



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 Detector¹

This standard is issued under the fixed designation F 2013; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a gas chromatographic procedure for the determination of the ppm residual acetaldehyde (AA) present in poly(ethylene terephthalate) (PET) homo-polymers and co-polymers which are used in the manufacture of beverage bottles. This includes sample types of both amorphous and solid-stated pellet and preform samples.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 4509 Test Method for Determining the 24-Hour Gas (Air) Space Acetaldehyde Content of Freshly Blown PET Bottles²

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

3. Terminology

3.1 The terms employed in this test method are commonly used in normal laboratory practice and require no special comment.

4. Summary of Test Method

4.1 A specified size (800 to 1000 μm) of granulated sample is weighed into a 20-mL head-space vial, sealed, and then heated at 150°C for 60 min. After heating, the gas above the

sealed sample of PET polymer is injected onto a capillary GC column. The acetaldehyde is separated, and the ppm of acetaldehyde is calculated.

5. Significance and Use

5.1 This test method is of particular use as a quality control tool for a molding or synthesis operation. Acetaldehyde is a volatile degradation product generated during melt processing of PET. Thus, it becomes trapped in the sidewalls of a molded article and desorbs slowly into the contents packaged therein. In some foods and beverages AA can impart an off-taste that is undesirable, thus, it is important to know its concentration in PET articles that are to be used in food contact applications.

5.2 The desorption conditions of 150 C for 60 min are such that no measurable AA is generated by the sample during the desorption process.

6. Sources of Error

6.1 A bias is known to exist if the ratio of sample mass (mg) to head-space vial volume (mL) exceeds a value of ten.

6.2 Acetaldehyde is very volatile and must be handled carefully to avoid sample loss during the calibration procedure. Storing the standard vials in a refrigerator is a must to minimize the error due to volatility.

6.3 Failure to achieve a tight seal on the head-space vial will result in the loss of acetaldehyde during storage and desorption, producing a false low value.

6.4 Failure to grind the sample to the appropriate particle size may lead to a false low value for residual AA due to the increased path length for desorption.

6.5 Samples submitted for 'residual AA measurement' should be stored in a freezer until they are tested. Failure to do so can result in lower than expected results.

6.6 Excessive grinding of samples can cause residual AA contained therein to be desorbed. Extensive excessive grinding can lead to actual melting of the polymer and AA generation. Samples which have been chilled in liquid nitrogen properly

¹ This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Materials and is the direct responsibility of Subcommittee F02.30 on Test Methods.

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² *Annual Book of ASTM Standards*, Vol 08.03.

³ *Annual Book of ASTM Standards*, Vol 14.02.

should only be in the grinder for ~30 s or less.

7. Apparatus

- 7.1 *Gas Chromatograph*, Hewlett-Packard 6890 series⁴
 7.2 *Integrator*, Hewlett-Packard Model 6890 series, H-P ChemStation, H-P LAN.⁴
 7.3 *Head-Space Sampler*, Hewlett-Packard 7694.⁴
 7.4 *Column*, 30-m by 0.53-mm inside diameter (megabore capillary column).⁵
 7.5 *Vials*, 20-mL, head-space, with 20-mm septa, 20-mm aluminum caps, and crimper for 20-mm caps
 7.5.1 *Vials*:
 Perkin-Elmer Part Numer 0105–0129⁶, or Kimble Glass Inc. Part Number 60827A-2375⁷, or Hewlett-Packard Part Number 5182–0837⁴.
 7.5.2 *Septa*:
 Pierce Part Number 12720, or Hewlett-Packard Part Number 9301–0807.⁴
 7.5.3 *Caps*:
 Pierce Part Number 13214, or Hewlett-Packard Part Number 9301–0721.⁴
 7.6 *Crimper*, 20-mm, Wheaton Part Number 224303.⁸
 7.7 *Decrimper*, 20-mm, Kimble Part Number 69903–20.⁷
 7.8 *Wiley Mill*, equipped with an 800 to 1000- μm screen, or equivalent
 7.9 *Syringe*, calibrated, with certificate of calibration.⁹
 7.10 *Small Vacuum Cleaner*, with host attachment for cleaning.
 7.11 *Analytical Balance*, capable of accurately weighing to at least ± 0.0001 g.
 7.12 *Hammer*.

