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BTEX Emissions in the Logistics of Middle Distillates: Diesel Oil

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Abstract: Besides technological processes; logistics; and the use of petrol and light solvents, which are widely known as pollutants, the sources of BTEX hydrocarbon contributing to air pollution may also include other petroleum products and fuels that feature higher boiling points and that have not yet been associated with this issue. In this study, the contents of benzene; toluene; ethylbenzene; and o-, m-, and p-xylene were evaluated in 25 commercial samples of diesel oils; the gaseous phase in thermodynamic equilibrium with liquid diesel oil at 40 °C was then evaluated. Based on the experimental results, it was found that benzene concentration in the gaseous phase is five to more than fifteen times higher than the limits set by regulations for benzene concentration in the air at a work place (1.6 mg/m^3) and cannot be compared with the limits set by regulations for annual average basal levels of benzene concentration in the air (5 µg/m^3). The research revealed that diesel oil is a potential source of environmental contamination from BTEX hydrocarbons, in particular benzene.

Keywords: diesel oil; BTEX emission; air pollution

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1. Introduction

In Europe, air pollution is perceived to be the second biggest environmental problem after climate change [1]. The directives related to air quality presented by the European Commission [2,3], the European Parliament [4], and national documents [5] set the expectations for effective measures that abate air pollution and its effects. The emission of VOC hydrocarbons, in particular benzene, toluene, ethylbenzene, and xylene (BTEX), to the atmosphere is an especially important issue. In the European Union, benzene emissions are monitored by the European Environment Agency (EEA) [6], while in Poland, the Chief Inspectorate of Environmental Protection is responsible for their monitoring [7].

In Poland, this issue is regulated by a few pieces of legislation, the first of which is an act [8] that specifies the general requirements. Subsequent regulations have specified a threshold limit value (TLV) of benzene concentration in the air [9], with an annual average basal level of 5 µg/m^3 and with an average level in a place of work of 1.6 mg/m^3 [10,11].

Monoaromatic hydrocarbons such as benzene, toluene, ethylbenzene, and xylenes (also known as BTEX) are the source of many adverse health effects, such as asthma; dizziness; tiredness; and irritation of the eyes, nose, and throat. Contact with these substances can also cause nausea and other non-specific health symptoms [12]. Negative health effects from BTEX may be significant even at low concentrations in the case of prolonged exposure [13]. Benzene is considered a special case. The International Agency for Research on Cancer (IARC) classified benzene as a 'carcinogen for people' (group 1) [14] based on confirmed evidence that this compound causes acute granulocytic leukaemia. Contrary to benzene, toluene was classified under group 3 (not classified due to its carcinogenic nature for people).

The most frequent case of exposure to BTEX is the inhalation of vapours, which is facilitated by the physical properties of these compounds, in particular their low boiling points and high vapour pressures under standard environmental conditions [15].

BTEX hydrocarbons, xylenes in particular, are environmental pollutants as well as ozone precursors contributing to adverse atmospheric warming [16]. In the atmosphere, BTEX is subject to degradation, mainly as a result of photolysis and/or chemical reactions with reactive structures, such as $OH^{\bullet}$ radicals ('day reactions') and $NO^{\bullet}$ nitrate radicals ('night reactions'). Other free radicals are formed in both reactions, such as organic peroxide ($ROO^{\bullet}$) and hydrogen peroxide ($HOO^{\bullet}$) radicals, which promote the formation of nitrogen oxides (NOx), which are ozone precursors that contribute to increased amount of tropospheric ozone. BTEX also plays a very important role in other chemical processes that occur in the atmosphere. They are considered precursors of other strongly oxidising substances, such as peroxyacetyl nitrate (PAN); they also contribute to the formation of secondary organic aerosols (SOA) [17,18].

A few main sources may be assigned to BTEX emissions into the air. The first type of source is stationary, such as fuel stations, petrol stations, vehicle service workshops, industrial plants, and refineries; the second source is strictly related to transport and traffic [19]. The combustion processes of raw materials such as petroleum and gas as well as forest fires may be defined as a separate group [20].

Benzene exists naturally in fossil fuels and is produced as a result of natural processes or human activity related to the combustion of organic matter, such as wood, coal, and petroleum products. The natural sources of benzene emissions are estimated to be 3–5%, while anthropogenic sources account for more than 90% (petrol vapours, exhaust gas, paints, and chemical production) [21].

Toluene exists naturally as a component of petroleum and is the main aromatic component of petrol, which contains approx. 5–7 wt.% of toluene. It is released during the production of petrol and other fuels from petroleum, during the production of coke from coal, and as a by-product of styrene production. It is used as a semi-finished product in the petrochemical industry [21].

Ethylbenzene is naturally present in petroleum. It is also a by-product of biomass combustion. Ethylbenzene is almost exclusively (>99%) used as a semi-finished product in the production of styrene monomer. Ethylbenzene emissions are related to the logistics of fuels and solvents, and to petrol combustion [21].

Xylenes exist in the environment as a mixture of *orto*, *meta*, and *para* isomers. Xylenes exist naturally in petroleum and coal tar. Xylenes are released to the atmosphere primarily as emissions from industrial sources (e.g., refineries, or petrochemical and chemical plants) and emissions from internal combustion engines in cars [21].

