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LOW-FIELD MAGNETIC SUSCEPTIBILITY OF ASBESTOS

A. A. WINER AND D. KARPOFF

MINERAL PROCESSING DIVISION

AND

D. T. A. SYMONS

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A. A. Winer*, D. Karpoff** and D.T.A. Symons***

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ABSTRACT

An instrument developed at the Geological Survey of Canada has been used for measuring the amount of magnetite present in chrysotile asbestos and for measuring the low-field susceptibility of asbestiform minerals such as amosite and brucite. The instrument has been found to be sensitive, accurate, and reliable for these purposes.

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Direction des Mines

Rapport de Recherches R 232

SUSCEPTIBILITÉ D'AMIANTE DANS UN CHAMP MAGNÉTIQUE FAIBLE

par

A.A. Winer*, D. Karpoff** et D.T.A. Symons***

RÉSUMÉ

Un appareil mis au point par la direction géologique du Canada a été employé pour la mesure de la quantité de magnétite présente dans l'amiante chrysotile ainsi que pour la mesure des susceptibilités, dans un champ magnétique faible, des matériaux amiantifères come l'amosite et la brucite. L'appareil utilisé s'est relevé comme étant sensible, précis et convenant bien à cet effet.

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SUMMARY OF RESULTS

Chrysotile, amosite, and brucite are diamagnetic minerals. The relative degree of diamagnetism appears to be: amosite > brucite > chrysotile. However, this order may not be entirely correct because there may be a ferromagnetic effect, due to the presence of magnetite even in the "cleanest" chrysotile sample, which would modify the result. Magnetite appears to be the major controlling factor in susceptibility variation for the different mixtures used in these experiments

The calculated or theoretical values of magnetite present in the mixtures (Figures 4 & 5) agree very well with the observed results. These values are within the experimental error (5×10^{-5} emu/g) which is mainly due to sampling. It is well known that representative sampling of asbestos fibre is very difficult and detailed procedures have been developed in the asbestos industry to minimize this error. Representive sampling becomes even more difficult where magnetite is concerned. The instrumental error (0.5×10^{-5} emu/g) contributes much less to the overall experimental error.

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This study has shown that the Geological Survey of Canada apparatus used for measuring magnetic susceptibility is an accurate and reliable instrument for measuring the amount of magnetite and/or other ferromagnetic minerals present in asbestos.

The measurement procedure adopted to offset susceptibility variations while the fibre is rotated on its axis does compensate sufficiently for this potential source of error.

Because of the sensitivity and ease of operation of this method, it would be possible to use the instrument for on-line quality control.

Further study of the low-field susceptibilities of asbestos and its contaminants, and of the low-field susceptibility in relation to the saturated-field susceptibility is in progress. Such studies may prove of value in the determination of the percentages of other mineral contaminants in asbestos.

CONTENTS

INTRODUCTION

A continuing program in the investigation of the properties of asbestos fibre is being carried on at the Mines Branch. The behaviour of chrysotile asbestos in an electrical field has previously been investigated[1].

Magnetite is a common adulterant in Canadian chrysotile asbestos. It is removed wherever feasible, particularly if the asbestos is to be used for electrical insulation applications. Therefore, a method for analyzing the asbestos ore and the asbestos fibre during selected stages of the milling process is important. The present study utilizes a low-field magnetic susceptibility meter which was constructed by the Geological Survey of Canada. It was used in this study to measure magnetic effects in asbestos fibre and mixtures produced by as little as 0.01 per cent magnetite. Minerals such as brucite and serpentine are at times associated with chrysotile fibre, and therefore their magnetic effect in the mixtures is also of interest.

This method is believed to be more sensitive and rapid than previous methods.

SAMPLES

The samples used in this study were Canadian chrysotile and associated minerals except where noted. They were obtained from various mines in Canada and included both processed and raw fibre.

APPARATUS

The apparatus used in this study, described more fully in another goverment publication[2], consists of two accurately balanced ac transformers

coupled to a low-noise, high-gain amplifier (Fig. 1). The transformers have ferrite cores in the form of toroids with an air gap (Fig. 2). The pick-up or secondary coils (S_{M1}, S_{M2}, S_{R1}, S_{R2}) are located on either side of the air gap with the energizing or primary coils (P_M, P_R) on the opposite side of the toroid (Fig. 3). The transformers are connected in series opposition as a transformer balance. A small ferrite slug is moved into, or out of, the air gap in the reference transformer to exactly balance the circuit. When a sample is placed in the air gap of the measuring transformer, which has an axially directed field with a peak intensity of 0.6 Oe, the magnetic moment of the sample changes the reluctance of the air gap and unbalances the circuit. Because the conductive component is rejected by the phase-sensitive detector of the amplifier, the inductive component, or susceptibility, is directly related to the amount of unbalance. This is amplified and displayed directly on the digital voltmeter. The amplifier-voltmeter scale is set so that ferromagnetic components give positive readings and diamagnetic components give negative readings. The sample holder is free to rotate, to facilitate measurement along different sample axes.

