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Thermoanalytical study of purine derivatives compounds

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Abstract: Thermal methods of analysis are now used in a very large range of scientific investigations. In this work simultaneous thermogravimetry-differential thermal analysis (TG-DTA), X-Ray powder diffractometry and infrared spectroscopy were used to study the derivative compounds of purine, i. e. aminophylline, theophylline, caffeine and uric acid. The results led to informations about the thermal stability and thermal decomposition of these compounds.

Keywords: aminophylline; theophylline; caffeine; uric acid; TG; DTA.

Introduction

Several investigations have been carried out on the application of thermoanalytical techniques for the study of drug substances, excipients and drug substances in dosage forms. Montagut and co-work [1] investigated dipyrone by employing DTA and TG, and examined the possibility of applying TG in quantitative studies. Wendlandt and Collins [2] used DTA and TG as aids in the characterization and identification of commercial non-prescription analgesics. Other investigations of the use of thermoanalytical techniques for the study of drug substances and for applications in routine pharmaceutical analysis and in the pharmaceutical industry have also been described [3-20].

In this paper derivative compounds of purine were investigated by using thermoanalytical techniques (TG, DTA), X-ray diffractometry and infrared spectroscopy.

Experimental

Caffeine (3,7-dihydro-1,3,7-trimethyl-1H-purine-2,6-dione); Theophylline (3,7-dihydro-

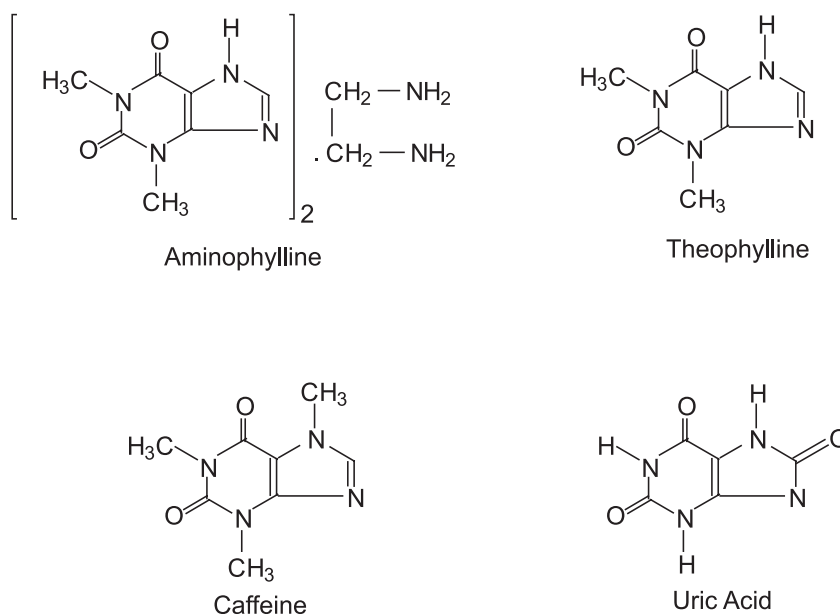
1,3dimethyl-1H-purine-2,6-dione) and Aminophylline (3,7-dihydro-1,3-dimethyl-1H-purine-2,6-dione compd. with ethylenediamine) were obtained from Quimidrol LTDA. Uric Acid (7,9-dihydro-1H-purine-2,6,8(1H,3H,9H)-trione) was obtained from Riedel de Haen.

The formula of these compounds are shown in scheme I

TG, DTG and DTA curves were obtained by using a simultaneous SDT 2960 from TA Instruments. The purge gas was nitrogen flow of 100 mL min⁻¹, heating of 20°C min⁻¹ and with a sample weighing about 6-8 mg. Alumina crucible was used for the TG, DTG and DTA studies.

X-ray powder patterns were obtained with a Simens D-500 X-ray diffractometer using CuK_α radiation (λ=1,544 Å) and setting of 40 kV and 20 m A. Infrared spectra for these compounds were run on a spectrophotometer model FTIR-8400 Shimadzu, within the 4000-400 cm⁻¹ range. The solid samples were pressed into KBr pellets.

