CHAPTER 3

EXPERIMENTAL RESULTS

3.1 Introduction

The apparatus which was described in the previous chapter are not the same as those used by F. Habbal, G.E. Watson, and P.R. Elliston[12] The circuit of the tunnel diode oscillator and its amplifier and the temperature measurement are quite different. Various magnetic material such as ferrite and ferrous oxalate ($FeC_2O_4.2H_2O$) were used to check if it was possible to obtain consistent and resonable results using our experimental apparatus. Since the magnetic susceptibility of cupric acetate monohydrate ($Cu(CH_3COO)_2H_2O$) has well known our measurement of it was used to obtain values for some of the parameters such as the filling factor, required in our method for measuring the magnetic susceptibility. Finally, the measurement of the susceptibility of cupric acetate monohydrate could also used to cross check the temperature measurements.

3.2 Results

3.2.1 Frequency Fluctuation

The resonant frequencies were set by adjusting the d.c. voltage of the oscillator. The variations of the frequency, have over a period of time been recorded. The fluctuations in the frequency while the coil was empty and while the sample was inserted into the coil are plotted. In Figure 19, we show the variation of frequency at room

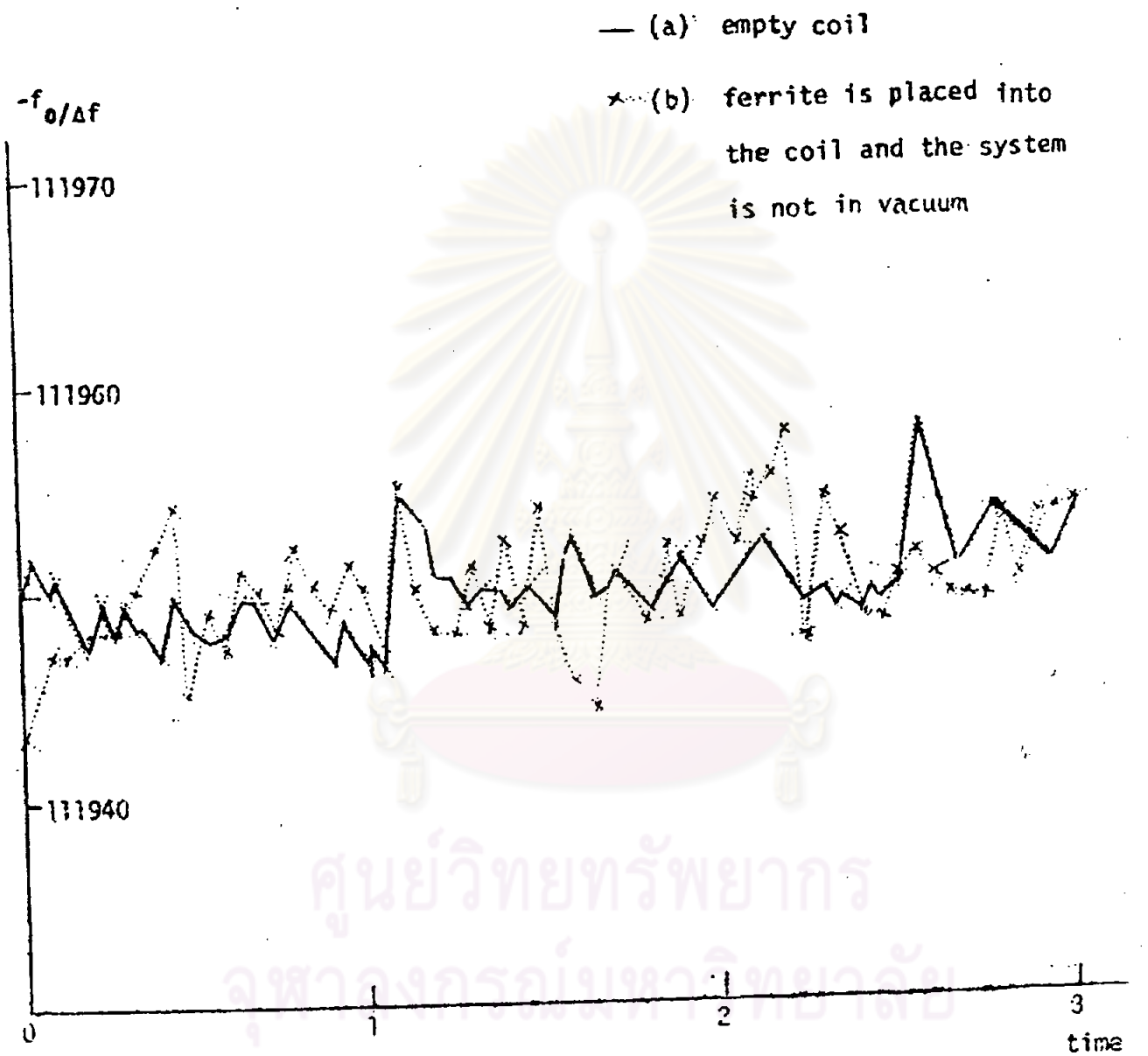

Figure 19 The variation of the frequency at room temperature

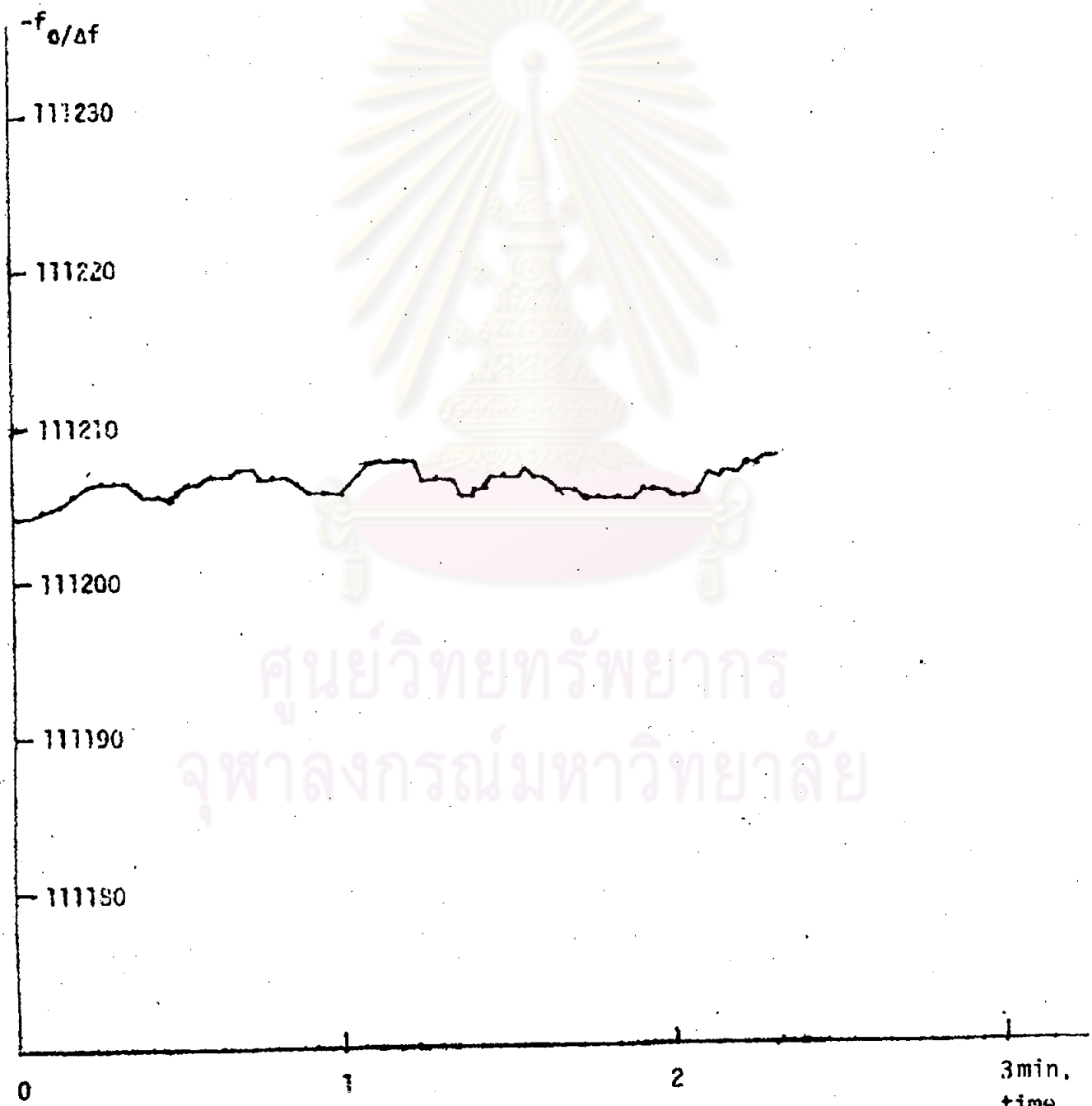

Figure 20 The fluctuation of the frequency of $FeC_2O_4 \cdot 2H_2O$

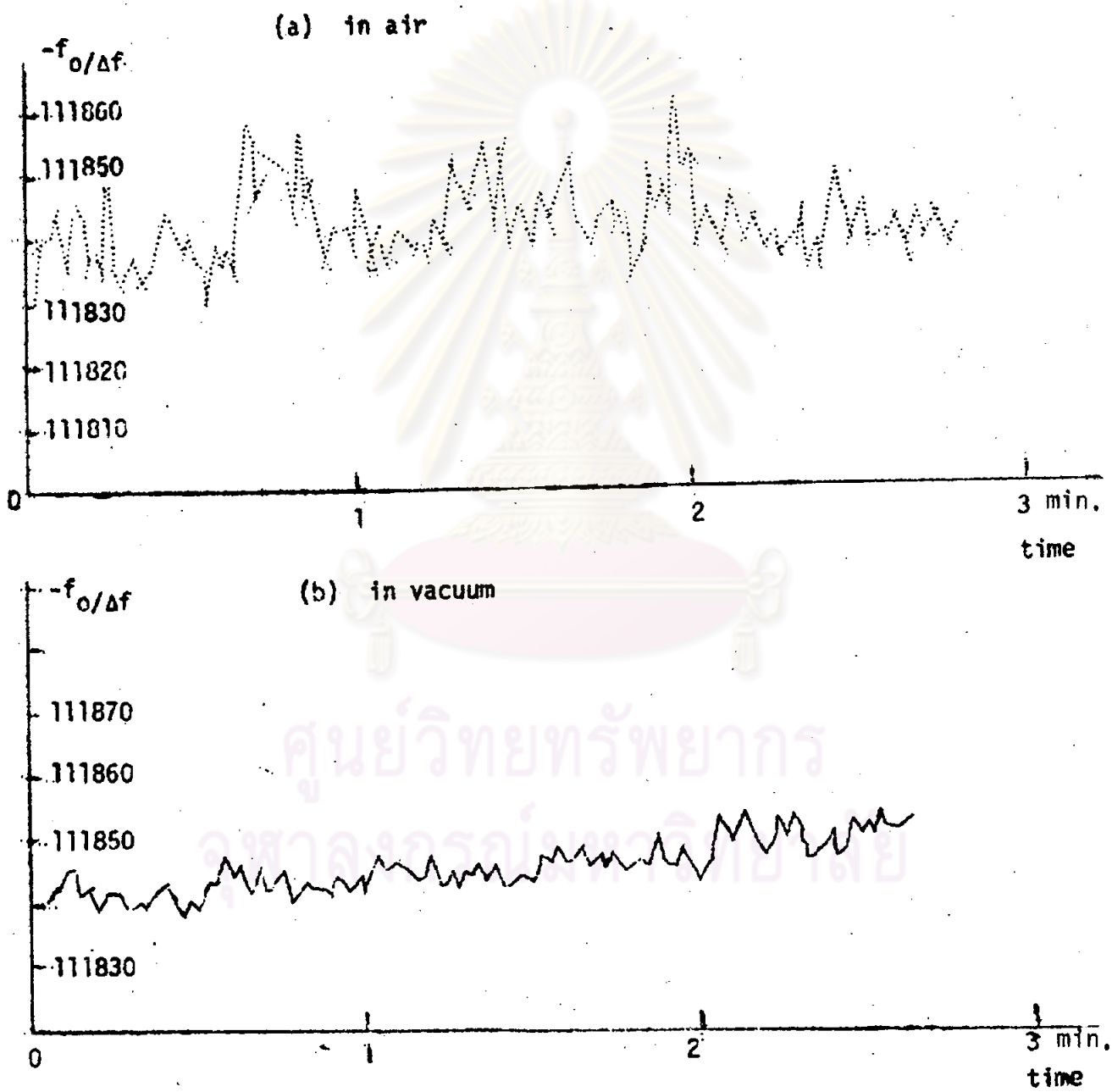

Figure 21 The fluctuation of the frequency of $FeC_2O_4 \cdot 2H_2O$

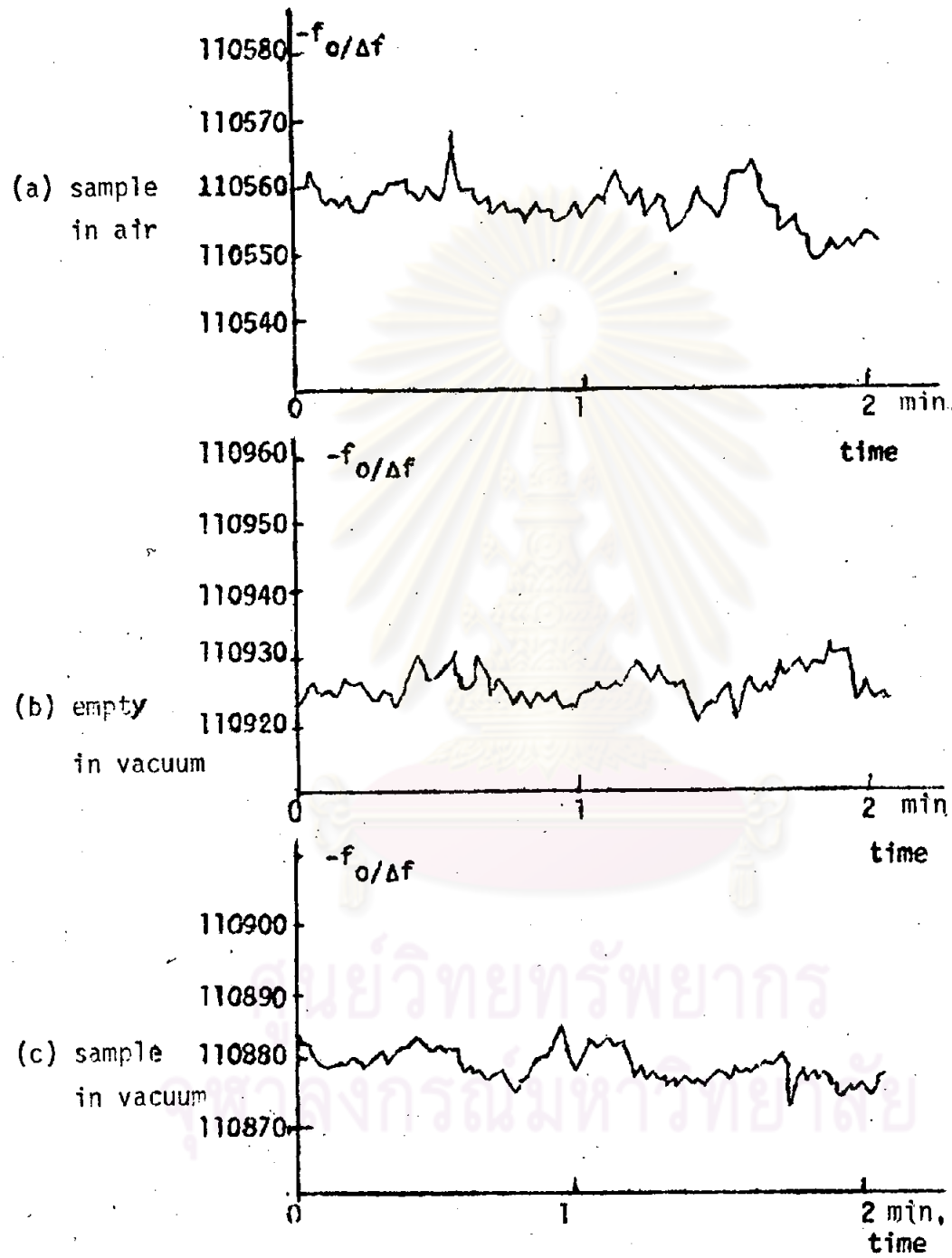

Figure 22 The fluctuation of the frequency of $Cu(CH_3COO)_2 \cdot H_2O$ at room temperature