Besides technological processes; logistics; and the use of petrol and light solvents, which are widely known as pollutants, the sources of VOC pollution may also include other petroleum products and fuels that feature higher boiling points and that have not yet been associated with this issue. It is necessary to recognize the scale of the problem to start effectively monitoring this problem and to develop prevention methods.

Hydrocarbon fractions with a declared initial boiling point above 150 °C contain small amounts of lighter fractions. This causes the gaseous phase remaining in equilibrium with the liquid phase (e.g., in tanks) to be enriched with these components. This results from Raoult's law.

Because of the range of the boiling point, no study has yet considered the potential threat of BTEX hydrocarbons from diesel oil, for which the typical range of boiling points is 160–360 °C, in detail.

2. Materials and Methods

Table 1 presents the selected properties of the 25 samples of commercial diesel oils used in the experiments.

Table 1. Properties of the studied diesel oils (measured by INiG-PIB).

Determined Parameter	**Unit**	**Diesel Oil**																								
		1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**23**	**24**	**25**
Cetane number	-	51.5	53.1	51.7	52.3	52.0	52.3	52.0	52.4	52.9	54.1	52.5	52.5	51.7	52.0	51.8	52.1	51.7	52.4	51.9	51.8	51.5	51.8	52	52.4	52.3
Density at 150 °C	kg/m^3	835.7	832.6	829.0	829.9	837.5	833.9	829.4	830.7	835.6	832.8	831.4	832.6	835.8	835.7	835.5	828.2	834.2	836.0	835.9	835.0	836.4	835.8	835.9	831.3	832.9
Ignition temperature	°C	62.0	64.0	64.0	58.0	64.5	61.5	60.5	61.5	62.5	59.5	60.5	62.5	61.5	63.5	59.5	60.5	64.5	64.5	62.5	63.5	62.5	62.5	62.5	59.5	61.5
FAME content	%(V/V)	5.9	6.9	6.8	6.7	6.4	6.4	6.3	6.4	6.5	<0.05	6.7	6.6	6.7	6.7	6.6	2.63	3.9	6.4	6.6	6.7	6.6	6.7	6.7	6.4	6.2
Fractional composition:																										
up to 250 °C distils	%(V/V)	37.6	37.0	41.2	37.1	37.3	37.7	39.5	38.8	36.2	39.8	36.1	37.8	39	38.7	39.1	40.3	37.6	36.1	38.5	38.4	37.7	38.5	38.5	38.3	39.2
up to 350 °C distils	%(V/V)	94.4	93.5	94.2	91.4	94.2	92.6	91.9	92.4	93.6	93.5	91.4	92.8	93.4	93.3	93.6	91.6	93.7	93.9	93.3	93.6	94.9	93.4	93.4	92.2	93.2
95 % (V/V) distils up to	°C	351.9	354.5	353.8	359.8	352.8	359.3	360.7	358.9	354.2	355.7	363.3	359.4	355.6	356.7	354.9	363	355.4	353.9	356.2	354.7	357	356.4	356.3	359.4	365.3

The analysis was carried out on a Clarus 500 gas chromatograph with a mass detector (GCMS) (PerkinElmer, Waltham, MA, USA) equipped with a Phenomenex polar capillary column Zebron ZB-50 0.25 µm that was 30 m long with a 0.25 mm inner diameter, with a 50% phenyl/50% dimethylpolysiloxane stationary phase and a quadruple mass detector.

The tests were carried out in accordance with the following procedure.

Approximately 4 cm^3 of the tested diesel oil was introduced to a 9 cm^3 test tube and tightly closed with a plastic septum. The test tube was conditioned at 40 °C for 2 h; a gas syringe was then used to collect 200 µL of the sample in the gaseous phase, which was analysed on the Clarus 500 GCMS chromatograph. The temperature programme of the separation maintained an initial temperature of 40 °C for 10 min before the column was heated to 220 °C at a rate of 15 °C/min; the final temperature was maintained for another 10 min. Helium 5.0 was used as the carrier gas. The mass detector worked in the selected ion recording (SIR) mode, recording selected molecular weights with the summary signal originating from three ions—78 Da (benzene), 92 Da (toluene, xylenes, and ethylbenzene), and 106 Da (xylenes and ethylbenzene)—or in the total ion chromatogram (TIC) mode, recording all weights from a specified range. The ion weights recorded in the experiment ranged from 5 to 450 Da.

3. Results

The following plan for the experiments was selected based on a literature review.

The contents of benzene and toluene, and the total amounts of ethylbenzene and the o-, m- and p-xylenes were evaluated in 25 samples of various commercial diesel oils. The gaseous phases of those samples were then analysed in thermodynamic equilibrium with the liquid phase at 40 °C. The potential threat of BTEX emission during diesel oil use and during logistics was assessed with an analysis of the results.

