**Title:** INKS FOR MARKERS AND PENS

**Abstract**

The invention relates to inks, particularly marker inks and is especially concerned with the problem known to be associated with conventional inks, the drying out of ink in the tip or point of a marker pen or the nib of a pen. This problem has been the subject of considerable research with various proposals for the incorporation of an anti-drying agent. The object of the present invention is to provide an improved ink that provides a substantial retardation of the drying out of the ink over many days and at the same time leave the pen in a condition for the immediate commencement of marking or writing. This objective is met by an ink that includes a dispersion of a wax or wax-like material. The invention also involves a process for preparing an ink employing an organic solvent, and comprises preparing an ink product by heating to a temperature in excess of the melting point of a selected wax or wax-like material and at which temperature the wax or wax-like material will be soluble, adding said wax or wax-like material to said ink product and dissolving the wax to obtain a homogeneous solution, allowing the combination of wax and ink to cool such that the wax becomes essentially insoluble and creates a fine dispersion of wax in the ink.
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INKS FOR MARKERS AND PENS

This invention relates to inks and is particularly but not necessarily exclusively concerned with marker inks.

Marker and pen products in general have provided as a part of their construction and disposition a means of closing off the writing tip or point from the atmosphere in order to prevent the tip or marking nib from drying out. This facility is usually provided by means of an air tight cap or other closure. With inks of current design should said cap or closure be removed and not replaced the ink evaporates and the ink dries out. The drying out mechanism often proceeds to completion when the cap is left off for prolonged periods. The result is invariably a product which, whilst it still may contain a considerable portion of the original quantity of ink, is unusable and is therefore rejected or disposed of. Occasionally it is possible to revivify the product by replacing the cap if the exposure time minus the cap is not too great. The performance of the product however in this case usually greatly impaired and may still be rejected.

There have been many ways proposed and utilised to overcome some aspects of this process usually with the employment of solvent(s) which are non or slowly volatile in company with the major solvent content. For example water based inks have traditionally employed organic solvents of the glycol type in an attempt to retard the evaporation process. However when utilised in this manner glycols have the drawback of retarding the drying process even when upon paper and may also affect the manner and extent to which the ink penetrates paper. The rate of ink drying and its visual perception and other qualities such as the sharpness of line may also therefore be impaired. Similar high boiling, involatile additives may be be employed to a small extent in order to assist in solvent based ink products. However owing to the need for these inks to dry rapidly without tackiness on impermeable substrates, the degree to which the non dry out aspect of these type of inks can be enhanced is very limited. It is specifically in this connection that the current invention is to be employed. Water based inks on the other hand have reached a
state of development where improvements in the exposed or cap off state utilising additives, whilst easily demonstrated, yield no great advantage in performance or cost as compared to existing products. The demonstration of such ink qualities using agents dissolved or dispersed into the ink medium, are therefore more of a curiosity value than utility.

In so far as the prior art is concerned, mention can be made to such as GB A 208 991 which reveals an ink that is water based and contains a pigment or pigments, a dispersing agent and an additive that prevents drying, the latter being referred to as an "anti-drying" agent. The anti-drying agent was noted to retard the drying out of the ink in the point or nib although no mechanism was proposed for this. The anti-drying agent is stated to be urea, thiourea or water soluble derivatives thereof. This seminal document teaches for the first time that the use of dissolved additives other than solvents can be used to retard the drying process in capillary systems.

JP-A-60-84369 describes an ink for marking pens containing an ink based upon volatile alcohol or glycol ether and employing a sucrose ester as the "anti-drying" agent noted in the above GB application. The sucrose ester is a solid white powder. 

JP-A-63-61065 teaches that the use of ascorbic acid and its derivatives may be employed in marker inks expressly for the purpose of preventing the drying out of the marker or pen tip whilst the cap has been removed. It is expressly noted therein that the reason or mechanism invoked by the ascorbic acid is not known. However the effect is noted and claimed.

