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Short Communication: Sulphur Levels and Fuel Quality in Peninsular Malaysia

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This paper presents the investigation on sulphur levels in fuels collected from different petrol stations in Peninsular Malaysia. Diesel and gasoline samples were analysed for sulphur using X-ray fluorescence spectrometry technique. In general, diesel concentrations of sulphur were only slightly lower than the Deprtment of Environment's current sulphur limit of 500 p.p.m. across 2010–2014 (the range was 289–698 p.p.m., 374–410 p.p.m., 319–436 p.p.m., 447–605 p.p.m. and 300–477 p.p.m. for 2010, 2011, 2012, 2013 and 2014, respectively). Sulphur concentrations were relatively low in gasoline samples: the mean figures were 95, 77, 96, 74, 240 and 125 for 2009, 2010, 2011, 2012, 2013 and 2014, respectively when compared with the sulphur limit of 500 p.p.m.. The importance of determining sulphur concentration in fuel oils was critical in the efforts to determine if the vehicles used quality fuels that comply with *Euro 2* Standards; and also was relevant in meeting *Euro 4* Standards requirement of 50 p.p.m. sulphur that was proposed to be adopted in the future.

Key words: Sulphur; diesel; gasoline; *Euro Standards*; fuel quality; regulation; Department of Environmen Malaysia; ASTM Standards

Sulphur occurs naturally in crude oil and is found in diesel and gasoline fuels. When fuel is combusted, the sulphur in vehicle fuel causes the release of environmentally harmful compounds such as sulphur oxides and sulphate particles that can contribute to decreased air quality and thus have negative environmental and health effects (Blumberg *et al*. 2003; Alberta Ministry of Transportation 2006). The main environmental concerns related to sulphur emissions are acid rain and the formation of particulate matter (International Marine Organisation 2013). Environmental impacts of acid rain include the acidification of aquatic systems, increasing soil acidity and damage to vegetation (Blumberg *et al*. 2003; Alberta Ministry of Transportation 2006). PM emissions can lead to reduced visibility and also can cause effect to human health (Blumberg *et al*. 2003; Alberta Ministry of Transportation 2006).

Developed countries have several air pollution control programme that have shown to be effective for cleaner air, including adoption of cleaner fuels. Most of these countries also have adopted standards which have ultra-low sulphur content. For example, India, and Thailand adopted *Euro 2 Standards* way back in 2001 and 2002, respectively (Asian Development Bank 2006). Conversely, Hong Kong moved a step forward and adopted *Euro 3 Standards* in 2001, while Singapore, South Korea and Taiwan have migrated to *Euro 4 Standards* (Asian Development Bank 2006). Similar to other countries, the Malaysia government has also enacted the introduction of sulphur-free fuel (with a maximum tolerable level of 500 parts per million (p.p.m.) sulphur) in 2007, which coincides with the *Euro 2 Standards*. The related regulation is *Environmental Quality (Control of Petrol and Diesel Properties) Regulations 2007* (Environmental Quality Act

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Malaysia 1974). However, it was only adopted in Malaysia in 2009 (Asian Development Bank 2006). Since then, all oil companies in Malaysia were required to upgrade their fuel quality to comply with the *Euro 2 Standards*. Formerly, the sulphur limit was 3000 p.p.m. for diesel and 1500 p.p.m. for gasoline (Asian Development Bank 2006).

In Malaysia, sulphur in diesel and gasoline is measured and controlled for environmental and regulatory reasons. Low sulphur fuel is essential for cleaner environment and better engines (Blumberg *et al*. 2003; Zhang *et al*. 2009a; Zhang *et al*. 2009b). The Malaysia government, as such, recommends lower sulphur to assure the vehicles use fuels that comply with the *Euro 2 Standards* levels. The vehicle growth and modern emission reduction technology, which demand an adequate supply of low sulphur fuel, with levels of 50 p.p.m. or 10 p.p.m., however, incited Malaysia to move toward *Euro 4 Standards* implementation, the next step up in fuel quality. The *Euro 4 Standards* restricts sulphur content to a maximum of 50 p.p.m. in fuels. The use of high quality fuel, as part of clean fuel initiative, is expected to improve the air quality and allow for improvements in the emissions control technologies. Unfortunately, the new proposal has been delayed many times and is still underway. On the contrary, the European Union mandated the process of sulphur reduction in fuels to 10 p.p.m. by 2009 (European Commission 2003).