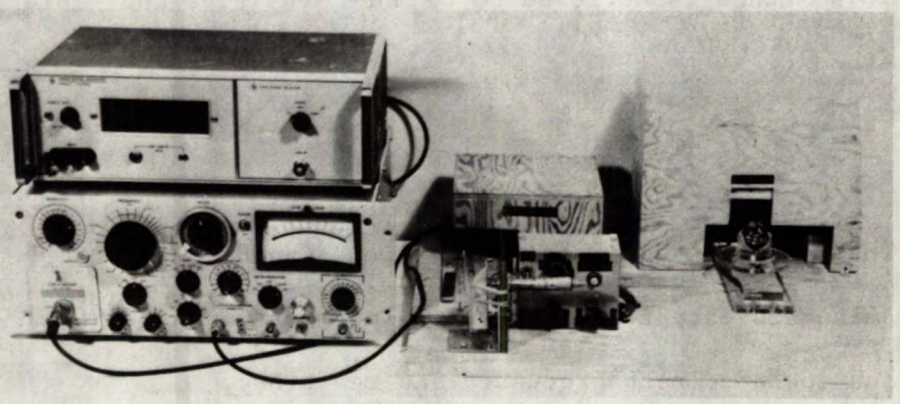

Figure 1*. Toroid transformer balance showing the operating layout with the amplifier unit on the left surmounted by the digital voltmeter and the transformers on the right covered with draught shields.

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* (after Christie & Symons).

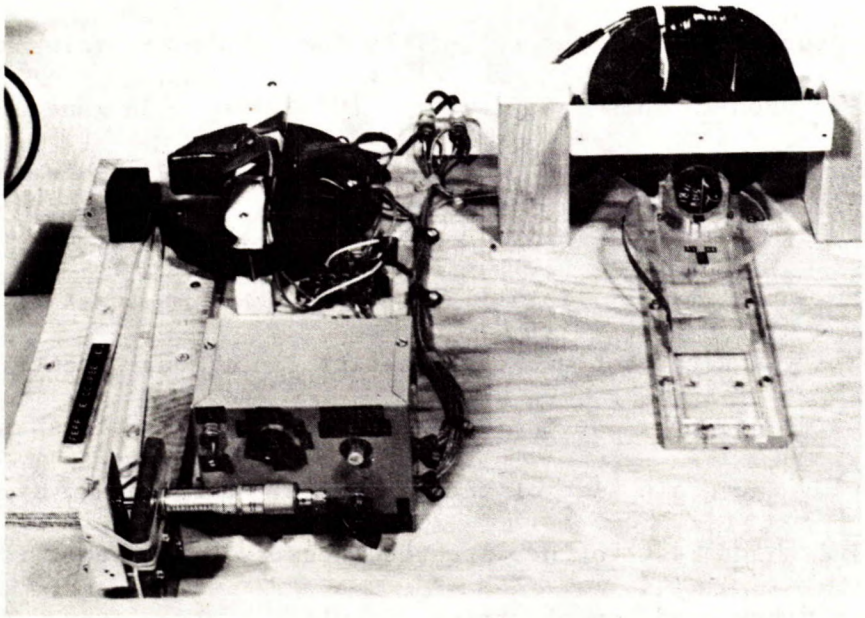


Figure 2*. Close-up view of the transformers. The reference transformer (upper left) is mounted so that the slugs (extreme upper left) can be moved in and out of its air gap using the micrometer (lower left) fine adjustment. The measuring transformer (upper right) is mounted so that the specimen can be in the gap and rotated in its holder (middle right). The variable resistor, variable capacitor and reasonating capacitor are in the box (middle left).

