Application Note

Instrument: TGM800

Determination of Total Solids in Milk

LECO Corporation; Saint Joseph, Michigan USA

Introduction

The TGM800 is a thermogravimetric analyzer designed to determine moisture content of materials using a loss-on-drying technique. Mass loss of the sample is measured as a function of the oven temperature by controlling the atmosphere and ventilation rate.

An accurate determination of moisture content in milk products provides important information related to the food quality and safety (texture, taste, microbial stability). Moisture is also a key variable used to calculate a products purity, yield, and/or resulting constituent analysis on a dry basis.

Sample Preparation

Sample should be mixed thoroughly before analysis.

Method Reference

AOAC Official Method 925.23 Solids (Total) in Milk

Fixed Drying Time Method

With 2.4 in crucible using ~3 g sample mass.

Accessories

621-010-956 Aluminum Foil Crucible (2.4 in Crucible),
621-011-237 Carousel (11 place)

Sample Mass ~3.0 g

Analysis Time ~3 h

Method General Parameters

Crucible Type	Large Foil
Minimum Crucible Weight	1.1200
Maximum Crucible Weight	1.6800
Crucible Density	0.5
Sample Type	Other
Sample Density	1.0
Minimum Sample Weight	2.500
Maximum Sample Weight	3.500

Method Step Parameters

Step Type	Preset
Preset Method Step	Moisture
Start Temperature	25.0 °C
End Temperature	102.0 °C
Ramp Rate*	10.0 °C/min
Hold Time	180 min
Maximum Time	240 min
Flow Rate**	5.0 L/min
Final Weight	At End of Step

**A ramp rate of 20 °C/min can be used, and may speed up the analysis and improve the temperature overshoot without any detrimental effects.*

Method Step Calculations

Calculation Type	Preset
Calculation Name	Moisture
Measurement Type	Mass Ratio
Calculation Equation	

(((Initial Mass – Moisture Mass)) ÷ Initial Mass)

Calculation Type	Custom
Calculation Name	Solids
Measurement Type	Mass Ratio
Calculation Equation	(1 - Moisture)

Procedure

1. Create and/or select a method using the parameters described above following the procedure in the TGM800 Instruction Manual.
2. Login and load samples following the procedure outlined in the TGM800 Instruction Manual.

Typical Results—Fixed Drying Time, 2.4 in Crucible

Sample	*Mass (g)*	*% Moisture*	*% Solids*
Skim Milk	3.0080	90.63	9.37
	3.0318	90.55	9.45
	2.9966	90.56	9.44
	3.0069	90.59	9.41
	3.0105	90.52	9.48
	3.0137	90.50	9.50
	3.0095	90.64	9.36
	Avg =	***90.57***	***9.43***
	s =	***0.05***	***0.05***
Half and Half	3.0604	80.76	19.24
	3.0092	80.72	19.28
	3.0059	80.78	19.22
	3.0241	80.72	19.28
	3.0295	80.69	19.31
	3.0360	80.68	19.32
	3.0155	80.69	19.31
	Avg =	***80.72***	***19.28***
	s =	***0.04***	***0.04***
Vitamin D Milk	3.0534	87.63	12.37
	3.0067	87.65	12.35
	3.0105	87.74	12.26
	3.0249	87.64	12.36
	3.0100	87.65	12.35
	3.0177	87.58	12.42
	3.0269	87.73	12.27
	Avg =	***87.66***	***12.34***
	s =	***0.05***	***0.05***
Heavy Whipping Cream	3.0413	58.43	41.57
	3.0323	58.39	41.61
	3.0084	58.44	41.56
	3.0240	58.41	41.59
	3.0078	58.45	41.55
	3.0195	58.39	41.61
	3.0317	58.42	41.58
	Avg =	***58.42***	***41.58***
	s =	***0.02***	***0.02***

***The TGM800 analyzer was connected to Compressed Air (oil and water free) for the reported moisture analyses.*

Fixed Drying Time Method

With 1.5 in crucible using ~1 g sample mass.

Accessories

621-010-236 Aluminum Foil Crucible (1.5 in Crucible),
621-010-642 Carousel (17 place)

Sample Mass ~1.0 g

Analysis Time ~3 h

Method General Parameters

Crucible Type	Small Foil
Minimum Crucible Weight	0.8000
Maximum Crucible Weight	1.2000
Crucible Density	0.5
Sample Type	Other
Sample Density	1.0
Minimum Sample Weight	0.8000
Maximum Sample Weight	1.2000

Method Step Parameters

Step Type	Preset
Preset Method Step	Moisture
Start Temperature	25.0 °C
End Temperature	102.0 °C
Ramp Rate*	10.0 °C/min
Hold Time	180 min
Maximum Time	240 min
Flow Rate**	5.0 L/min
Final Weight	At End of Step

Method Step Calculations

Calculation Type	Preset
Calculation Name	Moisture
Measurement Type	Mass Ratio
Calculation Equation	

(((Initial Mass – Moisture Mass)) ÷ Initial Mass)

Calculation Type	Custom
Calculation Name	Solids
Measurement Type	Mass Ratio
Calculation Equation	(1 - Moisture)

**A ramp rate of 20 °C/min can be used, and may speed up the analysis and improve the temperature overshoot without any detrimental effects.*

Procedure

1. Create and/or select a method using the parameters described above following the procedure in the TGM800 Instruction Manual.
2. Login and load samples following the procedure outlined in the TGM800 Instruction Manual.

