1.0 Purpose

The purpose of this SOP is to define the method for the determination of the related compounds of glycerin in raw materials by GC using flame ionization detection.

2.0 Scope

This procedure applies to the determination of the related compounds of glycerin in raw materials by the QC laboratory at ION Labs.

3.0 Responsibility

3.1 It is the responsibility of QC Chemists to follow this procedure.

3.2 It is the responsibility of QC Laboratory Management to ensure that this procedure is being followed.

3.3 It is the responsibility of QC Laboratory and Analytical Development Management to keep this SOP aligned with current practices.

4.0 Definitions

4.1 **GC** – Gas Chromatography

4.2 **CofA** – Certificate of Analysis

4.3 **DEG** – Diethylene Glycol

4.4 **GLY** – Glycerin

4.5 **RT** – Retention Time

5.0 References

5.1 PRTCL-20-0117: Verification of an Analytical Method for the Organic Impurities of Glycerin

5.2 USP Monograph for Glycerin

6.0 Supplies

6.1 Chemicals: All reagents are GC grade or better.

6.1.1 DEG Reference Standard

6.1.2 GLY Reference Standard

6.1.3 Methanol

6.2 Compressed Gases

6.2.1 Hydrogen

6.2.2 Helium

6.2.3 Air

6.2.4 Nitrogen

6.3 Glassware

6.3.1 Volumetric glassware as required by standard and sample preparations

6.4 Equipment

6.4.1 Agilent 7890 GC

6.4.2 Analytical Balance

7.0 GC Conditions

7.1 Column: ZB-624, 30 m x 0.32 mm x 1.8 µm or equivalent

7.2 Inlet Liner: Restek, 4.0 mm ID x 6.3 mm OD x 78.5 mm length straight liner with glass wool or equivalent

7.3 Inj Temp: 220 °C

7.4 Det Temp: 250 °C

7.5 Equil Time: 0.5 min

7.6 Run Time: 20 min

7.7 Split ratio: 10:1

7.8 Septum purge: 3 mL/min

7.9 Air flow: 350 mL/min

7.10 Fuel flow: 30 mL/min

7.11 Makeup flow: 30 mL/min (column + makeup = constant)

7.12 Injection Volume 0.5 µL

7.13 Injection Type Standard

7.14 Plunger Speed Fast

7.15 Wash Solvent Methanol

7.16 Temperature Ramp:

Ramp Rate (°C/min)	Temp (°C)	Hold Time (min)
N/A	100	0
7.5	220	4

7.17 Flow Ramp:

Ramp Rate (mL/min)	Flow Rate (mL/min)	Hold Time (min)
N/A	2.1	0
0.025	1.7	4

8.0 Diluent

8.1 Use methanol.

9.0 Working Standard Preparation

9.1 Transfer 25 ± 2.5 mg of GLY reference standard to a small beaker or vial.

9.2 Transfer 25 ± 2.5 mg of DEG reference standard to a small beaker or vial.

9.3 Use small portions of Diluent to quantitatively transfer both reference standards to a single 50-mL volumetric flask.

9.4 Dilute to volume with *Diluent* and mix well.

10.0 Sample Solution Preparation

10.1 Transfer 1.25 ± 0.10 g of sample into a 25-mL volumetric flask.

10.2 Dilute to volume with *Diluent* and mix well.

11.0 Recommended Sequence

11.1 Make a single injection of the Blank (Diluent).

11.2 Make a single injection of the Working Standard.

11.3 Make a single injection of each Sample Preparation.

12.0 System Suitability Requirements

12.1 The USP resolution between DEG and GLY in the injection of *Working Standard* is NLT 7.0.

12.2 No significant (>0.1%) peaks are present in the blank injection.

13.0 Retention Times

13.1 DEG 7.03 min

13.2 GLY 7.85 min

14.0 Evaluation of Limit

14.1 Exclude any peak eluting prior to 2.5 min for calculations.

14.2 Exclude any peak that is present in the blank injection.

14.3 Calculate the percent area for each impurity peak with concentration higher than 0.01%.

14.4 Calculate the sum of the all impurity peaks with concentration higher than 0.01%.

15.0 Revision History

Revision	Date	Description of Changes	CCR #	By
0	01/11/21	New	N/A	S. Sassman