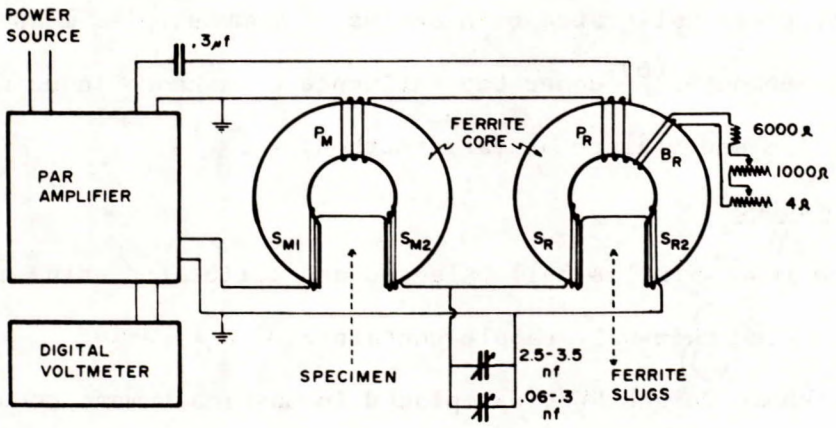


Figure 3*. Block diagram for the toroid transformer balance.

* (after Christie & Symons).

This instrument has a noise level of 2×10^{-8} emu (cgs) so that the minimum measurable weight susceptibility for samples weighing in the order of 6 g such as used in this study, is 2×10^{-7} emu/g. In general, the main ferromagnetic mineral present in asbestos fibres is disseminated magnetite but native nickel-iron may also be present.(3) Nagata(4) has shown that there is an approximately linear relationship between low-field bulk susceptibility and the content of magnetite (in mixtures containing less than 10 per cent magnetite). Deviation from the linear relationship is primarily a function of the grain size of the magnetite. Large grain size of magnetite is of definite interest to the asbestos industry, because it is deleterious in asbestos fibre used for electrical insulation, even though the total amount of magnetite may be small.

Calibration

The instrument is most easily calibrated by mixing known amounts of finely ground chemically pure magnetite and plaster of Paris in known amounts. Alternatively, various chemicals of known susceptibility may be used(5). This instrument was calibrated by a series of rock samples measured on a biastatic magnetometer(6) under the influence of a known inducing field and by the magnetite and plaster of Paris method.

Measurement Method

The instrument is null balanced and calibrated using a standard sample. A covered thin-wall sample container (1 x 1.25-in diameter) of known weight "Wc" was placed in the instrument to determine its diamagnetic contribution "D" to the susceptibility. In practice, calibration of the sample container need be done only on an occasional basis. Thus

$$D = F \left[\frac{1}{6} (R_{x+} + R_{y+} + R_{x-} + R_{y-} + R_{z+} + R_{z-}) - \frac{1}{2} (Z_s + Z_e) \right]$$

where:

R_{x+}, R_{x-}, R_{y+}, R_{y-}, R_{z+} and R_{z-} are the meter readings with the sampled aligned along the arbitrarily chosen orthogonal X, Y and Z axes in positive and negative orientation respectively (Fig. 4); Z_s and Z_e are the zero instrument readings at the start and end of the measurement sequence; and $F = A \times S$, where A is the attenuation setting and S the sensitivity setting on the amplifier.

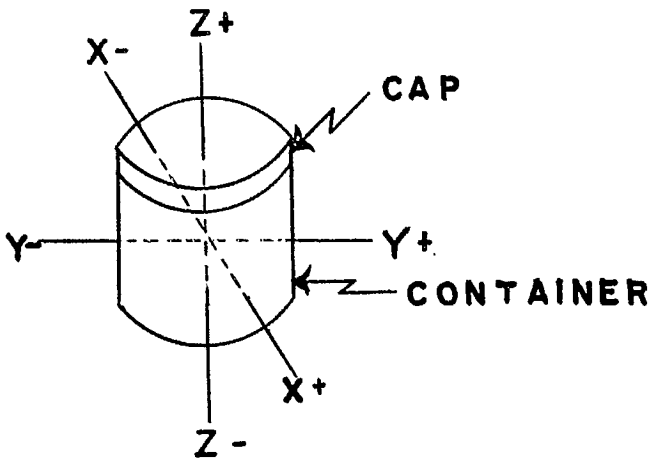


Figure 4. Alignment of sample along the X, Y, Z axes.

After weighing the capped plastic container, the asbestos fibre was manually packed into the container with the fibre aligned along the Z axis. The capped container and sample were again weighed to determine the sample weight.

The weight of sample, $W_s = W_{sc} - W_c$

where:

W_s = sample weight

W_{sc} = sample + container weight

W_c = weight of container.

The magnetic response "M" of the packed sample container is then determined, as before.

$$M = F \left[\frac{1}{6} (R_{x+} + R_{y+} + R_{x-} + R_{y-} + R_{z+} + R_{z-}) - \frac{1}{2} (Z_s + Z_e) \right]$$

The susceptibility (X) of the sample, in emu/g is:

$$X = \alpha (M-D)/W_s$$

where: α is the calibration constant of the instrument.

