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DETERMINATION OF FREE FORMALDEHYDE IN BABY SOAP AND BABY WIPES BY GAS CHROMATOGRAPHY TRIPLE QUADRUPLE MASS SPECTROSCOPY (GCMS/MS)

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ABSTRACT

Formaldehyde (HCHO) is a harmful chemical. It is used in the production of fertilizer, paper, plywood, and some resins. It is also used as a food preservative and in household products, such as antiseptics, medicines, and cosmetics. It can cause irritation of the skin, eyes, nose, and throat. High levels of exposure may cause some types of cancers. By considering its carcinogenicity and toxicity it is important to determine its amount in given product. In this article, an analytical method for determination of formaldehyde content is discussed that provides accurate amount of formaldehyde presents in Baby Soap and Baby wipes. The analytical was developed on Gas Chromatography triple quadruple mass spectroscopy (GCMS/MS).

KEYWORDS:

INTRODUCTION

Formaldehyde (systematic name **Methanal**) is an organic compound with the formula CH2O and structure H−CHO. Formaldehyde is a colorless, strong-smelling, flammable chemical.

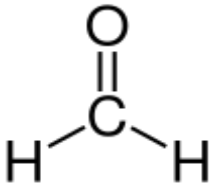

Figure: Structure of Formaldehyde.

Substances are defined as carcinogenic if after inhalation, ingestion, dermal application or injection they induce (malignant) tumours, increase their incidence or malignancy, or shorten the time of tumour occurrence.

Gas chromatography coupled to mass spectrometry (GC-MS) has also been used for the determination of formaldehyde in various samples. As formaldehyde is small molecule and have a single chromatophoric atom i.e. Oxygen (=O), so that the analysis of formaldehyde without derivatization lead many issue like response of the peak, peak shape and stability. Thus, a derivatization method was selected for the analysis. In this method derivatizatising reagent **2,4,Di-Nitrophenyl Hydrazine** was used to improve the chromatographic separation, peak shape, or response of the formaldehyde.

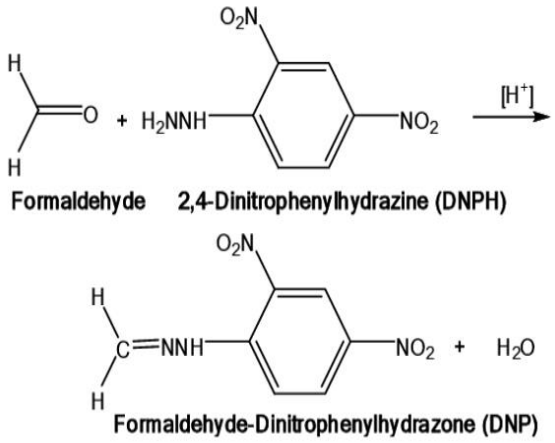

Figure: Reaction of Formaldehyde with DNPH.

Instrumentation

The HP-5 MS capillary column (Agilent Technologies, Santa Clara, CA, USA) was employed for chromatographic separation. A gas chromatograph equipped with a triple quadruple mass spectrometer (Agilent Technologies, **Model:** 7000C) was used for analysis. The balance shaker was a Dual range from Mettler Toledo.

A high speed centrifuge machine (up to 10000 RPM),Ultrasonic Bath and Oven (temperature range up to 150ºC) were used during analysis.

Chemicals

2,4,Di-Nitrophenyl Hydrazine used as derivatising reagent. Methanol used as diluent for standard and sample preparation. Hydrochloric Acid and Ethanol also were used in analysis.

Experiment

- **Instrument Parameter**

Column: HP-5 MS, 15m X 250µm X 0.25 µm (Make : Agilent).
Carrier gas: Helium
Column flow rate: 1 mL/min
Mode: Split less.
Injection volume: 2 µL.
Injection port temperature: 250 °C.

GC oven temperature gradient

Ramp	Temp	Hold
--	180	0
5	240	2

Ion Source: Electron ionization (EI+)
Source Voltage: 70 eV
Source Temperature: 300 °C
MSD Transfer line temperature: 280 °C
Stop Time: 14 min.

