

TA-MS and TA-FTIR Workshops in Korea

Motivated by our positive experience with workshops on a variety of Thermal Analysis topics in Europe and the USA, NETZSCH Korea (NKS) organized, for the first time, three seminars in Seoul and Taejon from October 17th to 19th, 2000.

TA-MS and TA-FTIR coupling and thermophysical measuring techniques were the main subjects. Because of the perfect organization by NKS and the participation of our

coupling expert, Mr. E. Kaisersberger from Selb, a high number of participants attended. The workshops enabled interested people and customers to exchange ideas and notes regarding modern TA methods and instrumentation as well as special application questions.

We would like to thank all participants and congratulate NKS, assuring our full support for future workshops on Thermal Analysis.



Participants of the workshop in Taejon

New on the bookshelf: **"Thermoanalytical Characterization of Pharmaceuticals"**

E. Marti, E. Kaisersberger, G. Kaiser, W.-Y. Ma

Thermal Analysis plays a central role for quality assurance and for characterization of drug substances and related products. It yields important information on melting points, heats of melting, polymorphous transitions, solvent and water content in crystalline solids, thermal or oxidative stability, eutectic purities, etc.

The application volume "NETZSCH Annual 2000: Thermoanalytical Characterization of Pharmaceuticals", which is available in English, gives an insight into the

various possibilities of Thermal Analysis in the pharmaceutical sector. The Annual features mainly DSC measurements extended by TG, TG-FTIR or TG/DSC-MS tests when it was necessary. Physical-chemical, kinetic and thermodynamic evaluation methods are presented to increase the effectiveness of the thermoanalytical data.

Twenty-three substances frequently used in pharmaceutical formulations are featured. The main emphasis, in the experimental section, was placed on the selection of measuring

conditions as well as pre-treatment of the samples. One chapter has been dedicated to the comparison of DSC results and the classical capillary method for determination of the melting point.

The Annual can be ordered via our web site Home Page at www.ngb.netzsch.com, "What's Hot" or contact Silke Popp (phone +49 9287 881 31, fax +49 9287/881 44, e-mail s.popp@ngb.netzsch.com).

The price of the "NETZSCH Annual 2000" is 49 euro (+ VAT and dispatch).

Contents

- TA-MS and TA-FTIR Workshops in Korea
- New on the bookshelf: "Thermoanalytical Characterization of Pharmaceuticals"
- Citius, altius, fortius ...
- Safe on French Roads Thanks to Thermal Analysis
- New Business Partners for Distribution of our Instruments in Greece and Spain
- Proceedings of the SKT 2000
- Trade Fairs and Symposia
- By the way

Citius, altius, fortius ...

This Olympic motto (faster, higher, stronger) also applies to the DSC 204 Phoenix®: fast heating and cooling rates and now, an even higher sensitivity.

One of the main features of the DSC 204 Phoenix® is its flexibility. Depending on users' requirements, a variety of exchangeable sensors, tailored to the corresponding application, can be employed.

After the introduction of the Phoenix® in 1997, its τ sensor has quickly established itself on the market. It features an extremely short time constant guaranteeing an excellent resolution of peaks that occur in close proximity.

We are pleased to present a semi-conductor sensor (see also ONSET edition of September), which distinguishes itself with a sensitivity approximately 15 times higher compared to conventional systems. Until recently, this sensational increase seemed impossible since it is generally recognized

that the law of physics does not allow a simultaneous increase in sensitivity with only a slight increase in time constant. The breakthrough was finally achieved in cooperation with a well-known German institute for aerospace technology.

On account of its working temperature range of between -150 and 400°C, the sensor is ideally suited for applications in the pharmaceutical, food, chemistry and polymer fields. The new sensor can fully utilize its abilities for detecting smallest impurities in trace analysis as well as for measuring weak or secondary transitions, especially in the low-temperature range.

