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(54) **LUBRICANT COMPOSITION FOR METAL WORKING.**

(57) A lubricant composition for metal working, which comprises: (a) a base oil mainly comprising $C_6$ to $C_{40}$ normal olefin, (b) at least one compound selected from among alcohol, glycol, polyalkylene glycol and its derivative and fatty acid, and/or (c) at least one compound selected between phenol and amine. The use of this composition serves to smooth plastic working, machining, or grinding of various metals and alloys.

EP 0 484 542 A1

## Technical Field

The present invention relates to a lubricating oil composition for metal working, particularly to a lubricating oil composition which comprises a specific type of straight chain olefin, alcohol and the like, and which has excellent detergency (removability) of abrasion dust produced in metal working of aluminum and aluminum alloy.

## Background Art

Generally, when straight chain-α-olefin is used as a lubricant in metal working such as plastic working (rolling or drawing), cutting, and grinding, working dust (abrasion dust) is adhered to the surface of a worked material after the working is finished. This results in deterioration of quality of a worked product and has unfavorable influence on the following working process.

For example, when an aluminum plate is rolled with the straight chain-α-olefin, aluminum abrasion dust is adhered to a rolled plate. If the rolled plate is annealed without removing such dust, annealing irregularity occurs very frequently. In case an aluminum foil is rolled by the use of the straight chain-α-olefin, there is afear that pinholes are made on the aluminum foil because of the presence of abrasion dust. Likewise, abrasion dust is adhered to a worked material in cutting and grinding processes to cause various troubles.

## Disclosure of the Invention

In view of the above, the present inventors have earnestly pursued the studies in order to develop a lubricating oil for metal working which has excellent removability (detergency) of abrasion dust produced in metal working such as plastic working, cutting, and grinding performed on various kinds of metal such as aluminum, steel and brass, and alloys thereof, which is excellent in workability and capable of finishing the surface of a worked material in an excellent condition.

As a result, it has been found out that the above-mentioned object is accomplished when a base oil containing straight chain olefin having 6 to 40 carbon atoms is used together with alcohol, glycol, polyalkylene glycol, or fatty acid.

The present invention is accomplished on the basis of the findings mentioned above.

According to the present invention, there is provided a lubricating oil composition for metal working, comprising (a) a base oil containing, as an essential component, straight chain olefin having 6 to 40 carbon atoms and (b) at least one member of compounds selected from the group consisting of alcohol, glycol, polyalkylene glycol, a derivative of polyalkylene glycol, and fatty acid.

According to the present invention, there is also provided a lubricating oil composition for metal working, comprising (a) the above-mentioned base oil and (c) at least one member of compounds selected from the group consisting of phenolic compounds and amine compounds.

## Best Mode for Carrying out the Invention

In the composition according to the present invention, the straight chain olefin constituting the base oil of the component (a) has 6 to 40 carbon atoms as described above. The straight chain olefin with the carbon atoms of less than 6 is undesirable because of a low flash point. If the number of carbon atoms is more than 40, the practical use is difficult because of a solid state. Furthermore, there become difficult the mixing with and the dissolving in mineral oil, synthetic oil, and other additives. In addition, the straight chain olefin having more than 40 carbon atoms is not popular and is difficult to be available. Among many kinds of the straight chain olefin, the composition having one double bond in a molecule and 6 to 30 carbon atoms is preferable. In particular, α-olefin (namely, n-α-olefin) having 12 to 30 carbon atoms are most preferable.

Specifically, examples for the straight chain olefin may include 1-octene, 1-decene, 1-dodecene, 1-tetradecene, 1-hexadecene, 1-octadecene, 1-eicocene, or mixtures thereof. The straight chain olefin can be produced by various methods. For example, there may be used ethylene oligomer obtained by polymerization of ethylene in an ordinary method.