US Patent 4,525,216 describes the use of particular glycerides, alkylamide alcohol and Sorbitan esters as evaporation reduction additives in a specific ink known as white board marker inks and which contain as principle components pigment(s), resin(s) and what are described as a separating agents but which those skilled in the art would recognise as release agents. The additives which impart the claimed solvent evaporation reduction attribute are stated to be wholly soluble in the solvent or solvent blends employed in the ink(s). Specifically the
mechanism of action by which the additives achieve the effect is by being precipitated at the solvent air interface when the solvent in the ink evaporates thereby forming a film which is later reincorporated into the ink when the product containing the ink is used and the film is disturbed.

In US Patent 5,279,652 there is described the use of a wide range of organic and some inorganic solid materials, all being solid materials at temperatures equal to or greater than 30°C Celsius and having molecular weights of \(<\) 500 and which exhibit a defining characteristic such that when incorporated in an ink, they crystallise at an air/liquid interface. Such interface being further defined by being the opening of a capillary structure wherein the ink is exposed to atmosphere. The mechanism behind such anti-drying behaviour is expressly noted as is also the range of concentrations useful in the described art. This patent is the most comprehensive in the field of prior art and claims a wide range of solid materials which exhibit the desired characteristic of forming a crystal structure under the conditions defined therein. The salient features of the claims being that the materials are all solid (M.P. greater than 30°C), are completely soluble in the solvent(s) employed and have a molecular weight equal or less than 500 Daltons.

The object of the current invention is to provide a means whereby the cap or closable portion of a marking or writing implement employing an ink based upon volatile solvent may be removed and left such that the product is in the unsealed mode for prolonged periods, usually many days, and yet remains immediately available for use.

According to a first aspect of the present invention an ink suitable for use in marker pens includes a dispersion of a wax or wax like material throughout the ink.

According to a second aspect of the present invention a process for preparing an improved marking ink employing an organic solvent comprises preparing an ink product by heating to a temperature in excess of the melting point of a selected wax or wax like material and at which temperature the wax of wax like material will be soluble, adding said wax or wax like material to said ink product and dissolving the wax to obtain an homogenous solution, allowing
the combination of wax and ink to cool such that the wax becomes essentially insoluble and creates a fine dispersion of wax in the ink.

Alternatively, the solution of wax and ink may be shock chilled by the addition of cool ink to the already hot solution of wax in ink.

Using such adjusted ink in an ink pen or marker to achieve a product that resists the dry out of the nib or point when the cap or closable portion of the product is removed or left open. This attribute is made available without in any marked or detrimental manner, impairing the performance of the ink as to drying time and other useful properties known to those skilled in the art of marker and pen ink manufacture. It is therefore the express purpose of this current application to note the above prior art and to provide an improvement whereby the demerits of the above techniques and those herein described, are avoided. The invention will provide for the use of additives which exhibit a notable anti-drying behaviour, which are waxy, liquid or semi-liquid at temperatures of less than 30° Celsius and below and which are largely, if not completely insoluble in the solvents employed in the ink or marking liquid vehicle. Finally and most importantly the additives will be shown to exhibit the defining characteristic of retarding the evaporation of the principle vehicle or solvent of a marking or writing ink to such an extent as to permit the cap or closure of the product to be removed and the pen or marker left in the un-capped state for prolonged periods. Further the concentration(s) of the active components in the finished ink are held to levels far below those at which prior art inclusions have proven to be effective, specifically and notably below one percent.

It has been found that the usage of solid materials added to an ink and dissolved therein as described in the above citations to achieve a desirable attribute of having an "anti-drying" capability all have the distinct disadvantage that the resultant dry ink film produced by such an ink suffers poor adhesion to many substrates, may not be water resistant and have poor gloss; none of which are desirable characteristics. It is preferable for any such ink film produced as a result of a
marker or pen ink usage and characterised as being of a permanent nature (Resistant to water and the elements as opposed to being lightfast) to have good water resistance, to have good adhesion to the applied surface and to exhibit a gloss. It is found that of the solid crystalline additives described in prior art when used at effective concentrations and when deposited as an ink film or more correctly as part of an ink film, produce relatively large crystal growths when the ink dries and these subsequently impair the integrity of the ink film to such an extent that the desirable characteristics noted above are jeopardised.

