AD-A183 406 PHYSICAL THERMAL AND OPTICAL CHARACTERIZATION OF 1/1
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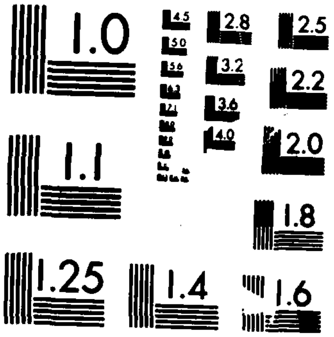

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Physical, Thermal and Optical Characterization of Rhodium(III) Acetylacetonate

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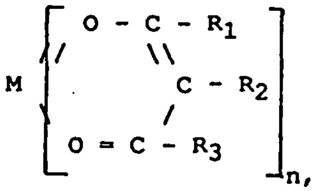
Abstract

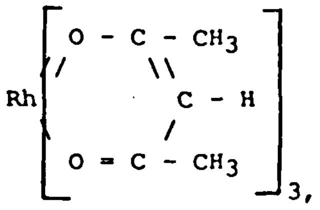
Rhodium(III) acetylacetonate was investigated for its physical, thermal and optical properties. Ultraviolet-visible absorption spectra show absorptions at 320 nm, 260 nm, and 210 nm. Density measurements yielded a value of 1.75 g/cm^3. Thermal characteristics were evaluated using differential thermal analysis(DTA). It was found that the compound is not volatile, decomposing upon heating. If heating rates are rapid enough, e.g. ≥ 2 °C/min, melting at 267 °C can be observed. If heating rates are slower, decomposition is complete below the melting point. If the compound is annealed at 267 °C for four hours decomposition is complete, yielding 99% pure rhodium metal. In an oxidizing atmosphere, on the other hand, the compound decomposes into a product containing 75% rhodium metal which appears to be RhO_2.

Introduction

As circuit dimensions continue to decrease in very large scale integration(VLSI) and ultra large scale integration(ULSI) designs, it has become increasingly important to look at alternative methods for thin film formation. This is due to 1) costly lithographic processes, and 2) the limitations of processing at high temperature due to the allowed thermal budget for device fabrication[1]. Studies of thin film preparation alternatives have prompted examination of the physical, thermal and optical properties of a series of metal organic compounds which have received limited characterization in the literature. Present research is currently involved with a series of metal organic compounds called ß-diketonates. These compounds have the general formula :



where M is the metal in a +n oxidation state, and R_1, R_2, and R_3 are carbon groups or hydrogen. An example of such a compound is rhodium(III) acetylacetonate(acac):



To varying degrees, these compounds possess desirable properties which make them potentially viable alternatives to the conventional costly metal lithographic, or insulator processes currently used in microelectronics. These properties are 1) most of them decompose at low temperatures(as low as 150-200 °C); 2) they are easily handled solids at room temperature; 3) some may be photoactive; 4) they are soluble in a wide variety of solvents and 5) some of them exhibit appreciable vapor pressures at temperatures below that at which decomposition is significant, whereas in others the decomposition products, other than the metal, are volatile. Those of interest for metal film formation may be attractive candidates for metal interconnection pattern repair for which there is no usable technology(metal organics have been used in metal lithographic mask repair[2]).

Ideally, a solution of a compound could be applied to a substrate, and then the resulting film either photolytically and/or thermally decomposed to produce a metal or an insulating material in a pattern. The undecomposed film could then be either 1) volatilized at low temperature, if the original compound possesses appreciable vapor pressure, leaving the remaining decomposed film or, 2) if the original compound is not volatile, but tends to decompose into fragments, the original compound could be redissolved in a solvent leaving the decomposed material behind. Other sequences are equally obvious, depending on the exact physical properties of the starting compound.

A survey of the literature revealed that few of the basic properties of rhodium(III) acetylacetonate had been fully characterized. This prompted the study of the thermal and photolytic properties, as well as the density of this compound. These are necessary parameters for the present thin film formation studies.

Thermogravimetric analysis(TGA)[3] as well as differential scanning calorimetry(DSC)[4] have been performed on selected acetylacetonates. Kettrup[5] applied TGA-DTA-mass spectroscopy to a series of divalent metal acetylacetonates(Cd, Fe, Co, Cu, Mn, Ni, Zn). Yoshida[6] has also applied DTA on selected divalent metal chelates(Cu, Be, Pd, Co, Ni). Charles[7] has studied the thermal gaseous decomposition products of several acetylacetonates with a mass spectrometer(Na, Cr, Al, Ni, Cu, Fe, Co, Mn). No attempts have been made to study the solid residue(s) remaining after an apparent themal anomaly is observed. In general, the heating rates applied were high(> 10 ^{o}C/min) which do not allow the system under study to obtain proper thermal equilibrium. None of the papers have dealt with the thermal decompositon of rhodium(III) acetylacetonate.

Several articles also deal with spectral absorption investigations of the metal ß-diketonates(Ba, Al, Ga, In, Zr, Th, Zn, Cd, Sc, Y, La, V, Mn, Co, Ni, Fe, Cu, Cr) [8-11]. DeArmond[12] has studied the phosphorescence and absorption of $Rh(acac)_3$ under conditions which were not defined precisely.

EXPERIMENTAL

A. Thermal Investigations

Thermal decomposition was studied with a differential thermal analyzer(DTA) designed by one of the authors and described previously[13]. Minor modifications were made to the assembly to assure the sample and reference materials were centered in the furnace, as shown in Fig. 1. These modifications were 1) squeeze clamps to assure the thermocouple (t.c.) rods would remain stationary and 2) an alumina block for the crucibles to rest upon. A furnace temperature profile was performed to position the samples in the hottest part of the furnace. The crucibles were made of quartz 4" long with a 7 mm inner diameter, and a 1/4" insert at the bottom to serve as a thermocouple well which insures that the t.c. is completely surrounded by the sample.