temperature when the coil is empty(19a) and when ferrite is placed into the coil(19b). A small drift of the frequency is seen as time passes. However, this small drift does not affect in our results.The results shown in Figure 19 are for the unevacuated system, and Figure 20 shows the result when the system is evacuated. Figures 20,21,and 22 show the frequency fluctuations when ferrous oxalate and cupric acetate monohydrate are placed into the coil. A study of the fluctuations indicates a standard deviation of about 5 cycles per 100 kilocycles. These values indicate that apparatus is quite stable over a short period of time.

3.2.2. Temperature Measurement

A chromel-alumel thermocouple consists of a pair of electrical conductors joined together so as to produce a thermal e.m.f. when the two junctions are at different temperature. The e.m.f. of chromel-alumel thermocouple has been well known for the range 77 to 300K. The magnitude of the e.m.f. increased with the temperature difference has been recorded in Table I, and the variation is shown in Figure 23. As we see the e.m.f. of the chromel-alumel thermocouple changes rather uniformly in the range 77 to room temperature. In this range, the e.m.f. as a function of temperature, is given by the equation

$$E = At + Bt^2 + Ct^3 \qquad (3.1)$$

Our recorded, e.m.f. values reported in the Table I at various temperatures agree with the true values. This indicates that our temperature measurements are accurate enough for this experiment.

Table I

The variation of the EMF of Chromel-Alumel Thermo-couple is corresponding to the temperature.

Temp°C	E.M.F.(mV)	Temp°C	E.M.F.(mV)	Temp°C	E.M.F.(mV)
100	4.18(4.10)*	64	2.68(2.60)	28	1.13(1.12)
98	4.08(4.01)	62	2.58(2.51)	26	1.05(1.04)
96	4.00(3.93)	60	2.48(2.43)	24	0.96(0.96)
94	3.91(3.85)	58	2.40(2.35)	22	0.88(0.88)
92	3.83(3.76)	56	2.32(2.27)	20	0.81(0.80)
90	3.74(3.68)	54	2.22(2.18)	18	0.72(0.72)
88	3.67(3.60)	52	2.15(2.10)	16	0.66(0.64)
86	3.59(3.51)	50	2.05(2.05)	14	0.57(0.56)
84	3.49(3.43)	48	1.98(1.94)	12	0.49(0.48)
82	3.41(3.35)	46	1.89(1.85)	10	0.39(0.40)
80	3.31(3.26)	44	1.80(1.77)	8	0.33(0.32)
78	3.24(3.18)	42	1.72(1.69)	6	0.25(0.24)
76	3.18(3.10)	40	1.63(1.61)	4	0.17(0.16)
74	3.08(3.01)	38	1.55(1.53)	2	0.09(0.08)
72	3.00(2.93)	36	1.45(.144)	0	0.02(0.00)
70	2.90(2.85)	34	1.36(1.36)	-66	-2.40(-2.41)
68	2.81(2.76)	32	1.27(1.28)	-108	-3.70(-3.72)
66	2.73(2.68)	30	1.20(1.20)	-184	-5.50(-5.50)
				-196	-6.00(-)

Referent temperature at 0.2°C

* ( ) the value from the table reference.

