Research Article

A Novel Gas Chromatography Method for Quantitative Determination of Benzene Content in Toluene

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ABSTRACT

A simple, precise, rapid, accurate and sensitive GC method was developed for determination of Toluene purity and quantitative determination of Benzene content in Toluene. The analysis was carried out on Perkin Elmer Clarus 600 Gas chromatograph. The optimized conditions are as follows: 1) Injector temperature 200°C, 2) Detector temperature 200°C, 3) Isocratic oven temperature 100°C for 12 min, 4) injection volume 1.5µl, 5) Split ratio 1:30 and 6) Attenuation -6. Under the optimized conditions, the linear equations were obtained in the concentration range of 4.4 - 150 ppm with good correlation coefficient values. The proposed method was found to be precise with % RSD <1 (n = 6). The method showed strict linearity ($r^2 > 0.999$) between 4.4 ppm to 150 ppm. The percentage recovery of Benzene in the optimized method was between 99.0 % to 99.8 %.

Keywords: Benzene, gas chromatography, perkin elmer clarus 600 gas chromatograph, toluene

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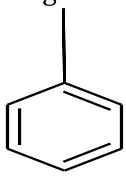
INTRODUCTION

Toluene is a mono-substituted benzene derivative. It is an aromatic hydrocarbon that is widely used as an industrial feedstock and as a solvent. Like other solvents, toluene is sometimes also used as an inhalant drug for its intoxicating properties; however, inhaling toluene has potential to cause severe neurological harm [1, 2]. Toluene is an important organic solvent, but is also capable of dissolving a number of notable inorganic chemicals such as sulfur [3] iodine, bromine, phosphorus, and other non-polar covalent substances. Inhalation of toluene in low to moderate levels can cause tiredness, confusion, weakness, drunken-type actions, memory loss, nausea, loss of appetite, and hearing and color vision loss. These symptoms usually disappear when exposure is stopped. Inhaling high levels of toluene in a short time may cause light-headedness, nausea, or sleepiness. It can also cause unconsciousness, and even death [4, 5].

Benzene is an organic chemical compound with the molecular formula C_6H_6. Its molecule is composed of 6 carbon atoms joined in a ring, with 1 hydrogen atom attached to each carbon atom. Because its molecules contain only carbon and hydrogen atoms, benzene is classed as a hydrocarbon. Benzene increases the risk of cancer and other illnesses. Benzene is a notorious cause of bone marrow failure. Substantial quantities of epidemiologic, clinical, and laboratory data link benzene to a plastic anemia, acute leukemia, and bone marrow abnormalities [6, 7]. The specific hematologic malignancies that benzene is associated with include: acute myeloid leukemia (AML), a plastic anemia, myleodysplastic syndrome (MDS), acute lymphoblastic leukemia (ALL), and chronic myeloid leukemia (CML) [8]. Human exposure to benzene is a global health problem. Benzene targets liver, kidney, lung, heart and the brain and can cause DNA strand breaks, chromosomal damage, etc. Benzene causes cancer in animals including humans. Benzene has been shown to cause cancer in both sexes of multiple species of laboratory animals exposed via various routes [9, 10].

As benzene occurs naturally in crude petroleum at levels up to 4 g/l, human activities using petroleum lead to exposure. These activities include processing of petroleum products, coking of coal, production of toluene, xylene and other aromatic compounds, and use in industrial and consumer products, as a chemical intermediate and as a component of petrol (gasoline) and heating oils. The presence of benzene in petrol and as a widely used industrial solvent can result in significant occupational exposure and widespread emissions to the environment. Automobile exhaust accounts for the largest source of benzene in the general environment. Off-gassing from building materials and structural fires lead to increased atmospheric benzene levels. Industrial discharge, landfill leachate and disposal of benzene-containing waste are also sources of exposure [11].

Toluene is used as a process solvent in the manufacturing process of APIs. Due to poisonous effect of benzene its possible presence in toluene cannot be neglected. So during our research work we established a novel and precise gas chromatography method for determination of toluene purity and quantitative determination of benzene content in toluene.