8. Reagents and Materials

- 8.1 *Acetaldehyde (AA)*, 500 ppm AA in water (or 1000 ppm), purchased certified standard.¹⁰
 8.2 *Liquid Nitrogen*, plant grade (R-3, S-3).

⁴ Hewlett-Packard apparatus, website: hewlett-packard.com, was used in the development of this standard. If you are aware of alternative suppliers, please provide this information to ASTM headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

⁵ J & W Scientific GS-Q (Catalogue Number 115–3435), available from 91 Blue Ravine Rd., Folsom, CA, website: JWscientific.com, was used in the development of this standard. If you are aware of alternative suppliers, please provide this information to ASTM headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

⁶ Available from Perkin-Elmer, website: perkin-elmer.com.

⁷ Available from Kimble/Kontes, Vineland, NJ, e-mail: cs@kimble-kontes.com.

⁸ Wheaton Part Number 224303, available from 1501 North 10th St., Millville, NJ 08332, website: algroupwheaton.com, was used in the development of this standard. If you are aware of alternative suppliers, please provide this information to ASTM headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

⁹ Hamilton Digital with 2- μL capacity, Model Number 7002KH, was used in the development of this standard. If you are aware of alternative suppliers, please provide this information to ASTM headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

¹⁰ Protocol Analytical Supplies, Inc., Middlesex, NJ 08846 (R-3, S-2). If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

9. Calibration and Standardization

NOTE 1—The following procedure should be performed and recorded once every three months.

- 9.1 Break open a certified AA standard ampule (ampules must be stored in a refrigerator) or prepare AA standard by the attached supplemental procedure. (See Appendix X5.)
 9.2 Using the syringe, fill it by placing the tip in the liquid standard and quickly moving the plunger up and down several times to evacuate any bubbles, then pull the plunger back past the 2.000- μL mark to 2.200 to 2.250 μL .
 9.3 Wipe the syringe needle with a tissue.
 9.4 Depress the plunger until the digital readout is 2.000 μL .
 9.5 Smear the excess liquid that is on the syringe tip on the OUTSIDE of the headspace vial.
 9.6 Place the syringe inside of the vial so that the tip just touches the bottom of the vial.
 9.7 Quickly inject the liquid standard into the vial and swirl the syringe tip around the inside of the vial to smear all liquid on the vial walls.
 9.8 Remove the syringe and IMMEDIATELY cap the vial.
 9.9 Calculate the weight of AA based on the standard's certified value and a 2.000- μL injection volume.

NOTE 2—Acetaldehyde is very volatile. The AA ampules must be stored in a refrigerator, and the standards prepared immediately after breaking open an ampule.

- 9.10 Analyze the working standard by the procedure described in Section 11, starting with 11.2.11.
 9.11 Calculate an AA response factor for the standard using the following equation:

$$\text{response factor of AA} = \text{Wt of AA in } \mu\text{g/area of AA} \quad (1)$$

NOTE 3—Due to the error associated with the certified standard, 9.1-9.11 should be performed five times using five different standard ampules.

- 9.12 Average the five response factors obtained, and use this value for the sample analyses.
 9.13 Manually enter the calculated response factor in the calibration list of the Hewlett-Packard 6890⁴ integrator or data system.

NOTE 4—During a series of sample analyses, a periodic check of instrument performance is recommended by placing a few liquid standard samples throughout the sample set. If these values fall out of the acceptable range as specified by the certificate of analysis, recalibration (9.1-9.12) should be performed.

10. Sample Preparation

10.1 *Parisons or Preforms or Plaques*—May be cryogenically ground whole, or can be broken into small pieces with a hammer (using liquid nitrogen) and then ground with the aid of grinding mill equipped with a 20-mesh or 850- μm screen. The grind should be thoroughly homogenized before sampling for AA. If the appropriate size screen is not available on the large grinding mill, then it is suggested that the sample be ground to 3 to 6 mm on the large mill and the sample thoroughly homogenized. A portion can then be taken to a smaller mill equipped with the 20-mesh or 850- μm screen and cryogenically ground again before analysis. Again the final sample should be thoroughly homogenized.

10.2 *Pellets*—May be cryogenically ground in a small grinding mill using liquid nitrogen. The final sample should be thoroughly homogenized before sampling for analysis.

NOTE 5—Samples, either preforms, plaques, or pellets, should be chilled in the liquid nitrogen for several minutes until the liquid nitrogen stops boiling and then dropped immediately into the grinder. Sample should be sufficiently ground in a few seconds. The grinder should not be allowed to operate more than 20 to 30 s as in such cases undesirable sample heating can occur.

