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(54) **Thermosensitive recording sheet for labeling foodstuff having direct contact with said foodstuff**

(57) The present invention refers to a thermosensitive recording sheet for labeling foodstuff having direct contact with said foodstuff comprising a substrate and on one surface of said substrate at least one thermosensitive layer comprising a ferric salt and a gallic acid ester, no reaction inhibitor being present in said layer.

In a further embodiment the thermosensitive recording sheet may comprise a substrate and on one surface of said substrate at least two layers comprising a ferric salt in one layer and a gallic acid ester in the other layer.

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Description

[0001] The present invention refers to a thermosensitive recording sheet for labeling foodstuff having direct contact with said foodstuff. The thermosensitive recording sheet comprises a composition that is not detrimental according to the regulations in respect of substances in direct contact with foodstuff.

[0002] In general, the obligation exists to identify foodstuff in the market place to enable identification of its origin. One possibility is by labeling said foodstuff.

[0003] The obligation to identify foodstuff is very important in the case of meat. Generally, cattle and pigs, and other meat stock, have an identification tag in their ear as proof of origin. Said marking allows to trace back their origin, age, and the name of the breeder. Additional information can also be included in such identification tag. It is important that after slaughtering such information is transferred to the carcass meat. Actually the veterinarian or the butcher puts down the required information on a label and affixes it immediately onto the meat. If these rather huge pieces of carcass meat are cut into smaller pieces for delivery to customers the information on said label is not transferred to the individual pieces of meat. This is so because the information has to be copied by hand in a time consuming manner.

[0004] The thermal printing technology is known in the prior art as a very reliable printing technology that can be carried out in environments where other printing technologies fail. For printing a label, for instance, a thermoprinter and a thermosensitive printing sheet is needed.

[0005] The thermosensitive recording material generally comprises a supporting substrate, for example, a paper sheet, synthetic paper sheet, or plastic resin film. A thermosensitive image forming layer is formed on the surface of the supporting substrate. Said layer generally comprises a dye precursor, such as a leuco basic dye, an acidic color developing agent, such as a phenolic compound, and a binder. Colored images are recorded on such a recording material by reaction of the dye precursor with the color developing agent upon heating.

[0006] In US-4,591,887 a heat-sensitive recording material is disclosed that is useful, for example, in the manufacture of labels used in connection with the sale of meat, produce, and the like. The material comprises a cellulosic or other suitable substrate, a thermally imprintable color producing layer, and a water-insoluble protective layer over the color-producing layer. The color-producing layer comprises a colorless or pale leuco dye, an acidic developer for producing an image upon heating of the recording material, a binder and a basic, particulate, neutralizing agent. The water-insoluble protective layer affixed to said color-producing layer comprises an organic-acid catalyzed cross-linked resin containing dispersed spacer particles. Such a label can be affixed to a package containing meat products. Meat discoloration beneath the label, known as "meat bloom", is avoided due to the sufficient oxygen-permeability of said label. The label cannot be affixed directly onto the meat because the ingredients of the thermosensitive layer, i.e. the leuco dye and the acidic developer are detrimental when in contact with meat.

[0007] JP-09150576 discloses a heat-sensitive recording material comprising a substrate provided with a heat-sensitive color developing layer containing a colorless or light colored electron donating precursor and an electron accepting substance capable of reacting with the dye precursor upon heating to cause color development, as the main component. A protective layer is applied to said heat-sensitive recording layer. A layer containing a styrene/butadiene copolymer and a pigment is applied to the back of the substrate. The purpose of the backcoat layer is to provide a heat-sensitive recording material having a water-resistant seal so that it can be affixed as label to a wet surface. Said recording material can be affixed to carcass meat without causing discoloration on said meat. It is not derivable from JP-09150576 that the label has direct contact with the meat and that it is not detrimental when in contact with meat.

[0008] DE-A-19853016 discloses a label for marking meat having a paper substrate of good printability and writability. Said paper substrate has an adhesive backside coating. Coating with the adhesive is carried out such that on each label there is at least one specific area which is coated with the adhesive and at least one specific area that is free of adhesive. The object of the alternating coating is to provide a substrate that can be affixed to either wet or dry meat.