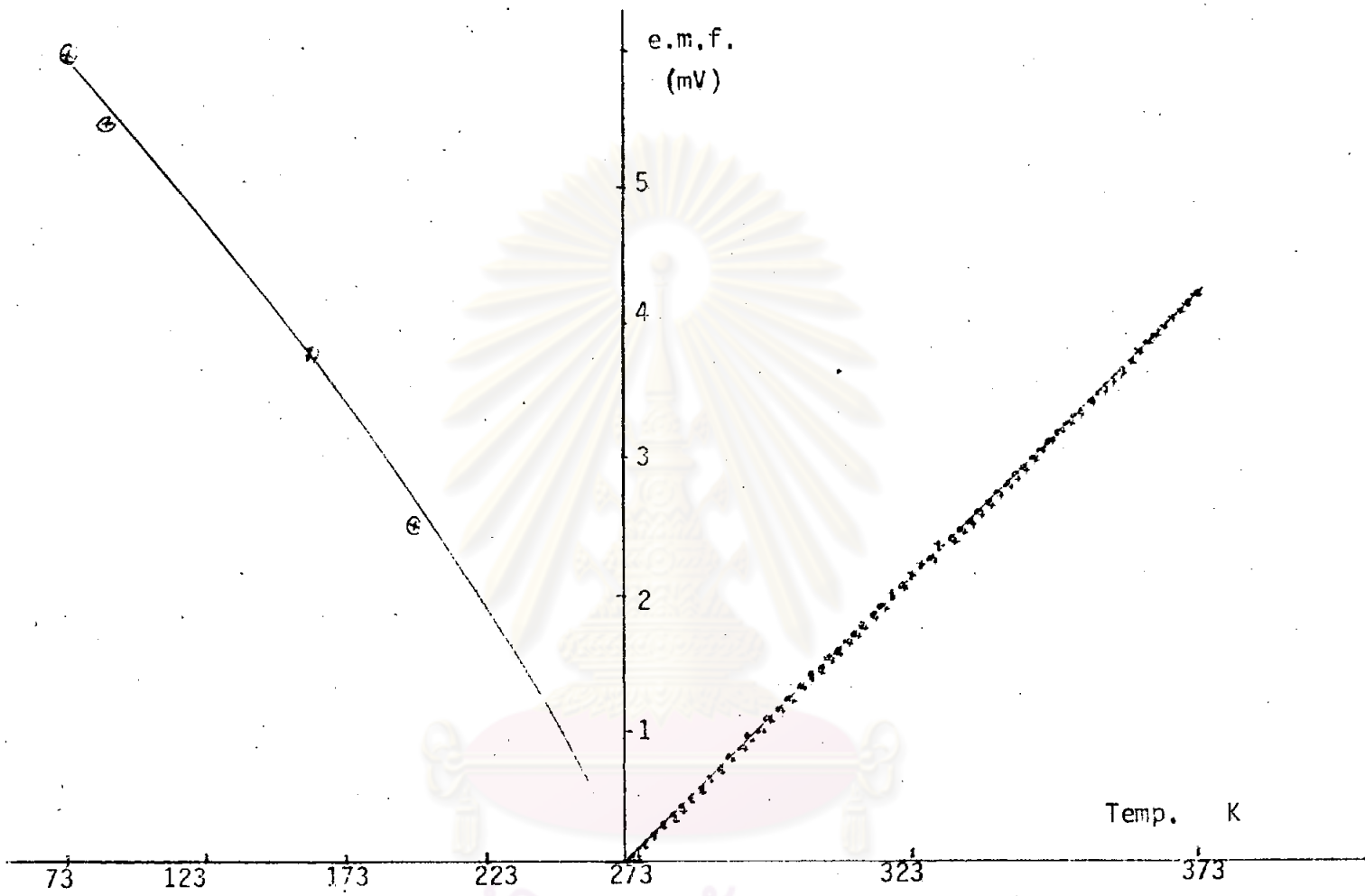

Figure 23 The thermal e.m.f. vary with the temperature

3.2.3 Ferrous Oxalate ($FeC_2O_4 \cdot 2H_2O$)

Ferrous oxalate in powder form which is in yellow colour has already been studied by others.[18] The magnetic susceptibility data obtained in the present measurements are shown in Table II. This compound shows an inverse susceptibility $\frac{1}{\chi}$ which increased slightly with T from room temperature down to 273K (0^oC). Since our temperatures did not go low enough, the maximum susceptibility could not be found for this sample. The dependence of susceptibility on temperature was determined and is shown on Figure 24.

3.2.4 Cupric Acetate Monohydrate ($Cu(CH_3COO)_2 \cdot H_2O$)

Crystalline cupric acetate monohydrate ($Cu(CH_3COO)_2 \cdot H_2O$) has the monoclinic prismatic crystals,[19] and is an example of antiferromagnetic substance. The magnetic susceptibility of this compound was studied by Bhatnagar, Singh, and Ghani,[20] subsequent investigations[21,22] confirm that the susceptibility of copper acetate at room temperature is anomalously low. The $Cu(CH_3.COO)_2 \cdot H_2O$ is transparent green or blue diamond-shaped plates. Grinding the crystals with an agate pestle and morter produces a powder which is of an opaque blue-green colour. The powder samples were then used in the magnetic measurements. The critical temperature T_C of hydrated copper is about 255K.[17] The relative magnetic susceptibilities of the sample at temperature between 77 to 300K were determined. The results show a well-defined maxima in the susceptibility

Table II

The temperature dependence of the inverse susceptibility of $FeC_2O_4 \cdot 2H_2O$ between 273K to room temperature

Temp. ^oC	Δf	$f_o/\Delta f$	Temp ^oC	Δf	$f_o/\Delta f$
2	2441	45	16	1879	59
4	2301	49	18	1705	65
6	2548	47	20	1385	80
8	2513	44	22	1421	76
10	2114	52	24	1304	85
12	2005	55	26	1167	95
14	1938	61			

