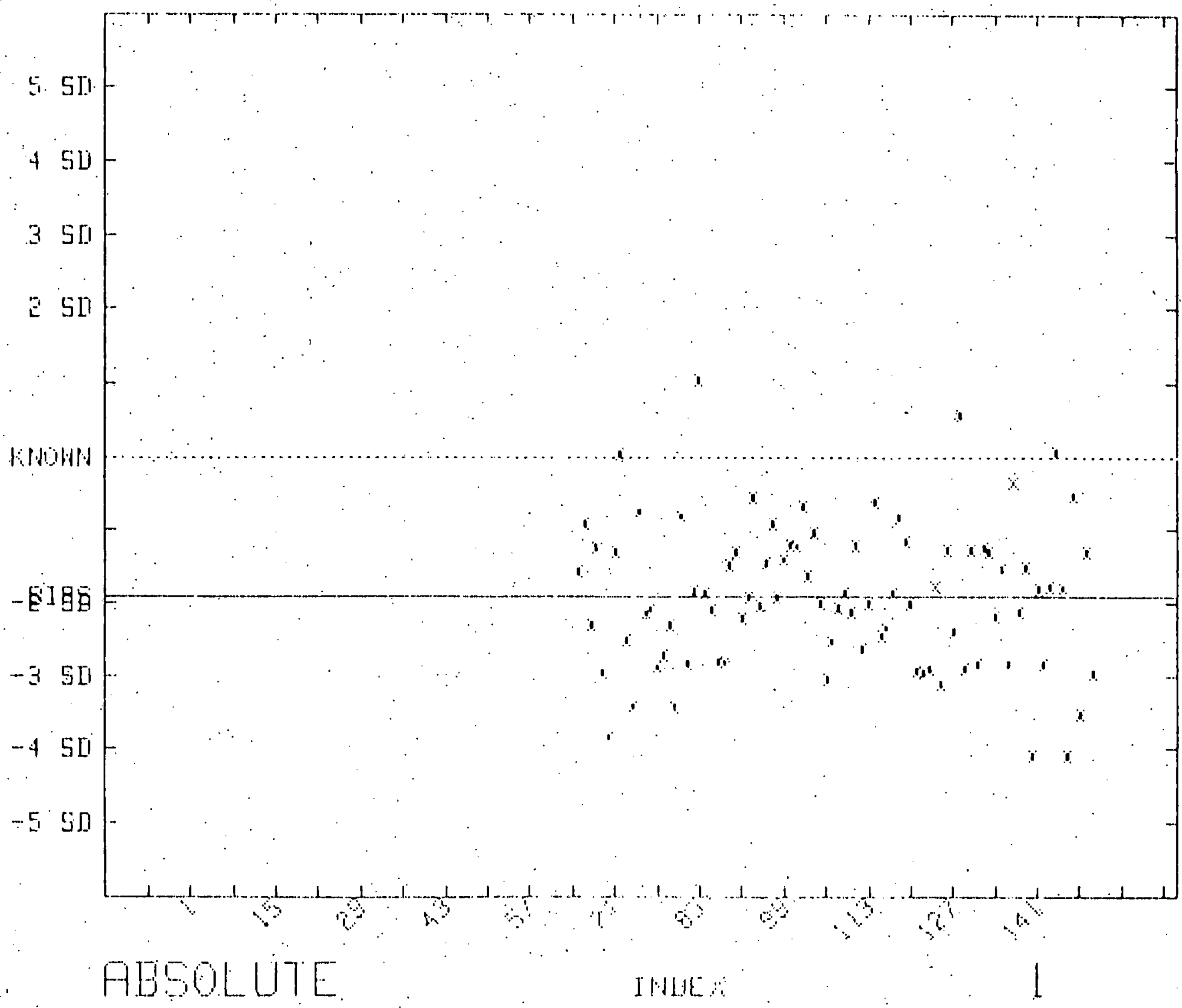
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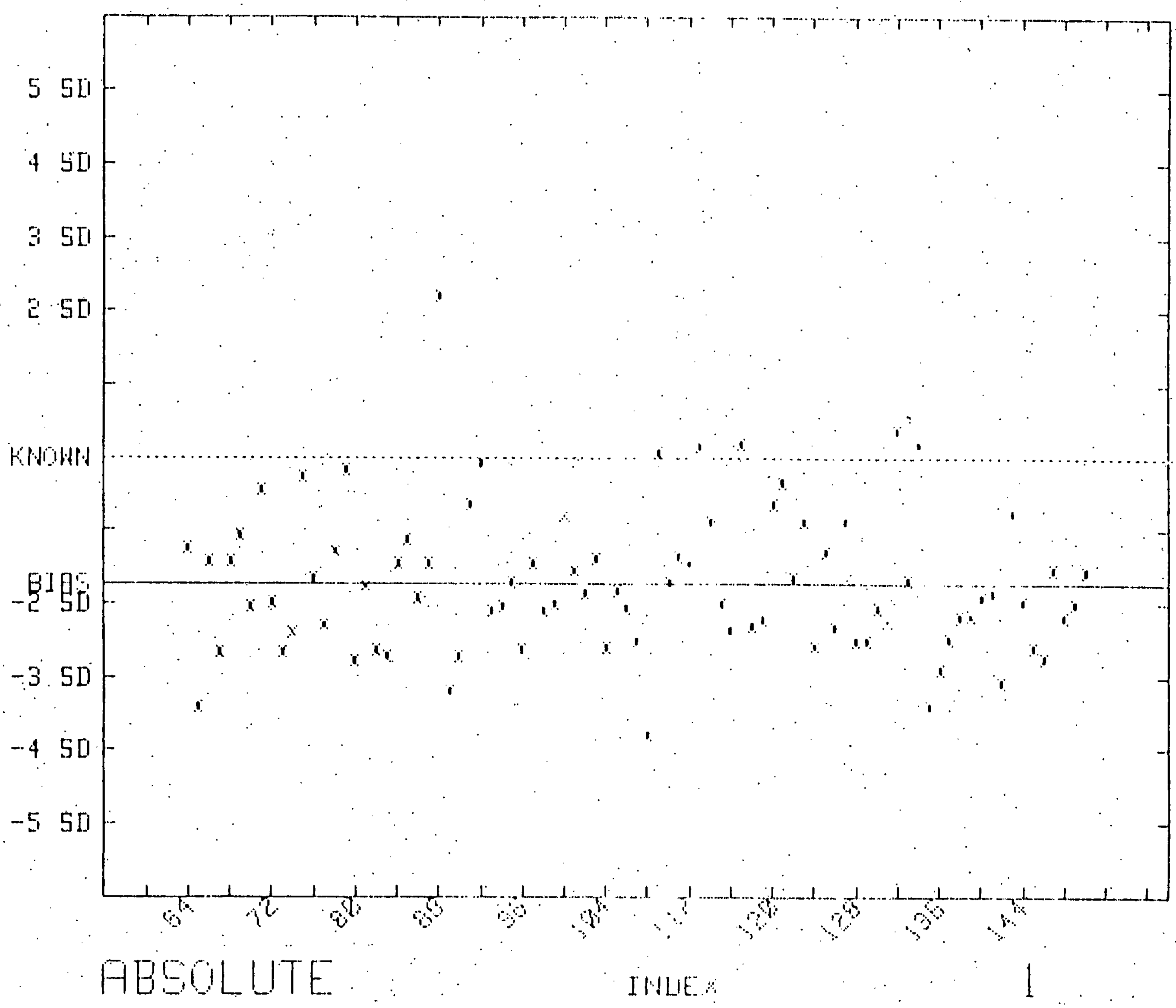
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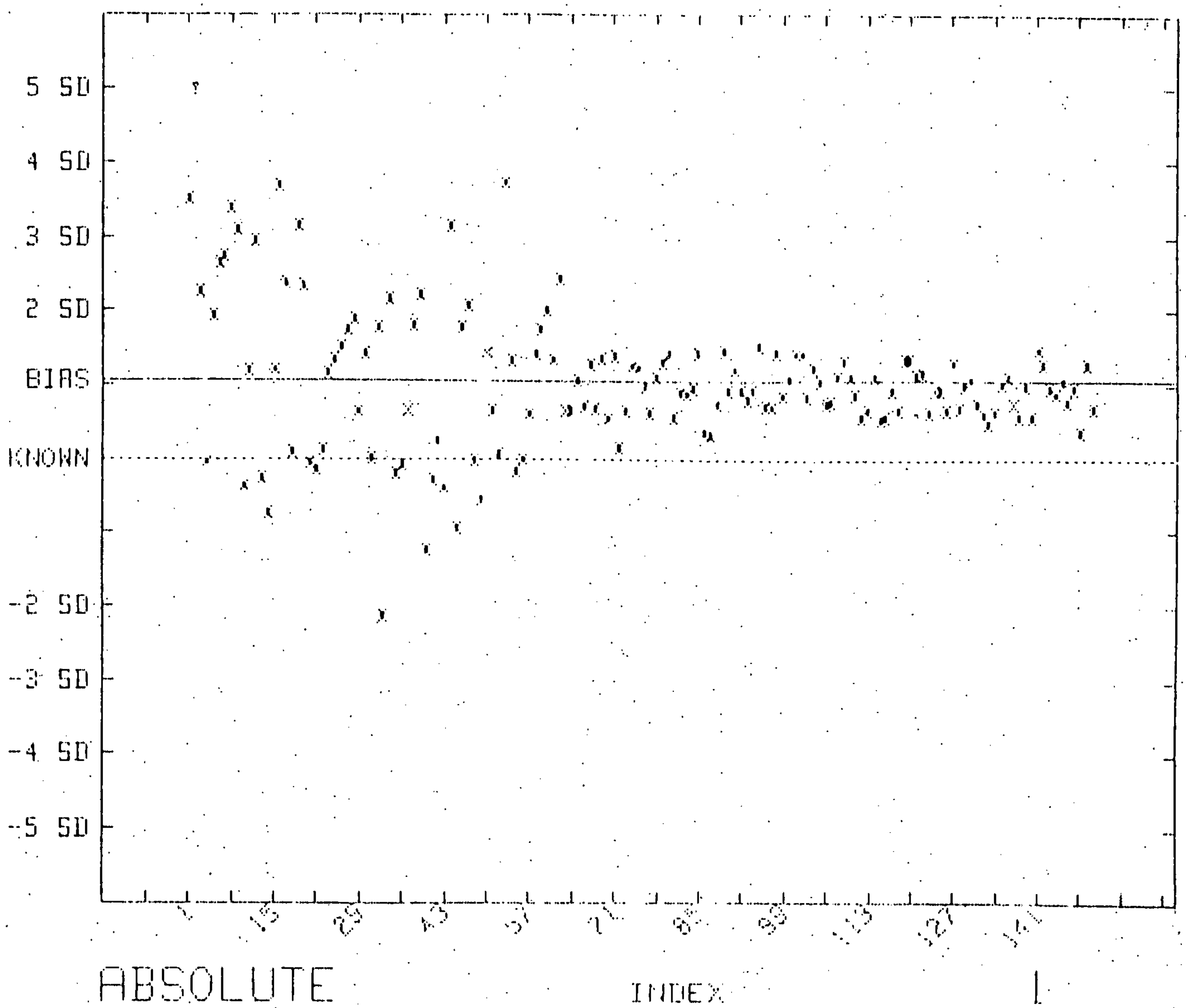
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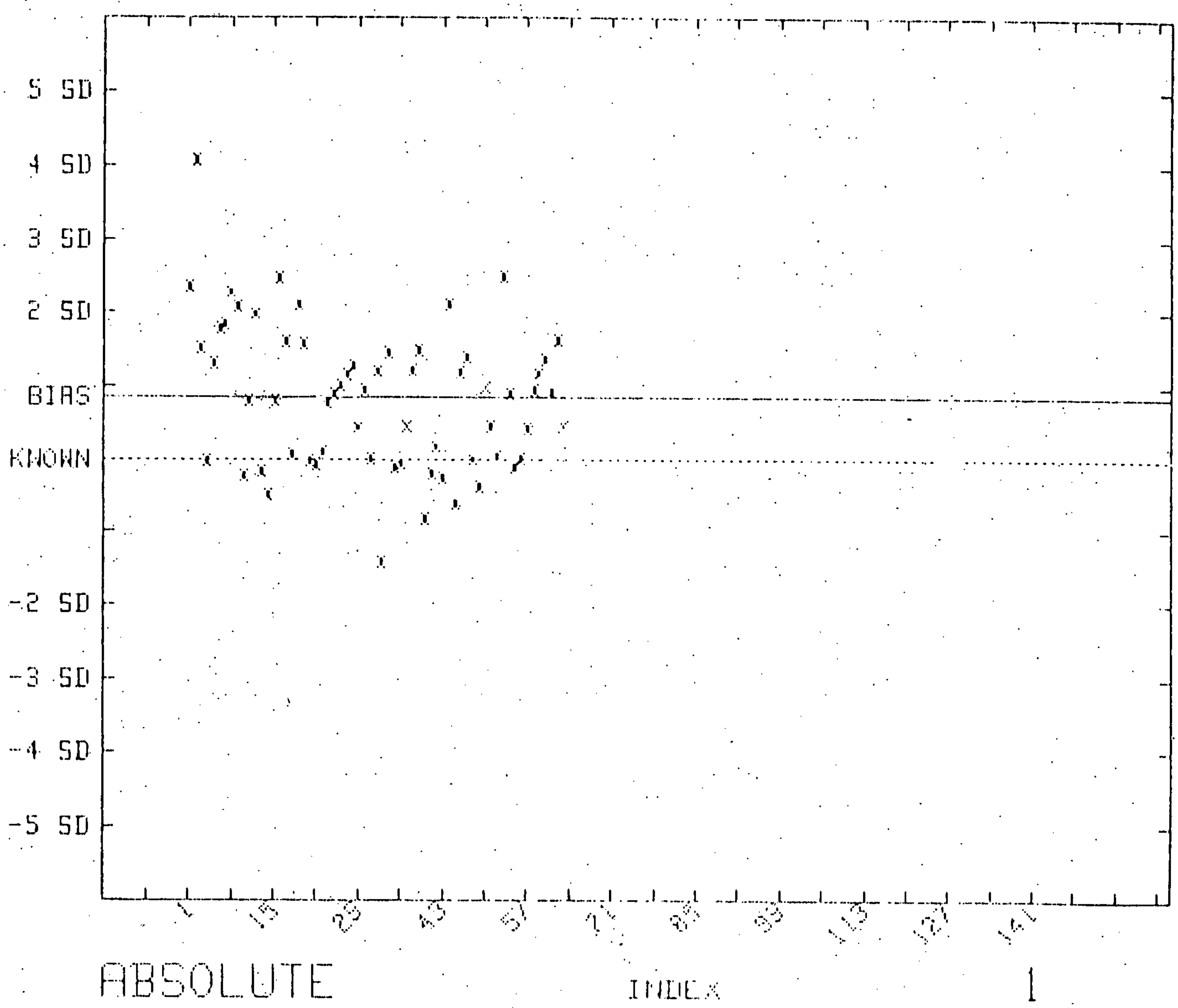