Samples were compared with a reference which consisted of a closed crucible of aluminium oxide, Al_2O_3(Fisher Sci. Co.,Raleigh Branch Office, P.O. Box 40339, Raleigh, N.C. 27629) at a heating rate of 2 °C/min, or less, under an argon or oxygen(Airco Industrial Gases, P.O. Box 12338 Research Triangle Park, N.C., 27709) flow rate of 0.1 slpm. Sample size ranged from 0.15-0.20 g. Data were plotted on an X-Y recorder(Houston Instruments, P.O. Box 15720 Austin, Tx., 78761). The X-sensitivity (differential signal) was 5 μV/in. and the Y-sensitivity was either 1mV/inch or 0.5mV/inch. Thermocouple wire used was platinum and platinum ten percent rhodium.

B. Photolytic Studies

Ultraviolet and visible spectra were obtained using a Cary 2300 Ultraviolet-visible(UV-VIS) spectrometer(Varian Associates, Instruments Group, 611 Hansen Way Box D-070, Palo Alto, Ca., 94303). Semiconductor grade methanol supplied by Allied Chemicals, 1599 Little John Road Parsidany, N.J. 07054) was used as solvent. Solutions were prepared at 10^{-5} M for UV and 10^{-2} M for the visible spectrum. No absorption was observed in the range 400 nm to 700 nm.

C. Density

A 5ml picnometer was used to measure density. It was calibrated with deionized water at 25 °C. Sample size of $Rh(acac)_3$ was ca. 0.3g, and measurements were made in paraffin oil(Fisher Sci. Co. Raleigh Office 3315 Winton Road, P.O. Box 40339 Raleigh , N.C. 27629). Density of the paraffin oil was determined prior to use. It was found to be 0.950 g/cm^3.

D. X-rays

Debye-Scherrer powder diffraction studies were performed on a Norelco diffractometer (type no. 12044, North American Phillips Co., 85 McKee Dr. Mahwah, N.J. 07430) using Cu K-α radiation.

E. Electron Probe

Electron probe studies were performed on a Phillips/AMR (North America Phillips Co.).

F. Material

$Rh(acac)_3$ was purchased as a light orange crystalline powder from Strem Chemicals Inc.(7 Mulliken Way, Newburyport, Ma. 01950), and was analyzed for purity by DC plasma emission spectroscopy. Since some acetylacetonates tend to hydrate, the material was analyzed also in terms of stoichiometry. The rhodium content of anhydrous $Rh(acac)_3$ should be 25.4 %. It was found to be 25.4 %. This result was also verified in a vacuum dehydration experiment over sulfuric acid for several days.

EXPERIMENTAL RESULTS AND DISCUSSION

A. Differential Thermal Analysis

DTA was carried out in either open or closed crucibles, in order to observe decomposition of the compound under its own vapor pressure or atmospheric pressure. The open crucible samples were heated in either an inert or an oxidizing ambient atmosphere to observe melting and decomposition behavior.

1. Open Crucible DTA in Argon

A heating trace taken at 2 °C/min of the rhodium compound in an open crucible in an argon atmosphere exhibits two endothermic peaks, as shown in Fig. 2. The first peak at 267 °C was found to be due to the melting of the compound. Upon completion of the peak, as determined by DTA, the sample was withdrawn rapidly from the DTA chamber. The sample was seen to be molten with a deep orange color. At the heating rate mentioned, the sample losses 6% of its weight. Since this does not correspond to the loss of a whole ligand molecule, the question arose as to whether decomposition commenced before the melting point, the results being due to a kinetic rather than a thermodynamic or steady state behavior of the material. For example, if decomposition begins below the melting point but occurs at a slow rate compared to the rate of heating, one could observe the melting point even though under more steady state conditions, decomposition would be complete before the melting point temperature was achieved. When a heating rate of 0.1 °C/min was employed, no peaks were observed, and it was found that decomposition was complete below the melting point. Thus decomposition in $Rh(acac)_3$ is unrelated to melting. The second endotherm, in Fig. 2, appears to represent rapid loss of the organic ligand since there is a weight loss of 75% which accounts for the three acac groups. Obviously, even at 2 °C/min the decomposition temperature detected is simply that at which the rate is very high. The decomposition kinetics should follow Arrhenius behavior. To test this, a sample was heated to 100 °C in argon and held at the temperature for four hours. No weight loss was observed. However at 184 °C under similar conditions, weight loss was observed, indicating decomposition was already measurable. Electron probe measurements indicated a rhodium residue of at least 99.9 % purity, following temperature elevation to 267 °C.

Thus, we can conclude from the annealing studies in an inert gas ambient atmosphere that significant decomposition begins to occur at 184 °C. If the sample is heated at 2 °C/min the decomposition is preceded by a melting phenomena and followed by a rapid loss of the organic ligand.

