

Moisture and Total Solid Determination of Milk-Based Products Comparing Automated Thermogravimetric and Air Oven Loss-on-Drying Techniques



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Introduction

Accurate determination of mass loss-on-drying in milk-based products provides important information related to product quality and safety (texture, taste, microbial stability). Mass loss also provides a key variable used to calculate the products purity, yield, and resulting constituent analysis on a dry basis. One of the most common moisture determination methods is **mass loss-on-drying** using an air oven in which the sample mass is taken before and after exposure to an elevated temperature within an oven with air ventilation. The resulting sample mass loss is calculated as total solids or moisture, depending on the method.

The AOAC total solid determination in milk and milk products method (Method 925.23) utilizes the air oven loss-on-drying technique that is widely used in the food industry. The AOAC moisture in cheese method (Method 948.12) also utilizes the air oven loss-on-drying technique for determination of moisture content. A new automated thermogravimetric moisture instrument (TGM800) is now available. This instrument meets the AOAC method requirements for sample mass, oven temperature, and air flow, while providing many additional benefits. Specific advantages of the TGM800 include improved work flow efficiency, minimal operator time, and sample batch throughput of up to 16 samples. The instrument also incorporates automated end point recognition based upon sample mass constancy, therefore delivering optimal analysis time.

This poster presentation will cover the comparison of mass loss-on-drying determination using an automated thermogravimetric moisture instrument (TGM800) and a manual air dry loss method using a 4-place balance, air oven, and desiccator. The comparison will include food matrix sample data measured with the TGM800 and air oven techniques following AOAC Methods 925.23 and 948.12.

Referenced Methods

- AOAC Official Method 925.23 Solids (Total) in Milk
- AOAC Official Method 948.12 Moisture in Cheese

TGM800 Theory of Operation

The TGM800 is a thermogravimetric analyzer designed to directly determine moisture content of materials using a mass 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. The instrument consists of a computer, an integrated four-place balance, and a multiple sample oven that allows up to 16 samples to be analyzed simultaneously with a maximum temperature of 150 °C.

Key Features

- Supports 1.5 in diameter (1 g nominal mass) aluminum foil crucibles
- Supports 2.4 in diameter (3 g nominal mass) aluminum foil crucibles
- Analysis can be performed in air or nitrogen atmosphere
- Automated crucible and sample mass recorded
- Optional fixed time or constancy method parameters



