The present invention relates to a cleaning composition for the use in treatment and cleaning of crude wool. The wool scouring composition comprises at least one dicarboxylic acid diester and at least one alcohol ethoxylate and/or amino alcohol ethoxylate surfactant.
CLEANING COMPOSITION FOR WOOL SCOURING
AND FELTING

Field of the Invention
The present invention relates to a cleaning composition for the use in treatment and cleaning of crude wool.

The process for the production of wool fiber involves several steps. One of the steps taken after wool is sheared is a scouring process; it involves the washing (scouring) of the wool to remove contaminants. The type and amount of contaminants can vary according to breed, nutrition, environment and position of the wool on the animal. Contaminants constitute of solvent-soluble fraction (wool grease and protein material) and a water-soluble fraction (largely perspiration salts collectively termed suint, dirt and vegetable matter in the form of burrs and seeds from pastures). A fleece may contain up to 30% by weight of contaminants, depending on the animal, so it is important that the wool is treated before use.

Typical process of wool scouring goes through a first wash where water is warmed up to 60 degrees Celsius followed by cold rinses. Washed wool is passed through squeeze rollers and a hot-air drying chamber bringing the moisture content to the right level for the next step in processing.

Background of the Invention
Until the 1960s wool was conventionally washed in an aqueous bath containing soap and alkali (usually sodium carbonate). It was necessary for soft water to be used because of the insolubility of calcium and magnesium soaps. Scouring wool with soap and alkali was a process which had to be undertaken with some care because of the susceptibility of wool to be
damaged by alkali.

Therefore it was with some relief that the industry greeted the introduction of synthetic detergents which were unaffected by hard water and were cost effective in degreasing wool.

Originally these were anionic and it was found that alkali was still needed to avoid undue loss of detergent by adsorption on to the wool.

The later introduction of nonionic detergents removed this constraint, and wool was able to be washed with these detergents alone.

For many years now, scouring has been carried out with nonionic detergents nonylphenol-ethylene oxide condensate detergents (described as NPEO\(X\) types, where \(X\) is a numeral denoting the number of moles of EO in the product, EO represents Ethoxylate).

The wool scouring detergent most commonly employed is a NPEO9 type.

More recently, primary alcohol ethoxylate detergents have been added to the list of regular use. There are two reasons for using these in preference to NPEO types. The first is that residues of NPEO detergents are undesirable in products produced from refined wool grease. The second is that NPEO detergents are comparatively difficult to biodegrade, and their use may result in the introduction of relatively long-lived and toxic phenolic residues into the
environment. Thus wool processors working to an "Environmental Choice" standard will prefer to use comparatively easily biodegraded and non-toxic alcohol ethoxylate products.

Unfortunately, alcohol ethoxylate detergents which address the needs of wool grease refining are not the best for wool scouring. If grease properties are paramount, detergents of the general type of \( \text{CH}_3(\text{CH}_2)_{7-9}\text{O}(\text{CH}_2\text{CH}_2\text{O})_6\text{H} \) may be used, while for wool scouring molecules with larger hydrophobes and longer EO chains are preferred, for example \( \text{CH}_3(\text{CH}_2)_{i_0-1_3}\text{O}(\text{CH}_2\text{CH}_2\text{O})_9\text{H} \). If the hydrophobe is linear, the biodegradability is also enhanced.

The mechanism involved for the removal of contaminants is based upon the structure of the detergent molecules which contain a hydrophobic part which is the hydrocarbon moiety and a hydrophilic portion formed of the polyoxyethylene chain.

When wool grease, a hydrophobic substance, is removed from the fibre surface by detergent action, a stable grease micelle is formed in which the hydrocarbon moiety is embedded in the emulsified grease, while the hydrophilic polyoxyethylene chain mingles with the surrounding water. Anionic compounds from the wool grease and suint (fatty acid anions) also act as surface active agents and assist in stabilizing the wool grease dispersion. Dirt particles (largely mineral) are also entrained in and emulsified with the grease. Soluble proteins, absorbed onto or interacting with the droplet surface, add to the complexity of the structures. The result of this process of grease emulsification (which takes place mostly at about 60°C, above the wool grease melting temperature) is an extremely stable dispersion of wool grease and dirt which defies most conventional methods of demulsification and phase separation.
This dispersion stability greatly complicates issues of effluent treatment, leading to problems of the recovery of wool grease as a by-product of scouring.