2. Open Crucible DTA in Oxygen

The heating and decomposition of the rhodium compound in an oxygen atmosphere appears to be quite different (Fig. 3-5) from that in an inert atmosphere, indicative of an oxidation of the compound. If the sample is heated at 2 °C/min, as shown in Fig. 3, one endotherm and two exotherms are observed. The first endotherm which is due to melting, as observed in an argon atmosphere, begins at 267 °C and is then overwhelmed by the first of the exotherms. At a heating rate of 1 °C/min, Fig. 4, two exotherms and no endotherms are observed. Figure 5 shown at a heating rate of 0.1 °C/min reveals only a single exotherm which begins at 184 °C. From these figures, it is evident again that the rate of heating can lead to erroneous interpretation of the DTA spectra. Thus, it appears at a slow enough heating rate that decomposition occurs in a single continuous process which begins almost 90 °C below the melting point. At greater heating rates, the melting transition can be observed just as in DTA in an inert atmosphere. At heating rates of 2 °C/min, a 6% weight loss is observed at 220 °C, a 23% loss at 322 °C, and at 384 °C a 75% weight loss was obtained. The latter is indicative of RhO_2. Electron probe studies indicated that a rhodium compound containing 74% rhodium resulted from the oxidation at 384 °C. Powder diffraction studies of the sample however, did not give data which exactly matched those of the ASTM X-ray #21-1315 (1982) of the standard rhodium oxide patterns for the composition RhO_2, even though weight loss experiments indicate that this is the final stoichiometry. One possible cause is that the RhO_2 resulting from the present studies was not crystallized well. An alternative explanation is that it exists in more than one polymorphic state. However, such data has not been reported.

Thus, unlike the decomposition of the compound in an inert gas, at high enough heating rates, the oxygen heat treatment shows exothermic anomalies, in addition to melting. The final stage of decomposition appears to involve oxidation of the rhodium to form RhO_2.

3. Closed crucible DTA

As shown in Fig. 6, thermal heating of the compound under its own vapor pressure yielded a trace similar to Figure 2, for the behavior on an open crucible in an inert atmosphere. At 420 °C the closed sample ruptured. Figure 7a shows the cooling curve of the compound after heating to the first peak which, as before, was found to be the melting point of the compound. During cooling, an exothermic peak was observed at 225 °C. Upon reheating this same sample, as shown in fig. 7b, a new endothermic peak appears at 217 °C, along with another one at 289 °C. This indicates that even under its own developed pressure the acac decomposes slowly. Heating of this sample above 289 °C resulted in the crucible exploding.

B. Optical Absorption Studies

Figure 8 shows the UV absorption spectrum of rhodium(III) acetlyacetonate in methanol at a concentration of 2.0 x 10-5 M. The sample was scanned to 205 nm. At this wavelength, the solvent absorbs too strongly for further analysis. The absorption peaks for the compound correspond to wavelengths of 320, 260, and 210 nm. These correspond to log molar absoptivites,ϵ, of 4.89, 3.84, and 3.95, respectfully. The 320 and 260 nm wavelength absorptions agree with the work DeArmond did(12). He did not scan to 210 nm. However, the molar absoptivities he obtained for the 320 and 260 nm peaks are different than ours. The purity of the compound was not specified in DeArmond's work, nor was the nature of the solvent used, or the temperature at which the spectra was obtained. It is possible that the spectrum was run at liquid nitrogen temperatures, but even this is unclear from the description given in the publication.

C. Density

Density values of 1.75 ± 0.05 g/cm^3 were obtained for rhodium(III) acetylacetonate in paraffin oil.

CONCLUSION

Thermal decomposition of rhodium(III) acetylacetonate in an inert atmosphere produces essentially pure rhodium metal. Depending on the rate of heating of the compound, it can be made to either melt before it decomposes, or to decompose before it melts. Using annealing techniques, decomposition became measurable at 184 $^{\circ}$C. In an oxidizing atmosphere, the compound decomposes exothermically resulting finally in the formation of what appears to be RhO_2. Density measurements of the rhodium(III) acetylacetonate indicate a value of 1.75 g/cm^3. The absorption spectrum of the compound shows sensitivity in the ultraviolet.

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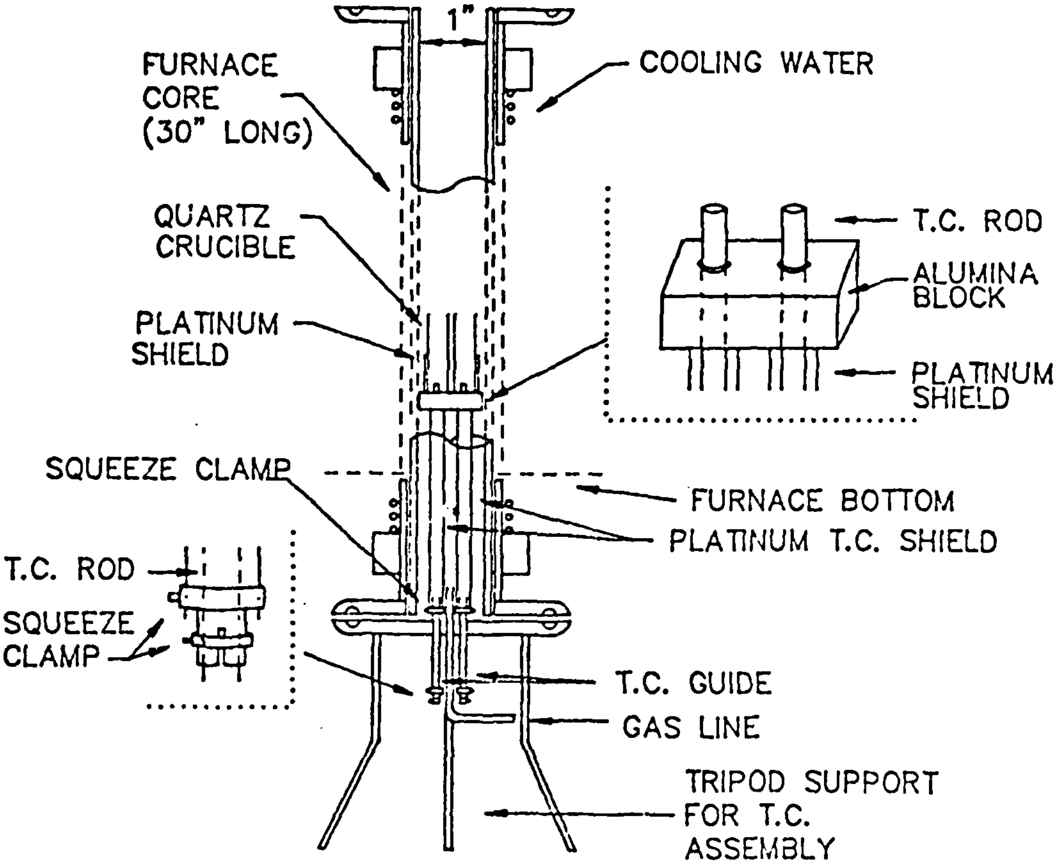

