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**Committee E37 on Thermal Measurements
Subcommittee E37.09 on Biological Calorimetry**

Research Report: E37-1046

**Interlaboratory Study to Establish Precision Statements for ASTM
E2603-15, Standard Practice for Calibration of Fixed-Cell Differential
Scanning Calorimeters**

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1. Introduction:

No formal Interlaboratory Study was conducted to establish a precision statement for E2603-2008, Standard Practice for Calibration of Fixed-Cell Differential Scanning Calorimeters. Instead, data contained in two interlaboratory studies published in the literature is used [1, 2]. Section 12 Precision and Bias for the E2603-2008 version of the standard reads.

12. Precision and Bias

12.1 Repeatability (Single Analyst):

12.1.1 Phosphatidylcholines: Studies conducted by F. P. Schwarz³ at the National Institute of Standards and Technology showed run-to-run reproducibilities of ± 0.1 K in the transition temperatures of the dialkylphosphatidylcholines of Table 1, when these measurements were performed in the range of scan rates for which the instrument showed no scan-rate dependence of the measured temperatures.

12.1.2 Chicken egg white lysozyme: An interlaboratory study, which incorporated several laboratories and several different models of calorimeters was conducted in the 1990s⁴. This interlaboratory study was conducted elsewhere and does not correspond to ASTM interlaboratory study protocols. Regardless, it suffices to give preliminary intralaboratory reproducibility estimates. The standard deviations of the means of the integrated enthalpies of reaction from four laboratories ranged from 0.3 % to 2 %. The unbiased standard deviations of the populations from the four laboratories ranged from 0.8 % to 4.5 %. The standard deviations of the means of the enthalpies of reaction calculated from the van't Hoff relation ranged from 0.2 % to 1.5 % and the standard deviations of the populations from the four laboratories ranged from 0.5 % to 3.2 %.

This statement is not in the appropriate style for an ASTM standard and does not provide users with the information needed to assess the precision and bias of their own work. It is the purpose of this study to re-examine the data from the referenced literature and to apply methods of ASTM Standards E1970 *Practice for Statistical Treatment of Thermoanalytical Data* and modified standard E691 *Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method* to obtain a Precision and Bias Statement in a style more consistent with the ASTM practice.

2. Test Method:

The Test Method used for this ILS is E2603-15. To obtain a copy of E2603-15, go to ASTM's website, www.astm.org, or contact ASTM Customer Service by phone at 610-832-9585 (8:30 a.m. - 4:30 p.m. Eastern U.S. Standard Time, Monday through Friday) or by email at service@astm.org.

3. Participating Laboratories:

For the study on temperature [1], a data from a single laboratory was used.

¹ Schwarz, F. P., *Thermochimica Acta*, Vol 177, 1991, pp.285–303.

² Hinz, H.-J., F. P. Schwarz, *Pure and Applied Chemistry*, Vol 732001, pp. 745–759.

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For the work on enthalpies of transition [2], data from five laboratories, found in the referenced publication, is used. This data is identified by laboratory number and apparatus type (see Table 1) but not by contributor.

4. Description of Apparatus

Data for the temperature precision study [1] was obtained on a Hart 7707 differential heat conduction scanning microcalorimeter (Hart Scientific, Provo UT now TA Instruments, New Castle DE) calibrated using internal heaters and checked by comparison of the heats of fusion of o-terphenyl and diphenyl ether [3].

Data for the enthalpy study was obtained on the five apparatus described in Table 1. The instruments were calibrated according to the manufacturer's recommendation usually using internal heaters.

Table 1 -Apparatus Used in Enthalpy Interlaboratory Study

Laboratory	Apparatus	Manufacturer
1	NanoDSC	TA Instruments
2	VPDSC	MicoCal
3	MC-2	MicoCal
4	SCAL-1	Russian Academy of Sciences [4]
5	DASM-4	V/O Mashpriborintorg

5. Test Materials

- 5.1 For the temperature measurements, a series of 10 % water suspension of di-alkylphosphatidylcholine were used. The suspensions were buffered at pH 7.0. The test materials were obtained from Avanti Polar Lipids Inc (Alabaster AL) with a stated purity of >99 %. The phosphocholines used include DTPC = 1,2-ditridecanoyl-sn-glycero-3-phosphocholine, DMPC = 1,2-ditetradecanoyl-sn-glycero-3-phosphocholine, DPPC = 1,2-dihexadecanoyl-sn-glycero-3-phosphocholine, DSPC = 1,2-dioctadecanoyl-sn-glycero-3-phosphocholine, DAPC = 1,2-dieicosanoyl-sn-glycero-3-phosphocholine, DBPC = 1,2-didocosanoyl-sn-glycero-3-phosphocholine, and DLPC = 1,2-ditetracosanoyl-sn-