TOLUENE

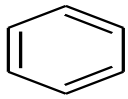

BENZENE

Figure 1: Chemical structures of Toluene and Benzene

MATERIALS AND METHODS

Instrumentation

Gas Chromatograph: Perkin Elmer Clarus 600

Analytical Micro balance: Mettler XP 26

Micro pipette: Erba, 0.5-10 µl

Reagents and chemicals

Reagents and chemicals used are mentioned in (**Table 1**).

Table 1: Reagents and Chemicals

Sr. No.	Reagents and Chemicals	B. No./Manufacturer	Purity/Assay
1	Benzene	SF2SF62382	99.9
2	Toluene	IG3IF63317	99.7
3	DMSO	R266C12	99.9

General procedure

Preparation of Solutions

Diluent:

Use Dimethylsulphoxide as a diluent

Blank Preparation

Use Dimethylsulphoxide as such

Standard solution preparation

Accurately take 2.8µl of Benzene standard sample and weigh about 125mg of Toluene standard sample into 25ml volumetric flask and dilute to mark with diluent

Test solution preparation

1) Accurately take 2.8µl of Benzene test sample into 25ml volumetric flask and dilute to mark with diluent
2) Weigh about 125mg of toluene Test into 25ml volumetric flask and add accurately 2.8µl of Benzene in it and dilute to mark with DMSO

Calculation:

$$\text{Benzene content in ppm} = \frac{At \times 100}{As}$$

Where,

At – Average peak area of Benzene in the test preparation.

As – Average peak area of Benzene content in the standard preparation.

RESULTS AND DISCUSSION

Toluene is used as a process solvent in the manufacturing process of APIs. Due to poisonous effect of benzene its possible presence in toluene cannot be neglected. So the proposed method is developed for quantitative determination of benzene content in toluene.

Analytical method validation

Method Precision

The method precision was checked after analyzing five different preparations of test sample of Toluene. The study shows that Benzene is not detected. To determine Benzene content spiking study was carried out by spiking 2.8µl of Benzene (**Fig. 2, 3 & 4**) into 125 mg of toluene test preparation (**Table 2**).

Table 2: Method Precision

For Toluene					For Benzene		
Sr.No.	Test Preparation	Inj. No	Result (%)	Mean (%)	Benzene Area	Result (PPM)	Mean (PPM)
1	1	1	99.97	99.97	Not detected	Not detected	--
		2	99.97		Not detected	Not detected	
2	2	1	99.97	99.97	Not detected	Not detected	--
		2	99.97		Not detected	Not detected	
3	3	1	99.97	99.97	Not detected	Not detected	--
		2	99.97		Not detected	Not detected	
4	4	1	99.97	99.97	Not detected	Not detected	--
		2	99.97		Not detected	Not detected	
5	5	1	99.97	99.97	Not detected	Not detected	--
		2	99.97		Not detected	Not detected	

