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Acute and chronic aquatic toxicity of aromatic extracts

Summary of relevant test data

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ABSTRACT

This report describes the experimental procedures and the results obtained in acute and chronic ecotoxicity tests on several aromatic extracts samples. The samples were tested for toxicity to the rainbow trout, *Oncorhynchus mykiss*, the crustacean zooplankter, *Daphnia magna* and the algae, *Selenastrum capricornutum* using water accommodated fractions. These results assist in determining the environmental hazard posed by aromatic extracts.

KEYWORDS

Ecotoxicity, fish, daphnia, algae, aromatic extracts, OECD guidelines, lethal loading, water accommodated fractions.

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SUMMARY

A series of toxicity tests have been performed on representative aromatic extracts from each CONCAWE category, Untreated Distillate Aromatic Extracts (DAE), Treated Distillate Aromatic Extracts (TDAE) and Residual Aromatic Extracts (RAE). The toxicity tests were conducted on rainbow trout (*Oncorhynchus mykiss*), *Daphnia magna*, and green algae (*Selenastrum capricornutum*) using OECD methods. As these are complex petroleum substances comprised of large numbers of poorly water soluble hydrocarbons substances, they were tested as water accommodated fractions (WAFs) in sealed test vessels. Test substances were equilibrated with water at each "concentration" or loading rate and the water phase ("WAF") tested for toxicity. The toxicity results were expressed as "lethal loading (LL)", or "effective loading (EL)" to cause a 50% response.

In acute studies with aromatic extracts, toxicity was observed only with DAE. The Daphnia 48 hour EL_{50} values range from 35.9 to >1000 mg/l. The algal 72 hour E_rL_{50} values range from 18.8 to >100 mg/l, based on the specific growth rate. The fish 96 hour LL_{50} is >1000 mg/l. All other acute data for TDAE and RAE were non-toxic, i.e. >100 - >1000 mg/l. In 21 day chronic studies with Daphnia, no effects were observed for DAE, TDAE or RAE in the range > 100 - >1000 mg/l.

Biomimetic extraction of WAFs has proved to be a successful screening technique for differentiating between aromatic extracts samples with high levels of water-soluble hydrocarbons (i.e. indicating toxicity) and those with lower or non-detectable amounts (i.e. no/low toxicity).

1. INTRODUCTION

CONCAWE has recommended that only ecotoxicity data generated using a “water accommodated fraction” (WAF) approach will be suitable for the purposes of classifying and labelling for environmental hazard in accordance with the criteria given in the CLP Regulation [1]. The experimental procedures and methods of presenting results using WAFs have been described [2].

Company data on the ecotoxicity of the generic category of petroleum substances known as aromatic extracts have been generated since 1992 and were subsequently published by CONCAWE in 2001 [3]. These data provided the basis for the environmental hazard classifications for aromatic extracts recommended in 2005 [4]. More recently, CONCAWE embarked upon a test programme to supplement the available data and all these data have been incorporated into this report.

2. AROMATIC EXTRACTS CATEGORY

Aromatic extracts are complex UVCB substances (Unknown or Variable compositions, Complex reaction products and Biological materials). These consist predominantly of carbon number range C_{15} through C_{50} and typically boil over the temperature interval 250 to 640°C. The generic chemical compositions of aromatic extracts depend on the nature of the crude oils from whence they are derived and the refinery processes that they have undergone

Aromatic extracts are produced as by-products during the manufacture of lubricating oils and waxes performed via petroleum refining. Crude oil is first distilled at atmospheric pressure to remove hydrocarbon streams that boil at temperatures lower than 350°C for further refinement in order to produce fuels. The residue (residuum) of atmospheric distillation of crude oil is, then, distilled under vacuum to produce vacuum distillates and vacuum residuum. These vacuum distillates and residuum contain impurities from the crude oil that can negatively impact lubricant performance, due to odour, oxidative stability or viscosity index. These impurities include polyaromatic compounds (PACs) as well as other aromatic compounds that contain sulphur, nitrogen and oxygen as heteroatoms.

Therefore, solvents with a high affinity for aromatic compounds (e.g. furfural, phenol, N-methyl-2-pyrrolidone) are used to extract these impurities from vacuum distillates and vacuum residuum. The resulting extracts are aromatic extracts. Vacuum residuum is solvent extracted to produce residual aromatic extracts (RAE), whereas treated and untreated distillate aromatic extracts are derived from solvent extractions of the vacuum distillate.

The types of aromatic extracts discussed in this report are:

- **Untreated Distillate Aromatic Extracts (DAE)**: A category comprising six aromatic extracts produced by solvent extraction of vacuum distillate fractions without further processing. This group consists predominantly of hydrocarbons with carbon numbers ranging from C_{15} through C_{50} that boil over the temperature interval of 250 to 640°C. Untreated distillate aromatic extracts consist of a high proportion of alkylated aromatics (mostly of one- or two- ring and three- to five- ring aromatics), with the remainder being naphthenic and iso-paraffinic hydrocarbons. Due to limited treatment, untreated distillate aromatic extracts typically contain higher concentrations of PACs when compared to treated distillate aromatic extracts.

- **Treated Distillate Aromatic Extracts (TDAE)**: A category comprising eighteen distillate aromatic extracts produced by solvent extraction of vacuum distillate fractions with further processing such as hydro-treatment, hydro-desulfurization, clay-treatment, acid-treatment, carbon-treatment, or further solvent extraction. This group consists predominantly of hydrocarbons with carbon numbers above C_{25} that boil over the temperature interval of 250 to 640°C. As for untreated distillate aromatic extracts, treated distillate aromatic extracts consist mostly of one- or two-ring and three- to five-ring aromatics, as well as naphthenic and iso-paraffinic hydrocarbons. While treatment can remove odorous sulphur compounds, traces of polar constituents, and other impurities, treatment does not change the composition of the aromatic extracts significantly. Hydro-treatment can reduce the amount of PACs within the extracts.

