



TENTH QUARTERLY PROGRESS REPORT

FERRITE DEVELOPMENT

CONTRACT NO.: DA-36-039-sc-5449

**GENERAL CERAMICS & STEATITE CORP.
KEASBEY, N.J.**

TENTH QUARTERLY PROGRESS REPORT

CONTRACT FOR SERVICES, FACILITIES, AND MATERIALS
REQUIRED TO INVESTIGATE AND STUDY MAGNETIC FERRITES

PERIOD COVERED: From July 1, 1953 to September 30, 1953

CONTRACT NO.: DA 36-039-sc-5449

DEPT. OF ARMY PROJECT NO.: 3-93-00-503, 3-99-04-042

SIGNAL CORPS PROJECT NO.: 32-2005-D, 29-194B

PLACED BY: Laboratory Office, Signal Corps Procurement Agency
Contracting Division, Fort Monmouth, New Jersey

CONTRACTORS: General Ceramics and Steatite Corporation
Keasbey, New Jersey

OBJECT: To develop ferromagnetic ferrites of different
permeabilities, and low magnetic losses, and to
investigate conditions favorable to their produc-
tion.

REPORTED BY: Dr. E. Albers-Schoenberg, Supervisor
Ephraim Gelbard, Project Engineer

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ABSTRACTS

PART I

- A) The investigation of temperature stable bodies of the lower permeability range (approximately 100-200) is continued. Two bodies of the nickel-zinc group are described and their properties discussed.
- B) The study of bodies of the system $\text{MgO-NiO-ZnO-CuO-Fe}_2\text{O}_3$ has still not come to an end. Out of a great variety of measurements, two materials have been selected to show the effect of nickel-oxide content upon the magnetic properties. All significant measurements are shown.
- C) A preliminary remark hints at the possibility of combining temperature stable bodies and high Q bodies into one and the same material.
- D) The field strength at the conditions of the μ_c test has been measured as a function of initial permeability. It is found that all measurements heretofore performed were at a field strength of less than 0.45 milli-oersted which validates the measurement technique.

PART II

- A) The uniformity and reproducibility test of MF-254 "G" body has been completed. The results of the G-2 preparation resemble those of the first part. Some conclusions are drawn and some general rules established.

PART I

Body Development and Measurements made during the Tenth Quarter
of the Contract

DA Project No.: 3-93-00-503 - SC Project No.: 32-2005-D

A) Temperature Stable Bodies of the Lower Permeability Range

The following investigation is, in some respect, a continuation of the previous work on oscillator slugs. Now, however, the objective of our experiments has been changed and has taken on a more general feature.

Up to the present time, it is known that temperature stability may be achieved in two ways:

1) a body of relatively high permeability in the dense-fired state can be brought down in temperature coefficient (simultaneously decreasing μ) by underfiring. The porous material represents a diluted state of magnetic matter with lower permeability.

2) a dense or almost dense fired body containing a small percentage of "impurities" or, in other words, of non-magnetic constituents, may also exhibit a somewhat reduced permeability in conjunction with a flattened out temperature curve of μ_0 . An example of this type of material is the MF-90 "B" material.

In both cases, of course, the basic composition has to be appropriate inasmuch as a moderate temperature coefficient of μ_0 is required right from the beginning. Nickel-zinc ferrites of relatively high nickel and relatively low zinc content lend themselves for the first of the two aforesaid types of material while, as the example of the MF-90-B shows, mixed 4 or 5 component bodies comply with the requirements of the second group.

Actually, both situations are special cases of the primary objective: i.e., to reduce the temperature coefficient by reducing the magnetic efficiency. Table XLIX summarizes the properties at several firings of two new materials for temperature stability.

The table explains the feature of the "stability through porosity" method although body MF-1832-C is not yet to be taken as the conclusion of the investigation. The initial permeability of 370 for the dense-fired body is reduced to about 175 in the underfired state. The temperature curves (toroidal measurement) are illustrated by Graph 167. Up to about 75°C temperature stability is very good. Between 75 and 125°C the temperature coefficient gradually reaches +0.03%/°C. Body MF-1829-C shows the same general principle, but in this case a

PART I

A) - Continued

relatively favorable temperature curve is associated with a water absorption of 5-6%, indicating a too soft structure of the material. However, it may be possible to increase the density of this material while retaining good stability.