MRM Table

Compound Name	Precursor Ion	MS1 res	Product Ion	MS2 res	Dwell (ms)	CE (V)
C7H6N4O4	210.1	Wide	210.1	Unit	20	20
C7H6N4O4	210.1	Wide	79	Unit	20	20
C7H6N4O4	210.1	Wide	63	Unit	20	20

C7H6N4O4 : DNPH-Formaldehyde

Preparation of DNPH Solution for derivataisation:
Weight about 2 gm of 2, 4 Di-Nitro phenyl hydrazine in a 100 mL volumetric flask. Add 10 ml of 10 N HCL in it and sonicate for 5 min with shaking and dilute up to mark with methanol.

Preparation of Blank

Take about 500 mg of methanol in a 20 mL GC Head Space vial and add 5 mL of DNPH Solution. Seal the vial with septa and crimp with aluminum cap. Keep the vial at 80ºC for 45 min in a oven. After 45 min withdraw the vial from oven and allow to cool it at room temperature. Transfer the solution in 20 mL volumetric flask and rinse the vial with methanol till to complete transfer and dilute up to the mark with methanol. Label this solution as Blank Solution.

Preparation of Standard Solution:

Take about 300 mg formaldehyde standard into 100 ml volumetric flask containing about 5 ml of Methanol. Dilute and make up the volume with Methanol. Mix well.

Further dilute 5 mL of above solution in 50 mL volumetric flask with Methanol and make up to volume with same solvent. (Label this solution as STD Stock-A).

Take 1 mL of STD Stock-A in a 20 mL GC Head Space vial and 5 mL of DNPH Solution. Seal the vial with septa and crimp with aluminum cap. Keep the vial at 80ºC for 45 min in a oven. After 45 min withdraw the vial from oven and allow cooling it at room temperature. Transfer the solution in 20 mL volumetric flask and rinse the vial with methanol till to complete transfer and dilute up to the mark with methanol. Label this solution as STD Solution.

(Standard weight and dilution can be adjusted according to the their potency to get the final concentration)

Preparation of Test Solution

Take about 500 mg of Test aliquots in a 20 mL GC Head Space vial and add 5 mL of DNPH Solution. Seal the vial with septa and crimp with aluminum cap. Keep the vial at 80ºC for 45 min in a oven. After 45 min withdraw the vial from oven and allow to cool it at room temperature. Transfer the solution in 20 mL volumetric flask and rinse the vial with methanol till to complete transfer and dilute up to the mark with methanol. Label this solution as Test Solution.

(Weight of test sample and dilution can be adjusted according to the their specification limit to get the final concentration)

Procedure

Inject the 2 µL of blank solution (in duplicate), standard solution (in six replicate) and Test sample (in duplicate). Minus the avg. response of formaldehyde obtained in blank injections from area obtained in Standard and test solution.