This opens up a separate topic rich in chemistry, the recovery, purification, and saponification (hydrolysis) of wool grease.

Wool grease is a very complex mixture, which consists mostly of esters of various long-chain fatty acids with long-chain alcohols and sterols. Technically it is a wax, rather than a fat, because glycerol esters are not present. Clearly the esters formed from these acids and alcohols form a bewildering variety, especially when one considers that the hydroxy-acids and the diols can form di-esters. In addition to the esters, free fatty acids are present, along with other impurities such as soaps (metal salts of fatty acids). Calcium soaps are particularly important. These arise mainly from the fellmongery production of slipe wool with lime-sulphide depilatories, and can cause problems in the centrifugal separation of wool grease as well as in wool grease refining.

Refined wool grease is known as lanolin, which is the product resulting from deodorisation, decolorisation, neutralisation and removal of entrained water (volatiles). Lanolin is an important item of commerce. However, the most sought-after product from wool grease is the mixed wool-wax alcohols. These are produced by hydrolysis (saponification) of the esters followed by solvent extraction of the alcohols. These are widely used in toiletries and medicinal preparations because of their ability to form stable emulsions entraining large amounts of water.

In brief, current state of the art uses nonylphenol ethoxylates (NPEO, a common surfactant used in the textile industry for wool cleaning) but there is a
shift of wool processors towards the use of products which fit better environmental profile. Indeed, the compelling regulation in textile prohibits NPEO used in any process of textile treatment. Primary alcohol ethoxylates are preferred as they are comparatively easily biodegraded and non-toxic but they are not efficient in terms of scouring.

The object of the present invention is thus replacing NPEO with environment-friendly wool scouring composition and keeping the good performance as NPEO.

**Brief description of the invention**

The Applicant has surprisingly discovered that the use of a mixture of a solvent and a surfactant produces greater efficiency in the removal of contaminants from crude wool and ease in wool grease refining. Indeed, a mixture of solvent and surfactant according to the present invention brings about an improved performance from the combination of surfactant and solvent cleaning mechanisms, for the removal of solvent-soluble and water-soluble contaminants.

To this end, the present invention provides a wool scouring composition comprising:

a) at least one dicarboxylic acid diester corresponding to the formula (I):

\[ R^\text{OOC-A-COO-R}^2 \]  

(I)

wherein:

R\(^1\) and R\(^2\) are identical or different, and represent a linear or branched, cyclic or noncyclic, C\(_1\)-C\(_{20}\) alkyl, aryl, alkylaryl or arylalkyl group, A represents a linear or branched divalent alkylene group;
b) and at least one alcohol ethoxylate and/or amino alcohol ethoxylate surfactant.

**Detailed Description Of The Invention**

According to a preferred embodiment of the invention, the dicarboxylic acid diester is provided in the form of a mixture of different dicarboxylic acid diesters of formula (I).

Preferably, A is a branched divalent C₃-C₁₀ alkylene group.

More preferably, A is chosen from the group comprising the AMG group of formula -CH(CH₃)-CH₂-CH₂⁻, the AES group of formula -CH(C₂H₅)-CH₂- and mixtures thereof.

According to a preferred embodiment of the present invention, the dicarboxylic acid diester is provided in the form of a mixture comprising the dicarboxylic acid diesters of following formulae (I’), (I’’) and optionally (II):

- R¹-OOC-AMG-COO-R² (I’),
- R¹-OOC-AES-COO-R² (I’’),
- optionally R¹-OOC-(CH₂)₄-COO-R² (II),

wherein:

- AMG is a group of formula -CH(CH₃)-CH₂-CH₂⁻,
- AES is a group of formula -CH(C₂H₅)-CH₂⁻,
- R¹ and R² are identical or different, and represent a linear or branched, cyclic or noncyclic, C₁-C₂₀ alkyl, aryl, alkylaryl or arylalkyl group.
In the composition of the present invention, \( R^1 \) and \( R^2 \) groups are preferably methyl groups.