- **Residual Aromatic Extracts (RAE)**: A category comprising two distillate aromatic extracts. The residuum from vacuum distillation is extracted with propane, which precipitates out resins, asphaltenes and particulates. The resultant stream is then stripped of the propane and the resultant viscous stream then undergoes the same extraction process used for vacuum distillate streams. Residual aromatic extracts consist of alkylated aromatics, mixed aromatic cycloalkanes, and cyclo-paraffins. This group consists predominantly of hydrocarbons with carbon numbers above C_{25} that boil above 380°C.

The complex and variable composition of such UVCBs, means that it is not possible to define precisely their physical-chemical and environmental properties, but they will fall into a range, defined by the properties and concentrations of the individual hydrocarbons present. Typical physical-chemical properties of the three types of aromatic extracts are presented in **Table 1**. They are typically viscous liquids to waxy solids at room temperature, with low vapour pressure and low water solubility.

Details of the substances included in each of the aromatic extract categories are included in **Appendices 1-3**.

Table 1 Physical-chemical properties of aromatic extracts

Untreated Distilled Aromatic Extracts			
Test Type	**Method [1]**	**Results**	**Reference**
Pour point	ASTM D97	0 - 50°C	5
Boiling range	ASTM D1160, ASTM D2887	250 - 640°C	5
Density absolute	EN ISO 12185, ASTM D1298	0.93 - 1.05 g/cm^3 at 15°C 0.96 - 1.01 g/cm^3 at 70°C	5
Vapour pressure	OECD 104	<0.1 kPa at 20°C	5
Flash point	EN ISO 2719, ASTM D93	240 - 289°C	5
Self-ignition temperature	DIN 51794	>280 - 410°C	5
Viscosity	ISO 3104, ASTM D445	50 – 21087 cSt at 40°C 16 - 24 cSt at 50°C 8.3 - 11 cSt at 70°C 3.8 - 124 cSt at 100°C	5
Treated Distilled Aromatic Extracts			
Test Type	**Method [1]**	**Results**	**Reference**
Pour point	ASTM D97	0 °C	5
Boiling range	ASTM D1160	350 - 550°C	5
Density absolute	EN ISO 12185	0.94 - 1.05 g/cm^3 at 15°C 1.02 - 1.031 g/cm^3 at 80°C	5
Vapour pressure	OECD 104	<0.1 kPa at 20°C	5
Flash point	EN ISO 2592	254 - 303°C	5
Self-ignition temperature	DIN 51794	>280°C	5
Viscosity	ISO 3104	400 cSt at 40°C 20 - 147 cSt at 100°C	5

Residual Aromatic Extracts			
Test Type	**Method** [1]	**Results**	**Reference**
Pour point	ASTM D97	3 - 9 °C	5
Boiling range	EN 15199	380 - 700°C	5
Density absolute	ASTM D1298	0.96 - 1.02 g/cm^3 at 15°C	5
Vapour pressure	OECD 104	<0.1 kPa at 20°C	5
Flash point	ASTM D93	298 - 335°C	5
Self-ignition temperature	DIN 51794	>300 - 397°C	5
Viscosity	ASTM D445, ISO 3104	2000 – 7000 cSt at 40° 55 - 189 cSt at 100°C	5

[1] Commonly accepted method guidelines set by American Society for Testing and Materials (ASTM), International Organization for Standardization (ISO), Deutsches Institut für Normung (DIN), and Organisation for Economic Co-operation and Development (OECD)

3. CHARACTERISATION OF TEST SUBSTANCES

Acute and chronic aquatic toxicity tests have been performed on several aromatic extracts samples obtained from various European refineries (**Table 2**). These test substances were selected as representative of the 3 aromatic extracts categories (**Appendices 1-3**).

Three samples (one each of DAE (PSG-1860), TDAE (PSG-1861) and RAE (PSG-1857)) were tested (acute and chronic) by BP Oil Europe in 1994-5 [6-14]. Further samples of RAE were tested by Kuwait Petroleum (EL 4199) in 2005 [15,16] and Total (Extrait 5) in 2007 [17]. For all of these samples, there were no data available on the characterisation of the test substances.

Other samples of DAE and TDAE tested in a more recent CONCAWE test programme were characterised by ExxonMobil Biomedical Sciences, Inc. (EMBSI) together with a RAE sample (**Tables 3** and **4**).

Table 2 Aromatic extracts samples tested for aquatic toxicity

Descriptor	Substance	EINECS No.	CAS No.	Lab. Code No.
Untreated Distillate Aromatic Extracts				
Light paraffinic distillate solvent	Extracts (petroleum) light paraffinic distillate solvent	265-104-2	64742-05-8	MRD-08-346
Heavy paraffinic distillate solvent	Extracts (petroleum) heavy paraffinic distillate solvent	265-103-7	64742-04-7	PSG-1860
Heavy paraffinic distillate solvent	Extracts (petroleum) heavy paraffinic distillate solvent	265-103-7	64742-04-7	MRD-08-347
Treated Distillate Aromatic Extracts				
Solvent refined heavy paraffinic distillate solvent	Extracts (petroleum), solvent refined heavy paraffinic distillate solvent	272-180-0	68783-04-0	PSG-1861
Solvent refined heavy paraffinic distillate solvent	Extracts (petroleum), solvent refined heavy paraffinic distillate solvent	272-180-0	68783-04-0	MRD-08-375
Residual Aromatic Extracts				
Residual aromatic extract	Extracts (petroleum), residual oil solvent	265-110-5	64742-10-5	PSG 1857
Residual aromatic extract	Extracts (petroleum), residual oil solvent	265-110-5	64742-10-5	EL 4199
Residual aromatic extract	Extracts (petroleum), de-asphalted vacuum residue solvent	295-332-8	91995-70-9	Extrait 5

Detailed compositional analyses of the seven samples tested by EMBSI has been carried out using a high-resolution approach involving comprehensive two-dimensional gas chromatography (GCxGC). A summary of the GCxGC compositional analysis [18,19] is provided in **Table 3**. This technique has previously been employed for the detailed characterisation of complex middle distillate fuels [20-22]. However, the upper volatility range of GCxGC currently limits its application to analysis of the lighter ($<C_{35}$) petroleum products and it is therefore recognised that this technique is often unable to provide a full and comprehensive description of all the components present in aromatic extracts, which typically cover the C_{15} - C_{50} range [5].

