#### **Technical Note**

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# An overview of the standard methods for hydrocarbon type detection of fuels

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#### Abstract

Generally, burning fossil fuels release large amounts of harmful air pollutants such as carbon dioxide and greenhouse gases into the air. Consequently, detection of feedstock's and petroleum products' specification could be essential to utilize the appropriate fuel. The detection of hydrocarbon types provides an insight to predict their performance. There are different gas chromatography based methods to identify the amount of aromatics, naphthenes, olefins and paraffins in the petroleum cuts. This investigation reviews the standard methods to compare the interfering substances, benefits and drawbacks of methods and their precision. Confirming the results of some methods need considering whole physical and chemical parameters as well as monitoring and verifying the method with higher sensitivity. Somewhat, the operative method should be selected regarding the precision of the method and considering the implications of inaccurate measurements. The identification of components contain some compromises and the best results are achievable just by conducting the optimum amounts of effective parameters, for instance the length and polarity of column or temperature programs in gas chromatography methods.

Keywords Hydrocarbon type, Gas Chromatography, Fuel, Gasoline.

#### 1. Introduction

Fossil fuels, formed from the remains of animals and plants over millions of years, are one of the most important sources of energy supply in the world. Although they are very effective in climate change and there are alternative renewable energy resources, many countries utilize fossil fuels indiscriminately [1-4].

Brudzewski et al. utilized the results of gas chromatography (GC) and Fourier Transform Infrared spectroscopy (FTIR) of 45 gasoline samples with different qualities to predict the amount of octane number by an artificial intelligence approach [5]. They reported remarkable correlation between chemical composition of the gasoline samples and predicted values of the octane number to identify gasoline quality [5]. Gasoline, which is one of the fossil fuels, is used as an energy source in cars. Gasoline contains various organic compounds, and increasing or decreasing the percentage of its constituent compounds, besides polluting the environment, causes damage to the car [6, 7].

Huber et al. utilized two hydrocarbon type analysis methods by gas chromatograph equipped with a single capillary column and a valve-switched multi-column system. They used a Hewlett-Packard 5880A Gas Chromatograph with a flame ionization detector. Although the amounts of the individual components were detected by their method but they reported overlapping of olefins with other groups and hard separation of all the paraffins and naphthenes in the range of C9 and higher carbon numbers especially when analysing naphthas made from highly naphthenic



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crude oils [8]. This separation was improved by adding a treated molecular sieve column in the multi-column valve-switched analyser. Their research had some limitations to use a second flame ionization detector (FID) and a second terminal [8].

There are some researches to determine physical properties including average molecular weight, density, carbon-to-hydrogen ratio and boiling range of petroleum associated condensate oil utilizing a GC equipped with capillary fused silica column and FID detector [9]. They considered the groups reference n-alkanes: n-C6 (C2– C9), n-C10 (C10–C13), and n-C14 (C14–C36), then detected the amount of density and molecular weight of the mixture of hydrocarbons considering the GC results and relative response factor calculated [9, 10].

By developing standards, the dangers of using fossil fuels can be avoided to some extent. In the field of developing standards related to fuel, Technical Committee 28 (Petroleum and related products, fuels and lubricants from natural or synthetic sources) of the International Organization for Standardization (ISO) develops the relevant standards. The main secretariat of this committee is in the Netherlands, and Iran is a participating member in this committee and plays an active role in drafting relevant standards. In addition, the American Society for Testing and Materials also compiles relevant standards in this field, many of which have global validity.

The specifications of gasoline were defined according to INSO 4904 and INSO 22920 [11, 12]. The detection of the accurate amount of total olefins, total aromatics and benzene is crucial to decide about the quality of this petroleum product. In international, regional and national standards, a practical limit has been defined for the mentioned parameters containing maximum total olefins of 18.0 % (V/V), maximum total aromatics of 35.0 % (V/V) and maximum benzene of 1.0 % (V/V). This review focus on the standard analytical methods to determine the hydrocarbon type regarding the amount of the precision and the potential interferences.