In Malaysia, there is little known about the levels of sulphur in diesel and gasoline. Determining the concentrations of sulphur in these fuels can be useful and vital because as well as being able to provide valuable information about the current situation of sulphur levels at petrol service stations, it may also provide some information about the fuel quality used before and after the adoption of *Euro 2 Standards*. Also the efficiency of the current regulation in controlling the sulphur levels in diesel and gasoline can be assessed. This study, therefore aimed to determine sulphur concentrations in diesel and gasoline using X-ray fluorescence spectrometry technique.

MATERIAL AND METHOD

The diesel and gasoline samples were provided by the Department of Environment Malaysia (DOE). These fuels are routine samples collected from different petrol stations located in Peninsular Malaysia from 2006 to 2014. The energy dispersive X-ray fluorescence (EDXRF) spectrometry method was used for analyzing sulphur concentration in the samples obtained from 2006 to 2009. For samples 2010 onwards, the analysis was performed using a PANalytical Axios wavelength diffractrometer X-ray fluorescence (WDXRF) spectrometer. All analysis was carried out at the Department of Chemistry Malaysia using validated methods, with reference to the American Society for Testing Materials Standards, *ASTM D4294-98 (EDXRF)* and *ASTM D2622-10 (WDXRF)*. These methods are recommended for analyzing sulphur in petroleum and petroleum products (*ASTM Standards* 1998; *ASTM Standards* 2010).

A series of diesel and gasoline fuel standard with known sulphur concentrations were used to set up the calibrations for the determination of sulphur in diesel and gasoline. In this case, sulphur in isooctane and sulphur in diesel fuel from AccuStandard brand were used. These standards were used to set up the calibration lines for quantitative determinations. Precise calibration curves were attained, with correlation of coefficient 0.9999–1.000 (*Figure 1*). The concentration of sulphur in mass % is automatically calculated from the obtained calibration curve (*ASTM Standards* 1998; *ASTM Standards* 2010). Later, it was converted to p.p.m. or μg/g by multiplying 10000.

