OIL AND GREASE AND TOTAL PETROLEUM HYDROCARBONS IN THE WATERS OF RAMSAR GAZETTED MANGROVE AREA, JOHOR

MUHAMMAD FAIS FADZIL¹, PANG SWEE YUN², AHMAD RUZAIMI RAZAL¹, POH SENG CHEE¹, SUHAIMI SURATMAN², NUR SYAIRAH DAGANG¹ AND NORHAYATI MOHD TAHIR^{1,2*}

¹School of Marine and Environmental Sciences, ²Institute of Oceanography and Environment, Universiti Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia.

*Corresponding author: hayati@umt.edu.my

Abstract: A study was carried out to evaluate the extent of total petroleum hydrocarbon (TPH) and oil and grease (O&G) pollution in the waters of Ramsar Gazetted mangrove area Johor, during monsoon (February) and post-monsoon (May), 2013. The concentrations of TPH obtained using UV-fluorescence spectroscopy ranged between 6.50-80.3 (mean 20.2 \pm 15.7) µg L⁻¹ Tapis crude oil equivalent and O&G using gravimetric analysis ranged from 0.06-1.50 (mean 0.37 \pm 0.28) mg L⁻¹. Higher TPH in area surrounding the Kukup Island is probably due to illegal desludging and active shipping activities. The O&G was found higher in the Strait of Kukup Island and Pulai River; high surface run-off of lipophilic matters from terrestrial and anthropogenic activities could have enriched the waterways. In general, anthropogenic activities, tidal influence and dynamic of mixing affected the distribution of O&G and TPH in the study area. Values obtained were comparable to reported data elsewhere in Malaysian waters.

Keywords: Anthropogenic activities, oil and grease, Ramsar site, seasonal change, total petroleum hydrocarbon, marine pollutant, South China Sea.

Introduction

Oil and grease (O&G) and total petroleum hydrocarbons (TPH) have been recognized as two major marine pollutants that could degrade marine water quality. The term O&G is used to describe solvent extractable organic matters from a sample matrix such as water and sediment (APHA, 1995). TPH on the other hand described the wide variety of chemical compound derived from crude oil and its by-product (ATSDR, 1999). Composition of O&G varies with organic solvent used in the extraction procedure. For instance, O&G extracted using polar solvent such as dichloromethane is primarily composed of biological lipids and mineral oil hydrocarbons. Mineral oil hydrocarbons are made up of diverse group of mixtures of hydrocarbons derived mainly from crude oil and its distillation products (UNEP, 1992).

Rapid industrialization has increased the demand for the petroleum related products. Transport of crude oil and its industrial derivatives by sea, use of fuel and lubricants in maritime activities and land based oily discharges have been known to cause oil pollution in the marine environment where tanker oil spills and illegal discharges have caught worldwide public attention. Oil pollution may directly or indirectly cause impact to the marine ecosystem such as coastal wetland, mangroves and also human health as the pollutant enters the food chain of the ecosystems. In Malaysia, O&G is being monitored by the Department of Environment (DOE) in her marine water quality monitoring program since 1976 (Mohd Tahir et al., 1999). This parameter is now listed as one of the seven parameters embedded in the Malaysian marine water quality index (DOE, 2013). A review of the environmental quality report published by the DOE from 2006 to 2012 clearly showed that O&G is one of the three parameters that frequently exceeded the Malaysian Marine Water Quality Criteria and Standard (Class E: mangrove estuarine and river mouth water). Sakari and his co-researchers (Sakari et al., 2012; Sakari et al., 2010) have conducted a study on the historical trend of aliphatic and polycyclic aromatic hydrocarbons in the core sediments collected in the Johor Strait in the southern part of Peninsular Malaysia. These authors reported the presence of both aliphatic and polycyclic aromatic hydrocarbons in the sediments and attributed the presence of these compounds to the input from port operations, shipping activities, accidental spillages and also vehicular emissions.