Examples of the chromatograms of the gaseous phase sample collected from diesel oil no. 16 are presented below; they were recorded in TIC mode (Figure 1) and SIR mode (Figure 2). The retention times for the compounds in question (Figure 2) were 4.22 min, 8.33 min, 12.89 min, 13.04 min, and 13.82 min for benzene, toluene, ethylbenzene, m-xylene, p-xylene, and o-xylene, respectively. For the purpose of this study, due to partial overlap in the peaks of ethylbenzene and the m- and p-xylenes, the total contents of ethylbenzene and all xylenes were evaluated. The peaks originating from benzene and toluene are not visible in the chromatogram recorded in TIC mode (Figure 1). In the case of benzene, it was obscured by the peaks of isomeric octanes with retention times of 4.15 min and 4.33 min. In both cases, in the summary spectrum of masses for these chromatographic peaks (Figures 3 and 4)—in addition to signals of fragmentation ions characteristic of octanes and the signal of molecular octane ion (114 Da)—weak signals were observed to originate from the ion of molecular benzene (78 Da).

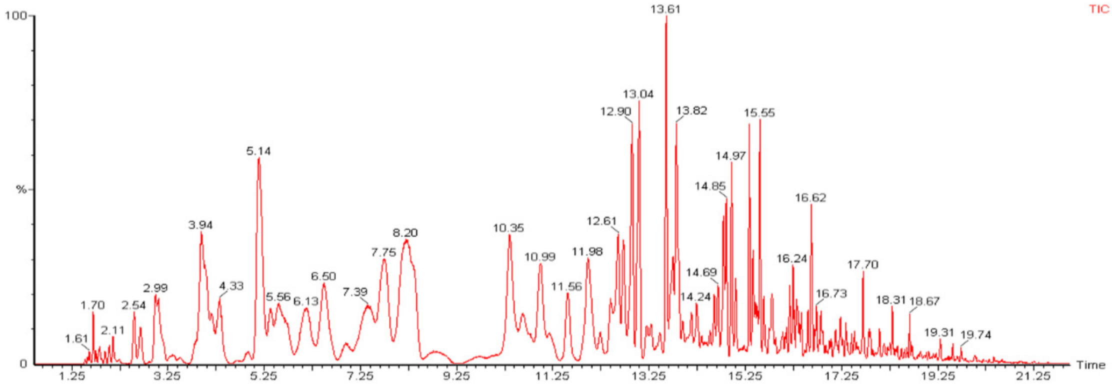

Figure 1. Chromatogram of a sample of diesel oil no. 16, recorded in TIC mode.

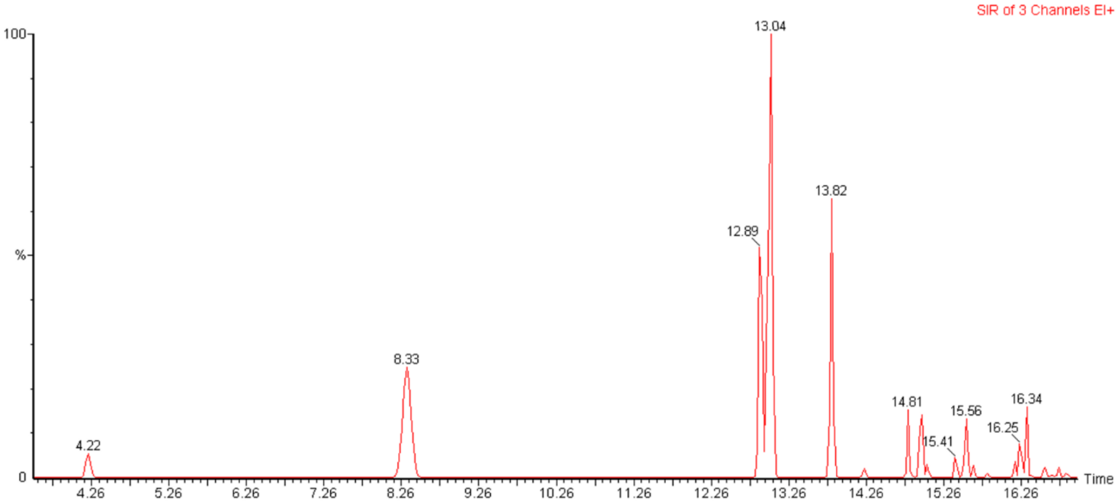

Figure 2. Chromatogram of a sample of diesel oil no. 16, recorded in SIR mode, for three masses: 78 Da, 92 Da, and 106 Da.