11. Procedure

NOTE 6—Refer to the general operating manual for the Hewlett-Packard 6890⁴ gas chromatograph, the Hewlett-Packard 7694⁴ head-space sampler, and the Hewlett-Packard 6890⁴ series integrator for instructions in performing steps in this procedure.

11.1 Adjust the H-P 6890⁴ gas chromatograph to the conditions specified in Appendix X1. Adjust the H-P 7694⁴ head-space sampler to the conditions in Appendix X2. Set the H-P 6890⁴ series integrator to the conditions in Appendix X3.

11.2 *Sample Analysis:*

11.2.1 Place 2 to 3 of polymer pellets (or crushed preform) into a small Dewar flask.

11.2.2 Cover the polymer with 20 to 40 mL of liquid nitrogen.

11.2.3 Allow the polymer to chill under the liquid nitrogen for approximately 3 min (or until most of the liquid N₂ has evaporated).

11.2.4 Turn on the Wiley mill equipped with a 800 to 1000- μ m screen.

11.2.5 Slowly pour the remaining liquid nitrogen from the Dewar flask through the Wiley mill, followed by the chilled polymer sample (tapping the sample may be required).

11.2.6 Collect the ground polymer in a small glass jar or small manila envelope.

11.2.7 Turn off the Wiley mill and clean it with a vacuum cleaner.

11.2.8 Allow the ground polymer sample to come to room temperature (approximately 10 min).

11.2.9 Weigh approximately 0.2000 (\pm 0.0200) g, recorded to the nearest 0.0001 g, into a 20-mL head-space vial.

11.2.10 Place a septum (with Teflon side down towards the inside of the vial) on the vial. Place an aluminum cap on top of the septum, and crimp the cap with a crimper UNTIL THE CAP CANNOT BE TURNED. Remove the center piece of the aluminum cap (if it exists).

11.2.11 Place the vial in the appropriate position in the head-space sampler.

11.2.12 Press the “Vial Parameter” key on the H-P 7964⁴ head-space sampler, and enter the starting and stopping vial positions.

11.2.13 Press the “START” button on the H-P 7694⁴ head-space sampler.

11.2.14 The head-space sampler will heat the sample for 60 min at 150°C and then automatically inject the head-space gas and start the gas chromatograph and integrator.

11.2.15 The final report will appear on the H-P 6890⁴ integrator or the data system when the GC is finished. This report will give the micrograms of AA.

11.2.16 To determine the concentration in ppm of AA in the

polymer sample, divide the micrograms of AA (reported in 11.2.15) by the sample weight in the vial (recorded in 11.2.9 as grams of polymer).

12. Calculation

12.1 The AA response factor is calculated as described in 9.11 and 9.12. The ppm of AA can be calculated manually by multiplying the response factor and the area of the AA peak, and then dividing this number by the sample weight in the vial (in grams).

13. Report

13.1 Report the ppm of AA to two decimal places.

14. Precision and Bias

14.1 The following was taken from work completed by the International Society of Beverage Technologists (ISBT) subcommittee concerning standardization of method to determine residual AA in PET.

14.2 The number of laboratories, materials, and determinations in this study meets the minimum requirements for determining precision in accordance with Practice E 691. A complete report is on file at ASTM Headquarters.¹¹

14.3 This round robin was conducted by having one laboratory mold PET preforms on a 48-cavity injection molding machine and selecting 6 of those cavities as the sample set. Even though these preforms all came from one PET sample (material), each cavity has its own unique AA value, and thus, were treated as six different materials. Also, two different types of precision and bias were calculated, one based on each laboratory using their own calibration standard solution and another when each laboratory calibrated with a “common” calibration standard.

	Practice E 691 Study	Minimum
Laboratories:	6	6
Materials:	6	4
Determinations:	3	2

14.4 *Precision and Bias With Each Laboratory Using Their Own Calibration Standard*—Precision, characterized by repeatability, *Sr* and *r*, and reproducibility, *SR* and *R*, has been determined for the materials to be as follows:

Materials	Average	<i>Sr</i>	<i>SR</i>	<i>r</i>	<i>R</i>
Material A	5.21	0.1812	0.6403	0.5074	1.7928
Material B	6.25	0.4060	0.7464	1.1368	2.0899
Material C	6.37	0.2880	0.6713	0.8066	1.8796
Material D	7.21	0.3285	0.7743	0.9198	2.1680
Material E	7.01	0.4217	0.8350	1.1808	2.3380
Material F	5.88	0.3930	0.7168	1.1003	2.0071

14.4.1 Since the materials used in this study are all from one specific type of material (PET), but have different AA levels because they are from different cavities, it makes more sense to have one set of precision values rather than one for each cavity. This will be derived by squaring each *Sr* and *SR*, averaging each of *Sr*² and *SR*² across materials and taking the square root.