Tanjung Piai, Kukup Island and Pulai River Forest Reserve, located in South West of Johor are important mangrove areas in Malaysia. These areas serve as important environment for migratory and resident birds, which includes the International Union for Conservation of Nature (IUCN) Listed Vulnerable Species, the Lesser Adjutant Stork. It is part of the Important Bird Area (IBA) of South-west Johore, which extends from Parit Jawa to Tanjung Piai (Awang et al., 2014). Mangroves are also known as natural barriers that secure and preserves coastline from physical attacks of wave action, coastal wind and storms that can cause erosion. The importance of these three adjacent areas were apparent when these areas were gazetted as a Ramsar Site (under the Convention of Wetland, Ramsar, Iran 1971) in 2003 (Jusoff & Taha, 2008).

These conserved mangrove areas have boosted the eco-tourism and other socioeconomic of local population tremendously. Although these sites are very interesting in terms of biodiversity, they are also facing threats of pollution due to heavy maritime activities in The Strait of Malacca (Thia-Eng et al., 2000), Johor, Singapore, and Riau Islands in Indonesia (Rusli, 2012a). The large number of vessels using the route could potentially introduce oil pollution in waterways of Johor. Oil spills are very common within shipping activities especially with congested shipping routes and port activities both in Johor (Tanjung Pelepas Port) and Singapore (Jurong Port). Oil pollution through operational, accidental discharges and illegal dumping of oil waste into the oceans could bring a lot of problems and harm to the marine ecological systems. For instance, when illegal discharges of oily wastes washed ashore

in mangrove ecosystem, the oil will deposit on sensitive surfaces such as sediments, mudflats, mangrove roots, seedlings and short plants for considerable length of time and in some cases resulting in death of animals and plants alike. Chronic impacts on mangrove have also been reported up to decades following an oil spill with symptoms such as death of trees with seedling regeneration, defoliation and canopy thinning, leaf yellowing and poor seedling establishment (Duke, 2016).

Considering the importance of this Ramsar site, efforts have been made in order to study the threat and to conserve this area. Nonetheless, the studies are more on the legislation (Rusli, 2012b), eco-tourism (Aminu et al., 2013), biodiversity (Kanniah et al., 2015) and physical threats such as coastal erosion (Awang et al., 2014). Study on the state of oil pollution in the study area is still limited. To our knowledge, Abdullah (1995) works on O&G and TPH in Kukup Island was the only oil pollution study done in this area. The study recorded O&G and TPH concentration at 3.5 mg L⁻¹ and 0.135 mg L⁻¹ (chrysene equivalent); 2.795 mg L⁻¹ (Seligi crude oil equivalent) respectively. This study aims to assess the O&G and TPH in the waters of the Ramsar gazetted area in Johor. The data generated would serve as baseline information on the extent of contamination for management purpose to mitigate the detrimental inputs of O&G and TPH to the study area.

Materials and Methods

Sampling was conducted during February 2013 (monsoon season) and May 2013 (post monsoon season). Details on the sampling stations are given in Figure 1 and Table 1. Briefly, surface water samples were collected using 2.5 L pre-cleaned amber glass bottles with polytetrafluoroethylene (PTFE) lined screw caps. Duplicate samples were collected for replicate analyses. The experimental procedure of the analysis was based on the APHA method 5520B (APHA, 1995). One litre of seawater was extracted three times with dichloromethane (total volume 100 mL) using liquid-liquid

partition method immediately within 4 hours of sampling. The extracts were then combined and stored in the amber bottle at low temperature (5 °C) before transported back to the laboratory. In the laboratory, prior to further analysis, any trace amount of water present in the extract was removed using anhydrous sodium sulphate. The dried extract was then evaporated to

almost dryness using rotary evaporator and then reconstituted in 1 mL of *n*-hexane. The concentrated extract was then stored in the vial and kept refrigerated (4 $^{\circ}$ C) until further analysis.

The O&G concentration was determined using gravimetric methods. An aliquot of the extract was blown to dryness using a gentle

