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Magnetic Properties of Apollo 12 Lunar Samples 12052 and 12065

by

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Abstract—Magnetization versus field and magnetization versus temperature data indicate that the ferromagnetic material in Apollo 12 samples 12052 and 12065 is spheroidal grains of iron having Curie temperatures of 782° and 788°C respectively. The samples carry natural remanent magnetization that is stable with respect to the earth's field, of the order of 6 to 20 x 10^{-6} emu/gm in intensity. This magnetization consists of several components, all of which are destroyed by heating in vacuum to temperatures below 600°C. The ability of the rocks to acquire thermoremanent magnetization below 200°C is also destroyed by heating to 600°C. The NRM in these rocks is concluded to have resulted from several magnetizing events that occurred at moderate temperatures some time later than the rocks' last cooling.

Introduction

Natural remanent magnetization (NRM) has been reported in several specimens of crystalline rock returned by the Apollo 11 expedition to the moon (Helsley, 1970; Nagata et al., 1970; Runcorn et al., 1970). These NRM's ranged from 2×10^{-6} to 2×10^{-5} emu/gm in intensity, and exhibited low to moderate stability against alternating-field and thermal demagnetization. If these NRM's were acquired by the rocks while on the moon, as seems likely, the presence of a lunar magnetic field, either steady or transitory, is implied; a brief discussion of this implication was given by Runcorn et al. (1970). The magnetic experiments reported here were performed on two Apollo 12 crystalline rocks (12052 and 12065) and were designed with the following objectives: (1) to determine the amount, stability, and carrier of NRM, (2) to ascertain whether any observed NRM could be thermoremanent magnetization (TRM) acquired by the rocks during their initial cooling, and (3) to estimate the magnitude of lunar magnetic field necessary to produce such magnetization. The available samples were in the form of two sawn cubes weighing approximately 10 gm and smaller fragments weighing approximately 0.2 gm. Heating experiments according to the method of Thellier and Thellier (1959) were done in fields of a few thousand gammas on the larger specimens to investigate the thermal stability of NRM and the distribution of partial thermoremanent magnetization (PTRM). The smaller fragments were used for strong-field magnetic experiments to determine some of the intrinsic magnetic characteristics of the rocks. Owing to the limited amount of material, no alternating-field demagnetization was done.

High-Field Magnetization

Magnetization curves for specimen 12052,48 at four temperatures are shown in Fig. 1. The magnetization is seen to be strongly temperature-

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Figure 1 near here

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dependent at low temperatures, indicating a relatively large paramagnetic and/or antiferromagnetic contribution to the total susceptibility. To evaluate the possible contribution of the superparamagnetism that was found to be characteristic of the Apollo 11 fines and breccia and, to a lesser extent, crystalline rock (Nagata et al., 1970), the same magnetization data are shown in Fig. 1 as a function of reduced field H/T. The presence of a ferromagnetic component is clearly evident from the fact that the curves do not coincide.

A notable feature of the magnetization curves in Fig. 1 is that saturation of the ferromagnetic component is not approached until fields of about 5000 oe are exceeded. This may be interpreted to mean that the ferromagnetic mineral is iron, or iron alloy of high permeability, in the form of multidomain spherical particles. Because of their large demagnetizing factor and high permeability, the self-demagnetizing field of such particles is so great that in moderate external fields their net magnetization is virtually zero. Therefore at temperatures well below the Curie point and in low fields the apparent susceptibility is temperature-independent and is given by $\chi_a = 3/4\ \pi\rho$, and assuming a density $\rho = 7.8$ for the spheres, $\chi_a = 0.03\ gm^{-1}$ (Senftle et al., 1964; Duncorn et al., 1970).

Figure 1.--Lower right: magnetization versus magnetic field, crystalline rock 12052,48. Upper left: the same magnetization data as a function of reduced field H/T.

By plotting the observed initial susceptibilities as a function of reciprocal temperature, the temperature-independent part of the susceptibility of specimen 12052,48 is estimated to be between 1.5 and 2.0 x 10^{-6} gm^{-1}. This value is only 25% to 30% of the total initial susceptibility, 6.0 x 10^{-6} gm^{-1}, measured at 291°K; the remainder is due to paramagnetic and/or antiferromagnetic minerals.

The amount of metallic iron in the rock may be estimated from the susceptibility data. Using the values given above, the percentage of iron in rock 12052,48 is 0.05 to 0.07 by weight. This proportion is in good agreement with those found using other magnetic methods in rock 12065 (Tsay et al., 1971), rocks 12021 and 12063 (Pearce et al., 1971), and rock 12053 (Nagata et al., 1971).

Magnetization versus temperature in the range -180°C to 850°C was measured using the recording magnetic balance described by Doell and Cox (1967). The temperature calibration was checked with electrolytic iron (measured Curie temperature 775°C) and pure natural magnetite (measured Curie temperature 583°C). Curie temperatures were determined using a graphical method (Grommé et al., 1969) that tends to give a slightly high value; Arajs and Colvin (1964) have reported an accurately determined ferromagnetic Curie temperature for pure iron of 771° ± 2°C. The relative precision of our Curie temperature measurements is about ± 2°C.