According to the present invention, the above-mentioned straight chain olefin can be used alone as the base oil of the component (a). Alternatively, mineral oil and/or synthetic oil may be used together with the straight chain olefin. Water may also be added. Mineral oil and synthetic oil to be added have usually a kinematic viscosity of 0.5 to 500 cSt at a temperature of 40 °C, preferably, 0.5 to 30 cSt. V arious kinds of mineral oil can be used, for example, distillate oil obtained through atmospheric distillation of paraffinic

crude oil, intermediate crude oil, or naphthenic crude oil, or through vacuum distillation of residual oil in atmospheric distillation. Alternatively, there may be used refined-oils obtained by refining the distillate oil in a usual method, such as solvent refining, hydrogenation refining, dewax treating, and clay treating. When such mineral oil is mixed with the above-mentioned straight chain olefin, the resultant lubricating oil has an improved oxidation stability.

As synthetic oil, there may be used various types of olefin other than the above-mentioned straight chain olefin (for example, branched chain olefin such as polybutene and polypropylene), and hydrogenated product of such olefin. In particular, polybutene having low molecular weight, polypropylene having low molecular weight, and $\alpha$-olefin oligomer having 8 to 14 carbon atoms are preferable. When such synthetic oil is mixed with the above-mentioned straight chain olefin, the resultant lubricating oil emits less odor during its use so as to improve working environment. Further, degreasing ability on the surface of a worked product is improved.

In case the mineral oil or the synthetic oil is mixed with the above-mentioned straight chain olefin to constitute the base oil of the component (a), the amount of the straight chain olefin is usually at least 3% by weight, preferably 5 to 60% by weight, while the amount of the mineral oil and/or the synthetic oil is at most 97% by weight, preferably, 95 to 40% by weight, although no specific limitation is given to the mixing ratio.

When water is used instead of the mineral oil or the synthetic oil, the lubricating oil of an emulsion type is obtained in which the straight chain olefin is dispersed in water. According to the present invention, the lubricating oil of this type can be used.

Then, as the component (b) in the composition according to the present invention, there is used one member selected from the group consisting of alcohol, glycol, polyalkylene glycol, a derivative of polyalkylene glycol, and fatty acid, or a combination of two or more members.

As alcohol, various alcohols may be used, and among them, aliphatic alcohol is preferable. Branched chain saturated or unsaturated aliphatic alcohol having 6 to 40 carbon atoms (especially 8 to 30 carbon atoms), or straight chain saturated or unsaturated aliphatic alcohol having 6 to 20 carbon atoms (especially 8 to 18 carbon atoms) is more preferable. The alcohol having less than 6 carbon atoms is rapidly consumed due to evaporation or splashing during its use and thus is economically disadvantageous. Branched chain alcohol having more than 40 carbon atoms or straight chain alcohol having more than 20 carbon atoms is sometimes undesirable since it may be insoluble in the base oil of the component (a).

Examples for such alcohol may include octyl (2-ethylhexyl) alcohol, decyl alcohol, lauryl alcohol, myristyl alcohol, cetyl alcohol, stearyl alcohol, eicosyl alcohol, oleyl alcohol, isostearyl alcohol, oxoalcohol, and the like. The amount of the alcohol is not limited, but it is suitable to use 0.05 to 50% by weight, preferably, 0.1 to 20% by weight, based on total amount of the composition.

As to glycol, various kinds of glycol may be used. Generally, there is used glycol having 2 to 6 carbon atoms, for example, ethylene glycol, propylene glycol, trimethylene glycol, 1,4-butanediol, 1,5-pentanediol, and the like. The amount of glycol is usually 0.05 to 50 % by weight, preferably, 0.1 to 20% by weight, based on the total amount of the composition, which is similar to that of the above-mentioned alcohol. Furthermore, there are various kinds of polyalkylene glycol or derivatives thereof. Among them, the compound which is represented by the following general formula (I) is preferable.

$$R^1\text{-}O(R^2\text{-}O)_n\text{-}R^3 \qquad (I)$$

(In the formula, $R^1$ and $R^3$ individually denotes a hydrogen atom, an alkyl group having 1 to 20 carbon atoms, an $C_7$-$C_{24}$- alkyl-substituted phenyl group, or a phenyl group; and $R^2$ denotes an ethylene group, a propylene group, or a butylene group; n is an integer of 2 to 50.)