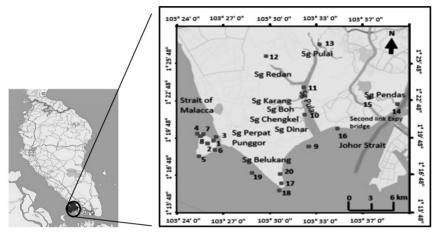


Figure 1: Location map of sampling sites

Stations	Description	Coordinate		Stations	Description	Coordinate	
		Latitude	Longitude	-		Latitude	Longitude
S1	Sg. Ular (estuary)	1.327°N	103.436°E	S11	Sg. Redan (estuary)	1.399°N	103.536°E
S2	Sg. Ular (middle)	1.324°N	103.430°E	S12	Sg. Redan (upstream)	1.442°N	103.494°E
S3	Straits of Kukup	1.332°N	103.439°Е	S13	Sg. Pulai (middle)	1.458°N	103.553°E
S4	Kukup Island (North)	1.337°N	103.418°E	S14	Sg. Pendas (estuary)	1.377°N	103.639°E
85	Kukup Island (West)	1.306°N	103.420°E	S15	Sg. Pendas (upstream)	1.385°N	103.608°E
S6	Kukup Island (South)	1.315°N	103.438°E	S16	Tg. Pelepas Port (mudflat)	1.344°N	103.573°E
S7	Sg. Solok	1.336°N	103.425°E	S17	Tg. Piai (sandbar area)	1.270°N	103.511°E
S8	Sg.Solok (middle)	1.333°N	103.422°Е	S18	Tg.P iai	1.260°N	103.509°E
S9	Tg. Bin	1.320°N	103.541°E	S19	Tg. Piai (West)	1.284°N	103.479°E
S10	Sg. Dinar	1.363°N	103.537°E	S20	Tg. Piai (infront Office)	1.282°N	103.510°E

Table 1: Coordinate and description of the sampling sites

stream of nitrogen gas in a tared aluminum foil and then measured using a 4-decimal place microbalance (model Radwag). The remaining extract was used to determine the TPH concentration using the spectrofluorometric technique. The relatively simple technique of fluorescence spectroscopy is a widely accepted analytical procedure for determining petroleum hydrocarbons (Ehrhardt, 1983), but accurate quantitative analyses based on the UVF technique are possible only where the samples and standard have the same aromatic composition. As the samples collected in the present survey are likely to contain hydrocarbons from a large number of sources, the accuracy of the method using a chosen arbitrary standard is limited and allows only semi-qualitative inter-comparison of available literature data (Mohd Tahir et al., 1997). Nonetheless, TPH concentrations obtained in the present study provide some relative measure on the extent of oil pollution in the area surveyed.

The TPH was measured using UV fluorescence spectrophotometer (model Varian Cary Eclipse) at 310 nm excitation and 374 nm emission wavelengths. Tapis crude oil and chrysene calibration standard were used as arbitrary standards. The method recovery efficiency for TPH from water samples was carried out in the laboratory using artificial seawater. Artificial seawater was spiked with Chrysene standard and extracted using similar method as described above. The mean recoveries were found to be $93 \pm 4\%$. The sampling sites is divided into three area consisting of stations around Pulau Kukup (S1-S8), riverine stations located at Sg. Pulai and Sg Pendas (S10-S15) and near shore stations located along the coast of Tg Piai (S9, S16-S20) and then the data was subjected to a one way ANOVA inter-station comparison and a paired t-test for inter sampling comparison using Microsoft Excel analysis.

Result and Discussion

Oil and Grease Distribution

The results of O&G for coastal areas are summarized in Table 2 and the distribution was

shown in Figure 2. O&G concentrations ranged from 0.06-1.50 (mean 0.41 \pm 0.35) mg L⁻¹ and 0.09-0.73 (mean 0.32 ± 0.17) mg L⁻¹ for the Feb-13 and May-13 sampling, respectively. Highest concentration was observed at S3 in the strait of Kukup and S11 near Sg Redan estuary, for the Feb-13 and May-13 sampling, respectively. S3 is located in vicinity of the Kukup floating village, Kukup terminal jetty and mariculture area. All these activities could influence the concentration of O&G in the area. S11, which is situated in the middle section of Sg. Pulai, could receive O&G inputs from the tidal effect and runoff from surrounding watershed and discharges from Sg Redan sub-basin, which flows into Sg Pulai slightly upstream of S11. Presence of lipophilic compounds from biological materials such as terrestrial plant waxes and algae could also contribute to the O&G content in the waterbody (Tong et al., 1999; Henderson et al., 1990). Nonetheless, despite a few stations exhibiting relatively higher O&G concentrations, statistical tests based on one way ANOVA analysis showed that the O&G concentrations was not significantly different between (p>0.05) and a paired t-test for inter-sampling comparison also showed no significant difference between sampling (p>0.05).