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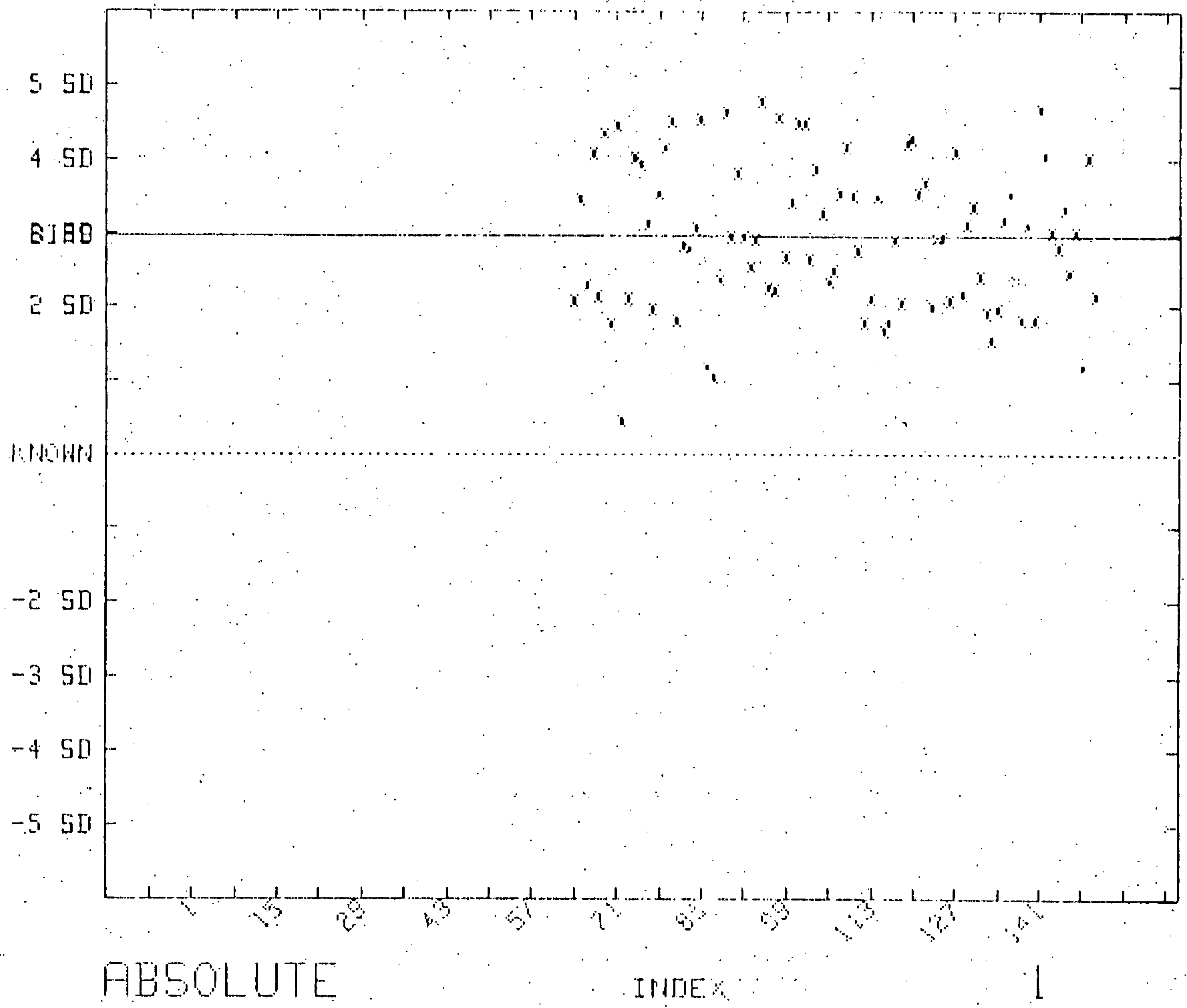
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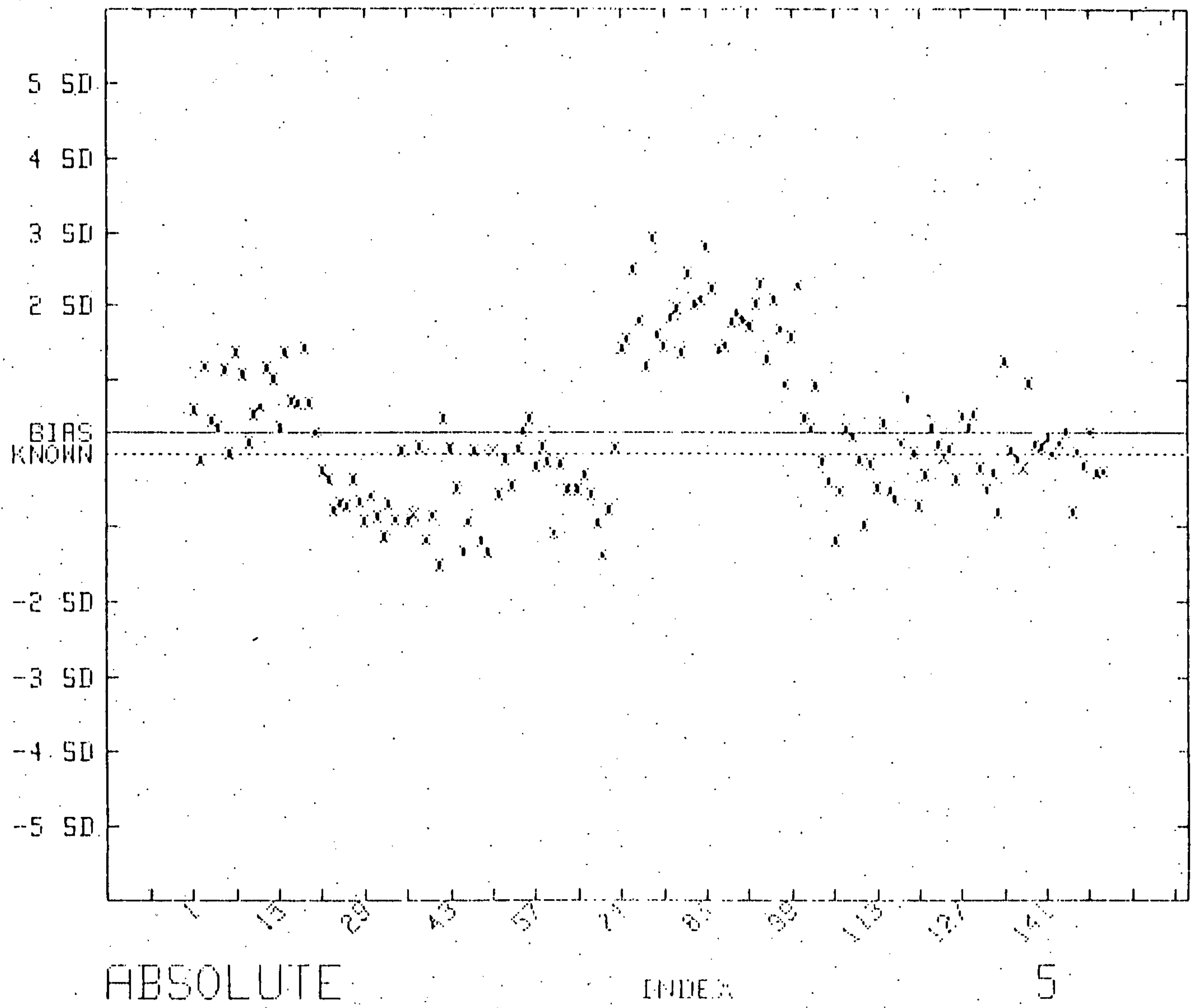
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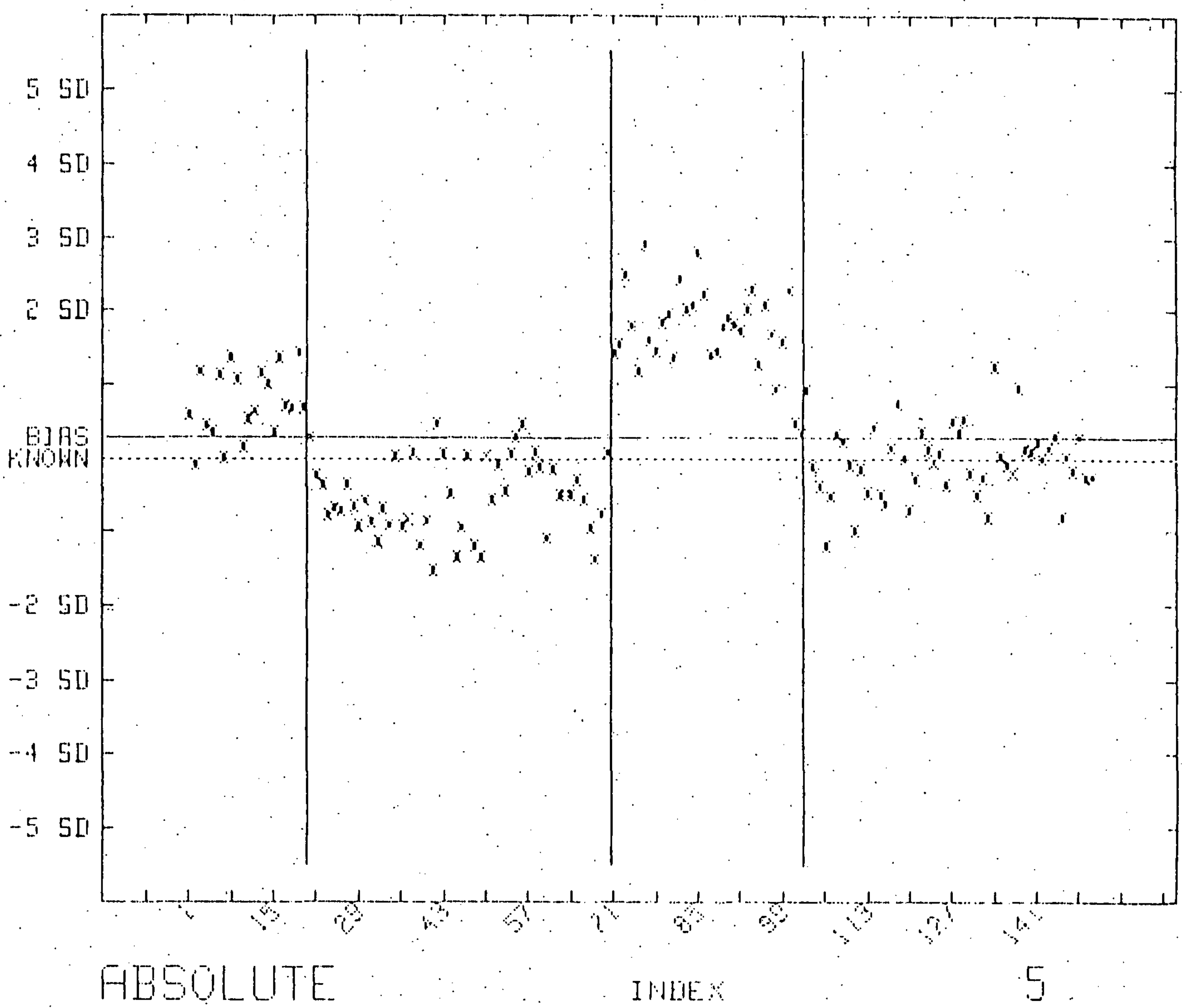
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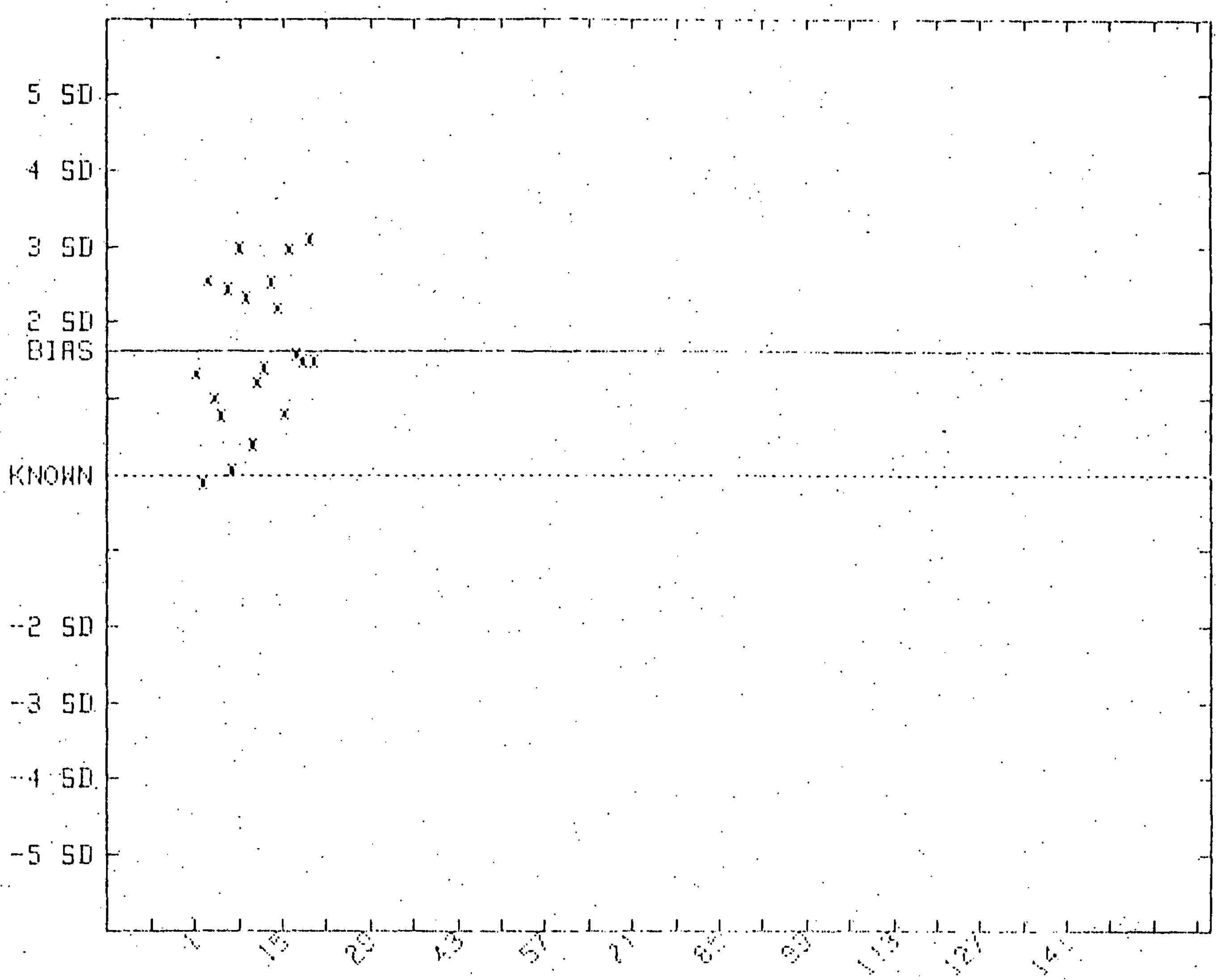