By following this procedure, a reading on the sample is made twice on each of three mutually perpendicular axes, i.e., a total of six times, in order to reduce any error due to inhomogenous distribution of magnetite in the sample since:

(1) the magnetite may be clustered preferentially in one portion of the container and the instrument response is sensitive to the location of such a cluster in the air gap, and

(2) the magnetite will likely be preferentially aligned with respect to fibre orientation either,

   (a) as crystals grown between fibres along the axis or across the fibre ends in the ore, or

   (b) as adhered dust along milled or processed fibre.

Alignment of the magnetite grains along the axis of the applied field in the air gap can give a significantly greater change in reluctance than alignment perpendicular to the axis by a factor of 2 or more. Aligning the fibres with the Z axis of the container provides a measure of the significance of this anisotropic effect, therefore the averaging of the six readings minimizes any anisotropic bias.

PREPARATION PROCEDURE OF THE ASBESTIFORM SAMPLES

All samples were weighed as previously noted.

The ends of raw or unfiberized samples were cleaned to remove any magnetite.

The processed or milled fibres were also cleaned to remove magnetite and other adulterants.

Processed fibre samples, consisting of groups 3, 4, 5, 6, and 7, were cleaned as noted above, and to these samples finely ground magnetite was added to form mixtures of 0.01, 0.1, 0.5, 1.0. 2.0 and 5.0 per cent magnetite by weight. One sample did not have magnetite added to it.

RESULTS AND DISCUSSION

Calculations

Sample calculations are shown below:

(a) Calibration Constant - Indirect Verification

Magnetite has an apparent susceptibility of 0.043 G cm^3 g^{-1} Oe^{-1}, i.e., 1 per cent magnetite = 4.3×10^{-4} emu/g sample. This compares with Nagata's optimum value of about 4.5×10^{-4} emu/g for volcanic rock[4].

For the G.S.C. instrument used in this study and using samples #20 and #21 as examples (Table 1), results were as follows:

	#20 (1 per cent magnetite added)	#21 (2 per cent magnetite added)
R/6 - Z/2	.193	.297
Sensitivity Correction (X20)	3.86	5.94
Container	.38	.38
Net Response	4.24	6.32
Weight of sample (g)	4.65	4.59
Net Response/g	.913	1.377

Therefore one per cent magnetite has response/g of

$$1.377 - 0.913 = 0.464$$

and the calibration constant should be:

$$0.464 \times \kappa = 4.3 \times 10^{-4}$$
$$= \frac{4.3}{0.464} \times 10^{-4} = 9.26 \times 10^{-4}$$

From a previous calculation, $\kappa = 4.25 \times 10^{-5} \times 21.6 = 9.18 \times 10^{-4}$. The value, 9.26×10^{-4}, compares favourably with the calibration constant, 9.18×10^{-4}, derived prior to this study by two methods, as discussed under CALIBRATION.

(b) Calculation for Bulk Susceptibility (emu x 10^{-5})

Sample # 10
Average value $R/6 = 0.4497 = (R_{x+}, R_{x-}, R_{y+}, R_{y-}, R_{z+} R_{z-}) /6$

$Z/2 = \underline{0.1691} = (Z_s + Z_e) /2$
Response = $R/6\ Z/2 = 0.2806$

Sensitivity Meter Correction (X20) = 5.612
Subtract Container Correction (-0.382) = 0.382
Net response 5.994
Sample weight $W_s = (W_{sc} - W_c) = 12.14 - 6.480 = 5.66$
Net response/g = 5.994/5.66 = 1.059
Calibration constant = 92 x 10^{-5}
Apparent isotropic (bulk) susceptibility (emu/g) = 92 x 10^{-5} x 1.059
= 97.5 x 10^{-5}

The bulk susceptibility results were obtained by the above method of calculation and a summary of the results is shown in Table 1.

Individual Minerals

From the bulk susceptibility values given in Table 1, it is evident that amosite (sample #2) and brucite (sample #3) are diamagnetic. Magnetite was not observed in either sample. Brucite, the fibrous variety of $Mg(OH)_2$, is associated with chrysotile from some deposits in the Eastern Townships of Quebec.