Thermomagnetic curves for specimens 12052,48 and 12065,37 are shown in Fig. 2. The Curie temperatures of these specimens are 788°C and

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Figure 2 near here

Figure 2.--Magnetizations of crystalline rocks 12052,48 and 12065,37 versus temperature in a field of 5500 oe. These are the first heating experiments on these specimens, and were done in a vacuum of 5×10^{-6} torr. Heating and cooling curves are indicated by arrows; the rate was 10°C per minute.

782°C respectively, significantly higher than that of pure iron. The only alloying elements of any importance that can raise the Curie temperature of iron are cobalt and vanadium (Bozorth, 1951). Gibb et al. (1970) report the presence of 1.5% Co, 1.9-2.3% Ni, and 0.1-0.2% Cu in metallic iron in rock 12052, while Cameron (1971) reports only 1.0 to 1.7% Ni in iron in rock 12065. To raise the Curie temperature of iron 13°C would require only 1.2% cobalt (Forrer, 1930), but the effect of nickel is opposite and the exact Curie temperature of this quaternary alloy is difficult to predict.

The thermomagnetic curves in Fig. 2 were obtained in a vacuum of about 5×10^{-5} torr. The cooling curves nearly retrace the heating curves, indicating that at this pressure the rate of inward diffusion of oxygen was sufficiently reduced to minimize oxidation of the iron within the specimens. To evaluate further the effects of oxidation, these experiments were repeated using the same specimens, first in nitrogen and then in air. The resulting thermomagnetic curves are shown in Fig. 3 in which the

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Figure 3 near here

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effect of oxidation is clear. All the curves have inflections at the Curie temperature of magnetite and the magnetizations above this point are significantly reduced.

The form of the magnetization curve at room temperature has turned out to be a sensitive indicator of oxidation in these rocks, as is

Figure 3.--Magnetizations of crystalline rocks 12052,48 and 12065,37 versus temperature in a field of 5500 oe. The second heating experiments on these specimens (left) were done in nitrogen, and the third heatings (right) were done in air. Heating and cooling curves are indicated by arrows.

illustrated in Fig. 4. Because the magnetite that results from oxidation

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Figure 4 near here

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has a much smaller self-demagnetizing field than do the iron spherules, it saturates in a much lower external field. This gives rise to convexity in the formerly straight parts of the curves below about 5000 oe. That a minor amount of oxidation occurred during heating in vacuum is evident from the curves marked a in Fig. 4. It is nevertheless clear from these experiments that heating to temperatures of the order of 800°C at pressures less than about 10^{-6} torr will produce no gross alteration of the ferromagnetic component of these rocks.

Time-Stability of NRM

All measurements of NRM were made using a spinner magnetometer in a field of a few hundred gammas to minimize possible effects of viscous magnetization. As received, specimens 12052,32 and 12065,67 had NRM's amounting to 2.1×10^{-6} and 6.0×10^{-6} emu/gm respectively. The specimens were remeasured after storage for five hours in the earth's magnetic field and again after four months. No significant changes in NRM direction or intensity were observed, and we conclude that viscous remanent magnetization in these rocks is negligible.

NRM-TRM Experiments

The thermal stability of NRM and the production of artificial TRM in specimens 12052,32 and 12065,67 were investigated using the double heating method of Thellier and Thellier (1959). The specimens were heated and

Figure 4.--Magnetizations versus magnetic field at room temperature, of crystalline rocks after successive heating experiments illustrated in Figs. 2 and 3. a, after heating in vacuum (5×10^{-5} torr); b, after heating in ~~dry~~ nitrogen; c, after heating in air. Magnetization curves for the same specimens before heating are shown dashed for comparison.

cooled in a vacuum of 5×10^{-6} torr in a nonmagnetic oven within a space that is field-free to within ± 10 γ. Results of Helsley (1970) and Nagata et al. (1970) on Apollo 11 crystalline rocks indicated that fields of the order of a few thousand gammas might be expected to produce TRM comparable in magnitude to the NRM; accordingly, fields of 5000 γ and 2000 γ were used.

The directions of magnetization after the successive heatings are shown in Fig. 5 and the corresponding intensities of NRM and PTRM are

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Figure 5 near here

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given in Table 1. After the first double heating, to 195°C in 5000 γ;

TABLE 1 NEAR HERE

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the NRM's in both specimens had diminished by about one-half to two-thirds and had changed directions significantly, and significant PTRM's were produced. As a consistency test and because of the direction change, the specimens were then heated to 195°C and cooled in null field. The NRM intensities agreed well with those calculated from the double heating, and the NRM direction in specimen 12052 remained constant but that in specimen 12065 continued to change (Fig. 5). A second double heating to 195°C was done in a field of 2000 γ, which showed that the PTRM from 195° to 20°C was linear with applied field (Table 1). The third double heating was to 395°C in 2000 γ, and as expected the NRM was further diminished.