Particularly, the mixture of dicarboxylic acid diester comprises:

- from 70 to 95\% by weight of the dicarboxylic acid diester of formula (\( \Gamma \)),
- from 5 to 30\% by weight of the dicarboxylic acid diester of formula (\( \Gamma' \)), and
- from 0 to 10\% by weight of the dicarboxylic acid diester of formula (II).

According to the present invention, the surfactant b) is preferably a mixture of alcohol ethoxylates and amino alcohol ethoxylates, comprising the compounds of the following formula (III) and (IIF):

\[
R^3(OCH_2CH_2)_x\cdot OH \quad (III)
\]
\[
R^4N[(CH_2CH_2O)_y\cdot H][(CH_2CH_2O)_z\cdot H] \quad (IIF)
\]

in which:
- \( R^3 \) is a linear or branched alkyl group having between 6 and 22 carbon atoms, preferably between 10 and 13,
- \( x \) is between 3 and 30, preferably between 8 and 10,
- \( R^4 \) is a linear or branched alkyl or alkenyl group having between 6 and 22 carbon atoms, preferably between 12 and 18,
- \( y+z \) is between 5 and 20, preferably between 10 and 15.

Compounds of formulae III and IIF correspond to mixtures with different carbon chains.

Preferably, the surfactant b) is a mixture, preferably in form of an emulsion,
comprising 30 to 60% by weight, preferably 50% by weight of the compound of formula (III), 10 to 40% by weight, preferably 30% by weight of the compound of formula (III') and 5 to 30% by weight, preferably 20% by weight of water.

In a specific embodiment of the present invention, the ratio by weight of the surfactant b) to the dicarboxylic acid diester b)/a) is between 9/1 and 5/5, preferably between 8/2 and 7/3.

In a particular embodiment of the present invention, the composition comprises:

a) from 10 to 50% w/w, preferably from 20 to 30% w/w of a mixture comprising:
   - from 70 to 95% by weight of the dicarboxylic acid diester of formula (I'),
   - from 5 to 30% by weight of the dicarboxylic acid diester of formula (I''), and
   - from 0 to 10% by weight of the dicarboxylic acid diester of formula (II);

b) from 50 to 90% w/w, preferably from 70 to 80% w/w of a mixture comprising:
   - from 30 to 60% by weight of the compound of formula (III),
   - from 10 to 40% by weight of the compound of formula (III'),
   and
   - from 5 to 30% by weight of water.

A particular composition of the present invention consists of a mixture of alcohol ethoxylates and amino alcohol ethoxylates, for example sent by
Rhodia under the reference Rhodaterge® 218, and a mixture of branched dibasic esters, for example sent by Rhodia under the reference Rhodiasolv® IRIS, as follows:

- from 50 to 90% by weight of Rhodaterge® 218 comprising:

  50% by weight of \(\text{CH}_3(\text{CH}_2)_{10-15}\text{CH}2\text{O}_{8-10}\text{H},\)
  
  30% by weight of \(\text{CH}_3(\text{CH}_2)_{10-16}\text{CHN}[(\text{CH}_2\text{CH}_2\text{O})_y\text{H}][(\text{CH}_2\text{CH}_2\text{O})_z\text{H}],\)
  
  with \(y+z = 10-15,\) and
  
  20% by weight of water;

- from 10 to 50% by weight of Rhodiasolv® IRIS comprising:

  85% of dimethyl methyl glutarate,

  \[
  \begin{align*}
    &\text{H}_3\text{COOC} \xrightarrow{\text{CH}_2\text{CH}_2\text{COOH}} \\
    &\text{COOCH}_3
  \end{align*}
  \]

  from 5 to 15% by weight of dimethyl ethyl succinate, and
  
  from 0 to 10% by weight of dimethyl adipate.