Figure 1: TGM800

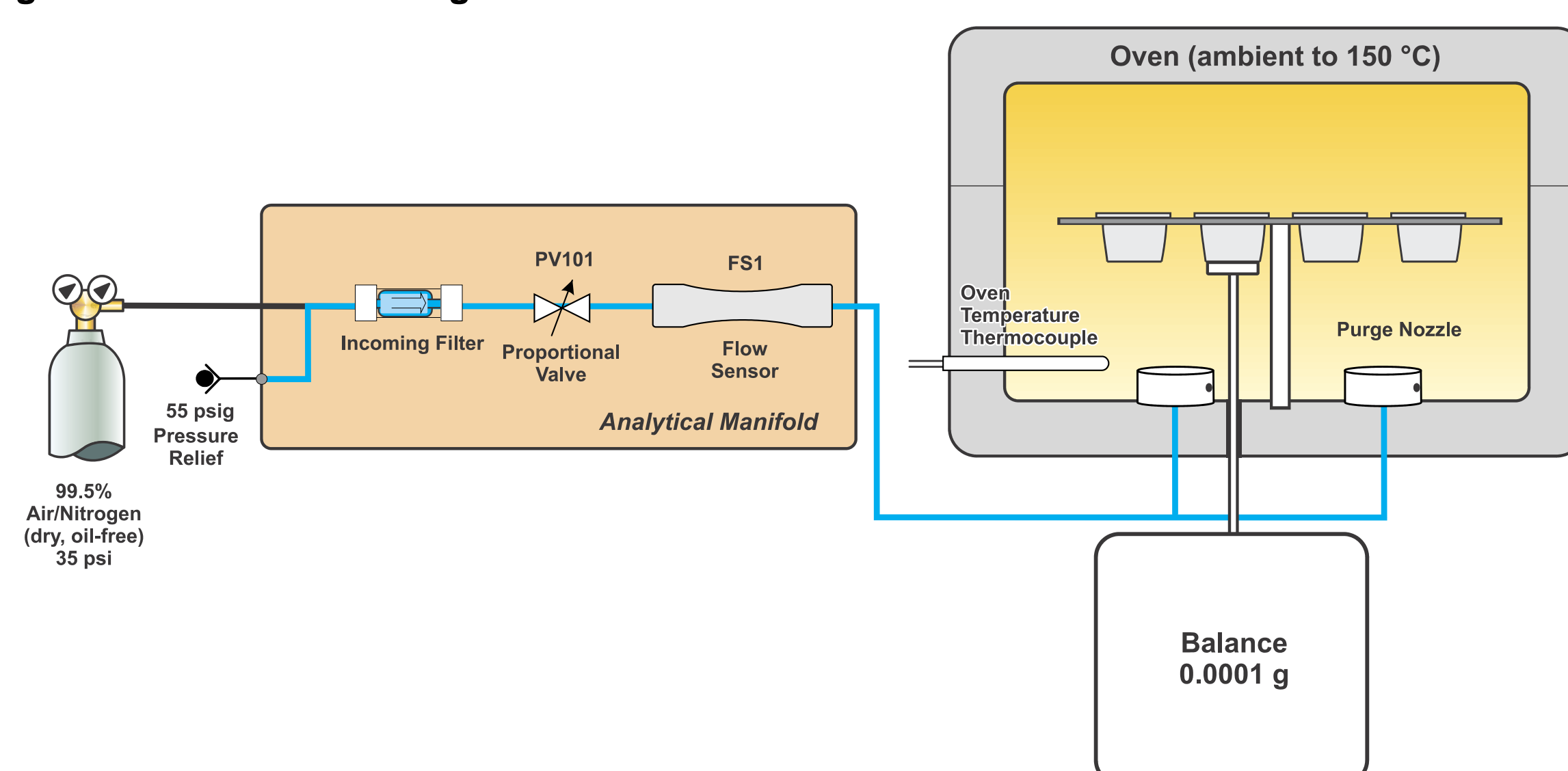


Figure 2: 16 sample carousel using 1.5 in aluminum foil crucible



Figure 3: 10 sample carousel using 2.4 in aluminum foil crucible

Figure 4: TGM800 Flow Diagram



Methodology

AOAC Official Method 925.23 Solids (Total) in Milk

A 2.5 to 3 g sample is weighed into a flat bottom dish with 5 cm diameter, and dried in an oven at 102 °C for 3 hours. The dish is covered before removal from the oven, then transported to a desiccator, and cooled before taking the final mass.

AOAC Official Method 948.12 Moisture in Cheese

A 2 to 3 g sample is weighed into a moisture dish with a tight fitting cover, and subsequently dried in an oven at 130 ±1 °C for 1.25 hours. The dish is covered before removal from the oven, then transported to a desiccator, and cooled before taking the final mass.

Moisture Analysis Parameters

Table 1: Manual Oven Loss-on-Drying Method Parameters

Method	AOAC 925.23	AOAC 948.12
Crucible Type	Glass Dish	Glass Dish
Nominal Mass	~3 g	~3 g
Oven Temperature	102 °C	130 °C
Hold Time	180 minutes	75 minutes

Table 2: TGM800 Milk Method Parameters

Method	Milk-Method 1	Milk-Method 2	Milk-Method 3
Crucible Type	2.4 in Ø Al Foil	1.5 in Ø Al Foil	2.4 in Ø Al Foil
Nominal Mass	~3 g	~1 g	~1 g
Start Temperature	Ambient	Ambient	Ambient
End Temperature	102 °C	102 °C	102 °C
Ramp Rate	10.0 °C/min	10.0 °C/min	10.0 °C/min
Hold Time	180 min	180 min	0 min
Ventilation Flow Rate	5.0 L/min	5.0 L/min	5.0 L/min
Final Mass	At End of Step	At End of Step	At Constancy

Table 3: TGM800 Cheese Method Parameters

Method	Cheese-Method 1	Cheese-Method 2	Cheese-Method 3
Crucible Type	2.4 in Ø Al Foil	1.5 in Ø Al Foil	2.4 in Ø Al Foil
Nominal Mass	~3 g	~1 g	~1 g
Start Temperature	Ambient	Ambient	Ambient
End Temperature	130 °C	130 °C	130 °C
Ramp Rate	10.0 °C/min	10.0 °C/min	10.0 °C/min
Hold Time	75 min	75 min	0 min
Ventilation Flow Rate	5.0 L/min	5.0 L/min	5.0 L/min
Final Mass	At End of Step	At End of Step	At Constancy

Sample Description

Four commercial milk samples were purchased representing a variety of milk product matrices.

