

Theme:

***Determination of Thermal Behavior of the Solid Products
and Materials
by Thermal Analysis Methods***

Contract CEEX: 02/2005 - AT

Contracting Authority: RENAR – UMM CEEX MODUL IV

Contractor: INCDIE ICPE – CA Bucuresti

Director of Project: dr. Chim. Petru BUDRUGEAC

Duration : 07.09.2005 – 10.02.2007

General objectives:

- Enlargement of the test range in the accreditation system by assimilation and implementation of new methods of thermal analysis (TG, DTG, DTA, DSC, DIL, separated and coupled) of the product and materials;
- Improvement of the test quality by acquisition of specific high technology devices and equipments, in agreement with the European directives in the field.

Final result : Enlargement of the test range in the accreditation system

Stages of Project achievement:

Stage I: Improvement of the technical-material base of the Laboratory
Duration: 07.09.2005 – 12.12.2005

Stage II: Arrangement of the Laboratory space and setting on function of the devices and equipments that were secured in the first stage of the Project. Thermal analysis of some standard substances and materials
Duration: 13.12.2005 - 16.06.2006

Stage III: Preliminary tests: thermal characterization of some materials and products. Laboratory specific activities. Dissemination of some information by participation at the following Symposia:

- 9-th European Conference on Thermal Analysis and Calorimetry, Cracovia – August 2006
- Iasi Academic Days– September 2006;
Duration: 17.07.2006- 10.11.2006;

Stage IV: Dissemination of the research results. Enlargement of the accreditation
Duration : 11.11.2006- 10.02.2007.

Obtained results:

■ **improvement of the technical-material base of the Laboratory by acquisition of the following devices and soft:**

1. Simultaneous TG-DTA/DSC Apparatus + FTIR- STA 409PC +FTIR- Produced by NETZSCH GERATEBAU GMBH –Germany
2. Differential scanning calorimeter-DSC 204 F1 Phoenix- Produced by NETZSCH GERATEBAU GMBH –Germania
3. Dilatometer DIL 402 PC/4 - Produced by NETZSCH GERATEBAU GMBH –Germany
4. Thermokinetics software for kinetic analysis of TG, DTA, DSC and DIL data - Produced by NETZSCH GERATEBAU GMBH –Germany

- training of the laboratory members that have activities in the thermal analysis of the products and materials;
- elaboration of specific procedures corresponding to the test range that are subject of the accreditation enlargement, as well as elaboration of the procedures corresponding to the new tests, which will be performed by improving the technical-material base;
- performing of some tests with the new devices and equipments. Elaboration of Test Reports;
- performing some specific activities;
- enlargement of the accredited test range.

Capitalization of the Project results:

By enlargement of the accredited test range, the following new tests were accredited:

- DSC analyses;
- dynamometric analyses (DIL);
- coupled analyses (TG+DTA; TG+DSC)

Dissemination of some obtained results:

- International Symposium: 9-th European Conference on Thermal Analysis and Calorimetry, ESTAC'9, Cracovia – August 2006

1. Use of thermal analysis methods and scanning electron microscopy to assess the damage in the patrimonial books from Romanian libraries, authors: P. Budrugeac, L. Miu, M. Souckova
2. Influence of Barium precursors on the characteristics of BaTiO₃ nanopowders obtained by solid-gel method, authors: A. Ianculescu, A. Brăileanu, M. Crișan, P. Budrugeac, N. Drăgan, G. Voicu, D. Crișan, V. E. Marinescu
3. Non-isothermal kinetic study of the decomposition of Zn acetate-based sol-gel precursor. II. The application of the IKP method, authors: P. Budrugeac, V. Mușat, E. Segal.

*These works were accepted for publication in the **Journal of Thermal Analysis and Calorimetry**, which is a ISI journal*

- National Symposium: Iasi Academic Days – September 2006

1. Accelerated electron effects on EVA based compound, authors: P. Budrugeac, T. Zaharescu;

- **Workshop**: “Thermal analysis of products and materials by the following techniques: thermogravimetry (TG), differential thermal analysis (DTA), differential scanning calorimetry (DSC) and dilatometry (DIL)”.

1. Information concerning thermal behaviour of the products and materials obtained by application of the thermal analysis methods, author Virgil Marinescu.
2. Application of the thermal analysis methods for prediction of the thermal lifetime of products and materials, author P. Budrugeac.

Services:

Now, the Laboratory of “Determination of thermal behavior of the solid products and materials by thermal analysis methods” from INCDIE – ICPE CA has a [modern and performing technical-material base](#), which allows a high level scientific activity, offering high quality services of characterization in different domains, comparable with those from some similar laboratories from European Union

LABORATORY: DETERMINATION OF THERMAL BEHAVIOR OF THE SOLID PRODUCTS AND MATERIALS BY THERMAL ANALYSIS METHODS

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- **Simultaneous TG-DTA/DSC Apparatus + FTIR- STA 409PC +FTIR**

Produced by:
NETZSCH GERATEBAU Gmbh – Germany

- Temperature range: 25 ... 1500°C
- Heating rate 0 ... 50 K/min
- Resolution for weight: better than 2 µg
- Maximum relative errors of DTA and DSC signal: ±3%
- Measurements under defined gas atmosphere; coupled with FTIR spectrometer (7500 ... 370 cm⁻¹)



- **DSC 204 F1 Phoenix**

Produced by:
NETZSCH GERATEBAU Gmbh – Germany

- Temperature range: -85 ... 600°C
- Sensitivity: < 0.3 µW
- Reproducibility: < 0.5 % for enthalpy changes and < 0.1 K for temperatures
- Measurements under defined gas atmosphere
- Heating rates: 0.001 K/min – 100 K/min
- Programmable Cooling rates



- **Dilatometer DIL 402 PC/4**

Produced by:
NETZSCH GERATEBAU Gmbh – Germany

- Temperature range: 25 ... 1600°C
- Reproducibility of temperature: ±0.5°C
- Accuracy for expansion coefficient α : $1 \times 10^{-8} \text{ K}^{-1}$ (or ±0.5% for most materials)
- Reproducibility α : $1 \times 10^{-8} \text{ K}^{-1}$; controlled atmosphere



- **Thermokinetics software for kinetic analysis of TG, DTA, DSC and DIL data**

- **Applications**

By using of these devices, it is obtained a complex „thermal spectrum” of a material from which we can directly determine:

- temperatures at which the phase transitions (melt, glass transition) occur;

- temperature range of material stability; dimensional and mechanical changes due to the heating of the material;
- caloric effects of some processes;
- chemical composition of the gaseous atmosphere resulted by decomposition or thermo-oxidation of the investigated material.

By working of the thermal analysis data, the following information may be obtained:

- crystallinity degree;
- compatibility of some compounds used for obtaining of composite materials;
- mechanisms of the processes put in evidence in thermograms;
- kinetic and thermodynamic parameters characteristic for these processes;
- relative thermal stability;
- thermal endurance (thermal lifetime) of a material corresponding to a certain temperature and an endpoint criterion.