[Cr(acac)$_3$] as a New Calibrant for Magnetic Susceptibility Experiments

R. P. SHARMA* and K. K. BHASIN
Department of Chemistry, Panjab University, Chandigarh-160014, India
(Received August 12, 1985)

Synopsis. The merits of a common coordination compound, [Cr(acac)$_3$] obtained by a new and useful method, as a calibrant for magnetic susceptibility experiments are discussed.

Recently, we have described a new and useful method[1] for the preparation of a well documented coordination compound; tris(acetylacetonato)chromium(III), [Cr(acac)$_3$], in high yields by adding acetylacetone to a warm aqueous solution of $M_2Cr_2O_7$ ($M=NH_4^+$, Na^+, K^+) or M_2CrO_4 ($M=Na^+$, K^+). The purity of the sample has been checked by elemental analysis (C, H, Cr), melting point and infrared spectrum.

The advantages of our method over the already existing ones[2-4] are (i) the starting materials are readily available (ii) the method is quick (iii) the final product is purer and (iv) the yield of the product is not pH dependent.

One of the most important techniques used in coordination chemistry to find out the number of unpaired electrons in the transition metal ion so as to throw light on the environment around the central metal ion in a coordination compound is the magnetic susceptibility.[5] The first step to find out the magnetic susceptibility always involves the calibration of the Gouy's tube with a standard substance known as calibrant. A calibrant is selected so to fulfil the following requirements:

(i) The calibrant should be easy to prepare and the starting materials cheaply and readily available.
(ii) The method for its preparation should be simple and quick.
(iii) The calibrant should be of high purity and if need be, easy to purify.
(iv) The calibrant should be nonhygroscopic and fairly stable, i.e., it should possess good keeping properties.
(v) The magnetic moment value should be fairly large.

As it was relatively difficult earlier to prepare pure [Cr(acac)$_3$] (which may be usually contaminated with the buffer used) it was not thought as suitable for calibration purposes. The most commonly used calibrant[6-8] has always remained Hg[Co(SCN)$_4$] so far. In view of our convenient method for the preparation of [Cr(acac)$_3$] coupled with other advantages over Hg[Co(SCN)$_4$] (Table 1), the use of [Cr(acac)$_3$] as a calibrant is strongly recommended.

References

1) R. P. Sharma and K. K. Bhasin, *Polyhedron*, in Press (1985).
2) W. C. Fernelius and J. E. Blanch, *Inorg. Synth.*, **1957**, 130.
3) G. Brauer, "Handbook of Preparative Inorganic

Table 1. Comparison of the Physical Characteristics of Hg[Co(SCN)$_4$] and [Cr(acac)$_3$]

	Property	Hg[Co(SCN)$_4$]	[Cr(acac)$_3$]
1.	Color and physical state	Deep blue crystalline compound	Reddish violet crystalline compound
2.	Nature	Polymeric[a]	Monomeric[b]
3.	mp/bp (°C)	.....	216°/340°[c,d]
4.	Density(g/ml)	.....	1.37
5.	Solubility	Insoluble in conventional organic solvents	Soluble in conventional organic solvents
6.	Preparation and purity	Easy to prepare but difficult to purify	Easier to prepare as well as to purify
7.	Stability	Stable	Exceptionally stable
8.	Toxicity	Toxic	Nontoxic
9.	Number of unpaired electrons	3	3
10.	Magnetic moments(B. M.) (observed at 300 °K)	4.33[e]	3.86,[f] 3,88[g]
11.	Gram susceptibility	16.44×10^{-6}[e,h]	18.07×10^{-6}[f]
12.	Weiss constant	+2 °K[h]	+7 °K[f]

a) J. W. Jeffery, *Nature*, **159**, 610 (1947); J. W. Jeffery, *Acta Cryst. Supp.*, **6A**, 66 (1963). b) B. Morosin, *Acta Cryst.*, **19**, 131 (1965). c) G. Urbain and A. Debierne, *Compt. Rend.*, **129**, 302 (1899). d) J. Von. Hene, R. G. Charles, and W. M. Hickam., *J. Phys. Chem.*, **62**, 1098 (1958). e) B. N. Figgis., *Trans. Faraday Soc.*, **56**, 1553 (1960). f) B. N. Figgis., J. Lewis, and F. E. Mabbs, *J. Chem. Soc.*, **1961**, 3138. g) Present work. h) H.-St. Rade, *J. Phys. Chem.*, **77**, 424 (1973).

Chemistry," Academic Press, New York (1965) Vol. 2, p. 1384.
4) H. Ssekaalo and S. O. Wandiga, *J. Inorg. Nucl. Chem.*, **39**, 769 (1977).
5) B. N. Figgis and J. Lewis, *Progr. Inorg. Chem*, **6**, 37 (1964).
6) B. N. Figgis and R. S. Nyholm, *J. Chem. Soc.*, **1958**, 4190.
7) B. N. Figgis and R. S. Nyholm, *J. Chem. Soc*, **1959**, 338.
8) B. N. Figgis, *Trans. Faraday Soc*, **56**, 1553 (1960).