The differences in sensitivity of the individual sensor systems are clearly seen in the $\mu\text{V}/\text{mg}$ presentation (Figure 1), i.e. display of the raw data. By conducting an enthalpy calibration (conversion of $\mu\text{V}/\text{mg}$ to mW/mg), all signals would be standardized to the same unit. Figure 1 clearly depicts the considerably

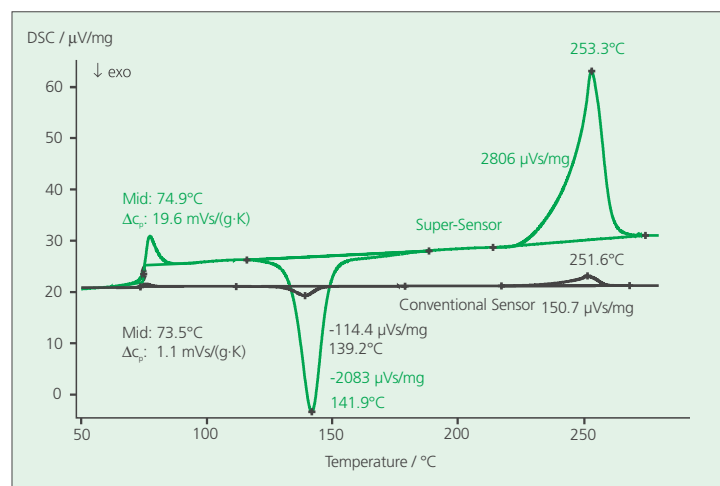


Figure 1. Comparison of the sensitivity on thermal effects of PET (green: new DSC sensor, black: conventional sensor)

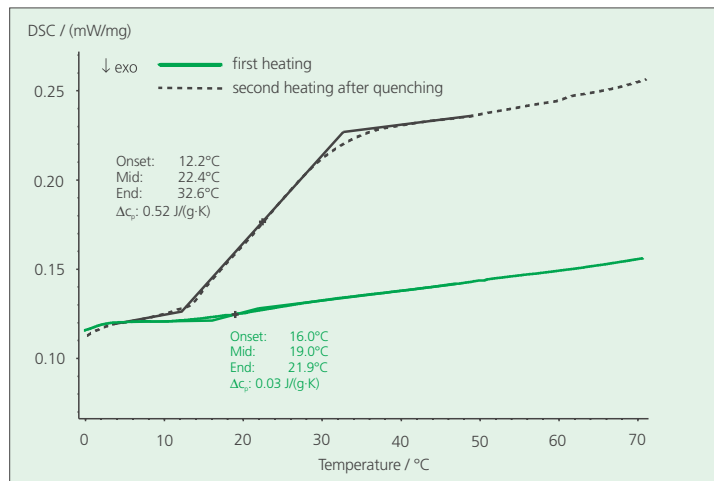


Figure 2. α -Lactose monohydrate, detection of amorphous parts (1st heating and 2nd heating after quenching)

higher sensitivity of the new sensor with the endo- and exothermal effects of an amorphous PET (polyethylene terephthalate).

By optimization of the signal-to-noise ratio combined with the high resolution electronics TASC 414/4, even the smallest effects can be detected with the new sensor.

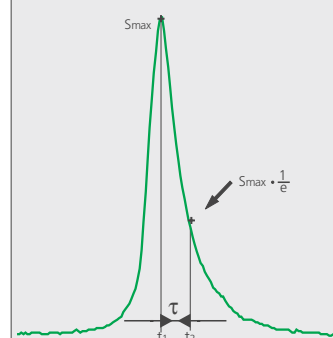
A readily available α -lactose monohydrate (milk sugar) contains a small percentage of amorphous parts which cannot usually be detected with a DSC measurement.

With the new sensor, analysis of the amorphous parts is not a problem, even when high-pressure crucibles are employed.

The T_g was detected at 19.0°C (midpoint) with a ΔC_p steps of only 0.03 J/(g·K).

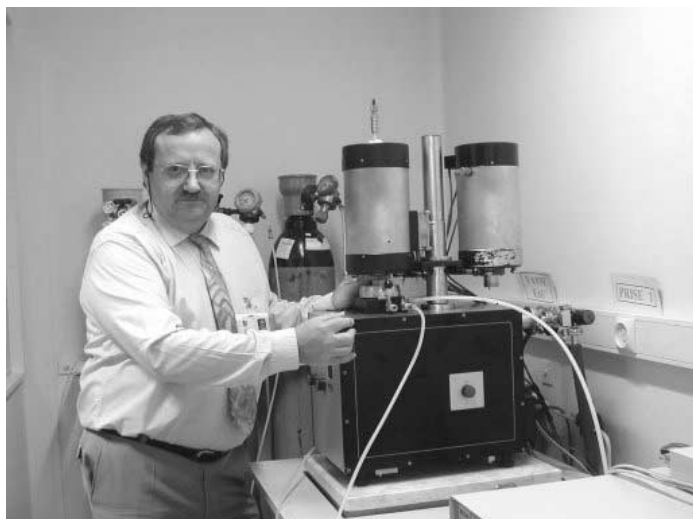
Quantitative evaluation of the amorphous parts is made by comparing the T_g step heights of the raw material and those of a quenched sample.

