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K-1292

MASTER

AEC RESEARCH AND DEVELOPMENT REPORT

A SENSITIVE RATIO RECORDER FOR MASS SPECTROMETERS

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OAK RIDGE GASEOUS DIFFUSION PLANT
Operated by
UNION CARBIDE NUCLEAR COMPANY
DIVISION OF UNION CARBIDE CORPORATION
for the Atomic Energy Commission

Acting Under U. S. Government Contract W7405 eng 26

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Printed in USA. Price .50 cents. Available from the

Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

Date of Issue: March 18, 1959 Report Number: K-1292

Subject Category: INSTRUMENTS
TID-4500 (14th Ed.)

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ABSTRACT

A ratio recorder measuring system has been developed for Oak Ridge Gaseous Diffusion Plant isotope ratio mass spectrometers. This measuring system uses many of the components of the galvanometer measuring system which it replaces. Improvements in both sensitivity and precision are achieved. Ratio recorders have been constructed and installed in twelve spectrometers.

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INTRODUCTION

One of the major limitations on the precision with which uranium isotope measurements could previously be made at the Oak Ridge Gaseous Diffusion Plant was the sensitivity and stability of the galvanometer-type measuring circuits (1) with which the mass spectrometers were originally equipped. When one analyzed natural uranium hexafluoride the balance decade resistance setting of these circuits could be determined to only approximately ± 0.1 per cent of itself in one reading, thereby requiring many readings to approach the desired precision. The sensitivity of the system was limited by the random noise which could be tolerated or read from a galvanometer.

A project was undertaken to improve the measuring sensitivity and precision, preferably by means of a recording system in which a higher noise level could be tolerated, since the errors from random fluctuations can usually be minimized by making a visual estimate of the average of a recorder scan (2). It was desired to economize by using as many of the existing spectrometer components in the new measuring circuits as possible.

A sensitive, inexpensive ratio recorder circuit was developed; recorders have now been installed on twelve spectrometers.

SUMMARY AND CONCLUSIONS

Performance records of the ratio recording instruments indicates the following advantages over those equipped with galvanometers:

1. Sensitivity is improved to ± 0.02 per cent for each reading.
2. Significant reductions in the confidence intervals of measured ratios are obtained.
3. A graphic indication of instrument operating condition facilitates trouble-shooting and repairing.
4. A convenient facility (uniform chart speed) for timing each step in the operating sequence is provided.

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(1) Nier, A. O. C., Ney, E. P., and Inghram, M. G., "A Null Method for the Comparison of Two Ion Currents in a Mass Spectrometer", Review of Scientific Instruments, 18, 294, (May 1947).

(2) McKinney, C. R., et al, "Improvements in Mass Spectrometers for the Measurement of Small Differences in Isotope Abundance Ratios", Review of Scientific Instruments, 21, 724, (August 1950).

OPERATION

The ratio recorder shown in figure 1, like the galvanometer circuit, is a null balance system in which the output of the "low-current" amplifier is balanced against a precisely-determined portion of the output of the "high-current" amplifier. The two ion currents to be compared are amplified by two dc. amplifiers; the low-current signal from m/e = 330 is fed directly into the amplifier of a self-balancing recording potentiometer, and the high-current signal from m/e = 333 is fed to the slide wire of the recorder through a precision decade resistor. In operation, the decade resistor is adjusted manually to the value which allows the recorder motor to drive the slide wire contact to the balance point. The recorder pen then indicates the balance point, and the reading from the decades and the recorder is proportional to the ratio of ion currents.

The system is used for relative measurements, in which the ratio reading of a sample is compared to that of a standard to obtain a ratio of mol ratios.

CONVERSION

The ratio recording system is designed to utilize as many of the components of the galvanometer circuit as possible. The high-current amplifier and power supply are those used in the galvanometer circuit; however, revisions to the high-current amplifier panel are necessary to change the output circuit and decades. The low-current amplifier is similar to the high-current, except that it has a zero stabilizing circuit. This amplifier uses, with only minor modifications, the power supply originally used for the FP54 electrometer. Thus the only new components required are the low-current amplifier, preamplifier, and the recorder.

The cost of conversion is therefore relatively low. The total material, labor, and plant expense cost for equipping one instrument, including conversion of a Micromax recorder to a Speedomax, is approximately $1200.

PERFORMANCE

The zero stability of the ratio recorder is indicated by figure 2. Ten small divisions are equal to 1 millivolt; the zero drift shown is approximately 0.1 mv. in 20 minutes. This drift is equivalent to a change in the ratio of 0.02 per cent during the 20 minutes required to make a determination.

Comparisons of past performance characteristics of several instruments with the results obtained on ratio recording spectrometers emphasizes the superiority of ratio recorders.