The O&G concentration in the study area is compared to the previously reported study in Malaysian environment (Table 3) and also the Marine Water Quality Criteria and Standards (MWQCS) for

Class E (Mangroves, Estuarine & Rivermouth water). The mean concentrations of O&G of the coastal and open sea stations were generally higher than the recommended MWQCS value of 0.14 mg L^{-1} (DOE, 2013) but generally lower than other reported values in previous studies.

The higher values of O&G found in the study area is probably due to the area being in the shipping lane and within the port limits of a major international seaport, Tanjung Pelapas Port and also the Kukup Ferry terminal (Marine Department Malaysia, 2016). Ships, tankers and ferries are allowed to anchor within the port

Stations	TPH (Chrysene equivalents, µg L-1)		TPH (Tapis crude oil equivalents, µg L-¹)		O&G (mg L ⁻¹)	
	Feb 2013	Feb 2013	Feb 2013	May 2013	Feb 2013	May 2013
S1	0.56	1.34	7.7	20.5	0.30	0.21
S2	1.31	2.45	20.6	38.5	0.60	0.59
S3	2.11	1.06	34.4	15.9	1.50	0.22
S4	0.49	4.03	6.50	64.2	0.40	0.53
S5	2.00	5.02	32.6	80.3	0.30	0.52
S6	0.69	1.98	9.90	30.9	0.10	0.09
S7	0.61	0.75	8.50	10.8	0.20	0.20
S8	2.71	0.63	44.8	8.90	1.00	0.34
S9	0.58	1.08	8.00	16.1	0.06	0.22
S10	0.59	0.76	8.30	11.0	0.90	0.28
S11	0.70	0.85	10.0	12.4	0.20	0.73
S12	1.13	0.61	17.5	34.1	0.20	0.20
S13	1.57	0.68	25.1	19.1	0.50	0.44
S14	1.19	2.28	18.6	35.6	0.30	0.36
S15	0.75	0.57	10.9	13.1	0.30	0.28
S16	0.60	0.51	10.2	6.90	0.20	0.24
S17	0.80	1.24	11.9	18.8	0.30	0.23
S18	0.51	1.13	6.80	17.5	0.20	0.13
S19	1.08	0.79	16.7	11.6	0.40	0.45
S20	1.41	0.73	22.4	10.5	0.30	0.22

Table 2: Concentration of O&G and TPH (in Chrysene standards and Tapis Crude equivalents)

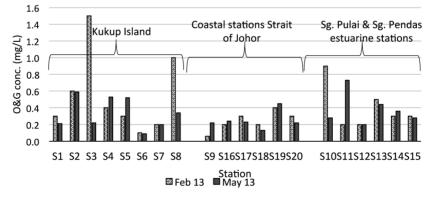


Figure 2: The surface distribution of O&G (mg L⁻¹) in the study area

limit thus operational discharges of O&G in the waters within this area is inevitable. Even though Pulau Kukup, Tanjung Piai, Sg. Pulai and Sg. Pendas are technically classified as Class E under the MWQCS, the fact that they are located within the port limit, the recommended limit for the O&G parameter for the whole area can be compared to that of Class 3 (port, oil and gas field) category value of 5.00 mg L⁻¹. The range (0.06-1.50 mg L⁻¹) and mean (0.32 mg L⁻¹) O&G values found in this study are still well below the recommended value of 5.00

mg L⁻¹ for Class 3 beneficial uses (port, oil and gas field). Annammala et al. (2013) studied the O&G in the waters of Kota Kinabalu and found high concentrations of O&G were recorded in Manukan, Memutik and Sapi Islands near Kota Kinabalu port; the authors attributed pollution due to the shipping and other maritime activities in the vicinity of the port area. Similar observations was found in some coastal areas of the Straits of Malacca and South China Sea (Abdullah, 1995; DOE, 2013); shipping and fishing activities as well as industrial discharges were identified as major contributors of O&G in these areas. In addition, Straits of Malacca is one of the world's busiest tanker routes, transporting crude oils from Middle East to Northeast Asia, where accidental and deliberate oil spills have been reported in the strait (Abdullah, 1995). Although the concentrations of O&G in this study were generally lower than previously reported studies (Table 3), nevertheless it seems that the general trend of O&G distribution was quite similar.

