Title

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Authors
Newton, Amos S.
DiGrazia, Herbert X.

Publication Date
1955-05-17

UCRL 2994

UNIVERSITY OF CALIFORNIA

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UCRL-2994
Unclassified Instrumentation

UNIVERSITY OF CALIFORNIA

Radiation Laboratory
Berkeley, California

Contract No. W-7405-eng-48

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Printed for the U. S. Atomic Energy Commission

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Radiation Laboratory
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ABSTRACT

The use of a bismuth wire coil for measuring the magnetic field in the Consolidated Engineering Corporation mass spectrometer is described. The method is based on the work of H. B. Keller (University of California Radiation Laboratory Report UCRL-2249). The resistance of the bismuth coil, which is kept at liquid nitrogen temperature, is calibrated against the field by measuring the mass and voltage of known materials. Assuming a radius of curvature (5 inches) this can be converted to a calibration of resistance _vs_ field in gauss.

The bridge circuit for measuring the resistance and the sensitivity of the measurement (0.1 gauss in the 2000- to 4000-gauss region) is discussed, and the stability of the setup in terms of drift, temperature cycling of the coil, and effect of room temperature, are described.

The use of the instrument for checking the field setting in normal operation, mass identification of peaks of unknown mass, and locating a given mass at a given voltage for appearance potential or voltage discrimination studies is discussed.

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There are definite advantages in the operation of any mass spectrometer to be able to meter the field. It is not necessary to do this in terms of absolute units of field intensity since other factors in the operation of the spectrometer, e.g., the radius of curvature, are not usually known to the precision to which the magnetic field can be measured (e.g., with a proton moment fluxmeter). For most purposes it is sufficient to assume a radius of curvature and calibrate the field on known mass peaks.

The relation between the mass of the ion collected, M, (in mass units per unit charge), the voltage V, and the field B, in gauss, is given in the relation

$$B = k\sqrt{MV}\,, \tag{1}$$

where k for a spectrometer of 5-inch radius is 11.336.[1] Any

1. Consolidated Engineering Corporation, Operation and Maintenance Manual, Model 21-103. Pasadena, California, 1951. Vol. I, p. 11, discusses the derivation of this relation.

device for measuring the field can be internally calibrated with the mass spectrometer itself in terms of MV <u>vs</u> response or in gauss <u>vs</u> response, the units being those defined by equation (1) and not necessarily absolute.

Of the methods available for measuring magnetic fields, by far the simplest and least expensive is that of measuring the resistance of a bismuth wire at a low temperature. Keller[2] has

2. H. B. Keller, University of California Radiation Laboratory Report UCRL-2249 (June 9, 1953).

discussed the development of this method and the use of the method for making precision measurements of the field distribution in model magnets. For Keller's purposes, where the fields were in the region of several kilogauss, it was expedient to operate at -28° C, the boiling point of Freon-12. However, with the Consolidated mass spectrometer where the fields of interest lie between 600 and 4000 gauss it is preferable to operate at liquid nitrogen temperatures when the change of resistance with field is much larger, as shown in Table I.

The bismuth coil is noninductively wound with 5-mil-diameter microcrystalline bismuth wire[3] on a small Teflon spool. The

3. Ductile bismuth wire is available from Fitzpatrick Electric Supply Company, Muskegon, Michigan. The wire comes already coated with Analac insulation.

spool is about 0.5 inch long, 0.25 inch diameter, with the center drilled out. The twisted leads are of No. 30 Formvar-covered copper wire and are connected to the bismuth wire and the spool so the bismuth wire is not under stress. Soldering can be done with either Wood's metal or conducting silver paint, but the Wood's metal has been found preferable for operation at liquid nitrogen temperatures, as the silver paint was found not to thermal cycle well. Some skill is necessary to solder with the Wood's metal to prevent grain growth in the bismuth wire near the junction and subsequent failure of the coil.[2] A pair of compensating leads are also soldered together with Wood's metal and are twisted with the coil leads. The junction is kept near the connections to the coil and the same length of copper wire is immersed in the liquid nitrogen in each case. This compensates for changes in resistance of the copper leads due to changes in the level of the liquid nitrogen. The coils used by the authors have had a zero field resistance of about 36 ohms at room temperature and about 15 ohms at liquid nitrogen temperature.