Several spectrometers analyze pairs of samples at approximately natural uranium concentration, and differing from each other by less than 0.5 per cent of their concentration. For the six months following installation of a dc. amplifier ratio recorder in one of these spectrometers, the internal confidence limits for this spectrometer were ± 0.07 per cent per determination, at the 95 per cent confidence level. These limits correspond to an internal variance only one third that of any of the other instruments without recorders used for the same type of analyses. (The entire precision improvement could not be attributed definitely to the measuring circuit alone, since other minor improvements were also made on the instrument.)

The internal variance of another spectrometer was decreased by a factor of approximately three by revision of the sample manifold and installation of a dc. amplifier ratio recorder.

After a ratio recorder was installed in a third spectrometer, the recorder revealed a drift problem which had not been detected previously. As a result, the analyzer tube was changed and the internal confidence limits for the instrument for a two-month period after that time were ± 0.10 per cent, compared to ± 0.14 per cent for seven months before. The proportion of precision improvement attributable to the elimination of the drift problem was not known; however, the improvement was all attributable to the ratio recorder either directly or indirectly, since the isolation and elimination of the drift trouble were a result of the recorder installation.

ACKNOWLEDGEMENTS

The first model of the ratio recorder was constructed by H. A. Kermicle, who also did much of the development work.