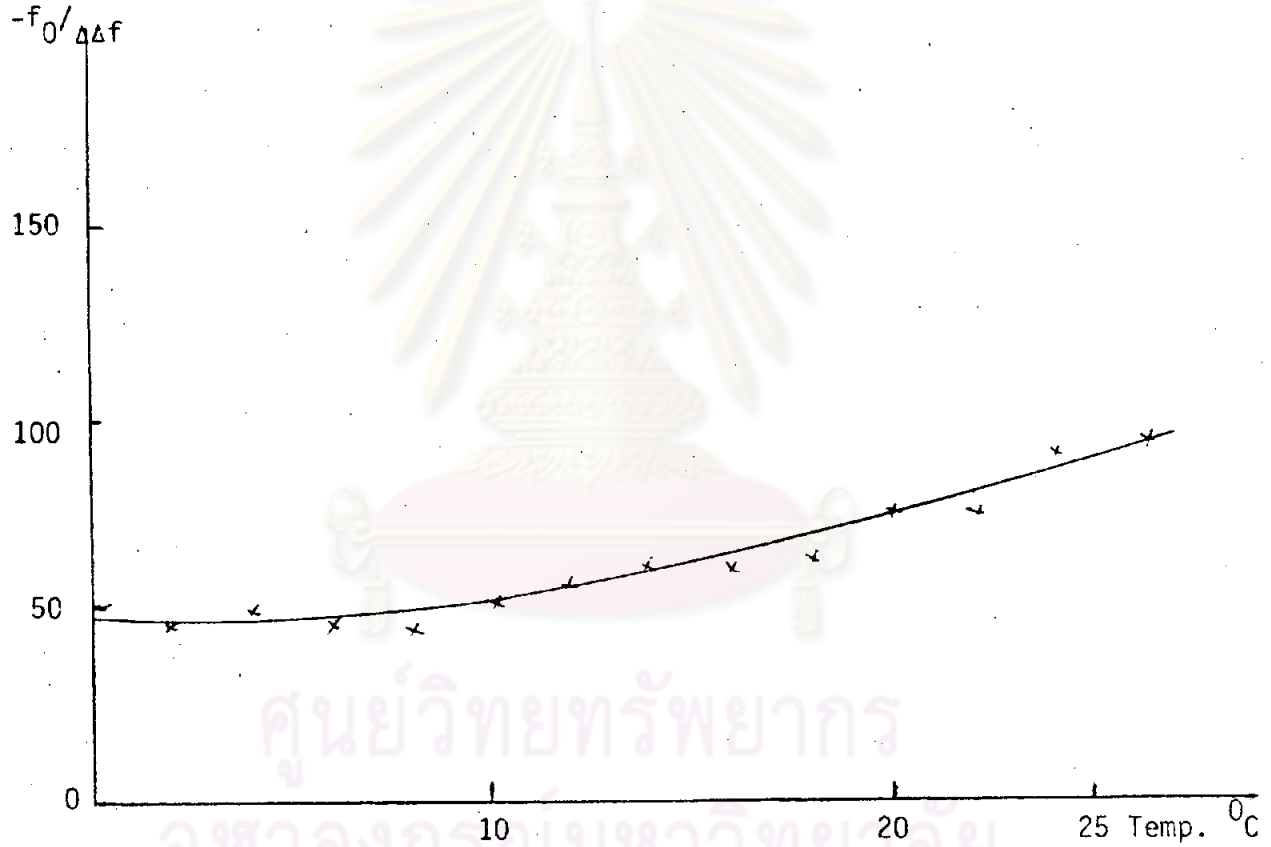

Figure 24 The temperature dependence of inverse susceptibility of $FeC_2O_3 \cdot 2H_2O$

curves at about 250K. The rough data is given in Table III, which the refined results are shown in Figure 25. The graph of the relative susceptibility shows that the inverse susceptibility vary with T. The susceptibility appears to obey some form of the expected Curie-Weiss law for an antiferromagnetic compound. At room temperature, in the paramagnetic region, the susceptibility increases as the temperature decreases. The inverse susceptibility appears to pass through maximum at around 250K. It then falls off rapidly below this temperature. Near room temperature, the susceptibility also decreased but at a lower rate. Our measurement shows the anomalous behavior of $Cu(CH_3COO)_2.H_2O$ which has been previously seen. According to our study, the curve in Figure 25 shows the temperature dependence of the inverse susceptibility of $Cu(CH_3COO)_2 \cdot H_2O$ and exhibits the maximum susceptibility at 250K. Thus the Néel temperature which is the critical temperature at which the susceptibility is maximum is 250K. The Curie-Weiss equation (1.44) is used to calculate the Curie's constant, by substituting for χ and T in the equation, the values of the maximum T in the equation, the values of the maximum susceptibility χ_m and the temperature at which it occurs T_m the values at temperature found on Figure 25, i.e.

$$\frac{1}{\chi} = \frac{T - \theta}{C} \qquad (3.2)$$

at $T_m = 250K$, $\frac{1}{\chi_m} = 870$

and $T_r = 300K$, $\frac{1}{\chi_m} = 1705$

From equation (3.2), the Curie constant C is calculated to 0.059 and the Curie temperature Θ to be 198 ± 5K.

The experimental results are used to determine two correction one due to the apparatus and the other sample. The secondary filling factor, f, which is the correction due to the density of the sample is approximately the ratio of the density of the sample to the density of the crystalline form[12]. The density of the powder sample was 0.454, and of the crystal form was 1.545. Thus we have

$$f = \frac{d_s}{d} = \frac{0.454}{1.545}$$

$$= 0.29$$

The next correction factor is the geometrical filling factor, F, which is the ratio of the volume of the sample to the volume of the coil. In Figure 26, the temperature dependence of the inverse susceptibility at temperature between 273K to 300K is a straight line since the sample is in the paramagnetic region. From equation (2.22), we see that the slope of the temperature dependence line is given

$$s = \frac{1}{2\pi FCf}$$

Looking at Figure 26, we find the slope to be 0.05×10^3. The filling factor F can be calculated if the Curie constant and the filling factor f are known. Substituting the measured values of C = 0.059

and f = 0.29, we find the filling factor of our apparatus to be F = 0.186. Using equation (2.17), with F = 0.186, we have determined the measured susceptibility as being

$$\chi_m = -\frac{1}{2\pi}\frac{\Delta\omega_m}{\omega_o}\cdot\frac{1}{F}$$

$$= -\frac{f_o}{\Delta f}\cdot\frac{1}{F}$$

$$= -\frac{900}{0.186} = -4838$$

Finally, the intercept of the graph in Figure 26 which is equal to γCf(2.22), we have calculated the molecular field constant, i.e.,

$$\gamma = \frac{\Delta}{Cf}$$

$$= \frac{650}{0.059 \times 0.29} = 37 \times 10^3$$

Some of the parameters for cupric acetate monohydrate are listed in Table V.