Figure 5.--Directions of remanent magnetization in crystalline rocks 12052,32 and 12065,67 after heating in vacuum (5×10^{-6} torr) in various applied fields. Directions are plotted on upper hemisphere of equal-area projection. Letters a through h indicate sequence of experiments. Direction of applied field is indicated by $\pm$ H and sign of applied field by short arrows; figures by arrows designate intensity of field in thousands of gammas. Point marked NRM is the natural remanent magnetization measured before heating. Maximum temperatures attained in successive heatings are shown in degrees C. Magnetization directions after paired heatings in opposing fields are joined by lines; midpoints of these lines are marked to indicate the calculated NRM directions. Note that heating d was done in zero applied field.

The PTRM acquired in 2000 γ was less after heating to 395° than it had been after heating to 195°C, indicating that the carrier of the PTRM was being progressively destroyed by the heating. After heating to 590°C and cooling in 2000 γ, both NRM and PTRM were below the sensitivity limit of the magnetometer (approximately 3×10^{-7} emu/gm). Finally, to confirm that an irreversible change in the carrier of the NRM and PTRM had occurred, both specimens were heated to 195°C and cooled in 5000 γ; again no remanent magnetization could be detected. During the heating experiments, the NRM

Table 1. Intensities of natural remanent magnetization and partial thermoremanent magnetization in specimens after heating and cooling in vacuum in controlled magnetic field

Experiment	T	H	Specimen 12052,32 NRM	12052,32 PTRM	12065,67 NRM	12065,67 PTRM
a	20	--	2.04	--	0.56	--
b	195	+5	(0.60)	0.23	(0.28)	0.11
c	195	-5				
d	195	0	0.61	--	0.31	--
e	195	+2	(0.62)	0.09	(0.29)	0.06
f	195	-2				
g	395	+2	(0.14)	0.04	(0.13)	0.02
h	395	-2				
i	590	+2	< 0.03	< 0.03	< 0.04	< 0.04
j	195	+5	< 0.03	< 0.03	< 0.03	< 0.03

T, maximum temperature in degrees Celsius.

H, ambient magnetic field in units of 10^{-2} oe.

NRM and PTRM in units of 10^{-6} emu/gm; NRM values in parenthesis calculated, values not in parenthesis directly measured.

direction in specimen 12065,67 changed continuously and unpredictably up to 395°C, while the direction in specimen 12052,32 changed during the first heating to 195°C but remained constant until the maximum temperature was raised to 395°C.

In order to test whether appreciable oxidation had occurred during these heating experiments, magnetization curves were determined on fragments from both specimens. The results are shown in Fig. 6 together

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Figure 6 near here

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with the magnetization curves of unheated samples for comparison. A small amount of iron was oxidized to magnetite, but the bulk magnetic properties of these specimens probably were little changed.

Discussion

From the experiments described above it is not possible to identify the carrier of the NRM and PTRM. One possibility is that it consists of grains of iron so small as to have very low blocking temperatures, and that were preferentially oxidized during the heating experiments. A difficulty with this hypothesis is that the transition from single domain to multidomain magnetic structure in iron occurs at a particle size of about 320Å (Néel, 1949), and the particle size dependence of blocking temperature distributions is not well known for multidomain iron grains.

Figure 6.--Magnetizations versus magnetic field at room temperature, of crystalline rocks after successive heating experiments described in Table 1 and Fig. 5. Magnetization curves for specimens 12052,48 and 12065,37 before heating are shown dashed for comparison.

Another possibility is that over a long period of time after the rocks cooled a structural state developed in the iron grains such that they were subdivided in part into extremely small effective grain sizes with correspondingly low blocking temperatures. A third and related possibility is exsolution from the original homogeneous iron alloy of very small grains of some other composition again having low blocking temperatures. In either case heating would result in homogenization of the iron grains and disappearance of NRM and PTRM. That exsolution of some kind did in fact occur is suggested by comparing the thermomagnetic curves in Figs. 2 and 3. In both specimens, the first reheating produced a small but unmistakable increase in Curie temperature, amounting to 8°C in specimen 12052,48 and 18°C in specimen 12065,37. The simplest explanation of these increases is resolution of exsolved cobalt in the iron. For homogenization of the iron grains to account for destruction of most of the NRM carrier at moderate temperatures would require appreciable resolution (and change in Curie temperature) to have occurred during the first heating of these specimens; the thermomagnetic curves in Fig. 2 do not preclude this.

