

Moisture (Loss on Drying) in Spices - Vacuum Oven Method

Purpose: To determine the moisture by loss on drying (LOD) content of capsicum spices (paprika, chili pepper, chilies, red pepper, etc.), dehydrated onion and garlic, and dehydrated vegetables.

Principle: Samples are dried for 6h under vacuum at 70°C and purged with a gentle stream of clean and dry air. The weight loss relative to the initial weight of the sample is interpreted as the percent (%) moisture within the sample.

A. Apparatus:

1. Aluminum dishes, 3" diameter by 3/4" with tight fitting aluminum covers.
2. Analytical Balance, sensitivity 0.0001g.
3. Air-tight desiccator with Drierite desiccant.
4. Vacuum oven with fittings for gas washing system, manometer, and thermometer.
5. Vacuum pump capable of maintaining pressure to 50-100 mm Hg (2-4 in)
6. Air drying system, connected in series to the vent of the oven, consisting for example of a Gilmont No. 10 flowmeter, two Drierite gas drying jars with desiccant and a Gelman Acro 50, 0.2 micron filter.
7. Gloves or tongs.
8. Timer capable of measuring at least a 6 h period

B. Reagents:

1. A 50/50 mixture of regular Drierite (8 mesh) and indicating Drierite (8 mesh) is recommended for use in both the desiccator and the gas washing system so that its effectiveness can be monitored.

C. Preparation of Sample:

1. Use Method 1.0.

D. Procedure:

1. Tare weigh an aluminum dish and cover. Do not handle aluminum dish or cover with bare hands, use gloves or tongs.

2. Accurately weigh 2.000 - 5.000 g of sample in to the dish. Use only enough sample to sufficiently cover the bottom of the dish but not less than 2 g.
3. Replace cover and store in desiccator until all samples have been weighed.
4. Before placing dishes in oven, remove lid and place under dish.
5. Place dish and cover in vacuum oven previously warmed to 70 C° (158°F). Do not stack dishes in oven.
6. With vent closed, attach gas washing system.
7. Open vacuum and adjust pressure to 50-100 mm Hg (2-4 in). During drying, open vent and adjust air flow through flowmeter to 60 - 80 mL/min.
8. Dry for 6 hours.
9. Close vacuum. Disconnect flowmeter from gas washing system. Slowly vent oven through desiccant until pressure in the oven returns to atmospheric pressure (760 mm Hg or 30 in).
10. Remove sample dishes from oven, replace matching cover and immediately transfer to desiccator and cool to ambient temperature.
11. Weigh samples to nearest 0.0001 g and calculate to percent. (See E. Calculation)

E. Calculation:

$$\% \text{ Moisture (LOD)} = [(\text{initial wt.} - \text{final wt.}) / \text{initial wt.}] \times 100$$

F. Statistics:

Coefficient of Variation:

		%	
	<u>Low</u>	<u>Med.</u>	<u>High</u>
Parsley	55.28	32.15	7.31
Onion	14.61	9.71	5.48
Paprika	7.50	7.37	6.55

G. Notes:

N/A

H. References:

1. AOAC Method 979.12 vacuum oven method II, Moisture (loss on drying) in roasted coffee, final action 1983.
2. Sanna. L.A. (1986) J. Assoc. Official Analytical Chemistry 69,834.

I. Revision history:

01/1997: initial document

04/2013: revision 1. Method title changed to specify “Loss on Drying”. A new section (principle) was added to follow the new ASTA method format. The graphic on page 4 was removed as being non related to the vacuum oven method. Significant rewording was added throughout the document.