

METHYLISOBUTYLKETONE (4-methyl-2 pentanone) IN URINE BY GC/MS
Headspace
Code GC10510

BIOCHEMISTRY

The methyl isobutyl ketone is a colorless and flammable liquid that is used as organic solvent in the laboratory and in industry as a solvent in paints, glues, dyes, etc. The absorption of methyl isobutyl ketone occurs mainly through the respiratory system and to a lesser extent, through the skin so it can be irritating to skin and mucous membranes. Exposure to high concentrations may cause dizziness, nausea, vomiting under rapidly reversible after exposure ceased. The proposed method determines the injection of methyl isobutyl ketone by GC-MS / headspace of a rate of urine sample previously treated in an appropriate solvent. Levels of methyl isobutyl ketone 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° 007	Methylisobutylketone in urine by GC/MS-headspace	11/2024
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TECHNICAL FEATURES

Principle of the Method :

This method enables the methyl isobutyl ketone urine by GC-MS / head space. The methyl isobutyl ketone is first diluted and is directly injected into GC.

Recovery: 100%

Sensitivity : 0,08 mg/l by MS

Dynamic Range: 0,08 – 1.600 mg/l by MS

Reference Range: Exposed < 1.0 mg/l urine after work shift

ACGIH 2020

Components of the Kit :

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

Reagent A – Diluting Solution, 1 x 200 ml

Reagent B – Test Solution 160 mg/l, 2 x 2 ml

For the sampling of Reagent B, the cap sealing requires the use of 100 ul Hamilton syringe Cod ZRE24563

Minimum Instrumental equipment required:

GC/MS
Autosampler for headspace
Operational Computer

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 about 7 days at 2-8 °C.

PREANALYTICAL PROCEDURE

Preparation of Test Solution

STEP 1 : Dispense in a headspace vial of 10 ml:

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

Close vials hermetically and mix at Vortex.

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. N.B.: the test solution should not be used to calibrate the samples

ANALYTICAL PROCEDURE

STEP 1 : Preparation of Calibration Standard Solution at **16 mg/l**

Dispense in a tube:

- 900 µl of **Urine free from Methyl Isobutyl Ketone**
- 100 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 Methyl Isobutyl Ketone, to prepare a Blank:

- 900 µl of **Urine**
- 100 µl of **H₂O HPLC grade**

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

STEP 2 : Sample preparation

Dispense in a headspace vial of 10 ml:

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

- The samples are directly injected transfer 1 ml in GC vials.

Close vials hermetically

Mix at Vortex

STEP 3 : Inject 1000 µl in GC with a syringe dedicated to Methyl Isobutyl Ketone only

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

METHYLISOBUTYLKETONE - Warnings

REAGENT B : TEST SOLUTION

METHYLISOBUTYLKETONE	160 mg/l
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SET OF GAS-CROMATOGRAPH:

- Factor Four VF-5ms Column 30 m x 0,25 mm, 0,25 µm (conditioned)
- Injector's Temperature 200 °C
- Temperature Column Heater: 40 °C x 4 minutes + 16 °C/min up to 200 °C (run 14 min)
- Helium Gas 1 ml/min
- Splitting ratio 1:20

SET OF AUTOSAMPLER COMBIPAL for HEADSPACE:

- Syringe Temperature: 80°C
- Mixer temperature: 80 °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 (4000):

- Range of Massa 30 – 110
- Temperature of Manifold: 80 °C
- Temperature of Massa: 180 °C
- Temperature of Transfer Line: 270 °C
- Filament ON: 1min

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 methyl isobutyl ketone to avoid any carryover effects and / or interfering due to other types of methods.

ACCESSORIES AND CONSUMABLES

CODE	DESCRIPTION	PACKAGING
GC10520	Chemical Standard for Methylisobutylketone	1 x 2 ml
ZRE13623	RxiSil 5-MS (30 m x 0,25 mm) 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

METHYLISOBUTYLKETONE IN URINE

(Reference Chromatograms/Spectrums GC-MS / Headspace)