<i>Sr</i>	<i>SR</i>	<i>r</i>	<i>R</i>
0.3466	0.7335	0.9705	2.0538

¹¹ A research report is available from ASTM headquarters. Request RR:F02-1015.

14.4.1.1 *Standard Deviation (Sr)*—*Sr* is the square root of the average within laboratory variance.

14.4.1.2 *Standard Deviation (SR)*—*SR* is the square root of the sum of the within laboratory variance and between laboratory variance of the laboratory means.

14.4.1.3 *Repeatability—r* is the interval representing the largest expected difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory. A difference larger than '*r*' indicates more variation is present than expected.

14.4.1.4 *Reproducibility—R* is the interval representing the largest expected difference between two test results for the same material, obtained by different operators using different equipment in different laboratories, not necessarily on the same day. A difference larger than '*R*' indicates more variation is present than expected.

14.5 *Precision and Bias When Each Laboratory Uses a Common Calibration Standard*—Precision, characterized by repeatability, *Sr* and *r*, and reproducibility, *SR* and *R*, has been determined for the materials to be as follows:

Materials	Average	<i>Sr</i>	<i>SR</i>	<i>r</i>	<i>R</i>
Material A	5.42	0.1849	0.4128	0.5178	1.1596
Material B	6.47	0.4123	0.6438	1.1545	1.8026
Material C	6.59	0.2703	0.5020	0.7567	1.4057
Material D	7.45	0.3113	0.6333	0.8716	1.7732
Material E	7.26	0.4014	0.5747	1.1240	1.6090
Material F	6.10	0.3854	0.5085	1.0792	1.4237

14.5.1 Since the materials used in this study are all from one specific type of material (PET), but have different AA levels because they are from different cavities, it makes more sense to have one set of precision values rather than one for each cavity. This will be derived by squaring each *Sr* and *SR*, averaging each of *Sr*² and *SR*² across materials and taking the square root.

<i>Sr</i>	<i>SR</i>	<i>r</i>	<i>R</i>
0.3376	0.5518	0.9453	1.5450

14.6 *Bias*—There are no recognized polymer standards by which to estimate bias of this test method. Testing a known liquid standard with all laboratories using common calibration did not show any laboratory bias between laboratories or between the average of all laboratories and the known value.

15. Keywords

15.1 acetaldehyde; carbonated soft drink; ground parison AA; PET bottles; 24 hours headspace; AA test; water

APPENDIXES

(Nonmandatory Information)

XI. HEWLETT-PACKARD 6890⁴ SERIES GC CONDITIONS

TABLE X1.1 Hewlett-Packard 6890⁴ Series GC Conditions

Temp 1 Isothermal	90°C
Time 1	8.00 min
Injector Temperature	250°C
Detector Temperature	250°C
Head Pressure	10 psi
Column-Flow	12.2 mL/min Helium
Velocity	85 cm/s
Split Ratio	2.0
Detector Air Flow	300 mL/min
Detector Hydrogen Flow	30 mL/min
Detector Makeup Flow	20 mL/min helium

X2. HEWLETT-PACKARD 7694⁴ HEAD-SPACE SAMPLER CONDITIONS**TABLE X2.1 Hewlett-Packard 7694⁴ Head-Space Sampler Conditions**

Oven Temp	150°C
Loop Temp	160°C
Transfer Line Temp	170°C
Carrier Pressure	11.5 psi
Vial Pressure	10.5 psi
Vial Eq. Time	60 min
Pressurize Time	0.2 min
Loop Fill Time	0.2 min
Loop Eq. Time	0.1 min
Inject Time	0.2 min
GC Cycle Time	9 min

