Mocrinis II Workshop

Manufacture of Mineral Oil and Wax Composition and Specifications

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Agenda

- Manufacture of Mineral Oil and Wax
- Impact on Substance Composition
- Definitions, Uses, Specifications, Regulations
Part 1

Manufacture of Mineral Oil and Wax

Impact on Substance Composition
Complex Flow Diagram of typical integrated Refinery

Lubricant Base Oils, White Oils, Wax: < 10% of total refinery production

Figure 4: Schematic flow diagram of a typical integrated oil refinery.

Source: EFSA Opinion 2012
Hydrocarbon type molecules from crude are selected during Refining

- **Crude Oil:** “complex” matrix of naturally occurring hydrocarbons ("UVCBs" - Substances of Unknown or Variable composition, Complex reaction products or Biological materials) which can be orderly classified
  - by molecular weight and by molecular structure
  - in limited number of chemical families because of natural origin of crude
- **Refining will select the desired molecules** for the targeted applications

<table>
<thead>
<tr>
<th>CRUDE OILS COMPONENTS</th>
<th>HYDROCARBON</th>
<th>CHEMICAL NAME</th>
<th>STRUCTURE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paraffinics</td>
<td>normal alkanes</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>iso alkanes</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>(with/without heteroatoms)</td>
<td></td>
<td></td>
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<tr>
<td>Naphthenics</td>
<td>Mono-cyclo alkanes</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>polycyclic alkanes</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>(with/without heteroatoms)</td>
<td></td>
<td></td>
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<tr>
<td>Aromatics</td>
<td>mono aromatics</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>polycyclic aromatics</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>(with/without heteroatoms)</td>
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<td></td>
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<tr>
<td></td>
<td>eg. Ni, V, Fe</td>
<td></td>
<td></td>
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<tr>
<td>Metals</td>
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</tr>
</tbody>
</table>
Content in various Hydrocarbon types vary with crude type and carbon number/boiling range

- Above example: typical hydrocarbon distribution of a lube crude (e.g. Arab Light)
  - Broad lumping of molecule types in the whole crude
  - Lubricant Base Oils / Wax / White Oils typically fall in 300°C to 700°C distillation range
- Petroleum **refining** primarily **controlled by Boiling range/temperature**
  - Carbon number of distillation cuts estimated from corresponding n-paraffin molecules
- Refining is needed to isolate the desired Lubricant Base Oils/Wax/White Oils components
- **Hydrocarbon solvents** (Carbon number below C20) **out of scope of mineral oils**
### Mineral Base Oils Constituents vs Performance

<table>
<thead>
<tr>
<th>Molecule</th>
<th>Lubricant Base Oils Characteristics</th>
<th>Wax</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Viscosity Index</td>
<td>Pour point</td>
</tr>
<tr>
<td>n-paraffins</td>
<td>Excellent</td>
<td>Poor</td>
</tr>
<tr>
<td>Iso-paraffins</td>
<td>Good/ Excellent</td>
<td>Good</td>
</tr>
<tr>
<td>Highly alkylated aromatics</td>
<td>Poor</td>
<td>Good</td>
</tr>
<tr>
<td>Naphthenics</td>
<td>Poor</td>
<td>Excellent</td>
</tr>
<tr>
<td>Polynuclear aromatics</td>
<td>Poor</td>
<td>Poor</td>
</tr>
</tbody>
</table>

- “Good” vs “Poor” performance depends on final application
  - eg “good” for wax is different from “good” for oil

- **Sulfur and nitrogen** in crudes significantly reduced through refinery processing
  - Sulfur can contribute to oxidation stability in conventionally refined base oil
  - Nitrogen can contribute to product color and is generally eliminated in processing
Molecular Structures Waxes and Oils

Waxes

Oils

Wax Fraction or Oil Fraction?
Molecular Structures Waxes and Oils: Iso-paraffins

Waxes

Oils

Wax Fraction or Oil Fraction
What Mineral Oil- and Wax is / is not

• The terms “mineral oil” or “wax” are generic and do not mean the same thing for everyone. Definitions and context is needed to understand the issue.

• Refined “mineral oils” and “waxes” are refinery products manufactured to meet specific standards.

• While Mineral oil can be chromatographically described as MOSH and MOAH fractions, these fractions do not always represent the products on the market.

• MOSH and MOAH does not necessarily mean “mineral oil”.
  • An isolated MOSH fraction does not imply that ALL saturated hydrocarbons are from mineral oil origin. It can contain natural n-alkanes.

• An isolated MOAH fraction does not imply PAC presence neither refinement level.