Table 3 Compositional analysis of some aromatic extracts samples

Analysis	Untreated Distillate Aromatic Extracts (average of 5 samples)	Treated Distillate Aromatic Extracts (1 sample)	Residual Aromatic Extracts (1 sample)
n-Alkanes (%wt)	3.27	0.55	0.92
iso-Alkanes (%wt)	5.27	1.04	0.77
Naphthenics (%wt)	7.15	3.10	2.58
Aromatics (%wt)	12.25	7.67	5.23
Naphthenic Aromatics (%wt)	16.26	3.41	3.23
Sum (%wt) of components $<C_{30}$	44.20	15.76	12.74

Review of the GCxGC data [18,19] confirms that a higher percentage of the hydrocarbon components (i.e. < C_{30}) could be analysed for DAE (~44% mean) compared to TDAE (~16%) or RAE (~13%). In addition, there was, on average, a lower carbon chain distribution for the DAE hydrocarbon components compared to TDAE and RAE. A comparison of the composition of the seven samples included in **Table 3** is presented in **Table 4**.

Additional PAH analysis was performed using a capillary GC-MS method based on that described in U.S. EPA SW-846 8270C (Modified). Details of the 16 Priority PAHs in the various aromatic extract samples analysed using this method are provided in **Table 5**.

Table 4 Detailed compositional analysis of some aromatic extracts samples

Sample description	EMBSI ID	Total Saturate Fraction (wt%)	Total Aromatic Fraction (wt%)	Total Fraction $<C_{30}$ (wt%)	Sum of 16 priority PAHs (ppm)
Untreated Distillate Aromatic Extracts					
Light paraffinic distillate solvent (CAS 64742-05-8)	MRD-08-346	13.06	86.94	100.00	2077
Heavy paraffinic distillate solvent (CAS 64742-04-7)	MRD-08-347	6.28	22.72	29.00	302
Heavy paraffinic distillate solvent (CAS 64742-04-7)	MRD-08-390	3.19	13.14	16.33	34
Heavy paraffinic distillate solvent (CAS 64742-04-7)	MRD-08-420	54.60	18.40	73.00	1.2
Heavy paraffinic distillate solvent (CAS 64742-04-7)	MRD-08-470	1.43	1.37	2.80	8.8
Treated Distillate Aromatic Extracts					
Solvent refined heavy paraffinic distillate solvent (CAS 68783-04-0)	MRD-08-375	4.67	11.08	15.75	9.6
Residual Aromatic Extracts					
Residual oil solvent (CAS 64742-10-5)	MRD-09-416	4.29	8.46	12.75	117

Table 5 Analyses (in ppm) of individual PAHs for various aromatic extracts

Analytes	DAE					TDAE	RAE
	MRD-08-346	MRD-08-347	MRD-08-390	MRD-08-420	MRD-08-470	MRD-08-375	MRD-08-416
Naphthalene	ND	ND	ND	ND	5.46	0.234	0.176
Acenaphthylene	0.094	ND	ND	ND	ND	ND	ND
Acenaphthene	0.474	ND	ND	ND	ND	0.143	0.077
Fluorene	3.42	0.167	0.812	0.144.	0.265	0.327	0.551
Anthracene	13.2	ND	0.291	ND	ND	ND	ND
Phenanthrene	208	1.05	3.85	0.688	0.746	0.662	5.37
Fluoranthene	120	ND	ND	ND	ND	0.137	0.593
Pyrene	924	9.98	2.06	0.358	0.289	0.432	3.42
Benz[a]anthracene	78.76	2.41	ND	ND	ND	ND	2.31
Chrysene/Triphenylene	716	22.7	9.62	ND	1.15	2.30	26.8
Benzo[b]fluoranthene	8.89	14.2	3.56	ND	ND	1.15	10.7
Benzo[k]fluoranthene	ND	ND	ND	ND	ND	ND	ND
Benzo[a]pyrene	4.14	23.0	4.28	ND	ND	1.25	9.20
Indeno[1,2,3-cd]pyrene	ND	ND	ND	ND	ND	ND	6.86
Dibenzo[a,h]anthracene	ND	19.3	ND	ND	ND	ND	10.3
Sum of 16 priority PAHs	2077	302	34	1.2	8.8	9.6	117

ND = Not Detected

4. TEST METHODS

4.1. GENERAL APPROACH

Mixtures of poorly water soluble, complex chemicals, like petroleum products, present special problems with regard to preparing aqueous solutions for toxicity testing. With soluble chemicals, the amount of chemical dissolved in water is varied in incremental steps to produce a range of toxic responses, from which a "dose - response" relationship and the associated median lethal concentration (LC_{50}) may be derived. With mixtures of poorly soluble complex chemicals, un-dissolved material appears as soon as the least soluble component reaches water saturation. Thereafter, the relative composition of the water phase varies in a non-linear fashion from the composition of the "neat" substance [23]. This does not apply to pure substances where the concentration will, if sufficient time is provided, equal the solubility limit when excess is added, regardless of the amount of excess. For poorly water soluble, complex chemicals, it has become a standard practice to test toxicity at substance additions far in excess of the amount that will dissolve, resulting in a two phase system.

There are, however, many divergent procedures for establishing and maintaining equilibrium between water and un-dissolved substance [24]. A recognised guideline [25] for testing mixtures of poorly water soluble substances has been developed by the Group of Experts on the Scientific Aspects of Marine Pollution (GESAMP). This method involves stirring various amounts (loading ratios) of test substance with water for a sufficient time to reach equilibrium, followed by separation of the water phase ("water accommodated fraction" or "WAF"). Toxicity testing of the WAFs generated in this manner allows the determination of the amount of the substance equilibrated with water which will cause 50% mortality. This end-point has been termed LL_{50} (lethal loading) to distinguish it from the LC_{50} [26]. (The LC_{50} is determined by completely dissolving the chemical in water and then making a dilution series to obtain a relationship between concentration and lethality). The LL_{50} procedure has also been described in a CONCAWE report [2]. It is also the approach specified by MARPOL for the marine pollution testing of poorly soluble mixtures [27] and by OECD for the aquatic toxicity testing of difficult substances and mixtures [28].