One experiment was performed to check the temperature stability of a liquid nitrogen trap. A Dewar fitted with a Styrofoam lid was filled with liquid nitrogen, a copper-constantin thermocouple was inserted, and left on the bench for 6 weeks. It was filled by the technicians every 4 hours according to their regular procedures. No care was given the setup beyond that normally given such a trap being serviced by the technicians. Over a 6-week period the temperature of the trap was measured three times daily. No variations greater than the sensitivity of the potentiometer used (about 0.1° C) were noted. Therefore it was concluded that the temperature stability of the nitrogen trap was adequate.

The side-arm Dewar for placing the coil in the field was constructed as shown in Fig. 1. The dimensions are not critical, but those given place the coil inside the metal block at the collector end of the analyzer tube. This Dewar was supported from the magnet in a stable position as shown in the photograph, Fig. 2.

The bismuth coil was wired as one arm of a Wheatstone bridge, as shown in the schematic diagram Fig. 3. The compensating leads and a resistance box were in the opposite arm. We have used a six-decade 10-K Leeds and Northrup resistance box in which only five of the decades were used. Different range boxes can be used by changing the 10 K resistor at the top of the bridge to a value to match the resistance of the resistance box used.

The 1 K resistor in the battery circuit limits the current through the coil to about 5 ma. Keller[2] recommends that not over 4 ma be used through coils made from 5-mil wire for operation at the boiling point of Freon-12. No difficulty has been experienced in using the higher current in several months' operation at liquid nitrogen temperatures. The 5-ma current was arbitrarily arrived at by trial and error at a time when a galvanometer was used as a null indicator. Using the Brown-Electronik Null Indicator instead of the galvanometer, plenty of sensitivity is available, so the 5-ma current could be reduced to 4 ma or less if trouble was encountered because of excess current.

The actual wiring diagram is shown in Fig. 4. The bismuth coil, compensating leads, battery leads, and ground terminals are connected to a terminal strip in a box mounted on the magnet as shown in Fig. 2. A four-wire shielded cable leads from this terminal strip, through the channel housing the amplifier cables to the control cabinet, and out through a special port above the magnet regulator to another terminal box. Leads are taken from here to the null indicator and the resistance box. All leads and terminal strips are grounded to minimize stray-current pickup. The magnet-measuring controls are situated just above the magnet regulator where operation is convenient. The setup is shown in the photograph in Fig. 5.

It was found necessary to change the magnet-control potentiometers to fully utilize the sensitivity of the bismuth coil, as the magnet "fine" control was not adequate and the stability of both fine and coarse controls was found to be lacking. Therefore, the fine control was eliminated, i.e., bypassed, and the coarse control was replaced by a 10-turn 50 K Helipot which has proven adequately sensitive and stable.

Operation

The calibration curve of a typical bismuth coil is shown in Fig. 6. This is a linear plot of resistance of the coil against the magnetic field as calculated from equation (1). The abscissa scale at the top of the plot is that of mass units times voltage. Other methods of plotting, such as resistance vs log(MV), are useful in practice. For actual operation the plot shown in Fig. 6 is on too small a scale to utilize the sensitivity of the method. In this figure the magnetic field at the usual magnet settings A, B, and C, corresponding to the three mass marker calibrations for the Consolidated instrument, are shown. Setting S is a mass marker calibration not given by Consolidated where the marker steps occur at masses 7, 9, 12, 14, 16, 19...etc; in steps of 2, 2, 3. This has been useful in looking at boron compounds and some multiply charged ions and is brought in at about 0.4 ampere magnet current on our machine. The hydrogen point is the field at which hydrogen is scanned when operating normally at field A but switched to the low magnet setting.

Measurements of the resistance are made to 0.001 ohm. There is sufficient sensitivity in the Brown Null Indicator to use another decade but the noise level in the system increases rapidly so it has not proven feasible to use the instrument beyond the 0.001-ohm range. In the region in which operation is normally conducted, 2000 to 4000 gauss, this measures the field to approximately 0.1 gauss. In Table I are shown some measurements on peaks at various mass X voltage settings to illustrate the sensitivity of the method. Since the peaks themselves are of the order of 0.030 ohm wide the center of each peak is located by setting the voltage, then changing the field to find the front and back lips of the peak. The mean is the resistance at the peak of mass M. It is seen from Table I that even in the most extreme cases of scanning at very low voltages, the measurements are sensitive to the order of 0.01 mass unit.