### 2. Measuring the hydrocarbon types by standard test methods

Many standards have been developed to measure the amount of olefins, aromatics and benzene in fuels, in this research the standard methods related to gasoline composition were studied.

#### 2.1. Olefins

The relevant standards for measuring olefins are ISO 22854, EN 15553, ASTM D6839, ASTM D1319 and ASTM D6730[13-17]. ISO 22854 and ASTM D6839 are the similar methods which cover the quantitative deter-

mination of saturates, olefins (in the range from 0,40 % (V/V) to 26,85 % (V/V)), aromatics, and oxygenates in spark-ignition engine fuels and ethanol (E85) automotive fuel by multidimensional gas chromatography[14, 16]. Whole mentioned methods report hydrocarbon types by carbon number and as a total amount. Additionally, the benzene and toluene content, oxygenated compounds and the total oxygen content can be determined. The test methods can also be practical for having similar boiling ranges of hydrocarbons, such as naphthas and reformates [14, 16]. They are also applicable to automotive motor gasoline with total aromatics of 19,32 % (V/V) up to 46,29 % (V/V) and benzene content from 0,38 % (V/V) up to 1,98 % (V/V).

ASTM D1319 and BS EN 15553 similarly specify a fluorescent indicator adsorption method for the determination of 0,3 % (V/V) to 55 % (V/V) olefins and 5 % (V/V) to 99 % (V/V)aromatics in petroleum fractions that distil below 315 °C [13, 17]. These methods can apply to concentrations outside these ranges, but the precision has not been determined. This test method is applicable to full boiling range products. Their precision does not apply to petroleum fractions with narrow boiling ranges near the 315 °C limit. Because, such samples are not eluted properly, and results are erratic[13, 17].

ASTM D6730 covers the determination of individual hydrocarbon components of spark-ignition engine fuels and their mixtures containing oxygenate blends (MTBE, ETBE, ethanol, and so forth) with boiling ranges up to 225 °C[15]. Other light liquid hydrocarbon mixtures typically encountered in petroleum refining operations, such as blending stocks (naphthas, reformates, alkylates, and so forth) may also be analyzed; however, statistical data was obtained only with blended spark-ignition engine fuels [15]. This test method is applicable to samples containing less than 25 % by mass of olefins. Table 1 shows the precision reported for determination of olefins.

As it could be observed, the amount of repeatability for ISO 22854/ ASTM D6839 are less than ASTM D1319 that could be related to the base of methods. It could be concluded that the Fluorescent Indicator Adsorption method has more error in detection by overlapping the layers in the column chromatography.

#### 2.2. Aromatics

The standard methods of ISO 22854, EN 15553, ASTM D6839, ASTM D1319, and ASTM D6730 could be similarly conducted to determine aromatic compounds [13-17]. Besides, some other methods of ASTM D5580 and D5986 are also recommended for determining aromatics [18, 19]. ASTM D5580 covers the determination of benzene, toluene, ethylbenzene, the xylenes, C9 and heavier

Standard	Repeatabili	ity (r)	Reproducibility (R)		
	For a range of data	Amount for X=15	For a range of data	Amount for X=15	
ISO 22854	r=0.0185 X + 0.1415	0.4	R=0.1176 X+0.5118	2.3	
ASTM D6839	r=0.13 $X^{0.46}$	0.5	R=0.72 X <sup>0.46</sup>	2.5	
ASTM D1319	X=4-33: r=0.26 X <sup>0.6</sup>	1.32	X=4-33: R=0.82X <sup>0.6</sup>	4.16	
ASTM D6730	Depends on the composition Depends on composition		Depends on the composition X=1.5:R=2.1	2.1	

