| ION LABS QUALITY FIRST | Standard Operating Procedure<br>Pre-Screening Bulk and Tapped Density of Key Materials | SOP Number<br>E-706 | Revision<br>1 |
|---|---|---|---|
| | | Effective Date<br>11/18/19 | Page<br>Page 1 of 4 |

| Written by/ Date | Reviewed by/ Date | Approved by/ Date |
|---|---|---|
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| Title: R&D Supervisor | Title: QS Manager | Title: QC Laboratory Director |

## 1.0 Purpose

This procedure outlines a pre-screening system for critical density materials and provides and early warning system to identify materials lots which could affect the processability of formulas/products. This procedure also provides instructions for performing the testing necessary to identify critical density material lots through bulk and tapped density measurements.

## 2.0 Scope

This procedure applies to key raw materials that affect the processability of products manufactured by Ion Labs, Inc.

## 3.0 Responsibility

3.1 It is the responsibility of R&D to follow this procedure. R&D must document acceptable bulk and tapped density ranges on the NPA and ensure RM Profiles are updated during sign- off. R&D will also determine if a raw material can be used through planned deviation should it fall outside of the acceptable ranges.

3.2 It is the responsibility of QC Laboratory to follow this procedure. QC Laboratory will conduct the bulk and tapped density test on all critical density raw materials and will alert the QS Manager and R&D Manager when materials are out of range.

3.3 It is the responsibility of QC Laboratory personnel to quarantine unacceptable materials and coordinate with Purchasing for the return of unacceptable materials.

## 4.0 Definitions

4.1 **Bulk Density** – the density of a powder "as poured" or passively filled into a measuring vessel

4.2 **Tapped Density** – density attained after "tapping down" a powder

4.3 **QC** – Quality Control

4.4 **QS** – Quality Systems

4.5 **R&D** – Research and Development

4.6 **NPA** – New Product Approval

4.7 **RM Profile** – A Raw Material Profile states the necessary specifications that a raw material must be tested against and pass

## 5.0 References

5.1 E-706-F1, Form, Bulk and Tapped Density Pre-Screening Form

5.2 E-801, SOP, Return of Materials and Destruction of Non-Hazardous Waste Materials

## 6.0 Procedure

6.1 NPA

6.1.1 R&D will create an NPA document which will include a section of all key materials that have densities which affect the processability of products. The NPA is shared with the QC Laboratory.

6.2 RM Profile

6.2.1 QC Laboratory personnel will create the RM Profile based on critical specifications outlined in the NPA.

6.2.2 Bulk and Tapped density cannot be COA challenged and must be tested on every lot.

6.3 Bulk Density

6.3.1 QC Laboratory Personnel will collect approximately 100 grams from containers to represent the lot of received material.

6.3.2 Using Form E-706-F1 Bulk and Tapped Density Pre-Screening Form:

6.3.2.1 Pass the powder sample through a sieve to remove agglomerates.

6.3.2.2 Tare a 100ml graduate cylinder of suitable dimensions for the AS-100 Tap Density Tester.

6.3.2.3 Gently transfer via a funnel sufficient powder to fill 50ml to 60ml of the cylinder with minimal shaking or vibrations.

6.3.2.4 Record weight on Form E-706-F1.

6.3.2.5 Determine the volume by gently moving the graduated cylinder to create a flat surface for accurate line to determine measured volume. Using only whole numbers, record as volume (b) on Form E-706-F1.

6.3.2.6 Density is calculated using the following equation:

- Density = (a) weight (gm) / (b) volume (ml)

6.3.2.7 Compare the results with the acceptable ranges specified on the RM Profile and determine if it has passed or failed. Record findings on Form E-706-F1 and continue with tapped density testing.

6.4 Tapped Density

6.4.1 Utilizing the sample used to test the bulk density, fix the cylinder to the AS-100 Tap Density Tester and tap 1000 cycles at a distance of 3mm +/- 0.2mm between 100-300rpm. This process should take about 5 minutes.

