

**METHYLTRICHLOROACETATE IN URINE BY GC/MS Headspace**  
**Code GC15510**

**BIOCHEMISTRY**

The Methyltrichloroacetate is trichloroacetic acid methyl ester. The latter is a solvent used fumigant for dry cleaning and degreasing as. It is absorbed through inhalation and dermal dose resulting, in a high action, dermal irritant and with a nephrotoxic effect. Was recovered in the urine of subjects professionally exposed to vapours of trichlorethylene and other chlorinated solvents, or urine drug used to sniff solvents or in cases of deliberate or accidental ingestion of trichlorethylene or derivatives. The proposed method determines the Methyltrichloroacetate injected into GC-MS / headspace of a urine sample rate earlier in an appropriate solvent. The levels are determined on a urine sample at the beginning and end of shift.

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This product fulfills all the requirements of Directive 98/79/EC on in vitro diagnostic medical devices (IVD). The declaration of conformity is available upon request.

Release N° 002	Methyltrichloroacetate in urine by GC/MS-headspace	March 2009
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## **TECHNICAL FEATURES**

### **Principle of the Method :**

This method allows to determine the Methyltrichloroacetate urine by GC-MS / headspace. Methyltrichloroacetate is first diluted and then is directly injected into GC.

**Recovery :** 96%

**Sensitivity :** 0,5 µg/l by MS

**Dynamic Range of the Method:** 1 – 10.000 µg/l by MS

**Normal Values:** n.d.

### **Components of the kit :**

All the Reagents are ready-to-use and stable 3 years at Temperature ≤ 20 °C.

**Reagent A** – Diluting Solution, 1 x 50 ml

**Reagent B** – Test Solution / Chemical Standard, 1 x 5 ml **Store at 2-8 °C**

### **Minimum Instrumental equipment required:**

GC/MS  
Operational Computer

### **Optional Equipment:**

Autosampler

### **Urine Collection Procedure:**

After work shift, collect 10 ml of urine in a suitable test tube.

Stable 7 days at 4 °C.

Stable 4 weeks at –20 °C.

## **PREANALYTICAL PROCEDURE**

Preparation of Test Solution.

Dispense in a vial of 10ml:

- 1800 µl of **Reagent A – Diluting Solution**
- 200 µl of **Reagent B – Test Solution**

Inject 1000 µl of solution into gas chromatograph GC. Verify that the Test solution has retention time similar to fig. 1. If the Test is all right you can start with the analytical procedure; if not, check the functionality of the analytical system.

## **ANALYTICAL PROCEDURE**

**STEP 1:** Preparation of Calibration Standard Solution at 150 µg/l

Dispense in a tube:

- 1800 µl of **Urine**
- 200 µl of **Reagent B – Chemical Standard**

**IMPORTANT: This Calibration Standard Solution is prepared from time to time**

To verify that the urine is effectively free from Methyltrichloroacetate, to prepare a Blank:

- 1800 µl of **Urine**
- 200 µl of **H<sub>2</sub>O HPLC grade**

**If the Blank urine presented the peak of Methyltrichloroacetate, proceed to identify the quantitative difference between the areas / heights of the peaks of Methyltrichloroacetate in urine enriched and blank.**

- The samples are directly injected transfer 2 ml in GC vials.

**Close vials hermetically**

**Mix at Vortex**

**Place them in the Sampler**

***Prepare work list and start the series according to the system's specifications***

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## **METHYLTRICHLOROACETATE - Warnings**

### **REAGENT B : TEST SOLUTION / CHEMICAL STANDARD**

<b>METHYLTRICHLOROACETATE</b>	1500 µg/l
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### **SET OF GAS-CROMATOGRAPH:**

<ul style="list-style-type: none"><li>• VF-5ms Column 30 m x 0,25 mm, 0,25 µm (conditioned)</li><li>• Injector's Temperature 200 °C</li><li>• Temperature 40 °C x 2 minutes + 5 °C/min up to 100 °C + 10 °C/min up to 160 °C (run 20 min)</li><li>• Helium Gas 1 ml/min</li><li>• Split Report Start: OFF; 10' ON Split 10</li><li>• Injection Splitless with opening after 0,10 min</li><li>• Syringe Temperature: 70 °C</li><li>• Mixer Temperature: 120 °C (mix and heat for 2 minutes)</li></ul>
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### **SET OF MASSA DETECTOR (4000):**

<ul style="list-style-type: none"><li>• Range of Massa 50 – 300</li><li>• Operative Method in Electronic Impact (EI)</li><li>• Set in SIM at Characteristic Masses – 83 – 85</li><li>• Temperature of Massa: 180 °C;</li><li>• Filament on: 1,5 minutes</li></ul>
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### **CONDITIONING OF VF-5 MS COLUMN**