Other more subtle hypotheses can be constructed to account for the changes in the magnetic properties of these specimens. The important point is that the heating experiments described in the previous section show clearly that the NRM in specimens 12052 and 12065 cannot be ordinary TRM that would have been acquired as the rocks initially cooled below their Curie temperatures. Furthermore, the NRM in specimen 12052,32 consists of at least two components and that in specimen 12065,67 apparently consists

of three components. For rock 12065 the multicomponent nature and moderate degree of stability of the NRM has also been clearly shown by the alternating-field demagnetization results of Hargraves and Dorety (1971). The NRM in rocks 12052 and 12065 must have resulted from several magnetizing events that occurred at temperatures not much greater than those prevailing on the lunar surface and at some time later than the rocks' initial formation or last cooling.

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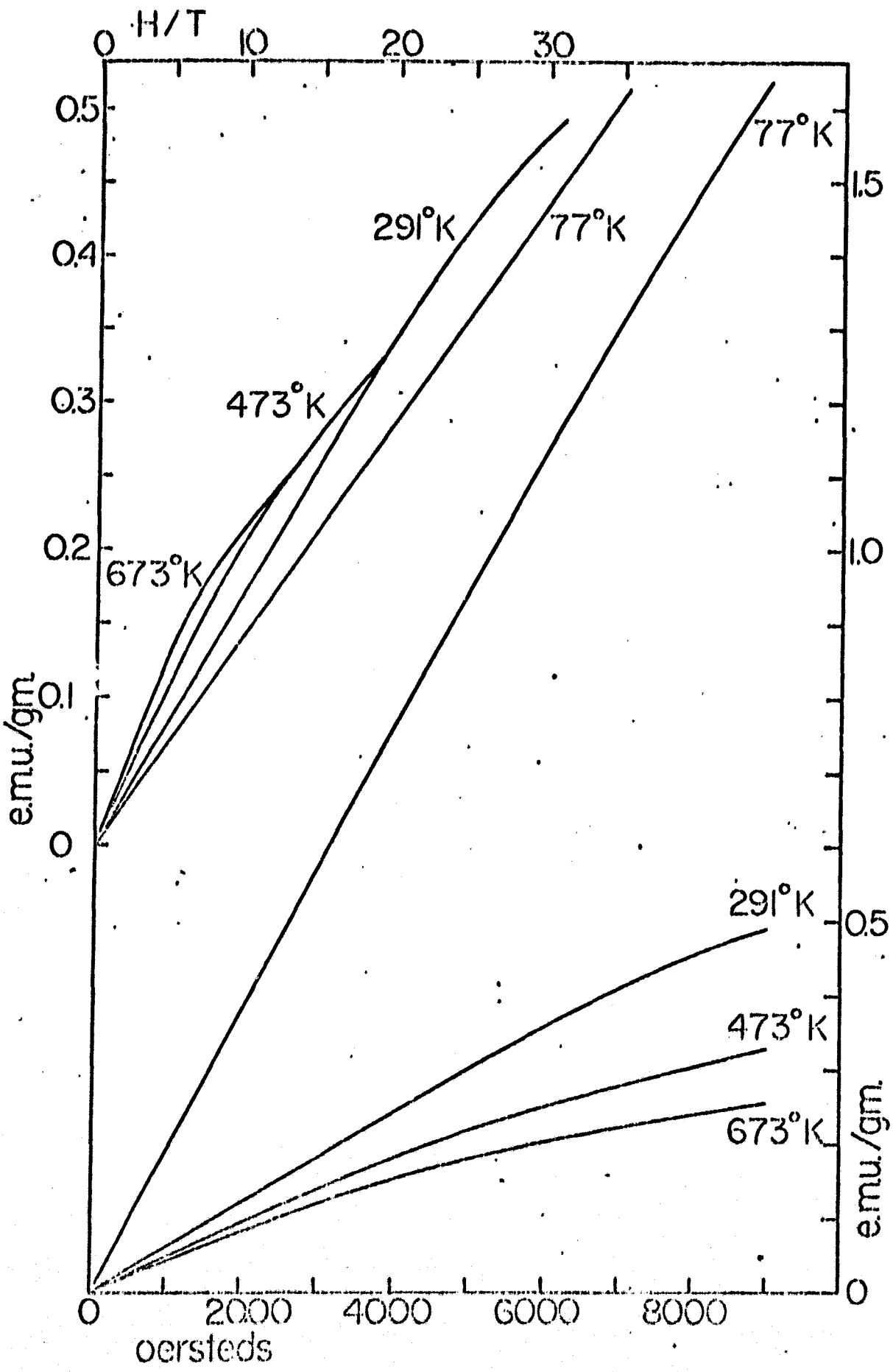
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Fig. 1