A further complication for the testing of hydrocarbon liquids is their volatility, particularly from aqueous solution. Although it may be environmentally unrealistic, it is necessary to prevent volatilization of the substance in order to maintain constant concentrations and, by doing so, to determine its inherent toxicity. This necessitates using closed test systems. In preparing WAFs, some headspace is necessary to achieve adequate interfacial area and mixing. In each test measured amounts of test substance are added to measured volumes of the appropriate test medium (for fish, daphnia and algae). The vessels containing the medium and the test substance are then sealed leaving only a small headspace, and the contents stirred with a 1-2 cm vortex depth for a period of time shown to be sufficient for the aqueous and test substance phases to equilibrate. After stirring, the contents of the vessels are left to stand to allow any un-dissolved material to separate out. The aqueous phases – the WAFs – are then drawn off for use in the tests. Control media are subject to the same regime but do not contain the test substance. It is important that mixing is sufficient to ensure that the aqueous phase is in equilibrium with the un-dissolved hydrocarbon phase. Mixing needs to be slow enough not to cause dispersion or emulsification of the un-dissolved hydrocarbon, yet vigorous enough and long

enough to attain equilibrium. In the current studies, mixing was done with a magnetic stirring bar set to develop a vortex at the surface of about 10% of the water height. Preliminary studies showed that this mixing condition was sufficient to reach equilibrium within 24 or 48 hours. After mixing for this period, solutions were allowed to stand for 1 hour before use in order to facilitate phase separation. The mixing vessel was either fitted with a stopcock at the bottom of the vessel or contained a glass tube for siphoning off the water phase, without contamination by the surface layer of un-dissolved hydrocarbon.

The exposure vessel for the fish test were typically a cylindrical bottle (volume 4.5 l) and with a stopcock at the bottom for removing liquid. The exposure vessel was filled to the top and stoppered with no headspace. The test chambers for the daphnia and algae studies were respectively 130-200 ml and 125 ml Erlenmeyer flasks with ground glass stoppers and were filled completely with test solution (no headspace).

All the studies were conducted in accordance with the principles of Good Laboratory Practice (GLP).

4.2. SCREENING STUDIES USING BIOMIMETIC EXTRACTION

Prior to any toxicity testing using daphnia or algae in the recent CONCAWE test programme, a screening exercise which involved analysing WAF samples of each of a number (6) of aromatic extracts by Biomimetic Solid Phase Micro-extraction or “SPME-biomimetic extractions” was undertaken [29]. WAFs were prepared at a loading rate of 100 mg/l and mixed for 48 hours. After settling for an hour, samples were taken and analysed in duplicate by GC-FID.

Sample aliquots (ca. 20 ml) taken directly from WAF systems were placed in septum sealed glass vials with no headspace and placed in an auto-sampler configured for automated SPME injections. A 30 µm poly-dimethyl siloxane (PDMS) SPME fibre (0.132 µl) was equilibrated with each sample for 100 minutes at 30ºC with rapid agitation (250 rpm) and no headspace. A single fibre was used for all automated sample analyses [29,30].

The SPME and liquid hydrocarbon calibration samples were analysed by GC-FID on a 15 m x 0.53 mm id capillary column with 1.5 µm Rtx-1 stationary phase (Restex). The SPME-BE method was calibrated by making 1 µl injections of a series of aromatic hydrocarbon standard solutions. The molar response factor of 2,3-dimethylnaphthalene was used for converting the observed GC-FID response to nanomoles of organic constituents on the PDMS fibre. Fibre results are normalized to the volume of PDMS and reported as micromoles (µmol) as 2,3-dimethylnaphthalene / millilitre (ml) PDMS.

4.3. FISH ACUTE STUDIES

The fish acute toxicity tests were conducted in accordance with OECD Guideline 203 (equivalent to EC methods for the determination of ecotoxicity, C1 – Acute toxicity for fish). The test species chosen for these studies was the salmonid, *Oncorhynchus mykiss*, the rainbow trout. The salmonids are considered to be one of the more sensitive test species, particularly to hydrocarbons. The rainbow trout is a common laboratory test species for determining toxicity to freshwater fish. Details of the source, husbandry and selection procedures are available in the laboratory

reports [7,9,11]. The fish used for the studies were 4.8 – 5.2 cm in length (mean weight range 1.06-1.31 grams) and were not fed during the exposure period. The loading rates evaluated were 0 and 1000 mg/l. 10 fish per vessel (1 for the control, 2 for the 1000 mg/l loading rate) were evaluated. The fish biomass loading was 0.53 - 0.66 g/l.

Fresh WAFs were prepared on a daily basis by mixing the test substance and test media approximately 24 hours and allowing 1 hour for settling. After settling, WAFs were used for daily renewal of the exposure medium (semi-static). Renewals were done by emptying most of the water (typically ≥ 80%) from the bottom port on each exposure vessel and then expeditiously re-filling by siphon from the mixing vessel. No specific hydrocarbon analysis of WAFs was performed, only TOC analysis.

The total exposure periods were 96 hours. Water hardness was 100 mg/l (as $CaCO_3$) with a pH of 7.3 to 7.7. The temperature was a constant 14°C and the light duration was 16 hours. Dissolved oxygen was 9.8 to 10.1 mg/l throughout all exposures and no reductions in oxygen concentrations sufficient to influence the results were observed during the tests. Observations were made at 3 hours after the commencement of exposure and once daily, thereafter.

4.4. DAPHNIA STUDIES

4.4.1. Daphnia acute studies

These tests were carried out in accordance with OECD Guideline 202, Part I (equivalent to EC methods for the determination of ecotoxicity, C2 – Acute toxicity for Daphnia). The test species was *Daphnia magna*, a fresh water invertebrate commonly used for toxicity testing. Details of the husbandry and selection of test organisms are provided in the laboratory reports [6,8,10,15,31,32]. The organisms used for testing were less than 24 hour old neonates, from parents ranging from 12 – 28 days. For definitive studies, either four replicates, each involving 5 organisms [31], or two replicates, each involving 10 organisms [6,8,10], were tested at each loading rate. The exposure period was 48 hours.

