

TETRACHLOROETHYLENE IN URINE BY GC/MS Headspace
Code GC15010

BIOCHEMISTRY

The Tetrachlorethylene or perchlorethylene, is an organic halide. Its structure is similar to that of a molecule of ethene which 4 hydrogen atoms have been replaced by four chlorine atoms. At room temperature is presented as a colorless liquid smelling of chlorine. It is a compound harmful if inhaled and harmful to the environment, and it is not flammable. Tetrachlorethylene is used as a degreaser in dry cleaning industry, in the chemical and pharmaceutical industries, in domestic use. Exposure to high concentrations of tetrachlorethylene can cause liver damage and kidney. The toxicity is related to metabolism via mercapturic acids with formation of premercapturic acid trichlorovinylcysteine and tiokethene activation via β -lyase. The proposed method determines the tetrachlorethylene injecting GC-MS/headspace rate of urine sample earlier in appropriate solvent. The levels of tetrachlorethylene 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.

N° 004	Tetrachloroethylene in urine by GC/MS-headspace	11/2024
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TECHNICAL FEATURES

Principle of the Method :

This method determines the tetrachlorethylene urinary GC / MS headspace. The tetrachlorethylene is first diluted and then is directly injected into GC.

Recovery: 100%

Sensitivity: 10 µg/l by MS

Dynamic Range of the Method: 10 – 2.000 µg/l by MS

CV%: 3,3 intra serie
4,3 inter serie

Normal Range: < 100 µg/l exposed

*Ref. Giornale italiano di Igiene Industriale e Ambientale, ACGIH,
Aprile 2010*

Components of the kit :

All the reagents are ready-to-use and stable 3 years at 2 – 8 °C.

Reagent A – Diluting Solution, 1 x 200 ml

Reagent B – Stabilizing Solution, 1 x 5 ml

Reagent C – Test Solution / Chemical Standard, 2 x 2 ml

**For the sampling of Reagent C, the cap
sealing requires the use of 100 ul Hamilton
syringe Cod. ZRE24563
Store at 2-8 °C**

Minimum Instrumental equipment required:

GC/MS
Operational Computer

Optional Equipment:

Autosampler for headspace

Urine Collection Procedure:

After work shift, collect 10 ml of urine in a suitable glass test tube hermetically sealed (Teflon coated aluminum caps and stoppers). The transfer of the sample must be made as soon as possible.

Stable 7 days at 4 °C.

PREANALYTICAL PROCEDURE

Preparation of Test Solution.

Dispense in a vial of 10 ml for headspace:

- 1800 µl of **Reagent A – Diluting Solution**
- 200 µl of **Reagent C – Test Solution/Chemical Standard**

Close vials hermetically and mix at Vortex for 10 sec.

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

Inject 1000 µl of solution into gas chromatograph GC. Verify that the Test solution has retention time similar to fig. below. If the Test is all right you can start with the analytical procedure; if not, check the functionality of the analytical system. **N.B.: the test solution should not be used to calibrate the samples.**

ANALYTICAL PROCEDURE

STEP 1: Preparation of Calibration Standard Solution at **202,5 µg/l**

Dispense in a tube:

- 1950 µl of **Urine without Tetrachloroethylene**
- 50 µl of **Reagent C – Test Solution/Chemical Standard**

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

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

- 1950 µl of **Urine**
- 50 µl of **Reagent B – Stabilizing Solution**

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

STEP 2 : Sample Preparation

Dispense in vials of 10 ml for headspace:

	Calibration Standard	Control	Sample	Blank
Calibration Standard	200 µl			
Control		200 µl		
Sample			200 µl	
Blank				200 µl
Reagent A – Diluting Sol.	1800 µl	1800 µl	1800 µl	1800 µl

Close vials hermetically

Mix at Vortex

Place them in the Sampler

STEP 3 : Inject 1000 µl in GC with a syringe dedicated to Tetrachloroethylene only

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

TETRACHLOROETHYLENE - Warnings

REAGENT C : TEST SOLUTION / CHEMICAL STANDARD

TETRACHLOROETHYLENE	8.100 µg/l
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SET OF GAS-CROMATOGRAPH:

- Restek Rxi-5Sil MS Column 30 m x 0,25 mm, 0,25 µm (conditioned)
- Injector's Temperature 200 °C
- Temperature 70 °C x 2 minutes + 20 °C/min up to 160 °C (run 6,5 min)
- Helium Gas 1 ml/min
- Split Report Start: 1:40

SET OF AUTOSAMPLER COMBIPAL for HEADSPACE:

- Syringe Temperature: 90°C
- Mixer temperature: 90 °C (incubation x 4 min at 750 rpm)
- Agitator ON: 5sec
- Agitator OFF: 2sec
- Plunger Fill Speed: 500ul/sec
- Plunger Injection Speed: 1000ul/sec
- Fill Strokes: 0
- Viscosity Delay: 1sec
- Pre-Injection Delay: 0.1sec
- Post-Injection Delay: 0.1sec
- Syringe Flush Time: 30sec

SET OF MASSA DETECTOR:

- Range of Massa 30 – 200
- Temperature of Massa: 180 °C
- Temperature of Transfer Line: 270 °C
- Temperature of Manifold: 80 °C
- Filament on: 1 minute

CONDITIONING OF VF-5 MS COLUMN

Follow the manufacturer prescription.
Do not condition column/s if connected to the Massa Detector.

