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MAGNETIC SUSCEPTIBILITY MEASUREMENT WITH A DUPONT'S THERMOGRAVIMETRIC ANALYSER

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The adaptation of a Du Pont Thermogravimetric Analyser for the measurement of magnetic susceptibility as a function of temperature in the range −80 °C to 300 °C is described.

For the measurement of magnetic susceptibility, X, the Gouy method has been employed [1, 2]. In this method a cylindrical sample is suspended in such a way that one end of the sample is in a region of strong uniform field and the other end in a region of negligible field.

Experimental

The equipment modified for this work was a Du Pont TGA, Model 951. This thermobalance has been described by various authors [3]; accordingly we show only (Figs 1 and 2) the modifications made to the envelope of the balance arm.

Figure 1 shows an upper view of the modifications made to the balance arm quartz envelope, where the electromagnets were placed, and how the Hall probe of a gaussmeter was placed to measure the magnetic field prior to the experiment. The configuration shown in Figure 1 was to measure X at ambient and subambient

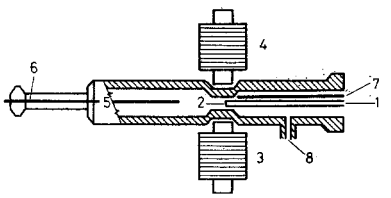

Fig. 1 Modified quartz envelope. 1) Quartz balance arm. 2) Sample position. 3)&4) Electromagnets. 5) Quartz envelope. 6) Gaussmeter Hall probe. 7) Sample thermocouple. 8) N_2 flow

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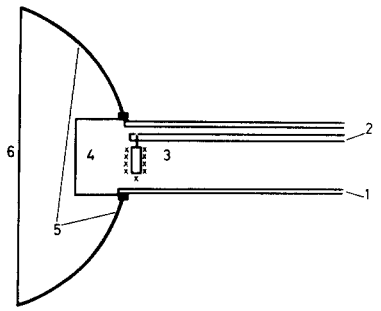

Fig. 2 Modified quartz envelope for radiation oven. (Drawing not to scale.) 1) Quartz envelope. 2) Quartz balance arm. 3) Magnetic field & sample tube. 4) Crucible, 5) Aluminized mirror surface. 6) Heating plate

temperatures. The quartz beam remains the same and the sample pan is substituted by a microtube, sealed at one end. For subambient temperature measurements, a flow of nitrogen, from a liquid nitrogen Dewar container, was introduced into the envelope, through the added tube (8 in Fig. 1).

Electromagnets

The electromagnets have cylindrical poles with a cross-section diameter of 1 cm and 12 cm long. The pole magnets can be changed very easily to try different materials. If iron poles are used, some hysteresis is present, but which such a small size it is very simple to interchange the pole positions and apply a field of sufficient magnitude to restore the poles to their initial conditions.

The power supply for the coils is a variable DC voltage (0–20 volt) and a maximum current of 25 ampere.

Magnetic field measurements were made by means of a Hall gaussmeter (Ealing Corp. Model 30–6217) with a long thin probe (Ealing 30–6233) that can easily be introduced into the arm envelope, so that direct measurements of the field can be made at site where the sample tube is placed.

Measurements above ambient temperature

Usually, the available X measurement balances include electrical heating jackets that are assumed not to alter the magnetic field. To avoid this, we decided to design a radiation furnace, following the work of Riveros and Rosenberg [4]. Figure 2 shows the modifications made to the balance arm envelope.

The temperature and weight (apparent) variations are directly plotted on the recorder of the 990 Du Pont Thermal Analyser as a function of time or temperature.

Sources of error

The main possible error is for powdered samples, due to inhomogeneous packing. This can be avoided if an electrical vibrator is used to pack the samples, with repetition of measurements until a constant value of X is obtained.

Experimental results

The following Table shows the experimental values obtained

Compound	$X \times 10^{-6}$ cgs units		T, °C
	obtained	reported*	
$HgCo(NCS)_4$	16.43	16.44	23
$CuSO_4 \cdot 5H_2O$	5.852	5.860	23
$CuSO_4 \cdot 5H_2O$	6.622	6.632	100
$TiCl_3$	1112.2	1112.0	23
$TiCl_3$	688.96	690.0	−60

X_g or X_m used indifferently for comparison with reported values.
* Handbook of Chem. & Phys., Chem. Rubber Co. Pub. Magnetochemistry, B.N. Figgis.

Conclusions

a) The results obtained are in good agreement with reported values.

b) Very small samples are needed (of the order of 1 mg).

c) It is possible to have the sample in any non-corrosive atmosphere. Corrections for buoyancy are negligible.

d) The temperature range is good.

e) Due to the dimensions involved, it is very simple to change poles and try another method such as Faraday's.

f) The system presents a different heating method. There is no alteration of the magnetic field.

References

1 P. W. Selwood, Magnetochemistry, Interscience Publ., New York, 1956, p. 279.
2 P. W. Selwood, Adsorption and Collective Paramagnetism, Academic Press, New York, 1962, p. 10.
3 W. W. M. Wendlandt, Thermal Methods of analysis, John Wiley & Sons, New York, 2nd Edition, p. 85 et seq.
4 H. G. Riveros, F. Rosenberg et al., Computational Design of a High Efficiency Melting Furnace, J. of Crystal Growth, 53 (1981).

Zusammenfassung — Die Adaptation des Du Pont's Thermogravimetric Analyzer zur Messung der magnetischen Suszeptibilität in Abhängigkeit von der Temperatur im Bereich von −80° bis 300 °C wird mitgeteilt.

Резюме — Показана адаптация термогравиметрического анализатора фирмы Дюпон для измерения магнитной восприимчивости в интервале температур −80°–300 °C.