X3. HEWLETT-PACKARD 6890 SERIES⁴ INTEGRATOR METHOD FILE

* LIST: METH @

RUN PARAMETERS

ZERO = 0
ATT 2^ = 0
CHT SP = 0.5
AR REJ = 200
THRSH = -2
PK WD = 0.04

TIMETABLE EVENTS
EMPTY

CALIBRATION
ESTD

REF % RTW: 5.000 NON-REF % RTW: 5.000

LEVEL: 1 RECALIBRATIONS: 3

CAL# RT LV AMT AMT/AREA
1R 2.545 1 1.5030E+00 1.4933E-04

CAL# NAME
1 ACETALDEHYDE

INTEGRATION PLOT TYPE FILTERED
Presentation plot NO

RUN DATA STORAGE
Store signal data YES
Device M
Bunched or raw data RAW
Store processed peaks NO

CALIBRATION OPTIONS
RF of uncalibrated peaks 0.0000E+00
Calibration fit P
Disable post-run RT update .. NO
SAMPLE AMT 1.0000E+00
MUL FACTOR 1.0000E-02

REPORT OPTIONS
Suppress local report NO
HEIGHT% report NO
Report uncalibrated peaks ... YES
Extended report YES

PRINT & POST-RUN LIST OPTIONS
Large font YES
Store post-run report NO
External post-run report NO
List run parameters NO
List timetable NO
List calibration table NO
List remote method NO
Form-feed before report NO
Form-feed after report NO
Skip perforations in report . NO
Skip perforations in plot ... NO
RANGE: C1,BUFFER 5!, 5!
INJECTOR 1 (not installed)
INJECTOR 2 (not installed)
TRAY (not installed)

FIG. X3.1 Hewlett Packard 6890 Series⁴ Integrator Method File

```

HP 6890 Series Gas Chromatograph 14 Feb 96 10:30
OVEN                                OVEN TEMPERATURE PROGRAM
  Temp ..... 90 deg C      # Rate Final temp Final time
  Initial time ..... 8.00 min  - ----  ---  ----
  Maximum temp ..... 450 deg C    1 0.00 (off)
  Equib time ..... 2.00 min
CRYO (not installed)
FRONT INLET (Split/Splitless)      BACK INLET (Split/Splitless)
  Mode ..... Split              Mode ..... Split
  Temp ..... 250 deg C          Temp .....250 deg C
  Pressure ..... 10.0 psi       Pressure ..... 10.0 psi
  Split ratio ..... 50.0        Split ratio ..... 2.0
  Split flow ..... 87.7 mL/min   Split flow ..... 25 mL/min
  Gas saver ..... Off           Gas saver ..... Off
  Gas type ..... He             Gas type ..... He
COLUMN 1                            COLUMN 2
  Length ..... 30.0 m          Length ..... 30.0 m
  Diameter ..... 320 u          Diameter ..... 530 u
  Film thickness ..... 0.00 u    Film thickness ..... 5.00 u
  Inlet ..... Front            Inlet ..... Back
  Detector ..... Front          Detector ..... Back
  Vacuum correct ..... Off      Vacuum correct ..... Off
  Pressure correct ..... Off    Pressure correct ..... Off
  Pressure ..... 10.0 psi       Pressure ..... 10.0 psi
  Flow ..... 1.8 mL/min         Flow ..... 12.2 mL/min
  Velocity ..... 32 cm/sec      Velocity ..... 85 cm/sec
  Mode ..... Constant pressure  Mode ..... Constant pressure
FRONT DET (Flame Ionization)        BACK DET (Flame Ionization)
  Temp ..... 250 deg C          Temp .....250 deg C
  H2 flow ..... Off             H2 flow ..... 30.0 mL/min
  Air flow ..... Off            Air flow ..... 300 mL/min
  Mode ..... Constant makeup    Mode ..... Constant makeup
  Makeup flow ..... Off         Makeup flow ..... 20 mL/min
  Makeup gas ..... He           Makeup gas ..... He
  Flame ..... Off               Flame ..... On
  Lit offset ..... 2.0 pA       Lit offset ..... 2.0 pA
  Electrometer ..... On        Electrometer ..... On
SIGNAL 1                             SIGNAL 2
  Type: ..... Back              Type: ..... Back
  Zero ..... 0.0                Zero ..... 0.0
  Range ..... 0                  Range ..... 0
  Attn ..... 0                   Attn ..... 0
COLUMN COMP 1 (no data)              COLUMN COMP 2 (no data)

```

FIG. X3.1 Hewlett Packard 6890 Series⁴ Integrator Method File (continued)