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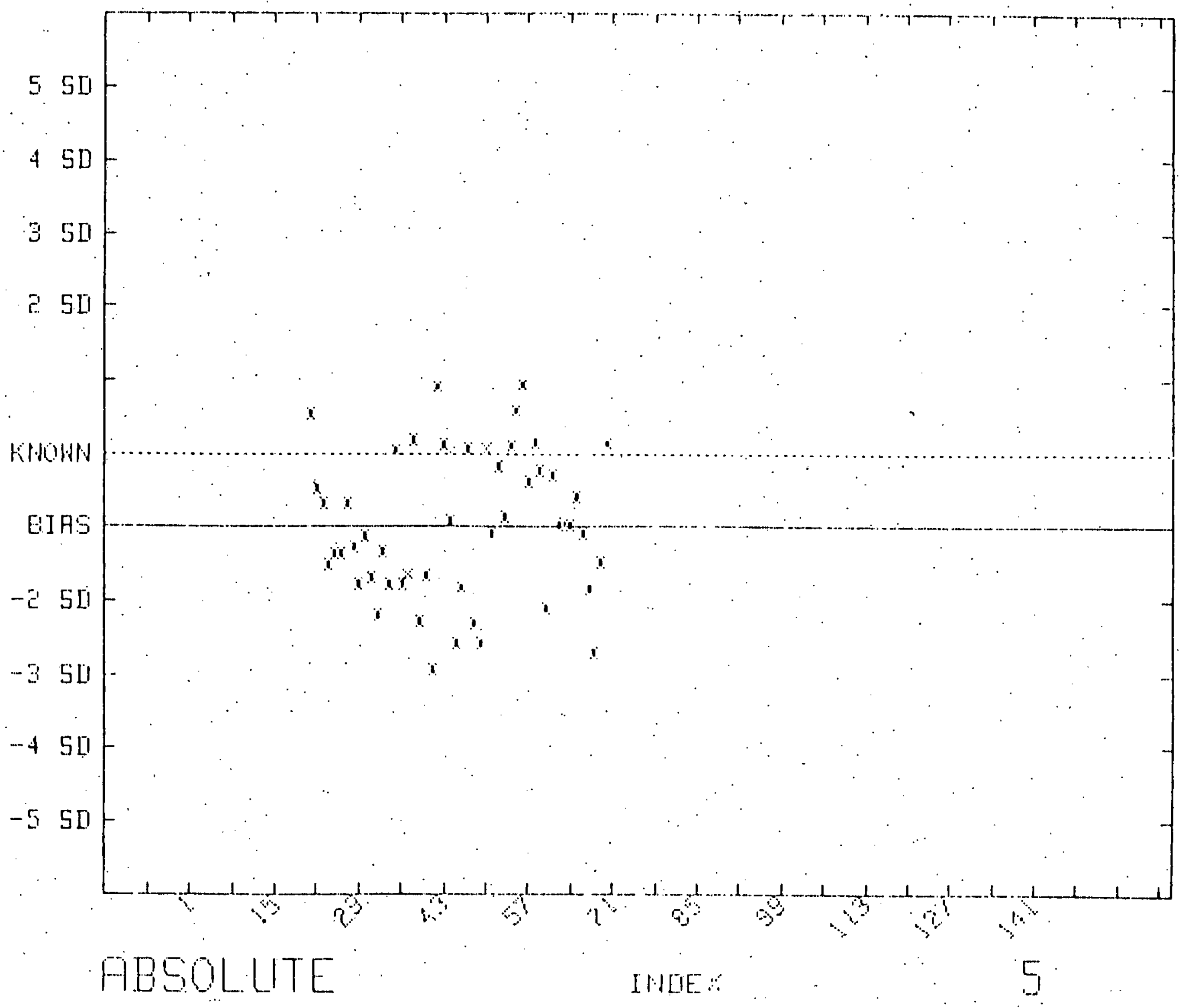
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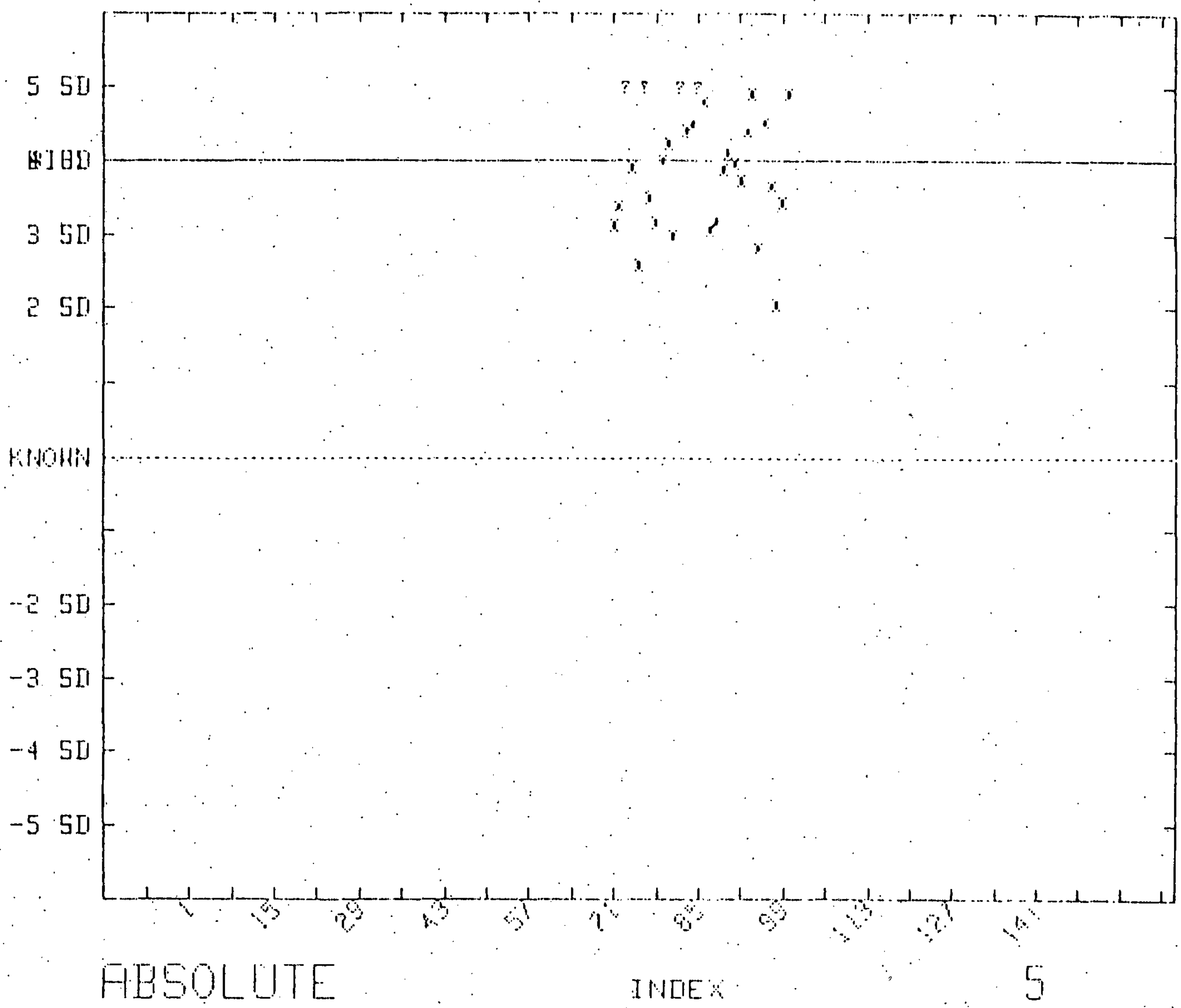
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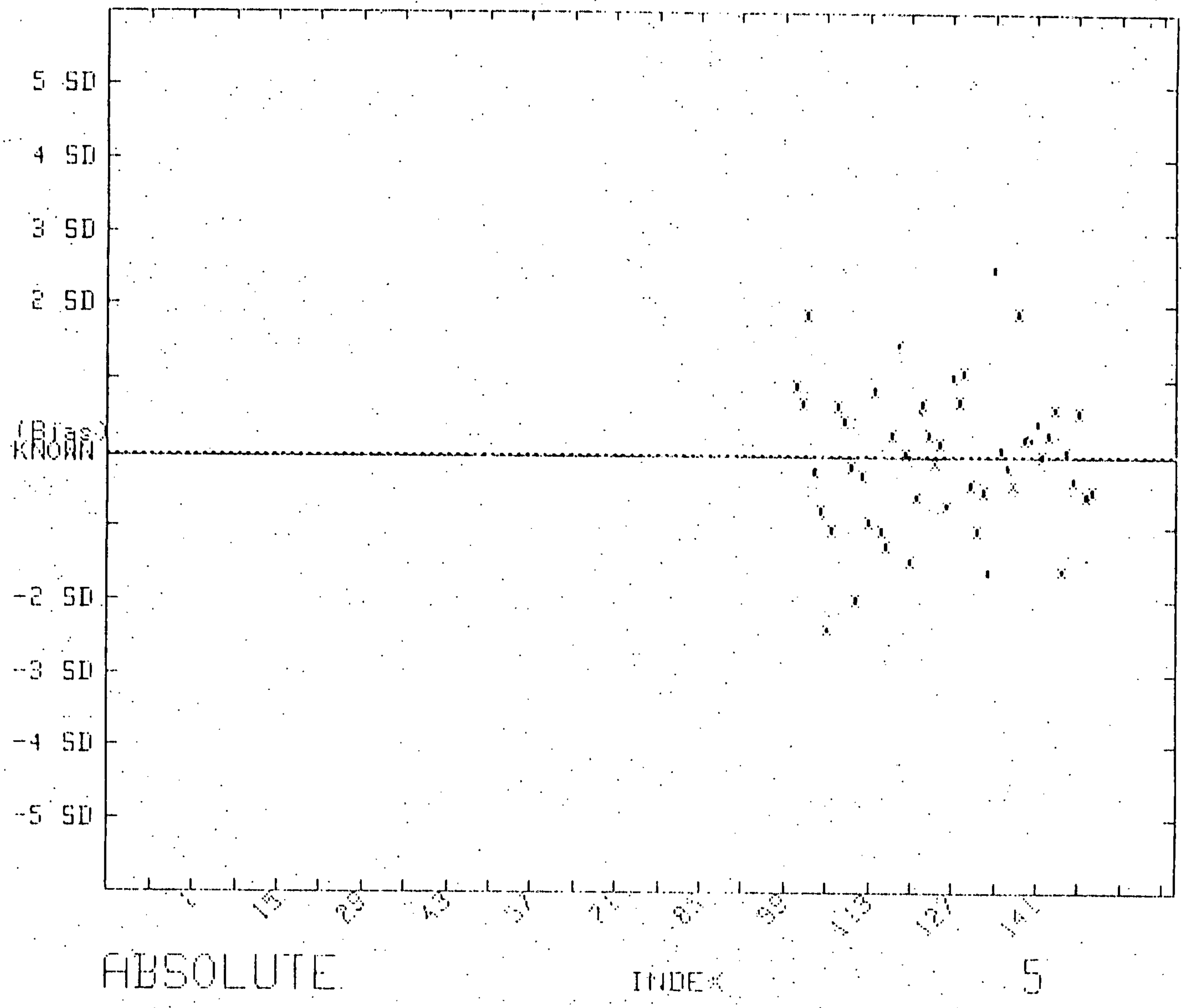
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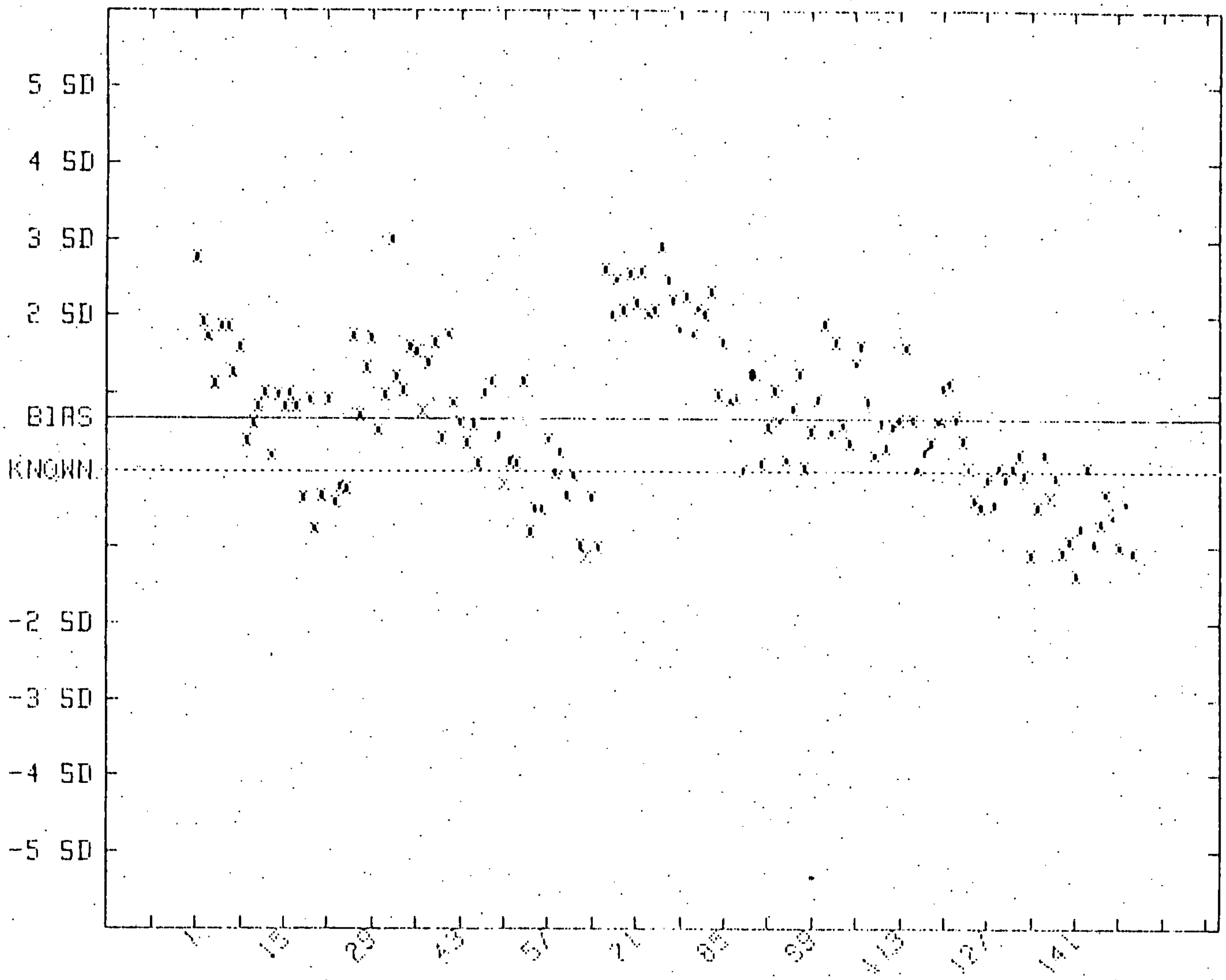