Table 1. The repeatability and reproducibility for measuring olefins

Table 2. The repeatability and reproducibility for measuring total aromatics

Standard	Repeatability(r)		Reproduci	ribility (R)		
Stanuaru	For a range of data	Amount for X=25	For a range of data Amount for X=2			
ISO 22854	0.0095 <i>X</i> + 0.1952	0.2	0.0450 <i>X</i> + 0.1384	1.3		
ASTM D6839	0.012 (10 + <i>X</i> )	0.4	0.036(10 + <i>X</i> )	1.3		
ASTM D1319	X=13-40 r= 1.3	1.3	X=13-40 R=3.7	3.7		
ASTM 6730	Depends on the composition	Depends on the composition	Depends on the composition X=24.5: R=1.3	1.3		
ASTM D5580	$X = (16.34-49.07)$ $r = 0.0899(X^{0.5})$	0.5	$X = (16.34-49.07)$ $R = 0.2619(X^{0.5})$	1.3		
ASTM D5986	X=13-41: r= 0.55	0.55	X=13-41: R=1.65	1.65		
X is the mean of the two results in $%$ (V/V) for one sample.						

aromatics, and total aromatics (10 % to 80 %) in finished motor gasoline by gas chromatography[18]. In this method, the C8 aromatics, containing p-xylene and m-xylene co-elute while ethylbenzene and o-xylene are separated. Moreover, the C9 and heavier aromatics are determined as a single group[18].

ASTM D5986 covers the quantitative determination of oxygenates: methyl-t-butylether (MTBE), di-isopropyl

ether (DIPE), ethyl-t-butylether (ETBE), t-amylmethyl ether (TAME), methanol (MeOH), ethanol (EtOH), 2-propanol (2-PrOH), t-butanol (t-BuOH), 1-propanol (1-PrOH), 2-butanol (2-BuOH), i-butanol (i-BuOH), 1-butanol (1-BuOH); benzene, toluene and C8–C12 aromatics, and total aromatics in finished motor gasoline by GC/FTIR [19]. Although this method could cover most of aromatics and oxygenate components, the connection of

	Repeata	bility (r)	Reproduc	ucibility (R)		
Standard	For a range of data	Amount for X=1.5	For a range of data	Amount for X=1.5		
	X< 0.8: r=0.02		X< 0.8: R=0.04			
ISO 22854	$X \ge 0.8$ : r=0.0147 X	0.03	X≥0.8: R=0.0777	0.09		
	+ 0.0031		X -0.025			
ASTM	$= 0.010 V^{1.6}$	0.04	$\mathbf{D} = 0.052 \ V^{1.6}$	0.10		
D6839	1-0.019 A	0.04	K-0.055 A	0.10		
ASTM	r=0.036 V	0.05	P-0.00 V	0.13		
D6730	1-0.030 A	0.05	K=0.09 A			
ASTM	X=0.11-1.5:	0.02	X=0.11-1.5:	0.14		
D5580	r=0.0259(X <sup>0.64</sup> )	0.05	R=0.1087(X <sup>0.64</sup> )			
ASTM	X=0.1-2	0.02	X=0.1-2	0.12		
D5986	r=0.0099(X+0.6824)	0.02	R=0.054(X+0.68)	0.12		
X is the mean of the two results in % $(V/V)$ for one sample.						

Table 3. The repeatability and reproducibility for measuring benzene

GC and FTIR by a light-pipe could be complicated. Table 2 depicts the range of repeatability and reproducibility of methods for aromatic detection. The amount of repeatability and reproducibility were determined while the total aromatics amount was 25%. The amount of repeatability and reproducibility for gas chromatography methods of ASTM D6839 and ASTM D 6730 were similar, although the amount of reproducibility of ASTM

D5986 was more that could be related to the operating

conditions such as type and length of column.

