concawe

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# the use of the dimethyl sulphoxide (DMSO) extract by the IP 346 method as an indicator of the carcinogenicity of lubricant base oils and distillate aromatic extracts

This report is based on a paper given on behalf of the CONCAWE Health Management Group to the Workshop on the Carcinogenicity of Coal and Petroleum Derived Substances held at the EC Joint Research Centre at Ispra, Italy, May 25-26, 1992.

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## ABSTRACT

Untreated lubricant base oils have been associated in the past with the development of human skin cancer. To give a better understanding of these health effects, industry has conducted an extensive range of long-term dermal carcinogenicity studies with the objective of identifying the influence of different types of refinery processing and to establish the important base oil compositional factors. The studies have led to improved refining techniques and to the development of simple markers for control purposes based on a standard analytical test.

However, with the increasing emphasis on the regulatory classification and labelling of petroleum products, it is proposed that the same markers can be effectively used for the classification of base oils.

The report describes the development of markers for the prediction of base oil carcinogenicity and examines the relative merits of two particular candidates, one based on dimethyl sulphoxide extraction by method IP 346 and the other based on benzo(a)pyrene (BaP) concentration.

## KEYWORDS

Animal, aromatic extract, base oil, benzo(a)pyrene, carcinogens, classification, dermal, DMSO extract, IP 346 test, ISPRA, labelling, lubricant base oils, marker, mice, PAC, RAE, skin, tumour.

## CONTENTS

# 1. INTRODUCTION

Untreated lubricant base oils have been associated in the past with the development of human skin cancer. To give a better understanding of these health effects, industry has conducted an extensive range of long-term dermal carcinogenicity studies with the objective of identifying the influence of different types of refinery processing and to establish the important base oil compositional factors. The studies have led to improved refining techniques and to the development of simple markers for control purposes based on a standard analytical test.

However, with the increasing emphasis on the regulatory classification and labelling of petroleum products, it is proposed that the same markers can be effectively used for the classification of base oils.

The report describes the development of markers for the prediction of base oil carcinogenicity and examines the relative merits of two particular candidates, one based on dimethyl sulphoxide extraction by method IP 346 and the other on benzo(a)pyrene (BaP) concentration.

Supporting experimental evidence for the study is a data base consisting of over one hundred skin painting studies conducted by CONCAWE member companies over a period of twenty years. This evidence is fully documented in the Appendix to the report.

The report is based on a paper given to the Workshop on the Carcinogenicity of Coal and Petroleum Derived Substances held at the EC Joint Research Centre at Ispra, Italy, May 25-26, 1992.

## 2. HEALTH ASPECTS OF MINERAL OILS AND DEVELOPMENT OF CONTROL MEASURES

Reports in the literature dating back to the early 1920s indicate that the use of poorly or unrefined mineral oils under conditions of poor personal hygiene has been associated with skin cancer in man. [1] The subject has been extensively reviewed in the literature and reference is made to the IARC study [2] and to the CONCAWE Dossier on Aromatic Extracts. [3]

Since the discovery that refinery processing can influence the carcinogenic potential of lubricants, the oil industry has conducted a wide range of studies to identify the factors involved. This has been one reason for the introduction of more severe refining techniques, such as solvent refining and hydrotreatment, and it is now possible to produce lubricating oils that are non-carcinogenic. As a result, older refining methods in Europe have been largely discontinued.

The standard procedure for assessing carcinogenic potential is by long-term animal skin painting studies. This involves applying test samples to the skin, several times a week for the major portion of the life span of the test animals. Such experiments may take two to three years to complete.

For industrial purposes, once the basic trends have been established, quicker alternative ways of assessing carcinogenic potential are advantageous for routine use. [4] There is also the need to minimize continual animal testing.

By the mid 1980s, a number of CONCAWE member companies had developed correlations between the results of animal studies and simple analytical parameters and were using such techniques for a variety of purposes. Recognising the importance of these developments, CONCAWE convened a multi-disciplinary study to review the subject. Member companies submitted data from their own records and altogether a data base of some 76 studies was established.

The conclusions of this work were reported to the EC in 1988. [5] CONCAWE recommended that, in the absence of any appropriate long-term animal test data, the carcinogenicity of lubricant base oils and distillate aromatic extracts should be assessed based on the dimethyl sulphoxide (DMSO) extract as determined by the IP 346 method. All substances with a DMSO extract of 3% (m/m) or greater should be considered as Class 2 carcinogens.

Subsequently, this proposal and the supporting experimental data have been reviewed by the TPC Working Group on the Classification and Labelling of Dangerous Substances, initially at the Working Group Meeting on 25-26 May 1992 held at the EC Joint Research Centre, Ispra, and later at following meetings in Brussels.

With the voluntary acceptance of this standard by industry in many parts of the world, the report describes the supporting evidence for the recommendations.

The **Appendix** includes all the information known to CONCAWE at the end of 1992, including the studies published after the Ispra Workshop in the paper by Chasey and McKee. [6]

# 3 PETROLEUM SUBSTANCES AND THE IP 346 METHOD

## 3.1 PETROLEUM SUBSTANCES

Most petroleum substances come into the category described in the European Inventory of Existing Commercial Chemical Substances (EINECS) as "substances of unknown or variable composition". In the petroleum industry, such substances are usually manufactured to meet specified physical and performance criteria, rather than a defined chemical composition. In consequence, the assessment of potential carcinogenicity can be difficult if based solely on the definitions given in EINECS.

## 3.2 LUBRICANT BASE OILS AND PAC CONTENT

It has been shown that the molecular species responsible for carcinogenic potential of lubricant base oils are principally the three to seven ring polycyclic aromatic compounds (PACs). [1] In the simplest case, the aromatic rings may consist of only hydrogen and carbon atoms and these compounds are termed polycyclic aromatic hydrocarbons (PAH). More complex structures may contain nitrogen, sulphur or oxygen atoms and as the number of rings increases, the possible steric arrangements increase rapidly. The molecules may also have side-chains of varying lengths, structures and complexity; such compounds are referred to as alkylated PACs.

It has been shown that polycyclic aromatic compounds can be selectively extracted from base oil streams by a dimethyl sulphoxide (DMSO) solvent. [7] The oil industry has therefore used this approach for a procedure to characterize the PAC content. A standard method for the extraction has been developed and is described in method IP 346. [8]

## 3.3 IP 346 METHOD

The IP 346 method is a gravimetric procedure in which a sample of oil is diluted with cyclohexane and extracted twice with DMSO. The sample is cut so as to exclude material boiling below 300°C. The resulting extract includes the three to seven ring polycyclic aromatic hydrocarbons in the test sample, but it is recognised that the method extracts other material as well. The resulting DMSO extract is therefore higher in percentage terms than the PAC content determined by a GLC analysis.

The IP 346 procedure is suitable for use with lubricant base oils and aromatic extracts from vacuum distillates, but it is not suitable for use with substances containing asphaltenes and/or resins such as some residual oils, residual fuels and bitumen. With such oils, the asphaltenic components prevent the separation of the DMSO extract.

In addition, the method is not suitable for use with used oils or formulated products containing additives. With these materials, the dimethyl sulphoxide may extract components from the additives as well as from the base oil, thus rendering the results inconclusive.

Experience has indicated that it is important to use a standardised method for determining the DMSO extract. The composition of the fraction obtained by DMSO extraction is dependent on the test procedure and different methods have been shown to give significantly different extract levels. For the correlations described later in this report, it is essential that only the IP 346 procedure is used.

## 3.4 RESIDUAL AROMATIC EXTRACTS

Typically, residual aromatic extracts have a boiling range from about 400°C to above 650°C. Because of the high boiling range, the molecules extracted by DMSO are likely to be highly alkylated or possess high molecular weight side chains and are therefore markedly different from those extracted from distillate aromatic extracts. There is little evidence to indicate that residual aromatic extracts are carcinogenic and, for this reason, it is not appropriate to base the classification on the DMSO extract. A high level of extract by IP 346 may not necessarily indicate potential carcinogenicity.

# 4. CARCINOGENICITY MARKERS

## 4.1 SUPPORTING EVIDENCE

At the time of the presentation to the EC Ispra Workshop in May 1992, the supporting data available for the evaluation of suitable markers consisted of some 76 skin painting studies. Most of this information was supplied to CONCAWE over the period 1981 to 1985 and came from individual studies conducted by member companies in the previous 15 to 20 years.

As these studies were conducted independently by member companies and did not form part of a co-ordinated programme, it was necessary to select appropriate samples from the information received that had the required manufacturing history, generic descriptions and analytical data. In particular, it was necessary to ensure that the DMSO extract had been determined by the IP 346 method, as other extraction procedures can give significantly different results.

The samples selected for the evaluation programme are listed in the Appendix. Table 1 gives a summary of the sample description, tumour incidence, DMSO extract and benzo(a)pyrene content, Table 2 lists details of test protocols and Table 3 lists the available analytical data on the test samples.

At the end of 1992, further information became available to CONCAWE with the publication of the paper by Chasey and McKee.[5] Some of the studies reported in this paper had already been included in the original CONCAWE data base, but others studies were new. The additional new data are listed in Table 4 of the Appendix.

The presentation to the Ispra Workshop in May 1992 was based on the information contained in Tables 1 to 3 of the Appendix. With the publication of the Chasey and McKee paper, the additional data listed in Table 4 has been used to confirm previous conclusions and to assess the accuracy with which potentially carcinogenic oils can be identified.

## 4.2 TUMOUR INCIDENCE CONSIDERED SIGNIFICANT.

For assessing the outcome of the dermal carcinogenicity studies, a sample has been considered as potentially carcinogenic if 4% or more of the test animals developed tumours. The 4% incidence rate was chosen as being above the typical background level for the untreated controls. For a typical study, this means that for a positive result, at least two of the 50 animals showed evidence of tumour formation.

Discussions in the TPC Classification and Labelling working group and with IARC have considered this 4% figure to be valid, although possibly severe.

## 4.3 INFLUENCE OF STRAIN OF MOUSE ON TUMOUR RESPONSE

Information on the test protocols used by member companies is listed in Table 2 of the Appendix. This shows that member companies have used two different strains of mouse, the C3H variety and the CF1 variety. It has been found that those studies conducted with the C3H mouse strain often gave a higher tumour response than those conducted with the CF1 mouse. This is illustrated in Figure 1 which shows tumour response against DMSO extract for each of the two strains. With the C3H mouse, tumour response rates in

excess of 70% can be obtained from samples with a DMSO extract in the 5 to 10% (m/m) range, whereas with the CF1 mouse, tumour response rates for similar samples are not in excess of 50%.