Follow the manufacturer prescription. Do not condition column/s if connected to the Massa Detector.
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### **CLEANING OF THE COLUMN**

Disconnect the detector. Keep the column at the highest temperature for the recommended time. (See the manufacturer 's instructions)
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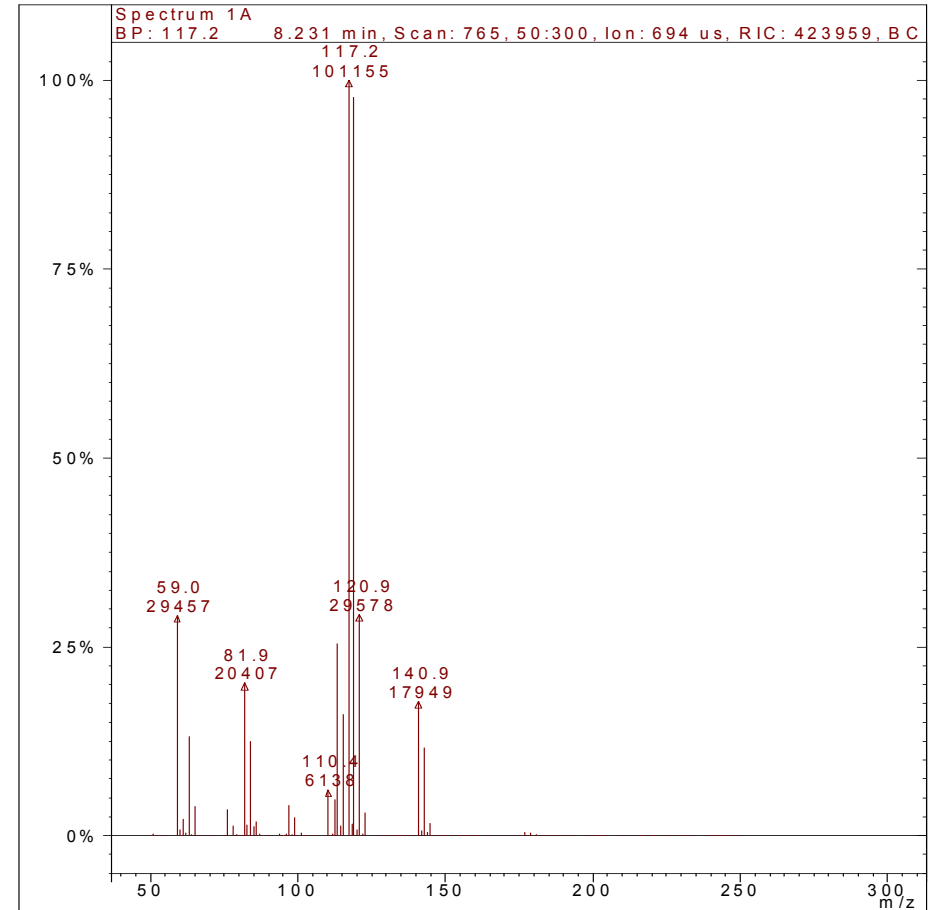
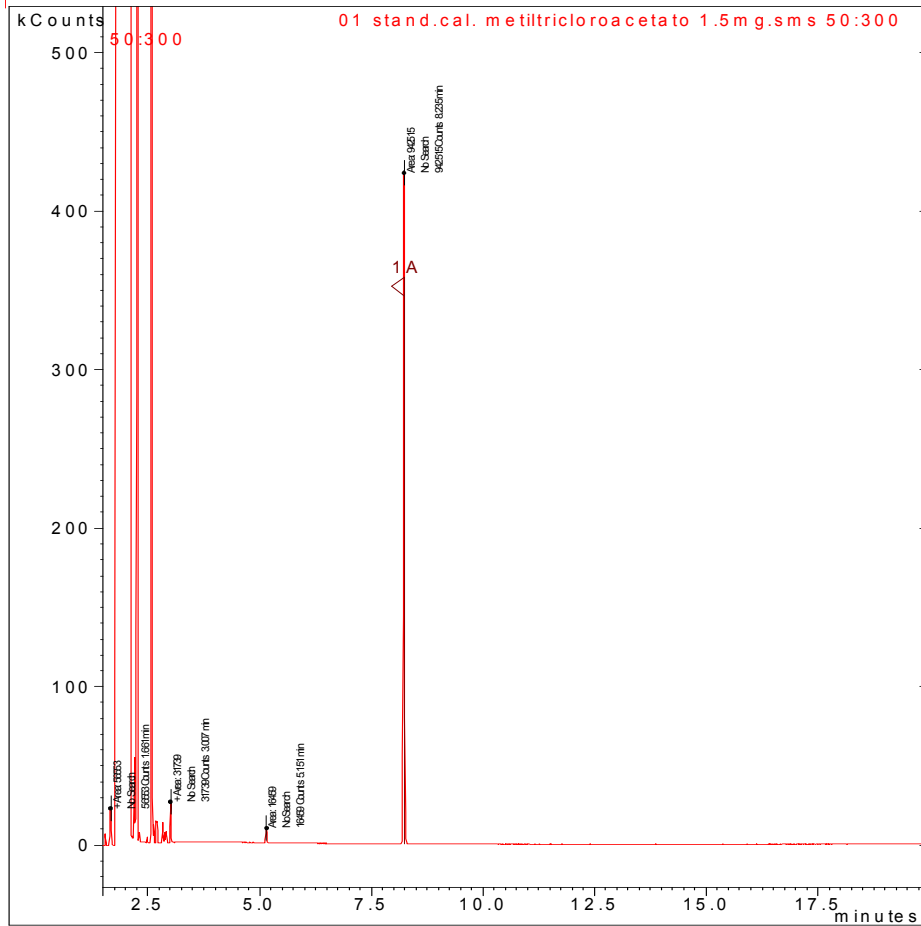
### **ACCESSORIES AND CONSUMABLES**