Chrysotile should theoretically give about the same diamagnetic response as brucite. Raw chrysotile (sample #6), with the magnetite cleaned

from the ends of the cross fibres, gave a low negative diamagnetic response. Within the limits of resolution of this low-field bulk susceptibility method, it is thought that a trace amount of magnetite 0.1 per cent) remains in the sample. This gave a low positive ferromagnetic response thereby reducing the negative diamagnetic response of the chrysolite. A slightly processed sample of raw chrysotile (sample #7), with its visible magnetite removed but from the same mine, gave a distinct low ferromagnetic response. The response is very likely due to minute particles of magnetite (0.3 per cent) which have been dispersed and trapped by the opened fibre.

Serpentine is also theoretically diamagnetic. The sample (#28) was cleaned and processed electrostatically to remove as much magnetite as possible, however, it still gave a ferromagnetic response equivalent to about 0.4 per cent magnetite. Magnetite is tenaciously held by opened fibre. It would appear that more effective methods would be required for removal of the remaining magnetite.

The relative order of diamagnetism of the minerals appears to be amosite > brucite > chrysotile; however the latter is in doubt because of the possible presence of minute amounts of magnetite in our most diamagnetic chrysotile sample (#6).

TABLE 1

Susceptibility Summary

Sample No. Coded	Sample Description	Apparent Bulk Susceptibility emu x 10^{-5}/g
1	Plastic capsule plus cap	-
2	Amosite	-6.900
3	Brucite (Eastern Twp., Quebec)	-4.048
4 - Mine A	Raw chrysotile sample, ends cleaned	41.03
5 - " "	" " " , as above, ends and fibres cleaned	7.544
6 - Mine B	Raw chrysotile, ends cleaned	-0.276
7 - " "	" " , hand cleaned	8.740
8 - Mine C	Processed chrysotile fibre, grade 3K, hand cleaned	159.3
9 - " "	" " " " 7R, " "	91.08
10 - " "	" " " " 4T, " "	97.5
11 - Mine D	" " " " 6D, " "	179.6
12 - " "	" " " " 4K, washed & dried	216.0
13 - " "	" " " " 7M, " "	152.5
14 - Mine E	" " " " 5R, " "	112.5
15 - Mine F	" " " " 4T, " "	189.2
16 - Mine A	" " 0.00% magnetite added" "	39.28
17 - " "	" " 0.01% " " "	40.66
18 - " "	" " 0.10% " " "	46.00
19 - " "	" " 0.50% " " "	58.24
20 - " "	" " 1.0% " " "	83.90
21 - " "	" " 2.00% " " "	129.90
22 - " "	" " 5.00% " " "	245.40
23 - Mine A	Processed chrysotile, 0.01% brucite added	41.86
24 - " "	" " 0.10% " "	41.49
25 - " "	" " 0.50% " "	42.14
26 - " "	" " 1.00% " "	42.60
27 - " "	" " 5.00% " "	43.42
28 -	Serpentine magnetically cleaned	12.51
29 - Mine A	Processed chrysotile 0.01% serpentine added	42.04
30 - " "	" " 0.10%	41.22
31 - " "	" " 0.50%	40.48
32 - " "	" " 1.00%	40.39
33 - " "	" " 5.00%	42.69
34 - Mine A	" " 1:1 mix, 0.01% serpentine + magnetite	40.10
35 - " "	" " 0.10% " "	42.40
36 - " "	" " 0.50% " "	48.60
37 - " "	" " 1.00% " "	60.80
38 - " "	" " 2.00% " "	78.50
39 - " "	" " 5.00% " "	137.0
40	Plastic cap - calibration at end	-

Processed Chrysotile

Samples 4 to 16 indicate the wide variation of magnetite content (one to five per cent) in the products from different Canadian mines and in the various grades from the same mine. Though processing in the asbestos mill removes some magnetite (samples 11 to 13; 9 and 10), it is apparent that a significant amount of magnetite is entrapped by the opened fibre and is not being removed. The cleaned, washed, and dried Canadian chrysotile (sample #16), which has the lowest bulk susceptibility, is used in our experiments as the basic constituent and to it are added known amounts of brucite, serpentine, and magnetite. This chrysotile has a susceptibility of 39.3 emu x 10^{-5}/g which is equivalent to 0.9 per cent magnetite; (43 x 10^{-5} emu/g is equivalent to one per cent magnetite, page 9).

Chrysotile-Brucite

Samples 23 and 27 consist of chrysotile with increasing amounts of brucite. They appear to show that additions of brucite do not contribute to the ferromagnetism of the mixture because the bulk susceptibility results are random. The variation of 2 x 10^{-5} emu significantly exceeds the noise level of the instrument which is 5 x 10^{-7} emu, but it is within the probable variation resulting from small variations in the amount of magnetite entrapped in the chrysotile of the different samples. Hence, this variation is attributed to sampling error.