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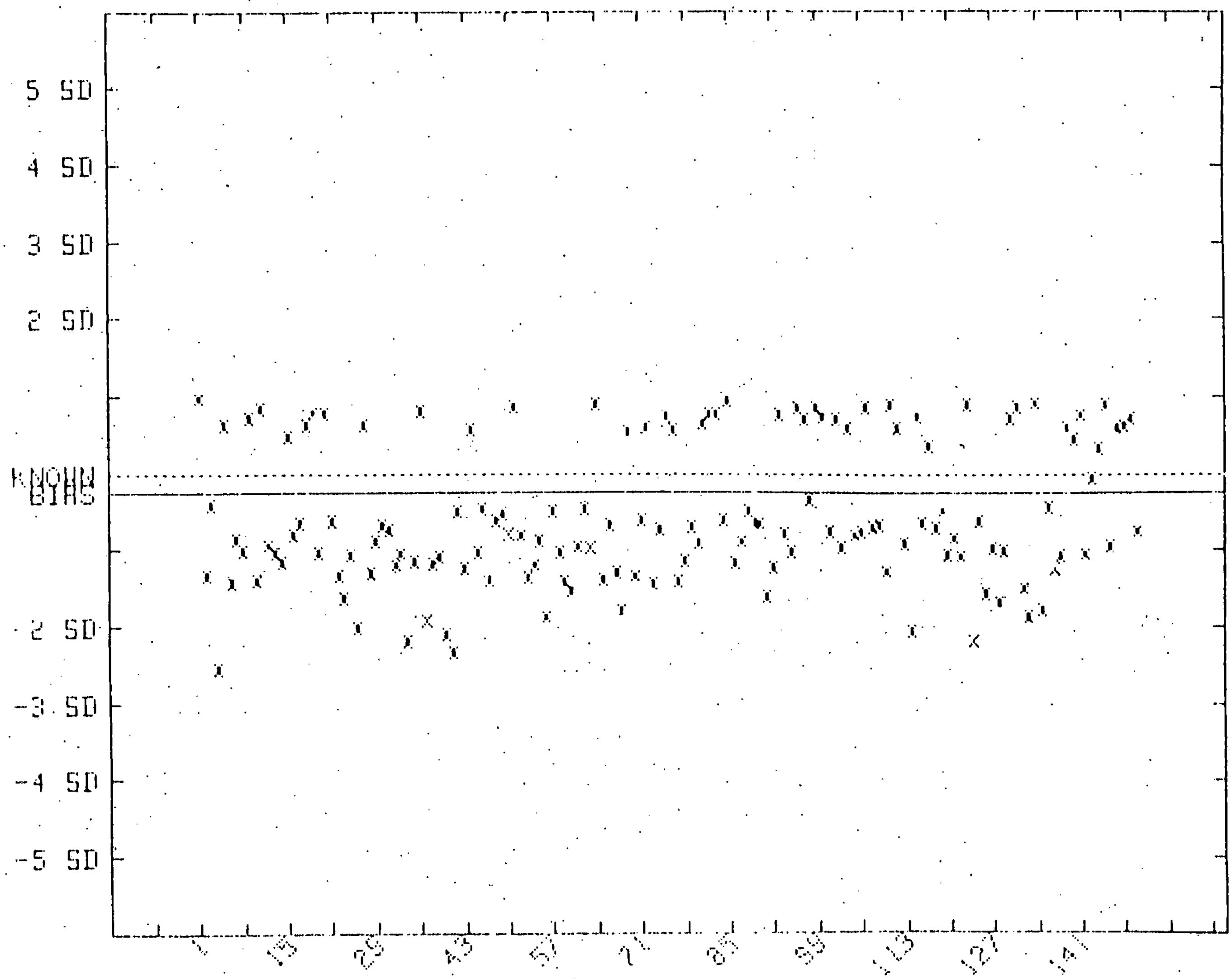