• The MOSH term applied to the saturated fraction of a wax is misleading because MO refers to an “oil”. At 25ºC an oil is liquid, a wax is solid.

Remember: MOSH and MOAH terms are highly contextual.
Crude Oil Initial Chemical Composition

The individual hydrocarbon components have diverse and well known performance characteristics and toxicological properties, that will drive refining.

Refining will select the molecules from the crude oil in controlled manner to set the final chemical composition (and properties) of the mineral oil.
Base Oil, Wax, White Oil Manufacture
Step 1 : Distillation

Atmospheric distillation
- Petroleum gases
- Naphta
- Kerosene
- Gasoils
- Heavy fuel oils
- Residue

Vacuum distillation

“Vacuum Distillate” = Feed for LBO, HRBO, Wax Manufacture
Atmospheric and Vacuum Distillation Impact

Distillation determines the Boiling/Carbon/molecular weight range of the final oil.

Boiling range / Carbon # range, Viscosity, Volatility are set by Distillation Units.

Diagram: Content (mass %) vs. Viscosity at 100°C (mm²/s) for different types of hydrocarbons:
- N-Alkanes
- Iso-Alkanes
- Naphthenes (mono and multi rings)
- Aromatics (1 and 2 rings)
- Aromatics (3+ rings)

Typical high viscosity distillate: C24 – C40.
Base Oil, Wax, White Oil Manufacture
Step 2: Aromatic Removal

Atmospheric distillation
- Petroleum gases
- Naphta
- Kerosene
- Gasolins
- Heavy fuel oils
- Residue

Vacuum distillation

Extraction of 3-7 PAC or hydrocracking or hydrogenation

Distillate aromatic extract

FAIL

PASS

IP 346

"Raffinate" = Feed to dewaxing unit
Extraction or Hydrocracking or Hydrogenation determines the total aromatics content and removes most of Polycyclic Aromatic Compounds.

- Removes most of Polycyclic Aromatic Compounds, down to IP346<3%
- Critical step to ensure mineral oils and wax are non-carcinogenic

Typical Group I heavy mineral oil
Aromatics:10-40%

Extraction or Hydrocracking or Hydrogenation determines the total aromatics content.
Base Oil, Wax, White Oil Manufacture
Step 3: wax separation

- Atmospheric distillation
- Vacuum distillation
  - Petroleum gases
  - Naphtha
  - Kerosene
  - Gas oils
  - Heavy fuel oils
  - Residue
- Extraction of 3-7 PAC or hydrocracking or hydrogenation
  - Distilled aromatic extract
- IP 346
  - FAIL
  - PASS
- Dewaxing (Safe)
- Base oil (Safe)

* Dewaxing not necessary for manufacture of Naphthenic base/white oils
Paraffin separation through Dewaxing

Solvent Dewaxing or Iso-Catalytic dewaxing removes solid waxy hydrocarbons (n-paraffins and some isoparaffins) from mineral oil. Creates Wax as a co-product.

**Typical dewaxed heavy oil**
- N-alkanes: 0%

**Solvent Dewaxing or Catalytic dewaxing** removes paraffins and some isoparaffins

Typical dewaxed heavy oil
- N-alkanes: 0%
Technical White Oil Manufacture

1. Atmospheric distillation:
   - Petroleum gases
   - Naphta
   - Kerosene
   - Gas oils
   - Heavy fuel oils
   - Residue

2. Vacuum distillation

3. Extraction of 3-7 PAC or hydrocracking or hydrogenation

4. Distillate aromatic extract

5. IP 346
   - FAIL
   - PASS

6. Safe
   - Slack-wax
   - Dewaxing*
   - Base oil
   - Technical white oil

7. Safe
   - Hydrothermal acid treatment 1

* Dewaxing not necessary for manufacture of Naphthenic base/white oils
Aromatic saturation through High pressure Hydrotreatment or Hydrocracking (1st step)

Hydrocracking or Moderate Hydrotreatment or Acid Treatment remove most of aromatics (to a few %), and Polyaromatics below ppm level => Technical White Oils

- Hydrocracking or Moderate Hydrotreatment or Acid Treatment removes most of aromatics (to a few%)

Typical Heavy Technical White Oil:
Aromatics%: 0.5-5%

- Total Aromatics% typically 0.5-5% level in technical white oils
- PAC% below ppm level in technical white oils
Full Aromatic saturation through High pressure Hydrogenation (2nd step)

Severe Hydrogenation or Acid Treatment remove nearly all remaining aromatics (to ~0.1%), and bring Polyaromatics to ppb level => Medicinal White Oils

- Total Aromatics typically around 0.1% level in pharmaceutical white oils
- PAC% at ppb level or below in pharmaceutical white oils

Hydrogenation or Severe Acid Treatment = ultimate severity for removal of aromatics

Typical heavy viscosity white oil
Aromatics %<=0.1%
Refining Summary: from crude to pharmaceutical white oil

Refining selects the molecules from the crude oil in a controlled manner to set the final chemical composition (and properties) of the mineral oil.