The present invention also aims the use of the above defined composition for the scouring of wool.

In a specific use according to the present invention, the concentration of the composition is less than 10%, preferably between 0.5 and 5%, and more preferably still between 0.8 and 1% w/w in water.

The cleaning performance of the invention is significantly better in-
comparison to the state of the art.

One of the advantages of the present invention is that there is less residual surfactant in the centrifuged grease. Moreover, the formulation makes stable micro-emulsion at concentration of below 10% in water. In addition, the preferred used dosage for wool scouring is 0.8-1% w/w of the composition of the present invention in water.

Besides, the surfactants and diester solvents according to the present invention are easily biodegradable.

The cleaning of the wool is often carried out in two steps. The first step is a washing step at a temperature preferably comprised between 30 and 70°C. The second step is a rinsing step in which the wool is rinsed at least one time with "cold" water at a temperature comprised between 10 and 20°C.

The application of the product would be in the wool fiber cleaning market, where it can be used as a cleaning detergent in the scouring of wool or as an antistatic agent in the use for cabling. The preferred applied dosage can be as low as 0.8 - 1% w/w.

**Description of Drawings**

Figure 1 represents three sheep fleece samples: before treatment, after a treatment according to comparative example 2 and after a treatment according to example 2.

**Examples**

The present invention promotes better cleaning and fluffier wool when compared to current art.
Test conditions are conducted as follow:

**Equipments:**

Launder-o-meter: SDLATLAS M228 Rotawash
Drying oven: Memmert UNB500
Whiteness and color meter: Wenzhou Jiayi Instrument Co. Ltd. JY9800

**Test Conditions:**

Wash time: 5 min
Rinse time: 4 min (under flowing water)
Wash temperature: 60°C
Drying temperature: 96°C

10 grams of crude wool is placed in 1 liter of water with 10 grams of scouring product. This mixture is put into the capsule of the launder-o-meter. 50 stainless steel ball bearings are added into the capsules to increase mechanical agitation, and a separate capsule is used of each sample.

Samples that are tested are as follows and in the same order in chart 1

**Comparative example 1**: Rhodaterge® 218

**Comparative example 2**: NP-9 Nonylphenol 9 ethoxylate

**Example 1**: Rhodaterge® 218 + Rhodiasolv® IRIS (DBE) (8:2)

**Example 2**: Rhodaterge® 218 + Rhodiasolv® IRIS (DBE) (7:3)

The temperature set of the water bath in the launder-o-meter is 60°C and the wash time is set at 5 minutes, rotation speed of the launder-o-meter for agitation of capsules is fixed at 40±2 rpm. After the washing is complete, the
wool is rinsed under cool running water (10-20°C) at room temperature for 4 minutes. The moist wool is then subjected to 96°C in the oven until it is completely dried.

Whiteness of the treated wool is taken using a color-meter, under which the whiteness is represented by two different indices, Berger and Taube, the higher the value of the index the whiter the material tested.

The whiteness equipment can separately give two values:

\[ W_{\text{Berger}} = 0.333 Y + 125Z/Zn - 125X/Xn; \]
\[ W_{\text{Taube}} = 400Z/Zn - 3Y. \]

The CIE Color Systems utilize three coordinates to locate a color in a color space, whiteness instrument can test the values of X, Y and Z using CIE XYZ standard at Standard Illuminant D65/10°. Zn and Xn are the calibrated values with the reference standard working white plate.

Result of the experiment is presented below.