Fusion of these compounds was determined by using a melting point apparatus model MQAPF-301 from Micro Química.



Scheme I: Structure of the derivative compounds of purine.

Results and Discussion

Theophylline

The TG, DTG curves of theophylline are shown in Fig. 1. These curves show that the compound is stable up to 250°C, and between 250-375°C the mass loss occurs in a single step, without formation of residue.

The DTA curve, Fig. 2, shows an endotherm between 256 and 375°C with two peaks

at 273°C and 350°C, respectively. The first endothermic peak is due to the fusion of the compound and in agreement with the literature [21]. The second endothermic peak is due to evaporation of the compound and in agreement with the TG curve, where the total mass loss is observed. The evaporation of the compound occurs without decomposition, in compliance with the infrared spectra of theophylline, and solidified sample collected after evaporation, as can be seen in the Fig. 3.

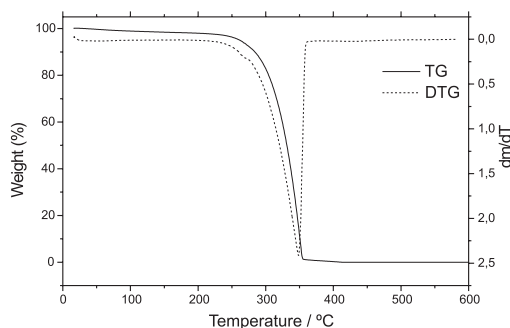


Figure 1- TG and DTG curves of theophylline sample ($m_i = 6.640$ mg).

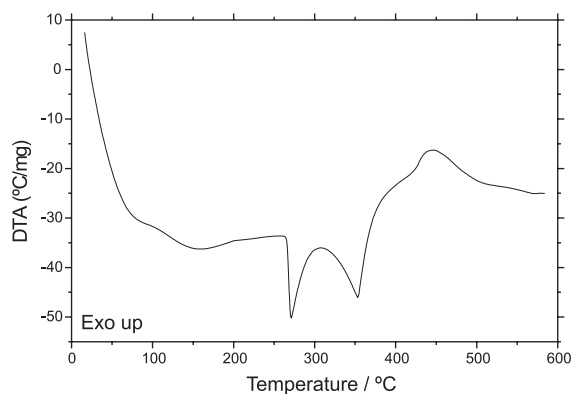


Figure 2 - DTA curve of theophylline sample ($m_i = 6.640$ mg).

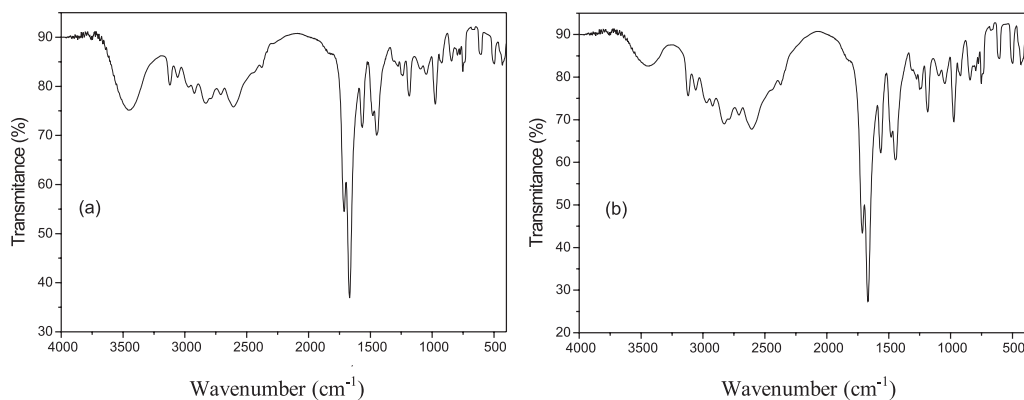


Figure 3 - FTIR spectra of (a) theophylline sample at room temperature (b) sample collected after condensation.

