TEST METHOD

Effective: 07 November 2017 DOWM 102819-E17A
Supersedes: New

Total Volatiles in Plastics by Moisture Analyzer

1. Scope

1.1 This method is applicable to the determination of percent volatiles in powder and pellet plastic materials.

2. Principle

2.1 This method uses a moisture analyzer to determine the volatiles content by weight loss on drying. The sample is spread onto a sample pan that is supported on a balance in a pre-heated chamber. The sample is then heated to vaporize the volatiles. The analysis is completed when the indicated weight loss falls below the rate specified in the test conditions. The total loss of weight is measured and displayed as percent volatiles.

3. Safety

3.1 Each analyst must be acquainted with the potential hazards of the equipment, reagents, products, solvents and procedures before beginning laboratory work. SOURCES OF INFORMATION INCLUDE: OPERATION MANUALS, MATERIAL SAFETY DATA SHEETS, LITERATURE AND OTHER RELATED DATA. Safety information should be requested from the supplier. Disposal of waste materials, reagents, reactants and solvents must be in compliance with applicable governmental and company requirements.

4. Interferences

4.1 Any volatile component will contribute to the volatile content reading. If results are suspect based on the analytical history of the product, the data should be confirmed by an alternate method.

5. Apparatus (Note 15.1)

5.1 Moisture analyzer: Mettler Model HX204, with halogen heat source, capable of weighing to 0.0001 g, available from Mettler-Toledo, Inc., or equivalent.

5.2 Weighing pans: aluminum, 102-mm diameter round, 8-mm height, catalog number 08-732-111, available from Fischer Scientific, or equivalent.

6. Reagents

6.1 Sodium tartrate dihydrate: with a known crystal water content of 15.66%, for statistical quality control checks, catalog number BP352-500, available from Fisher Scientific, or equivalent.

7. Analysis Conditions

Note: The parameters summarized below were used in the validation of the method. Parameters will depend on each individual moisture analyzer and polymer type and may differ from those stated below (Note 15.2).

Instrument:	Mettler HX204
Balance capability:	0.0001 g
Sample drying temperature:	160 °C
Sodium Tartrate drying temperature:	120 °C
End of analysis:	Automatic
Switch-off Criterion:	1 mg/50 seconds
Display mode:	%MC
Display format:	3 decimals (0.001%)
Sample size:	5.0 g

7.1 A representative end of analysis instrument display is illustrated in Figure 1.

8. Calibration

8.1 The moisture analyzer should be serviced and calibrated on a routine basis by a certified instrumentation expert (once every 12 months is suggested) according to the manufacturer's instructions.

9. Calibration Verification

Note: Perform the calibration verification on a regular basis (weekly is suggested) or prior to each set of analyses with sodium tartrate. Other materials with uniform moisture content that don't readily pick up moisture are acceptable to verify instrument performance. Adjust the analysis parameters as needed, if other materials are used.

9.1 Set the drying temperature to 120 °C and allow the instrument to equilibrate.

9.2 Ensure the other analysis parameters are set as detailed in Section 7.

9.3 Place a clean weighing pan (Section 5.2) on the holder in the moisture analyzer.

9.4 Add 5.00 g (recorded to the nearest 0.0001 g) of sodium tartrate dihydrate (Section 6.1) to the weighing pan and spread evenly.

9.5 Start the automated analysis.

9.6 When the analysis is complete, the instrument will display the volatiles content result to three decimal places (0.001%).

9.7 Dispose of pan and sample in the appropriate waste container.

9.8 Use statistical control charting methods to identify if calibration is in control.

10. Procedure

10.1 Ensure the temperature and analysis parameters are set as detailed in Section 7.

10.2 Place a clean weighing pan (Section 5.2) on the holder in the moisture analyzer.

10.3 Add 5.00 g (recorded to the nearest 0.0001 g) of sample to the weighing pan and spread evenly.

10.4 Start the automated analysis.

10.5 When the analysis is complete, the instrument will display the volatiles content result to three decimal places (0.001%).

10.6 Dispose of pan and sample in the appropriate waste container.

11. Calculations

11.1 The moisture analyzer automatically determines the volatiles content of the sample and displays the result as % (w/w) total volatiles.

11.2 If manual calculations are used, calculate the volatiles content (% w/w) in the sample as follows:

$$\text{Volatiles} = \frac{W_{wet} - W_{dry}}{W_{wet}} \times 100\%$$

where:

Volatiles	=	total volatiles (% w/w) in the sample
W_{wet}	=	weight (g) of sample before drying
W_{dry}	=	weight (g) of the sample after drying
100%	=	conversion to percent

12. Precision

12.1 Precision has been determined from multiple analyses of two powder batches [n = 20] and one pellet batch [n = 10]. The average volatiles content ranged from 0.60 to 0.81% (w/w). The precision data indicate a pooled standard deviation [s_{pooled}] of ± 0.022% and pooled relative standard deviation (RSD) [RSD_{pooled}] of ± 3.1%.

12.2 At the 95% confidence level, individual measurements on similar samples may vary from the long-term average by ± 0.045% [$t \times s_{pooled}$, where t = t-value of 2.05 at 27 degrees of freedom] and ± 6.4% relative [$t \times RSD_{pooled}$, where t = t-value of 2.05 at 27 degrees of freedom].

12.3 The distribution of the results was checked using the Shapiro-Wilk test for normality. The test confirmed that the results could originate from a normal distribution.

13. Accuracy

13.1 The accuracy of the method could not be determined due to the unavailability of characterized reference materials.

14. Limit of Detection/Limit of Quantitation

14.1 The limit of detection (LOD), defined as three times the standard deviation of a low concentration sample was determined to be 0.07% (w/w), and the limit of quantitation (LOQ), defined as ten times the standard deviation of a low concentration sample was determined to be 0.24% (w/w).

15. Notes

15.1 Analytical method performance can be affected by minor differences in instrumentation, reagents, and laboratory technique. Consequently, the method should be qualified in the performing laboratory to confirm its performance and suitability. In addition, analytical instruments should be calibrated at appropriate frequencies.

15.2 The settings defined is this procedure have been validated for acrylic polymers. To analyze other plastics, follow the instrument vendor's instructions for selection of operating conditions.

Figure 1. A representative instrument display at end of analysis