Chrysotile-Serpentine

Samples 29 and 33 consist of chrysotile with increasing amounts of serpentine. As has been concluded for the chrysotile-brucite mixture, the random variation of 4.5 x 10^{-5} emu/g in susceptibility is attributed to sampling error. Additional experiments with greater control on the chrysotile are

required to define the ferromagnetic effects of brucite and serpentine contamination, however, they appear to be minimal in our mixtures.

Chrysotile-Magnetite

Samples 16 to 22 consist of chrysotile with increasing amounts of magnetite. There is a definite linear relationship between the susceptibility results (Table 1) and magnetite content (Figure 4). The calculations for the theoretical results in Table 2 were obtained as follows:

Sample 18:

Magnetite added = 0.10 per cent = 0.001 g
Weight of asbestos = 1 g - 0.001 g = 0.999 g
Magnetite present in asbestos (assuming 0.914 per cent/g of asbestos) = 0.913
Total magnetite present in mixture = 0.10 + 0.913 = 1.013 per cent
Susceptibility Constribution and Magnetite Content (one per cent magnetite = 43×10^{-5} emu/g)
0.10 per cent mag. = $0.10 \times 43 \times 10^{-5} = 4.30 \times 10^{-5}$ emu/g
0.913 per cent asbestos = $\frac{0.913}{0.914} \times 39.3 = 39.3 \times 10^{-5}$ emu/g
Total equivalent susceptibility = 43.6×10^{-5} emu/g
The observed susceptibility = 46×10^{-5} emu/g
The error (46 - 43.6) = 2.4×10^{-5} emu/g

The observed and calculated values of susceptibility vs magnetite content are in good agreement as shown in Figure 4. The error is relatively constant at about 2.4×10^{-5} emu/g which is equivalent to 0.05 per cent magnetite. This error is believed to be mostly due to sampling error and to a lesser extent to measurement error.

Samples 34 and 39 are mixtures of chrysotile, with magnetite and serpentine, and again there is a linear relationship between the susceptibility values (Table 1) which agree reasonably with the theoretical values (Table 2) and the magnetite content.

It is clear that the low-field susceptibility in all chrysotile mixtures can be ascribed almost entirely to magnetite ferromagnetism with nearly negligible chrysotile, brucite, and serpentine diamagnetism.

During this study, it was found that fibre orientation can result in axial susceptibility variations of up to a ratio of 2:1 which must be compensated to derive an accurate low-field bulk susceptibility. The measurement procedure adopted, i.e., six measurements on three mutually perpendicular axes, does compensate sufficiently for the potential source of error.

The slope of the linear relationship between per cent magnetite and susceptibility will vary depending on the effective magnetite domain size. The effective size depends on the original grain size, the subdivision resulting from exsolution textures of such minerals as ilmenite or from the production of alteration products such as rutile, and the subdivision produced by the milling process. The slope of the line is likely to remain nearly constant for a given mine and milling process. By relating this slope to a standard or theoretical slope determined from pure magnetite and/or salt mixtures such as used in this study or by Collinson et al (5), the absolute and/or effective magnetite contamination in the produced fibre may de determined. Further detailed mineralogic study of the ferromagnetic minerals could potentially lead to accurate determination of other contaminants such as serpentinite by comparing the low-field and saturated-field susceptibility measurements. A study of asbestos minerals using saturated-field susceptibility measurements will be reported on in the future.

Routine operation of the instrument, based on this study, showed that a single determination could be made in six minutes. This included packing, labelling, and weighing of the container and sample as well as recording the meter readings and making the sample calculations.