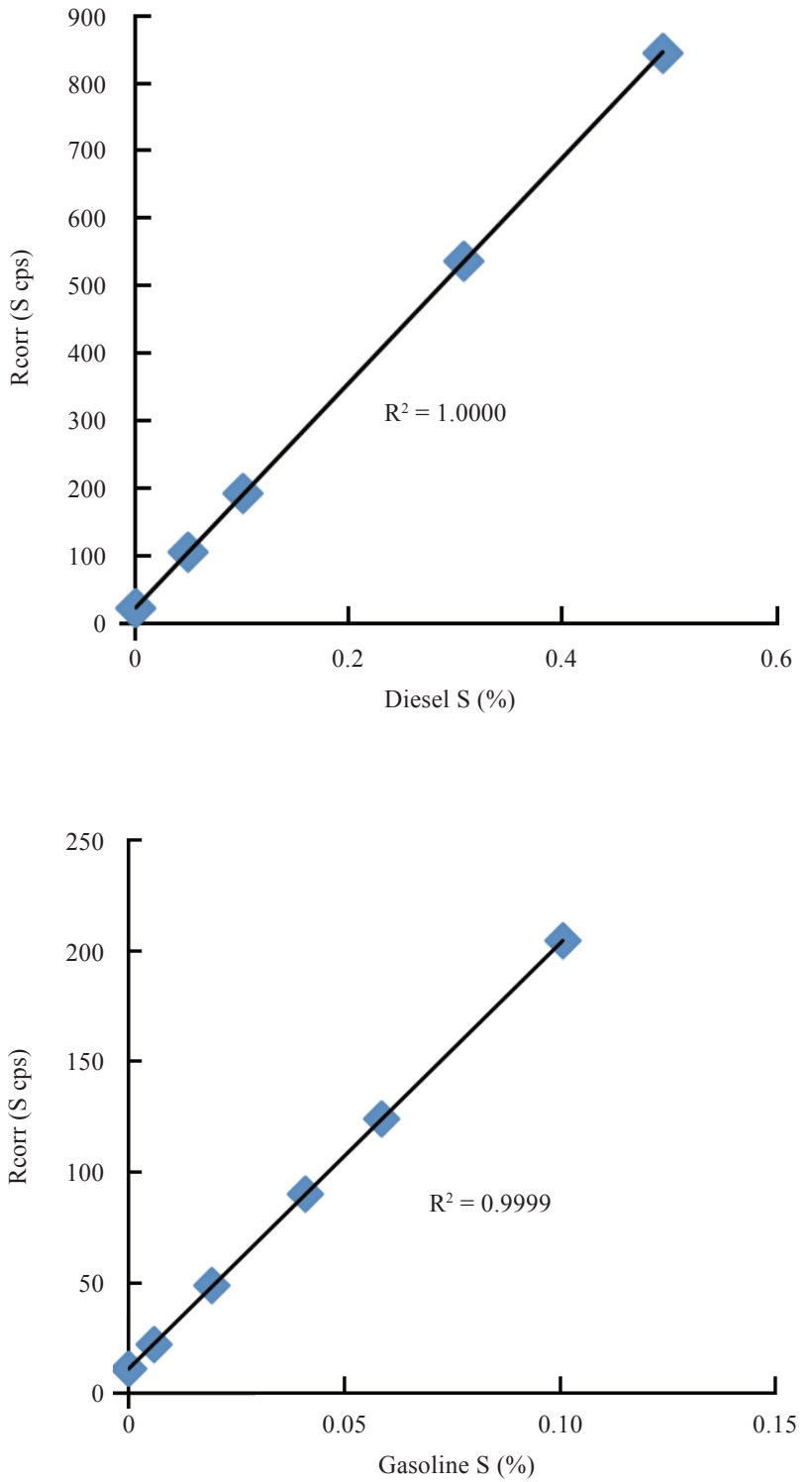

Figure 1. Diesel and gasoline calibration line.

A solution containing 0.003% or 300 µg/g of sulphur in diesel and 0.04% or 400 µg/g of sulphur in gasoline was used as a quality control (QC). The QC sample was analyzed after the measurement of every batch of routine samples.

RESULTS AND DISCUSSION

The concentrations of sulphur analyzed in diesel and gasoline samples across 2006 to 2015 are presented in *Table 1*. In total, 180 diesel samples and 189 gasoline samples were analyzed. Year 2008 was not included in the diesel list as only three samples were sampled for analysis. No values were reported for years 2008, 2007 and 2006 for sulphur in gasoline because there was no legislation requirement to monitor the levels at that time, and as such no samples were collected.

Table 1. Concentration of sulphur in diesel and gasoline samples (2006–2014).

Year		Diesel (p.p.m.)	Gasoline (p.p.m.)
2006	N	32	NA
	Mean	1464	NA
	Median	1170	
	Range	85–2964	NA
2007	N	15	NA
	Mean	1466	NA
	Median	1014	
	Range	283–4310	NA
2009	N	52	62
	Mean	994	95
	Median	434.5	86
	Range	259–3236	28–327
2010	N	22	22
	Mean	457	77
	Median	383	70
	Range	289–698	15–257
2011	N	14	19
	Mean	397	96
	Median	398.5	84
	Range	374–410	22–346
2012	N	15	20
	Mean	382	74
	Median	391	42
	Range	319–436	15–180
2013	N	20	32
	Mean	428	190
	Median	429	207
	Range	196–713	17–322
2014	N	10	34
	Mean	376	125
	Median	340	100
	Range	300–477	10–330

NA: Not avaiable

After *Euro 2 Standards* adoption, the sulphur content in diesel samples across 2010 to 2014 were found to be generally lower than the *Environmental Quality (Control of Petrol and Diesel Properties) Regulations 2007* limit, 500 p.p.m. (*Euro 2 Standards*) with the exception of a few samples. About 18% of the diesel samples in year 2010 and 35% in 2013 exceeded the current sulphur standards limit for diesel (*Figure 2*). On overall, only 4% of samples had levels below 250 p.p.m., 59% between 250 p.p.m.–500 p.p.m. and 37% above 500 p.p.m. (*Figure 2*). Sulphur concentrations tended to be much higher in diesel samples analysed in 2006, 2007 and 2009 (*Table 1*; *Figure 2*), which were consistent with the allowable maximum fuel sulphur content of 3000 p.p.m. at that time. During this period, 60% of diesel samples had sulphur levels above 1000 p.p.m. in 2006, followed by 67% in 2007 and 26% in 2009. Only two samples in 2006, three in 2007 and two in 2009 exceeded the standards limit of 3000 p.p.m.. Although, the concentration of sulphur in all diesel samples in 2011, 2012 and 2014, and in the majority of the 2013 samples were lower than 500 p.p.m., the mean and median concentration for all respective years was close to 400 p.p.m. (*Table 2*). This value was eight times higher than *Euro 4 Standards* Standards that were proposed to be adopted in the future.