Reconstituted water was used for the daphnia studies. Fresh WAFs were prepared on a daily basis by mixing the test substance and test media for either ~24 hours [6,8,10,15] or ~48 hours and then allowing 1 hour for settling. After settling, WAFs were used for daily renewal of the exposure medium (semi-static). No specific hydrocarbon analysis of WAFs was performed, only TOC analysis, for the earlier studies [6,8,10,15], whereas WAFs were extracted by SPME and analysed by GC-FID for the subsequent studies [31,32].

WAFs were prepared employing the same approach to those for the fish studies using similar equipment but they were not renewed daily as for the fish (i.e. static tests). The WAFs were siphoned into sealed flasks, typically 130 ml [6,8,10] or 200 ml [29,30], without headspace, and the daphnia were introduced. The light duration was 16 hours at 108-215 Lux. No reductions in dissolved oxygen concentration (range 7.8 – 9.0 mg/l) or pH (range 7.7 – 8.8) were seen at the end of the 48 hour exposure period. Observations were made for immobilization at 24 and 48 hours. The daphnids were not fed during the exposure periods, expect for the study on RAE EL 4199 [15]. No data are available on the analysis of WAFs at the beginning and end of the exposure.

4.4.2. Daphnia chronic studies

The chronic daphnia experiments were carried out in accordance with OECD Guideline 211 (equivalent to EC methods for the determination of ecotoxicity, C2 – Acute toxicity for Daphnia). Details of the husbandry and selection of test organisms are provided in the laboratory reports [12,13,14,16,17]. The organisms used are young neonates less than 24 hour old. This semi-static test involves 10 individuals per loadings, with four replicates for each. The duration of the test is 21 days.

Reconstituted water was used for the daphnia studies. WAFs were prepared in the same manner using the same equipment and analyses as for fish and were renewed three times per week .The WAFs were siphoned into covered glass flasks, typically 120 ml [18] and 400 ml [12-14], and the daphnia were introduced. The light duration was 16 hours. No reductions in dissolved oxygen concentration (range 7.8 – 8.4 mg/l) or pH (range 7.7 – 7.9) were seen at the end of the 21 day exposure period. The daphnids were daily fed with mixed unicellular algal suspension during the exposure periods.

At the renewal periods, young daphnids and unhatched eggs were collected and counted. The total number of living offspring produced per parent animal alive is assessed. No data are available on the analysis of WAFs at the beginning and end of the exposure.

4.5. ALGAL GROWTH INHIBITION STUDIES

The algal growth studies were conducted in accordance with OECD guideline 201. The test species was *Pseudokirchneriella subcapitata* [33,34] alternatively known as *Selenastrum capricornutum)*. Details of the culture methods are provided in the laboratory reports [33,34]. The algae used were taken from 9 day old stock cultures in the log phase of growth. Initial concentrations were approximately 1.0×10^4 cells/ml in each replicate test chamber. The exposure period was 72 hours.

WAFs were prepared in algal growth medium. WAFs were prepared employing the same approach to those for the fish studies using similar equipment but they were not renewed daily as for the fish (i.e. static tests). WAFs were analysed at the beginning of the test period, and again on a composite from test flasks after 72 hours. Test vessels, typically 125 ml, were filled completely with inoculated WAF and then closed with ground glass stoppers. Replicate vessels were set up for each treatment and the control to facilitate daily algal cell counting and pH measurements. The flasks were incubated at 22°C on an orbital shaker, at 100 cycles/min or rpm. Lighting was continuous and in the range of 6700 to 8300 Lux. Cell counts were determined at 24, 48, and 72 hours using a haemocytometer and microscope [33,34]. The pH changes during these studies were within the range 8.0 – 8.9.

5. RESULTS

5.1. SCREENING STUDIES

A summary of the biomimetic extraction (BE) results for six aromatic extracts samples [29] is shown in **Table 6**. SPME screening was carried out on WAFs prepared at 100 mg/l loading rates. On the basis of these screening data, one DAE sample with a positive BE result (MRD-08-346) together with two other samples (one DAE (MRD-08-347) and one TDAE (MRD-08-375)) with negative BE results (to confirm lack of toxicity) were taken forward for further ecotoxicity testing [31,33]. Both of the latter samples showed no effect to Daphnia (acute) and algae when tested at the 100 mg/l limit, whereas DAE sample MRD-08-346 showed significant immobilisation for Daphnia and growth inhibition for algae at the same dose level. Subsequent definitive testing [32,34] confirmed the toxicity values shown in **Tables 7** and **8.**

Table 6 Summary of biomimetic extraction screening results for six aromatic extracts

Sample description	CAS number	EMBSI ID	Mean BE result as µmol 2,3-DiMeNaph per ml PDMS	Further ecotox testing	Reference
Untreated Distillate Aromatic Extracts					
Light paraffinic distillate solvent	64742-05-8	MRD-08-346	10.9	Yes	29
Heavy paraffinic distillate solvent	64742-04-7	MRD-08-347	ND	Yes	29
Heavy paraffinic distillate solvent	64742-04-7	MRD-08-390	ND	No	29
Heavy paraffinic distillate solvent	64742-04-7	MRD-08-420	ND	No	29
Heavy paraffinic distillate solvent	64742-04-7	MRD-08-470	ND	No	29
Treated Distillate Aromatic Extracts					
Solvent refined heavy paraffinic distillate solvent	68783-04-0	MRD-08-375	ND	Yes	29

ND = Not Detected

5.2. AROMATIC EXTRACTS ECOTOXICITY DATA

A summary of all the relevant acute and chronic ecotoxicity data from studies of aromatic extracts samples on fish, Daphnia and algae generated using WAFs is detailed in **Tables 7** and **8**. These include previous data up to 2007 and all the ecotoxicity data generated from the more recent CONCAWE test programme.