Aminophylline

The TG and DTG curves of aminophylline are shown in Fig. 4. These curves show that the compound is stable up to 78°C, and this temperature the TG curve suggests mass loss in two steps, although the DTG curve shows three steps, being the two first as overlapping steps. The mass loss that occurs between 78 and 144°C is ascribed to the loss of ethylenediamine (TG=14.41%; Calcd=14,30%), with formation of theophylline. The second step that occurs between 238 and 362°C with total mass loss

is due to evaporation of theophylline.

The DTA curve of aminophylline is shown in Fig. 5. The first endothermic peak at 140°C is attributed to elimination of ethylenediamine and in agreement with the first mass loss of TG curve. The endotherm between 256 and 370°C with two peaks at 273 and 330°C, that show a great similarity with the DTA curve of theophylline, are attributed to fusion and evaporation, respectively. The infrared spectrum of solidified sample collected after evaporation is shown Fig. 6, in agreement with the FTIR spectra of theophylline, Fig. 3.

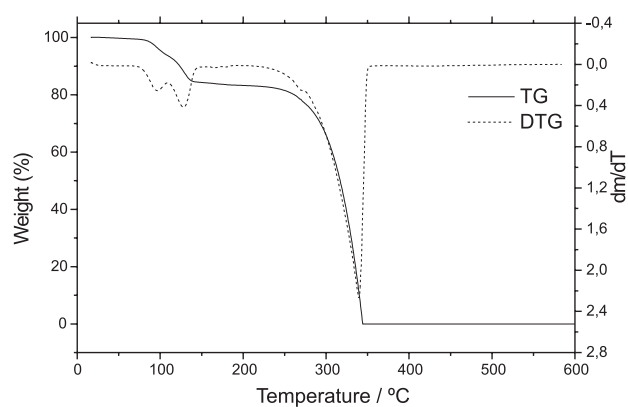


Figure 4 - TG and DTG curves of the aminophylline sample ($m_i = 8.607$ mg).

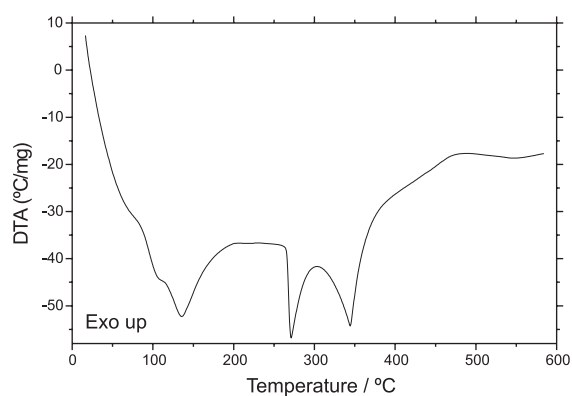


Figure 5 - DTA curve of aminophylline sample ($m_i = 8.607$ mg).

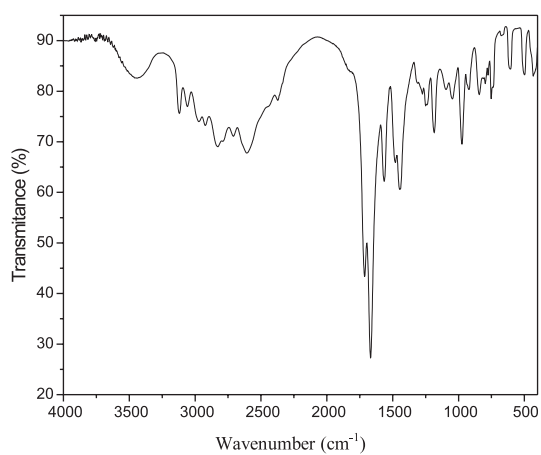


Figure 6 - FTIR spectrum of sample after condensation of aminophylline.

Caffeine

The TG and DTG curves of caffeine are shown in Fig. 7. These curves show that the compounds are stable up to 175°C and between 175-315°C, the total mass loss occurs in a single step.