This finding adds a further degree of variability to the data base in addition to those of test procedure, test duration, dosage rates, etc. The marker levels discussed below have been developed on a worst case basis and the variability in the data base adds to the strength and applicability of the markers proposed.

## 4.4 USE OF DATA BASE FOR PREDICTING CARCINOGENIC POTENCY

The relationship between skin tumour formation and DMSO extract or BaP content are shown as scatter diagrams in **Figures 1** to **3**. These plots readily show the number of points above the pass/fail limit of a 4% tumour incidence rate and give a simple graphical basis for setting pass/fail limits for possible markers. However, because of the different protocols used in developing the data, such diagrams do not give a sound basis for developing mathematical relationships for the prediction of carcinogenic response (the number of animals expected to develop tumours on the basis of the measured DMSO extract).

## 4.5 DMSO EXTRACT AND BAP MARKERS

The relationship between tumour incidence and DMSO extract is shown in **Figure 2**. This scatter diagram illustrates that, with one exception, all of the 26 samples with a DMSO extract greater than 3% gave rise to significant skin tumour formation in animal studies. Only one sample with a DMSO extract of less than 3% was positive. This so-called "false negative" sample was CONCAWE Sample No. 90, a solvent-extracted oil.

The alternative marker studied was benzo(a)pyrene (BaP). BaP can be considered as representative of the polycyclic aromatic hydrocarbons found in lubricant base oils and it can be determined by a variety of techniques including gas chromatography and high-performance liquid chromatography. The BaP data in this paper have been determined by the method described by Grimmer [9]. IARC [10] have reported that there is sufficient evidence to indicate that BaP is carcinogenic in animal studies.

However, the scatter diagram in **Figure 3** shows that BaP content alone is not sufficient for accurately discriminating between potentially carcinogenic and non-carcinogenic lubricant base oils as there is no clear dividing line between potentially carcinogenic and non-carcinogenic oils; with BaP contents between 0.03 and 0.3 mg/kg, test results could either be positive or negative.

**Figure 4** shows that the correlation between DMSO extract and BaP content is poor.

## 4.6 CONFIRMATORY STUDIES

### 4.6.1 Using basestock blends

Six of the samples included in the above evaluations were blends of lubricating oil basestocks where long-term dermal carcinogenicity studies had been conducted on both the individual components and the blends.

Three of the blends, CONCAWE Samples 108, 109 and 110, were based on components that had given rise to a significant tumour response in the initial studies. These were then cut back with a non-carcinogenic white oil so as to give blends with calculated DMSO extract levels, two below 3% and one above 3%. Samples 108 and 109 with DMSO extracts below 3% did not give rise to any tumour formation on further testing, whereas Sample 110 with a DMSO extract of 6.3% resulted in 10% tumours.

CONCAWE Samples 106 and 107 were made up from constituents that had all previously tested negative and had DMSO extract levels of below 3%. Both of these blends tested negative.

The final blend, CONCAWE Sample 95, had a DMSO extract of 13% and tested positive.

These studies give further experimental support to the choice of 3% DMSO extract as the control level for the marker, although it is not proposed that the use of the marker should be extended to cover such preparations.

### 4.6.2 Additional data

The additional data made available on the publication of the paper by Chasey and McKee [5] gave the opportunity of further checking and confirming the recommendations put to the Ispra Workshop. Inclusion of the new data increased the number of studies available from 52 to 104. Figure 5 shows the new data superimposed on Figure 2.

On the basis of all the data available to CONCAWE at the end of 1992, the accuracy with which the DMSO marker can discriminate between potentially carcinogenic and non-carcinogenic base oils is quantified in Table 1. This shows predictability based on DMSO marker levels set at 1%, 2% and 3%.

If the marker level is set at 1%, the marker can predict the onset of tumorigenic activity, but it is accompanied by an unacceptably high level of false-positives (16%). At 2%, one false negative is given in 104 samples and at 3%, three false negatives are given.

In view of the comments concerning the severity of the tumour incidence level considered positive by CONCAWE and the fact that two of the three false negatives only gave rise to a tumour incidence of 5%, it is considered that a DMSO extract marker set at 3% gives a satisfactory limit for classification purposes.

# 5 CONCLUSIONS

CONCAWE considers that the use of a marker based on the dimethyl sulphoxide (DMSO) extract by method IP 346 offers a simple and effective system for the prediction of the carcinogenic potential for lubricant base oils and distillate aromatic extracts.

The scope of the IP 346 method does not allow the proposed marker to be applied to products containing asphaltenes, such as residual fuel oils, or to formulated products containing additives. Also, the marker is not applicable to gas oils or residual aromatic extracts.

The method by which the extract is determined must be IP 346, other analytical methods are likely to give different results and should not be used for classification purposes. The IP 346 procedure only requires equipment that is routinely available in a manufacturing location.

# 6 REFERENCES


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3. CONCAWE (1992) Aromatic Extracts. Product Dossier No 92/101. Brussels: CONCAWE

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7. Van der Wiel, A. (1993) Selective determination of potentially hazardous polycyclic aromatics in luboil fractions by the DMSO extraction method - As development, evaluation and use. Report AMER 93.023 (External version of internal KSLA report issued 1979). Shell Research B.V., Kon/Shell Lab. Amsterdam.

8. IP (1993) Determination of polycyclic aromatics in unused lubricating base oils and asphaltene free petroleum fractions - dimethyl sulphoxide extraction refractive index method. IP 346/92. In: Standard methods for analysis and testing of petroleum and related products Vol 2. Chichester: John Wiley and Sons

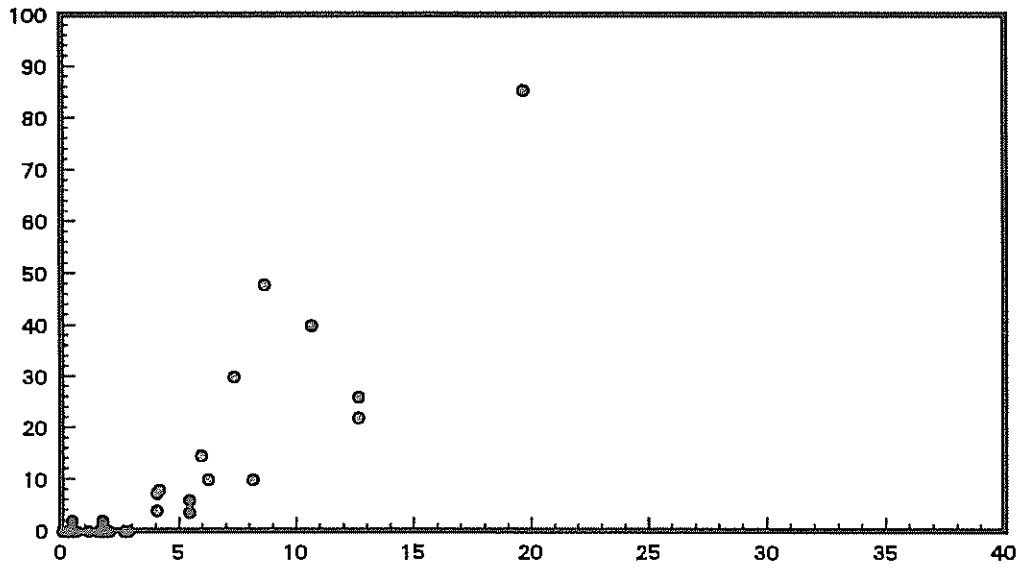
9. Grimmer, G. et al (1981) Profile of the polycyclic aromatic hydrocarbons from lubricating oils. Inventory by GC/MS-PAH in Environmental Materials, Part 1. *Fresenius Z Anal Chem 306, 347-355*

10. IARC (1983) Monographs on the evaluation of the carcinogenic risk of chemicals to humans. Polynuclear aromatic hydrocarbons, Part 1, Chemical, environmental and experimental data. Vol 32, 211-224. Lyon: International Agency for Research on Cancer

Table 1: Accuracy of prediction using DMSO extract marker

| Prediction | DMSO extract marker level (% m/m) | | |
|---|---|---|---|
| | 1% | 2% | 3% |
| Correct positive predictions | 37 | 36 | 34 |
| Correct negative predictions, | 51 | 57 | 64 |
| False positive predictions | 16 | 10 | 3 |
| False negative predictions | 0 | 1 | 3 |