- **Crude selection** determines the initial distribution of paraffinics vs naphthenics vs aromatics.
- **Distillation** determines the molecular weight range of the final oil.
- **Dewaxing** or **Iso-Catalytic dewaxing** removes paraffins and some isoparaffins.
- **Extraction** or **Hydrocracking or Hydrogenation** determines the total aromatics content.
- **Severe Hydrogenation / Acid Treatment** removes nearly all aromatics/PCAs.

**Severe Hydrogenation / Acid Treatment** = ultimate severity
- Typical heavy viscosity white oil: Aromatics% <= 0.1%

- Mineral Oils: Total aromatics range 0-50%, with PAC% << Aromatic%
- Pharma white oils: Total aromatics around 0.1%, PAC below ppb level
Chemical composition is adjusted through refining

Removal or conversion of undesirable molecules + Selection of desired molecules is obtained through the various refining units

Final chemical composition adapted to targeted properties and performance

Boiling/Carbon # range, Viscosity, Volatility set by Distillation Units

n-Paraffins removal / Wax production in Dewaxing Unit

Full aromatic saturation from severe Hydrotreatment or Acid Treatment (eg White Oil Units)

Poly-Aromatics (+some mono-/di-aromatics) removal in Extraction or Hydrocracking Units
Chemical composition is controlled by specifications

A set of **specifications** has been **developed** to efficiently and tightly **control** mineral oil **composition** according to its intended application.

- Specifications defined to ensure Performance in application and absence of Health and Safety concern for end consumers
- Specification **tests shall be simple and quick** to be run on each production batch

Volvatility or Flash point

Viscosity or GC distillation

Pour point or solid paraffin test

CaCpCn by ASTM D2140 or Density or Viscosity Index

Aromatics % in MO by ASTM D2007

Aromatics ppm in WO by direct UV

PACs % in MO by IP 346

PACs ppb in WO by UV DMSO
Petroleum Jelly Manufacture

Atmospheric distillation

Vacuum distillation

Extraction of 3-7 PAC or hydrocracking or hydrogenation

Distillate aromatic extract

FAIL

IP 346

PASS

Safe

Dewaxing*

Safe

Slack-wax

De-oiling

Base oil

Technical white oil

Hydrotreatment/acid treatment 1

Hydrogenation or clay treatment

Hydrotreatment/acid treatment 2

Blending

Hydrogenation or clay treatment

Food grade wax & microwax

Petroleum jelly

* Dewaxing not necessary for manufacture of Naphthenic base/white oils

Paraffin & microwax

De-oiling

Hydrotreatment/acid treatment 1

Hydrogenation or clay treatment

Pharmaceutical white oil

Technical white oil

Base oil

Hydrotreatment/acid treatment 2

Blending

Hydrogenation or clay treatment

Food grade wax & microwax

Petroleum jelly
Synthetic Wax and Oil Manufacturing (Fischer Tropsch)

- Process invented in 1925 by Franz Fischer and Hans Tropsch
- Uses several carbon sources
  - Biomass to Liquids/Solids
  - Coal to Liquids/Solids
  - Gas to Liquid/Solid
- Manufactures a variety of products
  - Diesel, Naphta, Jet Fuel, Base Oils, Waxes, etc
- Commercial product range includes oils of different viscosities and low and high melting waxes
Questions?

Laurent Jouanneau

Email: laurent.jouanneau@exxonmobil.com
COFFEE BREAK!
Part 2

Mineral Oil and Wax:
Definitions, Uses, Specifications, Regulations
Technical vs Medicinal White Oils
Definitions, Specifications

- **Definition: “white oils”** also defined as Highly Refined Base Oils (HRBO):
  - Colorless, highly refined mineral oils derived from non-carcinogenic LBO (excludes synthetic oils)
  - Hydrotreatment or Acid treatment to achieve **extremely low levels of aromatics**

- **Technical white oils:**
  - HRBOs not complying with pharmacopeia monograph purity
  - Meet requirements of US **FDA 21 CFR-178.3620(b) – color and UV-DMSO limits**
  - **Very low Aromatics,** mainly 1-2 ring highly alkylated structures (**typically 0.5 to 5%**)
  - **Uses:** Food Grade Lubricants, rubber extender oils, textile oils, Petroleum Jellies,…