The time constant τ is a measure of resolution. The smaller the time constant, the narrower the corresponding peak; overlapping peaks occur very seldom.



The sensitivity is a measure of the signal intensity. The more sensitive the system, the higher the peaks.

Safe on French Roads Thanks to Thermal Analysis



Mr. Platret with the NETZSCH STA 409 E

LCPC (**L**aboratoire **C**entral des **P**onts et **C**haussées) is the name of the French institute for applied research in the building industry. 600 employees with 250 scientists and 220 engineers are working in four facilities, located in Nantes, Marne-la-Vallée, Satory-Versailles and Paris. Their main research activities are focused on roads, bridges, geo techniques and environmental aspects.

The aim of LCPC is to forge links between science and practical application. The institute cooperates with numerous state laboratories (for example CNRS = **C**entre **N**ational de la **R**echerche **S**cientific), universities, building constructors and AFNOR (**A**ssociation **F**rançaise de **N**ormalisation), the French society for standards. LCPC is one of the leading reference laboratories in France.

On account of their comprehensive set of technical equipment and the variety of testing methods, LCPC has succeeded in continuously expanding their activity fields.

Thermal Analysis plays a key role here. G. Platret, in charge of Thermal Analysis at LCPC (see photo) carries out more than 250 experiments per year with his STA 409 E and DSC 200. A variety of materials such as clays, soils, slags, fly ashes, concretes, paints, asphalts, geo membranes (made of polymers such as polyethylene or polypropylene) are tested.

Thanks to the versatility of Thermal Analysis, not only the classical determination of temperatures (for example, melting temperatures, glass transition temperatures), but also complex studies on polymer additives in asphalt and bitumen (analysis of the crystalline content, ageing of polymers) are possible. Moreover, you can trace back the history of concretes (clinker bricks, alkali-silicate reactions, ect.).

The presented examples describe two typical applications in the fields of failure analysis and characterization of building materials.

Figure 1 depicts a TG/DSC measurement on a calcium and silicate-containing concrete. The DSC peaks for quartz and calcite at 578°C and 793°C, respectively, can clearly be seen. The following three successive dehydration steps occurred:

- at 117°C (peak temperature of the derivative DTG): water evolvment from the C-S-H-phases ($\text{CaO-SiO}_2\text{-H}_2\text{O}$)
- at 179°C: Dehydration of $\text{Ca}_4\text{Al}_2\text{O}_6\text{CO}_2\cdot 11\text{H}_2\text{O}$
- at 402°C: water evolvment due to an alkali-silicate reaction

As a result of the water evolvment, Portlandit can, for example, be identified by the DSC peak at 498°C.

Another characteristic temperature occurred at 901°C (DSC peak temperature): Formation (exothermal reaction) of wollastonit from the C-S-H phases, dehydrated at 117°C.

Shown in figure 2 is a comparison of two DSC measurements on bitumen. While the curve of the polymer modified sample (no. 2) clearly reveals two endothermal effects at 115°C and 118°C (peak temperatures), the curve of the non-modified bitumen sample (no. 1) shows no effect in this range. The corresponding melting temperatures indicate an admixture of polyethylene.

We would like to thank Mr. Platret for providing us with this contribution.