CLEANING OF THE COLUMN

Disconnect the detector. Keep the column at the highest temperature for the recommended time. (See the manufacturer's instructions)

NB: We recommend using a syringe of the autosampler for headspace ONLY for the injections of tetrachloroethylene to avoid any carryover effects and / or interfering due to other types of methods.

ACCESSORIES AND CONSUMABLES

CODE	DESCRIPTION	PACKAGING
GC15020	Chemical Standard for Tetrachloroethylene	1 x 2 ml
ZRE13623	Rxi-Sil 5MS (30 m x 0,25 mm) ID, DF=0,25 Capillary column	1 Pc
SCP8944	VF-5ms (30 m x 0,25 mm) ID DF=0,25 column	1 Pc
S51834475	Glass vial of 10 ml for headspace	1 x 100 Pcs
S80100165	Magnetic caps with teflon and sylicon septa for headspace vials	1 x 100 Pcs
S50404669	New Easy Grip Manual Crimper for 20 mm	1 x 100 Pcs

TETRACHLOROETHYLENE IN URINE

(Reference Chromatograms/Spectrums GC-MS / Headspace)

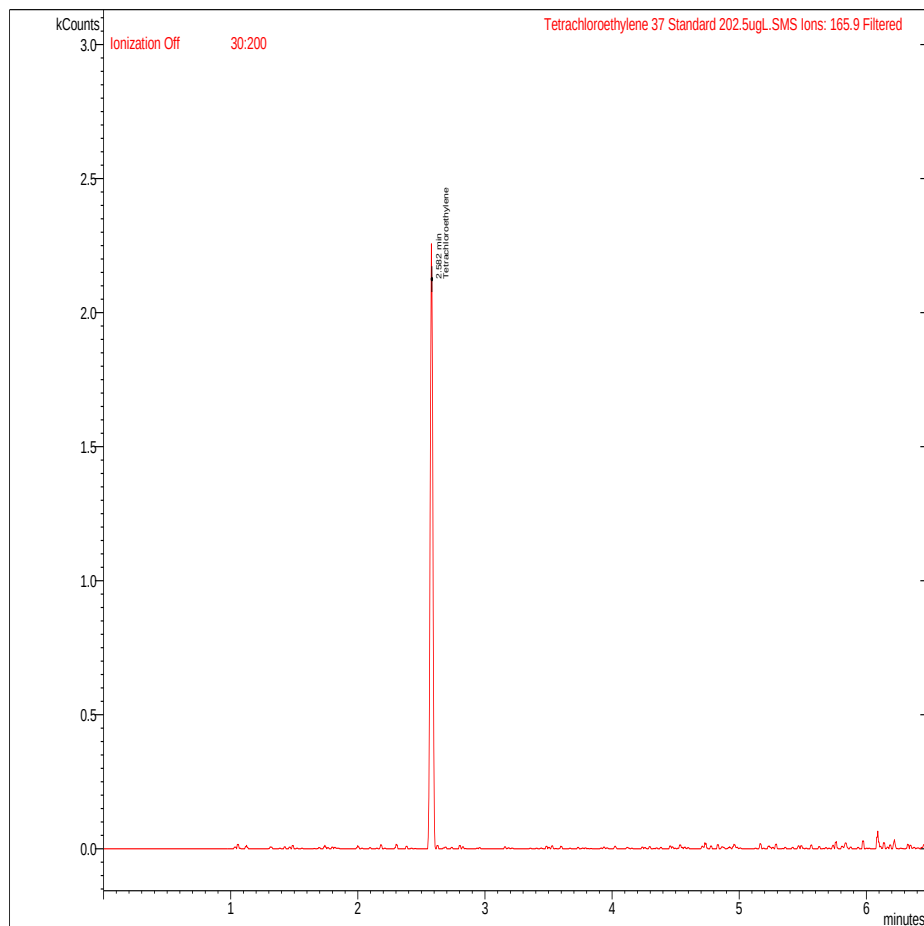


Fig. 1 :	Calibration Standard
	R.T. 2.6 TETRACHLOROETHYLENE 202,5 µg/l

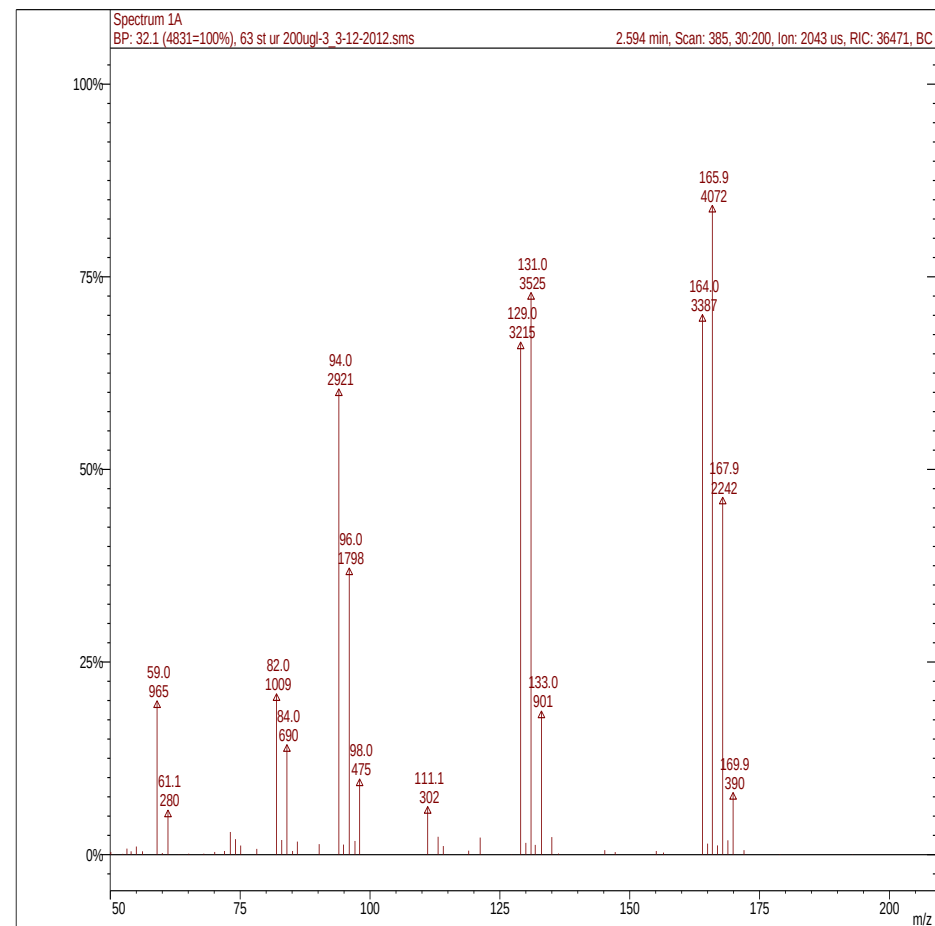


Fig. 2 :	Massa Spectrum of Calibration Standard
	MOLECULAR IONS : 166-131-94

TETRACHLOROETHYLENE IN URINE

(Reference Chromatograms/Spectrums GC-MS / Headspace)