Most gasoline sulphur concentrations were substantially lower than the current DOE standard limit of 500 p.p.m. and only 8% samples contained sulphur concentrations in exceedence of 250 p.p.m., while 35% of samples had concentrations above 100 p.p.m. (*Figure 2*). Although the measured concentrations are comparatively low, when compared with the *Euro 4 Standards* requirement (50 p.p.m.), the levels are considered still high. However, the recorded minimum concentrations of sulphur in gasoline samples across the years (2010, 2011, 2012, 2013 and 2014) were < 30 p.p.m. (*Table 1*), which could be considered as an optimistic sign in meeting the *Euro 4 Standards*.

A notable reduction in the annual median sulphur diesel content, close to 62%, was observed over the period 2006–2009 (*Figure 3*). From 2009 to 2013, there seems to be no drastic drop or increase in the annual median sulphur diesel content. The concentrations were rather steady for these years; however, there was a gradual drop in the sulphur content in 2014 (*Figure 3*). In the case of sulphur gasoline, there was no obvious diminution or raise in the annual median concentrations (*Figure 4*). The annual median concentrations were relatively stable, showing small annual variations but no consistent trend in recent years, except in 2013, where the concentration was almost 50% higher than the rest of the years (*Figure 4*).

The obvious sulphur levels in diesel and highly variable sulphur in both diesel and gasoline especially across 2009 to 2014 indicated that although the government had actively adopted stringent vehicle fuel standards, the less aggressiveness in the step towards the use of better quality fuels was a plausible factor impeding the full implementation of fuel emission standards. It was worth noting that regulation reforms were moving ahead to address issues related to fuel quality. More efforts therefore would be required to improve fuel quality by mandating reduced sulphur levels. Upgrading of hydro treatment units at refineries can be a potential solution as it aids the removal of sulphur from the fuel stream (Blumberg *et al*. 2003; Riegel *et al*. 1983; Zhang *et al*. 2009b) and there are refineries that are still using conservative approach and have inadequate advanced refinery equipment.

The results reported here need to be interpreted cautiously as only a number of routine samples were accessible. Moreover, no additional parameters were determined. It would have been informative to assess other parameters stated in the 2007 DOE (Malaysia) standards on fuel regulation in order to determine the quality of diesel and