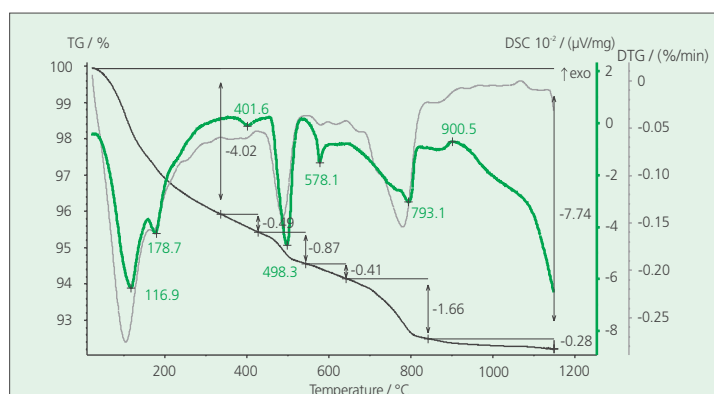


Figure 1. STA measurement on a concrete

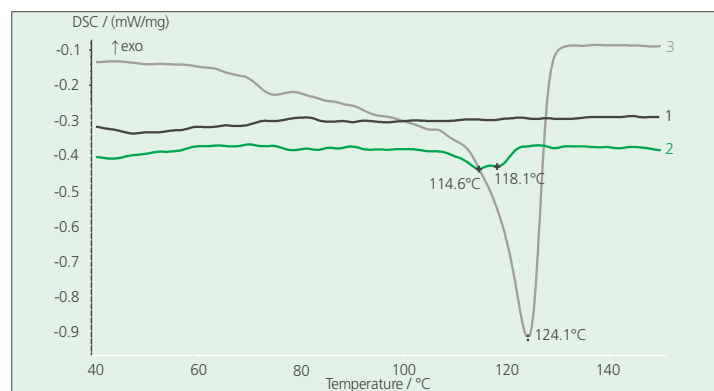


Figure 2. Comparison of two DSC measurements on bitumen (1 and 2); DSC measurement on pure PE (3)

New Business Partners for Distribution of our Instruments in Greece and Spain

Until recently we have begun exclusive cooperation with **Alfa Analytica Instruments**, Athens, headed by D. Haloulos, for the distribution and service of our instruments in Greece. We hope that we will successfully maintain existing customer relations as

well as establish new contacts.

In addition, we have started a partnership with **Bruker Espanola**, Madrid, headed by Mr. Victor Pidal, regarding distribution and service of our entire product line in Spain. Mr. Angel Sanz, responsible



Angel Sanz, Bruker Espanola

Trade Fairs, Symposia

We will attend and have exhibits at:

PittCon USA	04. - 09.03.01, New Orleans, LA
BRASILPLAST Brazil	05. - 10.03.01, Sao Paulo
EUROLAB 2001 Poland	07. - 09.03.01, Warsaw
CHEM 2001 Greece	09. - 13.03.01, Athens
Salon du Laboratoire France	13. - 16.03.01, Paris
EXPOFARMA Mexico	17. - 20.04.01, Mexico City
ACerS USA	22. - 25.04.01, Indianapolis, IN
Hannover Messe 2001 Germany	23. - 28.04.01, Hannover
ACHEMASIA 2001 PR China	08. - 12.05.01, Beijing
PM2TEC USA	13. - 17.05.01, New Orleans, LA
CERAMICS CHINA 2001 PR China	14. - 17.05.01, Guangzhou
45th Congresso Brasileiro de Ceramica Brazil	30.05. - 02.06.01, Florianopolis
CHINAPLAS PR China	26. - 29.06.01, Guangzhou
ANALTICIA 2001 Brazil	24. - 26.07.01, Sao Paulo
5th Brazilian Symposium on Glass Brazil	15. - 18.08.01, Foz d'Iguacu
NATAS USA	24. - 26.09.2001, St. Louis, MO

for the NGB product line, was trained as a product specialist in Selb.

The technical services staff of both countries have undergone intensive training courses on installation, maintenance and operation of our thermoanalytical instruments.

Proceedings of the SKT 2000

The proceedings of the 3rd SKT, held in Bad Orb, Germany (28 through 30 May 2000) are available under the title "Hyphenated Techniques in Thermal Analysis - 3rd SKT 2000".

The price is 39 euro (+ VAT and dispatch). The order form can be found on the internet

www.skt2000.com/skt2000/proceedings.htm

or contact Ms. Silke Popp (fax: +49 9287 881-44, e-mail: s.popp@ngb.netzsch.com).

by the way

... this newsletter is also available now in electronic form.

You can obtain the latest ONSET as well as the issues of April and September 2000 under the category "**What's Hot**" on our homepage

www.ngb.netzsch.com.

The corresponding pdf files can be opened with any version of the Adobe Acrobat® Reader. If this program is not yet installed on your computer, you can download this software free of charge by using the "Get Acrobat® Reader".

We hope you enjoy browsing through our newsletters!

Your ONSET Team

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