The stability of the instrument is shown in Table II. After the initial installation the resistance was very stable for at least 10 days or perhaps longer; measurements were not recorded in the period from 10 to 34 days. Then the resistance increased progressively until the apparatus was dismantled for moving the spectrometer, 47 days after the installation. The cause of this increase after about a month of operation is not known. One explanation (as yet untested) is that it is due to a change in bismuth coil temperature caused by the accumulation of ice crystals in the side arm. This would result in poor temperature control because of the limited access of liquid nitrogen to the coil. The liquid nitrogen trap was not warmed during the 47 days' operation.

Advantages of Magnetic Field Measurements

The advantages of the measurement of the field in a magnet where the magnet regulator controls the voltage and not the magnet current, and where the magnet exhibits considerable hysteresis, are apparent if the field is changed frequently. One can reset the magnet without introducing a standard material for calibration.

When appearance-potential studies or voltage-discrimination studies on a given peak are being made, these measurements are greatly facilitated by setting the field and the voltage to bring in the desired peak without searching. It also eliminates any uncertainty as to which peak is being studied.

The field calibration is also useful in regions where the mass marker does not give a good indication of the mass identification of peaks in a material being run for the first time. If there is any doubt, the measurement of the field and voltage at which the peak appears accurately defines the mass of the peak.

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

Table I

Effect of Temperature on the Change of

Resistance of Bismuth with Magnetic Field

Field Kilogauss	Relative Resistance at Temperature Cited			
	-192° C	-37° C	0° C	18° C
0	0.40	0.88	1.00	1.08
2	1.16	0.96	1.08	1.11
4	2.32	1.10	1.18	1.21
6	4.00	1.29	1.30	1.32
8	5.90	1.50	1.43	1.42

Data from Smithsonian Tables and Keller UCRL-2249[2].

Table II

Resistance Changes per Mass Unit under

Various Operating Conditions

Mass (M)	Voltage	ΔR/Mass Unit (M $\rightarrow$ M+1)	Field in gauss (M)
58	780	0.211	2422.6
150	320	0.081	2483.7
150	600	0.122	3400.9
150	1000	0.187	4390.6

Table III

Stability of Resistance Measurements

Days after Installation	Resistance (ohms) for m/e = 58 780 volts	Δ R (+)	Δ Mass Units at Mass 58
0	33.692	0.000	0.000
3	33.696	0.004	0.019
4	33.697	0.005	0.023
5	33.696	0.004	0.019
6	33.696	0.004	0.019
10	33.696	0.004	0.019
34	33.717	0.025	0.118
41	33.729	0.037	0.175
45	33.750	0.058	0.275
47	33.749	0.057	0.270