Table III

The temperature dependence of inverse susceptibility of $Cu(CH_3COO)_2 \cdot H_2O$ between 77K to room temperature

Temperature	$-f_o/\Delta f$	mean	Standard deviation
liquid N_2	9872, 9899	9904	45
6 m.V.	9902, 9857		
(77K)	9909, 9987		
Vapour N_2	2509, 2507	2485	28
5.5 m.V.	2487, 2459		
(89K)	2442, 2504		
dry ice &	1959, 2005	1966	36
Actone 3.7 mV	1989, 1995		
(165K)	1915, 1932		
dry ice	1195, 1219	1185	29
(2.4 mV)	1145, 1152		
(207K)	1201, 1197		
ice 0.25 mV	1011, 1079	1055	37
($\sim 0.2^oC$)	1111, 1025		
($\sim$ 273K)	1065, 1040		
Room Temp.	1701, 1712	1705	9
1.05 mV	1698, 1714		
(300K)	1692, 1713		

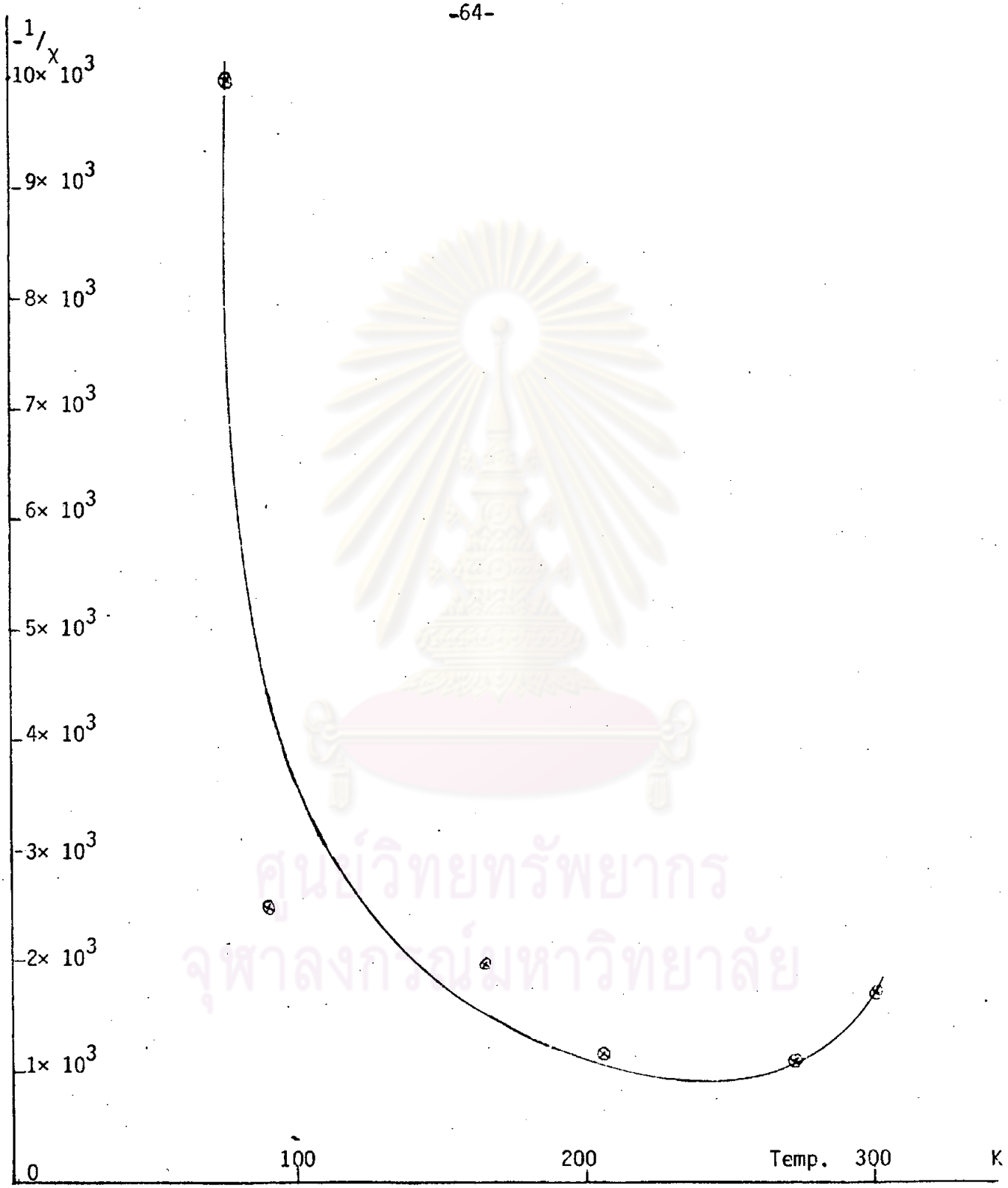

Figure 25 The temperature dependence of the inverse susceptibility of $Cu(CH_3COO)_2 \cdot H_2O$

Table IV

The temperature dependence of the inverse susceptibility of $Cu(CH_3COO)_2 \cdot H_2O$ between 273K to room temperature.