- **Pharmaceutical/Medicinal/Food Grade white oils (paraffinum liquidum)**
  - Derived from technical white oils, refined in a second step (Hydrotreatment or Acid treatment)
  - Comply with **purity of pharmacopeia monographs** (Eur or US) or FDA (US)
  - **Extremely low** levels of **aromatics** (1-2 ring highly alkylated structures) - **typically ~0.1%**
  - **Purity tests:**
    - **UV-DMSO:** tracks PACs, used in Pharmacopeias (EU/US) and FDA (US, food-contact)
      - Direct UV test was used in former DAB (German Pharmacopeia), indicator of «Total aromatics»
    - **Readily Carbonisable Substances:** tracks aromatics and impurities
  - Several categories based on **viscosity range** in pharmacopeias
  - Kin viscosity, Mol Weight and Carbon Nber used by JECFA to define mineral oils categories
    - Also used in EU Plastic Regulation
White Oils main purity test: UV-DMSO

- Based on **UV absorption of DMSO extract** of a white oil
  - WO first diluted with n-hexane
  - PAHs selectively extracted with DMSO
  - Absorbance of the extract (260-350 nm range) compared to a reference
  - described in ASTM D 2269 method

- **Pharmacopoeias Pass limits**
  - max extract absorbance (10 mm cell) $\sim 0.10$
  - Estimated equivalent to $\sim 0.3$ ppm max of PAHs
  - Typical PAHs contents in ppb range for most of commercial WOs

- **required in Pharmacopoeias and FDA specifications**
  - Max absorbance Limit 4.0 for Tech White oils (FDA (b))
  - Another UV-DMSO test procedure used for FDA(c) oils

- simple method, well suited to routine PAH content control of production batches in refinery labs
<table>
<thead>
<tr>
<th>Application</th>
<th>Example</th>
<th>EU Regulation</th>
<th>US Regulation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Food Additive</td>
<td>Glazing agent, anti-foaming, carriers, preservative for eggs or dried fruits</td>
<td>EU 1333/2008/EC (Directive 95/2/EEC): <strong>White Oils not on positive list¹</strong></td>
<td>21 CFR 172.878</td>
</tr>
<tr>
<td>Processing Aid</td>
<td>Release agent/lubricant, dedusting agent in grain, pan oil, demoulding oil</td>
<td><strong>No EU Directive</strong> Some specific local regulations²</td>
<td>21 CFR 172.878 (not differentiated from food additives)</td>
</tr>
<tr>
<td>Food Contact Materials</td>
<td>Extender oil in plastics, elastomers, paper, glass, metal, wood, cork, textiles, adhesives, pigments ...</td>
<td>Framework (EC) 1935/2004 <strong>Plastics: EU 10/2011</strong> Others: to be developed Some local regulations³</td>
<td>Various FDA chapters Require mineral oils that meet 21CFR178.3620 (a),(b) or (c) purity</td>
</tr>
<tr>
<td>Lubricant for incidental food contact</td>
<td>Formulation of lubricants for food machinery</td>
<td><strong>No EU regulation</strong></td>
<td>21 CFR 178.3570 (requires 178.3620(b) oils) NSF H-1 registration</td>
</tr>
</tbody>
</table>

- Most existing purity requirements are based on PACs using UV-DMSO methods

¹ Microcrystalline waxes are listed as E 905
² Eg French Arrêté for "Auxiliaires technologiques" (Food Processing Aids) – 21 Oct 2006 – demoulding uses (biscuits)
³ Germany: BfR recommendation XXV Purity requirements for mineral oil (155 BGA Mitteilung), microcristalline wax and paraffin
## EU Plastics Regulation 10/2011 – Food Contact

General requirements outlined in Art. 3 of EU framework Regulation (EC) N° 1935/2004 for materials and articles intended to come into contact with foodstuff.

<table>
<thead>
<tr>
<th>Products</th>
<th>FCM 95</th>
<th>Viscosity not less than 8,5 mm²/s at 100°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>White mineral oils</td>
<td></td>
<td>Carbon number amount &lt;C25, max 5 %</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Average molecular weight not less than 480</td>
</tr>
<tr>
<td>Microcrystalline wax</td>
<td>FCM 94</td>
<td>Viscosity not less than 11 mm²/s at 100°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Carbon number amount &lt;C25, max. 5%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Average molecular weight not less than 500</td>
</tr>
<tr>
<td>Paraffin wax*</td>
<td>FCM 93</td>
<td>Viscosity not less than 2.5 mm²/s at 100°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Carbon number amount &lt;C25, max 40%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Average molecular weight not less than 350</td>
</tr>
</tbody>
</table>

