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Note

THE THERMAL DECOMPOSITION OF THE Cu(II) COMPLEX OF 3,5-DINITROSALICYLIC ACID

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The thermal properties of metal complexes with organic chelating reagents is one of the least studied aspects of coordination chemistry. Salicylic and substituted salicylic acids are powerful chelating agents [1] and, due to their abundant medicinal properties, they have always been the subject of considerable attention by researchers [2]. The decarboxylation and thermal stability of salicylic [3] and *p*-amino salicylic acids and their sodium and calcium salts are known [4]. However, thermal analysis literature so far contains only a few reports dealing with the thermal decomposition of alkali and alkaline earth metal salicylates [5—9]. Little is known about the thermal decomposition of Th(II) salicylates [10] and ammonium salicylate [11]. Very recently, we have reported the thermal decomposition of Fe(II), Zn(II), VO(II) [12] and Cu(II) [13] complexes of salicylic acid. The present communication deals with the thermal decomposition of the Cu(II) complex of 3,5-dinitrosalicylic acid (3,5 DNSA).

EXPERIMENTAL

Preparation of the complex

The complex was prepared following the method described elsewhere [14]. Equimolar solutions of metal salt and ligand were prepared and mixed in the stoichiometric ratio (M : L = 1 : 2). The solid green complex obtained was washed thoroughly with ethanol, dried and recrystallized from dimethylformamide. All the reagents used were of BDH AnalaR grade.

Elemental analysis

The metal content was estimated by conventional methods [15]. Carbon, hydrogen and nitrogen analyses were carried out on a Colemann CHN analyser 29. The results of elemental analyses were

	Cu(%)	C(%)	H(%)	N(%)
Calcd.	11.48	30.35	1.80	10.11
Exptl.	11.72	30.16	1.93	10.84

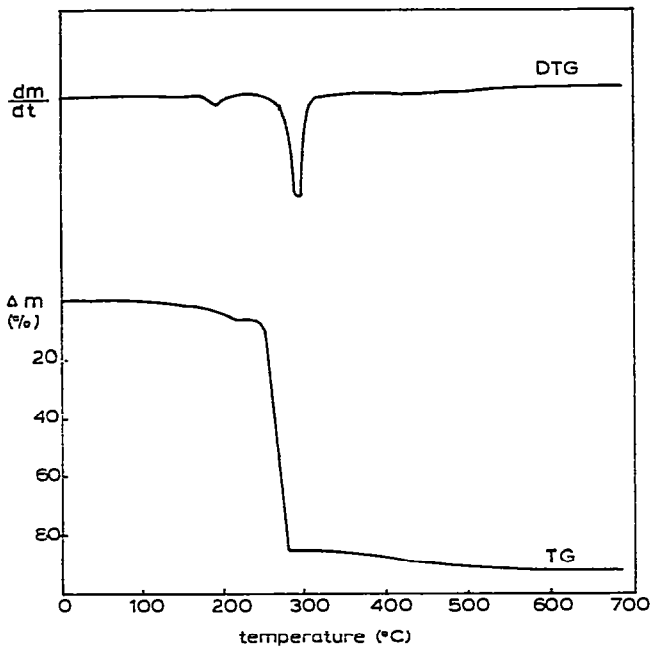

Fig. 1. TG and DTG curves of the Cu(II) complex of 3,5-dinitrosalicylic acid.

Thermal analysis

Thermogravimetry was carried out on a Stanton recording thermobalance (Model HT) of 1 mg sensitivity in static air. The sample (100 mg) was heated in a platinum crucible at a rate of 4°C min^{-1} to 700°C. The chart speed was maintained at 3 in. h^{-1}.

The TG and DTG results are shown in Fig. 1.

RESULTS AND DISCUSSION

The results of elemental analyses indicate that the complex formed has the composition $Cu(3,5\text{-DNSA})_2 \cdot 2\ H_2O$ where 3,5-DNSA is the 3,5-dinitrosalicylate anion.

Thermal decomposition

It was observed that the thermal decomposition of the complex began at 100°C with loss of water and the first inflection on the TG curve at 240°C corresponds to the weight loss of two water molecules. Beyond this, the thermal decomposition becomes extremely rapid and almost explosive. The complex exploded with fire and smoke at 280°C. Due to this explosive decomposition, some of the decomposing substance could have been easily ejected from the thermobalance. As a consequence of this, it was difficult to calculate the composition of the complex quantitatively by determination of the metal oxide content. The total weight loss observed in the thermal

decomposition was 92% and the end product was CuO. The oxide level began at 580°C. However, the CuO content obtained was lower than the theoretical, i.e. 14.11%, for the reason given above.

It is worthy of note that, in the thermal decomposition of the Cu(II) complex of salicylic acid, $Cu(SA)_2 \cdot 2\,H_2O$, no such explosive decomposition was observed [13] while, in the present case, explosion was noted which may be due to the formation of various nitro compounds during the thermal decomposition.

Results of this study on the thermal decomposition of the Cu(II) complex of 3,5-dinitrosalicylic acid showed that the complex decomposes in the range 100—560°C with explosion at 280°C giving a low CuO content.

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REFERENCES

1 D. Perrin, Organic Complexing Agents, Interscience, New York, 1964. S. Chaberek and A.E. Martell, Organic Sequestering Agents, Wiley, New York, 1959.
2 M.J.H. Smith and P. Smith, The Salicylates, Interscience, New York, 1966. A. Burger, Medicinal Chemistry, Parts I and II, Wiley-Interscience, New York, 3rd edn., 1970.
3 S. Rolski, Chemia Środków Leczniczych (Chemistry of Drugs) PZWL, Warsaw, 1964. E.M. Barrall and L.W. Roger, Anal. Chem., 34 (1962) 1106.
4 M. Wesolowski, Thermochim. Acta, 21 (1977) 243.
5 Y. Hara and H. Osada, Kogyo Kagaku Zasshi, 73 (1970) 1996; Kyushu Kogyo Diagaku Kenkyu Hokoku Kogaku, 22 (1971) 51.
6 R. Pirrisi and F. Mattu, Rend. Semin, Fac. Sci. Univ. Cagliari, 22 (1952) 163; 22 (1952) 170.
7 A. Radecki and M. Wesolowski, Thermochim. Acta, 17 (1976) 217.
8 A. Radecki, K. Kobayłczyk and B. Andrzejeczak, Ann. Acad. Med. Gedan, 4 (1974) 127; Rozpr. Wydz. III: Gdansk. Tow. Nauk, 9 (1972) 95.
9 A. Radecki and M. Wesolowski, J. Therm. Anal., 9 (1976) 16; 9 (1976) 29; 9 (1976) 357; 10 (1976) 233; 11 (1977) 39.
10 W.W. Wendlandt, Anal. Chim. Acta, 18 (1958) 316.
11 L. Erday, S. Gal and G. Liptay, Talanta, 11 (1964) 913.
12 B.D. Heda and P.V. Khadikar, Bull. Soc. Chim. Belg., 89 (1980) 1.
13 B.D. Heda and P.V. Khadikar, Univ. Indore Res. J. Sci., 6 (1980).
14 P.V. Khadikar, R.L. Ameria, M.G. Kekre and S.D. Chouhan, J. Inorg. Nucl. Chem., 35 (1973) 4301.
15 A.I. Vogel, Quantitative Inorganic Analysis, Longmann, London, 1973.