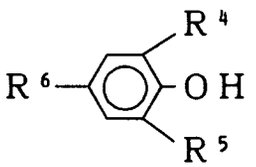
More specifically, poly(n = 6)ethyleneglycol-monobutylether, poly(n = 5)propyleneglycol-monononylether, poly(n = 10)ethyleneglycol-dipropylether, and the like may be used. Glycerol derivative of polyethylene glycol or fatty acid ester of polyethylene glycol can also be used besides the compound given by the above-mentioned general formula (I). The amount of glycol is usually 0.05 to 50% by weight, preferably 0.1 to 20% by weight, based on the total amount of the composition, which is similar to that of the above-mentioned alcohol.

As fatty acid, there are used straight/branched chain saturated/unsaturated fatty acid having 6 to 40 carbon atoms (preferably 8 to 20 carbon atoms). There is a risk that fatty acid having less than 6 carbon atoms are rapidly consumed due to evaporation or splashing during its use, while fatty acid having more than 40 carbon atoms is sometimes insoluble in the base oil of the component (a). Examples for such fatty acid may include caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, arachic acid, behenic acid, isostearic acid, undecylenic acid, oleic acid, linoleic acid, linolenic acid, arachidonic

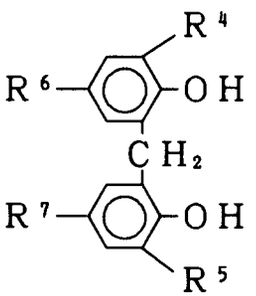
acid, and the like. The amount of fatty acid is usually 0.05 to 1.0% by weight, based on the total amount of the composition. If the amount of fatty acid exceeds 1.0% by weight, the anti-stain ability in annealing is unfavorably deteriorated.

As described, the composition according to the present invention comprises the above-mentioned components (a) and (b). Instead of the component (b) or, alternatively, in addition to the components (a) and (b), it is possible to blend the component (c) comprising at least one compound selected from the group consisting of phenolic compounds and amine compounds.

Various kinds of phenolic compounds can be used. Generally, the phenolic compound represented by the following general formula (II) or (III) is used.



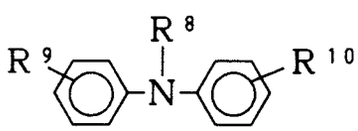
・・・ (Ⅱ)



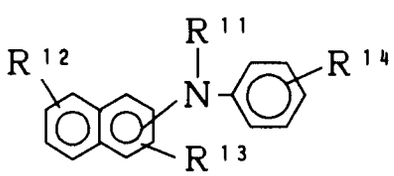
・・・ (Ⅲ)

(In the formula, each of $R^4$ and $R^5$ denotes an alkyl group having 1 to 8 carbon atoms, and each of $R^6$ and $R^7$ denotes a hydrogen atom or an alkyl group having 1 to 6 carbon atoms.) Specifically, 2,6-di-tert-butyl-4-metylphenol (DBPC), 2,6-di-tert-butyl-4-ethylphenol, 2,2'-methylenebis (4-methyl-6-tert-butylphenol), 2,2'-methylenebis (4-ethyl-6-tert-butylphenol), and the like are preferable.

As amine compounds, there are used various amine compounds, and it is suitable to employ usually the compounds represented by the following general formula (IV) or (V):



・・・ (Ⅳ)



・・・ (Ⅴ)

(In the formula, each of $R^8$ and $R^{11}$ denotes a hydrogen atom or an alkyl group having 1 to 10 carbon atoms, each of $R^9$ and $R^{10}$ denotes an alkyl group having 1 to 20 carbon atoms, and each of $R^{12}$ to $R^{14}$ denotes a hydrogen atom or an alkyl group having 1 to 20 carbon atoms.)