TABLE 2

A Comparison of Calculated vs Observed Values of Bulk Susceptibility

	1	2	3	4	5	6	7	8
Sample No.	Fe_3O_4 added Per Cent	Weight of Asbestos in Mixture grams	Fe_3O_4 present in Asbestos originally Per Cent	Susceptibility Contribution By:				Observed Fe_3O_4 $\frac{(7)}{43} \times 1$ Per Cent
				Fe_3O_4 added emu x 10^{-5}/g	Asbestos emu x 10^{-5}/g	Calculated (4 + 5) emu x 10^{-5}/g	Observed emu x 10^{-5}/g	
Chrysotile + Magnetite								
16	0.0	1	0.914	-	39.3	39.3	39.3	0.914
17	0.010	0.9999	0.914	0.43	39.3	39.7	40.66	0.947
18	0.100	0.9990	0.913	4.30	39.3	43.6	46.00	1.07
19	0.500	0.995	0.909	21.50	39.1	60.6	58.24	1.35
20	1.00	0.990	0.905	43.00	38.9	81.9	83.90	1.91
21	2.00	0.980	0.896	86.00	38.5	124.5	129.90	3.02
22	5.00	0.950	0.868	215.00	37.0	252.3	254.36	5.71
Chrysotile + Magnetite + Serpentine								
16					39.3	39.3	39.3	0.91
34	0.005	0.9999	0.914	0.215	39.3	39.5	40.1	0.93
35	0.050	0.9990	0.913	2.15	39.3	41.4	42.4	0.99
36	0.250	0.995	0.909	10.75	39.1	49.9	48.6	1.13
37	0.500	0.990	0.905	21.5	38.9	60.4	60.8	1.41
38	1.00	0.990	0.896	43.0	38.5	81.5	78.5	1.83
39	2.50	0.950	0.868	107.5	37.3	144.8	137.0	3.19

FIGURE 4 - MAGNETITE CONCENTRATION VS SUSCEPTIBILITY

Samples 16 to 22 (asbestos + magnetite)

