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## Prelude

### Your Personal Productivity Workstation

The Caliper Life Sciences Prelude is the most reliable personal productivity tool to be used in the laboratory to reduce the time, cost and tedium of manual sample preparation including dilution, weighing, mixing, filtration, extraction and other tasks. With Prelude, the analyst can increase their time by 50-70% while reducing the cost of reagents and disposal of those reagents by 70%. More work can be done on a continuous basis, freeing the analyst for other laboratory responsibilities. In addition, prepared samples can be automatically injected into HPLC systems, sent to UV/Vis spectrophotometers, or collected in the EasyFill Sample Collection Module. No other automated sample preparation system is as fast, flexible or easy to learn and operate as Prelude.

### Full Compliance with 21 CFR Part 11

*Electronic monitoring with Prelude provides an audit trail to assist in electronic record keeping, allowing implementation of regulations for electronic records and signatures that meet FDA requirements.*

*The Prelude ensures proper sequencing of entries, validity of data, and verification of records from those generated from the analytical balance and other sources. Use of secure electronic audit trails support electronic record integrity and authenticity, linking to electronic compliance.*

*The information generated can be easily and securely transmitted to laboratory information systems, chromatography database systems, as well as other spreadsheet applications used without compromising the integrity of the original record.*

## Automated Sample Preparation for Creams, Gels and Difficult Samples

**Lynn Jordan, Jayne Brown and Stephanie Wilson**

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### Abstract

Sample preparation for creams, lotions, gels and ointments is a difficult task. Their high viscosity or unique rheology causes analyte extraction of these sample types to be particularly difficult to disperse. They are most troublesome when the semi-solid base product is not miscible with the preferred solvent required for the assay. This poster will illustrate how automated sample preparation can be utilized to overcome these difficulties. One benefit of automated sample preparation is its ability to eliminate differences in various manual techniques such as sample dispersion. In addition, automated systems can provide a documented method and a complete audit trail of the sample handling which many laboratories require to meet 21 CFR Part 11 compliance. This poster will examine the parameters used for an efficient and reproducible sample preparation for these sample types, as well as the resulting documentation and audit trail.

### Objective

To compare manual and automated methods for three difficult sample types. For each of these samples we developed an automated Prelude Workstation application with the Sonicator accessory. Samples were run by manual and automated methods comparing time and solvent usage. Review documentation resulting from the manual and automated procedures.

### Conclusion

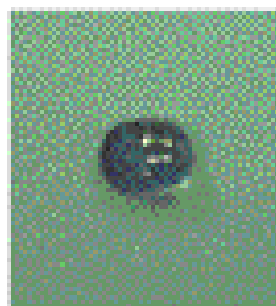
Sample Preparation of the gel, sun screen and caramel sample can be achieved by either a manual or automated procedure.

Although the automated method may or may not be faster than the manual method, the manual method requires hands-on operation, where the automated procedure provides unattended operation.

Manual sample preparation provides limited documentation. The Prelude provides total compliance to 21 CFR Part 11 by storing data in a secure database. In addition, the Prelude provides an Online Status Screen, and gravimetric confirmations for every step of the sample preparation documented in an Excel™ Spreadsheet.

# Gel Sample

This is a topical medication, based on Carbomer 940.