It has also been found that of the materials claimed in prior art and which are usable in organic solvent in amounts which are both sensible and cost effective, only very few have any true utility. As a class of materials, organic acids, more especially aromatic organic acids which are claimed in US patent 5,279,652 are relatively inexpensive and exhibit the desirable characteristics thereby permitting their use. In particular the isomeric forms of Methyl Benzoic Acid have been shown to be very effective in preventing the evaporation of solvent from the exposed surfaces of capillary conduits like for example openings at the extremities of marking nibs. Especially is this true when used in company with solvents of the simple aliphatic alcohol type. However this material when used as described yields very poor ink films and is for the most part by itself, useless as an ink additive. Also the material Phenanthrene as also claimed in the above patent when used in a similar manner and in solvent types and blends such as glycol ethers also demonstrates similar problems.

It is the intent behind the current application to show that wax like substances in particular those wax like materials derived from vegetable and animal fat sources can be added to inks in a suitable form or manner and which exhibit the characteristics of being insoluble or largely insoluble in the ink solvents employed, and further which can be demonstrated to form thin flake like lamellae when suspended in said solvent(s) and when prepared in the manner herein described.
It is the purpose of this invention to reduce solvent loss by evaporation from an ink as may be used in a marker or pen by employing wax like materials. Such wax or wax like materials are preferably added as an additive employing a carrier system. The wax or wax like materials form microscopic flakes in the marker ink. These flakes or lamellae readily align themselves one against another when subjected to the convection currents produced by the evaporative loss of solvent, forming at the solvent / air interface a thin barrier of overlapping platelets. This barrier effectively slows down the further loss of solvent to the point where losses become some orders of magnitude less than in the unprotected system. A vital part of this scheme is the fact that the wax like material should exhibit very low solubility in the ink solvent being employed such that migration through the platelets is low and again evaporative loss is prevented. At the same time the wax or wax like material whilst not being soluble in the solvent(s) employed should form stable micro dispersions of colloidal or near colloidal particle size.

Further the manner in which the wax or wax like like material is prepared for use in said ink systems affects desired result. The invention as detailed below will show that the wax or wax like materials will function if added directly to the ink but the effect is enhanced when a carrier material is carefully selected for the wax. Any such carrier material should be fully compatible with the ink formula being considered not only in the wet state (Solvent containing) but also in the dry or cured ink film. The carrier material must not be a true solvent for the wax like component, but should exhibit a small amount of affinity for it such that when the wax dispersion is prepared a stable, micron sized dispersion of the wax is formed and which readily demonstrates thin flake like lamellae as noted above.

The wax or wax like material suited for use in the present invention may be selected from the following non exhaustive listing.

Paraffin Wax.

Microcrystalline Paraffin Wax.
Carboxylic acid esters of Sterols.

Beeswax.

High Molecular Weight Fatty acids and Carboxylic Acids.

Mono and Di Glyceride Phosphate Esters.

Carnauba Wax.

Ethylene Glycol Di Esters of Fatty Acids.

Higher Fatty Acids.

Higher Fatty Alcohols.

Higher Fatty Alcohol Esters.

Higher Fatty Alcohol Fatty Acid Esters. e.g.

Cetyl Stearate.

Cetyl Palmitate.

Cetyl Stearyl Stearate.

Myristyl Myristate.

Hydrogenated Castor Oil.

Spermaceti - Natural & Synthetic.

Poly Ethoxylated Lanolin Alcohols.

Hydrogenated Lanolin.

Ozokerite Wax.

Montan Wax.

PolyAmide Wax.

The invention may be characterised by the following example. A Normal Propyl Alcohol based ink utilising a Black dye blend and a ketone resin film former is enhanced as to its ability to resist dry out of the nib when used in a marker pen by taking said ink and dissolving into it by means of intense mixing and under the influence of heat, a proportion of wax, in the order of
0.75% W/W namely Cetyl Palmitate. Ideally the ink should then be rapidly cooled so as to generate the flake like condition of the wax noted above.