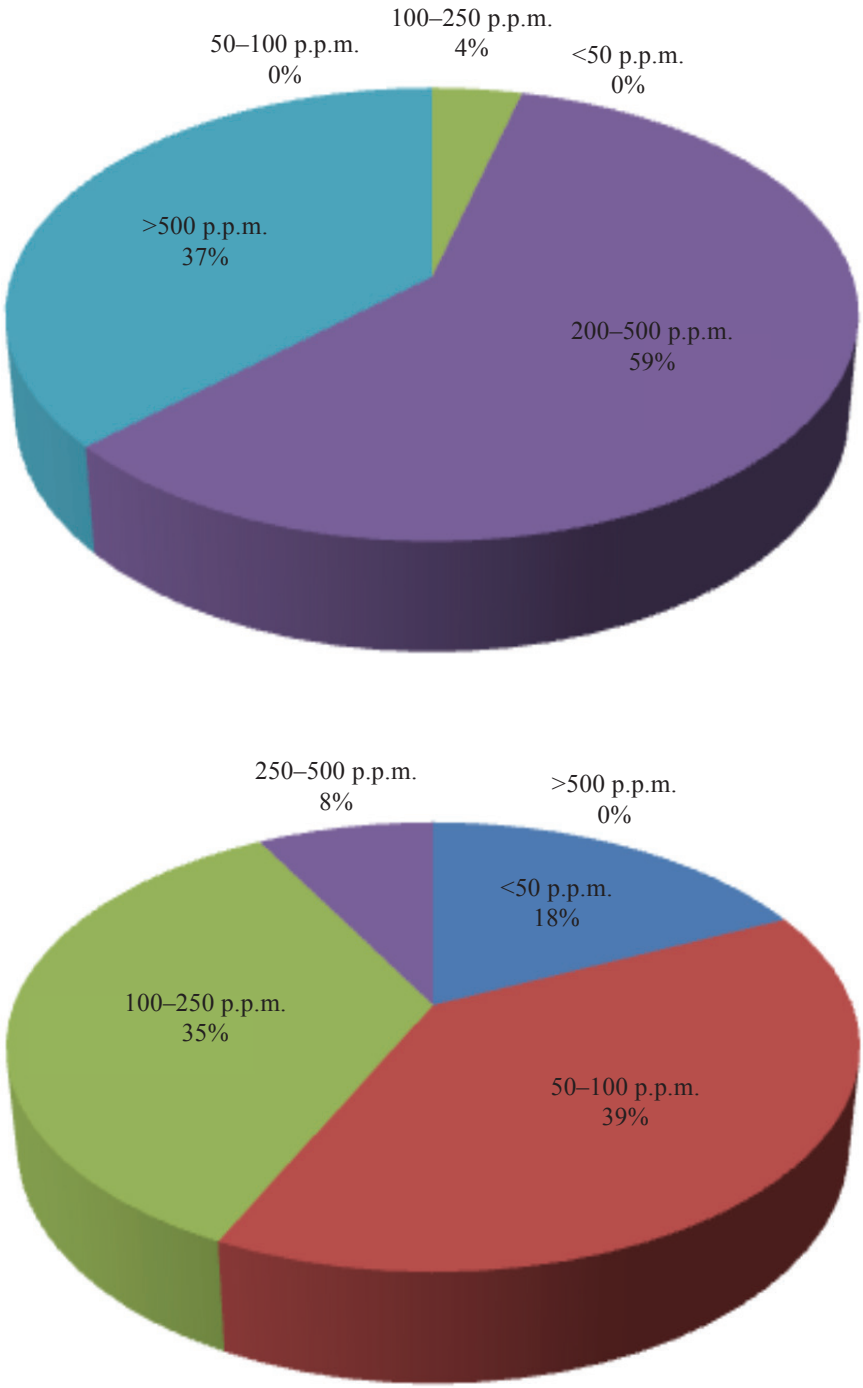

Figure 2. Diesel and gasoline sulphur levels distribution.

