METHOD FOR DETERMINING BTX (BENZENA, TOLUENA, AND XILENE) USING GAS-FID CHROMATOGRAPHY (FLAME IONIZATION DETECTOR)

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ABSTRACT
At present, the petrochemical industry is growing rapidly. Products from the petrochemical industry are produced from petroleum or natural gas which produces various compounds such as BTX (Benzene, Toluene, and Xylene). BTX is such a poisonous aromatic compound that the International Cancer Research Agency classifies benzene as carcinogenic to humans and other BTX species have a variety of detrimental health effects even at low concentrations. Thus the separation of benzene, toluene and xylene compounds is very important. The analysis technique that is commonly used to determine the BTX compound is gas chromatography, which in this case uses GC-FID. The results shown by this instrument show that all the data obtained meet the acceptance requirements with the test parameters performed.

Keywords: Benzene, Toluene, Xylene, Gas Chromatography

INTRODUCTION
Increasingly modern technology has been in dramatic changes in industrial construction materials. Developing creativity in building design and interior decoration is increasing the use of adhesives, resins, gasoline, and paints which increasingly contain volatile organic compounds such as BTX compounds. In even small amounts, these compounds have unpleasant effects on human health, ranging from irritation to the eyes and throat to carcinogenic effects (Kheirmand et al., 2014). BTX compounds which have volatile properties are widely used as solvents in industrial processes (Panggabean et al., 2019). BTX is also often found in the petrochemical industry such as gasoline, chemical reaction solvents, diluents, and others (Rushi et al., 2014). Benzene compound itself is a chemical that is toxic and carcinogenic to health, while toluene and xylene are not carcinogenic compounds but must exist at low levels in the environment because of their toxic properties. BTX is such a poisonous aromatic compound that the International Cancer Research Agency classifies benzene as carcinogenic to humans and other BTX species have a variety of detrimental health effects even at low concentrations. These effects mainly include non-communicable diseases, such as reproduction, sperm disorders, reduced fetal development, and effects on cardiovascular disease, respiratory tract damage, asthma, and sensitization to common antigens (Benzene & Sucahyo, n.d.). Aromatic hydrocarbon compounds, such as benzene, toluene, and xylene which have carcinogenic properties are quite a challenge for the gas or oil exploration industry, so that research on the adsorption of aromatic hydrocarbons is very important so that human health and the environment are maintained (Nojavan & Yazdanpanah, 2017). The traditional way to analyze BTX is to make use of a sample preparation technique that uses a lot solvent, takes a long time is laborious and demanding manipulation stage. Therefore, it is possible to cause errors and loss of sample especially when the compound is volatile analyzed (Paula et al., 2004). Continuous monitoring of volatile organic...
compounds (VOCs) helps to correlate their concentrations in open air with a major source of pollutants (Dori et al., 2000). Please note that VOC emissions will increase with increasing ambient temperature (Faber et al., 2013). The common analysis technique used to determine this BTX compound is chromatography (Panggabean et al., 2019).

Benzene

Benzene has the chemical formula C\(_6\)H\(_6\) and forms a ring, with one hydrogen atom attached to each atom. Because they only consist of carbon and hydrogen atoms, benzene compounds can be categorized into hydrocarbons. However, when compared to other hydrocarbons which contain six carbon atoms as well, it can be assumed that benzene has a higher degree of unsaturation. Benzene does not react like alkenes (addition, oxidation, and reduction). More specifically benzene does not react with HBr, and other reagents can normally react with alkenes. Benzene can be conjugated with glutation, sulfate and glicuronic acid to produce several metabolites, such as phenol, phenylcapture acid, trans, transmuconic acid and catechols (Paula et al., 2004).

Toluene

This compound is a colorless, aromatic liquid that is characteristic but not as sharp as benzene. Toluene, also known as methylbenzene or phenylmethane, is a clear, colorless liquid that is insoluble in water with an aroma like paint thinner and smells good like benzene. At room temperature, the vapor pressure of toluene is around 29,000 ppm so it exceeds the limits set by OSHA and ACGIH. Thus, toluene users are likely to be exposed to vapors of these compounds unless appropriate guarded vigilance. In addition, toluene vapor is denser than air so it will explode easily when mixed with free air. When poured, stirred, or pumped, they produce a static charge large enough to ignite vapor-air mixture. Toluene is generally produced together with benzene, xylene, and C\(_9\) aromatic compounds by the catalytic formation of naphtha. The resulting crude formation is then extracted and mostly occurs with sulfolane or tetraethylene glycol and solutes, into a well-mixed mixture of benzene, toluene, xylene and C\(_9\) aromatic compounds where the separation method is by fractionation. Toluene is included in volatile organic compounds (VOC) so it is highly volatile at room temperature and is one of the most commonly used solvents (Prayogi et al., 2020).