Specifically, di-p-octyldiphenylamine, di-p-butyldiphenylamine, di-p-nonyldiphenylamine, phenyl-$\alpha$-naphthylamine, phenyl-$\beta$-naphtylamine, and the like are preferable.

The amount of the component (c) is preferably not less than 0.1% by weight, more preferably 0.1 to 2.0% by weight, based on the total amount of the composition.

If desired, a suitable amount of an oiliness agent, an extreme-pressure agent, a rust inhibitor, a corrosion inhibitor, a defoaming agent, or the like may be blended with the composition according to the present invention.

## Examples

Then, more detailed description will be made regarding the present invention with reference to examples and comparative examples.

## Examples 1 to 12 and Comparative Examples 1 to 3

The following rolling test was carried out by the use of lubricating oil for rolling which has the composition shown in Table 1

## Rolling Test (Aluminum Plate Rolling)

A JIS A 5052 H16 aluminum plate (a coiled plate having a thickness of 1.2 mm and a width of 60 mm) was prepared as a material to be rolled. With each lubricating oil for rolling, the plate was rolled by a four-stage rolling mill having a work roll diameter of 135 mm at a rolling speed of 100 m/min. under forward tensions of 170 kgf and 400 kgf. The rolling process was carried out in such a manner that the reduction was stepwise increased per 20 m length during one pass as 50.0→ 54.2→ 58.3→ 60.8→ 63.3→ 65.8→ 68.3 (%). As to the plate having a thickness of 0.38 mm (a draft of 68.3 %) after the rolling process (after seven passes), the rolling load was measured. Abrasion dust detergency and anti-stain ability were evaluated by the following methods.

(a) Evaluation Method for Abrasion Dust Detergency

(Tape Test Method)

A cellophane tape was stuck onto the surface of the aluminum plate after rolling so as to pick up abrasion dust adhering thereto. Then, the tape was applied on a white paper sheet to visually determine a degree of contamination by abrasion dust. Thus, the detergency for abrasion dust adhering to the surface of the aluminum plate was evaluated. The results thus obtained are shown in Table 1.

(b) Evaluation Method for Anti-stain Ability in Annealing

The aluminum plate after rolling was cut into piece having a short length of 10 cm. The stack of several tens of the cut pieces were fixed by a thick steel plate and annealed in a small-size annealing furnace.

The heating process in the annealing furnace comprises the step of: heating in an air atmosphere with a temperature rising rate of 5 °C/min. until the temperature reaches 330 °C; holding for 30 minutes; and cooling. After completion of the heating process, a degree of occurrence of annealing stains on the annealed plate was visually determined. Thus, the anti-stain ability was evaluated. The results are shown in Table 1.

Table 1

| | Example | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
| Composition (% by weight) | | | | | | | | | | | | |
| n-$\alpha$-olefin *1 | 40 | 40 | 40 | 30 | 30 | 30 | 30 | 30 | 30 | 95 | 40 | 40 |
| Mineral oil *2 | 56.5 | 54.5 | 54.5 | 69 | 69 | 69 | 65 | 60 | — | — | 59.5 | 59.5 |
| Polybutene *3 | — | — | — | — | — | — | — | — | 69 | — | — | — |
| Decyl alcohol | 3 | — | — | — | — | — | — | — | — | — | — | — |
| Lauryl alcohol | — | 5 | — | — | — | — | 5 | — | — | 5 | — | — |
| Oleyl alcohol | — | — | — | — | — | 1 | — | — | — | — | — | — |
| Oxo alcohol | — | — | 5 | — | — | — | — | 10 | — | — | — | — |
| Lauric acid | — | — | — | 0.5 | — | — | — | — | 1 | — | — | — |
| Oleic acid | — | — | — | — | 0.5 | — | — | — | — | — | — | — |
| 2,6-di-t-butyl-4-methylphenol | 0.5 | 0.5 | 0.5 | 0.5 | — | — | — | — | — | — | 0.5 | — |
| Dioctyl diphenylamine | — | — | — | — | 0.5 | — | — | — | — | — | — | 0.5 |
| Methyl laurate | — | — | — | — | — | — | — | — | — | — | — | — |
| Abrasion dust detergency *4 | ○ | ○ | ○ | ○ | ○ | ○ | ○ | ○ | ○ | ○ | △ | △ |
| Anti-stain ability *5 | ○ | ○ | ○ | ○ | ○ | △ | △ | △ | △ | △~× | △ | △ |
| Rolling load (ton) | 14.6 | 14.2 | 14.1 | — | — | — | — | 14.7 | — | — | 14.6 | — |