[0009] JP-51/24257 discloses a heat-sensitive recording material for the manufacture of copying paper. The thermosensitive layer comprises ferric stearate, white pigment, binding resin, gallic acid ester and a reaction inhibitor. The coating is applied so that separately a first liquid comprising ferric stearate, white pigment, binding resin and solvent, and a second liquid comprising gallic acid ester, binding resin, reaction inhibitor and solvent, are mixed and then applied to a support and dried to form a heat-sensitive layer. This is because on mixing the first liquid components and the second liquid components together in a ball mill a fogging phenomenon arises. This leads to unexpected reactions. Additionally a reaction inhibitor is used as one of the essential components in the present invention because it inhibits the side reaction of ferric stearate and gallic acid ester in solution. The solvents used for preparing the coating composition are generally organic solvents and the gallic acid ester is generally soluble in these solvents.

[0010] The object of the present invention is to provide a thermosensitive recording sheet that is not harmful in direct contact with foodstuff. Especially, the thermosensitive recording layer does not comprise any substances harmful in contact with foodstuff. The thermosensitive recording sheet has to be printed using conventional thermal printers.

[0011] This object is attained by a thermosensitive recording sheet for labeling foodstuff having direct contact with said foodstuff comprising a substrate and on one surface of said substrate at least one thermosensitive layer comprising

a ferric salt and a water insoluble gallic acid ester. A reaction inhibitor is not necessary and therefore absent in said layer. In a further embodiment the thermosensitive recording sheet may comprise a substrate and on one surface of said substrate at least two layers comprising a ferric salt in one layer and a gallic acid ester in the other layer.

[0012] It was unexpectedly found that a reaction inhibitor is not required in a coating composition and the resulting coating comprising ferric salt and a water insoluble gallic acid ester. The disadvantageous fogging phenomenon of the prior art compositions was not observed if a thermosensitive recording layer comprising a ferric salt and a gallic acid ester were applied to a substrate. The coating composition of which the thermosensitive layer is applied to the substrate is preferably a water-based coating composition. The gallic acid ester should not be soluble in the fluid phase of the coating composition. The gallic acid ester should have a solubility in water at 23°C of less than 5 g/l. It is preferred that the solubility of the gallic acid ester is less than 3,5 g/l, more preferred less than 1,5 g/l and most preferred less than 0,5 g/l in the event that both reactive compounds are present in one layer.

[0013] In the event that the gallic acid ester and the ferric salt are present in different layers, alternatively gallic acid esters can be used having a higher solubility in water.

[0014] The coating weight of the thermosensitive layer is in the range of 5 to 27 g/m², preferably 10 to 22 g/m², and most preferred 15 to 17 g/m².

[0015] The thermosensitive recording sheet may comprise at least two layers on the same side of the substrate comprising ferric salt in one layer and gallic acid ester in the other layer. The coating weight of each layer is in the range of 3 to 13 g/m², preferably 5 to 11 g/m², and most preferred 7 to 9 g/m².

[0016] In a preferred embodiment an intermediate layer is present between the layer comprising the ferric salt and the layer comprising the gallic acid ester. Said intermediate layer may prevent a prereaction of thermosensitive compounds. Additionally, the resistance of the thermosensitive recording sheet to chemical and environmental influence may be improved. Said layer can comprise binders such as nitrocellulose, polyurethane, polyvinyl alcohol, vinyl acetates, acrylic latices or mixtures thereof. Additionally pigments such as silica, alumina or mixtures thereof can be present in said layer. The coating weight of said intermediate layer is in the range of 0,3 to 10 g/m², preferably 0,5 to 6 g/m², and most preferred 0,6 to 4 g/m².

[0017] In a further embodiment a back-coat may be applied to the side of the substrate opposite to that comprising the layer of ferric salt and gallic acid ester.

[0018] Additionally, adhesive may be applied to the side of the substrate opposite to that comprising the thermosensitive layer of ferric salt and gallic acid ester, or to the back-coat on the side of the substrate opposite to that comprising the thermosensitive layer of ferric salt and gallic acid ester. The adhesive may be applied over the entire area of the side of the substrate opposite to that comprising the thermosensitive layer of ferric salt and gallic acid ester. In a preferred embodiment the adhesive may be applied so that there is at least one specific area which is coated with the adhesive and at least one specific area that is free of the adhesive. It is preferred that the area coated with adhesive has an ellipsoidal or circular contour. The purpose of such alternating coating is to provide a thermosensitive recording sheet that can be affixed to either wet or dry meat. The adhesive may be any adhesive known not to be detrimental according to the regulations in respect of substances in direct contact with foodstuff, such as polyacrylic latices, derivatives of polyacrylic latices, UV-curable acrylic polymers, derivatives of UV-curable acrylic polymers, acrylic ester polymers, derivatives of acrylic ester polymers or mixtures thereof.

