

Thermal Analysis of Metal Complexes of SuccinylSulfathiazole and Sulfisoxazole

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Abstract: The thermal decomposition processes for succinylsulfathiazole (SSTZ) and its metal complexes viz: Cd(SSTZ), Cu(SSTZ)(H₂O)₂, Ru(SSTZ)OH; sulfisoxazole (SUL) and Cd(SUL)(H₂O)₂ OH have been accomplished on the basis of TG, DTG and DTA studies..

Keywords: TG, DTG, DTA, succinylsulfathiazole, sulfisoxazole, metal complexes

INTRODUCTION

The investigation of metal sulfonamide compounds has received much attention as these were the first effective chemotherapeutic agents to be employed for the prevention and cure of bacterial infections in humans¹. Succinylsulfathiazole[2-{N(4)-succinylsulfanilamido}thiazole] (SSTZ) is the most effective of the poorly absorbed bacteriostatic sulfonamides used for sterilization of the intestinal tract. SSTZ was employed in some dietary treatments to further enhance the level of folate depletion both before cancer initiation and during promotional/progressions phase of carcinogenesis². Sulfisoxazole [4-amino-N-(3, 4-dimethyl-5-isoxazolyl) benzenesulfonamide] (SUL) is used to treat urinary tract infections³. The structure of SSTZ and SUL is given in Fig.1. Studies on thermal analysis of acid derivatives of sulphonamides have been reported⁴. We have reported previously the synthesis and characterization of metal- succinylsulfathiazole and metal - sulfisoxazole complexes⁵. The present communication describes the thermal decomposition

studies (TG, DTG and DTA) of SSTZ, SUL and some of their metal complexes.

EXPERIMENTAL

Succinylsulfathiazole [2-{N(4)-succinylsulfanilamido}thiazole] (SSTZ) and sulfisoxazole [4-amino-N-(3, 4-dimethyl-5-isoxazolyl) benzenesulfonamide] (SUL) were procured from Sigma Aldrich. The preparation and characterization of the metal complexes of succinylsulfathiazole and sulfisoxazole has been reported earlier⁵. The thermogravimetric (TG), differential thermogravimetric (DTG) and differential thermal analysis (DTA) studies were carried out on Perkin Elmer (Pyris Diamond) at Institute Instrumentation Centre, IIT, Roorke. Al₂O₃ was used as standard. The measurements were made at a heating rate of 10 °C min⁻¹ and a chart speed of 20 cm per hr⁻¹ in an atmosphere of nitrogen (flow rate 400 ml⁻¹). The representative curve of TG, DTG and DTA curve for Cd(II)-SSTZ complex is shown in Fig. 2.

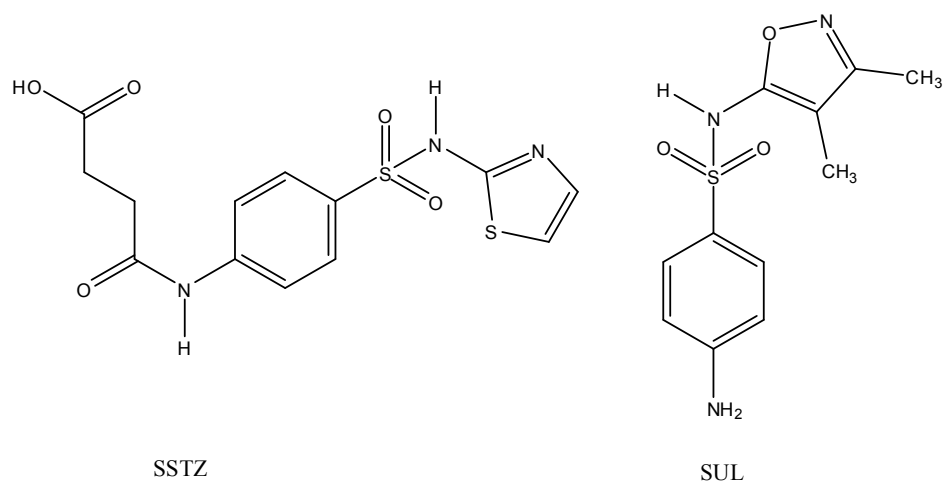


Fig.1. Structure of succinylsulfathiazole (SSTZ) and sulfisoxazole (SUL)