* Restriction: 0.05 mg/kg food
Not to be used for articles in contact with fatty foods
EFSA and JECFA have set ADIs to various oil categories (food additive use)

EU Plastic regulation specifies oils and waxes that meet EFSA/JECFA categories
  - No direct regulatory link between EFSA/JECFA ADIs and the Plastic regulation

Some local regulations have set same oil requirements as EU Plastics Regulation, even if for different materials than plastics
  - e.g. German Draft Ordinance on Printing Inks, Elastomerleitlinie (Leitlinie zur hygienischen Beurteilung von Elastomeren im Kontakt mit Trinkwasser), Swiss Ordinance 817.023.21 April 2010 for food contact

<table>
<thead>
<tr>
<th>JECFA/EFSA Categories Specifications and ADIs</th>
<th>ADI JECFA</th>
<th>ADI EFSA</th>
<th>Kinematic viscosity at 100°C (cSt)</th>
<th>Average molecular weight</th>
<th>Carbon number at 5% boiling point</th>
<th>EU 10/2011 Plastic Regulation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microcrystalline wax</td>
<td>0-20 mg/kg</td>
<td>0-20 mg/kg</td>
<td>≥ 11</td>
<td>≥ 500</td>
<td>≥ 25</td>
<td>X</td>
</tr>
<tr>
<td>Paraffin wax</td>
<td>-</td>
<td>-</td>
<td>≥ 2.5</td>
<td>≥ 350</td>
<td>Max 40% C&lt;25</td>
<td>X</td>
</tr>
<tr>
<td>Mineral oil (high viscosity)</td>
<td>0-20 mg/kg</td>
<td>0-12 mg/kg</td>
<td>&gt; 11</td>
<td>≥ 500</td>
<td>≥ 28</td>
<td>X</td>
</tr>
<tr>
<td>Mineral oil (medium and low viscosity) Class I</td>
<td>0-10 mg/kg</td>
<td>0-12 mg/kg</td>
<td>8.5-11</td>
<td>480-500</td>
<td>≥ 25</td>
<td>X</td>
</tr>
<tr>
<td>Class II - (removed)</td>
<td>- (removed)</td>
<td>- (removed)</td>
<td>7.0 – 8.5</td>
<td>400-480</td>
<td>≥ 22</td>
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<tr>
<td>Class III - (removed)</td>
<td>- (removed)</td>
<td>- (removed)</td>
<td>3.0 – 7.0</td>
<td>300-400</td>
<td>≥ 17</td>
<td></td>
</tr>
</tbody>
</table>
### Legislative framework – Paraffins and Microwax

<table>
<thead>
<tr>
<th>Product</th>
<th>Food Contact Legislation</th>
<th>Food Additive</th>
<th>Pharmacopea</th>
<th>Cosmetic</th>
<th>Pharmacopoeia and Cosmetic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hard Paraffin/ Microcrystalline</td>
<td>FDA.178.3710</td>
<td>US Petroleum Wax Monograph</td>
<td>FDA 172.886 (PCA -UV Absorption)</td>
<td></td>
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<td></td>
<td>EC-1935/2004</td>
<td>Framework regulation</td>
<td></td>
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<tr>
<td></td>
<td>EC-42/2007 (Regenerated Cellulose film)</td>
<td>Bfr - Recomendations for Paraffin: XXI, XXXV, XXXVI, XLIV, XLVIII, LII.</td>
<td></td>
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</tr>
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<td></td>
<td>EC-42/2007 (Regenerated Cellulose film)</td>
<td>Bfr - Recomendation XXV</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hard Paraffin</td>
<td>EU 10/2011 (PLASTIC), German Elastomer Guidance, Swiss Ordinance 817.023.21</td>
<td>95858 LVP</td>
<td>Eur /Ph. 9.0 USP -40</td>
<td>EC/1223-2009 and Cosmetic Europe recommendation</td>
<td>PAH Level ( &lt; 1/3 of absorbance of a solution containing 7 ppm naphthalene in DMSO at 278 nm)</td>
</tr>
<tr>
<td></td>
<td>95859 HVP</td>
<td>JECFA (CODEX: INS-905); EU 95/2 (E-905)</td>
<td>EC-231/2012</td>
<td>Eur /Ph. 9.0 USP -40 (*)</td>
<td>EC/1223-2009 and Cosmetic Europe recommendation</td>
</tr>
<tr>
<td>Microcrystalline</td>
<td></td>
<td>95859 HVP (PCA-UV Absorption)</td>
<td></td>
<td></td>
<td>PAH Level (EC-1223/209) and (KV ≥ 11, MW≥ 500, Carbon number at 5% boiling point ≥25)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Colipa Recommendation n° 14</td>
<td></td>
</tr>
</tbody>
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(*)- Microcrystalline Monograph in progress for European Regulation
Hydrocarbon Waxes as Food Contact Materials - Regulations