% mice with tumours - CF1 mouse strain




% mice with tumours - C3H mouse strain

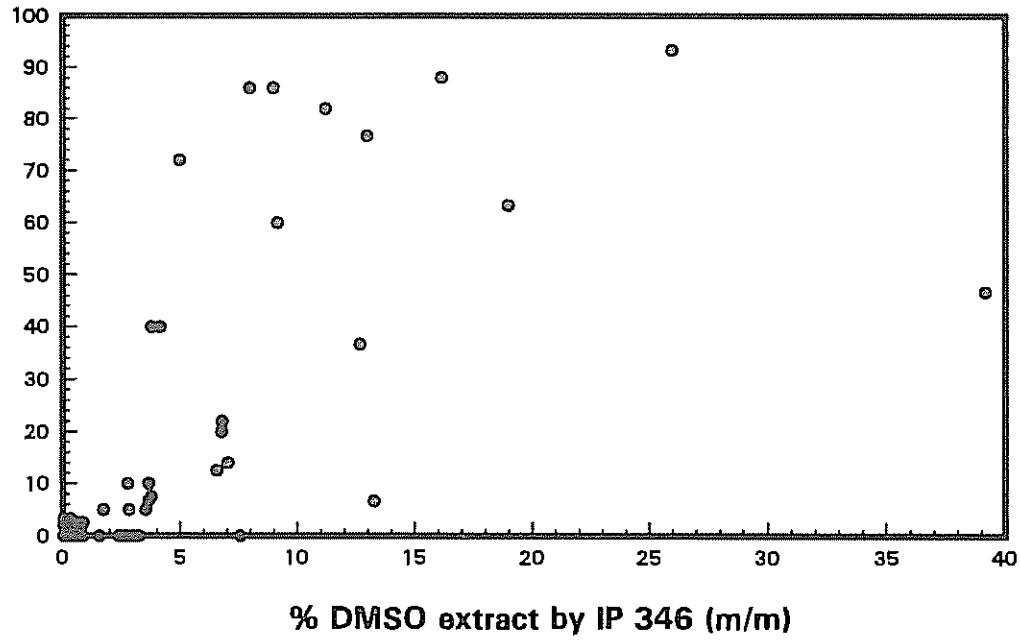



Figure 1: Influence of strain of mouse on tumour response

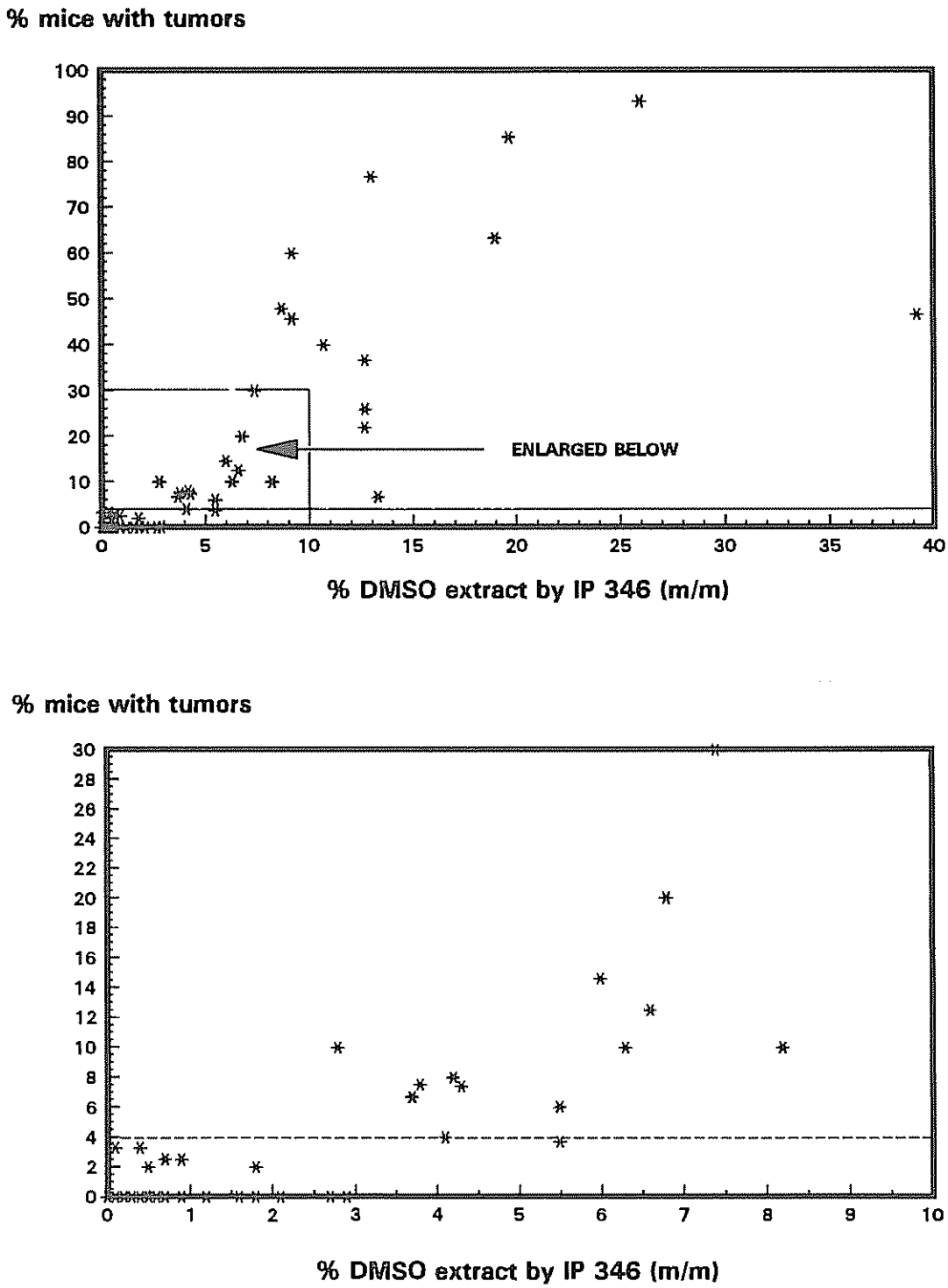
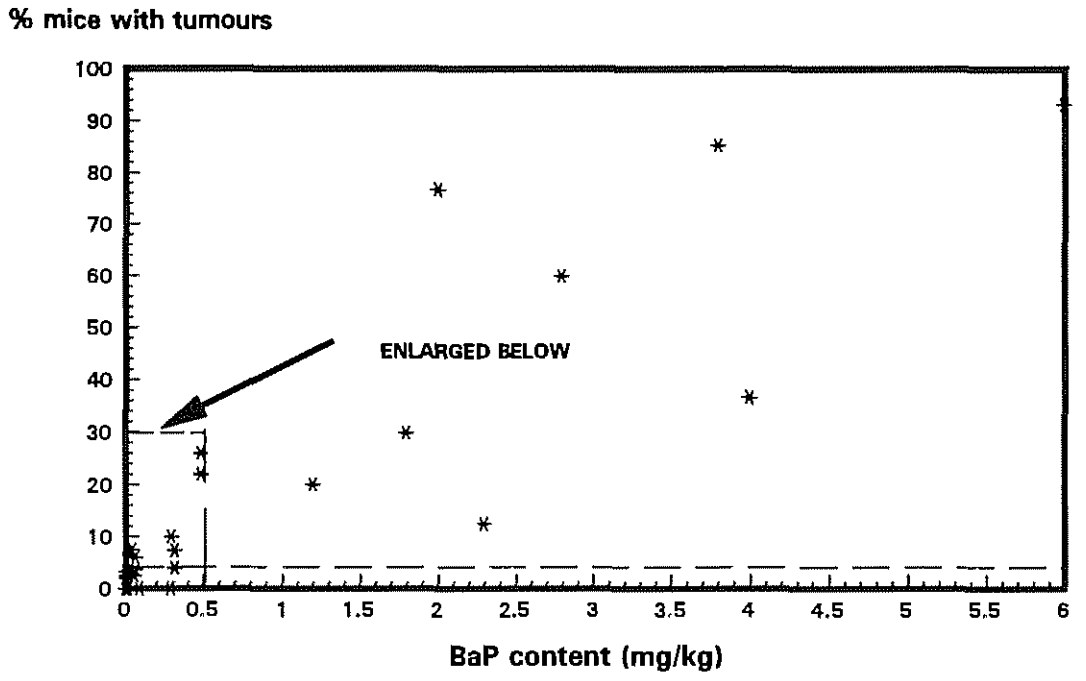



