This Research Report is issued under the fixed designation RR: F02-1015. You agree not to reproduce or circulate or quote, in whole or part, this document outside of ASTM International Committee/Society activities, or submit it to any other organization or standards body (whether national, international or other) except with the approval of the Chairman of the Committee having jurisdiction and the written authorization of the President of the Society. If you do not agree to these conditions, please immediately destroy all copies of this document. *Copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. All rights reserved.*

3 January 2001

Committee F02 on Flexible Barrier Packaging
Subcommittee F02.15 on Chemical/ Safety Properties

Research Report RR # F02-1015

Inter-Laboratory Study to Establish Precision Statements for ASTM F2013, Standard Test Method for Determination of Residual Acetaldehyde in Polyethylene Terephthalate Bottle Polymer Using an Automated Static Head-Space Sampling Device and a Capillary GC with a Flame Ionization Detector

ASTM International
100 Barr Harbor Drive
West Conshohocken, PA 19428-2959

Precision and Bias Report for ASTM F-2013

(Determination of Residual Acetaldehyde in Polyethylene Terephthalate Bottle Polymer Using an Automated Static Head-Space Sampling Device and a Capillary GC with a Flame Ionization Detector)

Summary
In January of 2000 a round-robin study was conducted in accordance with Practice E691-99, involving six materials tested by six laboratories. This study was conducted in conjunction with the International Society of Beverage Technologists sub-committee concerning standardization of ground preform AA testing.

Sampling – The six materials were from six cavities of a 48 cavitation Husky Injection Molding Machine selected for the test, some with high, some with medium, and some with low AA concentrations.
Multiple shots were collected from each cavity so as to have enough of a common sample from each cavity for all six labs. Each set of preforms from each of the selected cavities were cryogenically ground together and homogenized before loading in vials and sending out to the participating labs. Each sample was tested in triplicate fashion for each lab thus making 18 vials for testing.

Calibration - A common calibration vial set consisting of 3 vials containing 3 concentrations of acetaldehyde standard were also sent out to the labs for a 'common' calibration. The labs were to also use their own calibration curve which had been developed using their personal acetaldehyde standard.

Calculations - Labs were to calculate results based on their individual lab calibration curve and also on the calibration curve developed from the standards supplied by in the round robin.

Data Analysis
JMP® software V.4.0.0 was used for the data analysis. E691 V.2 software was also used to analyze the data and provide Tables 1 and 2 for the precision statement.
Attachment 1 illustrates the variation seen when each lab uses their own calibration solution. The 'preform-to-preform' variation is evident as expected but so is the 'lab-to-lab' and 'within lab' variation. The lab-to-lab variation is strongly illustrated in the Plot entitled "Difference in Labs After Accounting for Preform Differences".

Attachment 2 illustrates the variation seen when a common calibration is done. This plot shows Labs A & B as standing out from the rest but given the limited # of labs involved in the study it was decided to include them and treat this as part of the expected 'Lab-to-Lab' variation. Notice the decrease in 'Lab-to-Lab' variation (though not statistically significant with 5 df using an F-test, this is in the direction expected.). The 'preform-to-preform' and 'within lab' variation stays about the same as expected.

Observation:

The labs need to standardize on the number of decimal places to report.