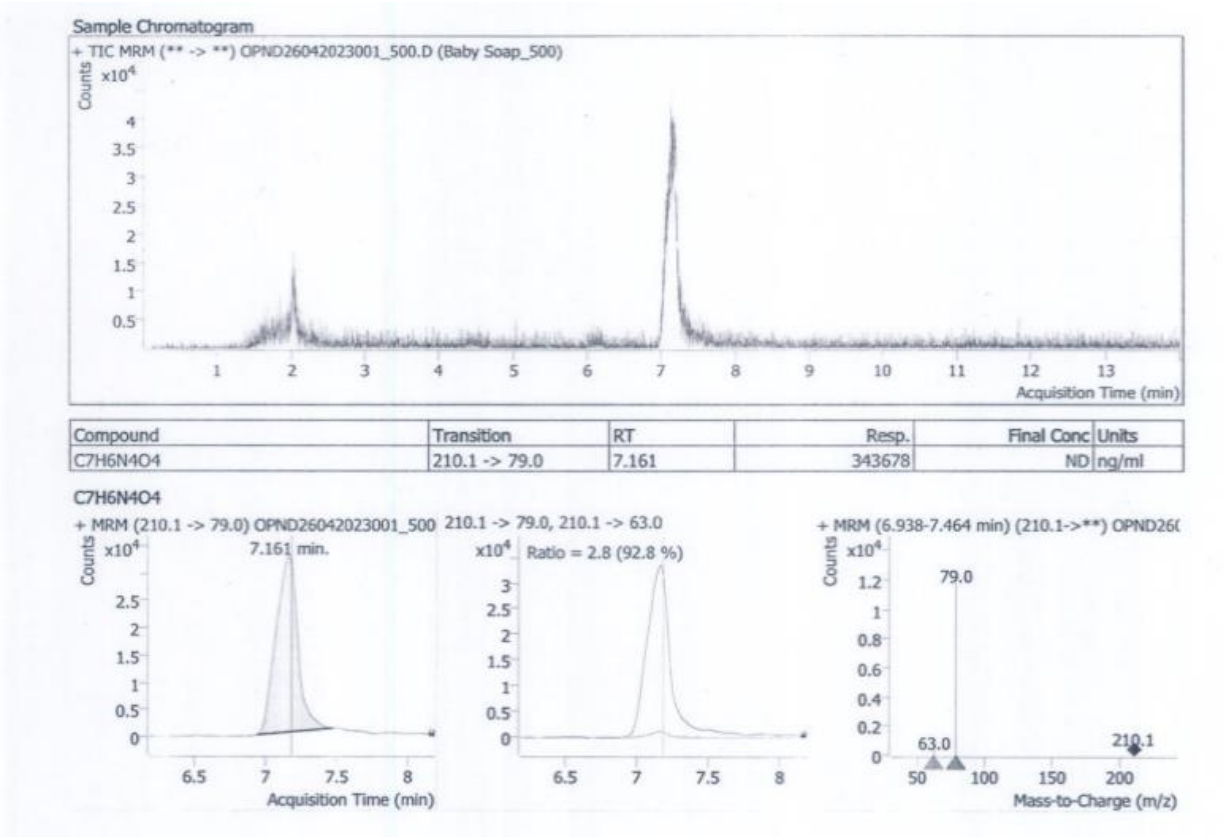

Figure: Chromatogram of Blank.