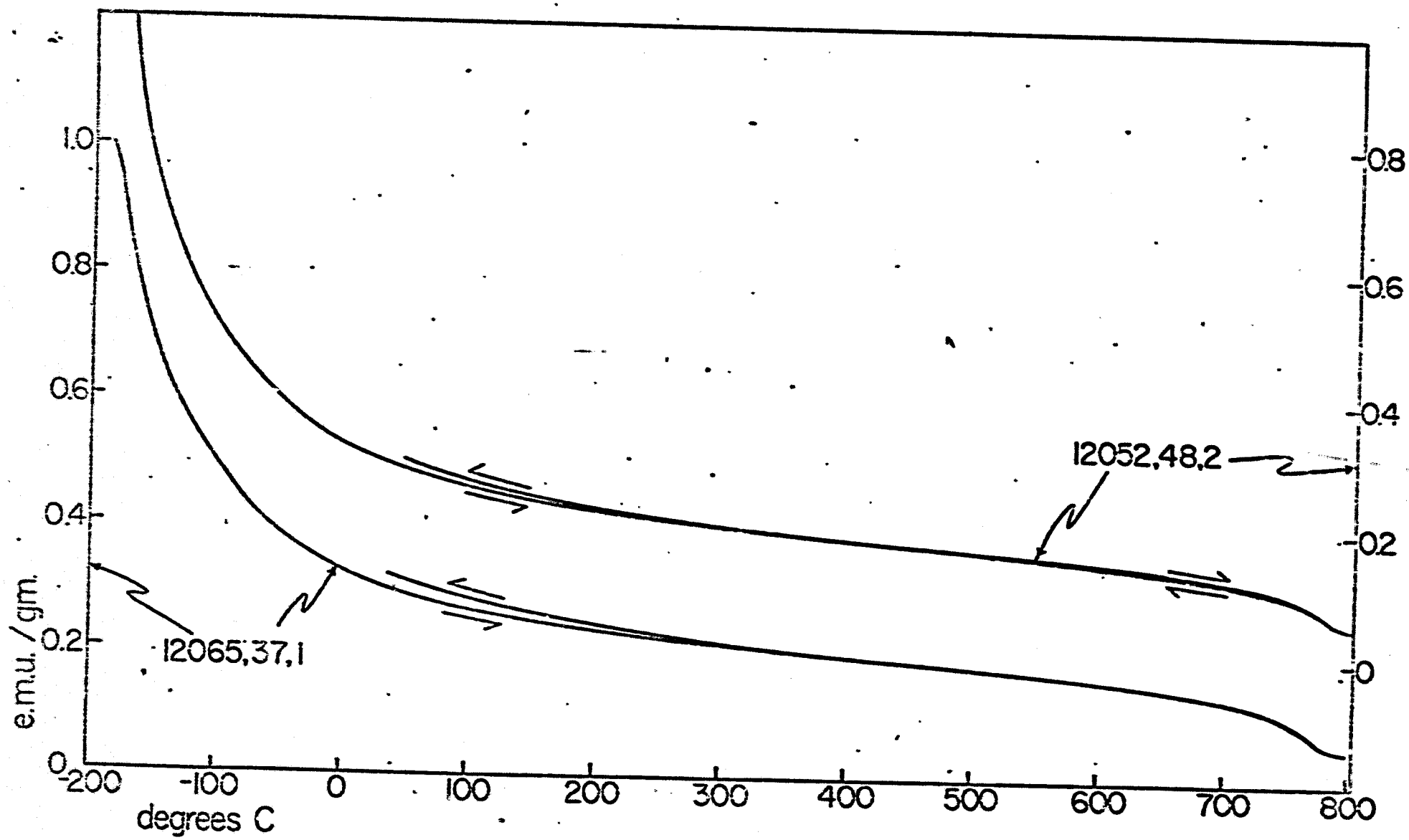
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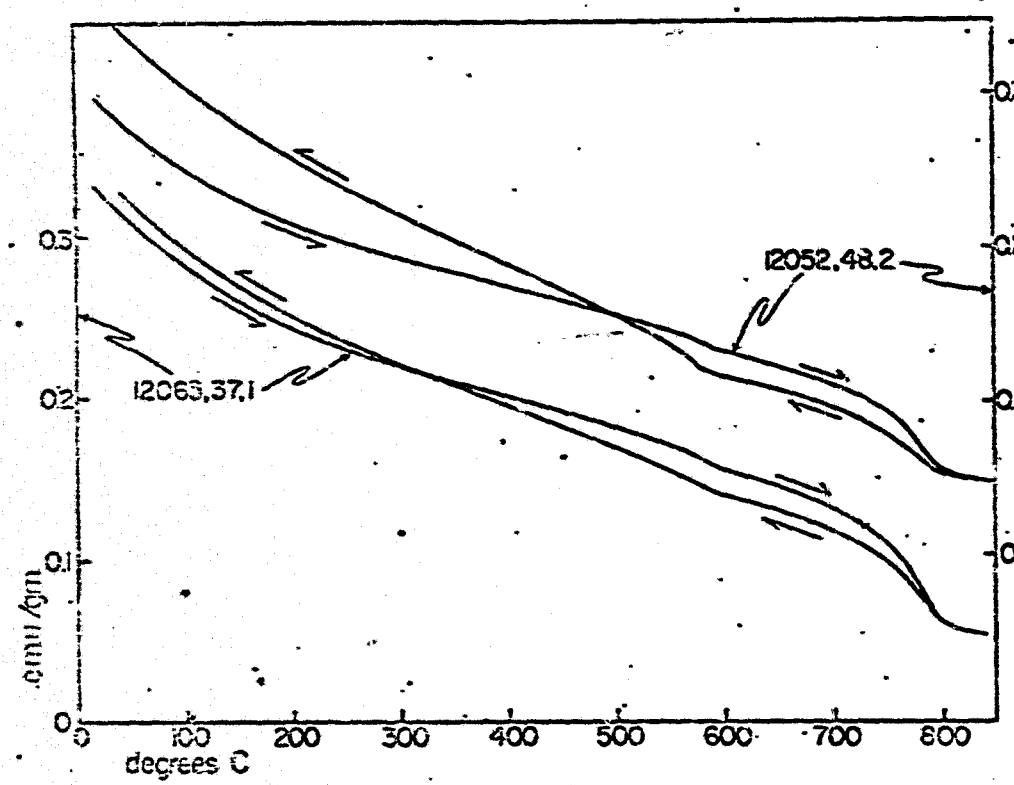
Fig. 2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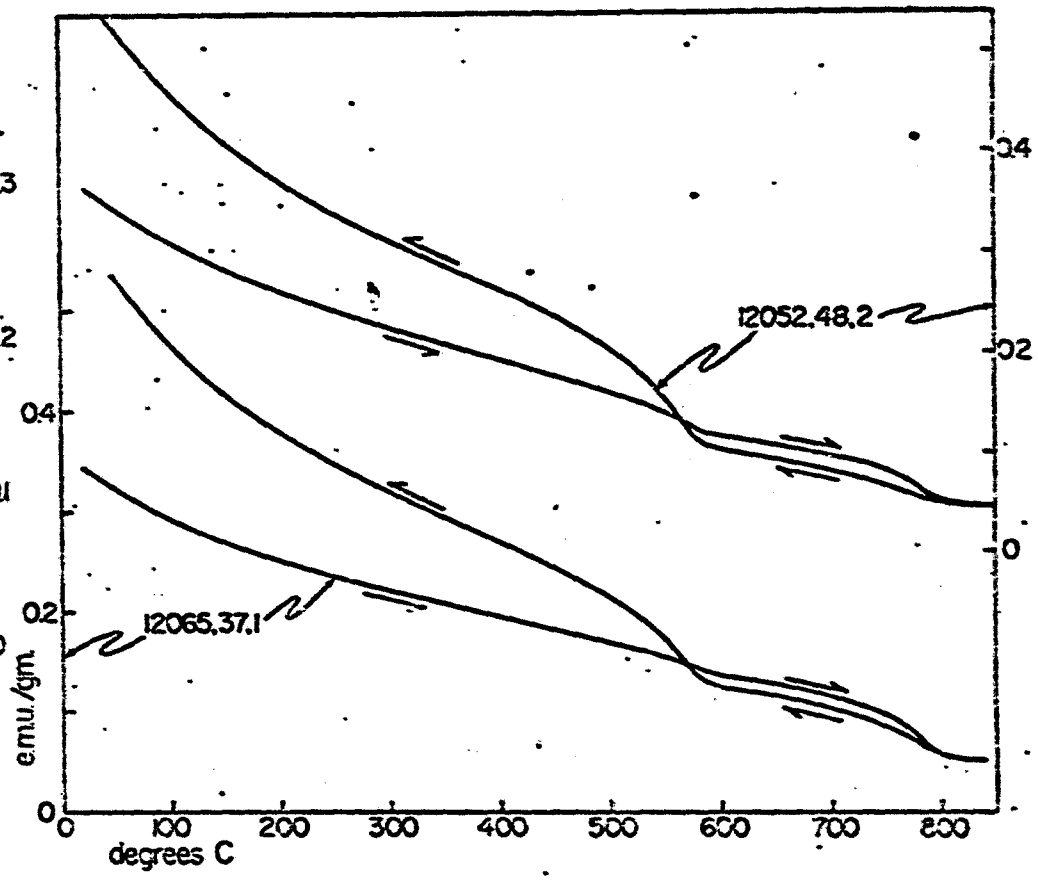
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Fig. 3