**Chart 1.**

<table>
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<tr>
<th>Composition</th>
<th>Comparative example 1</th>
<th>Comparative example 2</th>
<th>Example 1</th>
<th>Example 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rhodaterge® 218</td>
<td>100</td>
<td>0</td>
<td>80</td>
<td>70</td>
</tr>
<tr>
<td>Rhodiasolv® IRIS</td>
<td>0</td>
<td>0</td>
<td>20</td>
<td>30</td>
</tr>
<tr>
<td>NP-9</td>
<td>0</td>
<td>100</td>
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<td>0</td>
</tr>
<tr>
<td>BERGER</td>
<td>5.92</td>
<td>3</td>
<td>6.45</td>
<td>6.86</td>
</tr>
<tr>
<td>TAUBE</td>
<td>22.93</td>
<td>19.15</td>
<td>25.53</td>
<td>28.59</td>
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</table>

It is shown that the cleaning effect of Rhodaterge® 218 is better than NP-9 when comparing the Berger and Taube indices which are two values to indicate the whiteness. Tests with different ratios show that the best
performance comes when the ratio of Rhodaterge® 218 to Rhodiasolv® IRIS is 7 to 3.

The composition of the present invention not only is a wool cleaner, but also makes the wool evidently fluffier than that washed with NP-9, where matting of wool is observed, as shown in Figure 1.
CLAIMS

1. Wool scouring composition comprising:

   a) at least one dicarboxylic acid diester corresponding to the formula (I):

   \[ \text{R}^\text{OOC-A-COO-R}^2 \quad \text{(I)} \]

   wherein

   - \( R^1 \) and \( R^2 \) are identical or different, and represent a linear or branched, cyclic or noncyclic, \( C_{1-20} \) alkyl, aryl, alkylaryl or arylalkyl group,
   - \( A \) represents a linear or branched divalent alkylene group;

   b) and at least one alcohol ethoxylate and/or amino alcohol ethoxylate surfactant.

2. Composition as claimed in claim 1, wherein the dicarboxylic acid diester is provided in the form of a mixture of different dicarboxylic acid diesters of formula (I).

3. Composition as claimed in claim 1 or 2, wherein \( A \) is a branched divalent \( C_{3-10} \) alkylene group.

4. Composition as claimed in any one of the preceding claims, wherein \( A \) is chosen from the group comprising the \( A_{MG} \) group of formula \(-\text{CH(CH}_3)\text{-CH}_2\text{-CH}_2^-\), the \( A_{ES} \) group of formula \(-\text{CH}(_2\text{H}_5)\text{-CH}_2^-\), and mixtures thereof.

5. Composition as claimed in any one of the preceding claims, wherein the
dicarboxylic acid diester is provided in the form of a mixture comprising the dicarboxylic acid diesters of following formulae (Γ), (I") and optionally (II):

- $R^\text{OOC} - A_{\text{MG}} - COO - R^2$ (F),
- $R^\text{OOC} - A_{\text{ES}} - COO - R^2$ (Γ'),
- optionally $R^1\text{-OOC-(CH}_2\text{)}_4\text{-COO-R}^2$ (II),

wherein:
- $A_{\text{MG}}$ is a group of formula $-\text{CH(CH}_3\text{-CH}_2\text{-CH}_2\text{-}$,
- $A_{\text{ES}}$ is a group of formula $-\text{CH(CH}_3\text{-)}$-
- $R^1$ and $R^2$ are identical or different, and represent a linear or branched, cyclic or noncyclic, C$_1$-C$_2$0 alkyl, aryl, alkylaryl or arylalkyl group.

6. Composition as claimed in any one of the preceding claims, wherein $R^1$ and $R^2$ groups are methyl groups.

7. Composition as claimed in any one of the preceding claims, wherein the mixture comprises:
- from 70 to 95% by weight of the dicarboxylic acid diester of formula (Γ'),
- from 5 to 30% by weight of the dicarboxylic acid diester of formula (I''),
and
- from 0 to 10% by weight of the dicarboxylic acid diester of formula (II).

8. Composition as claimed in any one of the preceding claims, wherein the surfactant is a mixture of alcohol ethoxylate and amino alcohol ethoxylate, comprising the compounds of the following formula (III) and (III'):

$$R^3(\text{OCH}_2\text{CH}_2)^x\text{-OH (III)}$$
\[ R^4N[(CH_2CH_2O)_x-H][(CH_2CH_2O)_{y'-H}] (\Pi^\Gamma) \]

in which:
- \( R^3 \) is a linear or branched alkyl group having between 6 and 22 carbon atoms, preferably between 10 and 13,
- \( x \) is between 3 and 30, preferably between 8 and 10,
- \( R^4 \) is a linear or branched alkyl or alkenyl group having between 6 and 22 carbon atoms, preferably between 12 and 18,
- \( y+y' \) is between 5 and 20, preferably between 10 and 15.