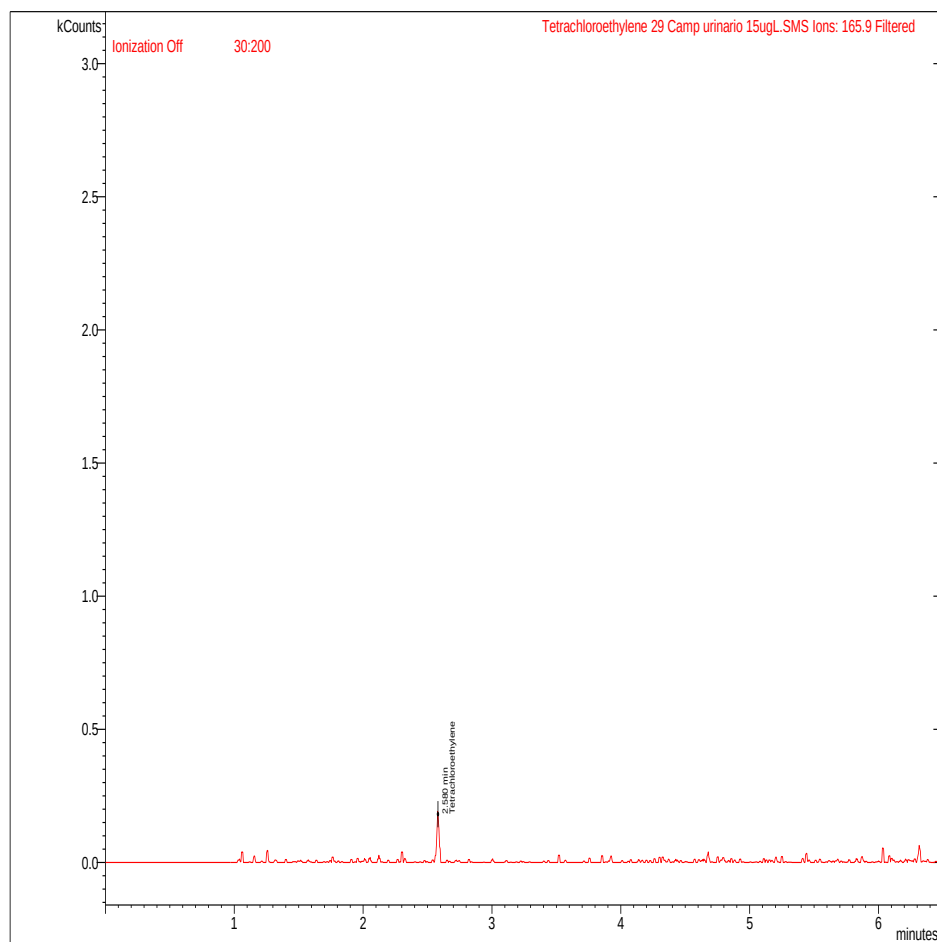


Fig. 3 :	Urine Sample
	R.T. 2.6 TETRACHLOROETHYLENE 15 µg/l

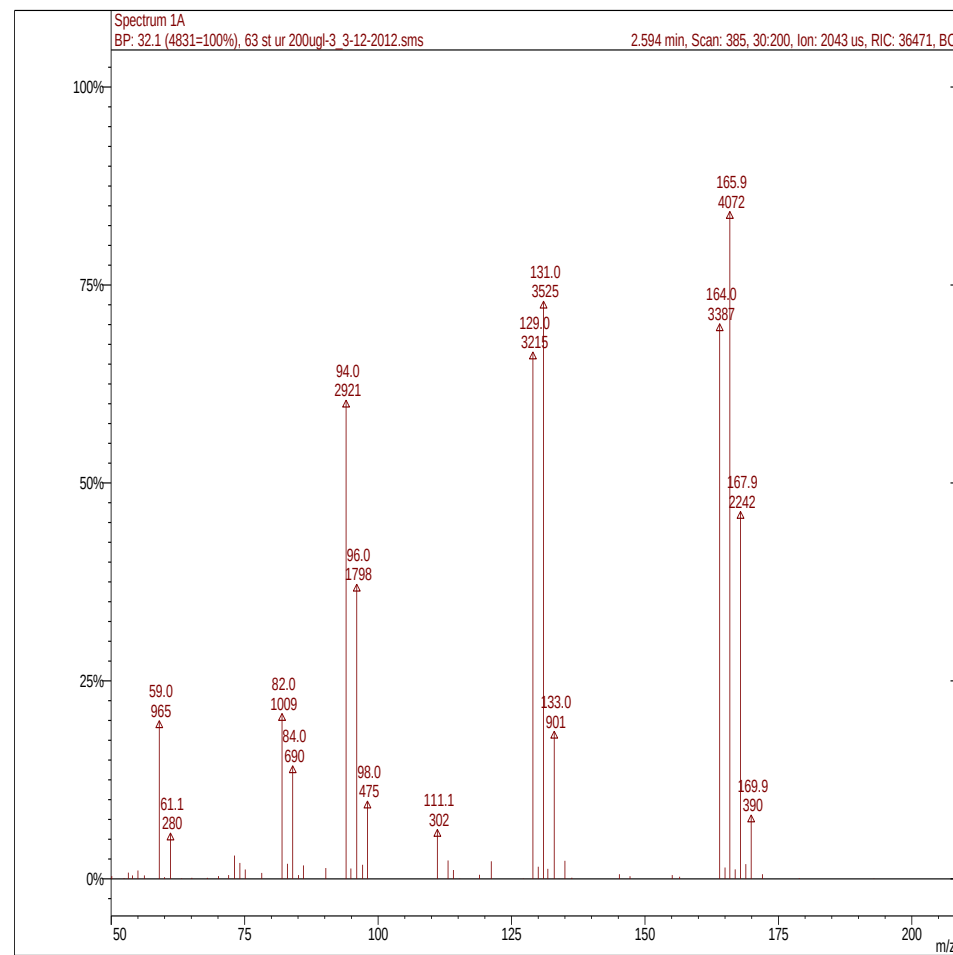


Fig. 4 :	Massa Spectrum of Urine Sample
	MOLECULAR IONS : 166-131-94