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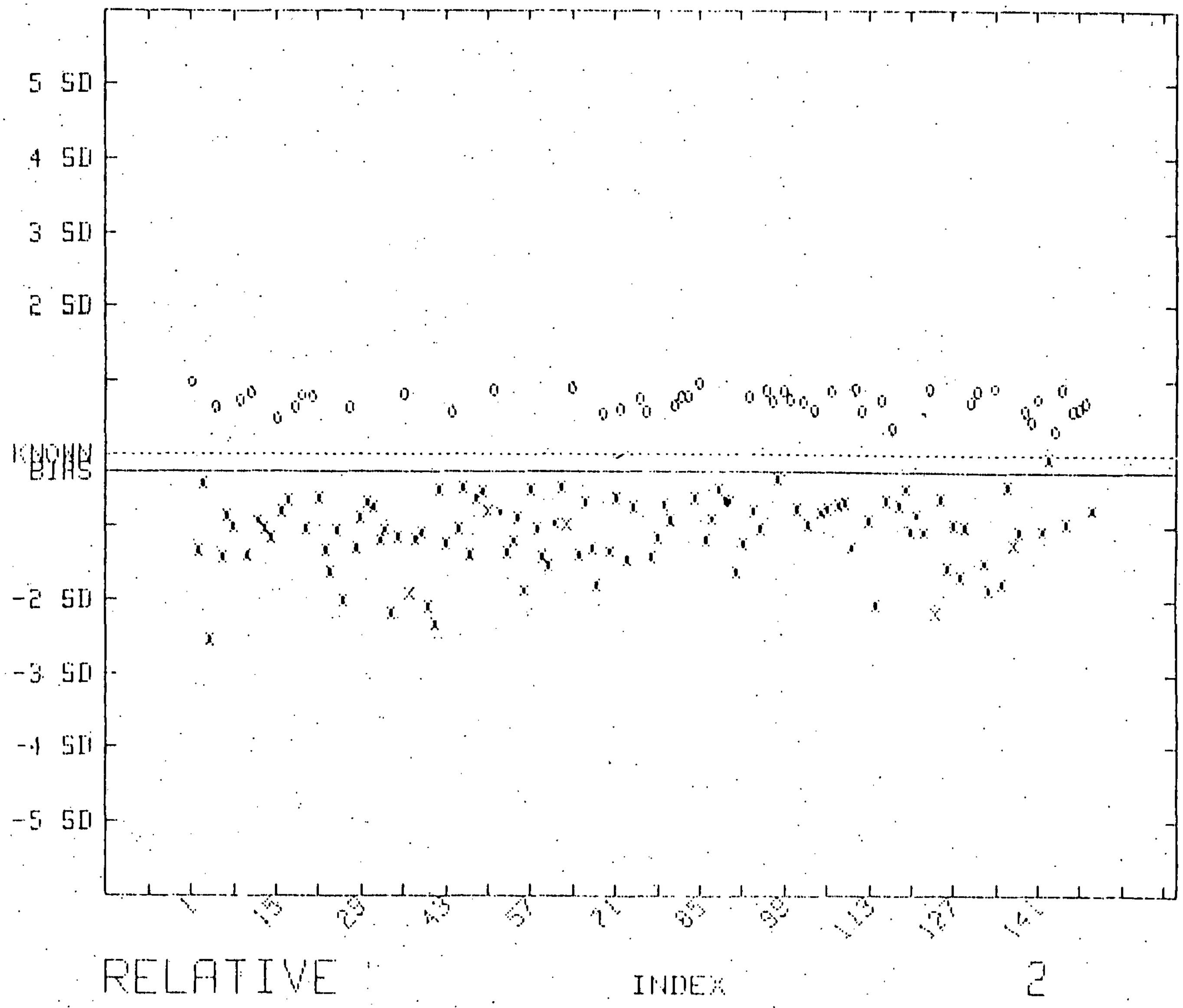
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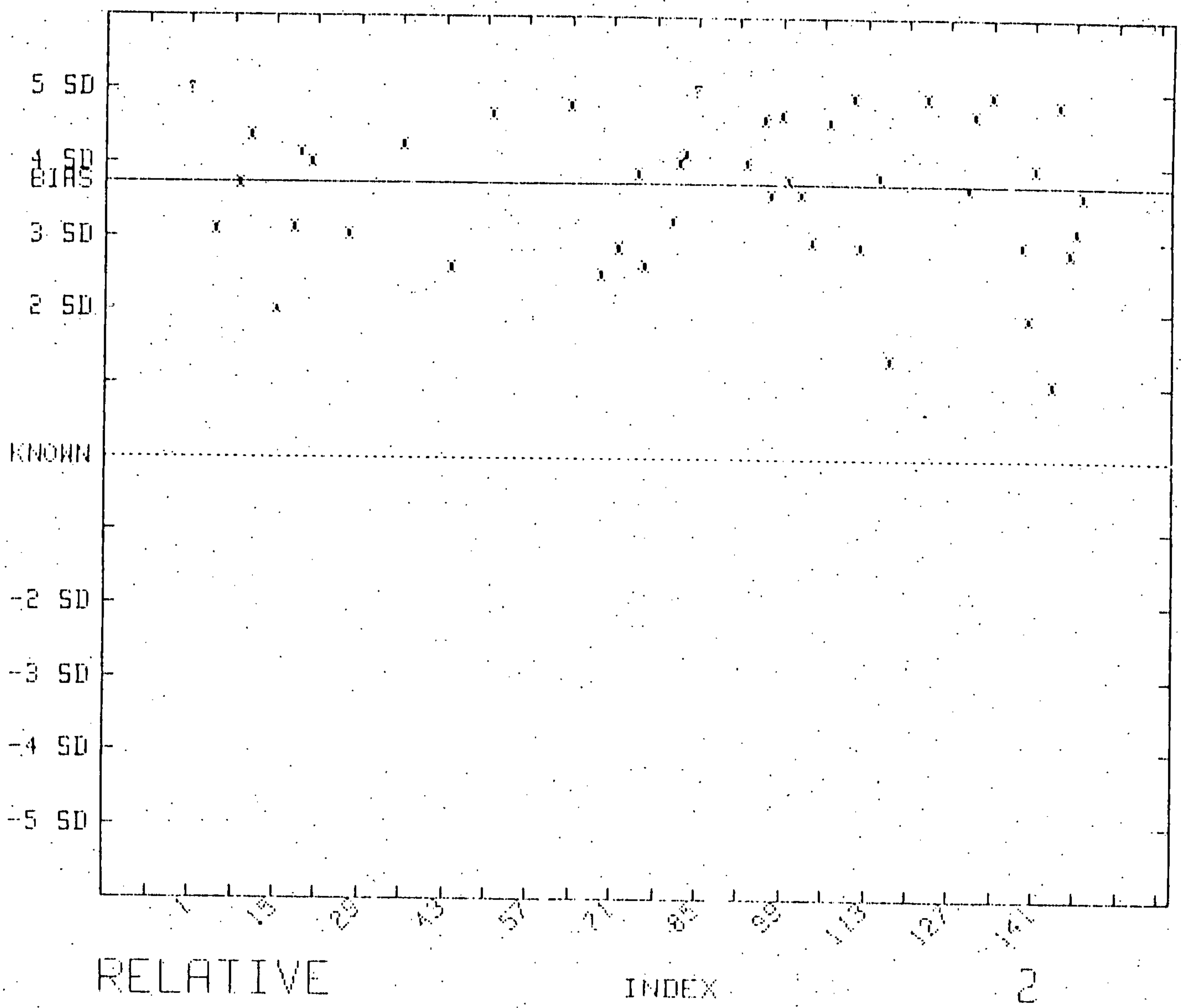
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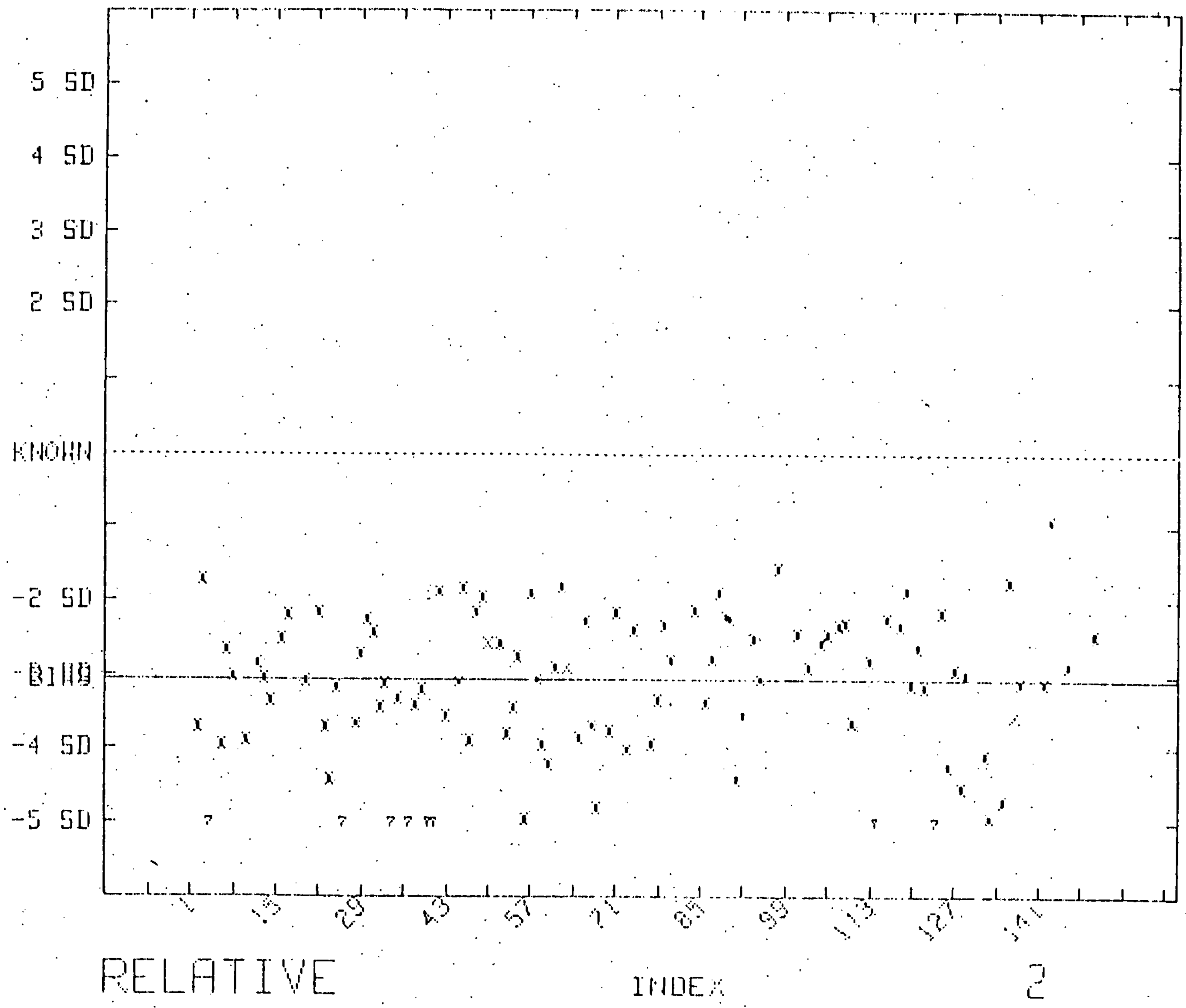
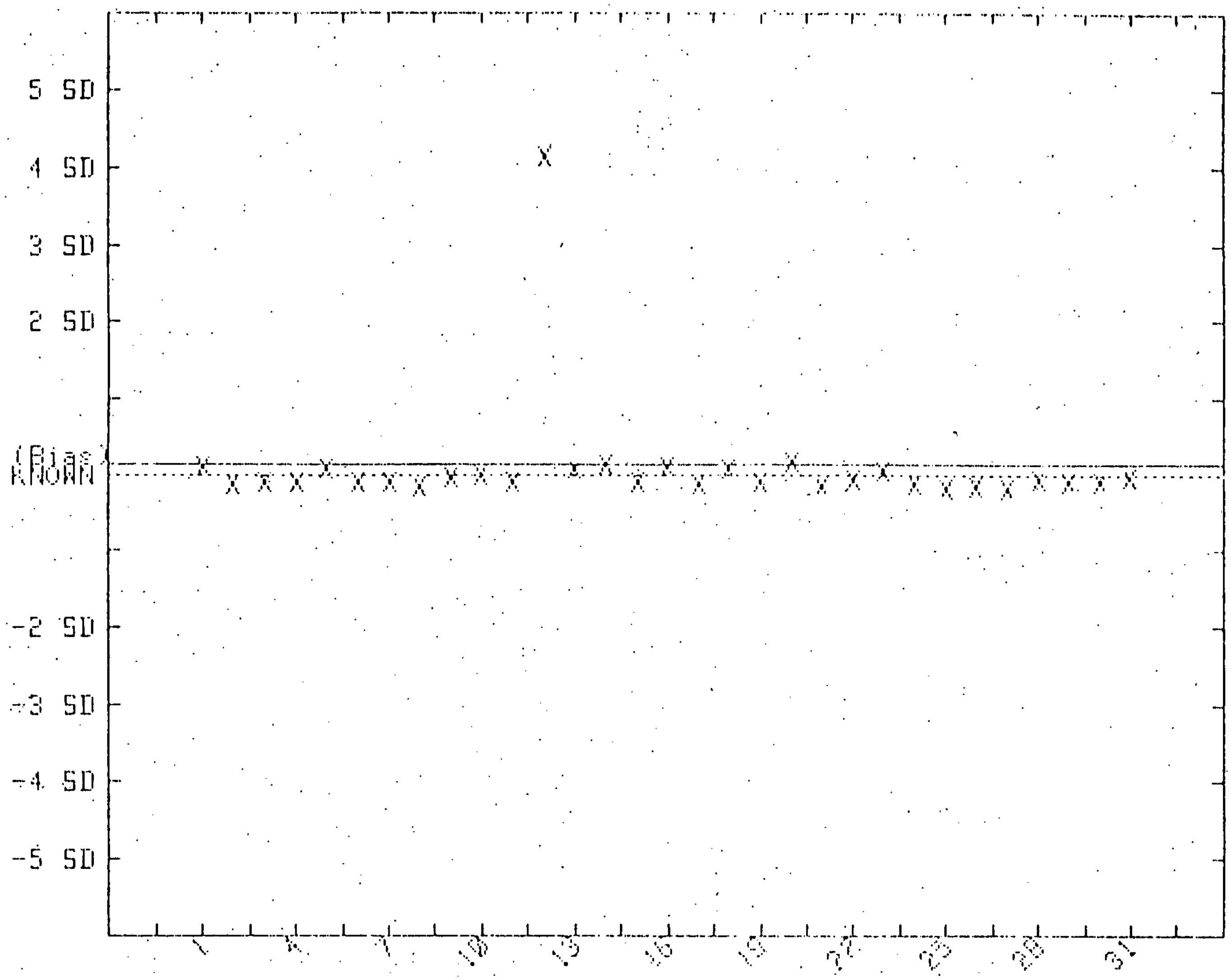
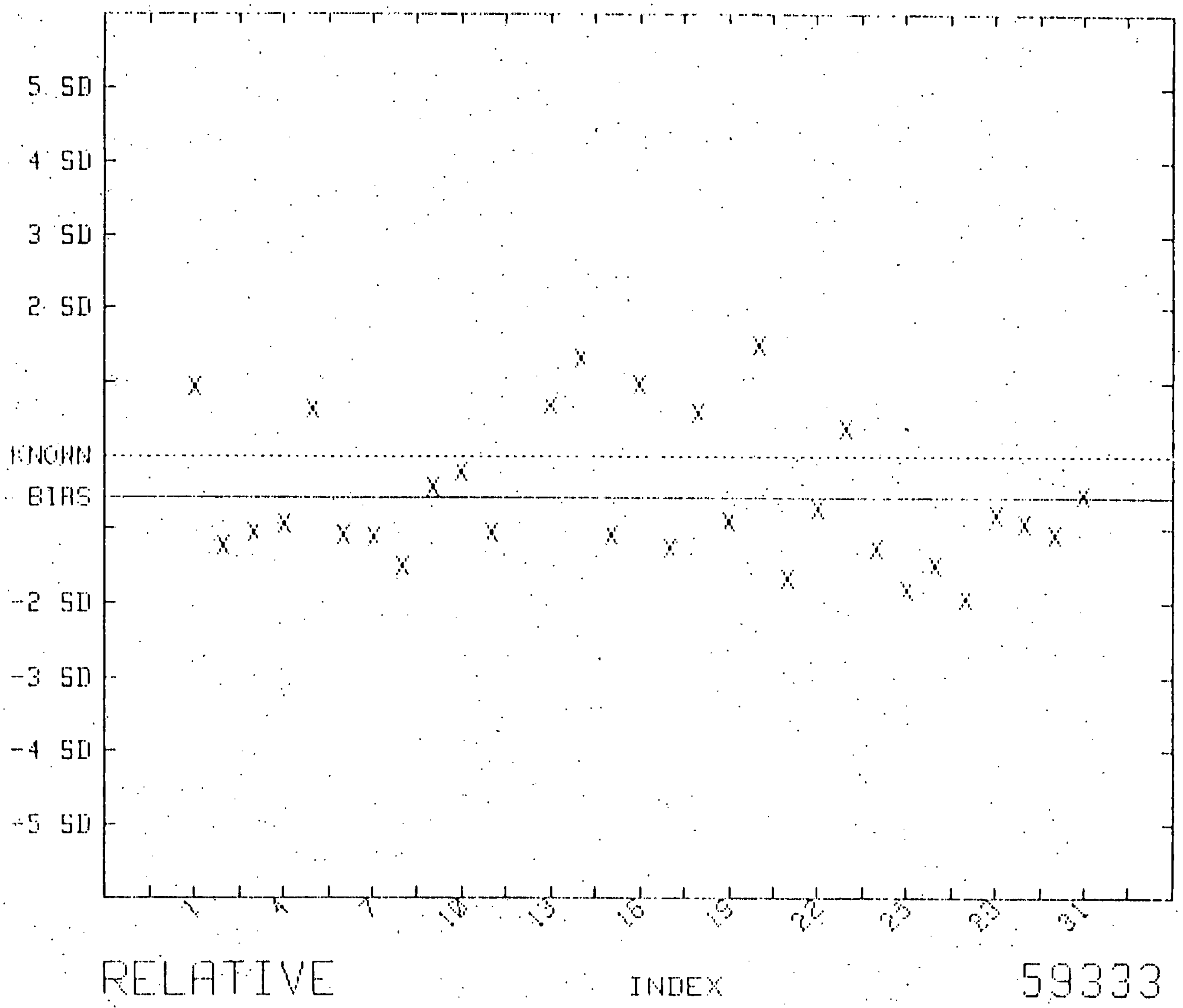