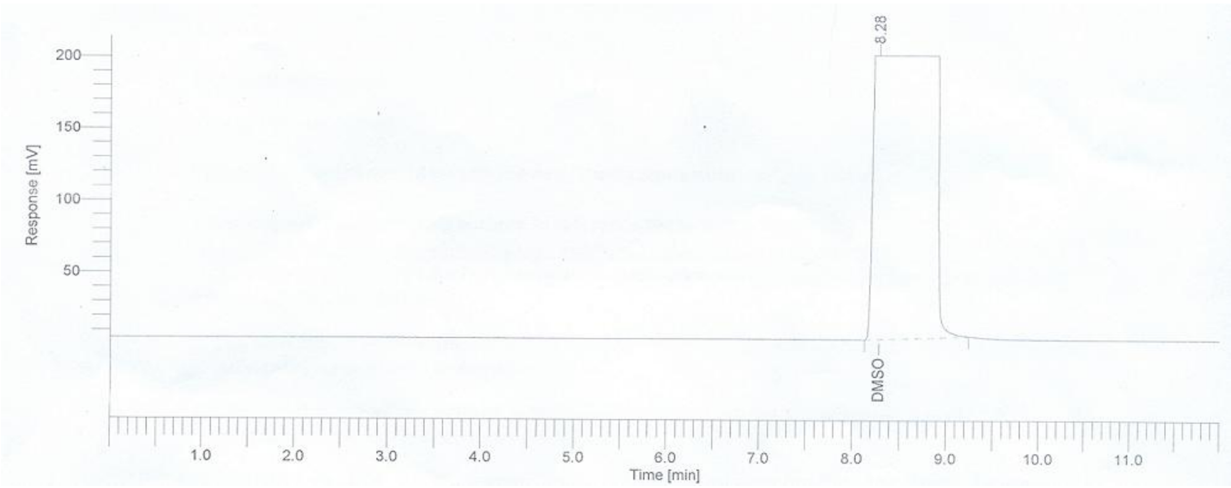


Figure 2: Blank

Linearity

For the establishment of method linearity seven different test preparations (4.4 ppm to 150 ppm) were prepared and analysed on Gas chromatograph (**Table 4**).

Table 4: Linearity

Test Preparations	Concentration (ppm)	Area
1	4.4	3152
2	25	795697
3	50	1638393
4	75	2465741
5	100	3251065
6	125	4116728
7	150	4938834