**Figure 2: Tumour formation as a function of DMSO extract by IP 346.**



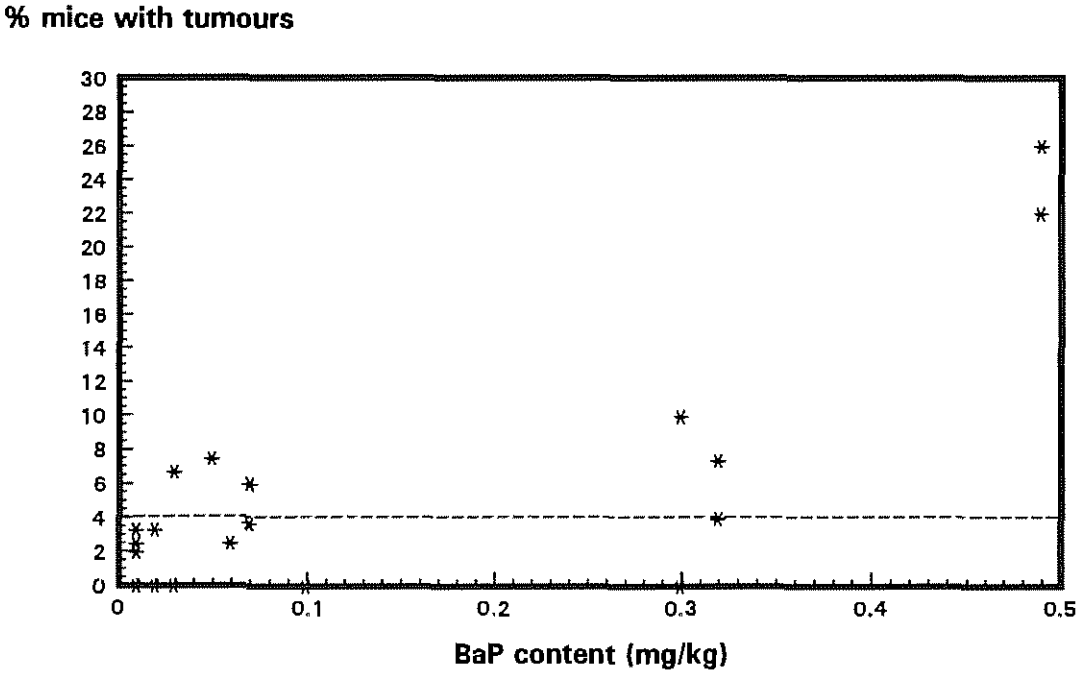




**Figure 3: Tumour formation as a function of BaP content**

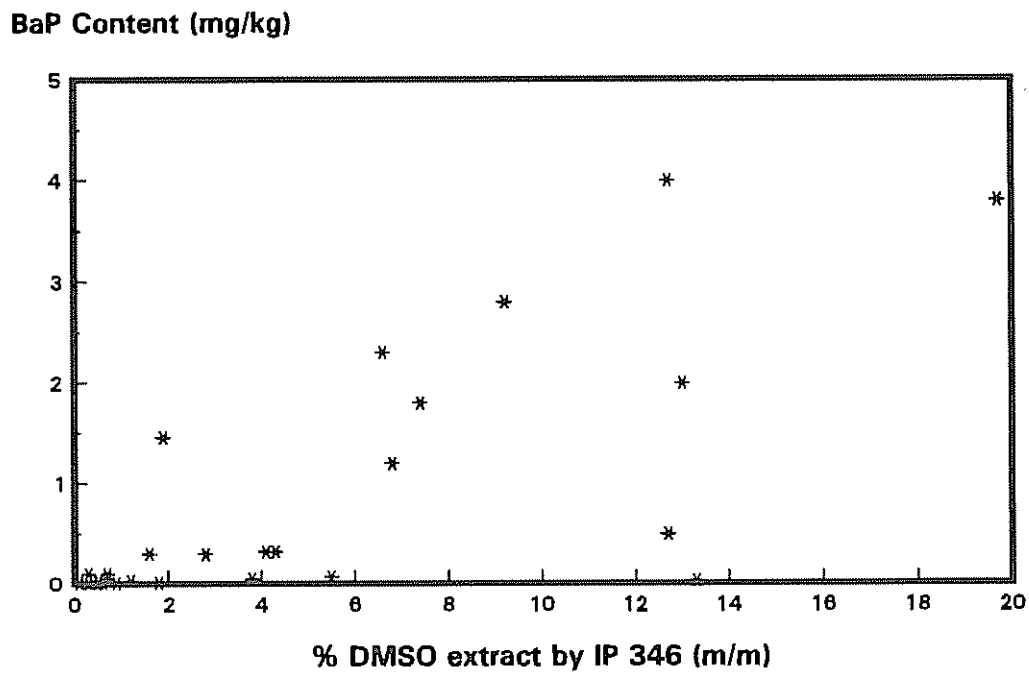
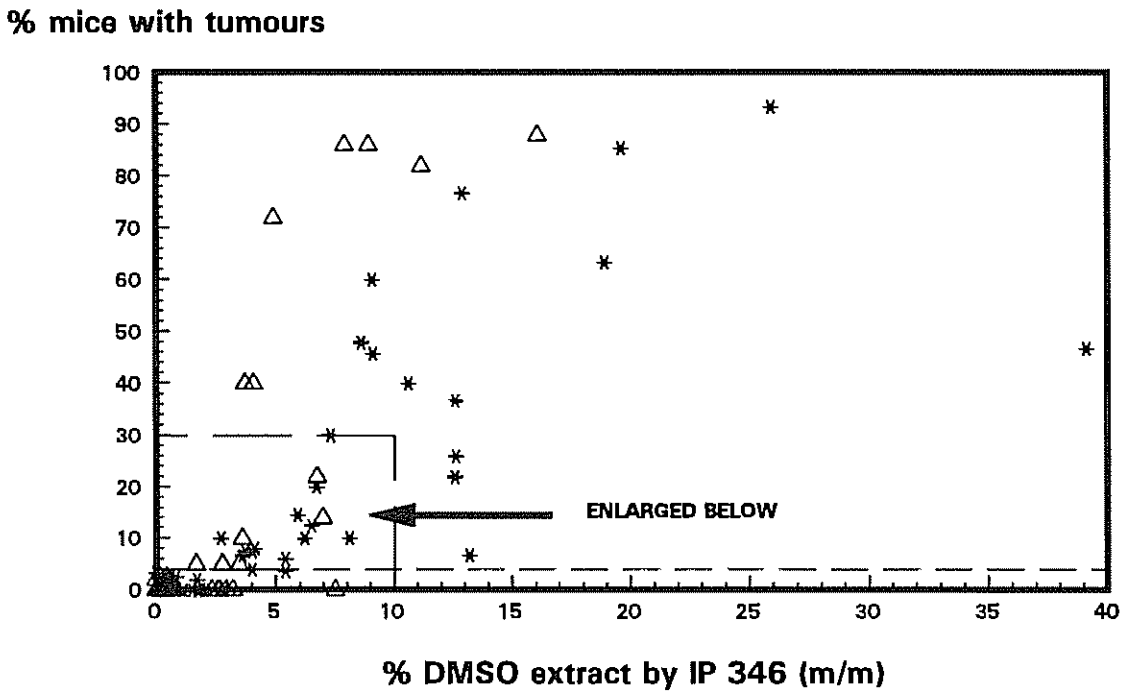



Figure 4: Relationship between DMSO extract and BaP Content



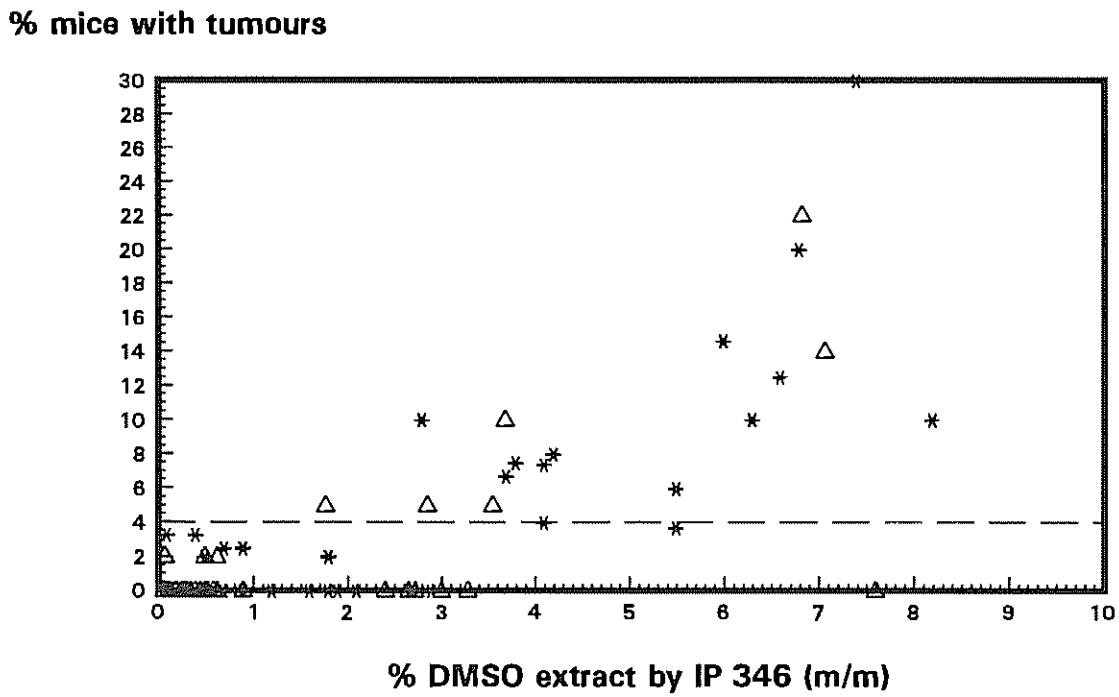




**Figure 5: Tumour formation as a function of DMSO extract: Addition of new data, February 1993.**

APPENDIX

SUPPORTING DATA FOR THE IP346 DMSO MARKER

The tables listed below summarize the data submitted by member companies to CONCAWE as part of its investigations into the relationship between the chemical composition of mineral oils and their ability to cause skin cancer in mice :

Table 1: Study data grouped according to refinery processing. The table includes relevant data received by CONCAWE up to 31/12/91.

Table 2: Summary of test protocols giving information concerning date of test, strain of mouse, dose, frequency of dosing and test duration for the studies listed in Table 1.

Table 3: Physical and chemical properties for the test, samples listed in Table 1.

Table 4: Study data received since 31/12/91 from the paper by Chasey and McKee.

The CONCAWE paper on the DMSO extract marker given in May 1992 to the Ispra Workshop on the Carcinogenicity Classification of Complex Petroleum Substances was based on the information given in Tables 1, 2 and 3 only.

*Table 1* Summary of CONCAWE mineral oil skin painting data

| CONCAWE No. | Generic Description | Crude type | DMSO Extract % m/m | BaP Content mg/kg | Tumours % |
|---|---|---|---|---|---|
| Vacuum distillates (raw untreated or dewaxed only) | | | | | |
| 93 | Vacuum distillate | Nephthenic | 6.6 | 2.30 | 12.5 |
| 62 | Dewaxed vacuum distillate | Paraffinic | 9.2 | 2.80 | 60.0 |
| Acid Treated Oils | | | | | |
| 37/71 | Acid/earth treated distillate (Acid refinded pale spindle) | Naphthenic | 12.7 | 0.49 | 22.0/26.0 |
| Solvent-extracted oils | | | | | |
| 46 | Dewaxed, solvent extracted vacuum distillate | Paraffinic | 0.6* | 0.01 | 0 |
| 58 | Dewaxed, solvent extracted vacuum distillate | Paraffinic | 0.5 | 0.02 | 0 |
| 8 | Furfural extracted, solvent dewexed | Paraffinic | 1.2 | 0.03 | 0 |
| 102 | Solvent extracted, vacuum distillate | Naphthenic | 1.6 | 0.30 | 0 |
| 19 | Solvent extracted, solvent dewaxed, vacuum distillate | Paraffinic | 1.6 | - | 0 |
| 50 | Solvent extracted vacuum distillate | Paraffinic | 1.9 | 0.20 | 0 |
| 90 | Dewaxed, solvent extracted vacuum distillate | Paraffinic | 2.8 | 0.30 | 10.0 |
| 78 | Dewaxed, solvent extracted vacuum distillate | - | 13.3 | 0.03 | 6.7 |
| Solvent-extracted, earth treated | | | | | |
| 98 | Liquid SO2 extracted, earth treated distillate (150 solvent pale) | Naphthenic | 0.5 | 0.01 | 2.0 |
| 100 | Liquid SO2 extracted, earth treated distillate (60 solvent pale) | Naphthenic | 0.6 | 0.01 | 0 |
| 79 | Furfural extracted, earth treated distillate (600 solvent pale) | Naphthenic | 0.7 | 0.03 | 0 |

continued....

*Table 1* Summery of CONCAWE mineral oil skin painting data

| CONCAWE No. | Generic Description | Crude type | DMSO Extract % m/m | BaP Content mg/kg | Tumours % |
|---|---|---|---|---|---|
| Solvent extracted end hydrogenated (including hydrofinished or terrofined) | | | | | |
| 28 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | Paraffinic | 0.1 | 0.01 | 3.3 |
| 61 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | Paraffinic | 0.2 | - | 0 |
| 69 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | Paraffinic | 0.2 | 0.02 | 0 |
| 68 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | Paraffinic | 0.3 | 0.10 | 0 |
| 103 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | Paraffinic | 0.4 | 0.02 | 3.3 |
| 81 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | Paraffinic | 0.7 | 0.06 | 2.5 |
| 9 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | Paraffinic | 0.9 | 0.01 | 0 |
| 57 | Solvent extracted and hydrogenated vacuum distillate | Naphthenic | 0.9 | 0.01 | 2.5 |
| 91/40 | Furfural extracted, ferrofined distillate (100 solvent neutral) | Paraffinic | 1.8 | 0.01 | 0/2.0 |
| 74 | Dewaxed solvent extracted and hydrogenated vacuum distillate | Paraffinic | 3.8 | 0.05 | 7.5 |
| 6/32 | Solvent (liquid SO2/C6H6) refined,ferrofined 100 solvent neutral | Paraffinic | 4.1 | 0.32 | 7.4/4.0 |
| Hydrogenated | | | | | |
| 104(e) | Hydrotreated distillate | Naphthenic | 2.9 | - | 0 |
| 12 | Severely hydrogenated vacuum distillate | Naphthenic | 3.7 | 0.18* | 6.7 |
| 67 | Hydrofinished distillate | Naphthenic | 4.2 | - | 8.0 |
| 63/43 | Hydrofinished distillate | Nephthenic | 5.5 | 0.07 | 3.7/6.0 |
| 70 | Hydrogenated vacuum distillate | Naphthenic | 6.8 | 1.20 | 20.0 |
| 87 | Dewaxed, hydrogenated vacuum distillate | Paraffinic | 7.4 | 1.80 | 30.0 |
| White oils | | | | | |
| 16 | Light medicinal white oil, phenol extracted, oleum treated, clay neutralised | Paraffinic | 0.3 | - | 0 |
| 89 | Technical white oil. Liquid SO2 extraction, hydrotreated distillate | Naphthenic | 0.4 | 0.01* | 0 |
| 10 (e) | Heevy medicinal white oil, solvent extracted, oleum treated, clay neutralised | Nephthenic | 0.1 | - | 0 |

continued....