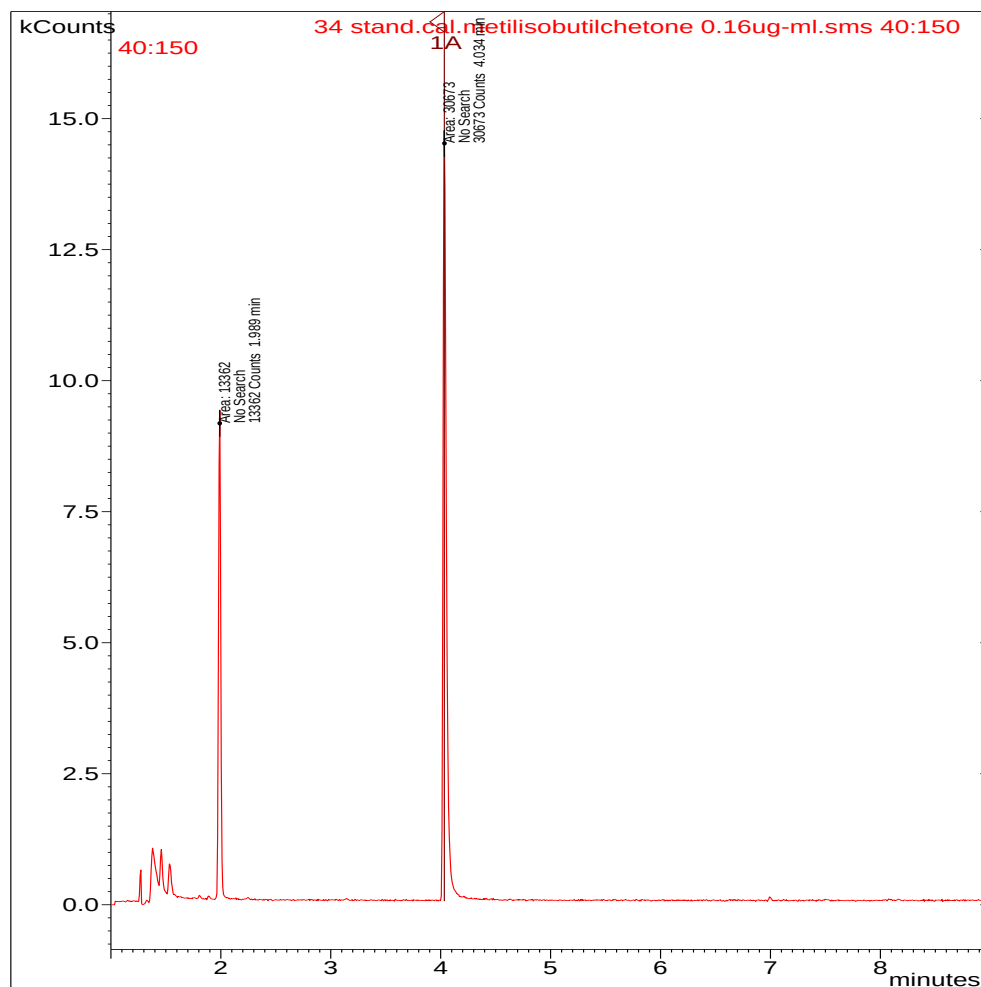


Fig. 1 :	Calibration Standard
	R.T. 4.2 METHYLISOBUTYLKETONE 0,16 mg/l

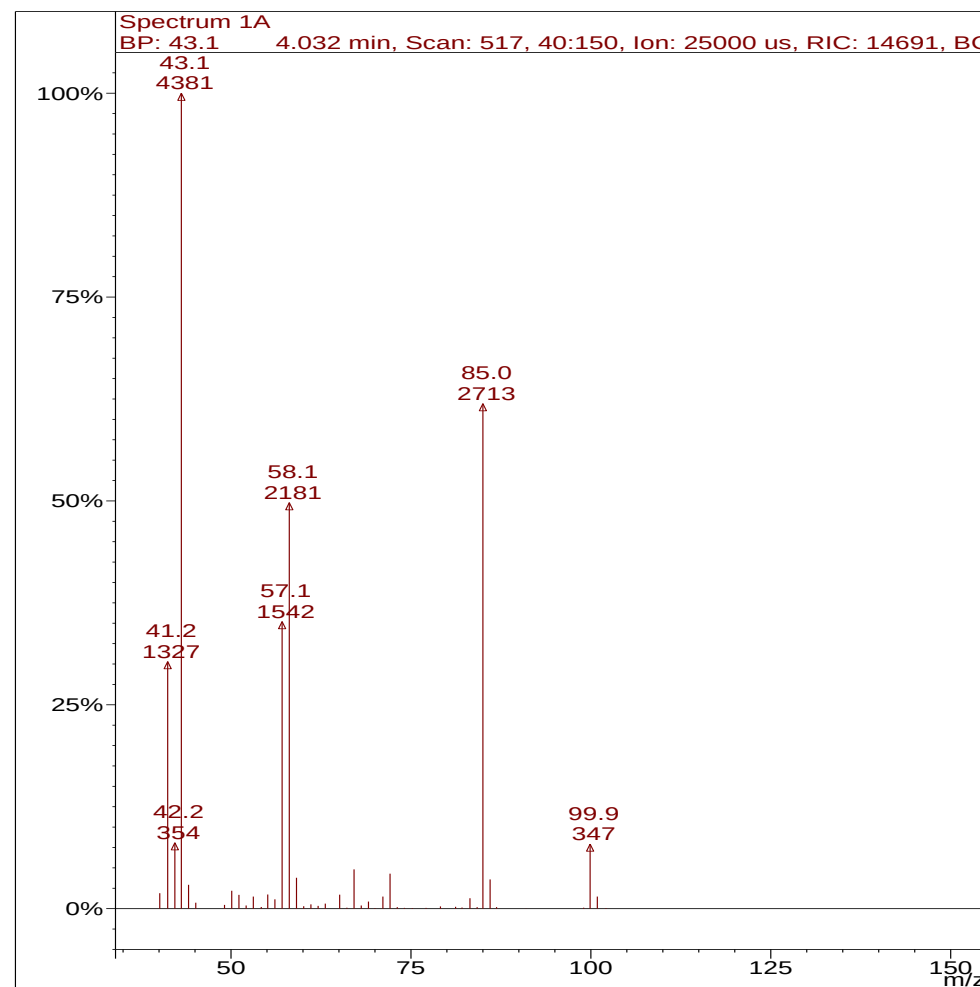


Fig. 2 :	Massa Spectrum of Calibration Standard
	MOLECULAR IONS: 43-57-58-85

METHYLISOBUTYLKETONE IN URINE

(Reference Chromatograms/Spectrums GC-MS / Headspace)