As regards the behaviour at various frequencies 1829 maintains its initial permeability and Q-factor reasonably well up to 5 Mcs. μ_0 remains quite stable up to 15 Mcs. This is in accordance with previous experiences that bodies of the system $\text{NiO-ZnO-Fe}_2\text{O}_3$, when in an underfired state, are useful up to frequencies of the $10^6 - 10^8$ range.

The next report will supplement these findings with further work on refinement of the second class of temperature stable bodies (dense MF-90-"B" type).

B) Bodies of the System $\text{MgO-NiO-ZnO-CuO-Fe}_2\text{O}_3$

The Ninth Quarterly Progress Report, April/June 1953 contains a brief statement on bodies of the above-named system where MgO , NiO and CuO are present in about the same weight percentage (4-5%).

The body referred to was MF 1459. In continuation of this investigation, several more variations have been made with the objective of determining the changes in magnetic properties which occur when small changes of composition are made. Each compositional variation was subjected to calcine temperature variations, but in order to simplify any conclusions that may be drawn, a series of compositions which used the same calcine ($\text{MgO-ZnO-Fe}_2\text{O}_3$) fired at a temperature of 2260°F is reported here. The firing temperature of the test samples was 2380°F . Table L shows the chemical compositions of two ferrites as well as a summary of the important magnetic properties. All measurements were performed upon C-shaped cores having about a 2 sq. cm. cross section and a mean magnetic path length of about 7 cm. The R. F. measurements were made at a frequency of 0.55 Mc. Each magnetic circuit had two air gaps, but the adjacent surfaces of the two C-cores were well ground to minimize the effects of the gaps.

If the variations in properties are related to the nickel oxide content of the body (which has proven through experience to be one of the most important parameters upon the efficiency of bodies of low nickel and reasonable zinc content), then it is to be expected that the magnetic properties improve as the nickel oxide content increases.

Graph 168 shows the μ_0 vs. temperature behaviour for these two materials on the same plot. The overall temperature behaviour of μ_0 is better for MF 1480 than for MF 1459. Although the μ_0 for MF 1480 is shown lower than that for MF 1459 (which has less nickel oxide) toroidal ring measurements have shown that μ_0 is actually higher. These results are also supported by the other magnetic measurements which are shown in Table L. In overall efficiency, MF 1480 (with the higher nickel-oxide content) is equal or better than MF 1459.

It should be pointed out that MF 1480 is a reproduction of MF 1429 (reported upon in the Sixth Quarterly Progress Report July/Sept. 1952), but using raw materials of a lesser degree of purity. In respect to this variation, MF 1480 compares favorably to MF 1429.

Photos of hysteresis loops (60 cps) of a low nickel oxide content body (MF 1102 - "H-1"), a medium nickel oxide content body (MF 1459), and a high nickel oxide content body (MF 419 "H") are shown in Graph 169. As far as can be seen from the shape of

B) Continued

the curve, these materials are quite similar. The actual magnetic properties, however, vary more than the 60 cps hysteresis loops indicate. Graph 170 shows the μ_0 vs. temperature curves for these same three ferrites. It is interesting to note that the ferrite with the medium nickel oxide content has the best temperature behaviour, which is explained by a difference in the ZnO content.

C) Preliminary Remark

Recent experiments have given evidence that bodies basically belonging to the system $\text{NiO-ZnO-Fe}_2\text{O}_3$ can be made that combine the following properties:

- a) μ_0 around 110
- b) Q-factor, in the frequency range of 500 Kc to 2 Mc, of several hundred
- c) a temperature coefficient of approximately $+0.1\%/^{\circ}\text{C}$ for the toroidal core

Further details will be given in later reports.

D) Measurement of Field Strength (H) at the Conditions of the μ_0 (1 Mc) Test

The purpose of this investigation was to again evaluate the test procedure for toroidal measurements of μ_0 and Q (at 1 Mc) using the General Radio 916-A Impedance Bridge in conjunction with a suitable signal generator and null detector. (See First Quarterly Progress Report April/June 1949). The need for this re-evaluation of the measuring techniques arises from the fact that it is known that many ferrites (especially those of low coercive force) have a rather narrow region where the μ_0 is independent of the magnetizing field strength. One objective of this work was to try to plot a curve of H_{ac} vs. μ_0 so that for any particular measurement heretofore made or for future measurements, the exact conditions of magnetizing field can be found for a ferrite of any measured initial permeability.