*Table 1* Summary of CONCAWE mineral oil skin painting data

| CONCAWE No. | Generic Description | Crude type | DMSO Extract % m/m | BaP Content mg/kg | Tumours % |
|---|---|---|---|---|---|
| Distillate aromatic extracts - untreated | | | | | |
| 66 | Distillate aromatic extract from furfural extraction of LMO distillate | - | 19.7 | 3.80 | 85.4 |
| 11 | Raw solvent extract from vacuum distillate | Paraffinic | 26.0 | 6.00 | 93.3 |
| Distillate aromatic extracts - treated | | | | | |
| 27 | Hydrotreated MMO aromatic extract | - | 6.0 | - | 14.6 |
| 52 | Blend of hydrotreated extracts | - | 8.2 | - | 10.0 |
| 51 | Hydrotreated MMO aromatic extract | - | 8.7 | - | 47.9 |
| 86 | Hydrotreated aromatic extract | - | 9.2 | - | 45.8 |
| 80 | Blend of hydrotreated aromatic extracts | - | 10.7 | - | 40.0 |
| 5 | Hydrogenated solvent extract from vacuum distillate | Paraffinic | 12.7 | 4.00 | 36.7 |
| 24 | Hydrogenated solvent extract from vacuum distillate | Paraffinic | 19.0 | - | 63.3 |
| 33 | Hydrogenated solvent extract from vacuum distillate | Paraffinic | 39.2 | - | 46.7 |
| Residual Oils | | | | | |
| 45 | Solvent extracted residual oil | Paraffinic | 0.1 | <0.1 | 0 |
| 26 | Hydrogenated (mild), dewaxed, deasphalted atmospheric residium | Paraffinic | 0.3 | 0.01 | 0 |
| 105 | Deesphalted and dewaxed cylinder oil | Paraffinic | 2.7 | - | 0 |

continued....

*Table 1* Summery of CONCAWE mineral oil skin painting data

| CONCAWE No. | Generic Description | Crude type | DMSO Extract % m/m | BaP Content mg/kg | Tumours % |
|---|---|---|---|---|---|
| Blends: 1:1 (v/v) | | | | | |
| 106 | Solvent extracted, earth treated distillate, 60 solvent pale (100) + medicinal white oil (10) | Naphthenic | 0.3 | - | 0 |
| 107 | Solvent extracted, earth treated distillate, 150 solvent pale (98) + medicinal white oil (10) | Naphthenic | 0.3 | - | 0 |
| 108 (d) | Solvent extracted and hydrogenated, 100 solvent neutral (6) + medicinal white oil (10) | Mixed | 2.1 | - | 0 |
| 109 (c) | hydrofinished distillate (43) + medicinal white oil (10) | Naphthenic | 2.7 | - | 0 |
| 110 (b) | acid/earth treated distillate (37) + medicinal white oil (10) | Naphthenic | 6.3 | - | 10 |
| 95 | dewaxed solvent extracted and hydrogenated vacuum distillate (103) +raw solvent extract from vacuum distillate (11) | Paraffinic | 13.0 | 2.00 | 76.7 |

Note:
1) The letters in parenthesis after the sample number refer to the temporary codes used in the CONCAWE paper dated April 1992 presented to the Ispra Workshop

2) LMO denotes Light Machine Oil
MMO denotes Medium Machine Oil

First issued: 12-10-92
Revised: 16-02-93 New information and corrections marked (*)

*Table 2* **CONCAWE mineral oil skin painting data: Summary of test conditions**

| CONCAWE No. | Generic Description | Date (Start) | Animal Strain | Animal No | Dose µl | Frequency (times/week) | Duration (Note 1) | % Tumours 18 m | % Tumours 24 m |
|---|---|---|---|---|---|---|---|---|---|
| Vacuum distilletes (raw untreated or dewaxed only) | | | | | | | | | |
| 93 | Vacuum distillate | 1975 * | C3H | 40 | 25 * | 3 | LT * | 5.0 | 12.5 |
| 62 | Dewaxed vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 55.0 | 60.0 |
| Acid Treated Oils | | | | | | | | | |
| 37 } | Acid/earth treated distillate | 1971 | CF1 | 27 | 250 | Note 2 | 18 m | 22.0 | - |
| 71 } | | 1971 | CF1 | 50 | 250 | Note 3 | 18 m | 26.0 | - |
| Solvent-extracted oils | | | | | | | | | |
| 46 | Dewaxed, solvent extracted vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 0 | 5.0 |
| 58 | Dewaxed, solvent extracted vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 0 | 0 |
| 8 | Furfural extracted, solvent dewaxed | 1971 | CF1 | 50 | 250 | 1 | 18 m | 0 | - |
| 102 | Solvent extracted, vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 0 | 0 |
| 19 | Solvent extracted, solvent dewaxed, vacuum distillate | 1971 | CF1 | 50 | 250 | 2 | 18 m | 0 | - |
| 50 | Solvent extracted vacuum distillate | 1980 | CF1 | 48 | 200 | 2 | 18 m | 0 | - |
| 90 | Dewaxed, solvent extracted vacuum distillate | 1975* | C3H * | 40 | 25 * | 3 * | LT * | 7.5 | 10.0 |
| 78 | Dewaxed, solvent extracted vacuum distillate | 1977 | C3H | 30 | 25 | 3 | LT | 6.7 | 6.7 |
| Solvent-extracted, earth treated | | | | | | | | | |
| 98 | Liquid SO2 extracted, earth treated distillate (150 solvent pale) | 1971 | CF1 | 50 | 250 | 1 | 18 m | 2.0 at 15 m<br>0 at 18 m | |
| 100 | Liquid SO2 extracted, earth treated distillate (60 solvent pale) | 1971 | CF1 | 50 | 250 | 1 | 18 m | 0 | - |
| 79 | Furfural extracted, earth treated distillate (600 solvent pale) | 1971 | CF1 | 50 | 250 | 2 | 18 m | 0 | - |

continued ...

*Table 2* **CONCAWE mineral oil skin painting data: Summary of test conditions**

| CONCAWE No. | Generic Description | Date (Start) | Animal Strain | Animal No | Dose µl | Frequency (times/week) | Duration (Note 1) | % Tumours 18 m | % Tumours 24 m |
|---|---|---|---|---|---|---|---|---|---|
| Solvent extracted and hydrogenated (inc.hydrofinished or ferrofined) | | | | | | | | | |
| 28 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | 1977 | C3H | 30 | 25 | 3 | LT | 3.3 | 3.3 |
| 61 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | 1977 | C3H | 30 | 25 | 3 | LT | 0 | 0 |
| 69 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 0 | 0 |
| 68 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 0 | 0 |
| 103 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | 1977 | C3H | 30 | 25 | 3 | LT | 3.3 | 3.3 |
| 81 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 2.5 | 2.5 |
| 9 | Dewaxed, solvent extracted and hydrogenated vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 0 | 0 |
| 57 | Solvent extracted and hydrogenated vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 0 | 2.5 |
| 91 | Furfural extracted, ferrofined distillate (100 solvent neutral) | 1971 | CF1 | - | 250 | 1 | 18 m | 0 | - |
| 40 | | 1971 | CF1 | 50 | 250 | 2 | 18 m | 2.0 | - |
| 74 | Dewaxed solvent extracted and hydrogenated vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 0 | Note 5 |
| 6 | Solvent (liquid SO2/C6H6) refined,ferrofined 100 solvent neutral | 1971 | CF1 | 27 | 250 | Note 2 | 18 m | 7.4 | - |
| 32 | | 1971 | CF1 | 50 | 250 | Note 3 | 18 m | 4.0 | - |
| Hydrogenated | | | | | | | | | |
| 104(e) | Hydrotreated distillate | | CF1 * | - | | | | 0 | |
| 12 | Severely hydrogenated vacuum distillate | 1977 | C3H | 30 | 25 | 3 | LT | 6.7* | 6.7* |
| 67 | Hydrofinished distillate | 1977 | CF1 | 50 | 200 | 2 | 18 m | 8.0 | - |
| 63 | Hydrofinished distillate | 1971 | CF1 | 27 | 250 | Note 2 | 18 m | 3.7 | - |
| 43 | | 1971 | CF1 | 50 | 250 | Note 3 | 18 m | 6.0 | - |
| 70 | Hydrogenated vacuum distillate | 1976 | C3H | 40 | 25 | 3 | LT | 7.5 | 17.5(6)* |
| 87 | Dewaxed, hydrogenated vacuum distillate | | | | | | | 12.5 | 30.0 |
| White oils | | | | | | | | | |
| 16 | Light medicinal white oil, phenol extracted, oleum treated, clay neut | | CF1 | 50 | 250 | 1 | 18 m | 0 | - |
| 89 | Technical white oil. Liquid SO2 extraction, hydrotreated distillate | | CF1 | - | 250 | Note 3 | 18 m | 0 | - |
| 10(a) | Medicinal white oil | | CF1 | - | 250 | 2 | 18 m | 0 | - |

continued ...

*Table 2* **CONCAWE mineral oil skin painting data: Summary of test conditions**

| CONCAWE No. | Generic Description | Date (Start) | Animal Strain | Animal No | Dose µl | Frequency (times/week) | Duration (Note 1) | % Tumours 18 m | % Tumours 24 m |
|---|---|---|---|---|---|---|---|---|---|
| Distillate aromatic extracts - untreated | | | | | | | | | |
| 66 | Distillate aromatic extract from furfural extraction of LMO distillate | 1977 | CF1 | 48 | 200 | 2 | 17 m | 85.4 at 17 m | |
| 11 | Raw solvent extract from vacuum distillate | 1977 | C3H | 30 | 25 | 3 | LT | 93.3 | 93.3 |
| Distillate aromatic extracts - treated | | | | | | | | | |
| 27 | Hydrotreated MMO aromatic extract | 1977 | CF1 | 48 | 200 | 2 | 18 m | 14.6 | - |
| 52 | Blend of hydrotreated extracts | 1977 | CF1 | 50 | 200 | 2 | 18 m | 10.0 | - |
| 51 | Hydrotreated MMO aromatic extract | 1977 | CF1 | 48 | 200 | 2 | 18 m | 47.9 | - |
| 86 | Hydrotreated aromatic extract | | | | | | | | |
| 80 | Blend of hydrotreated aromatic extracts | 1977 | CF1 | 50 | 200 | 2 | 18 m | 40.0 | - |
| 5 | Hydrogenated solvent extract from vacuum distillate | 1977 | C3H | 30 | 25 | 3 | LT | 36.7 | 36.7 |
| 24 | Hydrogenated solvent extract from vacuum distilatte | 1977 | C3H | 30 | 25 | 3 | LT | 40.0 | 63.3 |
| 33 | Hydrogenated solvent extract from vacuum distillate | 1977 | C3H | 30 | 25 | 3 | LT | 20.0 | 46.7 |
| Residual Oils | | | | | | | | | |
| 45 | Solvent extracted residual oil | 1980 | CF1 | 48 | 200 | 2 | 18 m | 0 | - |
| 26 | Hydrogenated (mild), dewaxed, deasphalted etmospheric residium | 1976 | C3H | 40 | 25 | 3 | LT | 0 | 0 |
| 105 | Deasphalted and dewaxed cylinder oil | | | | | | 18 m | 0 | - |

continued ...