The DTA curve of caffeine is shown in Fig. 8. The first endothermic peak at 163°C is due to a change of crystalline structure in compliance with

the X-ray powder patterns, as can be seen in Fig. 9 and in agreement with the literature [10]. The endotherm between 225-320°C, with two peaks at 270°C and 312°C are due to fusion and evaporation respectively. The evaporation of caffeine occurs without decomposition, in compliance with the infrared spectra of caffeine and solidified sample collected after evaporation, as can be seen in Fig. 10.

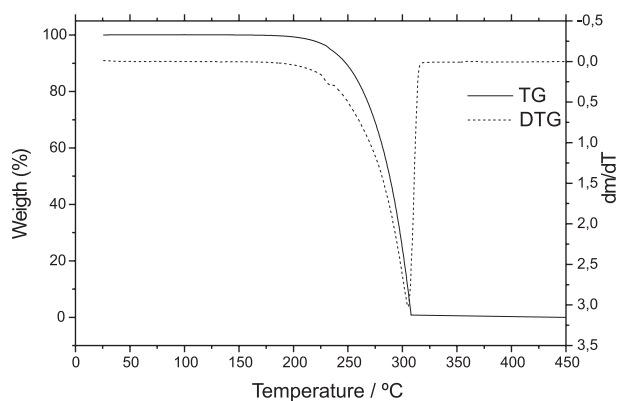


Figure 7 - TG and DTG curves of caffeine sample ($m_i = 7.435$ mg).

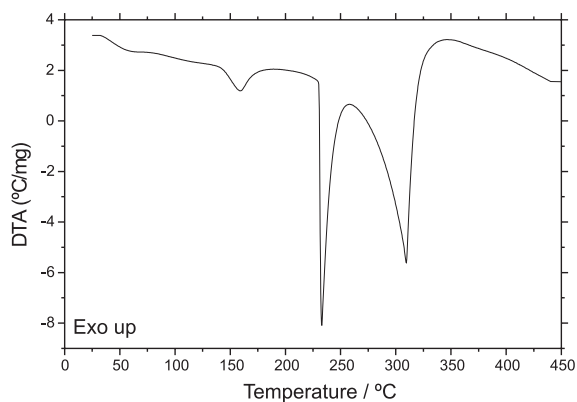


Figure 8 - DTA curve of caffeine sample ($m_i = 7.435$ mg).

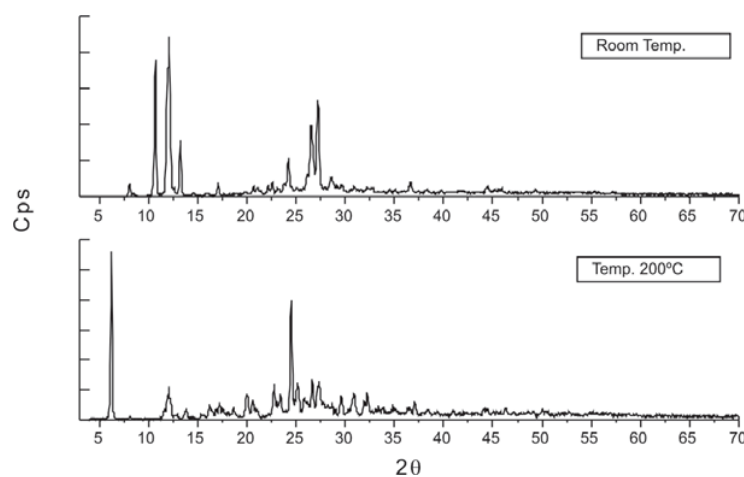


Figure 9 - X-ray diffraction powder patterns of the caffeine sample at room temperature and at 200 °C.