*1 An equivalent mixture of 1-hexadecene and 1-octadecene

*2 A paraffinic mineral oil having a kinematic viscosity of 4cSt at 40℃

*3 The one having a kinematic viscosity of 6cSt at 40℃

*4 ○, △, × indicate little contamination, some contamination, and much contamination, respectively.

*5 ○, △, × indicate little stain, some stain, and much stain, respectively.

Table 1 (continued)

| | Comparative Example | | |
|---|---|---|---|
| | 1 | 2 | 3 |
| Composition (% by weight) | | | |
| n-$\alpha$-olefin *1 | 100 | 97 | — |
| Mineral oil *2 | — | — | 94.5 |
| Polybutene *3 | — | — | — |
| Decyl alcohol | — | — | — |
| Lauryl alcohol | — | — | 5 |
| Oleyl alcohol | — | — | — |
| Oxo alcohol | — | — | — |
| Lauric acid | — | — | — |
| Oleic acid | — | — | — |
| 2,6-di-t-butyl-4-methylphenol | — | — | 0.5 |
| Dioctyl diphenylamine | — | — | — |
| Methyl laurate | — | 3 | — |
| Abrasion dust detergency *4 | × | × | ○ |
| Anti-stain ability *5 | × | × | ○ |
| Rolling load (ton) | — | — | Unable |

*1 An equivalent mixture of 1-hexadecene and 1-octadecene

*2 A paraffinic mineral oil having a kinematic viscosity of 4cSt at 40°C

*3 The one having a kinematic viscosity of 6cSt at 40°C

*4 ○, △, × indicate little contamination, some contamination, and much contamination, respectively.

*5 ○, △, × indicate little stain, some stain, and much stain, respectively.

Examples 13 and 14 and Comparative Examples 4 and 5

The following cutting test was carried out by the use of lubricating oil for cutting which has the composition shown in Table 2.

Cutting Test (Cutting for End Face of Cylinder)

A cylindrical aluminum alloy material (AC-4A-T6) having a diameter of 80 mm was prepared as a material to be cut. The aluminum alloy material was cut to form an end face by the use of a cutting tool (cemented carbide P20) under the cutting condition that a cutting depth (t) is 1 mm and a feed rate (f) is 0.1 mm/revolution.

After the cutting, the surface roughness ($R_{max}$) of the cut end face was measured while the surface thereof was observed. The results are shown in Table 2.

Table 2

| | Example 13 | Example 14 | Comparative Example 4 | Comparative Example 5 |
|---|---|---|---|---|
| n-α-olefin *1 (wt.%) | 40 | 90 | 100 | — |
| Mineral oil *2 (wt.%) | 50 | — | — | 100 |
| Lauryl alcohol (wt.%) | 10 | 10 | — | — |
| Roughness ($R_{max}$)(μm) | 2 | 2 | 5 | 10 |
| Result of observation | Feed mark alone (theoretical roughness) | Feed mark alone (theoretical roughness) | Disturbance in feed mark | Fragments dropping from the built-up edge are adhered |

*1,2 Same as those in Table 1

Industrial Applicability

As described above, by the use of a lubricating oil composition according to the present invention, abrasion dust detergency is improved in metal working (plastic working such as rolling, drawing, blanking, drawing, cold forging; cutting; and grinding) for various kinds of metals and alloys thereof while anti-stain ability in annealing of a worked material is improved.