6.4.2 Measure and record the apparent volume of the powder on Form E-706-F1.

6.4.3 Density is calculated using the following equation:

6.4.3.1 Density = (c) weight (gm) / (d) volume (ml)

6.4.4 Compare the results with the acceptable ranges specified on the RM Profile and determine if it has passed or failed. Record findings on Form E-706-F1 and continue to notification step.

6.5 Notification

6.5.1 Pass - If the critical density material has a bulk and tapped density within the acceptable ranges provided on the RM Profile.

6.5.1.1 QC Laboratory Supervisor will review results reported on Form E-706-F1.

6.5.1.2 Form E-706-F1 is added to Raw material testing packet be delivered to Document Control to be scanned and filed.

6.5.2 Fail – if the critical density material has a bulk or tapped density that fall outside of the acceptable ranges provided on the Raw Material Profile, QC Laboratory must immediately notify R&D Manager via email or phone.

6.5.2.1 Form E-706-F1 will be forwarded to R&D after notification.

6.5.2.2 R&D will evaluate the material and determine if the material can be used or should be returned.

6.5.2.3 If the material is to be returned:

- R&D/QC/Purchasing will follow SOP E-801 Return of Materials and Destruction of Non-Hazardous Waste Materials to have the material returned to the vendor.

- QC will ensure that the material is labeled appropriately and placed in quarantine.

6.5.2.4 If the material can be used:

- R&D will determine any changes that need to be made in order to use the material (i.e. roller compaction, fill reduction, fill increase, etc.)

- R&D will initiate a planned deviation to add processes and/or make formula revisions in order to use the material.

## 7.0 Revision History

| Revision | Date | Description of Changes | CCR # | By |
|---|---|---|---|---|
| 0 | 06/13/16 | New | 16-0424 | L. Titolo |
| 1 | 09/12/19 | Updated SOP to reflect current practice. Deleted reference to critical density material list and added RM Profile and NPA references. | 19-0663 | K. Westphal |

| ION LABS QUALITY FIRST | Bulk and Tapped Density Pre-Screening Form | | |
|---|---|---|---|
| | Form: E-706-F1 | CCR No. 19-0663 | Revision: 1 |

Raw Material _____Test Date: _____ Time: _____

RMID: _____ R # _____ Customer Lot# _____

**Bulk Density**

Instructions: QC will collect approximately 100 grams from the lot of material to be tested.

Method:

- Tare a 100 ml graduated cylinder and gently transfer via a funnel sufficient powder to fill 50 ml to 60 ml of powder with minimum shaking or vibrations.
- Weigh the net weight of powder transferred into 100 ml graduated cylinder, record as weight (a).
- Determine volume by gently moving graduated cylinder to create a flat surface for accurate line to determine measured volume using only whole numbers, record as volume (b).

Calculations: Density = weight (gm) / volume (ml)

Acceptance Range: Acceptance Range attained from RM Profile Dated _____

Bulk Density Range Min _____ g/ml Max_____ g/ml

**Results:** Bulk Density

| Sample | a. Weight (gm) | b. Volume (ml) | Bulk Density (a / b) | Pass / Fail |
|---|---|---|---|---|
| Bulk sample | | | | |

**Tapped Density**

Method:

- Transfer the weight in Bulk Density section (a) to weight in Tapped Density section (c)
- Utilizing the sample used to test bulk density, Fix the cylinder to the AS-100 Tap Density Tester and tap 1000 cycles at a distance of 3mm +/- 0.2mm between 100-300 rpm. The process should take about 5 minutes.
- Determine volume by gently moving graduated cylinder to create a flat surface for accurate line to determine measured volume using only whole numbers, record as volume (d).

Calculations: Density = weight (gm) / volume (ml)

Acceptance Range: - Acceptance Range attained from RM Profile Dated _____

Tapped Density Range Min _____ g/ml Max_____ g/ml

**Results:** Tapped Density

| Sample | c. Weight (gm) | d. Volume (ml) | Tapped Density (c / d) | Pass / Fail |
|---|---|---|---|---|
| Tapped sample | | | | |

***IF MATERIAL IS NOT WITHIN ACCEPTANCE RANGE, NOTIFY QC AND R&D IMMEDIATELY**

**Performed by:** _____ **Date:** _____