Since most of the ferrite developmental work is carried out on the F 108 ring (approximate dimensions: O. D. = 4.7 cm, I. D. = 3.6 cm, H = 0.6 cm), the subsequent measurements were performed on this size toroid. The actual toroidal dimensions varied from material to material by as much as $\pm 15\%$ (due to shrinkage variations).

Measurements of 50 toroids were made; about 5 rings each of all the commercially available "Ferramic" types so as to get the complete range of possible μ_0 values. The following measurement (1 Mc) were made on all the rings:

- 1) X_L - inductive component of the toroidal impedance
- 2) R - resistive or loss component of the toroidal impedance
- 3) E peak-peak-voltage across the toroidal impedance
- 4) The dimensions of the ring

Using the data of the preceding measurements, the field strength can be computed. The following formulas show the relationships:

- 1) $L = \frac{X_L}{2\pi f}$ where L = toroidal inductance in microhenrys
 X_L = bridge reactance reading divided by frequency in megacycles
f = frequency in megacycles

D) Continued

$$2) L_o = 0.0046 N^2 h \log(10) \frac{OD}{ID}$$

where L_o = toroidal inductance in microhenrys (air core)

h, OD, ID = toroidal dimensions in cm

N = number of turns

$$3) \mu_o = \frac{L}{L_o}$$

where μ_o = initial permeability

(For a complete development of the validity of these above expressions, see First Quarterly Progress Report April/June 1949)

$$4) Q = \frac{X_L}{R}$$

where Q = Q of toroidal inductance

R = bridge resistance reading (ohms)

Since the voltage across the toroidal inductance contains a resistive and reactive component, it is sometimes necessary to solve for these components in order to accurately find the field strength.

$$5) \phi = \tan^{-1} \left(\frac{R}{X_L} \right) = \tan^{-1} \left(\frac{1}{Q} \right)$$

where ϕ = loss angle
(also $\tan \phi = \frac{1}{Q}$)

$$6) \theta = \tan^{-1} \left(\frac{X_L}{R} \right) = \tan^{-1} (Q)$$

where θ = Phase angle

$$7) E_{\text{reactive r.m.s.}} = \frac{E_{\text{peak to peak}}}{\sqrt{2}} \times (\sin \theta)$$

where $E_{\text{reactive r.m.s.}}$ = r.m.s. active component of the total voltage appearing across the inductance

D) Continued

$$8) B_{max} = \frac{E_{\text{reactive}}}{4.44 (f) (N) (A) (10^{-2})} \quad \text{r.m.s.}$$

where B_{max} = maximum value of
sinusoidal flux density
in core (gauss)

A = cross sectional area of the
core in square cm.

f = frequency in Mcs.

$$9) H_{max} = \frac{B_{max}}{\mu_0}$$

where H_{max} = maximum value of the
sinusoidal field
strength in core
(oersteds)

It can be seen that when the Q exceeds 10, the value of $\sin \phi$ in (7) very nearly equals 1 so the reactive and resultant components are approximately equal.

The results of the measurements and calculations of the 50 rings shows that for μ_0 values up to 1000 (at least) the magnetizing field strength was always less than 0.45 millioersteds. Since this situation existed, no curve need be plotted for H_{ac} vs. μ_0 . During the course of the measurements, it was also found that a reduction of the bridge input voltage (nominally 1 volt at 1 Mc) had no effect upon the measured μ_0 values. This indicates that all the μ_0 measurements are valid and within the range where the initial permeability is independent of field strength.

PART II

Body Development and Measurements made during the Third Quarter
of the Contract

DA Project No.: 3-99-04-042 - SC Project No.: 29-194-B

A) Investigation of Reproducibility and Uniformity of Ferrite Materials

From the second batch of G-body (preparation G-2), toroids have been pressed fired and measured, and the results are communicated below.

Again three different grain sizes were available:

- a) all through 20 mesh
- b) through 20 mesh and on 80 mesh
- c) all through 80 mesh

From these fractions, the third (all fine) has been set aside. (Very fine powders, because of their high air content and difficulty in pressing, are usually discarded in favor of heavier grain size materials.)