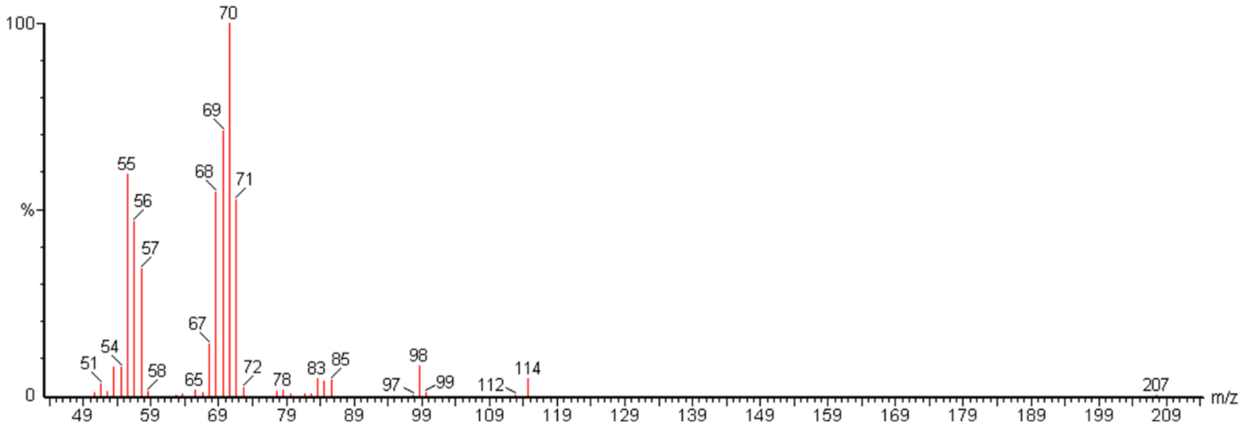

Figure 3. MS spectrum for the peak with a retention time of 4.15 min.

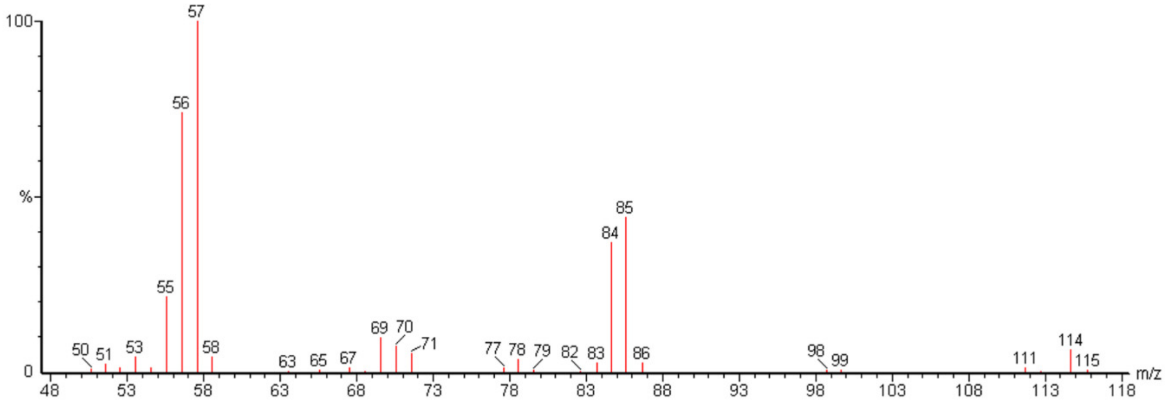

Figure 4. MS spectrum for the peak with a retention time of 4.33 min.

In the case of toluene, the chromatographic peak originating from it was obscured by a broad peak originating from one or more nonane isomers (retention time of 8.20 min). In the summary mass spectrum (Figure 5) for this chromatographic peak—apart from the signals of fragmentation ions characteristic of nonane and the signal of a molecular ion (128 Da)—a weak signal was observed to originate from a molecular ion of toluene (92 Da).

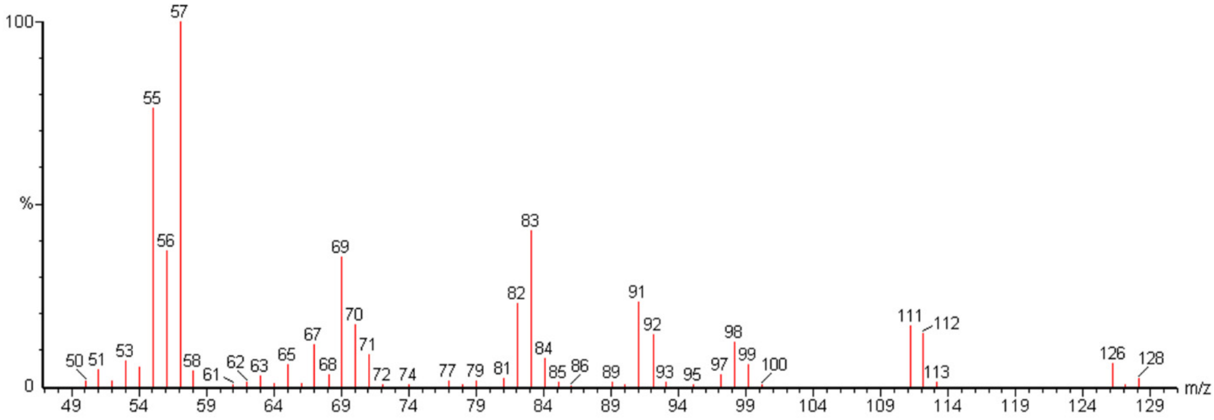

Figure 5. MS spectrum for the peak with a retention time of 8.20 min.

On the chromatogram recorded in TIC mode, for retention times ranging from 12 to 14 min, four peaks were clearly visible, of which three corresponded to the retention times of ethylbenzene, a mixture of m- and p-xylene, and o-xylene. The summary mass spectra (Figures 6–8) confirm the good separation of those compounds from other components in the hydrocarbon matrix. In addition, for retention times between 12 and 14 min, a clear peak of another hydrocarbon was observed for a retention time of 13.61 min. The MS spectrum (Figure 9) shows that it originated from a branched decane.

