Precision of DSC Measurements: Results of Interlaboratory Tests

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The determination of the precision or uncertainty of a measurement is a key problem of almost any analytical method. The correlation of measured data with real facts, the method validation, is inherently not trivial in most cases. One possibility to realize such a validation is to perform Interlaboratory Tests. Comparing data, generated from different laboratories on the same sample, afford a lot of valuable information regarding precision of this certain analytical method. In order to suppress biased behavior and to obtain a high independency Interlaboratory Tests are often organized by neutral federal institutions like Empa (Swiss Federal Institute for materials testing and research), BAM (German Institute for materials research) or DACH (German Accreditation for Chemistry).

Empa organizes Interlaboratory Tests on polymeric materials on a biennial basis. The participants are usually industrial laboratories and laboratories at institutes that test, research and develop polymeric materials. Several Interlaboratory Tests with differential scanning calorimetry (DSC) was realized in the past few years. Besides glass transition (T_g) and melting point (T_m) measurements, investigations regarding oxidation processes (OIT), curing and crystallization was made as well. The measured data was collected by Empa and evaluated using a robust statistical method. Repeatability and reproducibility data, which describes the precision of a method mathematically, are of special interest.

The most important factors that produce deviations between individual measured results are: - the operator, - the equipment and the analytical instruments, - the calibration of the equipment and the instruments and - the environmental effects during the test procedure (i.e. influence of temperature, humidity, light, pollution etc.). These factors are mathematically described by the robust **standard deviation of repeatability s_r** (factors a), b), c) and d) are identical; repeatability conditions) and the robust **standard deviation of reproducibility s_R** (factors a), b), c) and d) are varied; reproducibility conditions).

The five already mentioned DSC methods: - oxidation induction time and temperature (OIT_{time}/OIT_{temp}), - determination of glass transition temperature (T_g), - determination of melting point (T_m) and crystallization and - curing of adhesives are presented in the lecture in detail. The methods will be introduced in principle and the calculated essential statistic values about the individual precision of every method will be discussed as well as the statistic data in over-all comparison.

At a glance, the DSC Interlaboratory Tests have shown clearly, that it must be differentiated between one point temperature measurements (T_g, T_m, OIT_{temp}) and evaluations, which requires user-dependent attitudes (e.g. definition of base lines and integration limits). For the “pure” temperature measurements tests good agreements for repeatability (s_r) and reproducibility standard deviation (s_R) was found. If special cases, like Polyamide during the glass point determination are neglected, s_r is in-between 0.3°C and 1.0°C and s_R within the range of 1.0°C to 2.1°C. The evaluation of data which requires user defined manner increases the values for repeatability (s_r) and reproducibility standard deviation (s_R) significantly.