

	Standard Operating Procedure Cryogenic Grinding of Chewable Gels		SOP Number D-793	Revision 0
			Effective Date <i>02/24/20</i>	Page Page 1 of 3
Written by/ Date <i>Cyber 02-10-20</i>		Reviewed by/ Date <i>SSS 02/10/20</i>		Approved by/ Date <i>JM 02/10/20</i>
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1.0 Purpose

This document describes the procedure for the cryogenic grinding of chewable gels (gummies).

2.0 Scope

This procedure applies to any sample preparation involving chewable gels where reducing them to powder may be desirable. Examples may include the facilitation of chemical and/or microbiological assessments.

3.0 Responsibility

- 3.1 It is the responsibility of QC and Analytical chemists who have verified their ability to execute this procedure to follow this procedure.
- 3.2 It is the responsibility of the QC Laboratory Management to implement this procedure and to ensure that the procedure is being followed.
- 3.3 It is the responsibility of the QC Laboratory Management and AD Personnel to keep this procedure current with the associated monographs and laboratory practices.

4.0 Definitions

- 4.1 **QC** – Quality Control
- 4.2 **AD** – Analytical Development
- 4.3 **Cryogenic** – Being or relating to very low temperatures.
- 4.4 **LN2** – Liquid Nitrogen
- 4.5 **Dewar** – Vacuum insulated vessel used for storing cryogenic liquids.
- 4.6 **Phase Separator** – Metallic frit terminating LN2 transfer line designed to minimize hazardous splashing and vaporization.

5.0 References

- 5.1 Liquid Nitrogen Container Handling Instructions – Chart, Inc. Ref 10468660, Rev C, 2010.
- 5.2 Operating Manual for MVE Liquid Nitrogen Dewars – Chart, Inc. Ref 11624417, Rev T, 07/19.

- 5.3 Operating Instructions for Waring Commercial Spice Grinder – Conair, Inc. Ref WSG30 IB 08WC050, Rev F IB-8373, 2009.

6.0 Supplies

- 6.1 Chemical – Food Grade or Better
- 6.1.1 LN2 – 180 Liter Dewar @ 22 psi (Airgas P/N NI 180LT22)
- 6.2 Supplies and Hardware
- 6.2.1 72” Nitrogen Hose Assembly (Transfer Line) w/ Phase Separator
- 6.2.2 5 Liter Dewar w/ Pour Spout
- 6.2.3 1 Liter Dewar
- 6.3 Equipment
- 6.3.1 Waring Commercial Spice Grinder (Waring P/N WSG30)

7.0 Procedure

- 7.1 CAUTION
- 7.1.1 LN2 freezes human tissue almost instantaneously! (At atmospheric pressure, LN2 boils at -196°C!) When handling, always wear insulated gloves and safety glasses!
- 7.1.2 LN2 produces massive volumes of gas that do not support respiration! (1L of LN2 vaporizes into ~25 cubic feet of nitrogen gas!) Only use in large, well ventilated areas!
- 7.2 LN2 Storage & Use
- 7.2.1 In order to facilitate use, LN2 is stored / used / dispensed out of three different on-site dewars. A 180 liter dewar located in the warehouse provides for bulk storage.
- 7.2.2 A 5 liter dewar located in the QC Laboratory provides for convenient intermediate bulk storage capable of supporting multiple sample preparations.
- 7.2.3 Individual sample preparations are performed in a 1 liter dewar located in the QC Laboratory.
- 7.3 Sample Preparation
- 7.3.1 Connect the 72” Nitrogen Hose Assembly (Transfer Line) w/ Phase Separator to the 180 liter LN2 tank at the port labeled *Liquid*. Open the port labeled *Pressure Building* until gas can be heard exiting the safety valve, then close it.
- 7.3.2 Place the end of the transfer line into the 5 Liter dewar and slowly open the *Liquid* valve. Do not overfill the dewar! When full, the liquid level should be

- 2-4 inches below the shoulder of the dewar. Gently install the dewar cap, being careful not to damage the delicate Styrofoam cylinder.
- 7.3.3 When ready to powder the chewable gels, precool the removable grinder bowl by placing it (with lid attached) into a freezer.
- 7.3.4 Gently remove the cap from the 5 liter dewar and apply the pour spout. Tighten firmly!
- 7.3.5 Add enough LN2 to the 1 liter dewar to completely submerge the required number of chewable gels. Then, with the mouth of the 1 liter dewar pointed away from yourself, slowly introduce the gels one or two at a time.
- 7.3.6 Confirm that the sample is totally submerged, then gently replace the 1 liter dewar cap and allow to freeze for 10 minutes.
- 7.3.7 Decant off the LN2 and transfer the frozen gummies into the cold grinder bowl as quickly as possible. Grind to powder and weigh the sample as quickly as possible into an appropriately sized beaker.
- 7.3.8 Dissolve the powder in the applicable diluent, being attentive (if applicable) to any sensitivities (e.g. exposure to light, air, heat etc.) pertinent to the analyte of interest. Quantitatively transfer the contents of the beaker into the appropriate volumetric glassware.

8.0 Revision History

Revision	Date	Description of Changes	CCR #	By
0	02/10/20	New	N/A	C. Perry