<table>
<thead>
<tr>
<th>Hydrocarbon Waxes as Additive or Formulation Ingredient</th>
<th>Waxes as FCM Group (EC 1935/2004) Framework Regulation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Harmonized FCM (Member State Legislation)</td>
<td>Non-Harmonized FCM (Member State Legislation)</td>
</tr>
<tr>
<td>Plastics Regulation 10/2011</td>
<td>Germany: Recommendation XXV</td>
</tr>
<tr>
<td>Adhesives</td>
<td>Holland: Warenwet Chapter X</td>
</tr>
<tr>
<td>Coatings and Varnishes</td>
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<td>Printing Inks</td>
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<tr>
<td>Rubber</td>
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<td>Paper and Board</td>
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<td>Textiles</td>
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</tbody>
</table>

The **principle of mutual recognition** allows for the legal importation and sale into one Member State of products that are legally marketed in another Member State, even if the products do not comply with the specific regulatory requirements of the country of import.
## Hydrocarbon Waxes in the (Food Contact) Plastics Regulation EU 10/2011: specifications and purity

<table>
<thead>
<tr>
<th>EU 10/2011</th>
<th>FCM 93</th>
<th>FCM 94</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Description</strong></td>
<td>Waxes, paraffinic, refined, derived from petroleum based or synthetic hydrocarbon feedstocks, <strong>low viscosity</strong></td>
<td>Waxes, refined, derived from petroleum based or synthetic hydrocarbon feedstocks, <strong>high viscosity</strong></td>
</tr>
<tr>
<td><strong>Specification</strong></td>
<td><strong>Average Mol weight &gt; 350 Dalton. Viscosity 100 °C &gt; 2,5 cSt. Hydrocarbons with Carbon number less than 25, &lt; 40 %</strong></td>
<td><strong>Average Mol weight &gt; 500 Dalton Viscosity 100 °C &gt; 11 cSt Hydrocarbons with Carbon number less than 25, &lt; 5 %</strong></td>
</tr>
<tr>
<td><strong>Typical Products covered</strong></td>
<td>Mineral paraffin wax and synthetic (low viscosity) paraffin wax (both <strong>foodgrade</strong>)</td>
<td>Mineral microcrystalline wax and synthetic (high viscosity) paraffin wax (both <strong>foodgrade</strong>)</td>
</tr>
<tr>
<td><strong>Source</strong></td>
<td>Vacuum distillate mineral oil</td>
<td>Residue vacuum distillate mineral oil</td>
</tr>
<tr>
<td><strong>Abbreviation</strong></td>
<td>LMPW</td>
<td>IMPW</td>
</tr>
<tr>
<td><strong>Carbon distribution</strong></td>
<td>C20 - C35</td>
<td>C25 - C45</td>
</tr>
<tr>
<td><strong>N-alkane content (%)</strong></td>
<td>85-90</td>
<td>50 - 70</td>
</tr>
<tr>
<td><strong>Melting point (°C)</strong></td>
<td>52 - 60</td>
<td>60-68</td>
</tr>
<tr>
<td><strong>Viscosity 100 °C (cSt)</strong></td>
<td>3</td>
<td>7</td>
</tr>
<tr>
<td><strong>Average Mol weight</strong></td>
<td>350</td>
<td>475</td>
</tr>
<tr>
<td><strong>SML</strong></td>
<td>0.05 mg/kg</td>
<td>0.05 mg/kg</td>
</tr>
<tr>
<td><strong>Purity requirements</strong></td>
<td>Based on absence PAH</td>
<td>Based on absence PAH</td>
</tr>
<tr>
<td><strong>MOAH by GC (%)</strong></td>
<td>Virtually absent</td>
<td>Virtually absent</td>
</tr>
<tr>
<td><strong>MOAH by NMR (%)</strong></td>
<td>Virtually absent</td>
<td>Virtually absent</td>
</tr>
<tr>
<td>Product</td>
<td>Food Contact Legislation</td>
<td>Food Additive</td>
</tr>
<tr>
<td>-------------------------</td>
<td>--------------------------</td>
<td>---------------</td>
</tr>
<tr>
<td></td>
<td>Regulation</td>
<td>Purity test</td>
</tr>
<tr>
<td>EU 10/2011 (PLASTIC)</td>
<td>95833</td>
<td></td>
</tr>
<tr>
<td>21 CFR 178.3620 (a), (b) or ©- Food contact Plastics</td>
<td>FDA 172.878 Food Additive or Process Aid (e.g. Release Agent)</td>
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<tr>
<td>21 CFR 178.3570 (incidental food contact)</td>
<td>PCA-UV Absorption</td>
<td></td>
</tr>
<tr>
<td>Bfr-Recommendations V, VI, IX, XXI, XXXVI, XLIV</td>
<td>Bfr - 155 BGA Mitteilung=DAB 8</td>
<td>JECFA Monographs and 21 CFR 178.3570</td>
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<tr>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yellow Petrolatum</td>
<td></td>
<td></td>
</tr>
<tr>
<td>White Petrolatum</td>
<td></td>
<td></td>
</tr>
<tr>
<td>White and Yellow Petrolatum</td>
<td>FDA 178.3700 and 178.3710 indirect food contact</td>
<td>FDA 172.886(b)</td>
</tr>
</tbody>
</table>