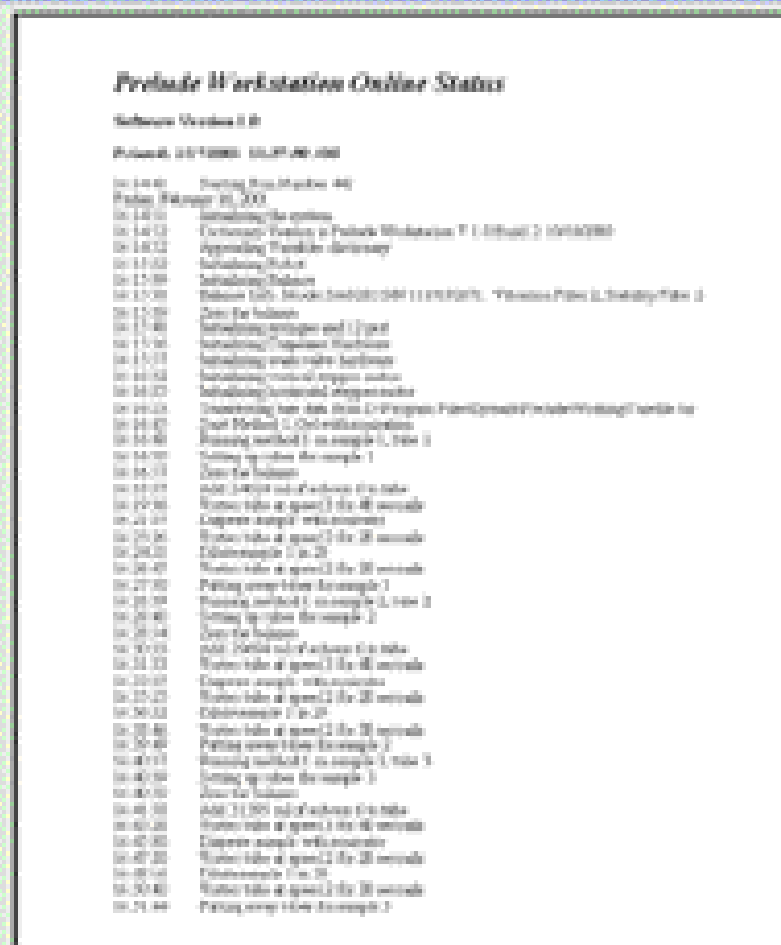
## Prelude™ Automated Procedure



## Gel Manual Procedure: Documentation



## Gel Automated Procedure: Documentation



Automated Workstation Online Status

### Gel: Manual Method

1. Weigh exactly 500 mg of gel into a 50 ml volumetric flask.
2. Add about 40 ml of water - acetonitrile, 40-60 v/v
3. Dissolve accurately with an ultrasonic bath.
4. Bring solution to temperature
5. Bring to volume with the water - acetonitrile, 40-60 v/v solution.
6. Mix thoroughly