<b>CODE</b>	<b>DESCRIPTION</b>	<b>PACKAGING</b>
<b>GC15520</b>	Chemical Standard for Methyltrichloroacetate	1 x 2 ml
<b>ZRE13623</b>	Rxi-Sil 5MS (30 m x 0,25 mm) ID, DF=0,25 Capillary column	1 Pc
<b>SCP8944</b>	VF-5ms (30 m x 0,25 mm) ID DF=0,25 column	1 Pc
<b>S51834475</b>	Glass vial of 10 ml for headspace	1 x 100 Pcs
<b>S80100165</b>	Magnetic caps with teflon and sylicon septa for headspace vials	1 x 100 Pcs
<b>S50404669</b>	New Easy Grip Manual Crimper for 20 mm	1 x 100 Pcs



# METHYLTRICHLOROACETATE IN URINE

(Reference Chromatograms/Spectrums GC-MS / Headspace)

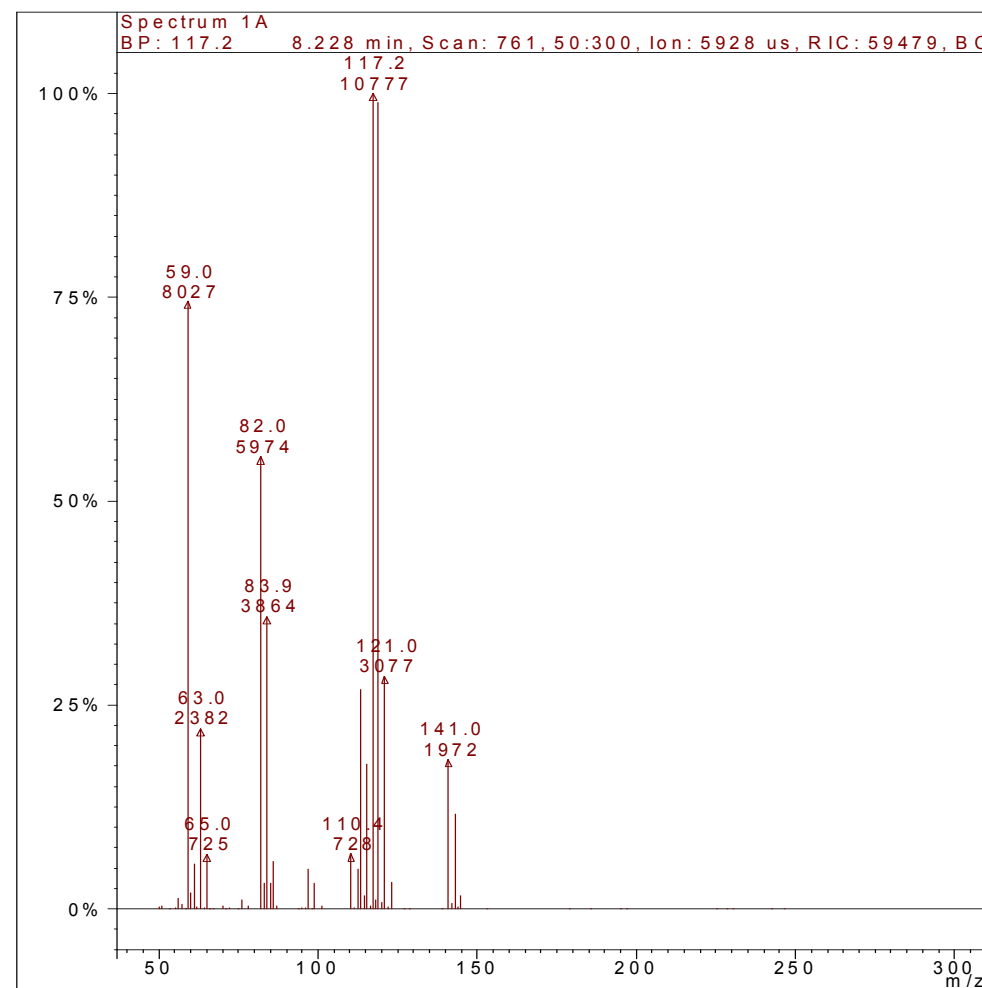
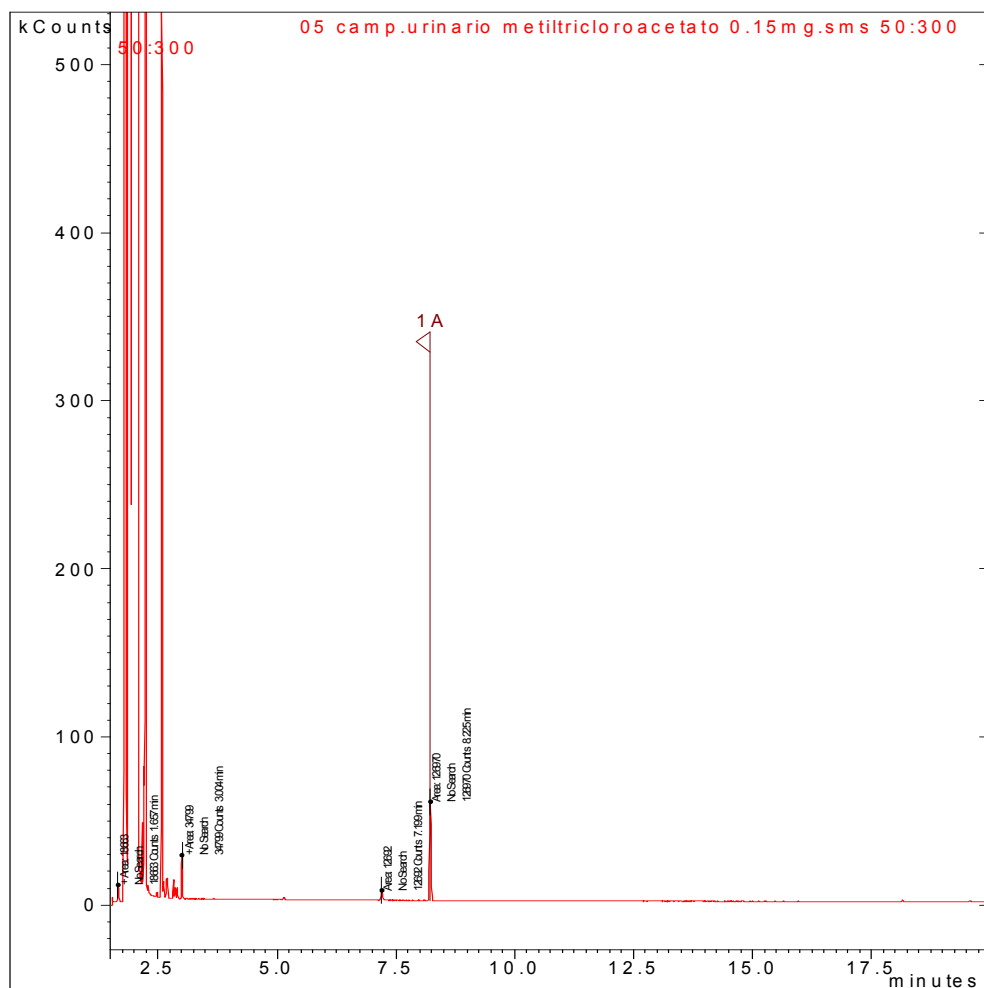


<b>Fig. 1 :</b>	<b>Calibration Standard</b>
	R.T. 8.2 METHYLTRICHLOROACETATE 150 µg/l

<b>Fig. 2 :</b>	<b>Massa Spectrum of Calibration Standard</b>
	MOLECULAR ION : 117

# METHYLTRICHLOROACETATE IN URINE

(Reference Chromatograms/Spectrums GC-MS / Headspace)



<b>Fig. 3 :</b>	<b>Urine Sample</b>
	R.T. 8.2 METHYLTRICHLOROACETATE 15 µg/l

<b>Fig. 4 :</b>	<b>Massa Spectrum of Urine Sample</b>
	MOLECULAR ION : 117