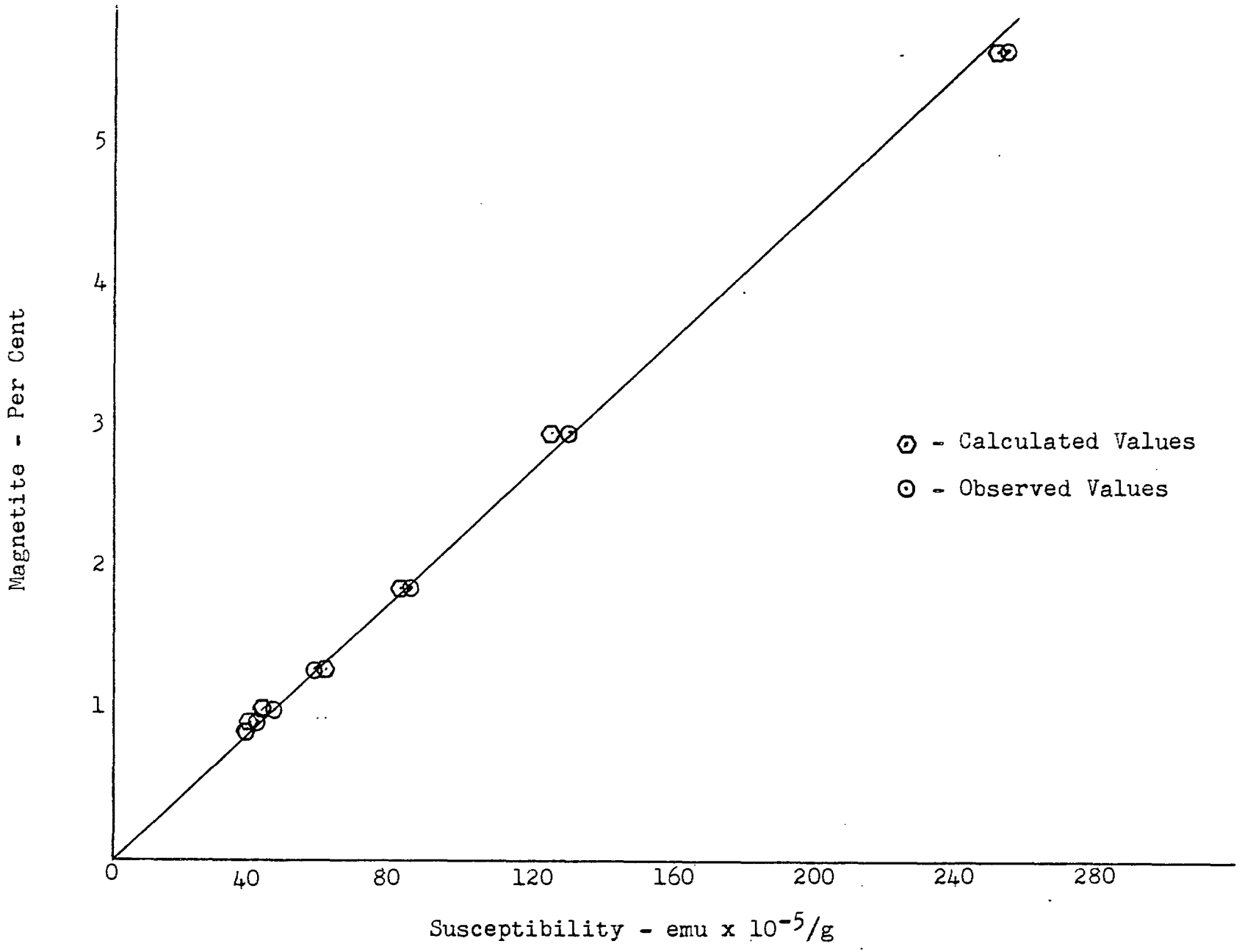
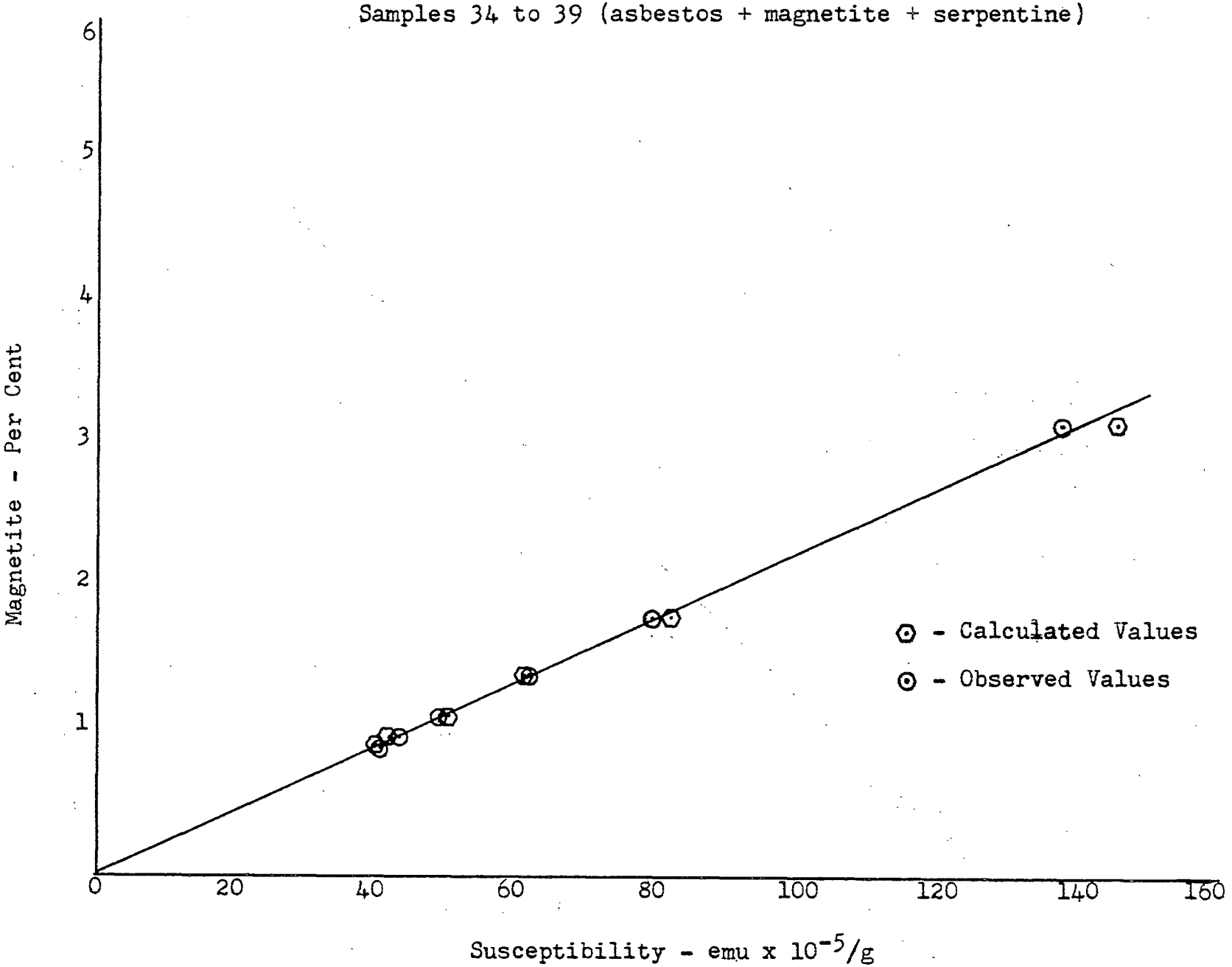


FIGURE 5 - MAGNETITE CONCENTRATION VS SUSCEPTIBILITY

Samples 34 to 39 (asbestos + magnetite + serpentine)



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