*Table 2* **CONCAWE mineral oil skin painting data: Summary of test conditions**

| CONCAWE No. | Generic Description | Date (Start) | Animal Strain | Animal No | Dose µl | Frequency (times/week) | Duration (Note 1) | % Tumours 18 m | % Tumours 24 m |
|---|---|---|---|---|---|---|---|---|---|
| Blends: 1:1 (v/v) | | | | | | | | | |
| 106 | Solvent extracted, earth treated distillate, 60 solvent pale (100) + medicinal white oil (10) | | CF1 | - | 250 | Note 4 | 18 m | 0 | - |
| 107 | Solvent extracted, earth treated distillate, 150 solvent pale (98) + medicinal white oil | | CF1 | - | 250 | 1 | 18 m | 0 | - |
| 108 | Solvent extracted and hydrogenated, 100 solvent neutral (6) + medicinal white oil (10) | | CF1 | - | 250 | Note 4 | 18 m | 0 | - |
| 109 | Hydrofinished distillate (43) + medicinal white oil (a) | | CF1 | - | 250 | Note 4 | 18 m | 0 | - |
| 110 | Acid/earth treated dististillate (37) + medicinal white oil (10) | | CF1 | - | 250 | Note 4 | 18 m | 10.0 | - |
| 95 | Dewaxed solvent extracted and hydrogenated vacuum distillate (103) + Raw solvent extract from vacuum distillate (11) | 1977 | C3H | 30 | 25 | 3 | LT | 76.7 | 76.7 |

Notes (1) Study duration: 18 m denotes 18 months, LT denotes lifetime study

(2) Frequency of dosing: twice per week for 22 weeks, none thereafter

(3) Frequency of dosing: once per week for 22 weeks, once every two weeks thereafter.

(4) Frequency of dosing: twice per week for 22 weeks, once per week thereafter

(5) Tumours: 2.5% at 24 months, 7.5% at 27 months

(6) Tumours variously quoted as 15%, 17.5% and 20% at 24 months

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Revision 1: 10-02-93 New information and corrections marked (*)
Revision 2: 13-05-93 Number of test animals in each study added.

*Table 3* Vacuum distillate (raw untreated or dewaxed only)

| | | 93 | 62 | |
|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 35.3 | 21.3 | |
| Relative density 15/15°C | | 0.977 | 0.927 | |
| K. Viscosity at 37.8°C | (mm2/s) | 11.34 | 51.1 | |
| K. Viscosity at 98.9°C | (mm2/s) | 2.0 | 6.11 | |
| Sulphur content | (% m/m) | 0.17 | 2.67 | |
| DMSO Extract: IP346 | (% m/m) | 6.56 | 9.18 | |
| DMSO Extract: Ref.Index/25°C | | 1.6154 | 1.6605 | |
| PCA Content | (mg/kg) | | | |
| Fluoranthene | | 2.5 | 4 | |
| Pyrene | | 10 | 4.8 | |
| Chrysene | | 19 | 95 | |
| Benzo(b)fluoranthene | | 15 | 12 | |
| Benzo(e)pyrene | | 13 | 44 | |
| Benzo(a)pyrene | | 2.3 | 2.8 | |
| Perylene | | 7.6 | 1 | |
| Benzo(g,h,i)perylene | | 12.7 | 0.7 | |
| 1-2 Benzofluorene | | 11 | 1 | |
| 2-3 Benzofluorene | | 11 | 1 | |
| Benzo(a)anthracene | | 0.8 | 3.5 | |
| Benzo(k)fluoranthene | | <2 | 0.5 | |
| Indeno(1,2,3)pyrene | | 4.3 | 0.3 | |
| Dibenzoanthracenes | | 0.5 | 0.7 | |
| C18H10S | | 11 | 140 | |

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Revised: 10-02-93 Revised format

*Table 3* **Acid Treated Oils**

| | | 37/71 | |
|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 22.9 | |
| Relative density 15/15°C | | 0.927 | |
| K. Viscosity at 37.8°C | (mm2/s) | - | |
| K. Viscosity at 98.9°C | (mm2/s) | - | |
| Sulphur content | (% m/m) | 1.9 | |
| DMSO Extract: IP346 | (% m/m) | 12.7* | |
| DMSO Extract: Ref.Index/25°C | | 1.616 | |
| PCA Content | (mg/kg) | | |
| Fluoranthene | | 23.2 | |
| Pyrene | | - | |
| Chrysene | | 85.9* | |
| Benzo(b)fluoranthene | | 2.71 | |
| Benzo(e)pyrene | | 2.33* | |
| Benzo(a)pyrene | | 0.49 | |
| Perylene | | - | |
| Benzo(g,h,i)perylene | | <0.01 | |
| 1-2 Benzofluorene | | - | |
| 2-3 Benzofluorene | | - | |
| Benzo(a)anthracene | | 8.19* | |
| Benzo(k)fluoranthene | | - | |
| Indeno(1,2,3)pyrene | | <0.01 | |
| Dibenzoanthracenes | | - | |
| C18H10S | | - | |

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*Table 3* **Solvent-extracted oils**

| | | 46 | 58 | 8 | 102 | 19 | 50 | 90 | 78 | |
|---|---|---|---|---|---|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 7.6 | 6.8 | 5.8 | 17.1 | 6.0 | 10.8 | 3.8 | 6.8 | |
| Relative density 15/15°C | | 0.869 | 0.861 | 0.862 | 0.930 | - | 0.905 | 0.896 | 0.863 | |
| K. Viscosity at 37.8°C | (mm2/s) | 30.35 | 14.58 | - | 253.5 | - | 89.21 @ 40C | 39.1 | 22.42 | |
| K. Viscosity at 98.9°C | (mm2/s) | 5.10 | 3.81 | - | 12.95 | - | 9.35 @ 100C | 5.60 | 4.20 | |
| Sulphur content | (% m/m) | 0.68 | 0.05 | 0.66 | 0.10 | - | 1.28 | 1.64 | 0.91 | |
| DMSO Extract: IP346 | (% m/m) | 0.59 | 0.45 | 1.2* | 1.62 | 1.6 | 1.9 | 2.82 | 13.3 | |
| DMSO Extract: Ref.Index/25°C | | 1.5924 | 1.5794 | 1.586 | 1.6075 | 1.558 | 1.613 | 1.6430 | 1.6174 | |
| PCA Content | (mg/kg) | | | | | | | | | |
| Fluoranthene | | 0.1 | 0.02 | 0.38 | 0.12 | - | <0.1 | 0.3 | 0.15 | |
| Pyrene | | 0.5 | 0.04 | - | 1.3 | - | 0.2 | 0.4 | 1.2 | |
| Chrysene | | 0.6 | 0.80 | 4.55* | 3.1 | - | 2.0 | 2.5 | 6 | |
| Benzo(b)fluoranthene | | 0.03 | 0.12 | 0.29 | 4.7 | - | - | 0.8 | 1.2 | |
| Benzo(e)pyrene | | 0.08 | 0.18 | 0.25* | 3.5 | - | 1.1 | 1.6 | 1.3 | |
| Benzo(a)pyrene | | 0.007 | 0.02 | 0.03 | 0.3 | - | 0.2 | 0.3 | 0.03* | |
| Perylene | | 0.009 | 0.02 | - | 0.2 | - | 0.3 | <0.04 | <0.09 | |
| Benzo(g,h,i)perylene | | 0.005 | ND | <0.01 | 2.0 | - | 0.3 | 0.1 | <0.04 | |
| 1-2 Benzofluorene | | 0.08 | 0.07 | - | 1.1 | - | - | 0.7 | 3.6 | |
| 2-3 Benzofluorene | | 0.04 | 0.13 | - | 1.7 | - | - | 0.3 | 2.0 | |
| Benzo(a)anthracene | | 0.02 | 0.03 | 0.20* | 0.1 | - | <0.1 | 0.5 | 0.3 | |
| Benzo(k)fluoranthene | | <0.002 | ND | - | - | - | - | <0.1 | 0.08 | |
| Indeno(1,2,3)pyrene | | 0.003 | ND | <0.01 | 0.7 | - | 0.2 | <0.2 | <0.06 | |
| Dibenzoanthracenes | | 0.009 | ND | - | 0.04 | - | 0.1 | <0.1 | <0.1 | |
| C18H10S | | 0.2 | 0.02 | - | 4.1 | - | - | 10 | 4.2 | |

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*Table 3* **Solvent-extracted, earth treated**

| | | 98 | 100 | 79 | |
|---|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 4.4 | 3.2 | 7.6 | |
| Relative density 15/15°C | | 0.884 | 0.868 | 0.906 | |
| K. Viscosity at 37.8°C | (mm2/s) | - | - | - | |
| K. Viscosity at 98.9°C | (mm2/s) | - | - | - | |
| Sulphur content | (% m/m) | 0.34 | 0.14 | 1.0 | |
| DMSO Extract: IP346 | (% m/m) | 0.5* | 0.6 | 0.7* | |
| DMSO Extract: Ref.Index/25°C | | 1.516 | 1.484 | 1.576 | |
| PCA Content | (mg/kg) | | | | |
| Fluoranthene | | 0.02 | 0.01 | 0.13 | |
| Pyrene | | - | - | - | |
| Chrysene | | 0.16* | 0.01* | 0.85* | |
| Benzo(b)fluoranthene | | 0.02 | <0.01 | 0.15 | |
| Benzo(e)pyrene | | 0.02* | 0.01* | 150.0* | |
| Benzo(a)pyrene | | <0.01 | 0.01* | 0.03 | |
| Perylene | | - | - | - | |
| Benzo(g,h,i)perylene | | 0.07 | <0.01 | 0.06 | |
| 1-2 Benzofluorene | | - | - | - | |
| 2-3 Benzofluorene | | - | - | - | |
| Benzo(a)anthracene | | 0.02* | 0.01* | 0.11* | |
| Benzo(k)fluoranthene | | - | - | - | |
| Indeno(1,2,3)pyrene | | <0.01 | - | - | |
| Dibenzoanthracenes | | - | - | 0.01 | |
| C18H10S | | - | - | - | |