Fig. 1. Representation of the crucible and thermocouple assembly and their position in the furnace.

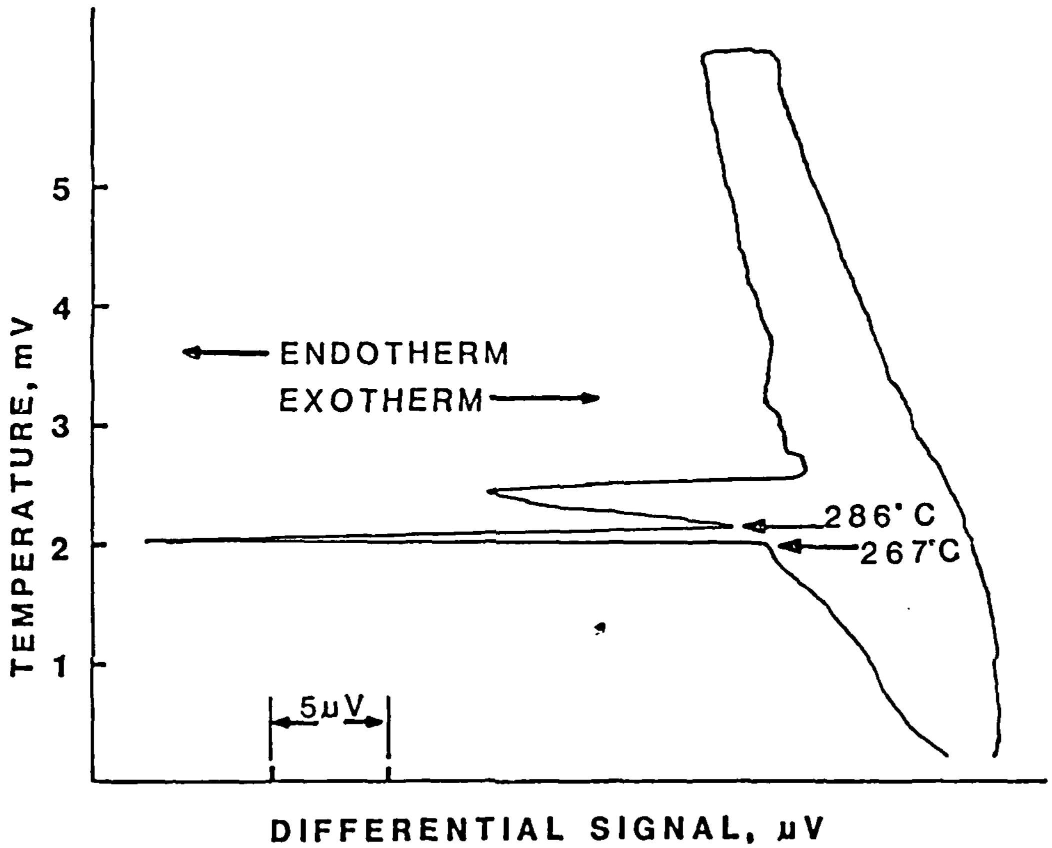

Fig. 2. Open crucible DTA of $Rh(acac)_3$ in argon at 2° C/min.