5 SD
4 SD
3 SD
2 SD
KNOWN
-2 SD
-4 SD
-5 SD
1
15
29
43
57
71
85
99
113
127
141
INDEX

RELATIVE 2

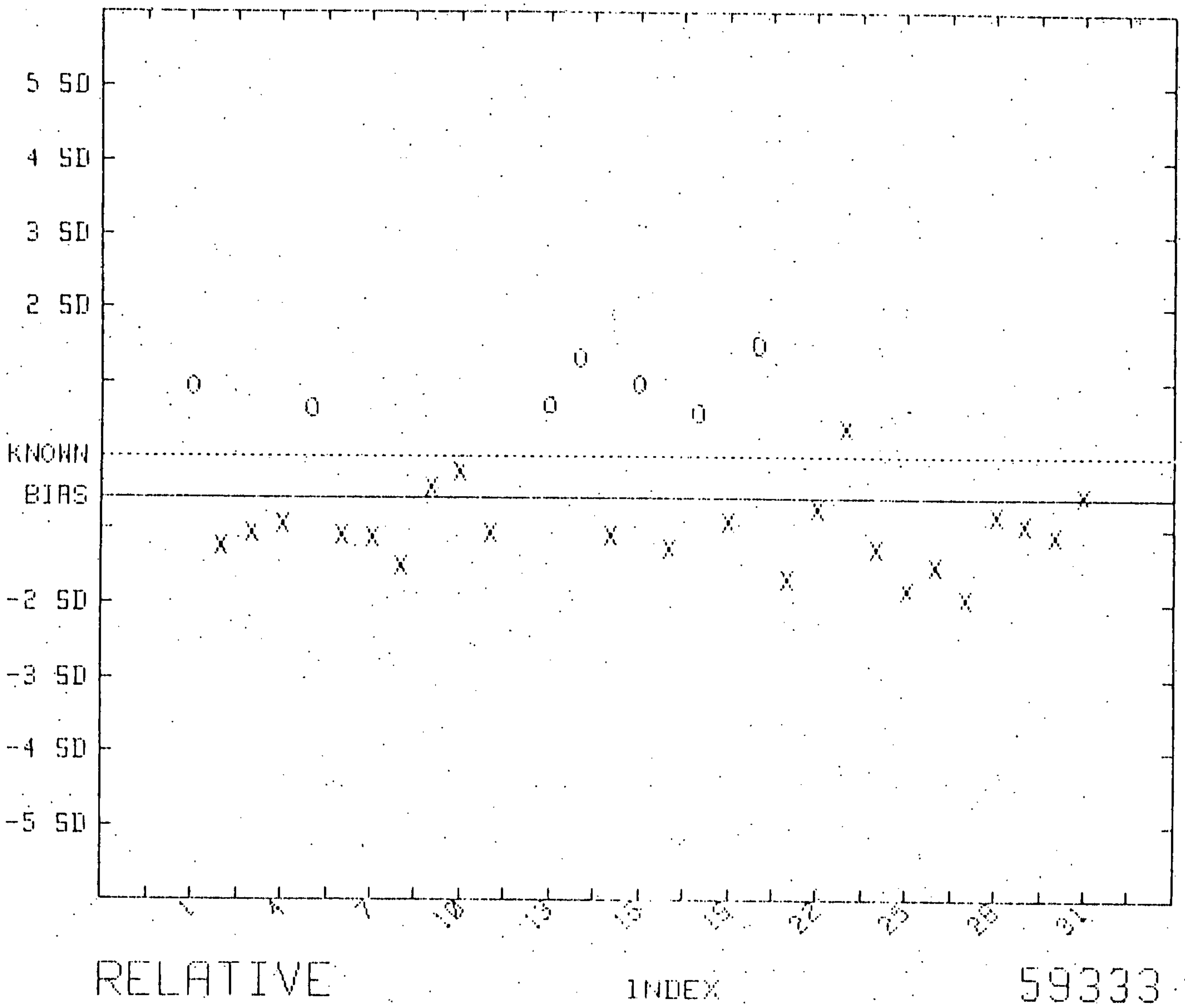
59333 Chi-Square Normalized RELATIVE 05/01/79

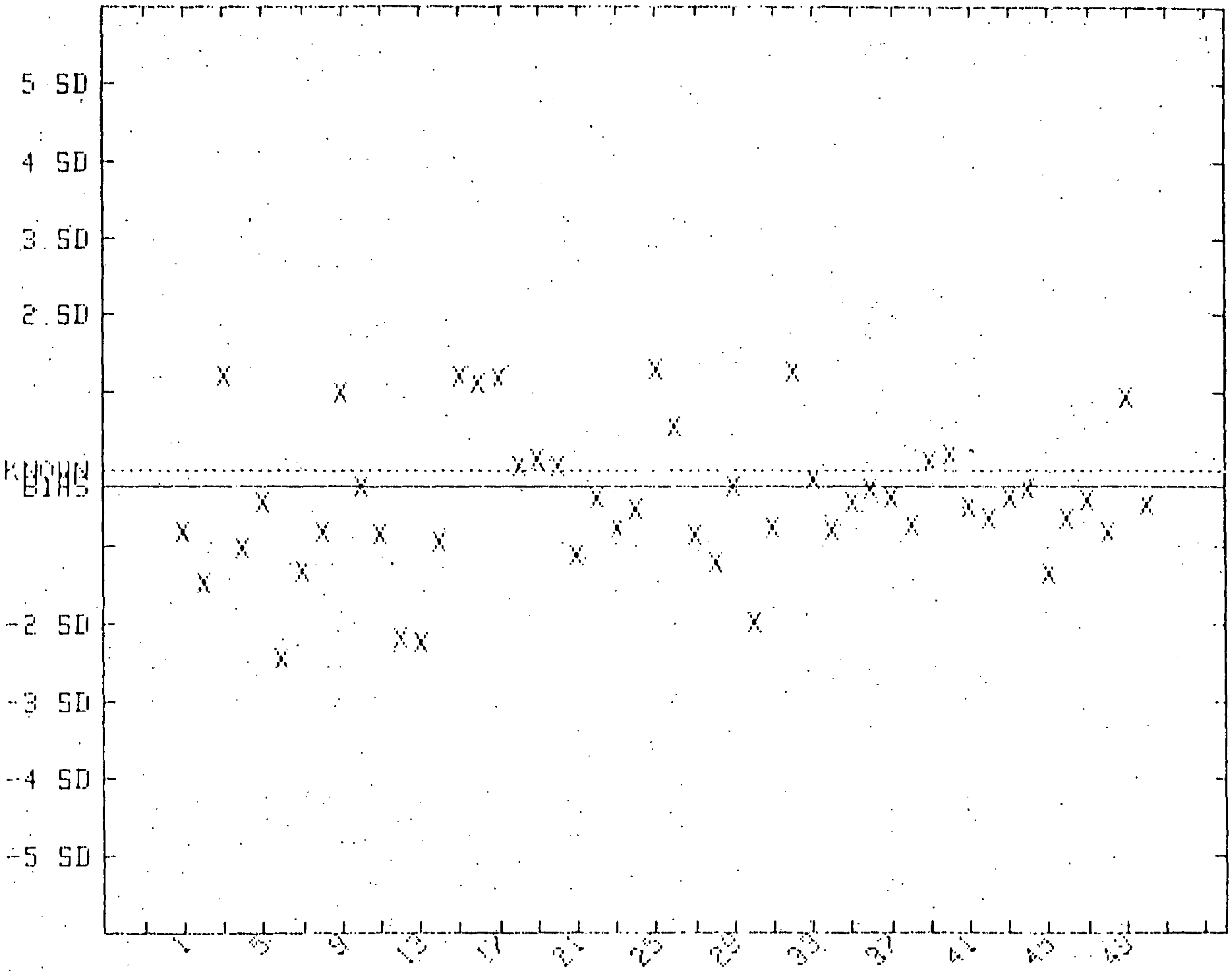


RELATIVE INDEX 59333


5 SD
4 SD
3 SD
2 SD
KNOWN
BIAS
-2 SD
-3 SD
-4 SD
-5 SD
1
4
7
10
13
16
19
22
25
28
31
RELATIVE
INDEX
59333

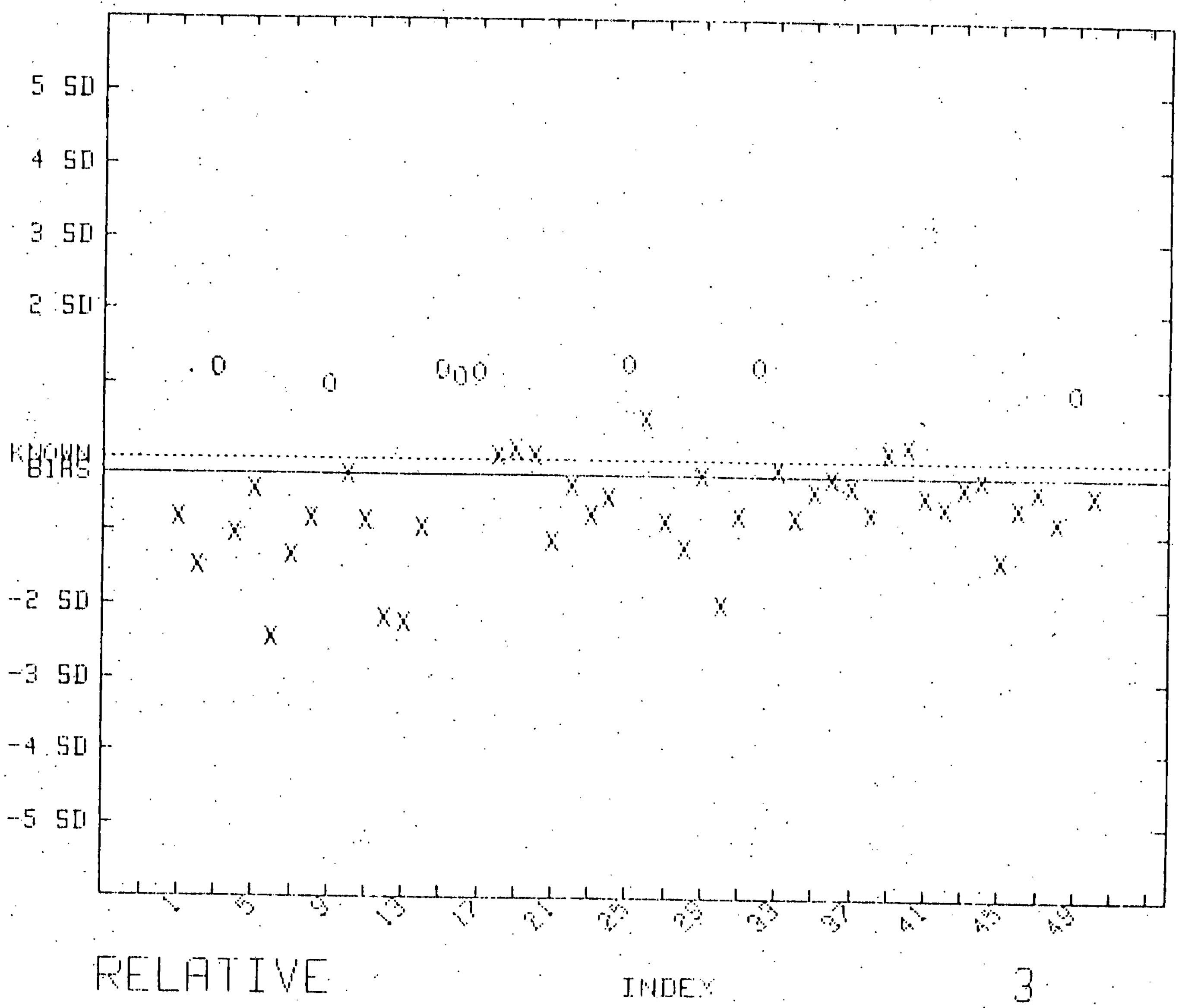
59333 Chi-Square ANALYST: CDAB RELATIVE 05/01/79