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*Table 3* **Solvent extracted and hydrogenated**

| | | 28 | 61 | 69 | 68 | 103 | 81 | 9 | 57 | 91/40 |
|---|---|---|---|---|---|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 9.2 | 2.6 | 10.5 | 9.6 | 9.0 | 10.7 | 10.4 | 8.4 | 7.4 |
| Relative density 15/15°C | | 0.899 | 0.850 | 0.889 | 0.868 | 0.870 | 0.871 | 0.863 | 0.881 | 0.873 |
| K. Viscosity at 37.8°C | (mm2/s) | 128 | 18.12 | 124 | 29.45 | 33.21 | 33.1 | 20.47 | 20.37 | - |
| K. Viscosity at 98.9°C | (mm2/s) | 12.46 | 3.89 | 12.07 | 4.95 | 5.38 | 5.37 | 4.02 | 3.71 | - |
| Sulphur content | (% m/m) | 0.49 | 12.0 | 0.54 | 0.09 | 0.30 | 0.42 | 0.17 | 0.03 | 1.20 |
| DMSO Extract: IP346 | (% m/m) | 0.09 | 0.17 | 0.18 | 0.29 | 0.4* | 0.71 | 0.87 | 0.92 | 1.8* |
| DMSO Extract: Ref.Index/25°C | | 1.5402 | 1.4972 | 1.5844 | 1.5686 | 1.5638 | 1.5964 | 1.5536 | 1.5542 | 1.585 |
| PCA Content | (mg/kg) | | | | | | | | | |
| Fluoranthene | | 0.01 | · | 0.04 | 0.1 | 0.06 | 0.56 | 0.30 | 0.04 | 0.13 |
| Pyrene | | 0.01 | · | 0.07 | 0.08 | 0.06 | 0.62 | 0.54 | 0.10 | - |
| Chrysene | | 0.08 | - | 0.26 | 0.14 | 0.16 | 11.2 | 0.30 | 0.07 | 0.44* |
| Benzo(b)fluoranthene | | 0.04 | - | 0.06 | 0.1* | 0.04 | 0.09 | 0.01* | 0.02 | 0.08 |
| Benzo(e)pyrene | | 0.05 | - | 0.13 | 0.1* | 0.06 | 0.14 | 0.01* | 0.01 | 0.07* |
| Benzo(a)pyrene | | 0.01* | · | 0.02 | 0.1* | 0.02 | 0.06 | 0.01* | 0.01 | 0.01 |
| Perylene | | ND | · | 0.01 | <0.1 | 0.02 | 0.04 | (1) | 0.01 | - |
| Benzo(g,h,i)perylene | | ND | - | ND | 0.8 | ND | 0.02 | (1) | ND | <0.01 |
| 1-2 Benzofluorene | | 0.01 | - | 0.03 | 0.08 | 0.06 | 0.60 | 0.30 | 0.20 | - |
| 2-3 Benzofluorene | | 0.03 | - | 0.04 | 0.12 | 0.09 | 0.09 | 0.60 | 0.62 | - |
| Benzo(a)anthracene | | 0.02 | · | 0.03 | <0.01 | 0.01 | 0.02 | 0.01 | 0.01 | 0.01* |
| Benzo(k)fluoranthene | | ND | · | ND | <0.2 | ND | ND | (1) | 0.01 | - |
| Indeno(1,2,3)pyrene | | ND | - | ND | <0.6 | ND | 0.06 | (1) | ND | <0.01 |
| Dibenzoanthracenes | | - | · | ND | <0.2 | ND | 0.03 | (1) | ND | - |
| C18H10S | | - | - | 0.16 | <0.08 | 0.22 | 0.24 | (2) | 0.01 | - |

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*Table 3* **Solvent extracted and hydrogeneted (continued)**

| | | 74 | 32/6 | |
|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 13.8 | 10.3 | |
| Relative density 15/15°C | | 0.848 | 0.882 | |
| K. Viscosity at 37.8°C | (mm2/s) | 4.48 | - | |
| K. Viscosity at 98.9°C | (mm2/s) | 1.54 | - | |
| Sulphur content | (% m/m) | 0.06 | 1.6 | |
| DMSO Extract: IP346 | (% m/m) | 3.85 | 4.1 | |
| DMSO Extract: Ref.Index/25°C | | 1.579 | 1.618 | |
| PCA Content | (mg/kg) | | | |
| Fluoranthene | | 0.9 | 0.94 | |
| Pyrene | | 0.8 | - | |
| Chrysene | | 0.05* | 18.0* | |
| Benzo(b)fluoranthene | | 0.02* | 1.24 | |
| Benzo(e)pyrene | | 0.05 | 0.85* | |
| Benzo(a)pyrene | | <0.1 | 0.32 | |
| Perylene | | <0.05 | - | |
| Benzo(g,h,i)perylene | | <0.1 | <0.01 | |
| 1-2 Benzofluorene | | 0.24 | - | |
| 2-3 Benzofluorene | | 0.08 | - | |
| Benzo(e)anthracene | | 0.26* | 0.56* | |
| Benzo(k)fluoranthene | | <0.1 | - | |
| Indeno(1,2,3)pyrene | | <0.3 | <0.01 | |
| Dibenzoanthracenes | | <0.1 | - | |
| C18H10S | | <0.1 | - | |

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*Table 3* **Hydrogenated**

| | | 104 | 12 | 67 | 63/43 | 70 | 87 | |
|---|---|---|---|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | | 16.5 | 14 | 19.3 | 37.6 | 22.2 | |
| Relative density 15/15°C | | | 0.887 | - | 0.909 | 0.978 | 0.915 | |
| K. Viscosity at 37.8°C | (mm2/s) | | 22.13 | - | - | 916 | 43.2 | |
| K. Viscosity at 98.9°C | (mm2/s) | | 3.78 | - | - | 17.95 | 5.69 | |
| Sulphur content | (% m/m) | | 0.13 | - | 0.63 | 0.19 | 1.71 | |
| DMSO Extract: IP346 | (% m/m) | | 3.69 | 4.2 | 5.3 | 6.82 | 7.39 | |
| DMSO Extract: Ref.Index/25°C | | | 1.6006 | 1.598 | 1.603 | 1.6196 | 1.645 | |
| PCA Content | (mg/kg) | | 2.78 | - | 1.89 | 2.7 | 4 | |
| Pyrene | | | 15 | - | - | 17.6 | 4 | |
| Chrysene | | | 12.72 | - | 13.6* | 29 | 43 | |
| Benzo(b)fluoranthene | | | 0.58 | - | 1.1 | 33 | 10 | |
| Benzo(e)pyrene | | | 0.90 | - | 3.54* | 18 | 21 | |
| Benzo(a)pyrene | | | 0.18 | - | 0.07 | 1.2 | 1.8 | |
| Perylene | | | 0.12 | - | - | 1 | <0.9 | |
| Benzo(g,h,i)perylene | | | 0.22 | - | 0.9 | 13 | <1 | |
| 1-2 Benzofluorene | | | 32.03 | - | - | 19 | 8 | |
| 2-3 Benzofluorene | | | 0.22 | - | - | 21 | 4 | |
| Benzo(a)anthracene | | | 0.8 | - | 0.21* | 1.1 | 0.20* | |
| Benzo(k)fluoranthene | | | ND | - | | 1.2 | <2 | |
| Indeno(1,2,3)pyrene | | | 0.53 | - | <0.01 | 5 | <2 | |
| Dibenzoanthracenes | | | 0.22 | - | - | 0.2 | <1 | |
| C18H10S | | | 1.04 | - | - | 26 | 65 | |

First issued: 12-10-92
Revised: 10-02-93 Revisions marked (*)

*Table 3* White oils

| | | 16 | 89 | 10 | |
|---|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 0 | 3.5 | | |
| Relative density 15/15°C | | 0.853 | 0.872 | | |
| K. Viscosity at 37.8°C | (mm2/s) | - | - | | |
| K. Viscosity at 98.9°C | (mm2/s) | - | - | | |
| Sulphur content | (% m/m) | 0.008 | 0.05 | | |
| DMSO Extract: IP346 | (% m/m) | 0.4 | 0.5 | | |
| DMSO Extract: Ref.Index/25°C | | 1.490 | 1.500 | | |
| PCA Content | (mg/kg) | | | | |
| Fluoranthene | | 0.02 | 0.01 | | |
| Pyrene | | - | - | | |
| Chrysene | | - | - | | |
| Benzo(b)fluoranthene | | - | 0.01 | | |
| Benzo(e)pyrene | | 0.02 | - | | |
| Benzo(a)pyrene | | - | <0.01 | | |
| Perylene | | <0.01 | - | | |
| Benzo(g,h,i)perylene | | - | <0.01 | | |
| 1-2 Benzofluorene | | <0.01 | - | | |
| 2-3 Benzofluorene | | - | - | | |
| Benzo(a)anthracene | | - | - | | |
| Benzo(k)fluoranthene | | - | - | | |
| Indeno(1,2,3)pyrene | | <0.01 | <0.01 | | |
| Dibenzoanthracenes | | - | - | | |
| C18H10S | | - | - | | |

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*Table 3* Distillate aromatic extracts - untreated

| | | 66 | 11 | |
|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 33 | 39.8 | |
| Relative density 15/15°C | | - | 1.005 | |
| K. Viscosity at 37.8°C | (mm2/s) | - | 175.3 | |
| K. Viscosity at 98.9°C | (mm2/s) | - | 9.55 | |
| Sulphur content | (% m/m) | - | 4.92 | |
| DMSO Extract: IP346 | (% m/m) | 19.7 | 26.0 | |
| DMSO Extract: Ref.Index/25°C | | 1.696 | 1.6510 | |
| PCA Content | (mg/kg) | | | |
| Fluoranthene | | - | 0.4 | |
| Pyrene | | - | 6 | |
| Chrysene | | - | 122 | |
| Benzo(b)fluoranthene | | - | 53 | |
| Benzo(e)pyrene | | - | 58 | |
| Benzo(e)pyrene | | - | 6 | |
| Perylene | | - | 2 | |
| Benzo(g,h,i)perylene | | - | 3 | |
| 1-2 Benzofluorene | | - | 16 | |
| 2-3 Benzofluorene | | - | 8 | |
| Benzo(a)anthracene | | - | 4.2 | |
| Benzo(k)fluoranthene | | - | 2.7 | |
| Indeno(1,2,3)pyrene | | - | ND | |
| Dibenzoanthracenes | | - | 4 | |
| C18H10S | | - | 250 | |