[0019] Other layers may be present in the thermosensitive recording sheet. It is further preferred that at least one intermediate layer is positioned between the substrate and the layer comprising the ferric salt and/or the gallic acid ester that is a thermal insulating layer known in the prior art. Thus, the layer may comprise a pigment such as a calcined clay, precipitated calcium carbonate, ground calcium carbonate, titanium dioxide, clay, talc, satin white, gypsum, silica, alumina, plastic pigment, hollow sphere pigment such as Ropaque® (manufactured by Rohm and Haas) or mixtures thereof. Binder, rheology modifier or wet-strength agents may also be present in said layer. The coating weight of said intermediate layer is in the range of 1 to 20 g/m², preferably 3 to 10 g/m², and most preferred 5 to 7 g/m².

[0020] Additionally, a cover layer may be applied to the layer comprising the ferric salt and/or the gallic acid ester. The cover layer may comprise a pigment, binder or mixture thereof. The coating weight of the cover layer is in the range of 0,1 to 8 g/m², preferably 0,5 to 5 g/m², and most preferred 1 to 3 g/m².

[0021] All layers present in the thermosensitive recording sheet can be applied by processes known to a person skilled in the art such as meyer bar, meyer rod, curtain coating, rotogravure, flexo printing, air brush coating, air knife coating, blade coating, spray coating and combinations thereof.

[0022] The substrate that is the carrier for at least one thermosensitive layer can be paper or board. The grammage of the paper or board can vary from 20-400 g/m², preferably 30-200 g/m², more preferred 35-150 g/m², and most preferred 40-90 g/m². The fibers used for preparing the paper or board may be of natural or artificial origin. A further embodiment of the substrate is to use a non-woven material, such as a valve prepared from fibers of natural or artificial origin. It is also possible that the substrate is a synthetic polymeric film, that is a PVC film, a polyethylene film (PE film), a polypropylene film, or a polyester film, like a polyethylene terephthalate film. Said film may be a unidirectional or biaxially oriented film.

[0023] If the substrate is paper or board it may have a Cobb value according to DIN 20535 after exposing the substrate for 60 seconds to water of less than 60. It is preferred that the Cobb value is less than 50, more preferred less than 40, even more preferred less than 30 and most preferred less than 20. Additionally, the substrate may be grease resistant.

[0024] The ferric salt used in the thermosensitive recording layer may be a ferric salt of a fatty acid such as ferric stearate (iron (III) stearate).

[0025] The gallic acid ester may be selected from lauryl gallate, stearyl gallate, octyl gallate, cetyl gallate, methyl gallate, ethyl gallate, propyl gallate and mixtures thereof.

[0026] The amount of the ferric salt present in the layer may be in the range of 2 to 10 wt% of the weight of the layer in total. It is preferred that the amount of the ferric salt in the layer is in the range of 4 to 9, more preferred 5 to 8, and most preferred 6 to 7 wt% based on the total weight of said layer.

[0027] The amount of the gallic acid ester present in the layer may be in the range of 5 to 20 wt% of the weight of the layer in total. It is preferred that the amount of the gallic acid ester in the layer is in the range of 9 to 17, more preferred 11 to 15, and most preferred 12 to 14 wt% based on the total weight of said layer. The thermosensitive recording layer may additionally comprise compounds such as binders, pigments, rheology modifiers, dyes, polyurethanes, starches, derivatives of starches, polyvinyl acetates, copolymers of polyvinyl acetates, polyvinyl alcohols, derivatives of polyvinyl alcohols, styrene butadiene latices, acrylic latices, waxes, nitrocellulose or mixtures thereof.

[0028] The thermosensitive recording sheet of the present invention may be affixed directly to the foodstuff. Foodstuff in the teaching of the present invention is any foodstuff that is adequate for human consumption. It is preferred that the foodstuff is selected from meat, sausages and similar products, fish, seafood, cheese, vegetables, fruit and baker's ware.