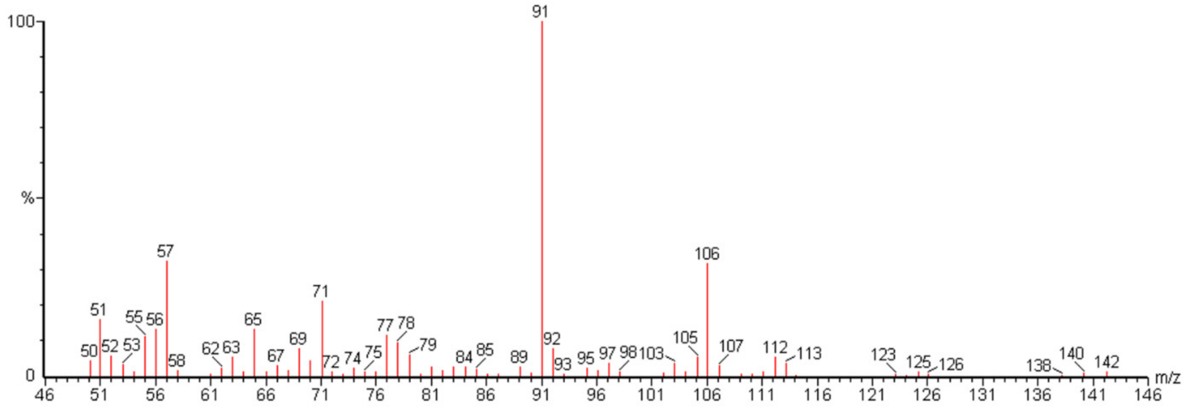

Figure 6. MS spectrum for the peak with a retention time of 12.89 min.

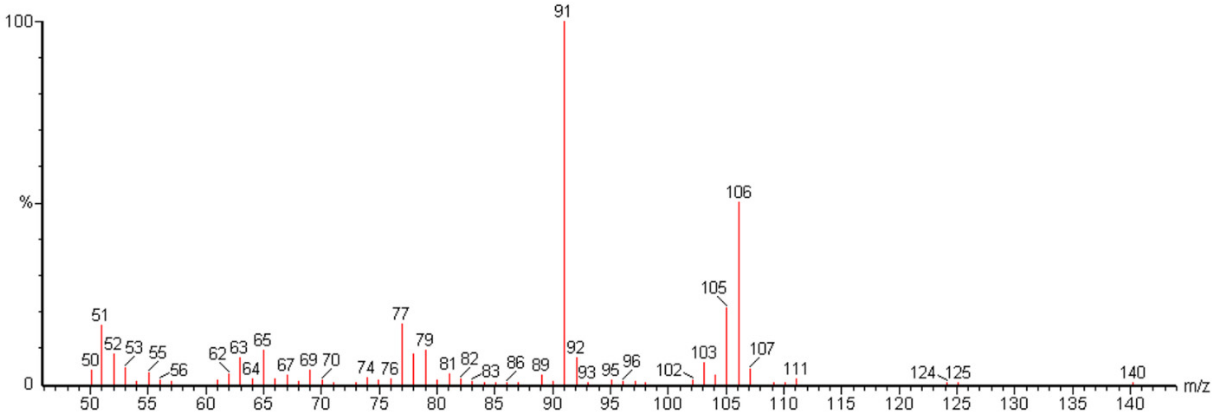

Figure 7. MS spectrum for the peak with a retention time of 13.04 min.

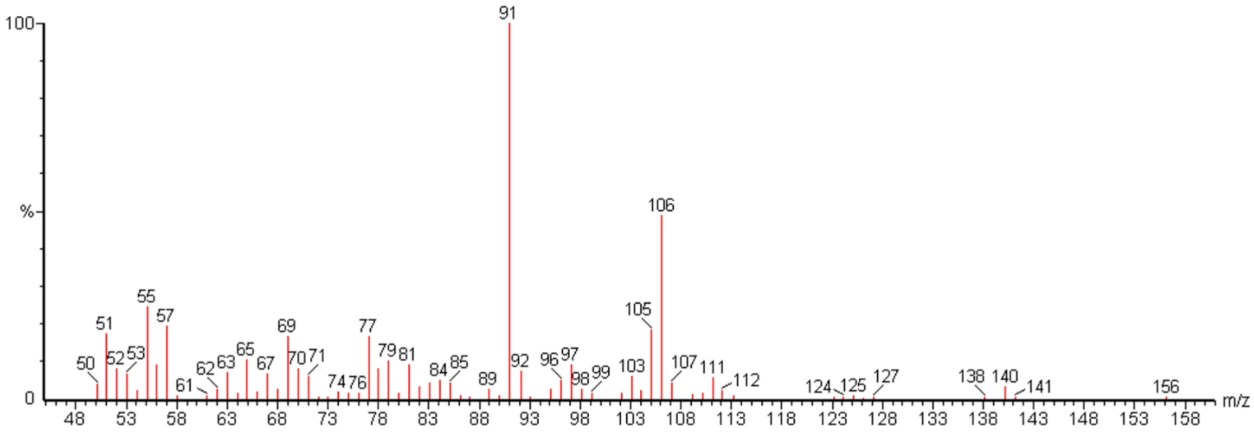

Figure 8. MS spectrum for the peak with a retention time of 13.82 min.