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*Table 3* **Distillate aromatic extracts - treated**

| | | 27 | 52 | 51 | 86 | 80 | 5 | 24 | 33 | |
|---|---|---|---|---|---|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 22 | 24 | 26 | 25 | 23 | 41.0 | >60 | >60 | |
| Relative density 15/15°C | | | - | - | - | - | 0.937 | 1.033 | 1.024 | |
| K. Viscosity at 37.8°C | (mm2/s) | | - | - | - | - | 23.15 | 17.19 | 15.21 | |
| K. Viscosity at 98.9°C | (mm2/s) | | - | - | - | - | 3.72 | 2.81 | 2.73 | |
| Sulphur content | (% m/m) | | - | - | - | - | 1.67 | 6.84 | 6.0 | |
| DMSO Extract: IP346 | (% m/m) | 6.0 | 8.2 | 8.7* | 9.2 | 10.7 | 12.75 | 19.0* | 39.2 | |
| DMSO Extract: Ref.Index/25°C | | 1.606 | 1.584 | 1.635 | 1.634 | 1.601 | 1.6316 | 1.6362 | 1.628 | |
| PCA Content | (mg/kg) | | | | | | | | | |
| Fluoranthene | | - | - | - | - | - | 5 | - | - | |
| Pyrene | | - | - | - | - | - | 25 | - | - | |
| Chrysene | | - | - | - | - | - | 96 | - | - | |
| Benzo(b)fluoranthene | | - | - | - | - | - | 12 | - | - | |
| Benzo(e)pyrene | | - | - | - | - | - | 10 | - | - | |
| Benzo(a)pyrene | | - | - | - | - | - | 4 | - | - | |
| Perylene | | - | - | - | - | - | 3 | - | - | |
| Benzo(g,h,i)perylene | | - | - | - | - | - | 1 | - | - | |
| 1-2 Benzofluorene | | - | - | - | - | - | 23 | - | - | |
| 2-3 Benzofluorene | | - | - | - | - | - | 26 | - | - | |
| Benzo(a)anthracene | | - | - | - | - | - | 3 | - | - | |
| Benzo(k)fluoranthene | | - | - | - | - | - | ND | - | - | |
| Indeno(1,2,3)pyrene | | - | - | - | - | - | 3 | - | - | |
| Dibenzoanthracenes | | ND | - | - | - | - | 0.5 | - | - | |
| C18H10S | | 0.04 | - | - | - | - | 14 | - | - | |

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*Table 3* **Residual Oils**

| | | 45 | 26 | 105 | |
|---|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | 5.6 | 10.7 | | |
| Relative density 15/15°C | | 0.896 | 0.903 | | |
| K. Viscosity at 37.8°C | (mm2/s) | 505.0 @ 40C | - | | |
| K. Viscosity at 98.9°C | (mm2/s) | 32.6 @ 100C | 32.85 | | |
| Sulphur content | (% m/m) | 0.7 | 0.69 | | |
| DMSO Extract: IP346 | (% m/m) | 0.1 | 0.31 | | |
| DMSO Extract: Ref.Index/25°C | | - | 1.5384 | | |
| PCA Content | (mg/kg) | | | | |
| Fluoranthene | | <0.1 | 0.06 | | |
| Pyrene | | <0.1 | 0.20 | | |
| Chrysene | | <0.1 | 0.70 | | |
| Benzo(b)fluoranthene | | <0.1 | 0.70 | | |
| Benzo(e)pyrene | | <0.1 | 0.70 | | |
| Benzo(a)pyrene | | <0.1 | 0.01* | | |
| Perylene | | <0.1 | <0.05 | | |
| Benzo(g,h,i)perylene | | <0.1 | <0.30 | | |
| 1-2 Benzofluorene | | - | 0.16 | | |
| 2-3 Benzofluorene | | - | 0.25 | | |
| Benzo(a)anthracene | | <0.1 | 0.01* | | |
| Benzo(k)fluoranthene | | - | <0.10 | | |
| Indeno(1,2,3)pyrene | | <0.1 | <1 | | |
| Dibenzoanthracenes | | <0.1 | <1 | | |
| C18H10S | | <0.1 | 2 | | |

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*Table 3* **Blends**

| | | 108 | 109 | 110 | |
|---|---|---|---|---|---|
| Aromatic Carbon (Brandes) | (%) | | | | |
| Relative density 15/15°C | | | | | |
| K. Viscosity at 37.8°C | (mm2/s) | | | | |
| K. Viscosity at 98.9°C | (mm2/s) | | | | |
| Sulphur content | (% m/m) | | | | |
| DMSO Extract: IP346 | (% m/m) | | | | |
| DMSO Extract: Ref.Index/25°C | | | | | |
| PCA Content | (mg/kg) | | | | |
| Fluoranthene | | | | | |
| Pyrene | | | | | |
| Chrysene | | | | | |
| Benzo(b)fluoranthene | | | | | |
| Benzo(e)pyrene | | | | | |
| Benzo(a)pyrene | | | | | |
| Perylene | | | | | |
| Benzo(g,h,i)perylene | | | | | |
| 1-2 Benzofluorene | | | | | |
| 2-3 Benzofluorene | | | | | |
| Benzo(a)anthracene | | | | | |
| Benzo(k)fluoranthene | | | | | |
| Indeno(1,2,3)pyrene | | | | | |
| Dibenzoanthracenes | | | | | |
| C18H10S | | | | | |

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*Table 4:* Summary of CONCAWE mineral oil skin painting data: Additional study data received since 31/12/91

| Chasey & McKee Sample No. | Tumour response Animals | %T | DMSO extract IP 346 % m/m | BaP Content mg/kg | Year of study | Crude type |
|---|---|---|---|---|---|---|
| **Vacuum distillates - raw untreated** | | | | | | |
| 2 | 36/50 | 72.0 | 5.02 | 1.2 | 1979 | P |
| 3 | 11/50 | 22.0 | 6.83 | - | 1981 | P |
| **Solvent extracted distillates** | | | | | | |
| 25 | 0/40 | 0 | 0.59 | <0.01 | 1975 | P |
| 27 | 0/50 | 0 | 0.19 | - | 1979 | P |
| 28 | 0/50 | 0 | 0.53 | <0.01 | 1979 | P |
| 29 | 0/50 | 0 | 0.12 | <0.01 | 1979 | P |
| 30 | 0/50 | 0 | 0.35 | <0.01 | 1979 | P |
| 31 | 0/50 | 0 | 0.25 | <0.01 | 1979 | P |
| 32 | 0/50 | 0 | 0.45 | <0.01 | 1979 | P |
| 33 | 0/50 | 0 | 0.18 | <0.01 | 1979 | P |
| 34 | 1/50 | 2.0 | 0.63 | - | 1981 | P |
| 35 | 0/50 | 0 | 0.27 | - | 1981 | P |
| 36 | 0/50 | 0 | 7.60 | <0.01 | 1981 | P |
| 37 | 0/50 | 0 | 0.27 | - | 1981 | P |
| 38 | 0/50 | 0 | 0.13 | - | 1981 | P |
| 39 | 0/50 | 0 | 0.53 | <0.01 | 1981 | P |
| 40 | 0/50 | 0 | 0.52 | <0.01 | 1981 | P |
| **Solvent extracted and hydrotreated distillates** | | | | | | |
| 51 | 0/50 | 0 | 0.13 | <0.01 | 1979 | P |
| 53 | 1/50 | 2.0 | 0.50 | <0.01 | 1979 | P |
| 54 | 0/50 | 0 | 0.07 | <0.01 | 1980 | P |
| 55 | 0/50 | 0 | 0.20 | <0.01 | 1979 | P |
| 56 | 0/50 | 0 | 0.29 | <0.01 | 1979 | P |
| 57 | 0/50 | 0 | 0.63 | <0.01 | 1979 | P |
| 58 | 0/50 | 0 | 0.13 | <0.02 | 1979 | P |
| 59 | 0/50 | 0 | 0.90 | <0.02 | 1979 | P |
| 60 | 0/50 | 0 | 0.08 | <0.01 | 1979 | P |
| 61 | 0/50 | 0 | 0.50 | - | 1981 | P |
| 62 | 0/50 | 0 | 0.30 | - | 1981 | P |
| 63 | 1/50 | 2.0 | 0.08 | - | 1981 | P |
| 64 | 0/50 | 0 | 0.20 | - | 1981 | P |
| 65 | 0/50 | 0 | 0.18 | - | 1981 | P |
| 66 | 0/50 | 0 | 0.30 | <0.01 | 1981 | P |
| 67 | 0/50 | 0 | 0.40 | <0.01 | 1981 | P |
| 71 | 16/40 | 40.0 | 4.17 | - | 1986 | P |
| 72 | 2/40 | 5.0 | 3.56 | - | 1986 | P |
| 73 | 2/40 | 5.0 | 2.86 | - | 1986 | P |
| 74 | 2/40 | 5.0 | 1.77 | - | 1986 | P |

continued...

| Chasey & McKee Sample No. | Tumour response Animals | %T | DMSO extract IP 346 % m/m | BaP Content mg/kg | Year of study | Crude type |
|---|---|---|---|---|---|---|
| **Hydrotreated distillates** | | | | | | |
| 80 | 0/40 | 0 | 3.29 | 0.20 | 1975 | P |
| 81 | 0/40 | 0 | 0.13 | <0.01 | 1975 | P |
| 82 | 7/50 | 14.0 | 7.07 | <0.70 | 1980 | P |
| 83 | 20/50 | 40.0 | 3.81 | <0.50 | 1979 | P |
| 86 | 0/40 | 0 | 3.01 | - | 1986 | P |
| 90 | 0/40 | 0 | 2.73 | - | 1985 | N |
| 91 | 0/40 | 0 | 2.41 | - | 1985 | N |
| 92 | 0/40 | 0 | 2.66 | - | 1985 | N |
| **Distillate aromatic extracts - untreated** | | | | | | |
| 12 | 41/50 | 82.0 | 11.25 | <2.5 | 1979 | P |
| 13 | 5/50 | 10.0 | 3.69 | <0.1 | 1979 | P |
| **Distillate aromatic extracts - hydrotreated** | | | | | | |
| 16 | 44/50 | 88.0 | 16.18 | <3.0 | 1980 | P |
| 18 | 43/50 | 86.0 | 8.02 | <1.0 | 1979 | N |
| 19 | 43/50 | 86.0 | 9.02 | <1.4 | 1979 | P |
| **Refined vacuum residuals** | | | | | | |
| 94 | 0.50 | 0 | 1.37 | <0.25 | 1979 | P |

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Notes: Crude type P denotes paraffinic, N danotes naphthenic