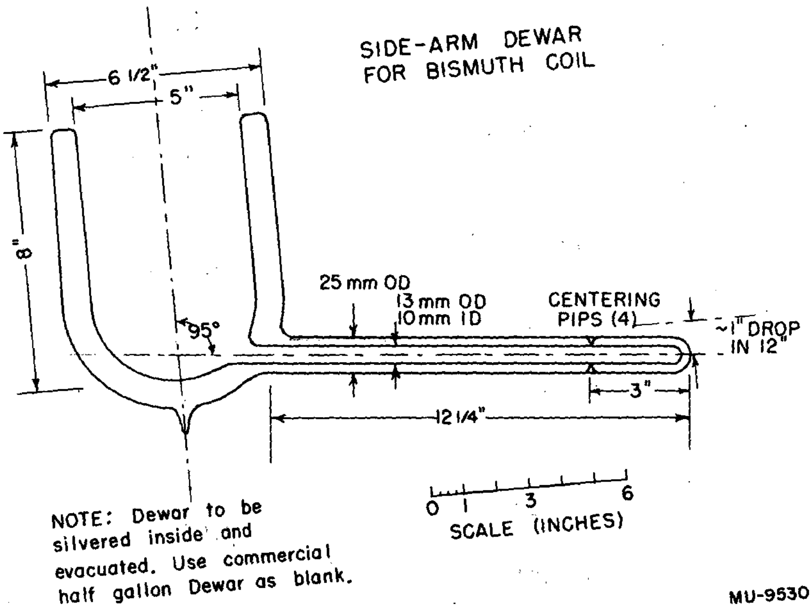
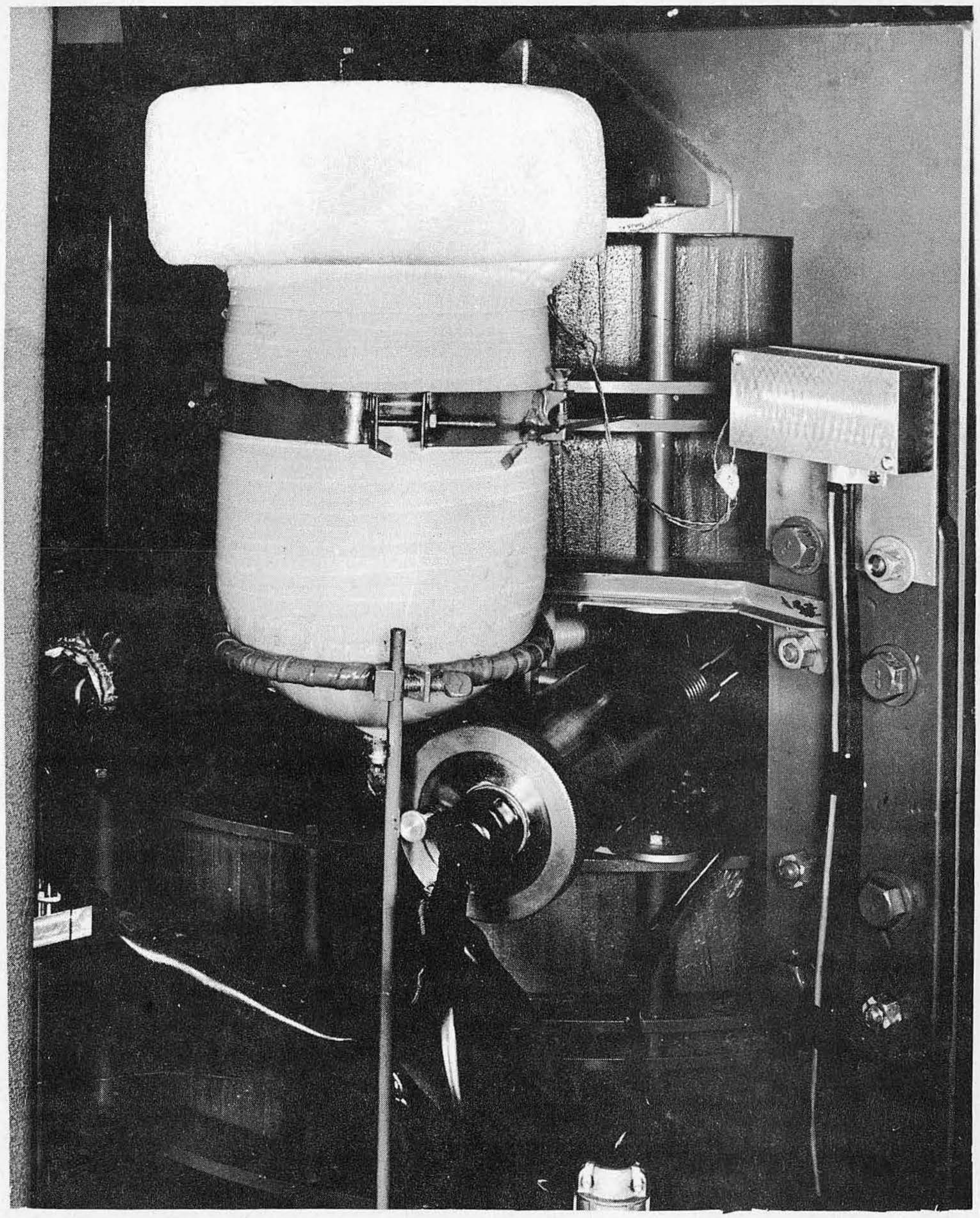


Fig. 1. Side-arm Dewar for bismuth coil.



ZN-1298

Fig 2. Photograph showing side-arm Dewar and junction box mounted on magnet.