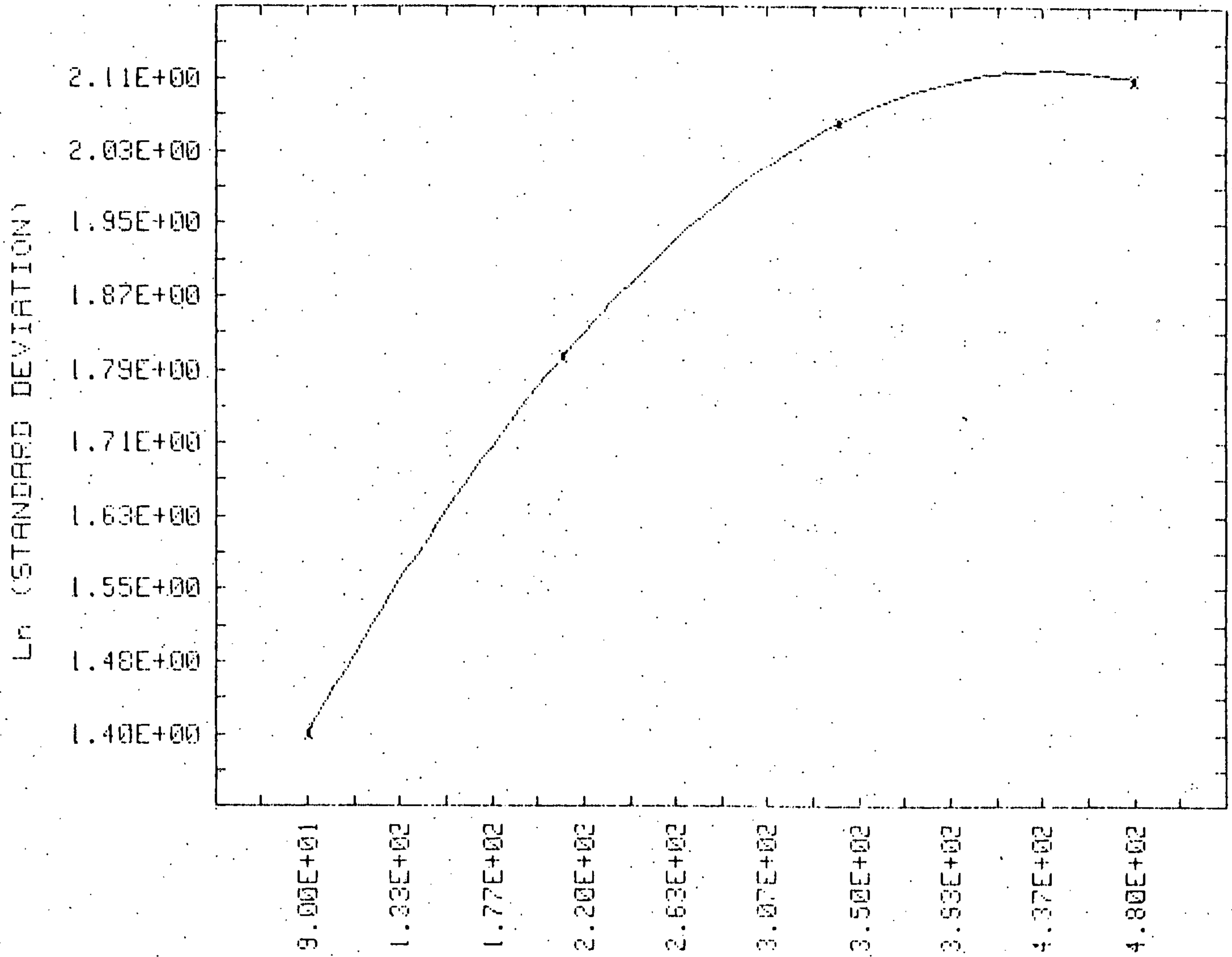

5 SD
4 SD
3 SD
2 SD
KNOWN
BIAS
-2 SD
-3 SD
-4 SD
-5 SD
1
5
9
13
17
21
25
29
33
37
41
45
49
INDEX

RELATIVE 3

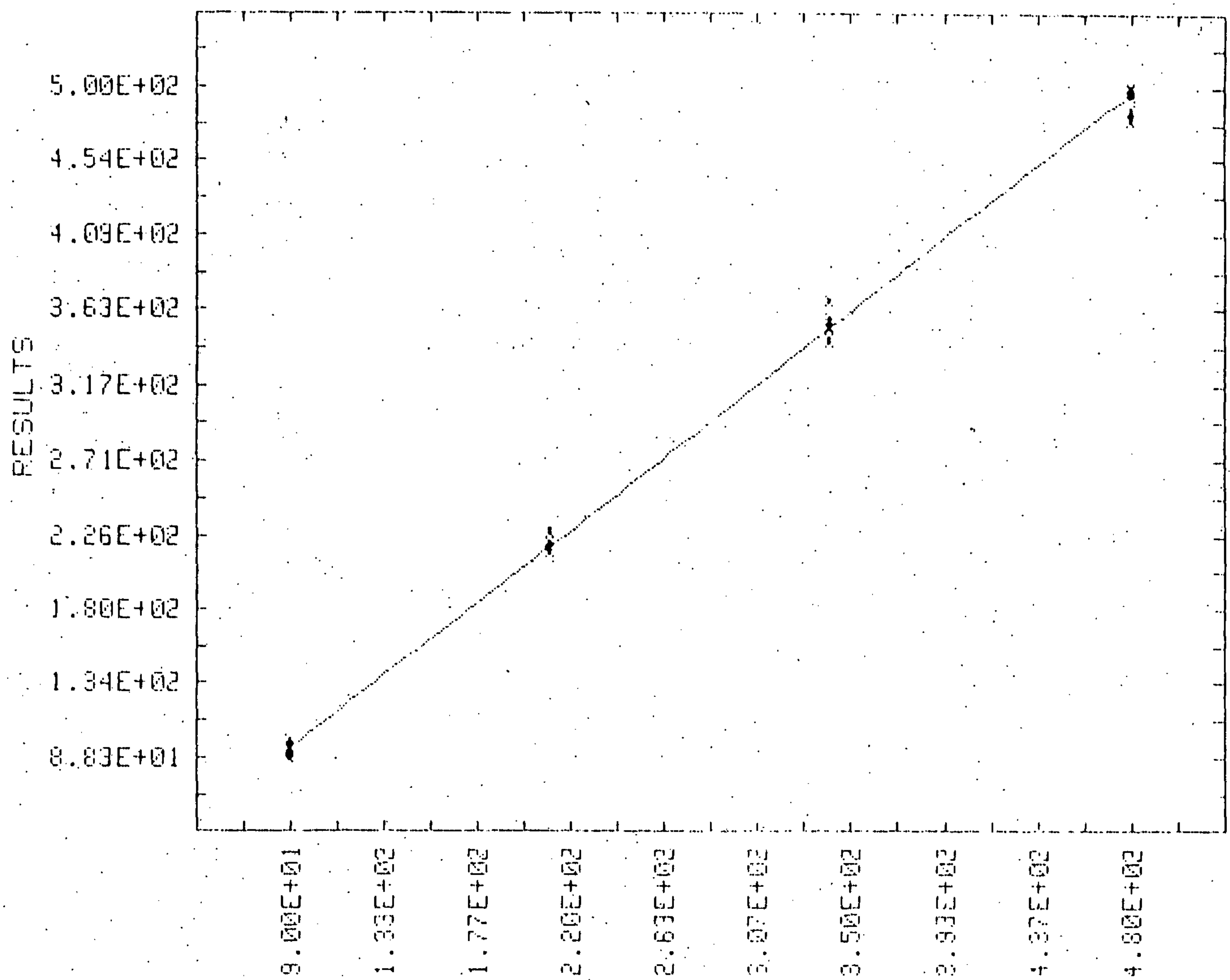
Normality ANALYST: HHA RELATIVE 05/31/79