Again three different pressures have been applied: 5 tons, 10 tons and 15 tons corresponding to 1040, 2080 and 3120 psi. The number of cores in each series, however, has been increased to 50. From these a small number has been used for preliminary firing tests so that finally a total number of 263 cores has been subjected to measurement and evaluated. The firing has been done in a small tunnel kiln of 8-3/4 feet length (the same as for the G-1 cores). The peak temperature was 2360°F. The appearance of the cores is very good. They are dense and shiny and have no deficiencies as to shape and surface. The evaluation of the μ_0 measurements results in a distribution curve Graph 171 similar to that for the G-1 series.

Table LI gives a detailed account for each of these six distribution curves of μ_0 . The table indicates that, for any particular pressure the dust-free granulation yields the better results. It also indicates that for any particular particle distribution, the higher pressing pressure gives better uniformity.

There are small differences between the percentage figures of the G-1 and G-2 experiment. It is believed that these can be accounted for by the difference in the number of cores subjected to each of the two test series. (13 to 15 for G-1; over 40 for G-2). The reasoning is the following:

PART II - A) Continued

- a) the variation through firing will be very small for so small a batch
- b) on the other hand, one single core deviating from the majority disturbs unduly the overall picture and percentage figures.

Surely the greater numbers of tested cores in the case of G-2 are more favorable from a statistical point of view, so that the G-2 experiment comes closer to practical working conditions.

With respect to absolute magnitudes of μ_0 , the G-2 mix is clearly superior. In the measurement of the G-2 rings, the ambient temperature was closely checked and corrections made ($+1.3\%/^{\circ}\text{C}$) when the temperature varied more than one-half degree centigrade from the 27°C norm. For the G-2 series, the average absolute magnitude of μ_0 was about 10% higher than that of the G-1 test. The Q values of all the rings of the G-2 series were within about $\pm 5\%$ and showed no discernable trends as a function of either pressure or particle size. Since the G-1 test showed that very good dimensional uniformity could be achieved by controlling particle size and pressing pressure, no dimensional evaluation has been made for the G-2 test.

The conclusions to be drawn from the whole uniformity experiment (G-1 and G-2) may be condensed to the following statements:

- a) A medium grain size (relatively large particles in mixture with a variety of smaller ones but excluding the very finest) is most suitable and offers optimum pressing conditions.
- b) The pressure should be high. A high die pressure narrow the magnetic tolerances.
- c) It is possible to keep almost 100% of the production within $\pm 5\%$ tolerance, (μ_0 and Q) and a tolerance of $\pm 2-1/2\%$ still gives a very satisfactory yield.
- d) The location of the peak of the distribution curve depends on the method of preparation and the firing temperature. Having once established the procedure reproducibility will not be in doubt.
- e) Dimensional uniformity can be good if the particle sizes and pressing pressures are held constant.

PART III

RESEARCH PLANNED FOR NEXT QUARTER*

- 1) The investigation of temperature-stable bodies of the lower permeability range (approximately 100-200) will be carried on. Bodies of the B-type, although somewhat lower in permeability (around 80), are also still under consideration (they offer the advantage of lower porosity).
- 2) The investigation of bodies of the system $\text{MgO-NiO-ZnO-CuO-Fe}_2\text{O}_3$ will presumably come to its final conclusion.
- 3) High-Q temperature-stable bodies will be included in conjunction with (1) above.
- 4) The uniformity and reproducibility test will be continued. Next body to be studied will be M. F. 141-"I". Reversible permeability measurements will be started.

* Nitrogen firing of manganese-zinc bodies, as was planned in the last Quarterly Report, has been delayed because of slow delivery of several items necessary for improved firing facilities.