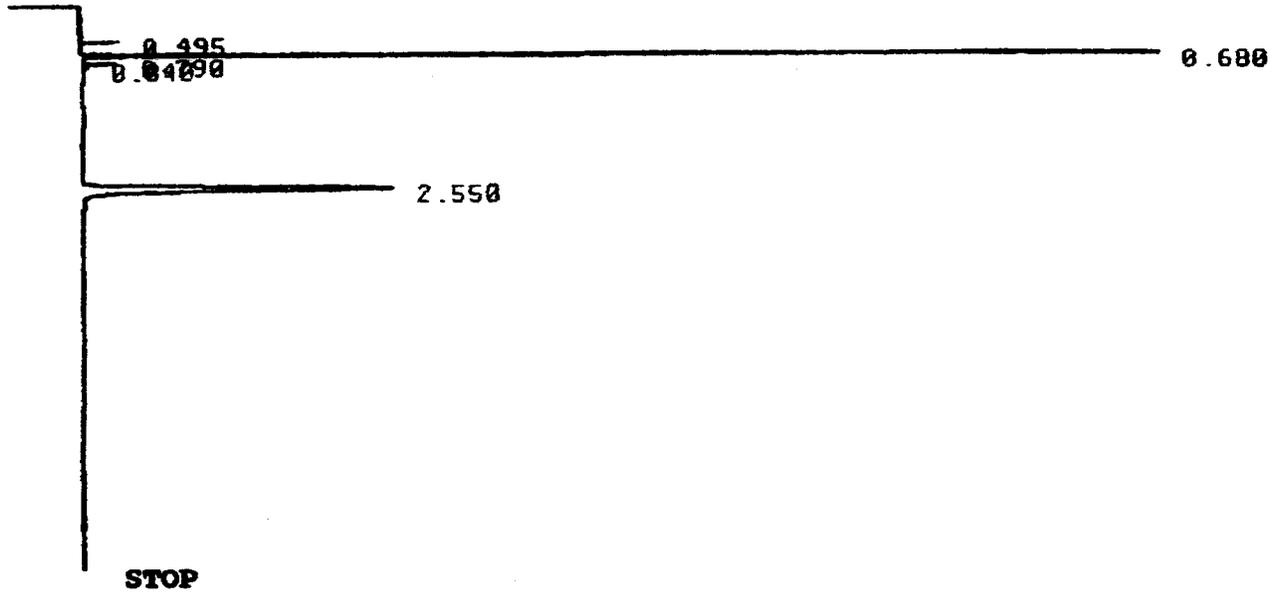
AUX 1 VALVES
Temp Not installed Valve 1 Not installed
Rate Final temp Final time Valve 2 Not installed
- ---- --- ----- Valve 3 Not installed
AUX 2 Valve 4 Not installed
Temp Not installed Valve 5 Not installed
Rate Final temp Final time Valve 6 Not installed
- ---- --- ----- Valve 7 Not installed
AUX 3 Valve 8 Not installed
Pressure Off
Initial time 0.00 min
AUX 3 PRESSURE PROGRAM
Rate Final pres Final time
- ---- --- -----
1 0.00 (off)
AUX 4
Pressure Off
Initial time 0.00 min
AUX 4 PRESSURE PROGRAM
Rate Final pres Final time
- ---- --- -----
1 0.00 (off)
AUX 5
Pressure Off
Initial time 0.00 min
AUX 5 PRESSURE PROGRAM
Rate Final pres Final time
- ---- --- -----
1 0.00 (off)
RUN TABLE
No entries

*.
FIG. X3.1 Hewlett Packard 6890 Series⁴ Integrator Method File (continued)

X4. CHROMATOGRAM OF A TYPICAL SAMPLE

* RUN # 238 JUN 5, 1996 08:36:16

START



Closing signal file M:SIGNAL .RAW

RUN# 238 JUN 5, 1996 08:36:16

METHOD NAME: M*M18AAF.MET

SIGNAL FILE: M:SIGNAL.RAW

ESTD%-AREA

RT	TYPE	AREA	WIDTH	CAL#	AMOUNT	NAME
.680	PB	27112	.009		.000	
2.550	PB	9753	.070	1R	1.456	ACETALDEHYDE

TOTAL AREA= 36865

MUL FACTOR=1.0000E-02

SAMPLE AMT=1.0000E+00

FIG. X4.1 Chromatogram of a Typical Sample

X5. PREPARATION OF ACETALDEHYDE CALIBRATION STANDARDS

X5.1 Introduction

X5.1.1 The acetaldehyde (AA) calibration standards are used for calibrating gas chromatographs in the determination of AA present in PET preforms which are used in the food and beverage industry.