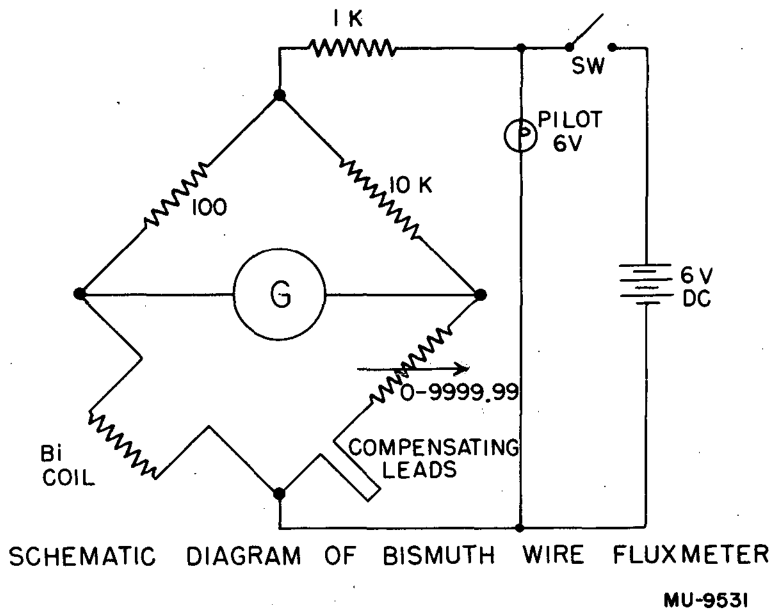


Fig. 3. Schematic diagram of bismuth wire flux-meter.

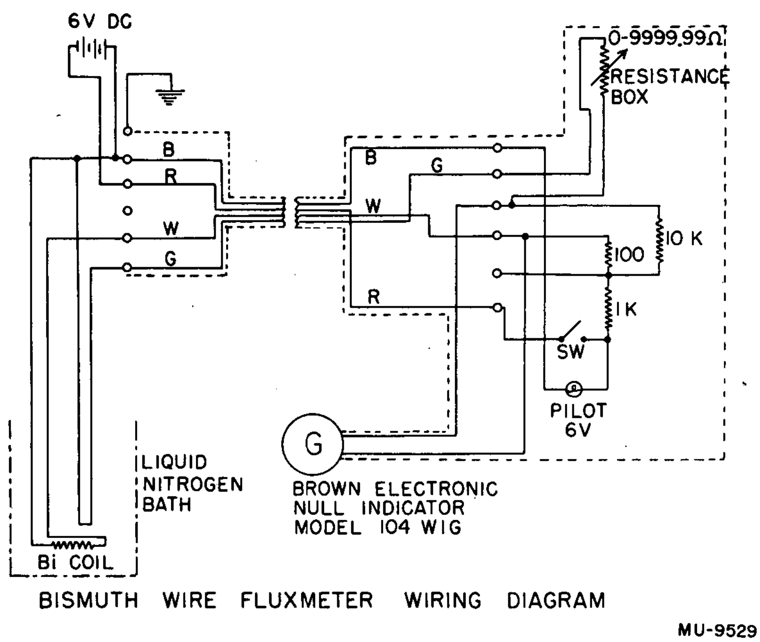


Fig. 4. Bismuth wire fluxmeter wiring diagram.