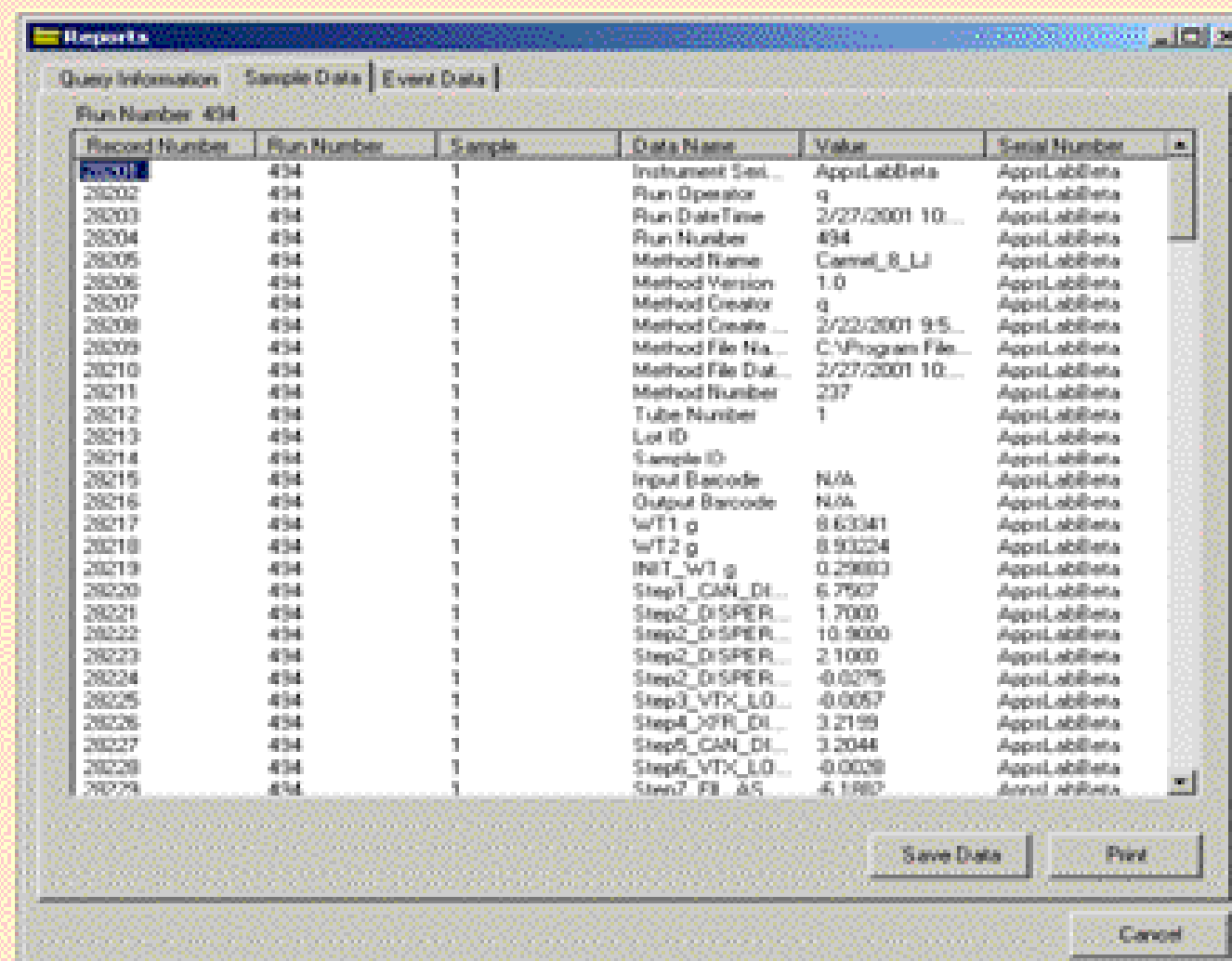
### Gel: Summary

Gel Sample Summary (n=5 Samples)		
	Sample Prep Time (minutes)	Total Solvent Usage (ml)
Manual Procedure	60	250
Automated Procedure	6.2	6.1

# Caramel Sample

continued

## Caramel Automated Procedure: Documentation



Secure Data Base 21 CFR Part 11 Compliant

Run Number: 434

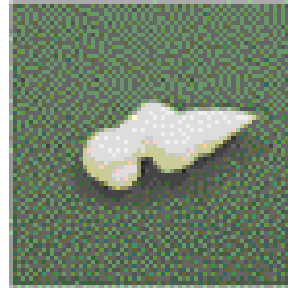
Run Date: 2/27/2001 10:00:00 AM

Sample	WT1 g	WT2 g	INIT_w1 g	Step1_CAN_DI...	Step2_DISPEN...	Step2_DISPEN...	Step2_DISPEN...	Step3_VTX_LO...	Step4_VPR_DI...	Step5_CAN_DI...	Step6_VTX_LO...	Step7_FR_AS
1	8.63341	8.93224	0.29883	6.7507	1.7000	10.9000	2.1000	-0.0275	3.2199	3.2044	-0.0028	4.1187

Excel™ Gravimetric Documentation

# Sun Screen

This is a SPF 15 sun screen. The active ingredient is titanium dioxide and zinc dioxide.



## Sun Screen Manual Procedure: Documentation



### Sun Screen: Manual Method

1. Weigh 6.3 g +/-0.1mg of sample into a 100ml volumetric flask.
2. Dilute to volume with Methanol/Ethyl Acetate Solvent.
3. Shake and sonicate the sample until it is totally dispersed.
4. Avoid excessive sonication.

Sonication should not exceed 10 minutes to prevent heat formation.

### Sun Screen: Summary

Sun Screen Sample Summary with Samples		
	Sample Prep Time (minutes)	Total Solvent Usage (ml)
Manual Procedure	120	600
Automated Procedure	60	78