Table 7 Summary of all the acute ecotoxicity data with fish, Daphnia and algae for aromatic extracts

Sample details	Fish 96 h LL_{50} (mg/l) OECD 203	Daphnia 48 h EL_{50} (mg/l) OECD 202	Algae 72 h E_rL_{50} (mg/l) OECD 201	Reference
Untreated Distillate Aromatic Extracts				
Light paraffinic distillate solvent MRD-08-346 CAS No 64742-05-8	NT	<100 35.9	<100 18.8	Daphnia [31,32] Algae [33,34]
Heavy paraffinic distillate solvent PSG 1860 CAS No 64742-04-7	>1000	>1000	NT	Fish [8] Daphnia [9]
Heavy paraffinic distillate solvent MRD-08-347 CAS No 64742-04-7	NT	>100	>100	Daphnia [31] Algae [33]
Treated Distillate Aromatic Extracts				
Solvent refined heavy paraffinic distillate solvent PSG 1961 CAS No 68783-04-0	>1000	>1000	NT	Fish [11] Daphnia [10]
Solvent refined heavy paraffinic distillate solvent MRD-08-375 CAS No 68783-04-0	NT	>100	>100	Daphnia [31] Algae [33]
Residual Aromatic Extracts				
PSG 1857 CAS No 64742-10-5	>1000	>1000	NT	Fish [7] Daphnia [6]
EL 4199 CAS No 64742-10-5	NT	>100	NT	Daphnia [15]

NT = Sample not tested

Table 8 Summary of the chronic ecotoxicity data with Daphnia for aromatic extracts

Sample details	Daphnia 21 day EL_{50} (mg/l) (OECD 211)	Reference
Untreated Distillate Aromatic Extracts		
Heavy paraffinic distillate solvent PSG 1860 CAS No 64742-04-7	>1000	12
Treated Distillate Aromatic Extracts		
Solvent refined heavy paraffinic distillate solvent PSG 1961 CAS No 68783-04-0	>1000	14
Residual Aromatic Extracts		
PSG 1857 CAS No 64742-10-5	>1000	3
EL 4199 CAS No 64742-10-5	>100	16
Extrait 5 CAS 91995-70-9	>500	17

5.3. FISH ACUTE STUDIES

The daily cumulative mortality data at each loading level were used to calculate the lethal loading causing 50% mortality (LL_{50}). The 96 hour LL_{50} values (**Table 7**) are >1000 mg/l for DAE (PSG 1860), TDAE (PSG 1961) and RAE (PSG 1857) samples.

5.4. DAPHNIA STUDIES

5.4.1. Daphnia acute studies

The cumulative immobilization at 48 hours at each loading level were used to calculate the effective loading causing 50% immobilization (EL_{50}). The data sets were all amenable to probit analysis. The 48 hour EL_{50} values (**Table 7**) exhibit a wide range of toxicity values from 35.9 - >1000 mg/l for DAE samples (MRD-08-346, PSG 1860, MRD-08-347) and >100 mg/l for both TDAE (PSG 1961, MRD-08-375) and RAE samples (PSG 1857, EL 4199).

5.4.2. Daphnia chronic studies

The cumulative assessment of alive juveniles at 21 days at each loading level were used to calculate the effective loading causing 50% of the reproductive output (EL_{50}). The 21 day EL_{50} (**Table 8**) is >1000 mg/l for the DAE (PSG 1860) and TDAE (PSG 1961) samples. The 21 day EL_{50} values for the three RAE samples tested (PSG 1857, EL 4199, Extrait 5) ranged from > 100 - >1000 mg/l.

5.5. ALGAL TOXICITY STUDIES

Acute toxicity results are expressed as the effect loading 50 (E_rL_{50}); that is the loading rate of test substance in dilution water which results in a 50% reduction in growth derived from the average specific growth rate (r) relative to the control for the specified time of exposure.

The 72 hour growth inhibition for each substance loading rate/concentration was estimated based on the percent inhibition relative to the control. The specific growth rate for each loading rate/concentration was determined by calculating the slope of the regression line of the Ln or ^{e}log (cell density) versus time using the PROC REGRESSION procedure from SAS [35]. The average specific growth rate was calculated in accordance with the formula listed in the OECD Guideline 201. The 72 hour E_rL_{50} values (**Table 7**) ranged from 18.8 mg/l for the DAE sample (MRD-08-346) to >100 mg/l for the RAE sample (MRD-08-375).

6. DISCUSSION

Company data on the ecotoxicity of the generic category of petroleum substances known as aromatic extracts have been generated since 1992 and were subsequently published by CONCAWE in 2001 [3]. These data provided the basis for the environmental hazard classifications for aromatic extracts recommended in 2005 [4]. More recently, CONCAWE embarked upon a test programme to generate typical acute and chronic data for several aromatic extracts with studies on daphnia and algae. All these data have been incorporated into this report.

When preparing a water accommodated fraction of a mixture which contains sparingly soluble components, two phases are present in the mixing system. Consequently, the individual components do not dissolve at their maximum water solubility, but equilibrate (partition) between the hydrocarbon and water phases. For this reason, the composition of the water phase varies for each component with the loading rate [23]. Petroleum products such as aromatic extracts will show toxicity at those loadings where the combined toxicities of the components in solution equal or exceed threshold levels.

Although in these tests, great care was taken to prevent volatilization losses during exposure, the mixing system, of necessity, had some headspace. It is important to standardise this aspect of test protocols, since for all hydrocarbon mixtures containing volatile components, the toxic constituents are likely to partition significantly to air. Accordingly, in conducting acute toxicity studies with volatile hydrocarbons, the headspace in the vessels should be kept as low as is practicable. Closed vessels were used for all of the toxicity studies.

In the **acute** studies of ten aromatic extract samples reported here, the ranges of acute results obtained for the three groups over the accepted periods that determine environmental classification, which are included in CONCAWE report 8/12 [36] were as follows:

<u>DAE (6 samples)</u>

fish (LL_{50}, 96h)	:	>1000 mg/l
Daphnia (EL_{50}, 48h)	:	35.9 - >1000 mg/l
algae (E_rL_{50}, 72h, specific growth rate)	:	18.8 - <100 mg/l

<u>TDAE (2 samples)</u>

fish (LL_{50}, 96h)	:	>1000 mg/l
Daphnia (EL_{50}, 48h)	:	>1000 mg/l
algae (E_rL_{50}, 72h, specific growth rate)	:	>100 mg/l

<u>RAE (2 samples)</u>

fish (LL_{50}, 96h)	:	>1000 mg/l
Daphnia (EL_{50}, 48h)	:	>1000 mg/l

RAE and TDAE on the basis of 2 samples each show no effects during acute exposures at 100 – 1000 mg/l to the three organisms (fish, Daphnia, algae). For DAE some effects to Daphnia and algae are observed in only 1 out of the 5 samples

tested; the other 4 DAE samples show no effects to these organisms at the levels tested (100 mg/l). As none of the samples have been tested on all three aquatic species, it is not possible to conclude whether any species is more sensitive than the other two.

