

Application of Combined Thermoanalytical Techniques on the Investigation of Organic Compounds

Cs. Novák^a, Zs. Éhen^a, T. F. Moura^b, L. P. Fernandes^b

^a Institute of General and Analytical Chemistry, Budapest University of Technology and Economics, Szent Gellért tér 4, H-1111 Budapest, Hungary

^b Universidade Federal do Rio Grande do Norte, Centro de Ciências de Saúde, Departamento de Farmácia, Av. Gal. Cordeiro de Farias s/n - Petrópolis, 59010-180, Natal, RN, Brazil

Application of different thermoanalytical methods in the pharmaceutical technology is commonly known [1]. They are simple and reliable techniques, but none of them gives information about the composition of the products evolved on heating. Determination of evolved gases using combined techniques (TG-FTIR, TG-MS) is a well established and successfully applied method in the analysis of inorganic and organic substances. These combined techniques allow the identification of the products of desolvation and/or dehydration and makes possible to follow the process of thermal degradation/fragmentation [2].

Cyclodextrin inclusion complex formation is a widely used technique to improve the solubility, to protect the thermal stability of pharmaceuticals against light, UV-radiation and also to reduce the loss of volatile and easy-to-sublime compounds. The application of thermoanalytical techniques is commonly applied to prove the complex formation between guest and host, but in all cases they do not give adequate information, when does the entrapped guest leaves from its inclusion upon heating.

Besides the thermoanalytical characterization of the cyclodextrin inclusion complexes of *Lippia sidoides* and *Mentha oil x villosa* essential oil extracts, the aim of the present study was to develop an experimental procedure to determine to follow the escape of the volatile compounds from their inclusion complexes. The obtained results using “classical” thermoanalytical methods were compared to the results provided by the TG-MS combined technique. Complementary investigations have been done with X-ray powder diffractometry.

All investigated inclusions have been prepared using “suspension” technique in aqueous medium, starting from 1:1 molar ratio between essential oil extracts (guests) and β -cyclodextrin (host). The amount of the entrapped guests has been determined using UV spectrophotometry.

The inclusion complex formation was confirmed by TG-MS combined technique. Moreover the elaboration of the entrapped fractions of the essential oils could be followed [3].

Summarizing the experiences of the TG-MS analysis it was concluded, that the application of this combined technique is powerful to prove the host-guest interaction and also to follow the evolution of the free, the loosely and/or tightly bound portions of guests.

Reference

- [1] F. Mallet, S. Petit, S. Lafont, P. Billot, D. Lemarchand, G. Coquerel: : Therm. Anal. Cal. 73(2) (2003) 459
- [2] J. M. Rollinger, Cs. Novák, Zs. Éhen and K. Marthi, J. Therm. Anal. Cal., 73(2) (2003) 519
- [3] L. P. Fernandes, Zs. Éhen, T. F. Moura, Cs. Novák J. Sztatisz: J. Therm. Anal. Cal., in press.