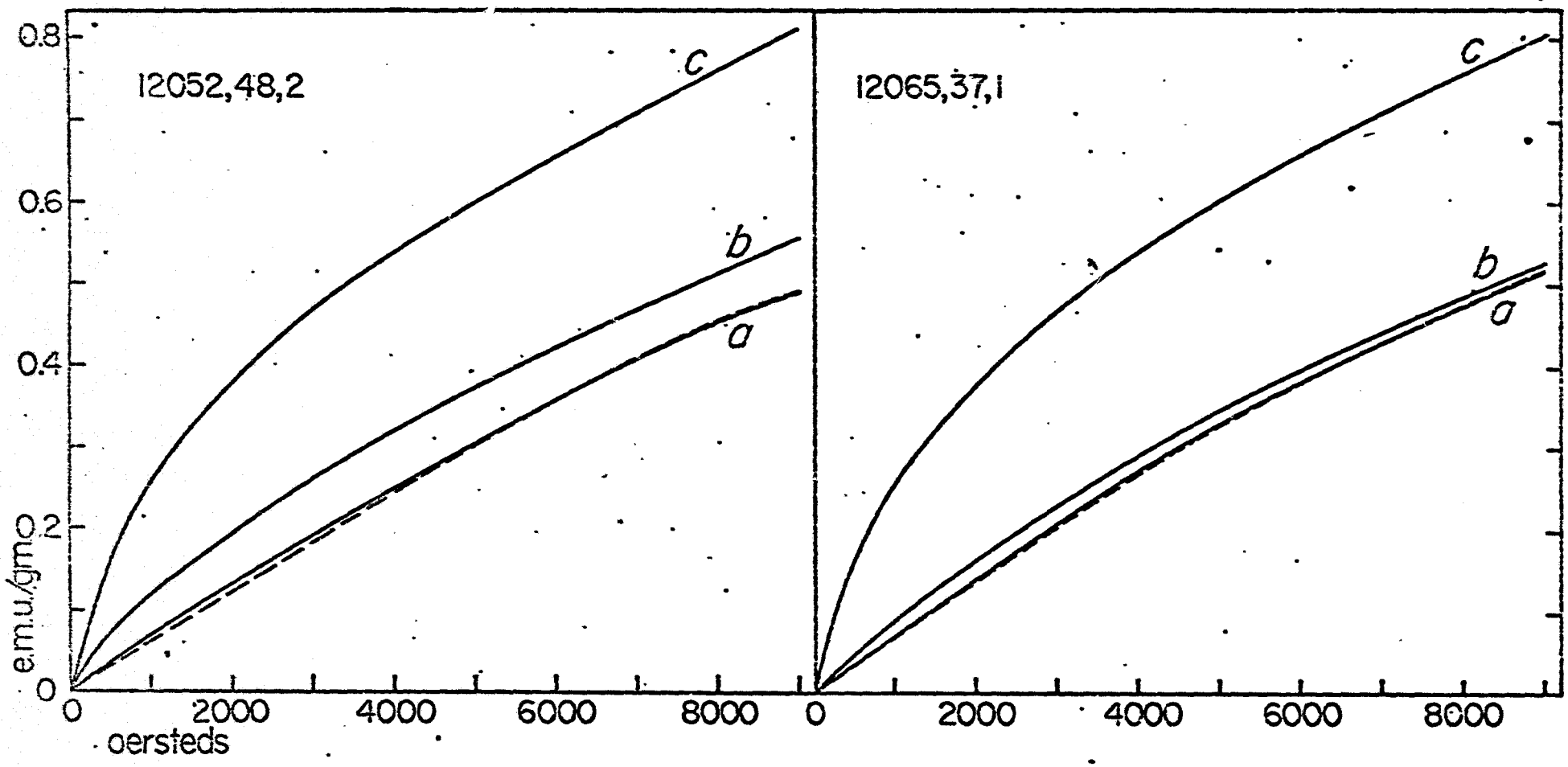
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Fig. 4