## Prelude™ Automated Procedure



## Sun Screen Automated Procedure: Documentation



Automated Workstation Online Status

# Sun Screen

continued

## Sun Screen Automated Procedure: Documentation

Record Number	Run Number	Sample	Data Name	Value	Serial Number
27307	446	1	Instrument Ser...	AppiLabBeta	AppiLabBeta
27308	446	1	Run Operator	q	AppiLabBeta
27309	446	1	Run DateTime	2/20/2001 10...	AppiLabBeta
27310	446	1	Run Number	446	AppiLabBeta
27311	446	1	Method Name		AppiLabBeta
27312	446	1	Method Version	1.0	AppiLabBeta
27313	446	1	Method Creator	q	AppiLabBeta
27314	446	1	Method Create ...	2/19/2001 1:5...	AppiLabBeta
27315	446	1	Method File Na...	C:\Program File...	AppiLabBeta
27316	446	1	Method File Dat...	2/20/2001 9:2...	AppiLabBeta
27317	446	1	Method Number	227	AppiLabBeta
27318	446	1	Tube Number	1	AppiLabBeta
27319	446	1	Lot ID		AppiLabBeta
27320	446	1	Sample ID		AppiLabBeta
27321	446	1	Input Barcode	N/A	AppiLabBeta
27322	446	1	Output Barcode	N/A	AppiLabBeta
27323	446	1	WT1 g	8.79904	AppiLabBeta
27324	446	1	WT2 g	9.43608	AppiLabBeta
27325	446	1	INIT_WT g	0.63674	AppiLabBeta
27326	446	1	Step1_CAN_DL...	7.9515	AppiLabBeta
27327	446	1	Step2_DISPERS...	1.6000	AppiLabBeta
27328	446	1	Step2_DISPERS...	6.1000	AppiLabBeta
27329	446	1	Step2_DISPERS...	2.1000	AppiLabBeta
27330	446	1	Step2_DISPERS...	-0.0218	AppiLabBeta
27331	446	1	Step3_FIL_AS...	6.6727	AppiLabBeta

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Run Number: 446

Method: Method 340.09, SR 11190071, Method File 2, Sample File 2

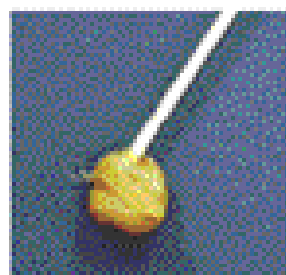
Instrument Serial #: AppiLabBeta  
Software Version: 1.0  
Test Method: 3200001-101913-00

Sample	WT1 g	WT2 g	INIT_WT g	Step1_CAN_DL...	Step2_DISPERS...	Step2_DISPERS...	Step2_DISPERS...	Step3_FIL_AS...
1	8.79904	9.43608	0.63674	7.9515	1.6000	6.1000	2.1000	6.6727
2	8.79904	9.43608	0.63674	7.9515	1.6000	6.1000	2.1000	6.6727
3	8.79904	9.43608	0.63674	7.9515	1.6000	6.1000	2.1000	6.6727
4	8.79904	9.43608	0.63674	7.9515	1.6000	6.1000	2.1000	6.6727
5	8.79904	9.43608	0.63674	7.9515	1.6000	6.1000	2.1000	6.6727
6	8.79904	9.43608	0.63674	7.9515	1.6000	6.1000	2.1000	6.6727

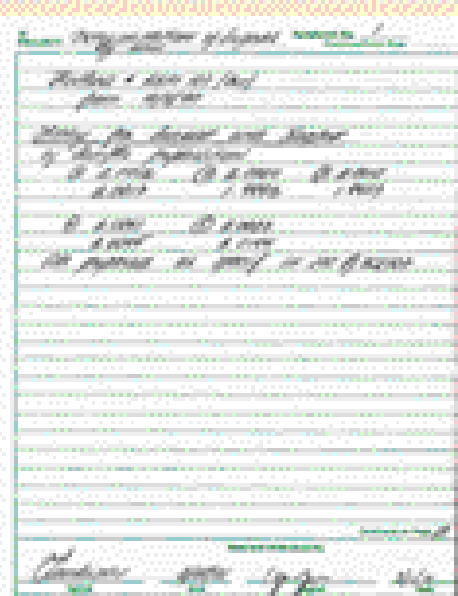
Excel™ Gravimetric Documentation

# Caramel Sample

This is a hardened caramel sample.



## Caramel Manual Procedure: Documentation



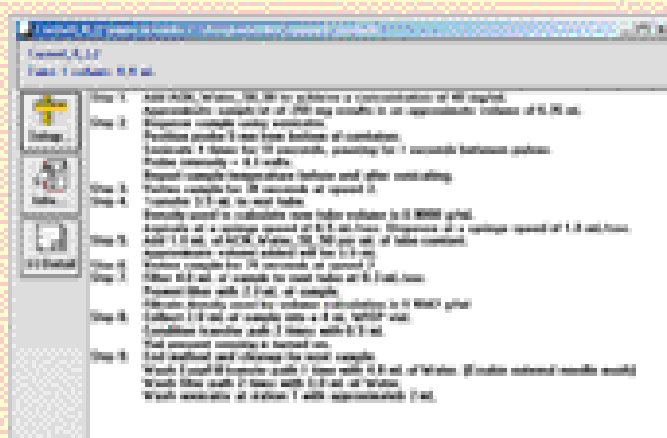
### Caramel Sample: Manual Method

1. Weigh accurately 2.0 g of each caramel sample and transfer into a 100 mL volumetric flask.
2. Add 50 mL of hot water (70-80°C)
3. Shake until sample is evenly dispersed
4. Dilute to volume with acetonitrile
5. Filter a portion of each sample through a 0.45 µm syringe filter into autosampler vials.