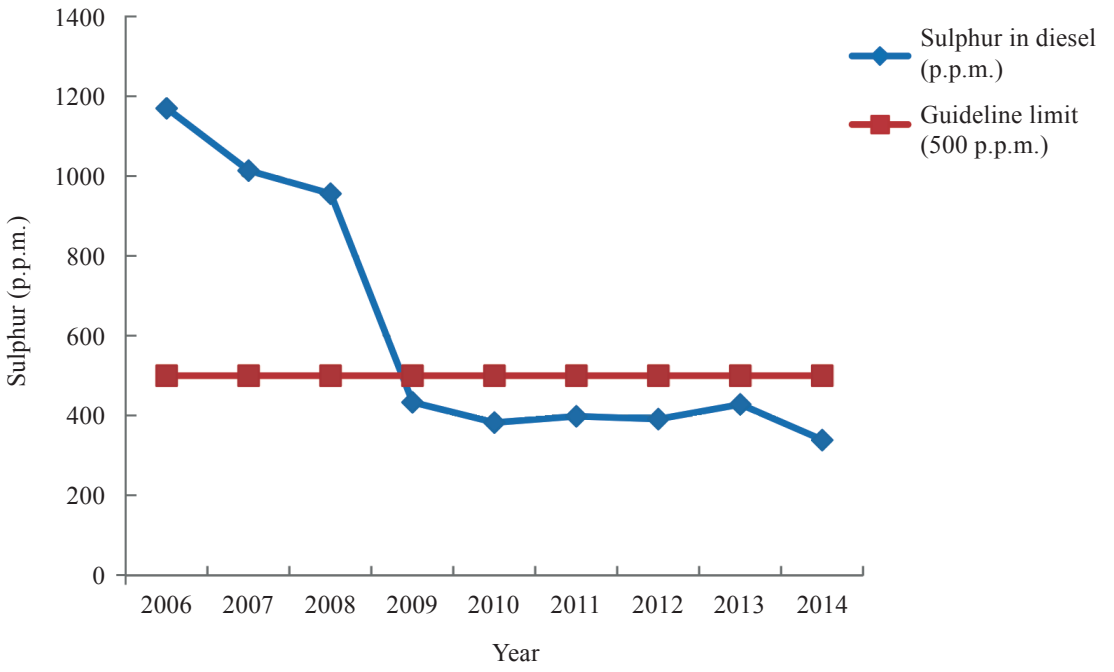

Figure 3. Median diesel sulphur concentration over the period 2006 to 2014.

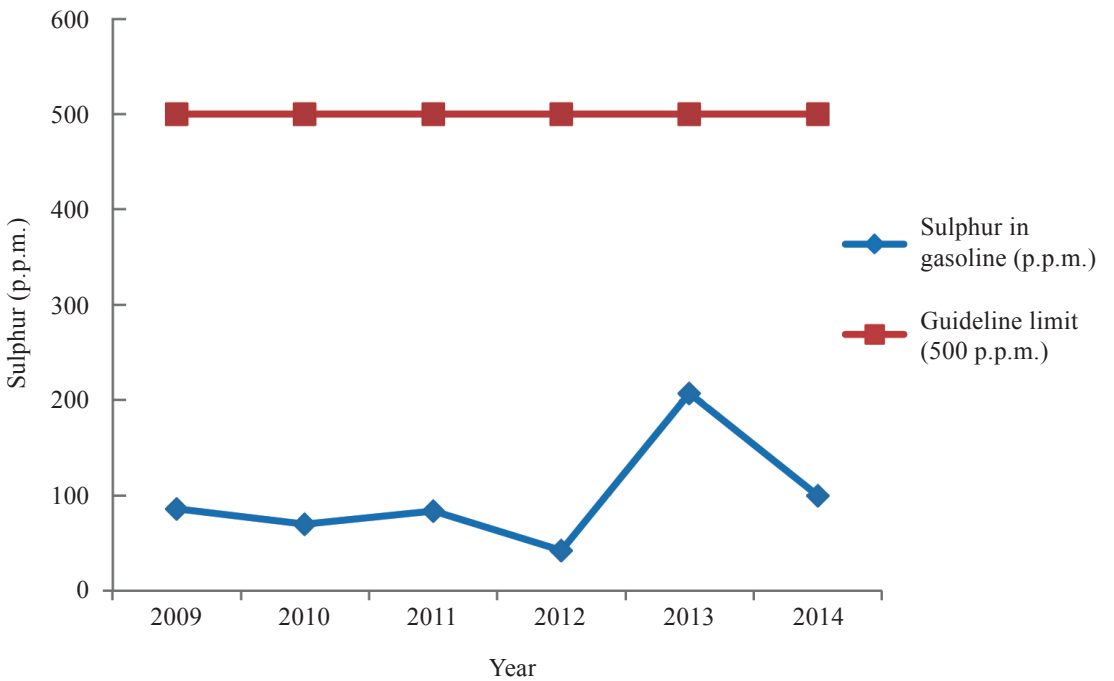

Figure 4. Median gasoline sulphur concentration over the period 2009 to 2014.

gasoline as other fuel specifications also impact emissions (Zhang *et al*. 2009a; Zhang *et al*. 2009b). Although, this was not possible in the current study, it is hoped that those characteristics would be addressed in future.

CONCLUSION

Some information was now available on the current situation of diesel and gasoline sulphur levels in Peninsular Malaysia, with the study which confirmed the presence of sulphur at noteworthy levels for most samples. While the sulphur concentration in fuels was overall slightly below the current DOE (Malaysia) allowable limits, for *Euro 2 Standards*, the concentrations were considered still high if compared with the *Euro 4 Standards*. The results presented here was only based on available samples; additional investigation with the inclusion of more recent samples was, however, recommended to ensure consistency. Finally, with the highly variable sulphur limits in samples, it seemed that there was a need for more rigorous enforcement concerning fuel emissions since there appeared to be inadequate regulation compliance.

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