Preferably however, the wax should be added to the ink in the form of a premix or additive, made as follows. A cetyl stearyl alcohol ester wax for example is added to a refined vegetable oil in a heated mixing vessel. The quantity of wax compared to oil is in the region 1 - 50% typically 10 - 20%. The mixture is heated through and to beyond the melting point of the wax typically circa 70° Celsius. Whilst stirring is continued the whole mixture is then allowed to cool or alternatively the dispersion as now formed is run off into collection vessels where more rapid cooling takes place. Alternatively only a portion of the required amount of oil is used to melt and combine with the wax material. After complete dissolution of the wax has occurred at temperature, the remaining oil is quickly added with thorough mixing to shock cool the whole blend to a temperature below the melting point of the wax. The relative amounts of the initial mix and the later added portion may be easily calculated to ensure that the correct temperature range is achieved following the last addition of oil.

The wax when added directly or in the form of a pre-dispersion in the proportion of 1 - 5% W/W into an ink (giving in all cases a maximum of 1% wax W/W in the ink), and then spotted onto a microscope slide exhibit clearly under low magnification the rapid migration of the platelets of wax to the surface where a thin semi permeable barrier rapidly forms. Hence the mechanism employed in this new device as compared with prior art is the aggregation of previously formed platelets or lamellae at the evaporating surface and which collectively form a barrier to said evaporation. In one system noted above the additives are required to be present in such quantities such that upon the loss of solvent from the ink, crystallisation occurs rapidly and uniformly owing to the limiting solubility of the additive in the solvent. In other systems claimed the solubilised oil based material is precipitated at the solvent air interface and requires much more in the way of active material than the present invention owing to the mechanism employed.

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As noted the present invention employs significantly less active component and is therefore much more effective economically and technically. The latter being accomplished by the relatively small amount of addition which naturally has far less impact upon the required performances of the ink and ink film than other systems.

It has been found that a wide variety of wax like materials may be employed to give the required effect. Additionally there have been found a selection of carrier liquids that can be selected to achieve the effect as required. Typically an ink manufacturer may choose a carrier liquid that is compatible with his ink ingredients and a wax material type and concentration that imparts the required performance to his ink. Alternatively by careful selection of the wax and carrier, an additive may be prepared which can be sold and utilised as a "Universal" additive to complement a wide range of ink formulae made and sold throughout the marker industry. It is the experience of the inventor that such inks will commonly employ the lower alcohols, glycol ethers and esters. It is likely that inks based upon simple aliphatic and aromatic solvents may have too much solvency for the wax like materials such that the anti-drying attribute will be diminished possibly to the point of ineffectivity.

Where used the carrier liquid may be selected from but not limited to the following and includes blends of said materials:-

- Castor oil.
- Liquid Paraffin.
- Vegetable oils.
- Capralactone and Poly Capralactone.
- Propylene carbonate.
- Polyether polyols.
- Polyester polyols.

The invention will now be illustrated by means of the following examples.
Black Ink

Mix.

N-Propanol 80
Propylene Glycol Mono Methyl Ether 10
Ketone Resin 5
Solvent Black 29 10

Cetyl Stearyl Alcohol 0.75.

Raise the temperature of the ink to 70°C Celsius with thorough intense shear mixing and then cool as rapidly as possible.

The result is a Black alcohol based marker ink that exhibits an open cap life of approximately one week when used with appropriate marker components for example:

Polyester fibre based reservoir capillarity 8cms.
Acrylic fibre based marker nib
Polypropylene marker components.

It should be emphasised at this point that the results achieved in terms of open cap life are very much dependent upon the marker or pen components utilised in company with the ink. Whilst the ink determines the type of effect achievable, the magnitude of it and thereby the open cap life is attained by the product as a whole. Altering for example the nib from a high flow relatively soft tip to a lower flow, denser type can reduce the open cap life by perhaps an order of magnitude.

Red Ink

Premix

Myristyl Myristate 20.
Vegetable Oil 80.

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Mix

N-Propanol 80
Propylene Glycol Mono Methyl Ether 5
Ketone Resin 4.5
Neopen Red 366 4
C.I.Pigment Red 112 dispersion* 4

Add 4% of the premix and stir thoroughly.

This Red ink achieves 6-8 days cap off life dependant upon conditions and components.