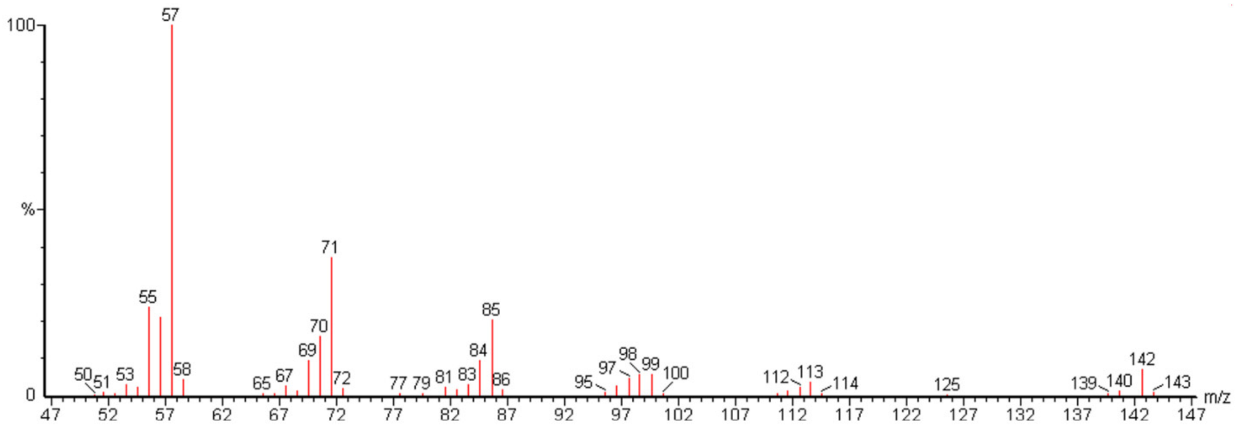

Figure 9. MS spectrum for the peak with a retention time of 13.61 min.

Table 2 presents the measurements of BTEX concentrations in the liquid and gaseous phases of diesel oil in equilibrium at a temperature of 40 °C. In the case of benzene, the concentration present in the liquid phase of the samples ranged from approx. 8 to approx. 27 mg/dm^3, while that in the gaseous phase ranged from approx. 7 to approx. 25 mg/m^3.

In the case of toluene, its concentration in the liquid phase of the samples ranged from approx. 140 to more than 600 mg/dm^3, while that in the gaseous phase ranged from approx. 90 to approx. 230 mg/m^3.

The total xylene and ethylbenzene content of the liquid phase in the samples ranged from approx. 1600 to approx. 3500 mg/dm^3, while that in the gaseous phase ranged from approx. 220 to more than 500 mg/m^3.

Raoult's and Dalton's laws control the composition of a gaseous phase in equilibrium with mixtures of liquids that do not react with each other. For a perfect solution, the partial pressure of a single component is described by the following formula:

$$p_i = x_i * p_i^*$$

where p_i is the partial pressure of component i in the gaseous phase in equilibrium with the liquid phase, x_i is the mole fraction of component i in the liquid phase (solution), and p_i^* is the equilibrium pressure of a pure component's vapours at a given temperature.

When the components of the solution reach thermodynamic equilibrium, the total vapour pressure above the solution p may be described by the following formula:

$$p = \sum_i x_i * p_i^*$$

Table 2. BTEX concentrations in liquid and gaseous phases of diesel oil in equilibrium at a temperature of 40 °C.

Diesel Oil	Benzene		Toluene		Xylenes + Ethylbenzene	
	Liquid Phase	Gaseous Phase	Liquid Phase	Gaseous Phase	Liquid Phase	Gaseous Phase
	mg/dm^3	mg/m^3	mg/dm^3	mg/m^3	mg/dm^3	mg/m^3
1	18.0	11.9	310	108	1906	227
2	14.2	19.5	242	157	1819	319
3	18.1	24.5	386	203	2474	402
4	11.7	15.4	154	148	2378	521
5	25.7	23.5	448	163	2474	291
6	14.4	24.6	632	201	3213	225
7	14.5	9.3	157	87	2697	352
8	11.4	12.4	151	116	2337	322
9	18.0	23.6	308	177	1643	321
10	22.3	23.6	346	203	2440	364
11	8.2	6.7	141	116	2469	460
12	16.8	15.4	271	155	2500	391
13	26.5	15.4	459	131	2439	243
14	14.3	20.6	302	234	1817	404
15	18.1	22.8	423	211	2185	357
16	11.6	8.2	221	132	3513	514
17	16.7	16.5	211	155	3513	313
18	15.4	14.8	254	155	1748	300
19	23.6	21.2	567	209	3073	287
20	27.8	17.9	634	192	3505	340
21	20.5	18.9	393	189	2622	344
22	20.5	17.7	421	188	2139	321
23	22.3	19.8	452	212	2688	389
24	11.9	8.1	237	107	2848	317
25	15.7	13.4	245	151	2307	360

Table 3 presents the equilibrium pressures of pure BTEX vapours p_i^* for the temperature at which the experiment was carried out, and for 10 °C and 25 °C, which are typical of the Central European climate.