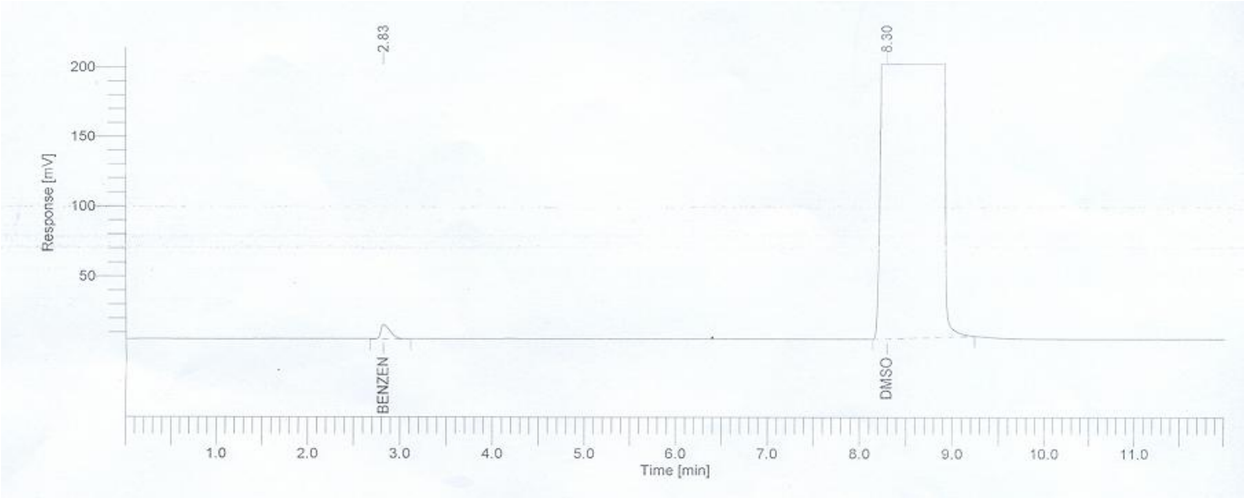


Figure 3: Benzene Standard

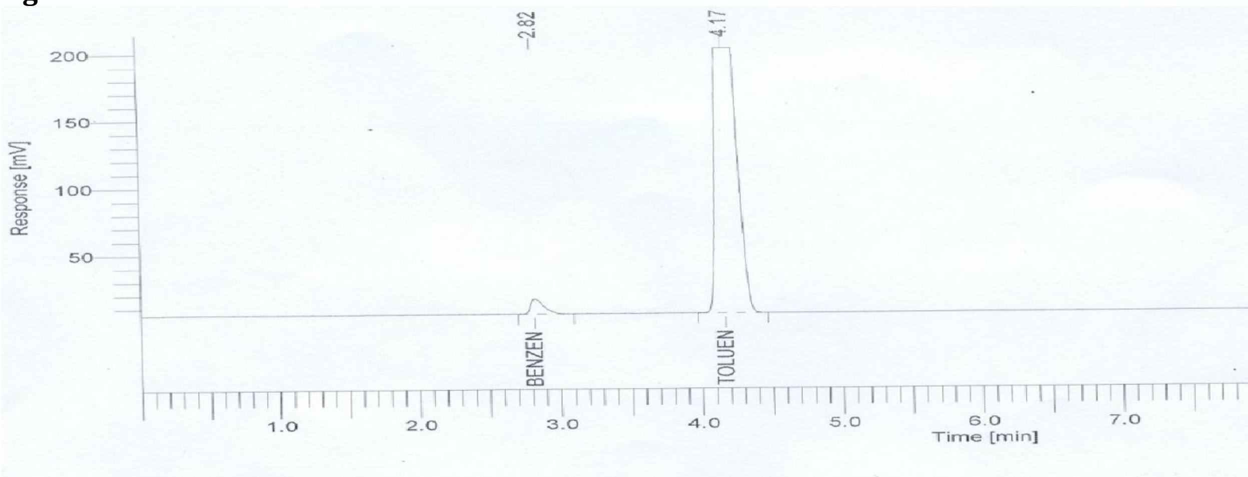


Figure 4: Standard with Benzene and Toluene

The analysis was performed at each level. Linearity curve was drawn (**Fig. 5**) by plotting sample concentration on X axis and values of Area on Y axis. And the regression values are calculated (**Table 5**).

Table 5: Regression values

Parameters	Benzene
Intercept	33562
Slope (S)	-79730
Correlation	0.9996
Co efficient Correlation R^2	0.9996

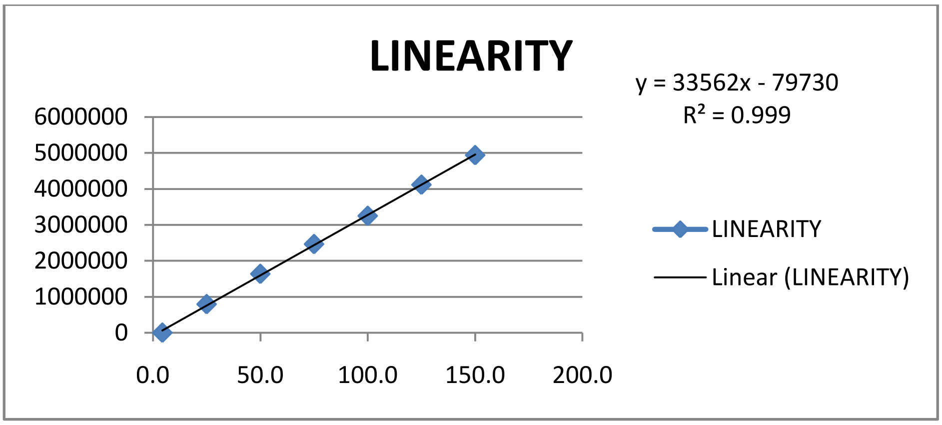


Figure 5: Linearity Curve

Accuracy and recovery

Accuracy was determined at three different levels i.e., 80 %, 100 % and 120 % of the nominal concentration. The analysis was performed in triplicate at each level the results obtained in linearity study was considered as true value during the calculation of percentage (%) recovery.

The percentage range recovery of Benzene content was found in the range of 99.0 % to 99.8 %. It confirms the accuracy of the proposed method (**Table 6**).

Table 6: Accuracy/Recovery

% Level	Benzene content (ppm) After spike	Recovery (%)
80 %	79.76	99.8
100 %	99.43	99.4
120 %	118.8	99.0

LOD-LOQ Determination

LOD-LOQ was determined at five different levels i.e., 0.5 ppm to 100 ppm of the nominal concentration. Analysis was performed at each level. The limit of detection obtained is 0.87 ppm and the limit of quantitation obtained is 2.64 ppm (**Table 7**).

Table 7: LOD-LOQ Determination

Parameters	Benzene
Standard Deviation(σ)	189.60
Slope (S)	717.72
Limit of detection (ppm)	0.87
Limit of Quantitation (ppm)	2.64

CONCLUSION

The proposed method for quantitative determination of Benzene content in Toluene was found to be precise, accurate and linear. The values of percentage recovery and standard deviation showed sensitivity. The method was validated. It showed satisfactory data for all the parameters of validation. Hence it can be applied for routine quality control application.

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