X5.2 Materials

X5.2.1 The materials for AA calibration standards are as follows:

- X5.2.1.1 Distilled water.
- X5.2.1.2 1-L Class A volumetric flask.
- X5.2.1.3 Sylon CT.¹²
- X5.2.1.4 Acetaldehyde.
- X5.2.1.5 Syringe (1 mL).
- X5.2.1.6 Amber caps (4 mL).¹³
- X5.2.1.7 Hole caps¹⁴ and TFE-fluorocarbon-faced septa.¹⁵
- X5.2.1.8 Glass tray.
- X5.2.1.9 Balance (capable of 1-mg readings).
- X5.2.1.10 Refrigerator, set at 4°C.

X5.3 SetUp

X5.3.1 Use distilled water that is stored in a container other than PET (that is, high-density polyethylene or polyvinyl chloride bottles)

X5.3.2 If this is the first time a 1-L Class A volumetric flask is being used or the flask has been used for something other than AA preparation, then proceed to X5.3.3. If the flask has been used previously for AA solution preparation, then proceed to X5.3.4.

X5.3.3 The flask must be deactivated which is done with Sylon CT, or equivalent.¹²

X5.3.4 Using the distilled water described in X5.3.1, fill the deactivated or previously used 1-L flask to the fill line using the meniscus.

X5.3.5 Place the flask in a refrigerator and allow it to cool and equilibrate to ~4°C.

X5.3.6 The following items should also be stored in the refrigerator at ~4°C including the acetaldehyde, a 1-mL syringe, a box of unopened 4-mL amber vials,¹³ and a glass tray.

X5.3.7 Prepare the caps by placing the TFE-fluorocarbon-faced septa¹⁵ in the aluminum 'hole-in-cap' lids¹⁴ (Supelco cat. # 2-712OU, or equivalent). Make sure that the TFE-fluorocarbon facing is on the inside so that it is between the rubber septum and the glass vial. Please note that caps do not have to be refrigerated.

X5.4 Procedure

X5.4.1 Remove the AA, syringe, and 1-L flask and place them beside the balance.

X5.4.2 Fill the syringe with AA, handling the syringe by the top, taking care not to touch the body of the syringe, thereby causing it to heat up. Place the syringe on the balance and tare it.

X5.4.3 Remove the glass stopper from the 1-L flask, then insert the tip of the syringe into the water and discharge the AA. Remove the syringe and touch the inside sidewall of the flask to remove any drops. Recap the flask and weight the empty syringe immediately and record the weight loss.

X5.4.4 Invert the flask and shake at least ten times to ensure proper mixing of the AA.

X5.4.5 Remove the glass tray and the box of vials from the refrigerator. Place no more than 10 vials in the tray. The purpose of the tray is to catch overflow material.

X5.4.6 Using the 1-L AA solution in the flask, remove the stopper and fill the vials to above the brim, then re-stopper the flask.

X5.4.7 Cap all of the vials with the caps prepared in X5.3.7. It is important to complete X5.4.6 and X5.4.7 as quickly as possible to prevent AA loss.

X5.4.8 Repeat X5.4.6 and X5.4.7 until the desired amount of vials have been filled, never filling more than 10 at a time.

X5.4.9 For every five vials prepared, prepare one control vial of distilled water. This can be done at room temperature. This should be done after X5.5.1 and X5.5.3 have been completed as to prevent mislabeling.

X5.4.10 Check all vials to ensure they have no air bubbles (discard vials with air bubbles).

X5.5 Labeling

X5.5.1 Prepare labels containing the concentration listed as XXX milligrams of AA/litre of solution using the value obtained in X5.4.3 as well as the current date.

X5.5.2 Prepare labels for the control vials saying distilled water.

X5.5.3 Place labels on the appropriate vials.

¹² Available from Supelco and other suppliers, Catalogue # 3-3065-U. It comes with instructions on deactivating glass.

¹³ Available from Supelco, Catalogue # 2-7115U, or equivalent.

¹⁴ Available from Supelco, Catalogue # 2-712OU, or equivalent.

¹⁵ Available from Supelco, Catalogue # 2-7144, or equivalent.

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