Table 3. Equilibrium vapour pressures of pure BTEX components at given temperatures.

	Pressure of Pure Hydrocarbon Vapours Being in Equilibrium with Liquid Phase *					
Temp.	Benzene	Toluene	O-Xylene	M-Xylene	P-Xylene	Ethylbenzene
(°C)	(kPa)	(kPa)	(kPa)	(kPa)	(kPa)	(kPa)
10	6.807	1.678	0.344	0.451	0.472	0.502
25	12.196	3.827	0.883	1.118	1.186	1.269
40	24.826	7.935	2.051	2.529	2.679	2.875

* calculated from the Antoine equation ($\log_{10}(P) = A - (B/(T + C))$) based on data from the National Institute of Standards and Technology (NIST) [22–26].

Diesel oil is a complex mixture of a large number of hydrocarbons at various concentrations, and therefore, the above description cannot be easily translated into an assessment of correlations between BTEX concentrations in the diesel oils under study and their concentrations in the accompanying gaseous phase.

Figures 10–12 depict the correlations between the composition of a gaseous phase in equilibrium with different mixtures of liquids.

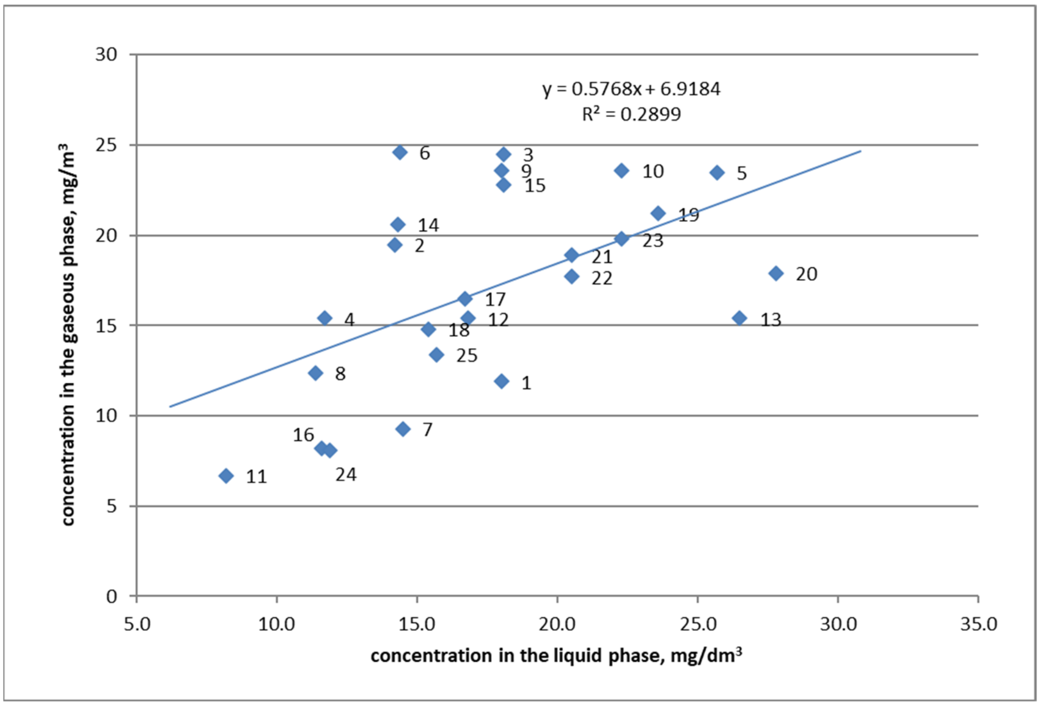

Figure 10. Concentration of benzene in the liquid and gaseous phases of diesel oil in equilibrium.