Xylene

Xylene or often also called dimethylbenzene with the formula C\(_6\)H\(_4\) (CH\(_3\)) \(_2\) is one of the organic aromatic hydrocarbons. Xylene is widely used for ethyl production benzene, polyester, plastics, and paint or varnish solvents. Individuals from xylene isomers include ortho-, meta-, and para-xylene (Niaz et al., 2015). Xylene has a molecular weight of 106.17 grams / mol with a carbon (C) composition of 90.5% and hydrogen (H) 9.5%. Xylene has three isomers namely ortho-xylene, meta-xylene and para-xylene. This compound is a colorless liquid produced from petroleum or liquid asphalt and is often used as a solvent in industry. Other names for xylene include xylol and dimethylbenzene.

Gas Chromatography

Gas chromatography is a technique for separating the components in a mixture between the stationary phase (column) and the mobile phase (gas). The scope of application of gas chromatography is volatile, volatile, and non-degraded samples when exposed to heat. For samples that do not meet these requirements it is still possible to be analyzed using the gas chromatography method through certain treatments such as derivatization and the use of additional techniques (headspace method, pyrolizer, etc.). In general, gas chromatography has been used extensively in various disciplines medical, analytical, and also biology. Meanwhile, the application is like estimation of critical or essential substances, namely phenolic microbes fermented flavonoid products (in food and beverage) in urine, plasma, feces, and others (Kamal & Klein, 2010). The use of gas chromatography has several advantages, namely the relatively fast analysis time and high sensitivity. Detectors that can be used for gas chromatography instruments include HHD (Heat
Hose Detector), FID (Flame Ionization Detector), NPD (Nitrogen-Phosphere Detector), ECD (Electron Capture Detector), and MS (Mass Spectrometer) (Pulungan et al., 2018). Gas chromatograph with a universal detector such as flame ionization detector (FID), is known to be an inexpensive analysis system and can include high-throughput analysis compounds containing carbon (Jumhawan et al., 2015). BTX analysis is usually carried out by gas chromatography using a flame ionization detector or with a polyethylene glycol stationary phase, however the use of this stationary phase cannot separate m-xylene from the p-xylene isomer. (Bahrami et al., 2011).

**METHODOLOGY**

The method used in this writing is literacy study. Determination of benzene, toluene and xylene can use gas chromatography with the FID type (Flame Ionization Detector) with a capillary column (30 mx 0.32 mm ID; 1 μm film 100% PEG or equivalent), a set of computer units, analytical balance, goiter pipette, suction rubber, volumetric flask, volume pipette, syringe, vial, charcoal tube and separating funnel. Chromatography is an analytical technique which is based on the principle of the separation of the two or some components due to the different distribution between the two phases namely the stationary phase and cellular phase (Zampetti et al., 2021). While the materials used in this study are carbon disulfide (CS2), helium carrier gas, standard solutions of benzene, toluene and xylene (E’Merck) with pro analysis quality and samples taken (can be from gasoline, soil, air or other compounds. others). For the preparation of the standard 1000 mg / L BTX solution, it is done by inserting each of the benzene, toluene and xylene solutions into a 50 mL volumetric flask until the weight of the solution is ± 0.05 g, then diluted with CS2 (carbon disulfide) solution then homogenized. The 1000 mg / L BTX main solution was diluted to 10 mg / L by pipette 0.5 mL of the mother liquor put into a 50 mL volumetric flask, then added with CS2 solution then homogenized. Then the respective concentrations of the standard series solutions of benzene (0.5; 1.0; 1.5; 2.0 and 3.0 mg / L), toluene (1.0; 1.5; 2.0; 2, 5 and 3.0 mg / L) and xylene (2.0; 3.0; 4.0; 5.0 and 6.0 mg / L) were diluted from a standard solution of 10 mg / L (Panggabean et al., 2019 ). After the standard solution is formed and sampling is taken, the next process is to conduct a sample test, namely linearity, LOD, and LOQ.

**RESULT AND DISCUSSION**

Qualitative analysis of BTX compounds by gas chromatography was carried out to determine the quality of separation of BTX compounds in the stationary phase column by determining the basic quantities of chromatography such as retention time (tr), capacity factor (k') and selectivity (α).