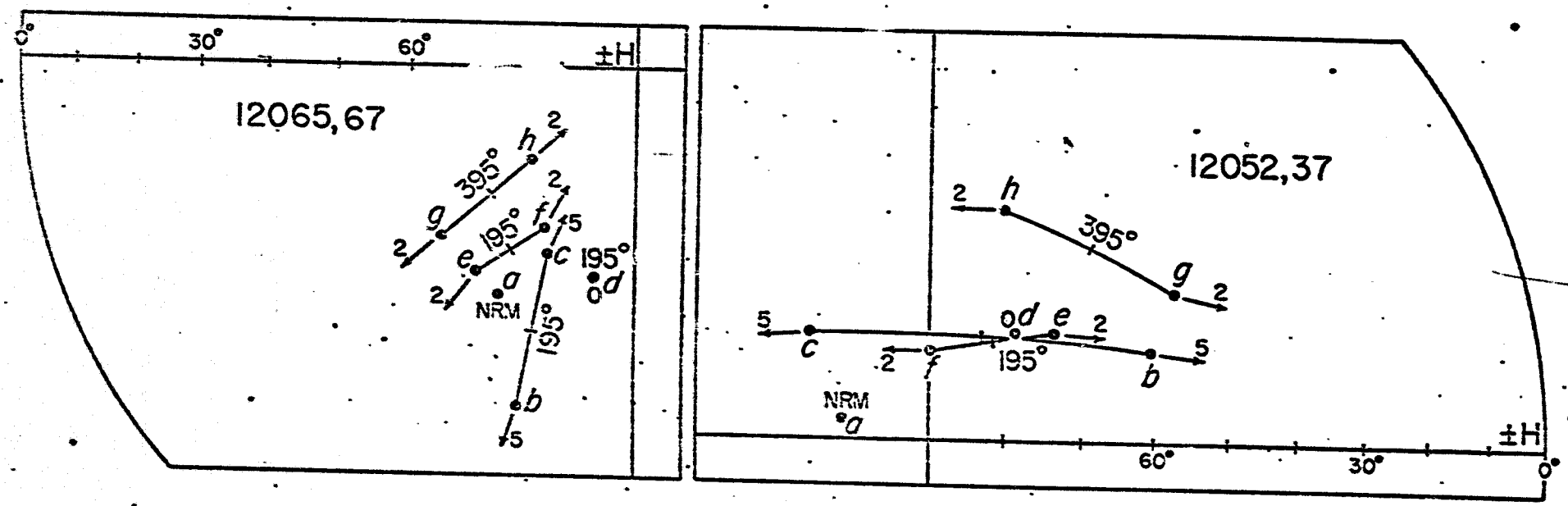
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Fig. 5