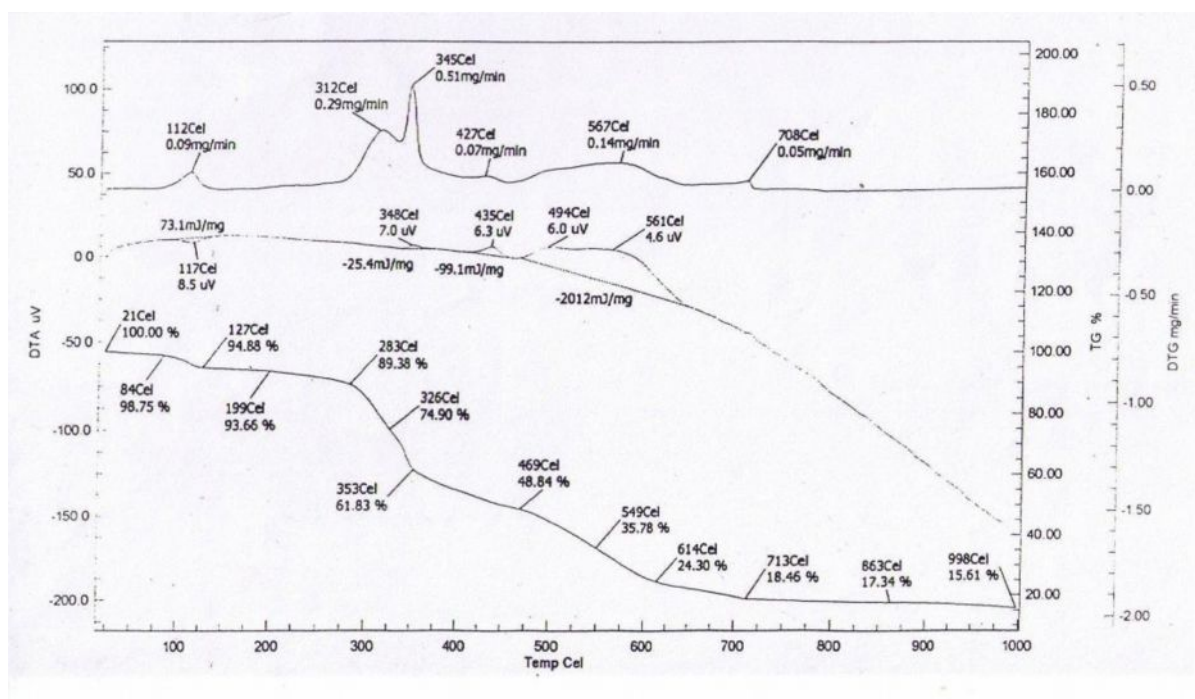


Fig. 2. TG (lower), DTG (upper) and DTA (middle) curve for Cd(II)-SSTZ complex

RESULTS AND DISCUSSION

The metal complexes of SSTZ and SUL were studied by thermogravimetric analysis from ambient temperature to 1273 °C in nitrogen atmosphere. The TG curves are shown as % mass loss versus

temperature, the DTG curves as the rate of loss of mass versus temperature, and DTA curves as enthalpy changes. TG, DTG and DTA data for the ligands and metal complexes are shown in Table 1.

Table 1. The character parameters of TG, DTG and DTA curves of SSTZ, SUL and their metal complexes.