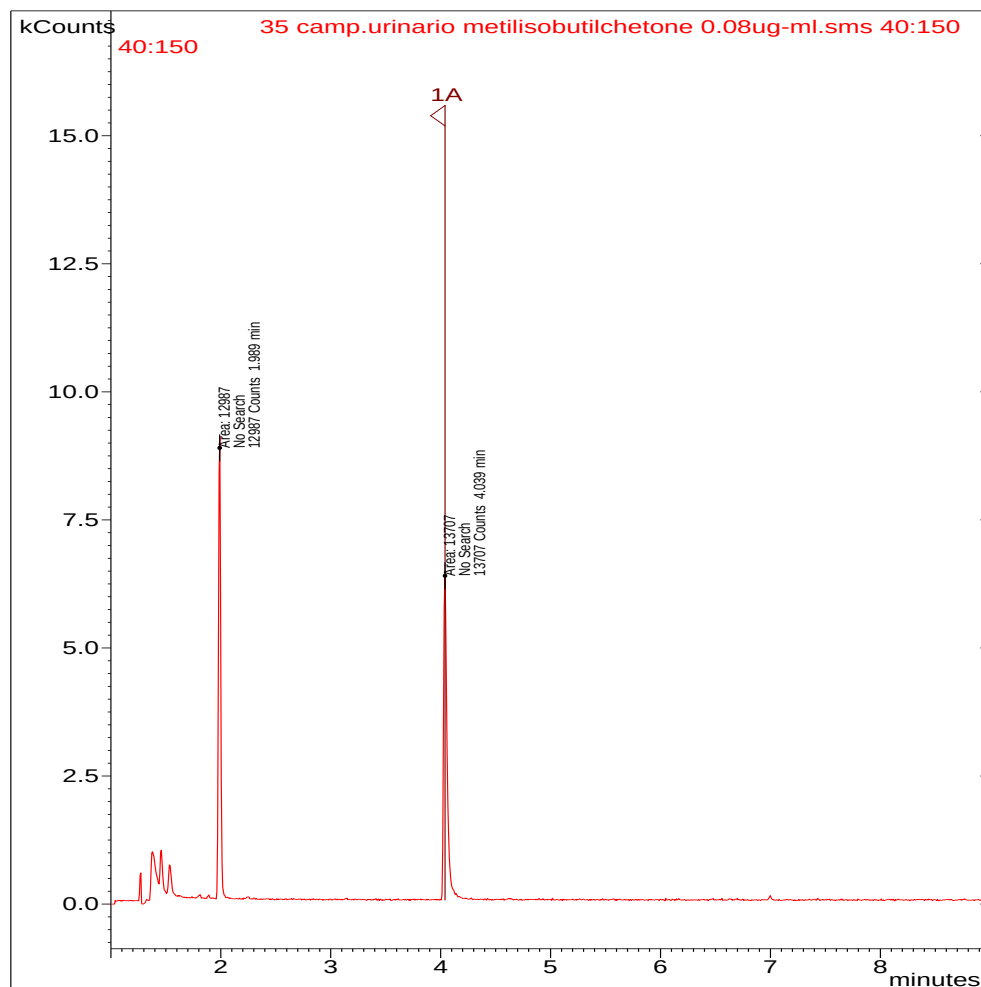


Fig. 3 :	Urine Sample
	R.T. 4.2 METHYLISOBUTYLKETONE 0,08 mg/l

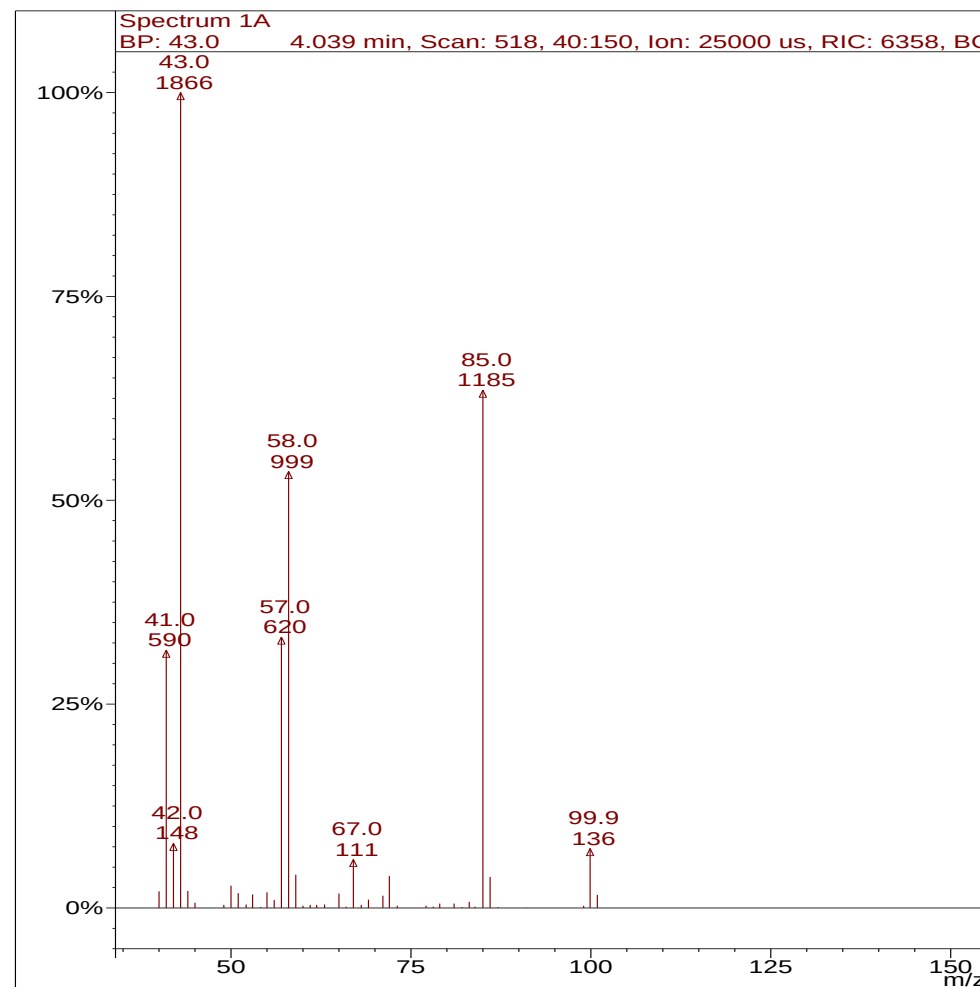


Fig. 4 :	Massa Spectrum of Urine Sample
	MOLECULAR IONS: 43-57-58-85