Typical Results—Fixed Drying Time, 1.5 in Crucible

Sample	*Mass (g)*	*% Moisture*	*% Solids*
Skim Milk	1.0426	90.59	9.41
	1.0211	90.55	9.45
	1.0492	90.62	9.38
	1.0132	90.58	9.42
	1.0352	90.58	9.42
	1.0049	90.60	9.40
	1.0212	90.56	9.44
	Avg =	***90.58***	***9.42***
	s =	***0.02***	***0.02***
Half and Half	1.0429	80.70	19.30
	1.0346	80.72	19.28
	1.0792	80.75	19.25
	1.0219	80.74	19.26
	1.0219	80.72	19.28
	1.0653	80.70	19.30
	1.0217	80.73	19.27
	Avg =	***80.72***	***19.28***
	s =	***0.02***	***0.02***
Vitamin D Milk	1.0551	87.71	12.29
	1.0321	87.79	12.21
	1.0239	87.81	12.19
	1.0064	87.83	12.17
	1.0277	87.76	12.24
	1.0317	87.78	12.22
	1.0098	87.81	12.19
	Avg =	***87.79***	***12.22***
	s =	***0.04***	***0.04***
Heavy Whipping	1.0129	58.39	41.61
Cream	1.0669	58.24	41.76
	1.0109	58.41	41.59
	1.0074	58.33	41.67
	1.0588	58.34	41.66
	1.0133	58.42	41.58
	1.0260	58.43	41.57
	Avg =	***58.36***	***41.63***
	s =	***0.07***	***0.07***

***The TGM800 analyzer was connected to Compressed Air (oil and water free) for the reported moisture analyses.*

Mass Constancy Drying Time Method

With 2.4 in crucible using ~1 g sample mass.

Accessories

621-010-956 Aluminum Foil Crucible (2.4 in Crucible),
621-011-237 Carousel (11 place)

Sample Mass ~1.0 g

Analysis Time ~1 h

Method General Parameters

Crucible Type	Large Foil
Minimum Crucible Weight	1.1200
Maximum Crucible Weight	1.6800
Crucible Density	0.5
Sample Type	Other
Sample Density	1.0
Minimum Sample Weight	0.8000
Maximum Sample Weight	1.2000

Method Step Parameters

Step Type	Preset
Preset Method Step	Moisture
Start Temperature	25.0 °C
End Temperature	102.0 °C
Ramp Rate*	10.0 °C/min
Hold Time	0 min
Maximum Time	240 min
Flow Rate**	5.0 L/min
Final Weight	At Constancy
Constancy Window	9 min
Constancy Level	0.0005 g

Method Step Calculations

Calculation Type	Preset
Calculation Name	Moisture
Measurement Type	Mass Ratio
Calculation Equation	
(((Initial Mass – Moisture Mass)) ÷ Initial Mass)	
Calculation Type	Custom
Calculation Name	Solids
Measurement Type	Mass Ratio
Calculation Equation	(1 - Moisture)

**A ramp rate of 20 °C/min can be used, and may speed up the analysis and improve the temperature overshoot without any detrimental effects.*

Procedure

1. Create and/or select a method using the parameters described above following the procedure in the TGM800 Instruction Manual.
2. Login and load samples following the procedure outlined in the TGM800 Instruction Manual.

Typical Results—Mass Constancy Drying Time, 2.4 in Crucible

Sample	*Mass (g)*	*% Moisture*	*% Solids*
Skim Milk	1.0325	90.50	9.50
	1.0186	90.29	9.71
	1.0148	90.43	9.57
	1.0224	90.35	9.65
	1.0065	90.46	9.54
	1.0130	90.31	9.69
	1.0225	90.48	9.52
	Avg =	***90.40***	***9.60***
	s =	***0.08***	***0.09***
Half and Half	1.0198	80.74	19.26
	1.0522	80.74	19.26
	1.0274	80.83	19.17
	1.0260	80.70	19.30
	1.0110	80.70	19.30
	1.0196	80.62	19.38
	1.0448	80.75	19.25
	Avg =	***80.73***	***19.27***
	s =	***0.06***	***0.06***
Vitamin D Milk	1.0393	87.76	12.24
	1.0254	87.69	12.31
	1.0051	87.87	12.13
	1.0499	87.59	12.41
	1.0298	87.78	12.22
	1.0038	87.62	12.38
	1.0244	87.71	12.29
	Avg =	***87.72***	***12.28***
	s =	***0.10***	***0.10***
Heavy Whipping	1.0092	58.30	41.70
Cream	1.0355	58.31	41.69
	1.0222	58.39	41.61
	1.0529	58.34	41.66
	1.0167	58.41	41.59
	1.0119	58.27	41.73
	1.0123	58.41	41.59
	Avg =	***58.35***	***41.65***
	s =	***0.06***	***0.06***

***The TGM800 analyzer was connected to Compressed Air (oil and water free) for the reported moisture analyses.*

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