Test Procedure SA-004
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Loss on Drying

Applicable Products: Carbopol®* Polymers, Pemulen™* Polymeric Emulsifiers and Noveon®* AA-1 Polycarbophil

Scope:

This procedure is for the determination of volatile materials in Carbopol® polymers, Pemulen™ polymeric emulsifiers and Noveon® AA-1 polycarbophil.

Abstract:

A weighed sample of polymer is placed in a vacuum oven at a vacuum of 29 inches (736 mm) Hg at the specified temperature and time. The sample is cooled, reweighed and the percent weight loss calculated.

Safety Precautions:

1. Wear safety goggles and gloves.
2. Polymer dust is irritating to the respiratory passages and inhalation should be avoided.
3. See all Material Safety Data Sheets (MSDS) for additional safety and handling information.

Interferences:

Care must be taken to avoid moisture pick-up from the atmosphere. The most accurate measurements can be expected from samples removed the first time the sample container is opened. Because of the hygroscopic nature of Carbopol® polymers, Pemulen™ polymeric emulsifiers and Noveon® AA-1 polycarbophil, moisture pick-up each time the sample container is opened will influence the loss on drying.

Apparatus:

1. Vacuum oven controlled at 80 ± 2°C (176 ± 4°F) with a vacuum of 29 inches (736 mm) Hg.
2. Vacuum oven controlled at 45 ± 2°C (113 ± 4°F) with a vacuum of 29 inches (736 mm) Hg.
3. Balance capable of ±0.0001 g accuracy.
4. Heat safe weighing bottle with glass stopper.
5. Desiccator with silica gel desiccant.
6. Vacuum pump.

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Procedure:

1. For Carbopol® polymers and Pemulen™ polymeric emulsifiers, dry a weighing bottle in the 80 ± 2°C vacuum oven with a vacuum set at 29 inches Hg for 30 minutes. For Noveon® polycarbophil, dry a weighing bottle and stopper at 45 ± 2°C in a vacuum oven with a vacuum of 29 inches Hg for 30 minutes.
2. Keep sample container tightly closed until ready to analyze. Do not allow the sample to absorb moisture from the atmosphere. The loss on drying measurement should be performed first for any series of polymer analyses so that the sample container is not open longer than necessary.
3. Remove the weighing bottle and stopper from the oven and place in a desiccator to cool to room temperature.
4. Determine the tare weight of the weighing bottle and stopper on the analytical balance and record the weight to the nearest 0.0001 gram.
5. Add approximately 1.0 gram (See Note 1) of polymer to the weighing bottle. Place the stopper on the weighing bottle immediately. This will minimize errors due to moisture absorption.
6. Reweigh the weighing bottle, stopper and sample on the analytical balance and record the value to the nearest 0.0001 gram.
7. By gentle, sidewise shaking distribute the polymer evenly in the weighing bottle.
8. For Carbopol® polymers and Pemulen™ polymeric emulsifiers, place the loaded weighing bottle in the vacuum oven at 80 ± 2°C and set at 29 inches (736 mm) Hg vacuum for 1 hour. For Noveon® AA-1 polycarbophil, conditions are 45 ± 2°C and 29 inches (736 mm) Hg vacuum for 4 hours. Remove the stopper and place in the vacuum oven with the loaded bottle.
9. Upon opening the oven at the end of the specified heating time period, stopper the weighing bottle immediately. If the sample is not covered immediately, moisture absorption will occur.
10. Place the weighing bottle in a desiccator and allow cooling to room temperature (approximately 15 minutes).
11. Reweigh the bottle and contents on the analytical balance. Record the weight to the nearest 0.0001 gram and perform the calculation.

Calculations:

% LOSS ON DRYING = (A-B)(100)/C

Where A = Weight of sample, weighing bottle and stopper before drying (to the nearest 0.0001 g).

Where B = Weight of sample, weighing bottle and stopper after drying (to the nearest 0.0001 g).

Where C = Weight of sample (to the nearest 0.0001 g).

Notes:

1. A one gram sample size is specified. This satisfies the United States Pharmacopeia requirement of a 1 to 2 gram sample. The European Pharmacopeia specifies 1.000 gram. Since the actual weight of sample is recorded and used in the calculation, a slight deviation from 1.000 gram is acceptable.

References:

- *Current edition of the United States Pharmacopoeia/National Formulary (USP/NF)*
- *Current edition of the European Pharmacopoeia*