(*)- Microcrystalline Monograph in progress for European Regulation

Lubricants: EN ISO 21469 "Security of machine lubricants with no foreseeable product contact - hygiene requirements (ISO 21459:2006)"
Requirements for Medicinal/Pharmaceutical uses based on Pharmacopeia Monographs

Purity requirements are very similar to those of FDA and JECFA

<table>
<thead>
<tr>
<th></th>
<th></th>
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</thead>
<tbody>
<tr>
<td>Initial Boiling Point</td>
<td>Medium Vis MO (2013 edition)</td>
<td>&gt;= 200°C</td>
<td>Light Mineral Oil</td>
<td>0.810-0.875</td>
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<tr>
<td></td>
<td>High Vis MO (2006 edition)</td>
<td>&gt;=350°C</td>
<td>Mineral Oil</td>
<td>0.827-0.890</td>
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<tr>
<td>Average Molecular Weight</td>
<td>480 - 500</td>
<td>500 min</td>
<td>Light Liquid Paraffin</td>
<td>25-80 cPo</td>
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<tr>
<td>Carbon number at 5% dist point</td>
<td>25 min (BP&gt;391°C)</td>
<td>28 min (BP&gt;422°C)</td>
<td>Liquid Paraffin</td>
<td>110-230 cPo</td>
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<tr>
<td>Rel. Density 20/20°C</td>
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<td>0.810-0.875-0.827-0.890</td>
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<tr>
<td>SpecGravity 25/25°C</td>
<td>0.818-0.880</td>
<td>0.845-0.905</td>
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<tr>
<td>Viscosity</td>
<td>8.5 - 11 cSt</td>
<td>11 cSt</td>
<td>3.0 - 34.4 cSt</td>
<td>34.5 - 150 cSt</td>
</tr>
<tr>
<td></td>
<td>at 100°C</td>
<td>at 100°C</td>
<td>at 40°C</td>
<td>at 40°C</td>
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<tr>
<td>IDENTIFICATION</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Infra-red spectrum</td>
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<td></td>
<td>Identical to reference spectrum</td>
</tr>
<tr>
<td>Reaction with NaOH</td>
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<td></td>
<td></td>
<td>Neutral</td>
</tr>
<tr>
<td>TESTS</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Acidity/Alcalinity</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td></td>
</tr>
<tr>
<td>Heavy Metal Content</td>
<td>Lead : 1ppm max</td>
<td>-</td>
<td>-</td>
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<tr>
<td>Sulfur Compounds</td>
<td></td>
<td>Pass</td>
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<tr>
<td>Solid Paraffin (4h/0°C)</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td></td>
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<tr>
<td>Polycyclic Arom. Hydrocarbons</td>
<td></td>
<td>Pass</td>
<td>Pass</td>
<td></td>
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<tr>
<td>UV Absorp. of DMSO extract</td>
<td></td>
<td>Pass</td>
<td>Pass</td>
<td></td>
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<tr>
<td>Readily Carbonizable Substances</td>
<td>Pass (USP)</td>
<td>Pass</td>
<td>Pass</td>
<td></td>
</tr>
</tbody>
</table>

Note: Reproduction permitted with due acknowledgement
# Purity Criteria for pharmaceutical and cosmetic uses
## Mineral Oil and Wax