**Blue Ink**

Premix.

10

Cetyl Stearyl Stearate 10
Capralactone 90

Mix.

Iso Propanol 80
Orasol Blue 2GLN 6.

15

Ketone Resin 5

Add 5% of the premix and stir thoroughly.

This Blue ink achieves a cap off life of 3-5 days dependant upon conditions and components.

The invention also encompasses writing implements such as for example marker pens so structured as to enable the employment of inks in accordance with the invention in its first aspect.
CLAIMS

1. A process for preparing an improved marking ink employing an organic solvent whereby the ability of the marker or pen product containing said ink to resist dry out of the nib or point is significantly enhanced, the process involving

preparing an ink as described heating same to a temperature in excess of the melting point

of a selected wax or wax like material and at which temperature the wax becomes soluble

adding said wax or wax like material and dissolving the wax to obtain an homogeneous solution. Allowing the combination of wax and ink to cool such that the wax becomes essentially insoluble and creates a fine dispersion of wax in the ink. Alternatively the solution of wax in ink may be shock chilled by the addition of cool ink to the already hot solution of wax in ink

using such adjusted ink in a pen or marker to achieve a product that resists the dry out of the nib or point when the cap or closable portion of the product is removed or left open.

2. A process according to claim 1 where the wax or wax like material may be selected from but not limited to the following:-

Paraffin Wax.

Microcrystalline Paraffin Wax.

Carboxyilic acid esters of Sterols.

Beeswax.

High Molecular Weight Fatty acids and Carboxylic Acids.

Mono and Di Glyceride Phosphate Esters.

Carnauba Wax.

Ethylene Glycol Di Esters of Fatty Acids.

Higher Fatty Acids

Higher Fatty Alcohols.

Higher Fatty Alcohol Esters.
Higher Fatty Alcohol Fatty Acid Esters. e.g.

Cetyl Stearate
Cetyl Palmitate.
Cetyl Stearyl Stearate.
Myristyl Myristate.

Hydrogenated Castor Oil.
Spermacetin - Natural & Synthetic.
Poly Ethoxylated Lanolin Alcohols.
Hydrogenated Lanolin.
Ozokerite Wax.
Montan Wax
PolyAmide Wax.

3. A process according to claim one or claim 2 whereby the proportion of wax or wax like material that is added or formulated into the ink in question is between 0.1 and 5% by weight of the final formula.

4. A process according to claim 1 to claim 3 whereby the wax or wax like material is of limited solubility in the solvent employed in the marker or pen ink. The ability of said wax or wax like material to form a fine dispersion of flake like lamellae in the ink at room temperature determines the suitability of the wax or wax like substance. The solubility of said wax or wax like material in said solvent and / or carrier determining the utility of such solvent or carrier. As noted the wax or wax like material shall form small (microscopic) platelets or lamellae under the conditions herein outlined.

5. A process according to claims 1 to 4 whereby the open cap storage time is increased by a factor of at least 5.
6. A process according to claims 1 to 5 whereby the attribute of open cap storage time is achieved by the physical aggregation of minute platelets under the influence of convection currents generated by the evaporation of solvent from the ink contained in a marker or pen device, more particularly from the writing tip or nib.

7. A process according to claims 1 to 6 whereby the limited solubility of the wax or wax like material in the solvent employed in the marker or pen ink and when utilised in company with the conditions as noted under claim 6, imparts the barrier properties essential to the mechanism noted in claim 6.

8. A writing or marking instrument comprising the ink composition claimed in claims 1-7 and a nib or point capable of passing the ink composition along with reservoir, casings and closure commonly employed in the art of such product manufacture.

9. A process for preparing an improved marking ink employing an organic solvent whereby the ability of the product containing said ink to resist dry out of the nib or point is significantly enhanced. The process involves:-

preparing a concentrate of the wax or wax like material in a carrier liquid or blend and
heating said carrier liquid to a temperature in excess of the melting point of a selected wax or wax like material at which temperature the wax becomes soluble. Adding said wax or wax like material and dissolving the wax to obtain an homogeneous solution. Allowing the combination of wax and carrier to cool such that the wax becomes essentially insoluble and creates a fine dispersion of wax in carrier liquid. Alternatively the solution of wax in carrier may be shock chilled by the addition of cool carrier liquid to the already hot solution of wax in liquid.