MANHOURS SPENT ON CONTRACT FOR THE PERIOD OF

July 1, 1953 to September 30, 1953

NAME	TITLE	HOURS
Dr. E. Albers-Schoenberg	Supervisor	116
Ephraim Goldbard	Project Engineer	168
Joseph Katz	Ceramist Engineer	4
Samuel Ortepico	Ceramist Engineer	136
Melvin Eisenberg	Electrical Engineer	20
James Callahan	Ceramist Engineer	455
Mary Holton	Meas. Technician	314
Ruth Wainwright	Meas. Technician	298
Agnes Arias	Ceramic Technician	120
Elizabeth Wetzel	Ceramic Technician	7
Dr. K. Wetzel	Ceramist Engineer	91
John Kurts	Technician	86
William Schirger	Technician	40
Peter Coverza	Technician	41
Anthony Koseski	Technician	105
Charles Purzas	Technician	95
R. Reseter	Technician	11
Sigmund Golion	Technician	160

TABLE XLIX

PROPERTY CHART OF TWO MATERIALS FOR TEMPERATURE STABILITY
OF INITIAL PERMEABILITY

BODY	OXIDE SYSTEM	FIRING TEMPERATURE OF 2380°F				FIRING TEMPERATURE OF 2180°F				FIRING TEMPERATURE OF 2160°F			
		μ_c (lMc)	T.C. (μ_o) %/°C	Water Absorp.	Q (lMc)	μ_o (lMc)	T.C. (μ_o) %/°C	Water Absorp.	Q (lMc)	μ_o (lMc)	T.C. (μ_o) %/°C	Water Absorp.	Q (lMc)
MF 1829-C	NiO-ZnO-Fe ₂ O ₃	500	Un- stable	Dense	13	-	-	-	-	120- 140	<4.03 (25- 125°C)	5-6	75- 80
MF 1832-C	NiO-ZnO-Fe ₂ O ₃ (less NiO than 1829)	370	Un- stable	Dense	36	170-180	<1.02 (25-100°C)	2.5 - 2.8	63	-	-	-	-

TABLE I

COMPOSITION AND MAGNETIC PROPERTIES* (at 25°C) OF A SERIES OF TWO PHASES
OF THE SYSTEM $\text{MgO-NiO-CuO-ZnO-Fe}_2\text{O}_3$

Body Number	COMPOSITION								MAGNETIC PROPERTIES					
	Weight Percent				Mol Percent				μ_{000}	Q^{**}	μ_{max}	B_S^{***}	H_c	
	NiO	MgO	ZnO	Fe_2O_3	NiO	MgO	CuO	ZnO						Fe_2O_3
MF 1459	4.59	4.08	16.30	70.40	7.08	11.83	6.72	23.20	51.16	780	7.7	2400	2860	0.19
MF 1480	6.62	3.06	16.30	69.70	10.40	8.88	6.31	23.39	50.99	585	9.9	3170	3220	0.19

* All measurements performed on well ground C-shaped cores.