Detailed compositional analysis using GCxGC of the six aromatic extracts has been carried out. A larger percentage of hydrocarbon components (i.e. < C_{30}) could be analysed by GCxGC in the DAE samples (~44% mean) compared to TDAE (~16%) or RAE (~13%) (**Table 3**). A more detailed analysis was undertaken of the composition of these six samples (**Table 4**) in comparison with their biomimetic (**Table 5**) and ecotoxicity data (**Table 6**). This confirmed that the one sample (light paraffinic distillate, CAS No. 64742-05-8, EMBSI code MRD-08-346) that had the only positive BE result (10.9 µmol 2,3-dimethylnaphthalene per ml PDMS) and subsequently classed as being 'harmful' to Daphnia and algae was also the one with the lowest carbon number range (all the material was shown by GCxGC to be <C_{30}) and was particularly high in aromatics (87 %wt), as well as a higher PAH content.

From the CONCAWE test programme, biomimetic extraction of WAFs has therefore proved to be a successful screening technique for differentiating between aromatic extracts samples with higher levels of water-soluble hydrocarbons (i.e. highest toxicity) and those with lowest levels or non-detectable amounts (i.e. no/low toxicity) [30].

Chronic 21 day daphnia reproduction studies of 5 aromatic extract samples are summarised for the three groups, confirming no chronic aquatic toxicity for any of the aromatic extract groups.

<u>DAE (1 sample)</u>		
Daphnia (EL_{50}, 21d)	:	>1000 mg/l
<u>TDAE (1 sample)</u>		
Daphnia (EL_{50}, 21d)	:	>1000 mg/l
<u>RAE (3 samples)</u>		
Daphnia (EL_{50}, 21d)	:	>100 - >1000 mg/l

7. GLOSSARY

ASTM	American Society for Testing of Materials
BE	Biomimetic Extraction
CAS no	Chemical Abstracts Service (Registry) Number
DAE	Untreated Distillate Aromatic Extract
EC	European Council
EINECS	European Inventory of Existing Commercial Chemical Substances
EL	Effective Loading
EL_{50}	Loading Rate of Test Substance (in dilution water) which causes adverse effects in 50% of the exposed population
E_rL_{50}	Loading Rate of Test Substance (in dilution water) which causes 50% reduction in algal growth rate
GC-FID	Gas Chromatography with Flame Ionisation Detection
GCxGC	Two-Dimensional Gas Chromatography
GESAMP	Group of Experts on the Scientific Aspects of Marine Pollution
GLP	Good Laboratory Practice
In	Cell density
LL	Lethal Loading
LL_{50}	Loading Rate of Test Substance (in dilution water) which causes lethal effects in 50% of the exposed population
MARPOL	Maritime Pollution
mg/l	Milligram per litre
ml	Millilitre
OECD	Organisation for Economic Co-operation and Development
PAC	Polycyclic Aromatic Compound
PAH	Polycyclic Aromatic Hydrocarbon

PDMS	Poly-dimethyl Siloxane
RAE	Residual Aromatic Extract
SAS	Statistical Analysis System
SPME	Solid Phase Micro-extraction
TDAE	Treated Distillate Aromatic Extract
UVCB	Substance of Unknown or Variable Composition, Complex Reaction Products or Biological Materials
WAF	Water Accommodated Fraction

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APPENDIX 1: UNTREATED DISTILLATE AROMATIC EXTRACTS

Untreated Distillate Aromatic Extracts Category Members			
CAS#	**EINECS #**	**Substance Name**	**Substance Description**
64742-03-6	265-102-1	Extracts (petroleum), light naphthenic distillate solvent	A complex combination of hydrocarbons obtained as the extract from a solvent extraction process. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C15 through C30. This stream is likely to contain 5 wt. % or more of 4- to 6-membered condensed ring aromatic hydrocarbons.
64742-04-7	265-103-7	Extracts (petroleum), heavy paraffinic distillate solvent	A complex combination of hydrocarbons obtained as the extract from a solvent extraction process. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C20 through C50. This stream is likely to contain 5 wt. % or more of 4- to 6-membered condensed ring aromatic hydrocarbons.
64742-05-8	265-104-2	Extracts (petroleum), light paraffinic distillate solvent	A complex combination of hydrocarbons obtained as the extract from a solvent extraction process. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C15 through C30. This stream is likely to contain 5 wt. % or more of 4- to 6-membered condensed ring aromatic hydrocarbons.
64742-11-6	265-111-0	Extracts (petroleum), heavy naphthenic distillate solvent	A complex combination of hydrocarbons obtained as the extract from a solvent extraction process. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C20 through C50. This stream is likely to contain 5 wt. % or more of 4- to 6-membered condensed ring aromatic hydrocarbons.
91995-78-7	295-341-7	Extracts (petroleum), light vacuum gas oil solvent.	A complex combination of hydrocarbons obtained by solvent extraction from light vacuum petroleum gas oil. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C13 through C30.
97722-04-8	307-753-7	Hydrocarbons, C26-55, arom. rich	A complex combination of hydrocarbons obtained by solvent extraction from a naphthenic distillate having a viscosity of 27cSt at 100°C (212°F). It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C26 through C55 and boiling in the range of approximately 395°C to 640°C (743°F to 1184°F).