- Skim Milk
- Vitamin D Milk
- Half and Half
- Heavy Whipping Cream

Four commercial pre-shredded cheese samples were purchased representing a variety of cheese product matrices.

- Colby-Jack
- Mild Cheddar
- Mozzarella
- Parmesan

Sample Results

Chart 1: Milk Moisture Results

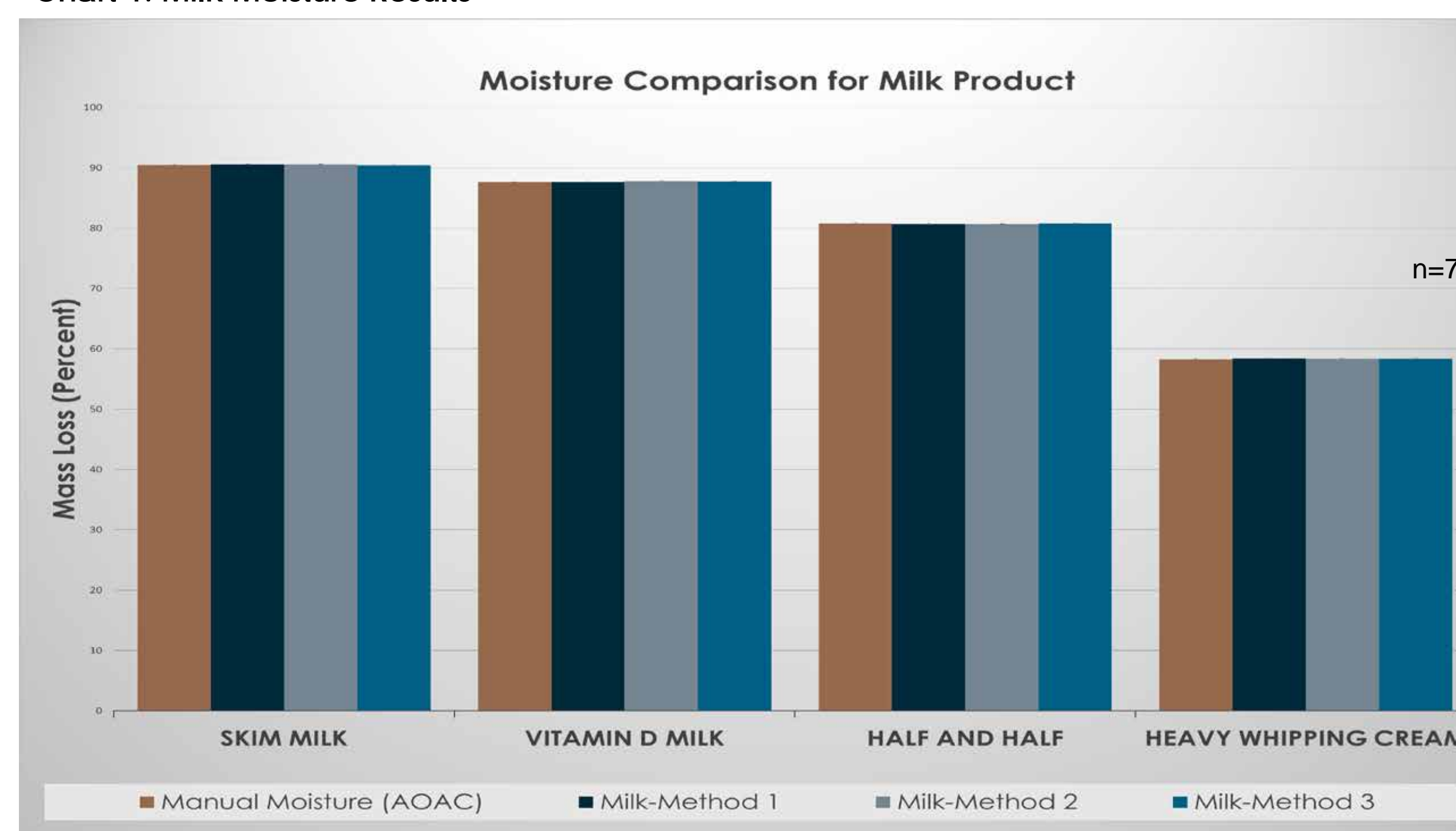


Table 4: Standard Deviations of the Various Milk Methods Tested

Sample	Manual Moisture (AOAC)	Milk-Method 1	Milk-Method 2	Milk-Method 3
Skim Milk	0.05	0.05	0.02	0.08
Vitamin D Milk	0.06	0.05	0.04	0.10
Half and Half	0.11	0.04	0.02	0.06
Heavy Whipping Cream	0.20	0.02	0.07	0.06