Accordingly, a lubricating oil composition according to the present invention is widely and effectively used as a metal working oil in plastic working, cutting, or grinding of various kinds of metals and alloys such as aluminum, aluminum alloy, aluminum foil.

## Claims

1. A lubricating oil composition for metal working, comprising: (a) a base oil containing, as an essential component, straight chain olefin having 6 to 40 carbon atoms; and (b) at least one compound selected from the group consisting of alcohol, glycol, polyalkylene glycol, derivative of polyalkylene glycol, and fatty acid.

2. A lubricating oil composition for metal working, comprising: (a) a base oil which is a mixture of straight chain olefin having 6 to 40 carbon atoms and mineral oil and/or synthetic oil; (b) at least one compound selected from the group consisting of alcohol, glycol, polyalkylene glycol, derivative of polyalkylene glycol and fatty acid.

3. A lubricating oil composition for metal working according to Claim 1 or 2, further comprising the additional component of (c) at least one compound selected from the group consisting of phenolic compounds and amine compounds.

4. A lubricating oil composition for metal working, comprising: (a) a base oil containing, as an essential component, straight chain olefin having 6 to 40 carbon atoms; and (c) at least one compound selected from the group consisting of phenolic compounds and amine compounds.

5. A lubricating oil composition for metal working, comprising: (a) a base oil which is mixture of straight chain olefin having 6 to 40 carbon atoms and mineral oil and/or synthetic oil; and (c) at least one compound selected from the group consisting of phenolic compounds and amine compounds.

# INTERNATIONAL SEARCH REPORT

International Application No PCT/JP91/00534

**I. CLASSIFICATION OF SUBJECT MATTER** (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC Int. Cl[5] C10M105/04, C10M169/04//(C10M169/04,C10M105:04,C10M129:06,C10M129:08,C10M129:40, C10M133:12,C10M129:10),C10N40:24,C10N40:22,C10N30:04

**II. FIELDS SEARCHED**

Minimum Documentation Searched [7]

| Classification System | Classification Symbols |
|---|---|
| IPC | C10M105/04, C10M169/00-04, C10N40:22-24 |

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

**III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]**

| Category [10] | Citation of Document, [11] with indication, where appropriate, of the relevant passages [12] | Relevant to Claim No. [13] |
|---|---|---|
| Y | JP, A, 2-84495 (Hitachi, Ltd.), March 26, 1990 (26. 03. 90), Example & EP, 341,688 | 1-2 |
| Y | JP, A, 1-282,297 (Hitachi, Ltd.), November 14, 1989 (14. 11. 89), Claim (Family: none) | 1-2 |
| Y | JP, A, 1-153794 (Hakuto Kagaku K.K., Showa Aluminum Ltd.), June 15, 1989 (15. 06. 89), Pages 2 to 4 (Family: none) | 1-5 |
| Y | JP, A, 63-202697 (Nippon Grease K.K.), August 22, 1988 (22. 08. 88), Page 3 (Family: none) | 3-5 |
| Y | EP, A, 48,216 (Schweiz Aluminium Ltd.), March 24, 1982 (24. 03. 82), Claim & US, A, 4,326,974 & JP, A, 57-80496 | 1-2 |

* Special categories of cited documents: [10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"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

"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

"&" document member of the same patent family

**IV. CERTIFICATION**

| Date of the Actual Completion of the International Search | Date of Mailing of this International Search Report |
|---|---|
| July 11, 1991 (11. 07. 91) | July 29, 1991 (29. 07. 91) |
| **International Searching Authority** | **Signature of Authorized Officer** |
| Japanese Patent Office | |

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

| | | |
|---|---|---|
| P | EP, A, 369,320 (Idemitsu Kosan Co., Ltd.), May 23, 1990 (23. 05. 90), Claim & JP, A, 2-133495 & JP, A, 2-281097 | 1-5 |

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [1]

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers . because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claim numbers , because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claim numbers , because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [2]

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.