9. Composition as claimed in any one of the preceding claims, wherein the surfactant \( b) \) is a mixture comprising 30 to 60\% by weight, preferably 50\% by weight of the compound of formula (\( \Pi \)), 10 to 40\% by weight, preferably 30\% by weight of the compound of formula (\( \Pi^\Gamma \)) and 5 to 30\% by weight, preferably 20\% by weight of water.

10. Composition as claimed in any one of the preceding claims, wherein the ratio by weight of the surfactant \( b) \) to the dicarboxylic acid diester \( b)/a) \) is between 9/1 and 5/5, preferably between 8/2 and 7/3.

11. Composition as claimed in any one of the preceding claims 8 to 10, comprising:

a) from 10 to 50\% w/w, preferably from 20 to 30\% w/w of a mixture comprising:
- from 70 to 95\% by weight of the dicarboxylic acid diester of formula (\( \Gamma \)),
- from 5 to 30\% by weight of the dicarboxylic acid diester of formula (\( \Gamma' \)), and
- from 0 to 10\% by weight of the dicarboxylic acid diester of formula (\( \Pi \));
b) from 50 to 90% w/w, preferably from 70 to 80% w/w of a mixture comprising:
   - from 30 to 60% by weight of the compound of formula (III),
   - from 10 to 40% by weight of the compound of formula (IIP), and
   - from 5 to 30% by weight of water.

12. Use of the composition according to any one of claims 1 to 11, for the scouring of wool.

13. The use as claimed in claim 12, wherein the concentration of the composition is less than 10%, preferably between 0.5 and 5%, and more preferably still between 0.8 and 1% w/w in water.
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

see the extra sheet

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC:C11D,D06M, D01C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

CNPAT,CNKI,WPEPODOC: DICARBOXYLIC, DIESTER, DIBASIC, ESTER, CARBOXYLIC, RHODIA, WASH+, ERAS+, ETHOXYLATE, "POLYETHOXY",WOOL?, CASHMERE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
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<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
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<td>X</td>
<td>EP0743358A1(TEXTIL COLOR AG),20 Nov.1996(20.11.1996), paragraphs 3048,005 1.0054-0058,0060</td>
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<td>WO2009013207(AI(RHODIA OPERATIONS),29 Jan. 2009(29.01.2009), claims 1,3,6,7,9, page 2 lines 19-30, page 3 line 9- page 4 line 15, page 12 lines 28-37</td>
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</table>

* Further documents are listed in the continuation of Box C.

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

Further documents included in the international search:

Date of the actual completion of the international search: 18 Oct.2010 (18.10.2010)

Date of mailing of the international search report: 18 Nov. 2010 (18.11.2010)

Name and mailing address of the ISA/CN

The State Intellectual Property Office, the P.R.China

6 Xitucheng Rd., Jinen Bridge, Haidian District, Beijing, China 100088

Facsimile No. 86-10-62019451

Authorized officer: Wang, Chenhong

Telephone No. (86-10) 62084566

Form PCT/ISA/210 (second sheet) (July 2009)
### INTERNATIONAL SEARCH REPORT

**International application No.**

**PCT/CN2010/070523**

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**C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT**

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<td>CN101503648A (SHENZHEN HEMING SCI &amp; TECHNOLOGY LTD), 12 Aug. 2009 (12.08.2009), claims 1,3,5</td>
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<td>WO2009013208A (RHODIA OPERATIONS), 29 Jan. 2009 (29.01.2009), the whole document</td>
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Form PCT/ISA 2/10 (continuation of second sheet) (July 2009)
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Continuation of classification of subject matter:
C11D1/66(2006.01)i
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