Chart 2: Cheese Moisture Results

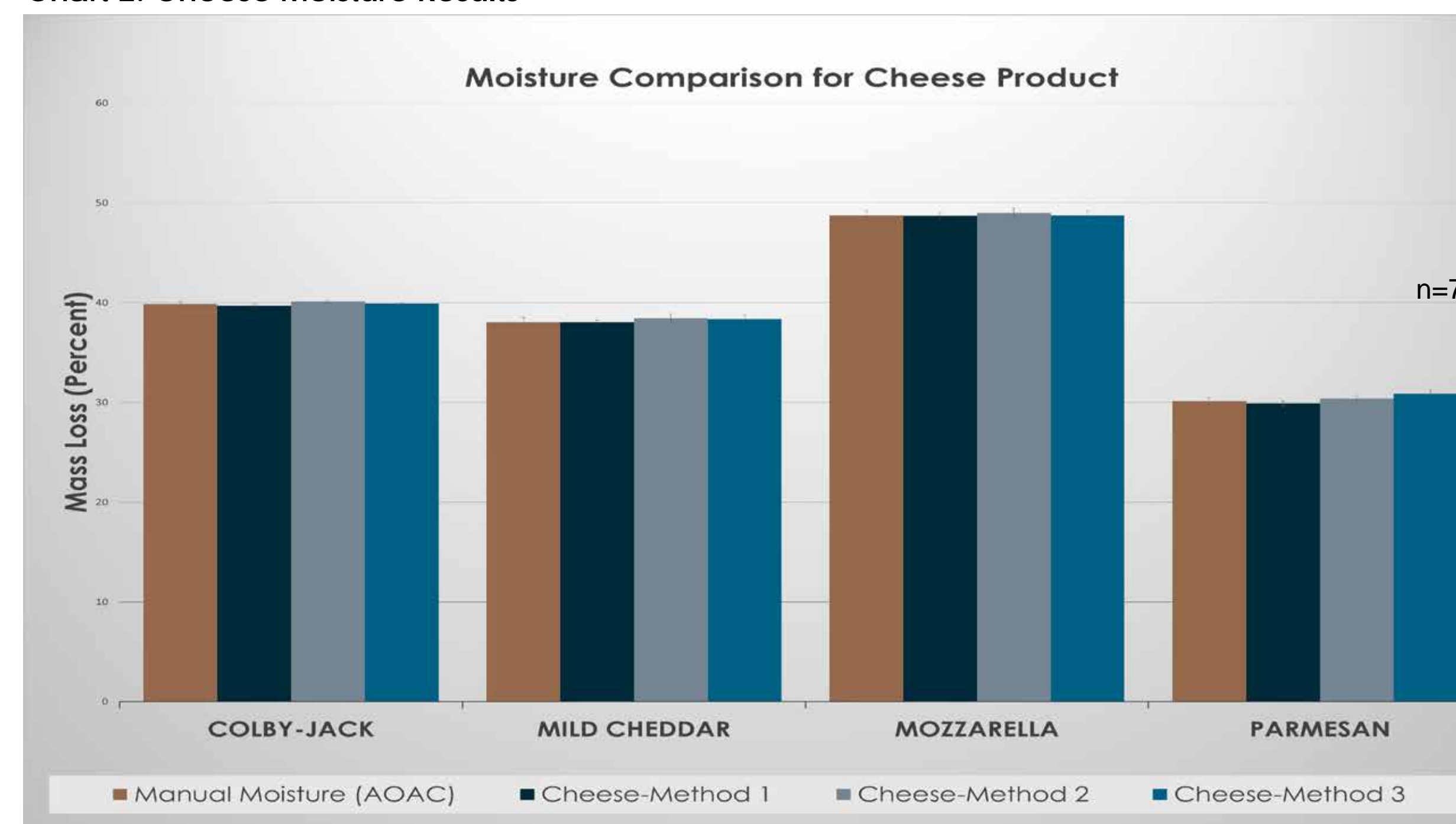


Table 5: Standard Deviations of the Various Cheese Methods Tested

Sample	Manual Moisture (AOAC)	Cheese-Method 1	Cheese-Method 2	Cheese-Method 3
Colby-Jack	0.28	0.17	0.13	0.09
Mild Cheddar	0.49	0.22	0.39	0.42
Mozzarella	0.49	0.30	0.47	0.48
Parmesan	0.33	0.27	0.24	0.39