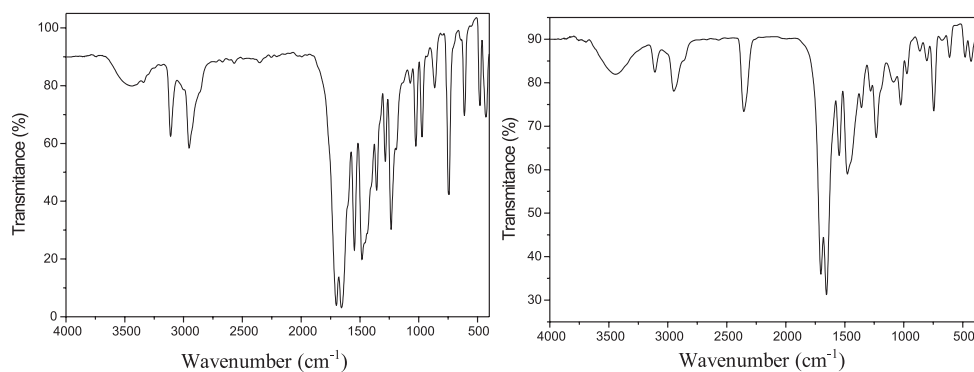


Figure 10 - FTIR spectra of the caffeine (a) sample at room temperature (b) evaporation product.

Uric Acid

The TG and DTG curves of uric acid are shown in Fig. 11, these curves show that the compound is stable up to 375°C, and above this temperature the mass loss begins through a fast process up to 480°C, followed by a slow process

between 480 and 600°C, with loss of 60% and 29%, respectively. These curves also show that the mass loss is still being observed up to 600°C.

The DTA curve of uric acid is shown in Fig. 12. The endothermic ascribed to thermal decomposition corresponding to the fast mass loss of the TG and DTG curves.

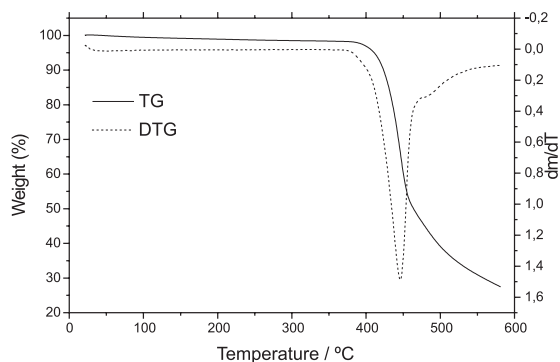


Figure 11 - TG and DTG curves of uric acid sample ($m_i = 7.581$ mg).

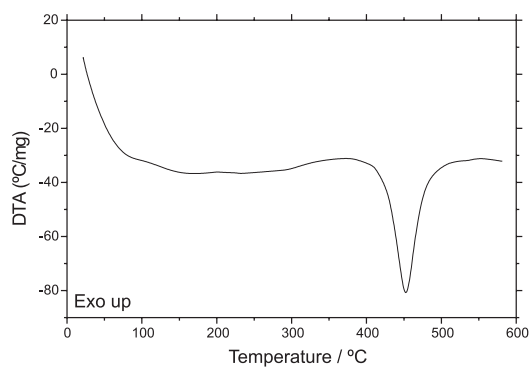


Figure 12 - DTA curve of uric acid sample ($m_i = 7.5809$ mg).

Conclusion

The TG, DTG, DTA curves, infrared spectroscopic spectra and X-ray powder patterns, provided information on the thermal stabilities and thermal decomposition of these compounds.

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E. Schnitzler, M. Kobelnik, G. F. C. Sotelo, G. Bannach, M. Ionashiro. Estudo termoanalítico de compostos derivados da purina.

Resumo: Os métodos térmicos em análises estão sendo atualmente muito utilizados nas investigações científicas. Neste trabalho a termogravimetria-análise térmica diferencial simultânea (TG-DTA), difratometria de raios X pelo método do pó, espectroscopia de absorção na região do infravermelho foram utilizadas para estudar os compostos derivados da purina. Sendo estes: aminofilina, teofilina, cafeína e ácido úrico. Os resultados forneceram informações com respeito à estabilidade térmica e decomposição térmica sobre esses compostos.

Palavras-chave: aminofilina; teofilina; cafeína; ácido úrico; TG; DTA.

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