<table>
<thead>
<tr>
<th>Substance</th>
<th>European Pharmacopoeia Designations</th>
<th>European Pharmacopoeia Test</th>
<th>Limit</th>
<th>INCI Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>White Mineral Oil</td>
<td>Paraffin, Light Liquid Paraffin, Liquid</td>
<td>Polycyclic aromatic hydrocarbons</td>
<td>Not more than 1/3 of the absorbance of a solution containing 7 ppm naphthalene in trimethylpentane at 275 nm</td>
<td>Paraffinum Liquidum (EU) Mineral Oil (USA)</td>
</tr>
</tbody>
</table>
| Microcrystalline Wax       | --- (no Ph. Eur.) USP/NF listed       | Polycyclic aromatic hydrocarbons from FDA 21 CFR 172.886 b | Max 0.15 at 280 – 289 nm  
Max 0.12 at 290 – 299 nm  
Max 0.08 at 300 – 359 nm  
Max 0.02 at 360 – 400 nm | Cera Microcrystallina (EU) Microcrystalline Wax (USA) |
| Paraffin Hard              | Hardparaffin                          | Polycyclic aromatic hydrocarbons  | Not more than 1/3 of the absorbance of a solution containing 7 ppm naphthalene in DMSO at 278 nm  | Paraffin                                                                |
| White Petrolatum           | Paraffin, White Soft                  | Polycyclic aromatic hydrocarbons  | Not more than 1/3 of the absorbance of a solution containing 6 ppm naphthalene in DMSO at 278 nm | Petrolatum                                                              |
| Yellow Petrolatum          | Paraffin, Yellow Soft                 | Polycyclic aromatic hydrocarbons  | Not more than 1/3 of the absorbance of a solution containing 9 ppm naphthalene in DMSO at 278 nm | Petrolatum                                                              |

**INCI**: International Nomenclature of Cosmetic Ingredients

- No European Pharmacopoeia exists for Microcrystalline wax
- However PACs are strictly controlled
Mineral Oil and Wax - Manufacturing and Quality Assurance Testing

- Refining operation
- Carbon# range selection
- Polycyclic Aromatic Compound (PAC) removal
- Waxy hydrocarbons separation
- Purification
- Petroleum jelly manufacturing

- Refining process
- Atmospheric and vacuum distillation
- Solvent extraction and/or catalytic hydrotreatment
- Solvent dewaxing or catalytic dewaxing
- Hydrogenation or acid/clay treatment
- Blending and optional purification

- Product
- Distillate or vacuum gas oil
- Base oil and slack-wax
- Technical white oil, white oil, paraffin wax and micro-crystalline wax
- White petroleum jelly and yellow petroleum jelly

- Safety qualifier (PAC % content)
  - Carcinogenic: IP 346 ≥ 3%
  - Non-carcinogenic: IP 346 < 3%

Pharmacopeia tests
Quality Assurance / Protection from Contamination

- Products are controlled vs **manufacturing and sales specifications**
  - defined at industry or company level
  - carefully designed to **control their chemical composition**

- **Absence of contamination** during refinery transfers, loading and packaging **controlled by Quality Assurance** procedures
  - Eg ISO 9001, ISO 14001, Good Manufacturing Practices (GMP)

Mineral oil / wax handling
Quality control from manufacture to shipment

- Mineral oil or wax plant ➔ Production control ➔ Q = OK
- Dedicated tank ➔ Batch release control ➔ Q = OK
- Direct loading ➔ Bulk transfer control ➔ Q = OK
- Packaging ➔ Quality control ➔ Q = OK

There are dedicated pipes and tanks for each step in the process.
Procedures are in place for manufacturing, storing, handling, packaging as part of the Quality Assurance process.
Q = Quality
Conclusion – Manufacture and composition of Mineral Oil and Wax

- **Refining** selects the molecules from the crude oil in a controlled manner to set the final chemical composition (and properties) of the mineral oil and wax
  - Removal/conversion of undesirable molecules obtained through various refining units

- **Product Specifications** tightly control mineral oil and wax composition
  - to ensure performance in application and no safety concern for consumer
  - tests shall be simple and quick to be run on each production batch

- **PACs in mineral oil/wax have been removed at desired level**
  - Absence of carcinogenicity controlled by IP346 <3.0% and known refining history
  - Mineral base oils: Total aromatics can be 0-50%, but PAC% << Aromatic%
  - Purity of products used in pharmaceutic/cosmetic/Food contact applications ensured by Pharmacopeia UV Tests and adequate Quality Assurance/Quality Control
    - Medicinal white oils: Total aromatics ~ hundreds of ppms, PACs at ppb level

- **Total aromatics content is not a correct safety indicator**

- Development of **harmonized EU regulations** needs to be pursued
  - compatible EU and US regulations are preferred (e.g. pharmacopeias)
Questions?

Laurent Jouanneau
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