5401   Zr-AA-1   UG/mL   DATA BASE   MULTI-LEVEL   05/01/79

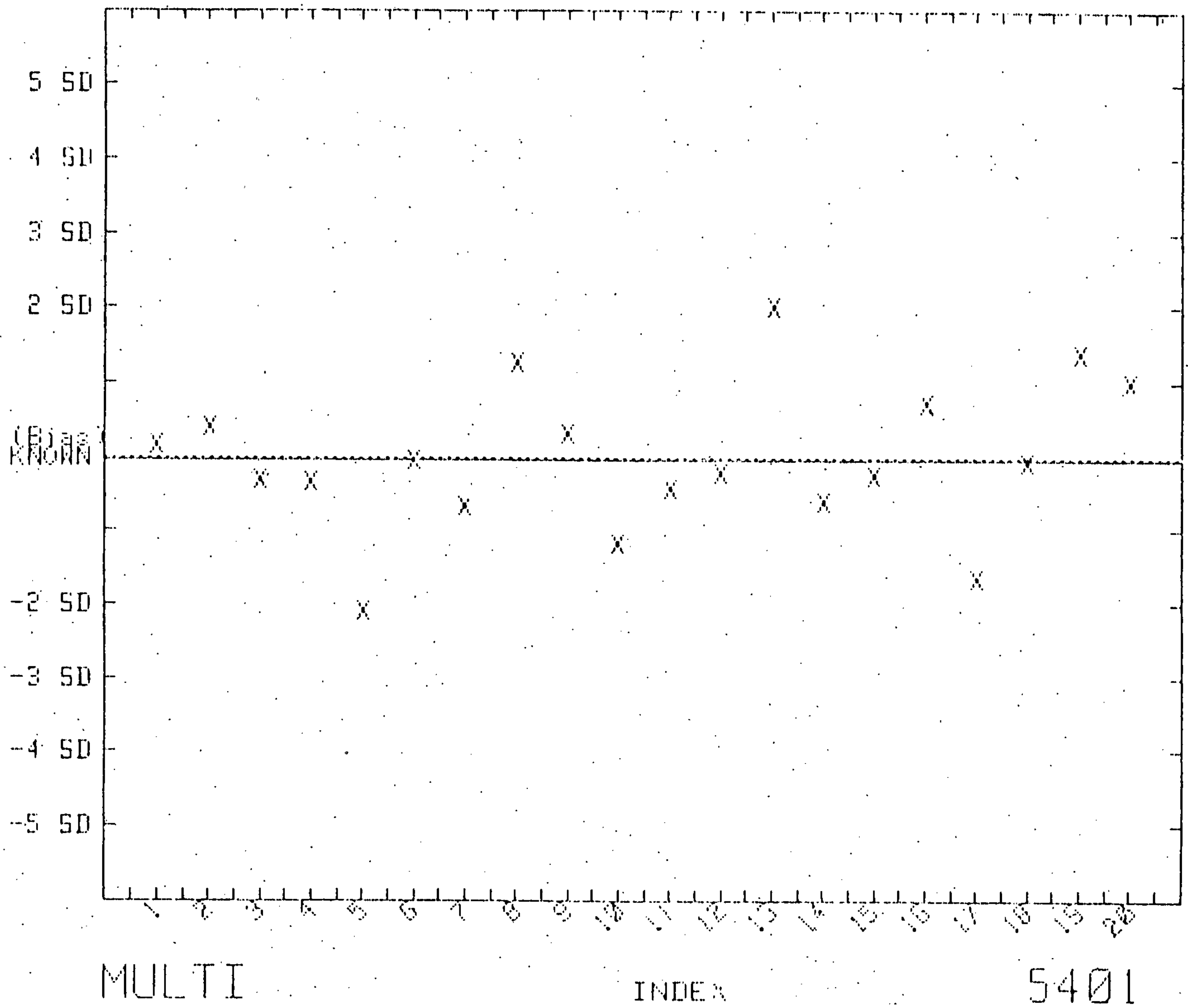


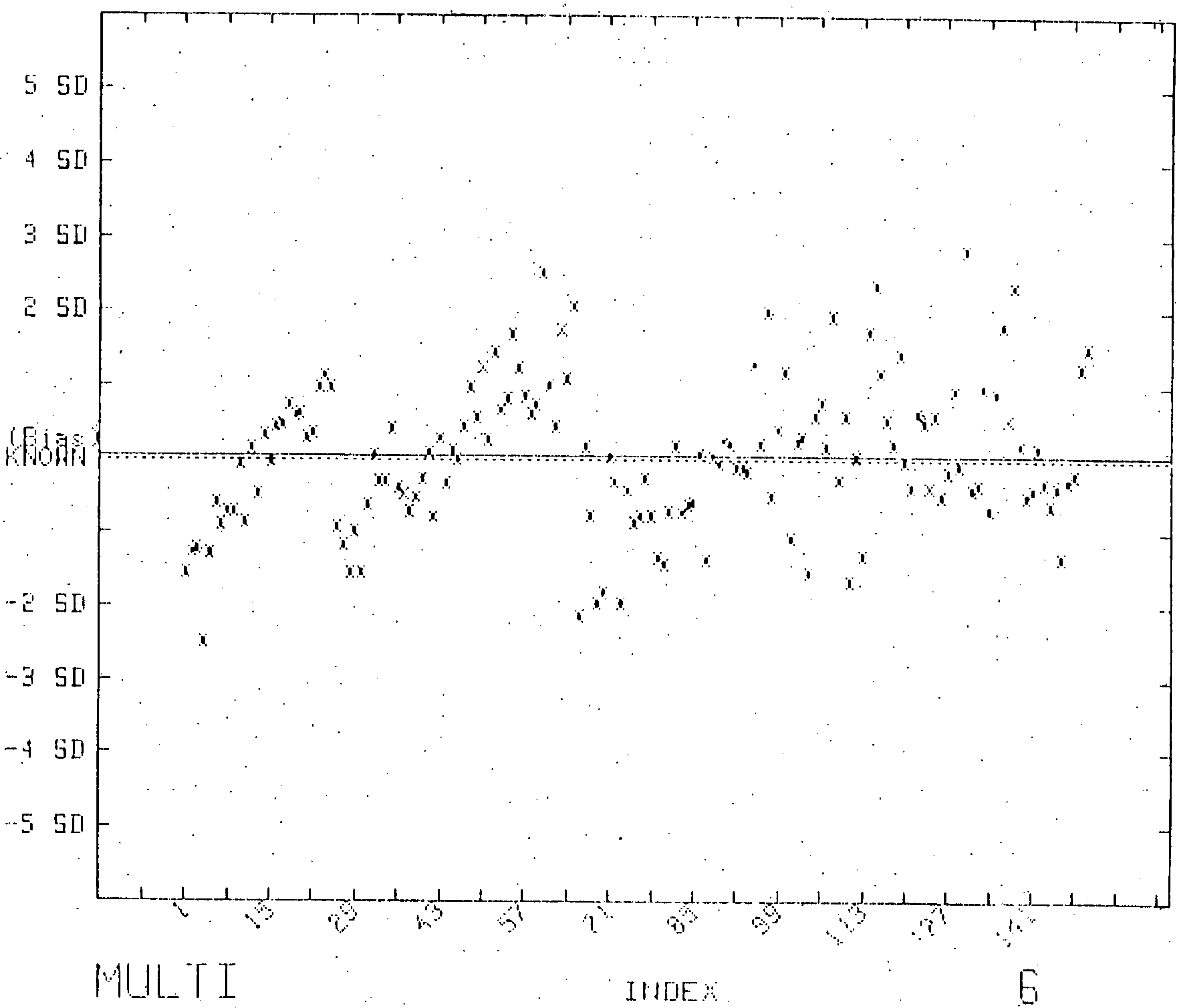
MULTI-LEVEL   KNOWN   5401



MULTI-LEVEL 5401

5401 Zr-AA-1 uG/mL MULTI-LEVEL 05/01/79




5 SD
4 SD
3 SD
2 SD
(Bias)
KNOWN
-2 SD
-3 SD
-4 SD
-5 SD
1
15
29
43
57
71
85
99
113
127
141
MULTI
INDEX
6

MESSAGES: The known and reported values appear to be related by a relative bias.

****

CURVE FORM: ln(Standard deviation) is a quadratic function of the known. Result is a linear function of the known.

****

NORMALITY TEST: No signifcant divergence from the normality assumption.

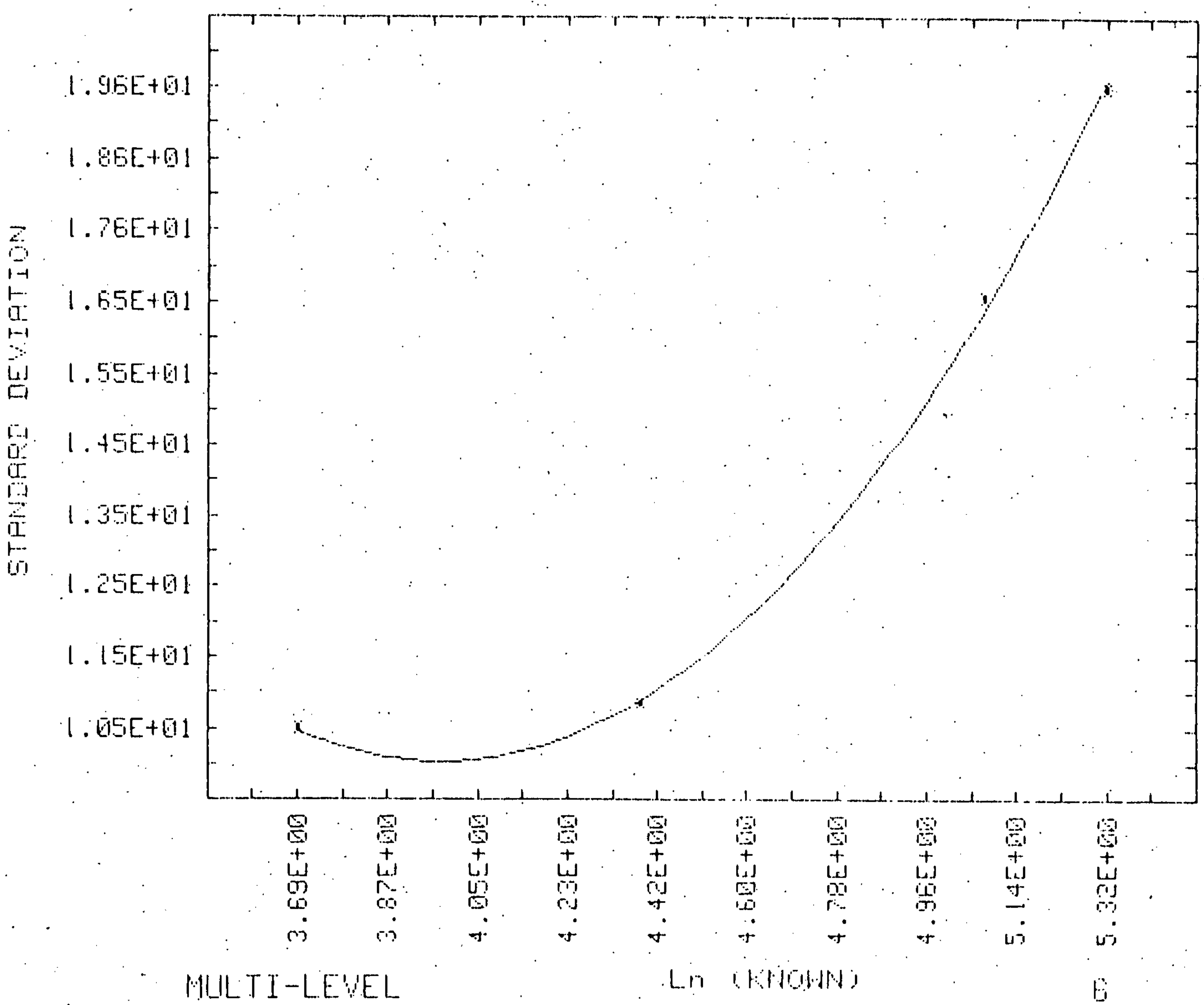
****

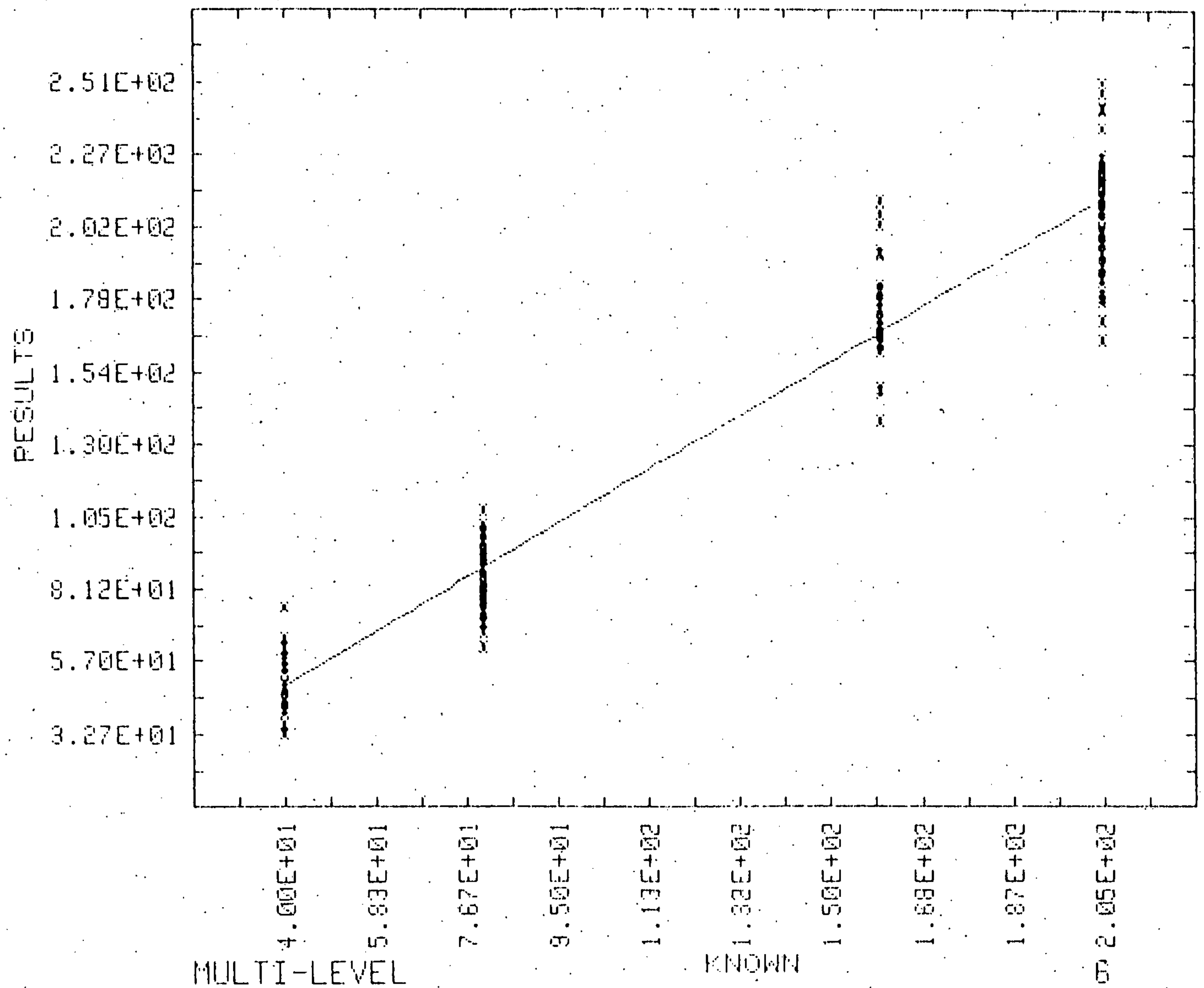
VON NEUMAN TEST: The Von Neuman ratio for autocorrelation is 2.0405456597 based upon 19 D.F.

****

RUNS TEST: The probability of 9 or fewer runs is .29661824436
The probability of 9 or more runs is .84091450345
Hence, the number of runs does <u>not</u> invalidate the assumption of random residuals.

****


STANDARD DEVIATION
1.96E+01
1.86E+01
1.76E+01
1.65E+01
1.55E+01
1.45E+01
1.35E+01
1.25E+01
1.15E+01
1.05E+01
3.69E+00
3.87E+00
4.05E+00
4.23E+00
4.42E+00
4.60E+00
4.78E+00
4.96E+00
5.14E+00
5.32E+00
MULTI-LEVEL
Ln (KNOWN)
6


2.51E+02
2.27E+02
2.02E+02
1.78E+02
1.54E+02
1.30E+02
1.05E+02
8.12E+01
5.70E+01
3.27E+01
RESULTS
4.00E+01
5.83E+01
7.67E+01
9.50E+01
1.13E+02
1.32E+02
1.50E+02
1.68E+02
1.87E+02
2.05E+02
MULTI-LEVEL
KNOWN
6