Chart 3: Milk Analysis Time Results

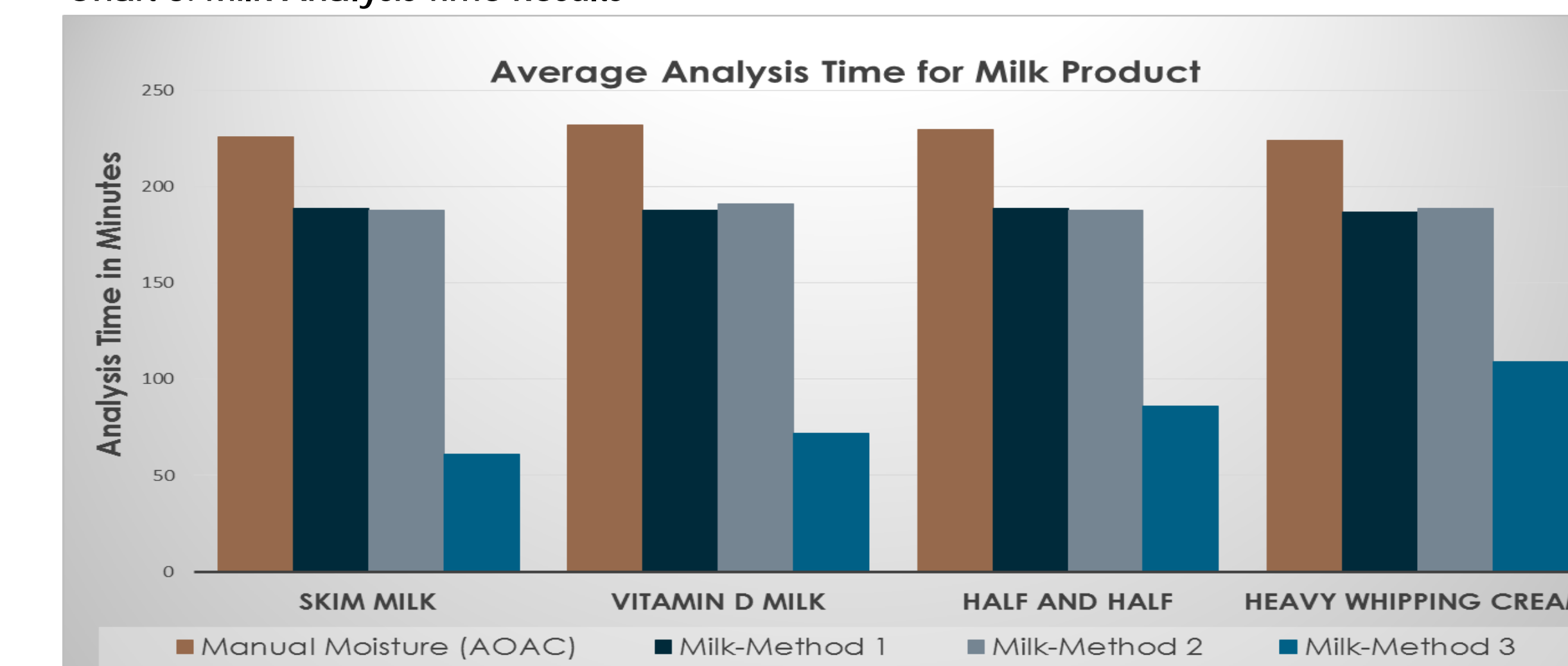
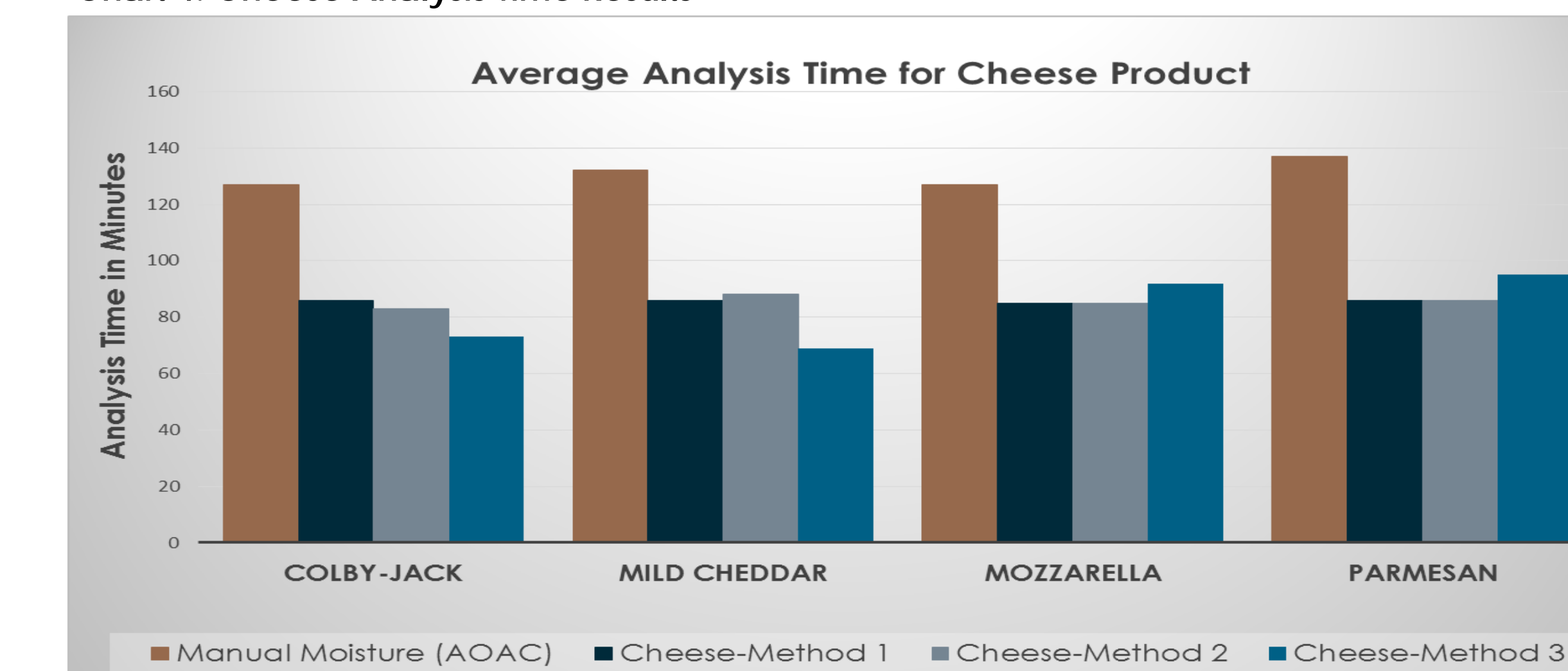