#### 2.3. Benzene

Benzene as an aromatic component could be determined by ISO 22854, ASTM D6839, ASTM D5580, ASTM D5986, ASTM D6277 and EN 238 [14, 16, 18-21]. ASTM D6277 and EN 238 cover the determination of the percentage of benzene in spark-ignition engine fuels utilizing infrared spectroscopy [20, 21]. According to table 3 the amount of the repeatability and reproducibility for measuring benzene (while X=1.5%V/V) are different, therefore the selection of methods could be performed regarding the precision on demand.

Table 4 depicts the detection limit of the methods to measure the amount of total olefins, benzene, total aromatics and the boiling range of components could be separated by each one.

To detect the linearity of the mentioned chromatography methods, the peak areas of an internal standard containing components such as benzene and toluene are checked regarding the actual amount of each constituent[18]. Some error are regularly encountered due to co-elution and a deficiency in components detection. Relative error is usually determined by the ratio of the difference between the real amount of a component concentration and detected concentration to its real one. On the other hand, a linear split injection is considered as a requirement to identify constituents [15]. Optimum column efficiency and detector linearity are affected by the actual chromatographed sample size[18].

In ASTM D6730, the relative error or the splitter linearity range of (3% or less) was reported while the method was conducted according to the recommended conditions of column, temperature and flow[15].

#### 3. Interferences in gas chromatography

Common sources of error include instrumental, environmental, procedural, and human. All of these errors can be either random or systematic. Instrumental error happens when the instruments being used are inaccurate, such as a balance that does not work. The main sources of error are contamination, measurement errors and mechanical/ instrument errors such as fluctuations of inlet pressure, outlet pressure, detector temperature, column temperature and sample size. In addition, errors can come from the noise and from the measurement itself. Improving accuracy and precision of the gas chromatographic system

		components			<b>Boiling range of</b>
Standard		Total Olefins	Benzene	Total aromatics	materials
ASTM D6839/ ISO 22854	Low Detection limit % (V/V)	1.5	0.01	20	Maximum
	Linearity range % (V/V)	1.5-30	0.01-2	20-50	
ASTM D1319/	Low Detection limit % (V/V)	0.3	Not detect	5	Maximum 315°C
<b>D</b> S EI (13335	Linearity range % (V/V)	0.3 – 55	Not detect	5-99	
ASTM D6730	Detection limit % by mass	0.01	0.01	15	Maximum
	Linearity range % by mass	0.01-25	0.01-30	15-50	225 °C
ASTM D5580	Low Detection limit % (V/V)	Not applicable	0.1	10	Not reported
	Linearity range % (V/V)	Not applicable	0.1 – 5	10- 80	

Table 4. Comparison of the detection limit and the linearity range of the methods

depends on the nature of the chemicals and hardware factors such as temperature dependence of flow controllers and sample fractionation, both prior to and during injection. The precision attainable could be affected by the type of injection with Indium tube sample injection or with syringe injection [22].

The impurity of the carrier gas could be a potential contaminant which should be reduced in order to achieve reproducible results and to extend the lifetime of GC columns [23].

Purity of gases, ranging from 99.996% (4.6 grades) to 99.99999% (7.0 grades) purity, that are used as carrier gas in gas chromatography or as calibration standard are important. Checking the impurities of gas by gas chromatography had an acceptable results for the analysis of various matrix of materials [24].

Mechanism of Interferences for gas chromatography are important especially the adequate separation of the compounds prior to mass spectrometry analysis or other detectors are essential to avoid the same mass spectrum for two components[25]. The ionization efficiency of the target compound may be affected by electron impact (EI) ionization. Besides, according to researches, many of the spectral interferences can be avoided by using high MS resolutions (35 000 or better)[26]. The concentration ratio of the coeluting compounds and/or the number of quantitation ions are effective on partially overlapping mass spectral profiles [27].