APPENDIX 2: TREATED DISTILLATE AROMATIC EXTRACTS

Treated Distillate Aromatic Extracts Category Members			
CAS#	**EINECS #**	**Substance Name**	**Substance Description**
68783-00-6	272-175-3	Extracts (petroleum), heavy naphthenic distillate solvent, arom. conc.	An aromatic concentrate produced by adding water to heavy naphthenic distillate solvent extract and extraction solvent.
68783-04-0	272-180-0	Extracts (petroleum), solvent refined heavy paraffinic distillate solvent	A complex combination of hydrocarbons obtained as the extract from the re-extraction of solvent-refined heavy paraffinic distillate. It consists of saturated and aromatic hydrocarbons having carbon numbers predominantly in the range of C20 through C50.
68814-89-1	272-342-0	Extracts (petroleum), heavy paraffinic distillates, solvent deasphalted	A complex combination of hydrocarbons obtained as the extract from a solvent extraction of heavy paraffinic distillate.
90641-07-9	292-631-5	Extracts (petroleum), heavy naphthenic distillate solvent, hydrotreated	A complex combination of hydrocarbons obtained by treating a heavy naphthenic distillate solvent extract with hydrogen in the presence of a catalyst. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C20 through C50 and produces a finished oil of at least 19cSt at 40°C (100 SUS at 100°F).
90641-08-0	292-632-0	Extracts (petroleum), heavy paraffinic distillate solvent, hydrotreated	A complex combination of hydrocarbons produced by treating a heavy paraffinic distillate solvent extract with hydrogen in the presence of a catalyst. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C21 through C33 and boiling in the range of approximately 350°C to 480°C (662°F to 896°F).
90641-09-1	292-633-6	Extracts (petroleum), light paraffinic distillate solvent, hydrotreated	A complex combination of hydrocarbons produced by treating a light paraffinic distillate solvent extract with hydrogen in the presence of a catalyst. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C17 through C26 and boiling in the range of approximately 280° to 400°C (536°F to 752°F).
91995-73-2	295-335-4	Extracts (petroleum), hydrotreated light paraffinic distillate solvent	A complex combination of hydrocarbons obtained as the extract from solvent extraction of intermediate paraffinic top solvent distillate that is treated with hydrogen in the presence of a catalyst. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C16 through C36.
91995-75-4	295-338-0	Extracts (petroleum), light naphthenic distillate solvent, hydrodesulfurized	A complex combination of hydrocarbons obtained by treating the extract, obtained from a solvent extraction process, with hydrogen in the presence of a catalyst under conditions primarily to remove sulfur compounds. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C15 through C30. This stream is likely to contain 5 wt.% or more of 4- to 6-membered condensed ring aromatic hydrocarbons.

91995-76-5	295-339-6	Extracts (petroleum), light paraffinic distillate solvent, acid treated	A complex combination of hydrocarbons obtained as a fraction of the distillation of an extract from the solvent extraction of light paraffinic top petroleum distillates that is subjected to a sulfuric acid refining. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C16 through C32.
91995-77-6	295-340-1	Extracts (petroleum), light paraffinic distillate solvent, hydrodesulfurized	A complex combination of hydrocarbons obtained by solvent extraction of a light paraffin distillate and treated with hydrogen to convert the organic sulfur to hydrogen sulfide which is eliminated. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C15 through C40 and produces a finished oil with a viscosity of greater than 10cSt at 40°C.
91995-79-8	295-342-2	Extracts (petroleum), light vacuum gas oil solvent, hydrotreated	A complex combination of hydrocarbons, obtained by solvent extraction from light vacuum petroleum gas oils and treated with hydrogen in the presence of a catalyst. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C13 through C30.
92704-08-0	296-437-1	Extracts (petroleum), heavy paraffinic distillate solvent, clay-treated	A complex combination of hydrocarbons resulting from treatment of a petroleum fraction with natural or modified clay in either a contact or percolation process to remove the trace amounts of polar compounds and impurities present. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C20 through C50. This stream is likely to contain 5 wt. % or more 4-6 membered ring aromatic hydrocarbons.
93763-10-1	297-827-4	Extracts (petroleum), heavy naphthenic distillate solvent, hydrodesulfurized	A complex combination of hydrocarbons obtained from a petroleum stock by treating with hydrogen to convert organic sulfur to hydrogen sulfide which is removed. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C15 through C50 and produces a finished oil with a viscosity of greater than 19cSt at 40°C.
93763-11-2	297-829-5	Extracts (petroleum), solvent-dewaxed heavy paraffinic distillate	A complex combination of hydrocarbons obtained from a solvent dewaxed petroleum stock by treating with hydrogen to convert organic sulfur to hydrogen sulfide which is removed. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C15 through C50 and produces a finished oil with a viscosity of greater than 19cSt at 40°C.
100684-02-4	309-672-2	Extracts (petroleum), light paraffinic distillate solvent, carbon-treated	A complex combination of hydrocarbons obtained as a fraction from distillation of an extract recovered by solvent extraction of light paraffinic top petroleum distillate treated with activated charcoal to remove traces of polar constituents and impurities. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C16 through C32.
100684-03-5	309-673-8	Extracts (petroleum), light paraffinic distillate solvent, clay-treated	A complex combination of hydrocarbons obtained as a fraction from distillation of an extract recovered by solvent extraction of light paraffinic top petroleum distillates treated with bleaching earth to remove traces of polar constituents and impurities. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C16 through C32.

100684-04-6	309-674-3	Extracts (petroleum), light vacuum, gas oil solvent, carbon-treated	A complex combination of hydrocarbons obtained by solvent extraction of light vacuum petroleum gas oil treated with activated charcoal for the removal of trace polar constituents and impurities. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C13 through C30.
100684-05-7	309-675-9	Extracts (petroleum), light vacuum gas oil solvent, clay-treated	A complex combination of hydrocarbons obtained by solvent extraction of light vacuum petroleum gas oils treated with bleaching earth for removal of trace polar constituents and impurities. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly in the range of C13 through C30.

APPENDIX 3: RESIDUAL AROMATIC EXTRACTS

Residual Aromatic Extracts Category Members			
CAS#	**EINECS #**	**Substance Name**	**Substance Description**
64742-10-5	265-110-5	Extracts (petroleum), residual oil solvent	A complex combination of hydrocarbons obtained as the extract from a solvent extraction process. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly higher than C25.
91995-70-9	295-332-8	Extracts (petroleum), de-asphalted vacuum residue solvent	A complex combination of hydrocarbons obtained by solvent extraction of a vacuum-de-asphalted residue. It consists predominantly of aromatic hydrocarbons having carbon numbers predominantly greater than C30. This stream contains more than 5 wt. % of 4- to 6-membered condensed ring aromatic hydrocarbons.

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