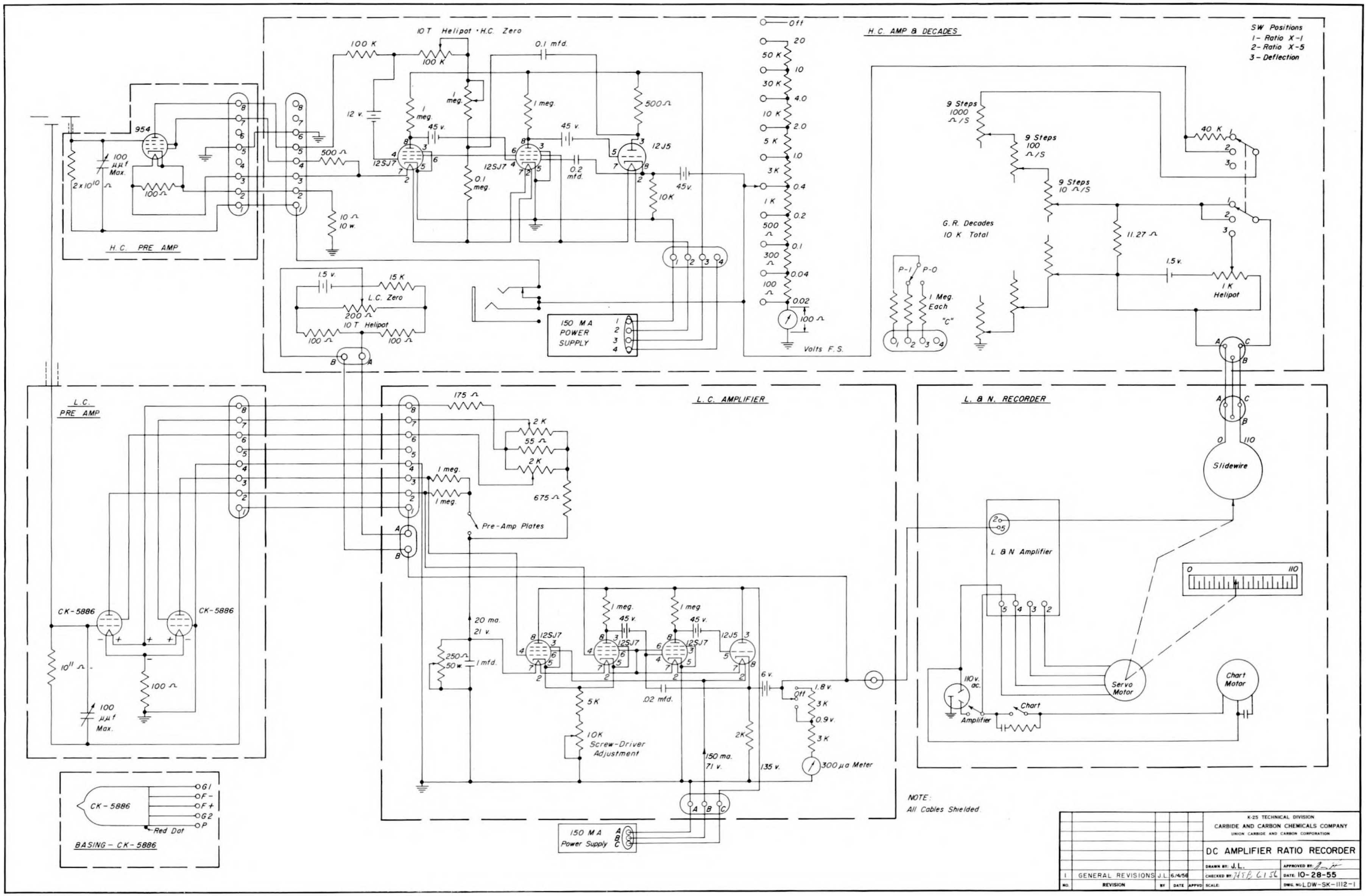

FIGURE 1
D. C. Amplifier Ratio Recorder

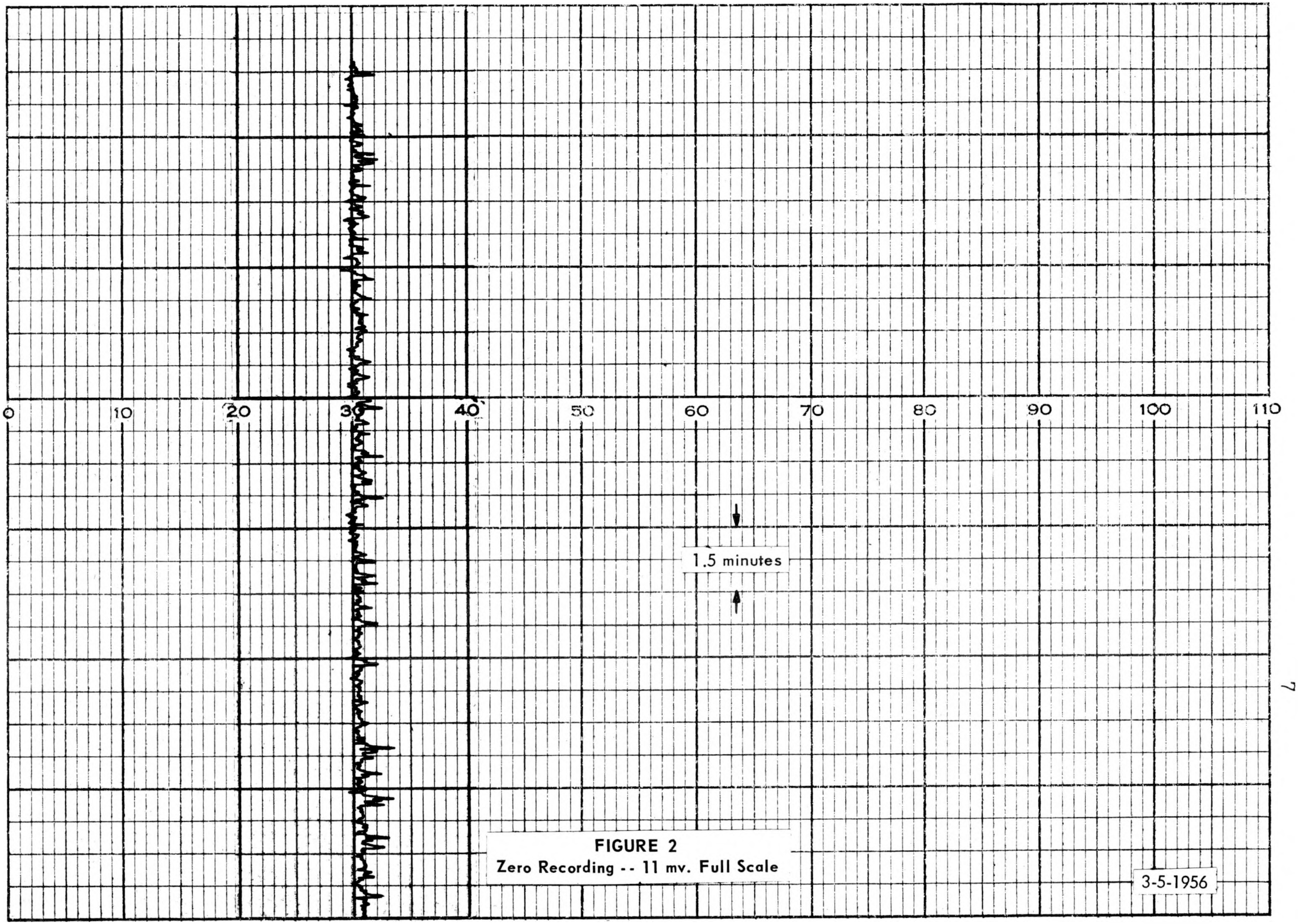

FIGURE 2
Zero Recording -- 11 mv. Full Scale

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