Mostly, the determination of the total amount of saturates, olefins, and aromatics in petroleum fractions is significant in illustrating the quality of gasoline. Table 4 shows the benefits and drawbacks of the standard methods to

Standard	Method	Benzene	Total	Total	Interferes/ drawbacks
			Aromatics	olefins	
ASTM D1319/	Adsorption	applicable	applicable	applicable	dark-color components may interfere
ISO3837/	Columns				in recognizing the chromatographic
INSO8403					bands
ASTM D6730/	GC	applicable	applicable	applicable	- olefins above C7 may be co-eluted,
INSO22888					particularly if blending components or
					their higher boiling cuts such as those
					derived from fluid catalytic cracking
					(FCC) are analyzed, and the total
					olefin content may not be accurate.
ASTM D6839/	GC	applicable	applicable	applicable	- Method doesn't able to determine
ISO22854/					individual
INSO19324					hydrocarbon components except
					benzene.
					-There are a relative bias section for
					total olefins
ASTM D5580/	GC	applicable	applicable	Not	-Nonaromatic with a boiling point
INSO17268				applicable	greater than <i>n</i> -dodecane may cause
					interferences with the determination
					of the C9 and heavier aromatics.
					- <i>p</i> -xylene and <i>m</i> -xylene co-elute.
					-The C9 and heavier aromatics will be
					detected as a single group.
					-Not applicable for olefins
ASTM D5986	GC/FTIR	applicable	applicable	Not	-Not applicable for olefins
				applicable	

Table 5. Comparison of the methods for hydrocarbon type detection of gasoline

detect hydrocarbon types of gasoline. As it could be observed, some methods aren't applicable determining olefin amount such as ASTM D5986 and ASTM D5580 [18, 19]. On the other hands, the amount of olefins determined by ASTM D6839 and ASTM D6730 need to be assessed by calibration of the GC and certified materials [14, 15]. The column temperature program rate and the length of the packed precolumn are very important to achieve enough resolution by ASTM D6730. For instance, to achieve a suitable separation of p-xylene and 2,3-dimethylheptane, the hold temperature should be lowered from 50 °C to 48 °C (figure 1.a)[15]. On the other hand, the column

temperature program rate should be enough to distinct 1-methylnaphthalene and tridecane (figure 1.b). If the resolution is incomplete, this rate may be adjusted to run sufficient separation, for example it could be decreased from  $1.5^{\circ}$  /min to  $0.1^{\circ}$  /min[15]. At all, the change of temperature program, the length of the columns, the flow of carrier gas, the amount of injected materials are some of effective parameters to achieve suitable separation. Today, some software such as Dragon could determine this physical properties of petroleum cuts using the composition of the sample according GC results consider-

ing the properties of pure materials such as density and



Fig 1. The separation of (a) p-xylene/2,3-dimethylheptane and (b) 1-methylnaphthalene/tridecane[15]

molecular weight. The input information in this types of software (including the amount of density or molecular weight and boiling point of each component) is imperative to achieve reliable results.

#### 4. Conclusion

This document compares the methodologies for the hydrocarbon type detection utilizing standard methods which most of them are gas chromatography based. Totally, conducting the analysis of trace-level impurities in petroleum products by gas chromatography technique could be a real challenge. Most of methods give the quantitative determination of paraffins, olefins, naphthenes and aromatics in total as well as by carbon-number. Variability in measurements made on the same subject could be related to different parameters and considering associated repeatability or reproducibility could be significant for decision about the acceptable results. Therefore, the proposed amount of repeatability and reproducibility of the available methods were discussed and the interferences in the detection of components were described. Besides, the advantages and limitations of methods were discussed. Changing some parameters such as the rate of temperature program and the types and length of the columns, type of detector and split ratio are effective to attain the acceptable the relative error or the splitter linearity range. In the main, defining analytes and effective parameters are essential for developing a successful method.

#### Funding

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

#### Declarations

**Ethics approval and consent to participate** Not applicable.

#### **Consent for publication**

Not applicable.

#### **Competing interests**

The authors declare that they have no competing interests.

Received: Nov. 2023 Accepted: Dec. 2023 Published online: Dec. 2023

#### DOI: 10.22034/ASAS.2023.431362.1049

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