

**Determination of Cyclical Volatile Siloxane Compounds in Seals Using Thermal Extraction and Gas Chromatography-Mass Spectrometry (GC-MS)****Application**

This procedure is used to quantitatively determine thermal extraction rates of selected cyclical volatile siloxane compounds (D3, D4, D5, D6 and D9) in elastomeric materials. Other cyclical volatile siloxane compounds (D7, D8, others) can be determined semi-quantitatively.

Apparatus and Materials Required**Thermal Desorption Tubes for the Gerstel Twister Thermal Desorption System (supplier Gerstel):**

Tubes with glass frit (Gerstel part # 013742-005-0)

Tubes with Tenax GR (Gerstel part # 015953-005-0)

GC-MS Chromatographic Column:

DB-5 MS, column dimensions: length: 30 m, diameter: 0.25 mm, film thickness: 0.25 μ m

(Supplier: Agilent or equivalent)

Gerstel Twister Inlet Liner: CS4 liner with silanized glass wool; ID=1 mm; length= 76 mm;

Gerstel Part# 013409-010-00

GC-MS Carrier and Gerstel ThermoExtractor Conditioning Unit Gas: Ultra High Purity Helium

(Supplier: AirGas or equivalent)

Individual Standards: neat dimethylsilicone cyclic compounds (purity 99.8% or better):

D3 (Cyclotrisiloxane, hexamethyl-; CAS # 541-05-9)

D4 (Cyclotetrasiloxane, octamethyl-; CAS # 556-67-1)

D5 (Cyclopentasiloxane, decamethyl-; CAS # 541-02-6)

D6 (Cyclohexasiloxane, dodecamethyl-; CAS # 540-97-6)

D9 (Cyclononasiloxane, octadecamethyl-; CAS # 556-71-8)

(Supplier: Ohio Valley Specialty Company or equivalent)

Glass volumetric cylinders: 10 mL and 50 mL

Pipets (9", Fisher Brand)

Stainless Steel Surgical Blade

Syringe: 10 μ L

Liquid Nitrogen: low pressure liquid nitrogen required for GC-MS "Cryo Cooling"

(Supplier: AirGas or equivalent)

Solvent: Methanol, certified (Supplier: Fisher Scientific or equivalent)

Thermal Desorption System: Gerstel Twister Thermal Desorption Unit (TDU) with MPS2 auto-sampler

Chromatography System: Agilent Gas Chromatograph, 6890N
Agilent Mass Spectrometer, 5975B VL MS

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2016 10 21	Released		

Documents
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Controlling Software: Agilent Chemstation GC-MS, Data Acquisition/Data Analysis
Software Version: D.03.00, SP1
Gerstel MASTer, Thermal Extraction Control Software, Version 2.8.0.0

Tube Conditioner: Gerstel TubeConditioner, model: TC (with a metal extension for short tubes)

Analytical Balance: Mettler-Toledo, model: PM400

Note: All the above listed equipment and/or software may be substituted with alternatives of equivalent performance.

Chemicals, materials, parts, and equipment referenced in this document must be used and handled properly. Each party is responsible for determining proper use and handling in its facilities.

Procedure

Preparation of standard and quality control solutions:

Prepare a stock standard solution in methanol (1,000 µg/mL mixture of each target volatile siloxane compound).

From the stock standard solution prepare calibration standards of 250 and 500 µg/mL mixtures of each compound by serial dilutions by volume.

Separately prepare a solution of the 500 µg/mL mixture of each compound from the stock solution. This solution will be used as a quality assurance /quality control check solution (QA/QC).

Store the solutions in sealed vials in a refrigerator; the solutions are stable for 2 months.