** Measured at 0.55 Mc

*** At $H_c = 27$ Oersted

TABLE LI

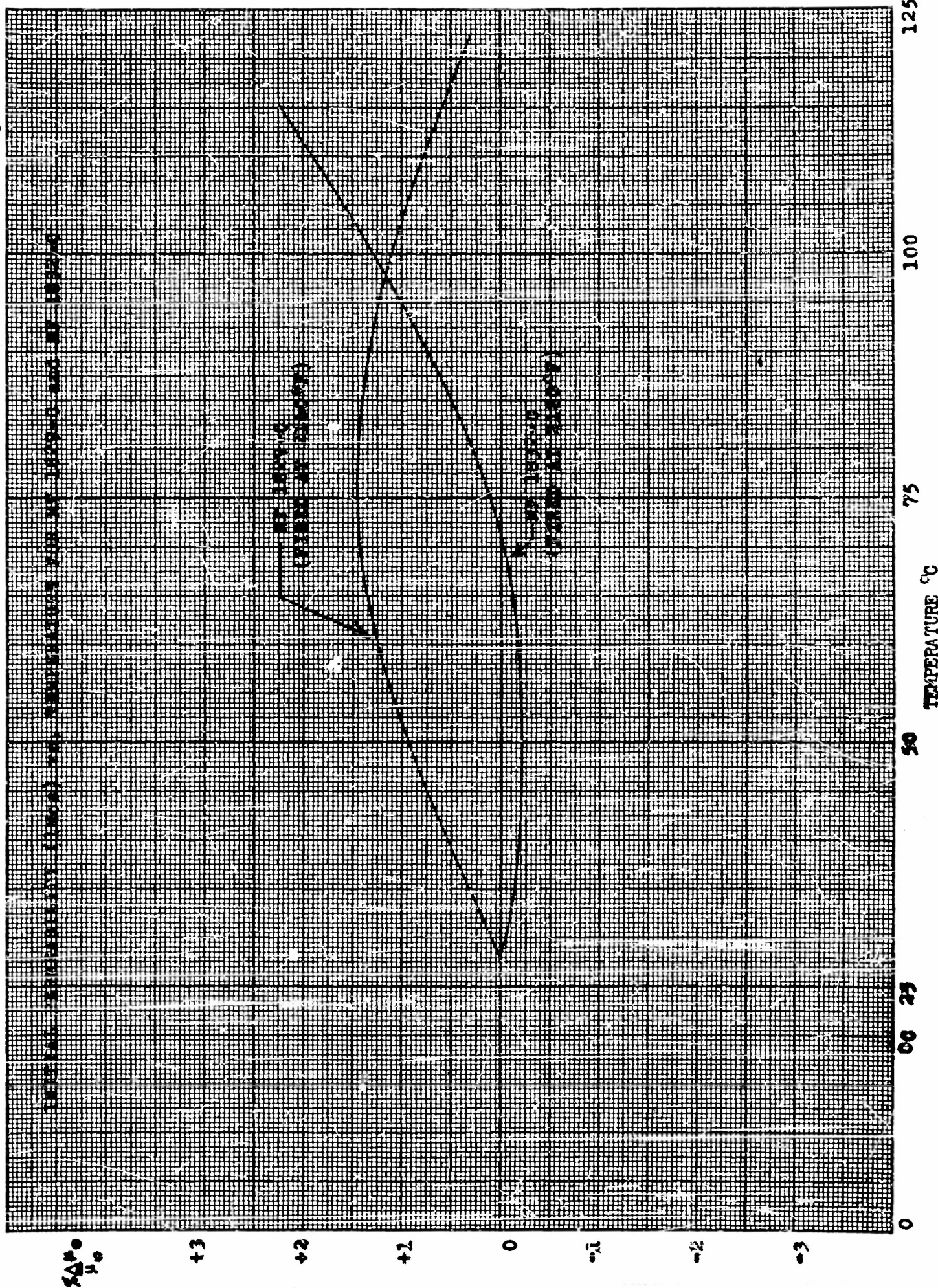
DISTRIBUTION OF μ_0 (1MG) MF 254-"G" AT 27°C FOR THREE DIFFERENT PRESSING PRESSURES AND TWO DIFFERENT PARTICLE SIZES (DIE F-109) PREPARATION G-2 (PERCENTAGES OF CORES WITHIN $\pm 2\text{-}1/2\%$ and $\pm 5\%$ FOR μ_0 (USING THE PEAK OF THE DISTRIBUTION CURVES AS THE CENTER VALUE)