Total Petroleum Hydrocarbons Distribution

Figure 3 shows the concentration of TPH (Tapis crude oil equivalents) in surface coastal water

of the study area as Tapis crude oil equivalents and Chrysene standard equivalents. The range of concentrations recorded were between 0.49 to 2.71 (mean 1.07 \pm 0.62) µg L⁻¹ and 0.51 to 5.02 (mean 1.42 \pm 1.21) µg L⁻¹ chrysene standard equivalents for the Feb-13 and May-13 sampling, respectively. The range of concentrations recorded in study area as Tapis crude oil equivalents were between from 6.5-44.8 (mean 16.6 \pm 10.7) µg L⁻¹ and 6.9-80.3 (mean 23.8 \pm 19.1) µg L⁻¹ for the Feb-13 and May-13 sampling, respectively. Highest TPH was found at S8 in Sg Solok of Kukup Island and S5 near western part of Kukup Island, for the Feb-13 and May-13 sampling, respectively. Interestingly, the range of TPH values obtained in the present study was found to be much lower than reported values by Abdullah (1995) for the area. Abdullah (1995) attributed the high TPH values due to the presence of active shipping activities and also the illegal desludging activities from vessel in vicinity of the study area at that time.

The distribution of O&G and TPH in coastal waters is expected to fluctuate throughout the year and is subjected to change due to the monsoon variation, surface run-off, tidal effect

Location	Mean concentration (range) (mg L ⁻¹)	References	
Kukup Island, Tg. Piai coastal & Sg Pulai estuaries	Kukup Island: 0.44 (0.09 - 1.50); Tg Piai: 0.28 (0.13 - 0.45); Sg Pulai, Sg Pendas, Sg Redan: 0.34 (0.06 - 0.90)	Present Study	
Brunei Bay costal & open sea	0.63 (0.20-1.00)	Waheed <i>et al.</i> (2007)	
Straits of Malacca coastal areas	Kedah: 2.79 (2.00 - 3.63); Melaka: 5.17 (5.00 - 7.00); P.Pinang: 4.38 (2.40 - 8.80); Selangor: 2.81 (1.45 - 5.60)	DOE (2013)	
Straits of Malacca	Perak: 0.51 (0.10 - 0.90); Selangor: 0.53 (0.30 - 1.00); N.Sembilan: 0.42 (0.10 - 0.60); Johor: 1.07 (0.30 - 3.50); Perlis: 0.75 (0.60 - 0.90); Melaka: 0.58 (0.50 - 0.60); P.Pinang: 0.86 (0.40 - 1.40)	_ Abdullah (1995)	
South China Sea	Sarawak: 0.68 (0.10 - 1.50); Pahang: 1.70 (0.80 - 2.40); Terengganu: 0.97 (0.40 - 1.90); Kelantan: 0.77 (0.30 - 1.30); Sabah: 0.45 (0.40 - 0.60)		
Labuan (Manukan, Memutik & Sapi Islands) 43.3 (23.30 - 76.8)		Annammala <i>et al</i> .	
Kota Kinabalu port	24.6 (8.53 - 40.6)	(2013)	

Table 3: Comparison of O&G contents in Malaysian marine waters

and local economic activities. Although the mean concentration of TPH were found higher during May 13 sampling periods, however the paired t-test result showed that the differences between sampling dates is not statistically significant (p>0.05), and the one way-ANOVA analysis also showed no significant difference between stations (p>0.05). These findings suggest that the seasonal change is less likely to exert changes in TPH values in the study area. The surface run-offs and various anthropogenic activities in the study area might play a more dominant role in influencing the distribution of TPH in the area.

TPH in study area is comparable to previously reported studies in Malaysian environment (Table 4). Higher levels of petroleum hydrocarbons were also observed near river mouths and recreational beaches, coastal water with oil refining activities like Port Klang in Selangor (Abdullah *et al.*, 1994). The TPH obtained in the present study is still considered in mid-range and comparable to other studies reported such as Setiu Wetland (Suratman *et al.*, 2012), South China Sea (Kuantan to P. Tioman coastal waters; Law & Mahmood (1988), K. Terengganu; Law & Yusof (1986), Kota Bharu to Kuala Dungun; Phang *et al.*, (1984), Sabah; Law (1990)) and Straits of Malacca (Tg. Piai; Abdullah (1994), nearshore P. Dickson; Law *et al.*, (1991)).