Temp. ^oC	$f_{o/\Delta f}$
0	998
2	972
3	829
5	1001
10	984
13	1275
15	1134
17	1790
18	1801
20	1565
22	2010
23	1956
25	1882
27	1950
28	2210
30	2840

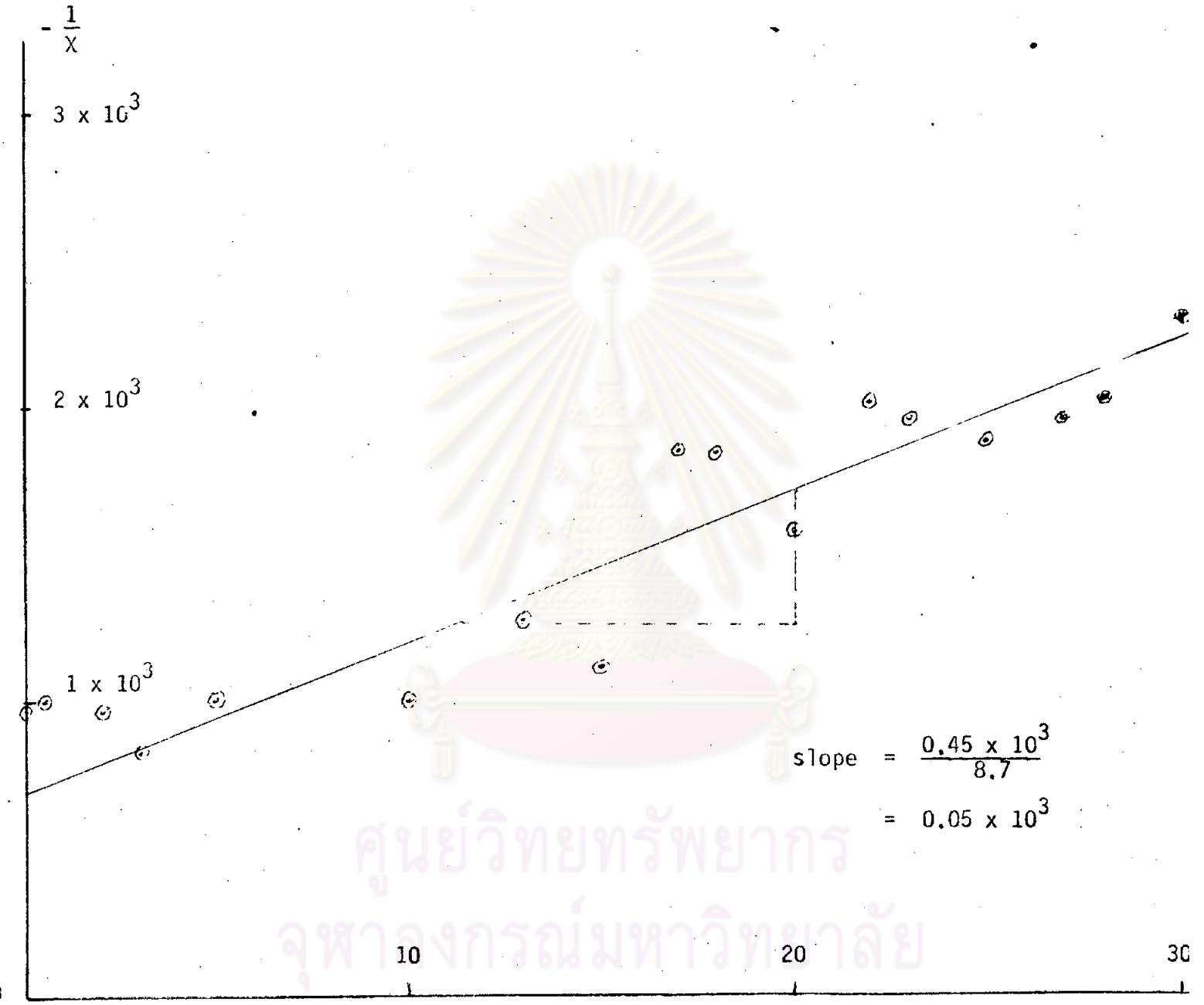

Figure. 26 The temperature dependence of the inverse susceptibility of $Cu(CH_3COO)_2 . H_2O$ between 273 K to room temperature

3.3 Conclusion

An apparatus has been built for the measurement of the magnetic susceptibility as a function of temperature over the temperature range between 77K to room temperature. The circuit of a tunnel diode oscillator to insure good stability is given. The magnetic susceptibility of various materials have been studied. Ferrite and ferrous oxalate were used to calibrate the apparatus. Cupric acetate monohydrate was studied in detail in order that the filling factor of the apparatus could be obtained. The susceptibility at some temperatures are different from those reported in the literature[17]. Most of the results obtained are however in agreement with the ones reported in the literature. The experimental susceptibility of copper acetate is anomalous low at room temperature in keeping with that reported in the literature[17]. Figgis and Martin reported that this behavior arises from some form of strong coupling to the crystalline field. The magnetic properties at very low temperature can not be studied by the present system because of the lack of liquid helium.

Table V

Material	m_s(g)	d(g/cm^3)	f	C(K)	θ(K)	Tm(K)	Magnetic Ion	Basic Level
$Cu(CH_3COO)_2.H_2O$	0.3698	1.545	0.29	0.059	198±5	250±5	Cu^{++}	$^2D_{5/2}$

m_s = mass of sample

f = second filling factor = $\frac{d_s}{d}$ = 0.29

d_s = density of the sample