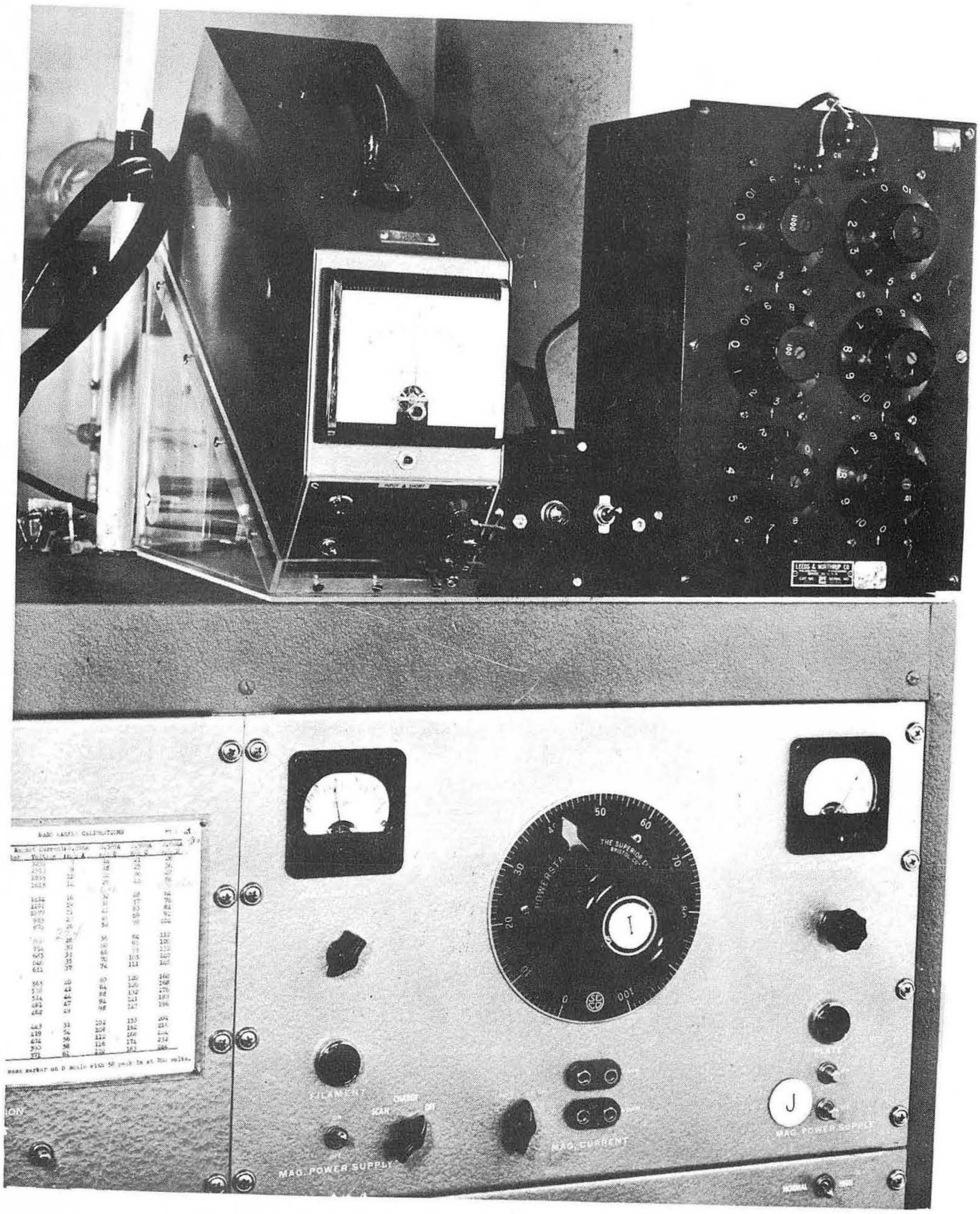


Fig. 5. Photograph showing mounting of Brown Electronik Null Indicator and resistance box over the magnet regulator. ZN-1299

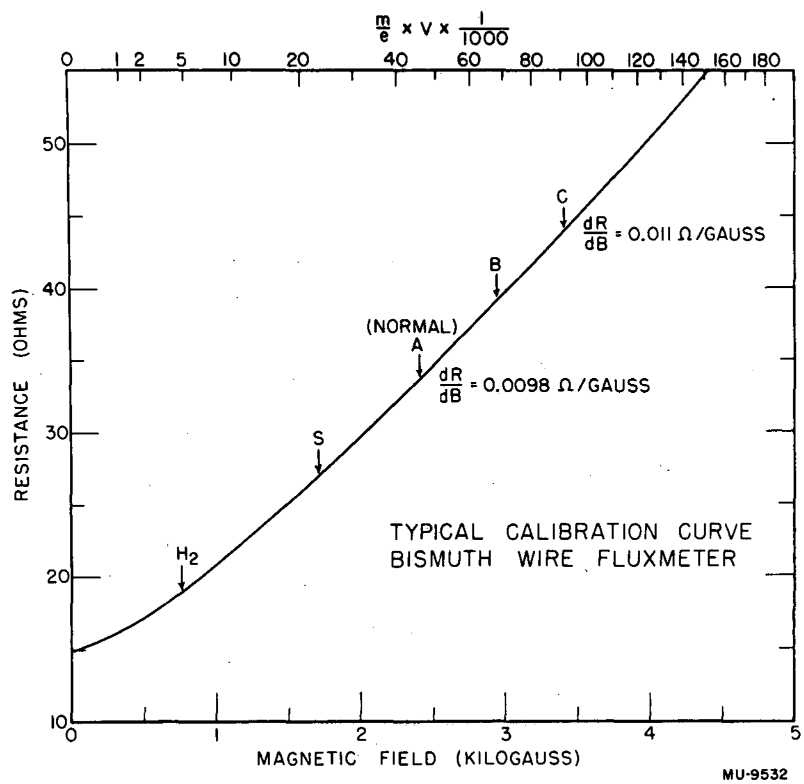


Fig. 6. Typical calibration curve of bismuth wire fluxmeter.