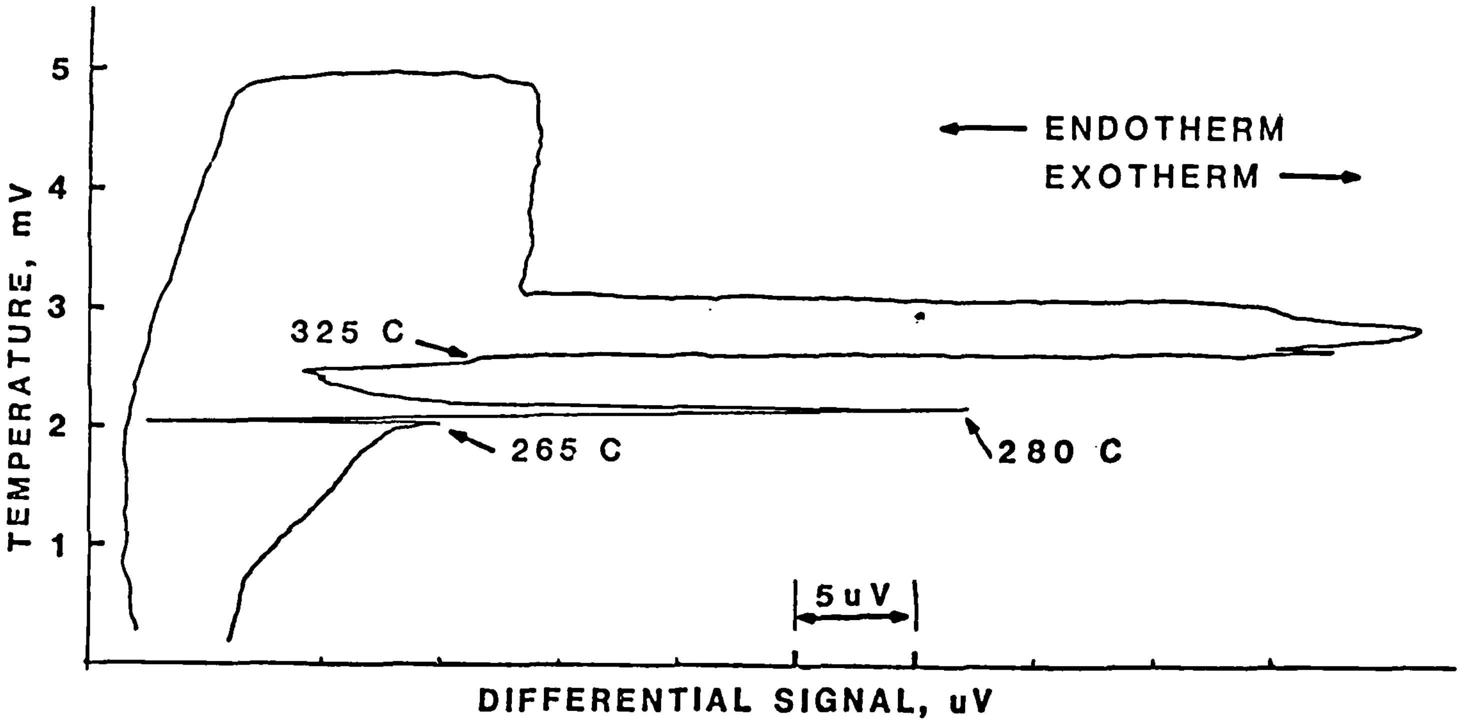

Fig. 3. Open crucible DTA of $Rh(acac)_3$ at 2 C/min in oxygen.

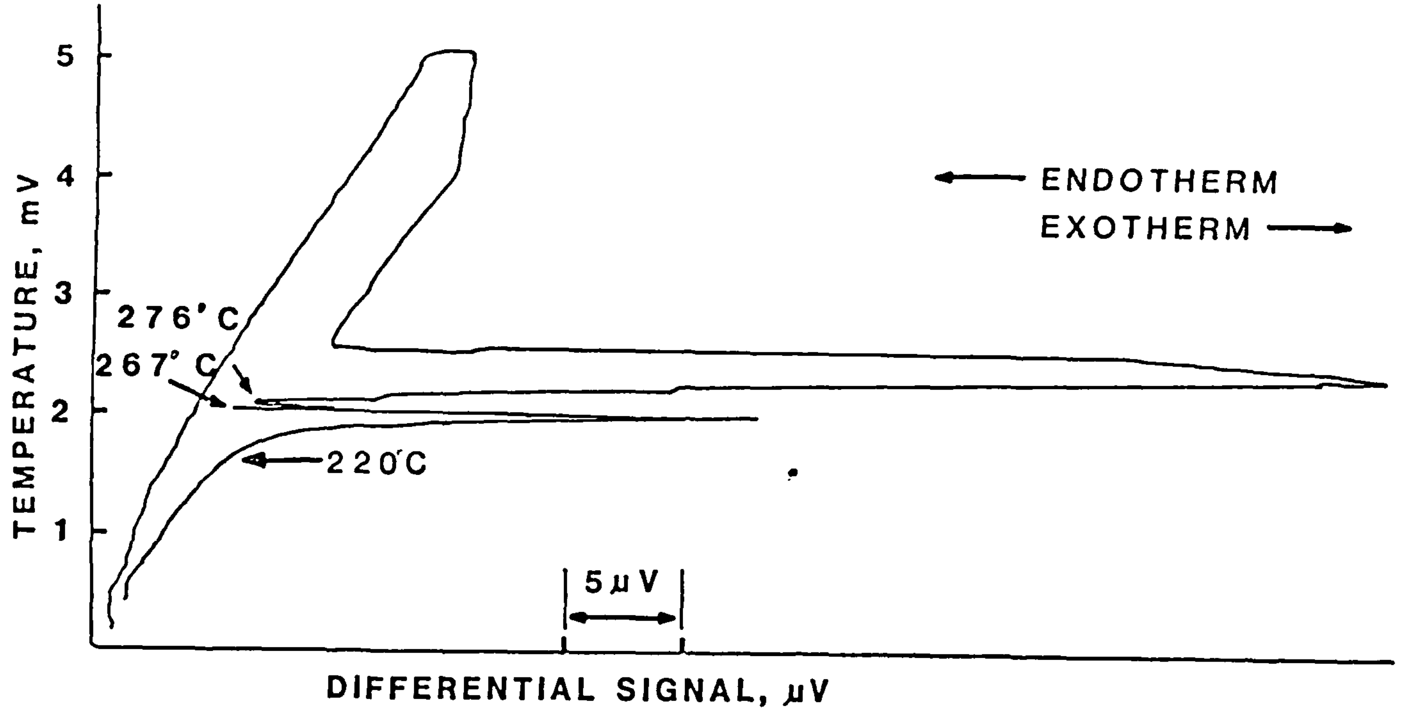

Fig. 4. Open crucible DTA of $Rh(acac)_3$ at 1°C/min in oxygen.