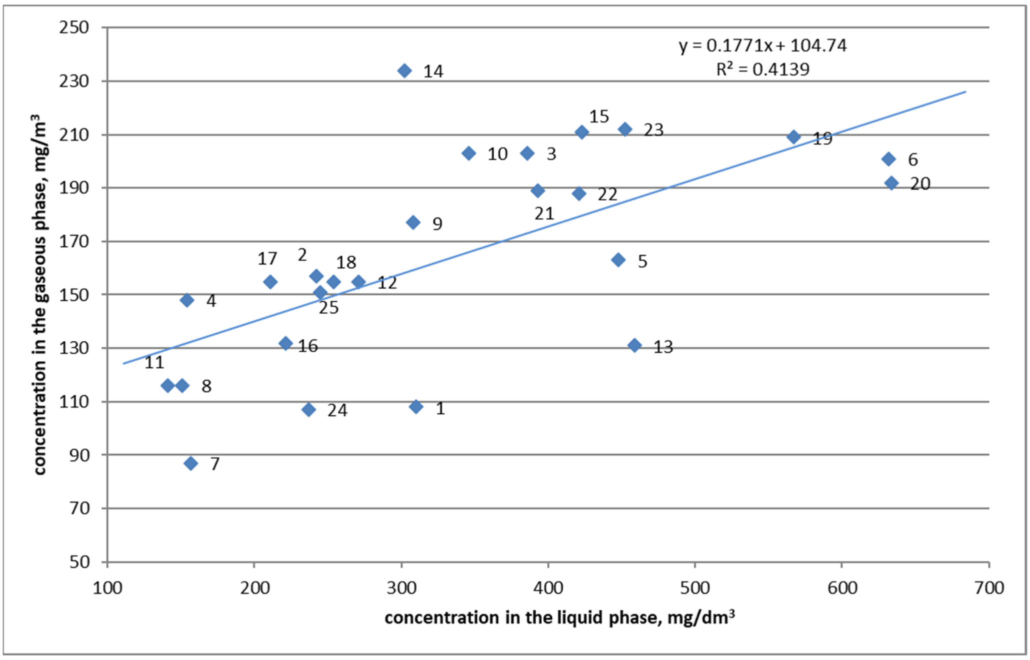

Figure 11. Concentration of toluene in the liquid and gaseous phases of diesel oil in equilibrium.

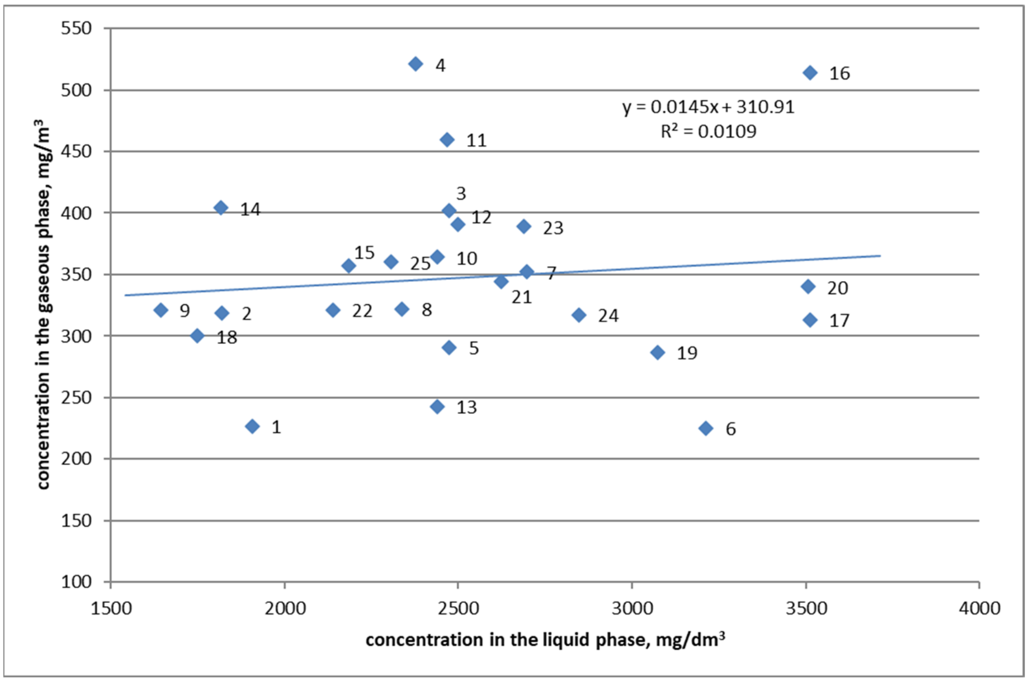

Figure 12. Total concentration of xylenes and ethylbenzene in the liquid and gaseous phases of diesel oil in equilibrium.

For benzene and toluene, a weak relationship was observed (Figures 10 and 11) between their concentrations in the liquid and gaseous phases: an increase in the hydrocarbon concentration in the liquid phase results in an increased concentration in the gaseous phase. In the case of benzene, the slope (coefficient *a*) is about 0.6, and in the case of toluene, the slope is about 0.2. The coefficients of determination R^2 are 0.3 and 0.4 accordingly. However, for the total xylene and ethylbenzene, practically no systematic relationship was observed (Figure 12); the slope is lower than 0.015; and R^2 is about 0.0, which was most likely related to the presence of varying amounts of other hydrocarbon components with similar equilibrium pressures in the studied diesel oils.

4. Discussion

In these experiments, the benzene concentration in the gaseous phase in equilibrium with diesel oil was found to be from five to more than fifteen times higher than the TLV of benzene concentration in the air for a place of work, which is 1.6 mg/m^3, and cannot be compared with the TLV of benzene concentration in the air on an annual average basis, which is 5 µg/m^3.

This research revealed that diesel oil is a potential source of environmental contamination with BTEX hydrocarbons, in particular with benzene. In the literature, the combustion processes of middle distillate fuels as a source of BTEX emissions into the air have been described as a concern. No work on such emissions in logistics and the storage of middle distillate fuels was found. The above observations should draw the attention of the authorities involved in environmental protection and in occupational health and safety to the necessity of considering the issue of VOC emission (including BTEX) accompanying the logistics of middle-distillate fuels, including diesel oil.

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Conflicts of Interest: The authors declare no conflict of interest.

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