10. A process according to claim 9 where the wax or wax like material may be selected from but not limited to the following:-

Paraffin Wax.

Microcrystalline Paraffin Wax.
-15-

Carboxylic acid esters of Sterols.

Beeswax.

High Molecular Weight Fatty acids and Carboxylic Acids.

Mono and Di Glyceride Phosphate Esters.

Carnauba Wax.

Ethylene Glycol Di Esters of Fatty Acids.

Higher Fatty Acids

Higher Fatty Alcohols.

Higher Fatty Alcohol Esters.

Higher Fatty Alcohol Fatty Acid Esters. e.g.

Cetyl Stearate

Cetyl Palmitate.

Cetyl Stearyl Stearate.

Myristyl Myristate.

Hydrogentated Castor Oil.

Spermaceti - Natural & Synthetic.

Poly Ethoxylated Lanolin Alcohols.

Hydrogenated Lanolin.

Ozokerite Wax.

Montan Wax.

PolyAmide Wax.

11. A process according to claim 9 or 10 whereby the carrier liquid may be selected but not limited to the following materials:-

Castor oil.

Liquid Paraffin.
Vegetable oils,
Capralactone and Poly Capralactone.
Propylene carbonate.
Polyether polyols.
Polyester polyols.

12. A process according to claims 9 to 11 whereby the proportion of wax or wax like material that is added or formulated into to a final ink is between 0.1 and 1% by weight of the final formula.

13. A process according to claims 9 to 12 whereby the wax or wax like material is of limited solubility in the carrier liquid and the final solvent employed in the marker or pen ink. The solubility of said wax or wax like material in said solvent and/or carrier determining the utility of such solvent or carrier. The wax or wax like material shall form small (microscopic) platelets or lamellae under the conditions herein outlined.

14. A process according to claims 9 to 13 whereby the open cap storage time is increased by a factor of at least 10.

15. A process according to claims 9 to 14 whereby the attribute of open cap storage time is achieved by the physical aggregation of minute platelets under the influence of convection currents generated by the evaporation of solvent from the ink contained in a marker or pen device, more particularly from the writing tip or nib.

16. A process according to claims 9 to 15 whereby the limited solubility of the wax or wax like material in the solvent employed in the marker or pen ink when utilised in company with the conditions as noted under claim 15, imparts the barrier properties essential to the mechanism noted in claim 15.

17. A writing or marking instrument comprising the ink composition produced by the methods of claims 1 to 8 and claims 9 to 16 and a nib or point capable of passing the ink
composition along with a reservoir, casings and closure commonly employed in the art of such product manufacture.

18. An additive that may be combined with an existing ink or an ink in the course of manufacture and which exhibits a combination of a wax or wax like material and a carrier liquid employing the ingredients claimed in claims 10 and 11.

19. An ink suitable for use in marker pens comprising a dispersion of a wax or wax like material throughout the ink.
# INTERNATIONAL SEARCH REPORT

**A. CLASSIFICATION OF SUBJECT MATTER**

| IPC   | C09D11/16 |

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   | C09D |

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 practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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<th>Relevant to claim No.</th>
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<td>FR 2 439 226 A (SAKURA COLOR PRODUCTS) 16</td>
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<td>May 1980</td>
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<td>vol. 013, no. 293 (C-615), 6 July 1989</td>
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<td>&amp; JP 01 087677 A (PENTEL KK), 31 March 1989,</td>
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Further documents are listed in the continuation of box C. Patent family members are listed in annex.

*S* Special categories of cited documents:

*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 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

*K* document member of the same patent family

Date of the actual completion of the international search: 13 May 1997

Date of mailing of the international search report: 26.05.97

Name and mailing address of the ISA:

European Patent Office, P.B. 5818 Patentlaan 2

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Fax (+31-70) 340-3016

Authorized officer:

Girard, Y

Form PCT/ISA/31/8 (second sheet) (July 1992)
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