### Caramel: Summary

Caramel Sample Summary n=6 Samples		
	Sample Prep Time (minutes)	Total Solvent Usage (mL)
Manual Procedure	100	600
Automated Procedure	122	115

## Prelude Automated Procedure



## Caramel Automated Procedure: Documentation



Automated Workstation Online Status

# Gel Sample

continued

## Gel Automated Procedure: Documentation

Reports

Query Information | Sample Data | Event Data

Run Number: 442

Record Number	Run Number	Sample	Data Name	Value	Serial Number
27125	442	1	Instrument Ser...	AppLabbeta	AppLabbeta
27126	442	1	Run Operator	g	AppLabbeta
27127	442	1	Run Date Time	2/16/2001 4:1...	AppLabbeta
27128	442	1	Run Number	442	AppLabbeta
27129	442	1	Method Name	Gel with sonicat...	AppLabbeta
27130	442	1	Method Version	1.0	AppLabbeta
27131	442	1	Method Creator	g	AppLabbeta
27132	442	1	Method Create ...	2/16/2001 11:...	AppLabbeta
27133	442	1	Method File Na...	D:\Program File...	AppLabbeta
27134	442	1	Method File Cat...	2/16/2001 4:1...	AppLabbeta
27135	442	1	Method Number	225	AppLabbeta
27136	442	1	Tube Number	1	AppLabbeta
27137	442	1	Lot ID		AppLabbeta
27138	442	1	Sample ID		AppLabbeta
27139	442	1	Input Barcode	N/A	AppLabbeta
27140	442	1	Output Barcode	N/A	AppLabbeta
27141	442	1	WT1 g	8.60214	AppLabbeta
27142	442	1	WT2 g	9.09462	AppLabbeta
27143	442	1	INT_WT g	0.49248	AppLabbeta
27144	442	1	Step1_GAN_DI...	2.1755	AppLabbeta
27145	442	1	Step2_VTX_LO...	-0.0177	AppLabbeta
27146	442	1	Step3_DISPEN...	1.7000	AppLabbeta
27147	442	1	Step3_DISPEN...	5.9000	AppLabbeta
27148	442	1	Step3_DISPEN...	1.9000	AppLabbeta
27149	442	1	Step3_DISPEN...	-0.0130	AppLabbeta
27150	442	1	Step4_VTX_LO...	-0.0004	AppLabbeta
27151	442	1	Step5_DISPEN...	2.1000	AppLabbeta

Save Data Print Cancel

Secure Data Base 21 CFR Part 11 Compliant

Run Number: 442

Sample: Wt1 (8.60214 g) (WT1) (Sample No. 1) (Sample No. 1)

Method: Gel with sonicat (Method No. 225)

Method Version: 1.0

Method Creator: g

Method Create Date: 2/16/2001 11:00:00

Method File Name: D:\Program Files\... (Method File Name)

Method File Category: 2/16/2001 4:10:00 (Method File Category)

Method Number: 225

Tube Number: 1

Lot ID:

Sample ID:

Input Barcode: N/A

Output Barcode: N/A

Step No.	Step Name	Start Time	End Time	Duration	Start Volume	End Volume	Dispensed Volume	Dispensed Volume (mL)	Dispensed Volume (mL)	Dispensed Volume (mL)	Dispensed Volume (mL)	Dispensed Volume (mL)	Dispensed Volume (mL)
1	Step1_GAN_DI...	0:00	0:00	0:00	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
2	Step2_VTX_LO...	0:00	0:00	0:00	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
3	Step3_DISPEN...	0:00	0:00	0:00	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
4	Step3_DISPEN...	0:00	0:00	0:00	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
5	Step3_DISPEN...	0:00	0:00	0:00	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
6	Step4_VTX_LO...	0:00	0:00	0:00	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
7	Step5_DISPEN...	0:00	0:00	0:00	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

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