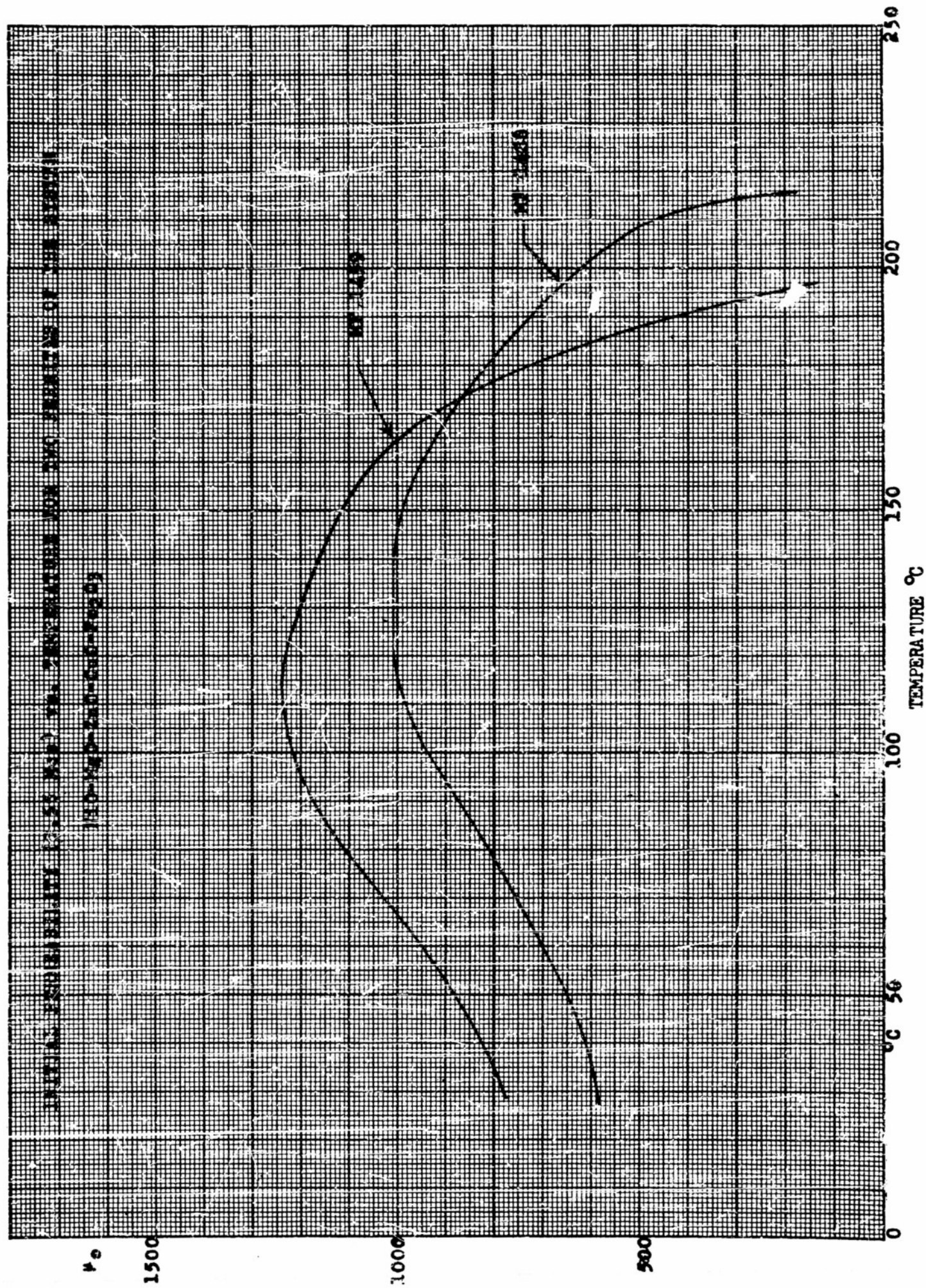
PRESSING PRESSURE	THROUGH 20 MESH		THROUGH 20 MESH ON 80 MESH	
	WITHIN $\pm 2\text{-}1/2\%$	WITHIN $\pm 5\%$	WITHIN $\pm 2\text{-}1/2\%$	WITHIN $\pm 5\%$
3120 PSI	86.0%	100%	88.6%	100%
2080 PSI	66.0%	95.5%	84.0%	100%
1040 PSI	59.0%	91.0%	70.5%	97.8%