Compound	TG		DTG	DTA	Remarks
	T _{range} /°C	Total mass loss %	Peaks/°C	Peaks/°C	
C ₁₃ H ₁₃ N ₃ O ₅ S ₂ (SSTZ)	0-750	100	108 186 289 664 ^b	109 (endo) 188(endo) 291(exo) 664(exo)	Melting of SSTZ. Continuous exothermic decomposition of SSTZ up to 750 °C
Cd(SSTZ)	21- 283 284-353 354-713 714-998	10.6223 8.17 81.54 84.39	112 312 345 427 567 708	117(endo) 348(exo) 435(exo) 450-614 ^b (exo)	(CdS) ₂ as end product agree with final residue mass
Cu(SSTZ)(H ₂ O) ₂	23-133 134-277 278-475 476-654 655-960	9.0 21.45 49.86 90.72 91.64	128 195 299 398 495 600 971	143(endo) 282(exo) 398(exo) 498 ^b (exo) 675(exo)	Loss of two water molecules up to 133 °C. Thermal decomposition occurs in a number of steps.
Ru(SSTZ)OH	25-167 168-306 306-386 387-462	8.21 34.62 69.51 94.75	313 390 415	324 ^b (exo) 390(exo) 414 ^b (exo)	A number of steps for thermal decomposition of the complex occurs
C ₁₁ H ₁₃ N ₃ O ₃ S (SUL)	24-180 181-308 309-699	1.21 49.58 100	200 285 640	200(exo) 653(exo)	Continuous exothermic decomposition of SSTZ up to 699 °C
Cd(SUL)(H ₂ O) ₂ OH	22-220 221-539 540-884 885-999	8.83 29.91 33.88 34.09	206 280 425 520 869	210(endo) 520(exo) 736(exo) 914(endo)	Loss of two water molecules up to 220 °C

b - broad

SSTZ

The DTA endothermic peak at 188 °C corresponds to melting of SSTZ ($\Delta H = 0.312 \text{ J mol}^{-1}$), followed by the continuous decomposition of SSTZ up to 750 °C. ΔH values for two exothermic peaks at 291 °C and 664 °C were found to be $-0.171 \text{ J mol}^{-1}$ and $-8.056 \text{ J mol}^{-1}$ respectively.

Cd(SSTZ)

The thermal decomposition of complex occurs in a number of stages with DTG curve maxima at 112, 312,

345, 427, 567 and 708 °C. The final residual mass left at 998 °C correspond to (CdS)₂ formation as end product. $\Delta H = 0.157 \text{ J mol}^{-1}$ for endothermic peak at 117°C, $\Delta H = -0.054 \text{ J mol}^{-1}$ for exothermic peak at 348°C, $\Delta H = -0.213 \text{ J mol}^{-1}$ for exothermic peak at 435°C, $\Delta H = -4.319 \text{ J mol}^{-1}$ were observed.

Cu(SSTZ)(H₂O)₂

The initial mass loss of about 9 % may be associated with loss of two water molecules in temperature range 23-133 °C. DTG data show that decomposition occurs

in a number of steps with temperature maxima at 128, 195, 299, 398, 495, 600 and 971 °C. Exothermic peaks at 282, 398, 498 and 675 °C have ΔH -0.174, -0.247, -3.106 and -0.026 J mol⁻¹. The residual mass left at 654 °C and 1002 °C suggests the existence of Cu₂O and Cu respectively as end products.

Ru(SSTZ)OH

DTG data indicates a number of steps for thermal decomposition with temperature maxima at 313, 390, 415 °C. Exothermic peaks at 324 °C (ΔH = -4.0072 J mol⁻¹), 390-414 °C (ΔH = -1.7840 J mol⁻¹) were observed.

SUL

A slight loss of mass up to 1.21% in temperature range 24-180 °C may be associated with moisture. The SUL decomposes in two steps in temperature range 221-539 °C and 540-999 °C. This is supported by two peaks in

DTG curve with temperature maxima at 285 °C and 640 °C. DTA data show two exothermic peaks at 200 °C and 653 °C with -2.117 J mol⁻¹ and -6.038 J mol⁻¹ respectively (Table 1).

Cd(SUL)(H₂O)₂ OH

First mass loss of 8.83% corresponds to loss of two water molecules in temperature range 22-220 °C. The next step of decomposition occurs in temperature ranges 221-539 °C and 540—999 °C as it is evident from DTG curve peaks with temperature maxima at 425 °C and 520 °C. The residual mass corresponds to CdS formation as DTG shows a peak at 869 °C. ΔH values at 117, 348, 435, 450-614 °C were found to be 0.09 J mol⁻¹, 0.084 J mol⁻¹, -0.158 J mol⁻¹ and -0.062 J mol⁻¹ respectively. The final mass loss of about 34% may be due to mixture of CdO and CdS or (CdS)₂ as end product.

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