Chart 4: Cheese Analysis Time Results



Conclusion

The moisture results obtained using the manual method and TGM800 for both milk-based products are in agreement. This includes utilizing multiple modifications of the AOAC methods using the TGM800.

Benefits of determining moisture and total solids of milk-based products on the LECO TGM800 include:

- Ability to follow both AOAC 925.23 and 948.12 methodology
- Ability to deviate from AOAC methods by analyzing a smaller sample mass and utilizing end of step hold times, resulting in higher sample throughput
- Ability to deviate from AOAC methods by analyzing smaller sample mass and utilizing the constancy option, resulting in reduced sample analysis time

The objective of this poster presentation was to demonstrate the analytical performance and application capabilities of the TGM800 instrument using multiple method parameters compared to the manual air oven methods used in the AOAC methods. The TGM800 moisture results utilizing all of the examined method modifications were comparable to the results obtained by the manual AOAC methods. The TGM800 moisture results have improved precision compared to the manual AOAC methods, with some samples showing as much as an order of magnitude improvement in precision. When using fixed drying time method conditions that are compliant with the manual AOAC methods, the total analysis time for the TGM800 was 30 to 40 minutes shorter than the manual AOAC methods. This is due to the elimination of sample transfer to a desiccator and the associated cooling time. When using sample mass constancy, a total time savings of 40 to 150 minutes can be achieved compared to the manual AOAC methods. This is accomplished by utilizing the automated drying time end point detection based on the sample mass constancy and the 2.4 in diameter aluminum foil crucibles with ~1 g sample mass.

The TGM800 provides the user with flexible method settings, automation, and hardware capabilities that maximizes moisture determination efficiency, productivity, and analytical performance. This is accomplished while offering the ability to meet the primary loss-on-drying method requirements for sample mass, oven temperature, and ventilation.