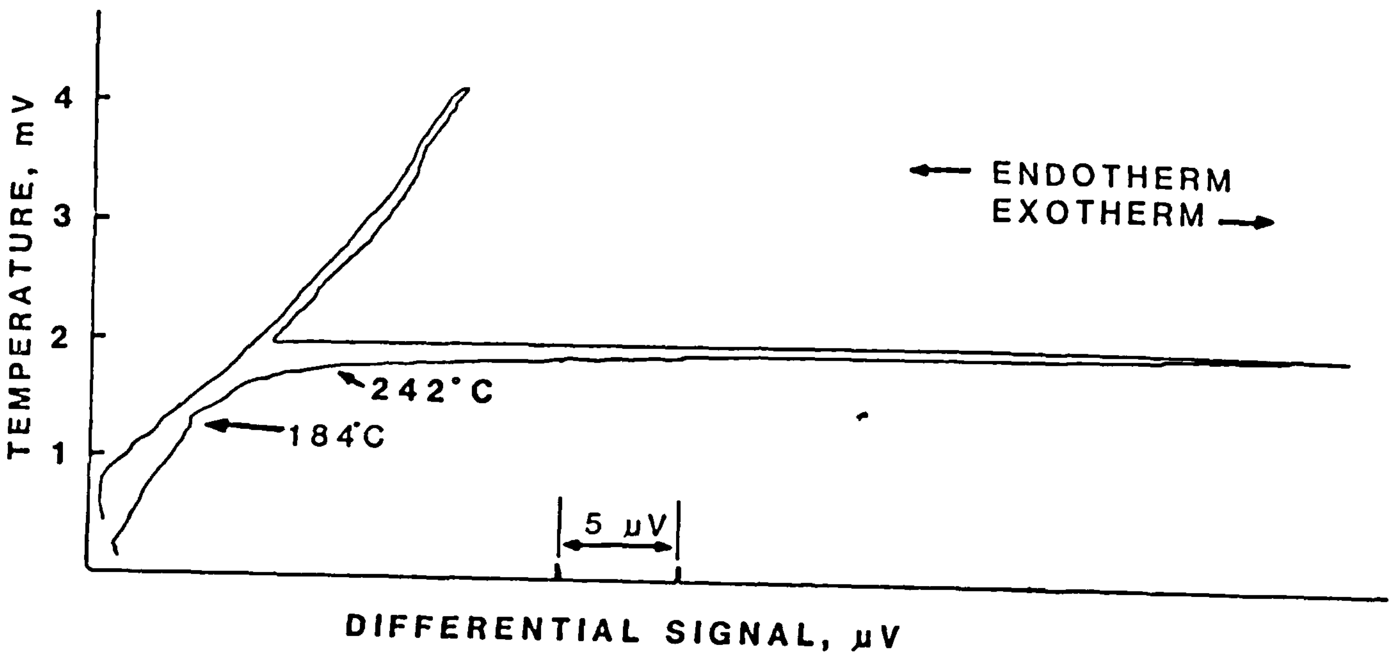

Fig. 5. Open crucible DTA of $Rh(acac)_3$ at 0.1°C/min in oxygen.

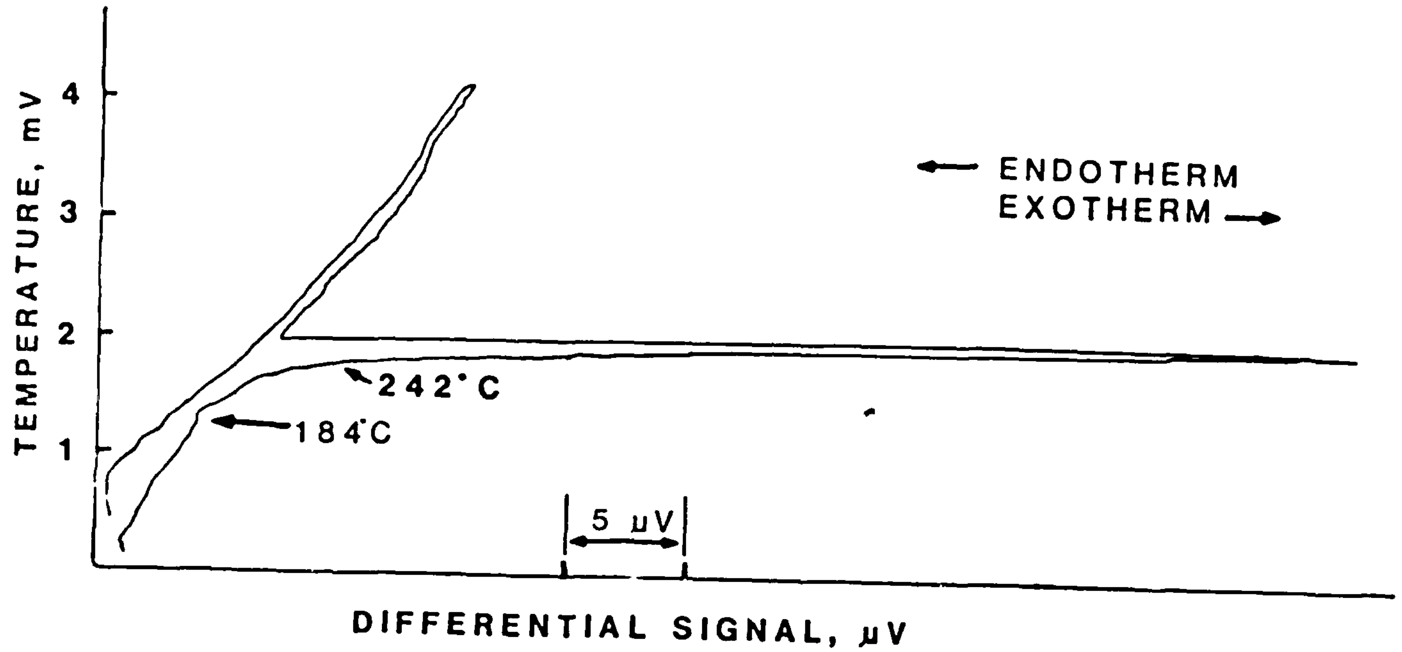

Fig. 5. Open crucible DTA of Rh(acac)$_3$ at 0.1°C/min in oxygen.