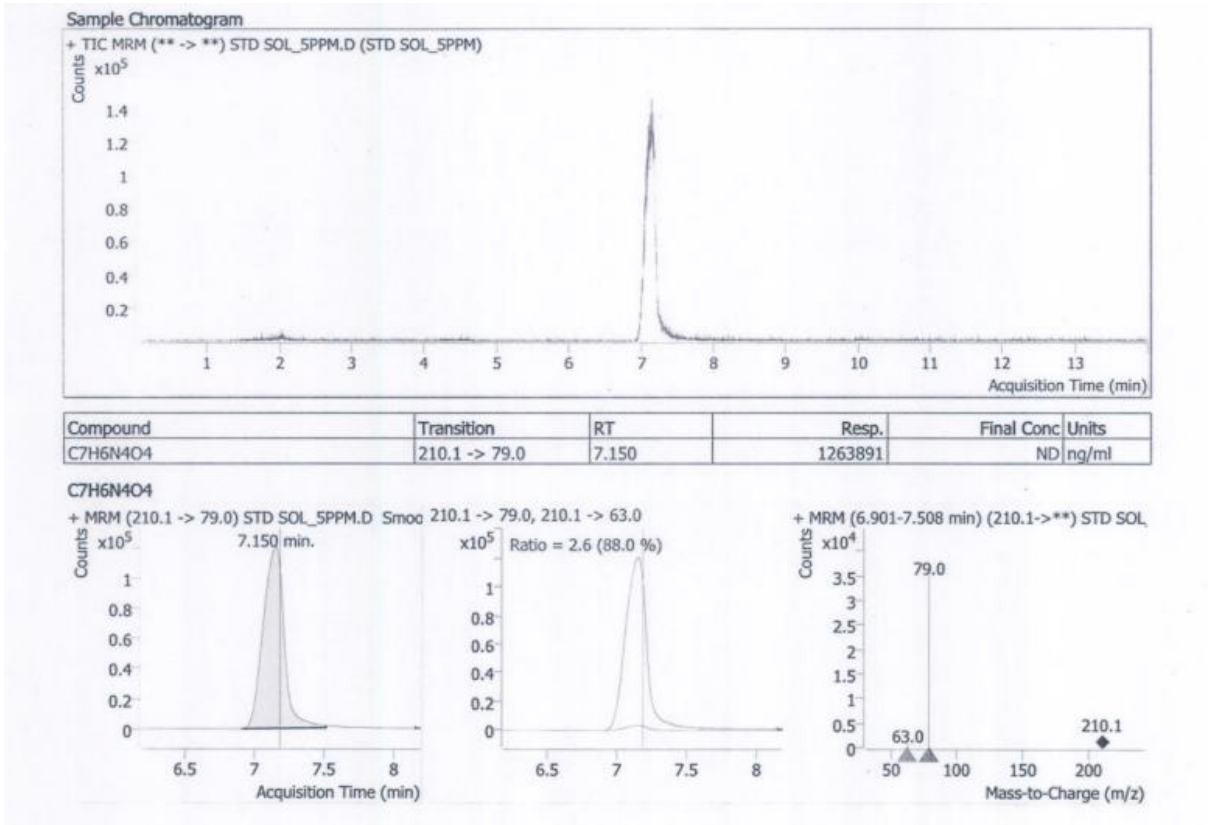

Figure: Chromatogram of Standard.

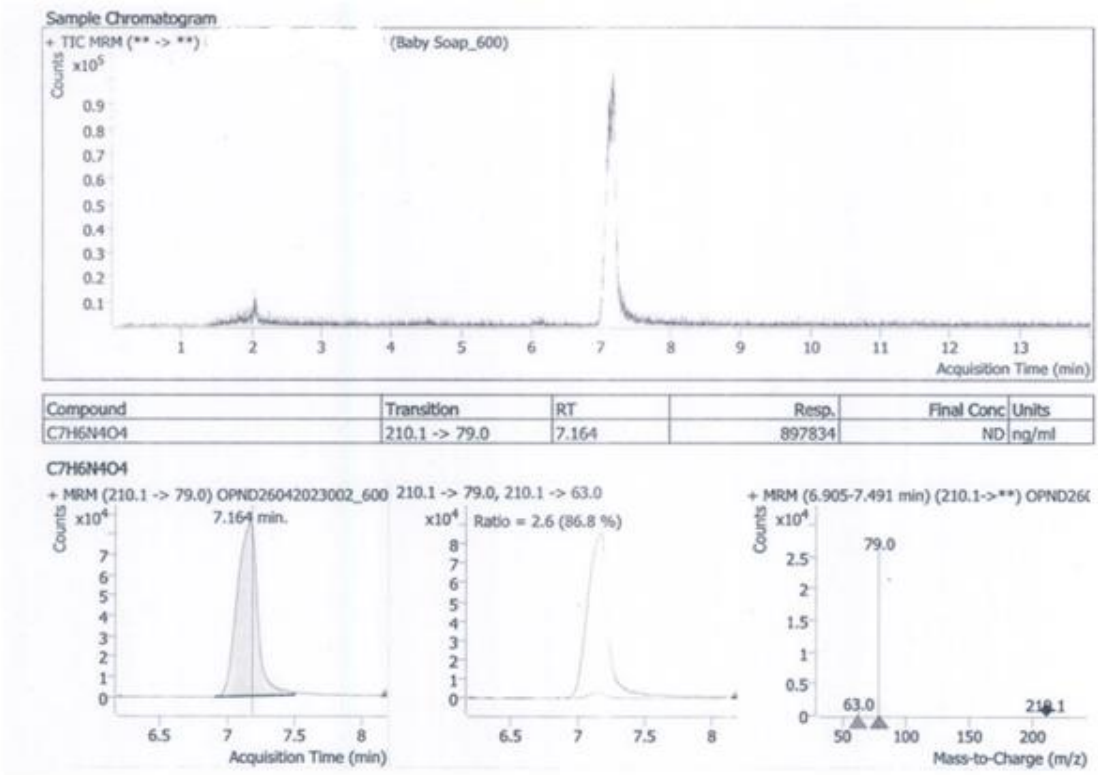

Figure: Chromatogram of Test –Baby Soap.