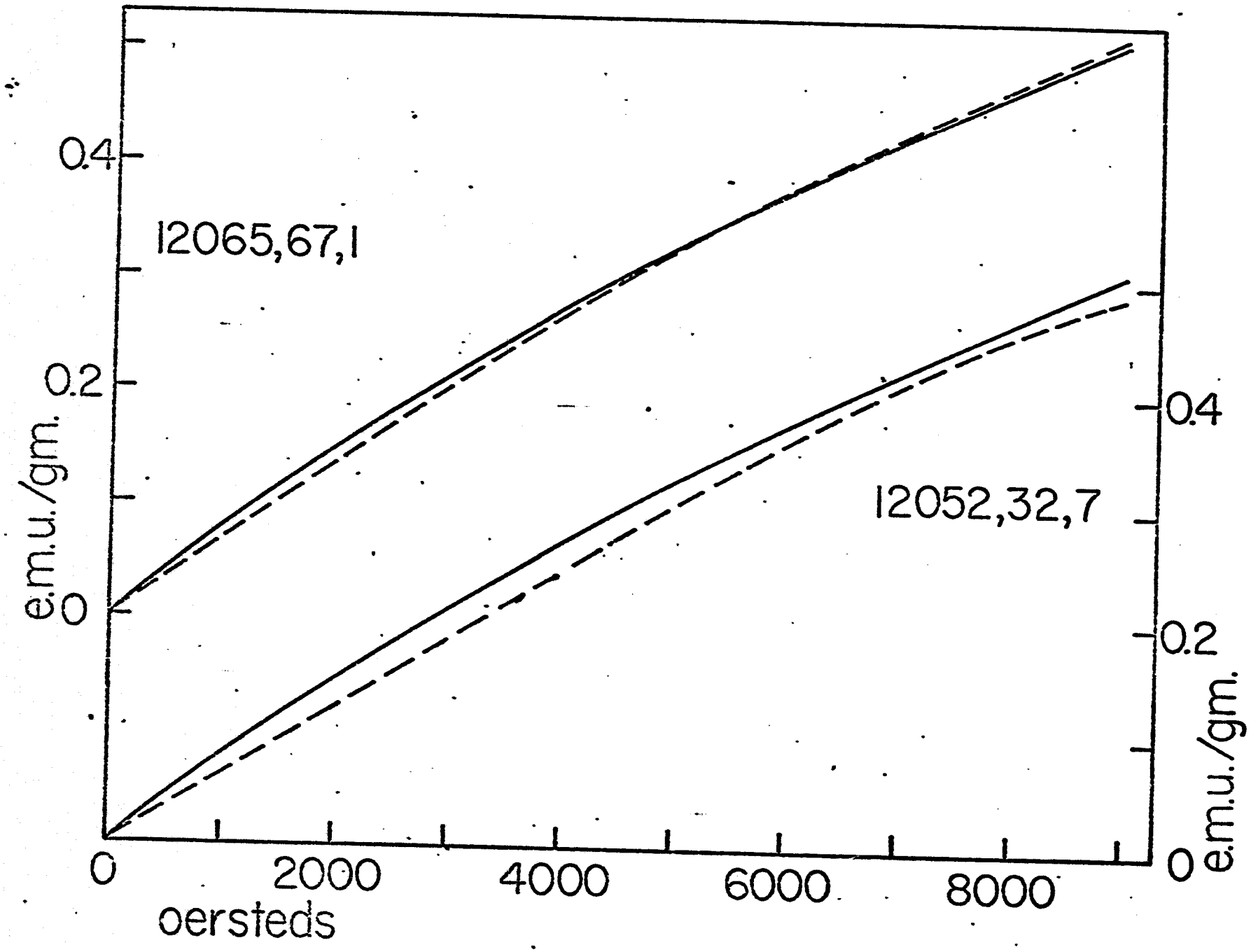
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Fig. 3-1