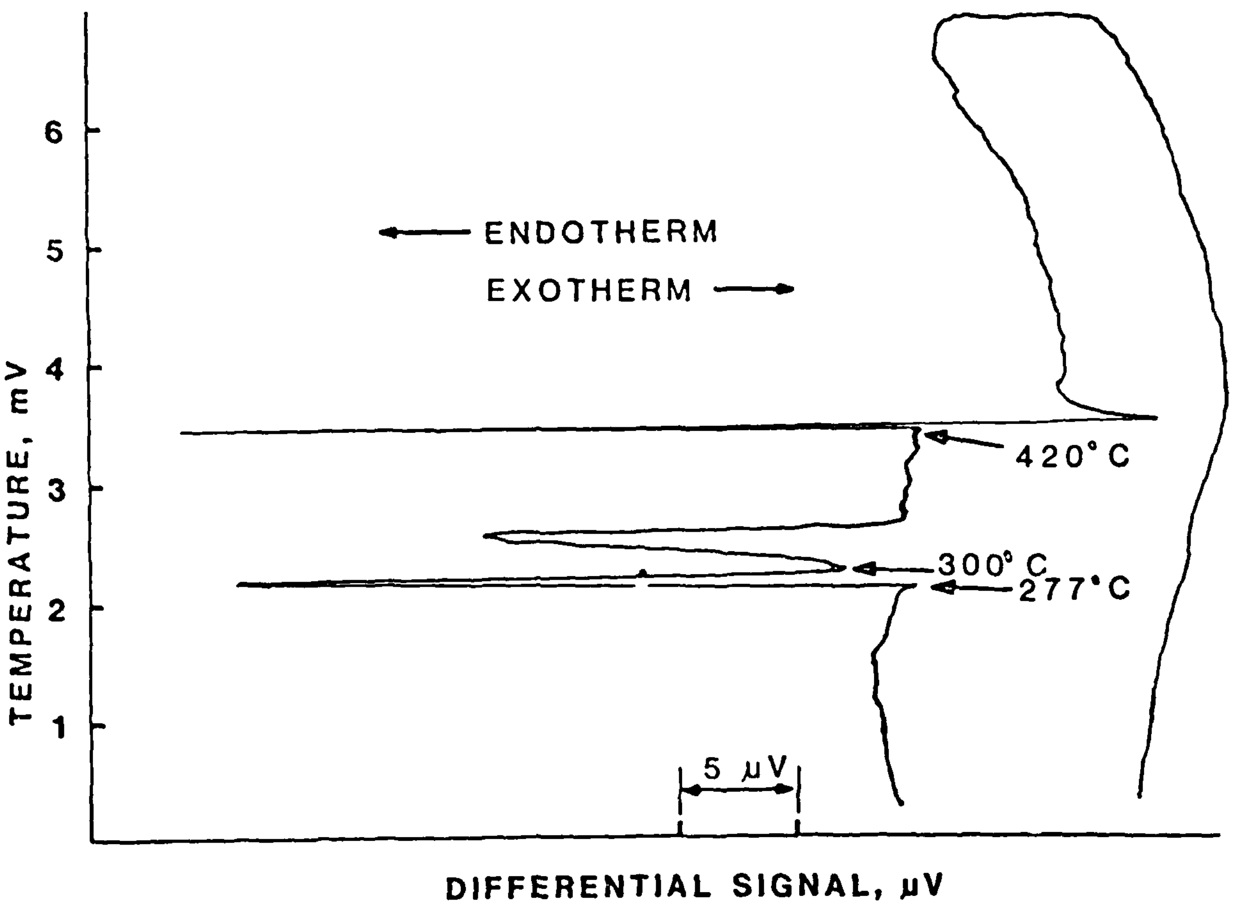

Fig. 6. Closed crucible DTA of Rh(acac)$_3$ at 2° C/min

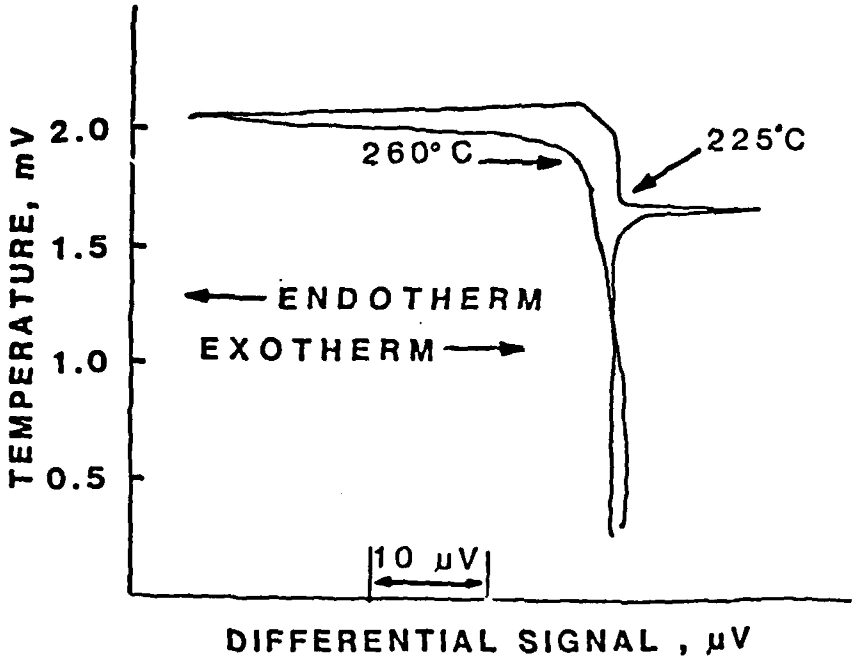

Fig. 7a. Cooling curve DTA of $Rh(acac)_3$ in a closed crucible at 2° C/min.