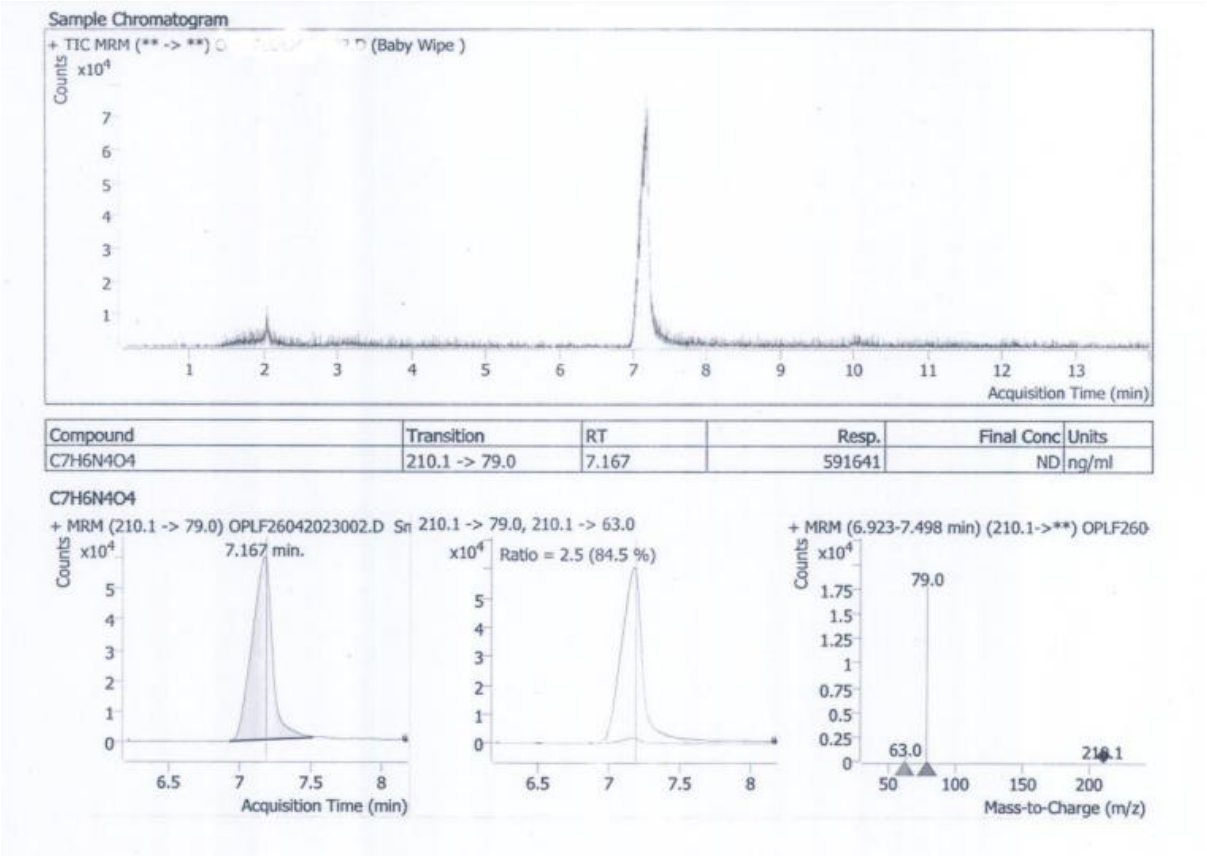

Figure: Chromatogram of Test –Baby Wipes.

Results of the content of formaldehyde (in ppm) calculated by following formula.

$$\frac{\text{Area in test}}{\text{Avg. area of Std.}} \text{ X } \frac{\text{Std. wt.}}{\text{Dilution}} \text{ X } \frac{\text{Dilution}}{\text{Test wt.}} \text{ X } \frac{\text{Std. Potency (in \%)}}{100} \text{ X } 1000000$$

RESULTS AND CONCLUSION

The % RSD of area response of formaldehyde was less than 2.0 %. That supports to method are well précised and peak of DNPH derivatized formaldehyde is stable. Also peak shape of peak was good. Results of formaldehyde in Baby Soap and Baby wipes obtained less than 100 ppm. Method found very accurate and précised during development and able to detect the content of formaldehyde even at lower level.

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