Conclusion

The O&G readings obtained in the study area (range 0.06-1.50 with mean 0.37 ± 0.28 mg L⁻¹), is higher than the MWQCS Class E recommended value of 0.14 mg L⁻¹ for mangroves, estuarine

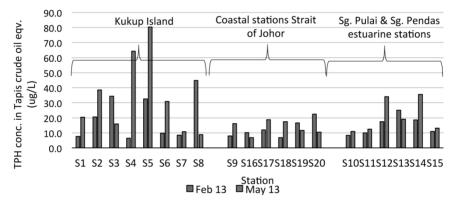


Figure 3: The distribution of TPH in study area (µg L⁻¹ Tapis crude oil equivalents)

and river-mouth area but much lower than the MWQCS Class 3 recommended values of 5.00 mg L⁻¹ for port, oil and gas field. The O&G values obtained were also lower than other reported studies in Malaysian waters. The TPH obtained in study area ranged from 6.50-80.3 (mean 20.2 ± 15.7) µg L⁻¹ Tapis crude oil equivalents, and these values were comparable to reported studies elsewhere in Malaysian waters, particularly with respect to levels of TPH in seawater. Owing to the potential conflict of the beneficial uses in the study area, there is a need to continually monitor the O&G and TPH level in the waters of the Ramsar Gazetted mangrove area to ensure that the water quality with respect to these parameters remains suitable to support Class E beneficial uses.

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Location	Mean concentration (range) (µg L-1)	References
Kukup Island, Tg. Piai coastal & Sg Pulai estuaries	Kukup Island: 27.2 (6.50 - 80.3); Tg Piai: 14.5 (6.80 - 22.4); Sg Pulai, Sg Redan, Sg Pendas: 16.1 (6.90 - 35.6)	Present Study ¹
Setiu Wetland, Terengganu	60 (4-121)	Suratman et al. (2012)
Straits of Malacca	Perak: n.a (20.5 - 186); Selangor: n.a (6.3 – 62.6); N.Sembilan: n.a (16.9 - 101); Johor: n.a (25.0 - 2795); Perlis: n.a (2.1 – 2.8); Melaka: n.a (17.8 - 140); P.Pinang: n.a (1.5 – 18.8)	Abdullah (1995) ²
South China Sea	Sarawak: n.a (3.9 – 63.4); Pahang: n.a (18.3 – 48.6); Terengganu: n.a (15.8 – 90.6); Kelantan: n.a (11.4 – 51.8); Sabah: n.a (30.8 – 44.5)	-
Straits of Malacca	Tg. Piai, Johore: 1115 (n.a)	Abdullah (1994) ³
Straits of Malacca coastal areas	Lumut, Perak: 344 (314 - 386); P.Klang, Selangor: 42 (9 - 129); P.Dickson, N.Sembilan: 28 (8 - 112); Muar, Batu Pahat, Johore Strait, Johore: 28 (8 - 81); K. Perlis, Perlis: 26 (18 - 31)	Abdullah et al. (1994)
Straits of Malacca	P.Dickson, N.Sembilan: 69 (n.a)	Law et al. (1991) ¹
South China Sea	Offshore Sabah: 65 (n.a.)	Law (1990) ²
South China Sea	Coastal waters off Kuantan, Pahang to P.Tioman, Pahang: n.a $(9.5 - 66)$	Law & Mahmood (1988) ¹
South China Sea	Offshore Pahang: 197 (n.a); Coastal waters off K.Terengganu, Terengganu: n.a.(10-175)	Law & Yusof (1986) ¹
Straits of Malacca	Penang: 60 (n.a)	Phang et al. (1984) 4
South China Sea	Coastal waters off K.Bharu, Kelantan to K.Dungun, Terengganu: n.a. (30-130)	

Table 4: Comparison of TPH concentration in Malaysian marine waters

 $n.a = not available; ^{1}$ concentration stated in ESSO Tapis crude oil equivalent; 2 concentration stated in ESSO Seligi crude oil equivalent; 3 concentration stated in Dulang whole oil equivalent; 4 concentration stated in hydrocarbon standard mixtures equivalent (37.5% iso-octane, 27.5% hexadecane, 25% benzene)

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