GENERAL CERAMICS & STEATITE CORPORATION

GRAPH 167

Sept. 29 - 1953





SATURATED HYSTERESIS LOOPS (60 cps) FOR THREE FERRITES COVERING A
WIDE RANGE OF NICKEL OXIDE CONTENT

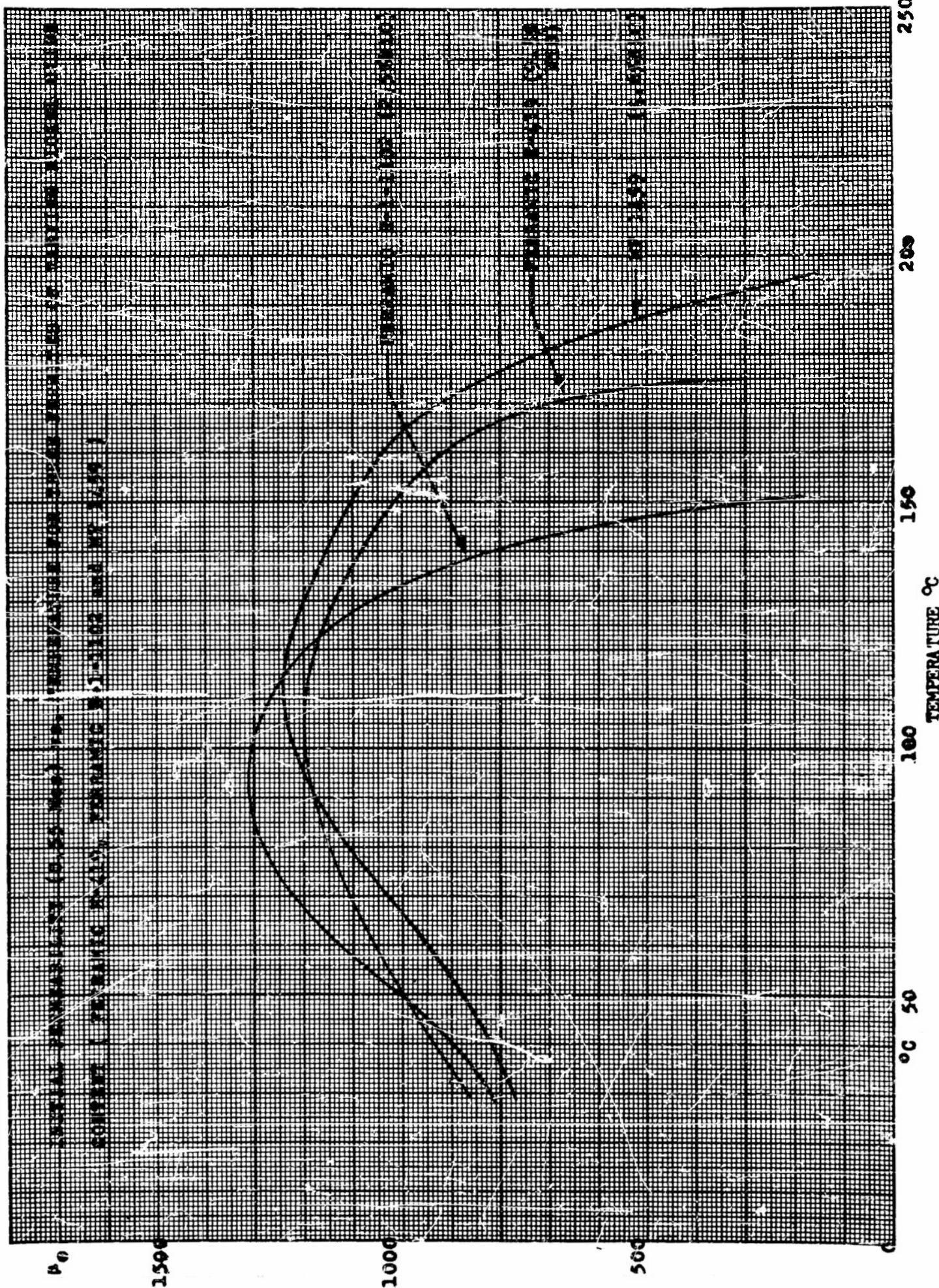


H-1 Body (MF 1102)
Oxide system $\text{MgO-NiO-ZnO-CuO-Fe}_2\text{O}_3$
Nickel Content: 2.5 %
Magnetic Specifications:
 $B_s = 2800$ gauss
 $H_c = 0.35$ oersteds (maximum)
 $\mu_{\text{max}} = 3800$

MF 1459
Oxide System $\text{MgO-NiO-ZnO-CuO-Fe}_2\text{O}_3$
Nickel Content: 4.6 %
Magnetic Specifications:
 $B_s = 2860$ gauss
 $H_c = 0.19$ oersteds
 $\mu_{\text{max}} = 3500$



H-Body (MF 419)
Oxide System $\text{NiO-ZnO-CuO-Fe}_2\text{O}_3$
Nickel Content 9.5 %
Magnetic Specifications:
 $B_s = 3400$ gauss
 $H_c = 0.18$ oersteds
 $\mu_{\text{max}} = 4300$



DISTRIBUTION OF λ , (144) OF MF 254 "G" AT 27°C; C-2 PREPARED
TWO DIFFERENT PARTIAL PRESSURES, THREE DIFFERENT FREQUENCIES
(DIS 3-309)