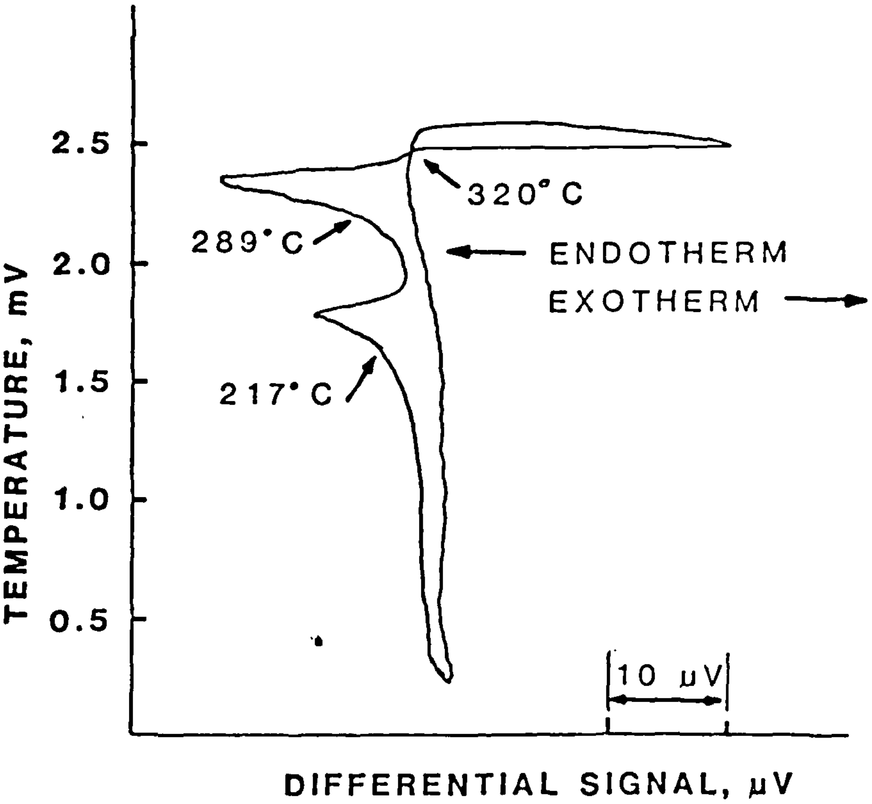

Fig. 7b. Sample of 7a reheated at 2° C/min.

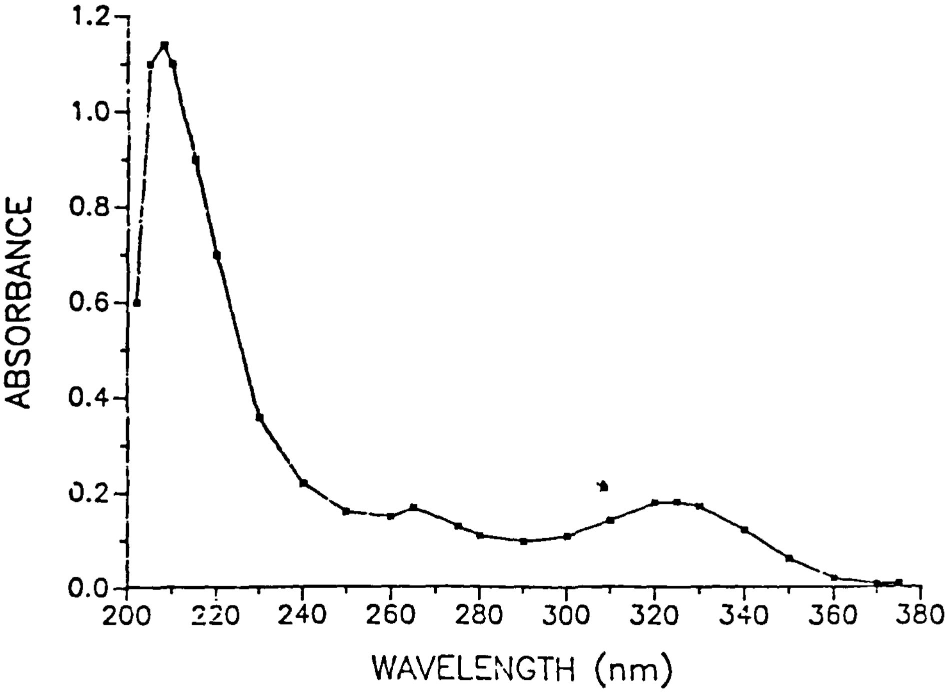

Fig. 8. Electronic absorption spectrum of $Rh(acac)_3$ at 2 x 10^{-5} M.

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