![Figure 1](image-url)  
*Figure 1. The chromatogram profile of the separation of BTX compounds by the gas chromatography (GC) method (Panggabean et al., 2019).*

<table>
<thead>
<tr>
<th>Compound</th>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
<td>Benzene</td>
<td>Retention Time (tr)</td>
<td>3.538 ± 0.012</td>
</tr>
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<td></td>
<td>Capacity Factor (k')</td>
<td>0.23</td>
</tr>
<tr>
<td></td>
<td>Selectivity Factors (α)</td>
<td>3.76 1.77 6.65</td>
</tr>
<tr>
<td>Toluene</td>
<td>Retention Time (tr)</td>
<td>5.317 ± 0.062</td>
</tr>
<tr>
<td></td>
<td>Capacity Factor (k')</td>
<td>0.86</td>
</tr>
<tr>
<td></td>
<td>Selectivity Factors (α)</td>
<td>3.76 1.77 6.65</td>
</tr>
<tr>
<td>Xilene</td>
<td>Retention Time (tr)</td>
<td>7.256 ± 0.025</td>
</tr>
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Figure 2. The Chromatograph of BTX in Gas Chromatography (Paula et al., 2004)

The test results show that the peak of the benzene compound is close to the peak of the CS2 solvent shown from the k' value of 0.23. The capacity factor (k') represents the ratio of the distribution of the number of analytes in the stationary phase and the mobile phase. A good k' value is between 1-10. This can be seen in the k' value of toluene and xylene compounds of 0.86 and 1.52, respectively; shows that these compounds are well separated (Pulungan et al., 2018). Carbon disulfide (CS2) can be used as a good solvent for extracting the BTX which is absorbed in the solvent (Gallego-Díez et al., 2016). After using the solvent, the chromatography test was then carried out which aims to ensure the elimination of traces of benzene. We assume that the absorption rate is the same for all samples (Wideqvist et al., 2003). The samples used in this case are air and soil.

Linearity

FID is included in the general differential detector the FID signal depends on linear sample mass flow rate used. Gas chromatography analysis requires absolute measurements or the relative quantity of each component which can easily be calculated from the detector signal integral with time. This provides a relationship between the detector signal and the linear mass flow rate within the intended measurement range and is then a function of linearity called linearity (Bruderreck, 1967). The linearity test is expressed as the correlation coefficient (r).

Figure 3. Creation of a calibration curve: (a) compound B (benzene), (b) compound T (toluene) and (c) compound X (xylene) using the gas chromatography method (Pulungan et al., 2018)

The requirements that meet the criteria for the correlation coefficient are r ≥ 0.990. The r value obtained indicates that the results of linearity data are declared valid and there is a very
strong correlation between concentration and area.

**LOD and LOQ**

LOD and LOQ determination was performed statistically using the standard BTX curve obtained in the previous linearity test. The determination of LOD and LOQ can be determined by a calibration curve. LOD of benzene, toluene, and xylene compounds were 0.02; 0.59; and 0.08 mg/L. Meanwhile, the LOQ is 0.07; 1.99; and 0.27 mg/L. This value indicates the lowest concentration of analyte and the smallest quantity that can be determined by a method by being fully applied to the method used under the conditions agreed in the test laboratory. Because there is no uniform definition precise guidelines for determining LOD and LOQ, therefore, during method validation, appropriate procedures for the determination of LOD and LOQ should be clearly stated in the document method. Therefore, that the estimated LOD and LOQ values are also used for comparison by other analysts or laboratory assistants (Rahman & Rahman, 2015).

**Accuracy Test**

Accuracy states the degree of closeness of the analysis results to the actual analyte content. Accuracy is expressed as the percent recovery of the analyte added. The accuracy results are declared good if a value is obtained in the range of 95 - 105% (Panggabean et al., 2019). % recovery of each compound of benzene, toluene and xylene when using air samples is 101.69 ± 5.77%; 102.08 ± 5.43% and 98.55 ± 5.11% so that the accuracy results can be said to be good.

**CONCLUSIONS**

Based on literacy studies, the determination of benzene, toluene, and xylene can be done using gas chromatography, which in this case uses FID. The test results show that the peak of the benzene compound is close to the peak of the CS2 solvent shown from the k' value of 0.23. The capacity factor (k') represents the ratio of the distribution of the number of analytes in the stationary phase and the mobile phase. A good k' value is between 1-10. This can be seen in the k' value of toluene and xylene compounds of 0.86 and 1.52, respectively; indicates the compound is well separated. After testing in the form of linearity, LOQ and LOD tests, and accuracy tests, it shows that all the data obtained meets the acceptance requirements with the test parameters performed.

**REFERENCES**


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