[0029] The assessment that the thermosensitive recording sheet of the present invention is not detrimental can be conducted according to § 35 of the German "Lebensmittel- und Bedarfsgegenständegesetz", EN 1186, EN 13130 and the XXXVI. recommendation for paper and board having contact with foodstuff of the ISEGA (XXXVI Empfehlung - Papiere, Kartons und Pappen für den Lebensmittelkontakt; 202. Mitteilung: Bundesgesundheitsbild-Gesundheitsforsch-Gesundheitsschutz 5/2001 44:546). Said recommendations regulate the properties of substances admissible for direct contact with foodstuff. The thermosensitive recording sheet of the present invention conforms to said recommendations.

[0030] The present invention is illustrated by the following examples.

[0031] All following coating compositions are manufactured using procedures known to a person skilled in the art. Said coating compositions are applied to a substrate by means of meyer rod coating.

Example 1:

[0032]

Table 1:

Compound	A1 [wt-%]
10 wt-% aqueous solution of Mowiol® 4/98 ¹	42
Aerosol OTB ²	0,2
lauryl gallate	26,3
water	rest

¹ polyvinyl alcohol

² sulfosuccinate (surfactant, Cytec)

[0033] The composition of master batch A1 is given in table 1. Aerosol OTB was added to a dispersion unit containing a 10 wt-% aqueous solution of Mowiol 4/98 and water. After dissolving the surfactant, lauryl gallate was added and the mixture was dispersed until a homogeneous mixture was obtained.

Table 2:

Compound	B1 [wt-%]
10 wt-% aqueous solution of Mowiol® 4/981	27,8

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Table 2: (continued)

Compound	B1 [wt-%]
Aerosol OTB ¹	0,3
iron (III) stearate	6,8
Martinal OL Q 107 ²	32,3
water	rest

¹ sulfosuccinate (surfactant, Cytec)

² aluminium hydroxide (Alusuisse)

[0034] The composition of master batch B1 is given in table 2. Aerosol OTB was dispersed in a dispersion unit containing a 10 wt-% aqueous solution of Mowiol 4/98 and water. Iron (III) stearate, and Martinal OL Q 107 were added subsequently. The master batch B1 was dispersed until a homogeneous mixture was obtained.

[0035] For manufacture of a thermal sensitive recording sheet master batch A1 comprising the gallic acid ester, master batch B1 comprising the ferric salt and binder are blended in a reaction vessel and subsequently applied to a substrate. The coat weight is in the range of 15 to 17 g/m². The composition of the applied coating color and the type of substrate is given in table 3, 4 and 5.

Table 3:

Compound	S1 [pbw] ¹	S2 [pbw] ¹	S3 [pbw] ¹	S4 [pbw] ¹	S5 [pbw] ¹	S6 [pbw] ¹	S7 [pbw] ¹
Substrate	paper	paper	paper	paper	paper	paper	paper
A1	2	1,3	1	1	1	1	1
B1	2		2	2	2	2	1
APU 1012 ²	1			2	1		1
Acronal S728 ³		1	2				
Neocryl XK14 ⁴						2	

¹ parts by weight

² polyurethane / polyacrylate dispersion (Alberdingk and Boley)

³ styrene/acrylate dispersion (BASF)

⁴ acrylic polymer (Neoresins)

Table 4:

Compound	S8 [pbw] ¹	S9 [pbw] ¹	S10 [pbw] ¹	S11 [pbw] ¹	S12 [pbw] ¹	S13 [pbw] ¹	S14 [pbw] ¹
Substrate	paper	paper	PE film	paper	paper	PE film	PE film
A1	1	1	1	1	1	1	1
B1	4	4	4	2	1	2	2
APU 1012 ²	1,5	1		3	3	2	2
Setalux 6774 EBL ³			4				
Neocryl XK14 ⁴							

¹ parts by weight

² polyurethane / polyacrylate dispersion (Alberdingk and Boley)

³ polyacrylate dispersion (Akzo Resins)

⁴ acrylic polymer (Neoresins)

Table 5:

Compound	S15 [pbw] ¹	S16 [pbw] ¹
Substrate	PE film	PE film
A1	1	1
B1	2	2
UC 80 ²	2	
U911 ³		2

¹ parts by weight

² polyurethane / polyacrylate dispersion (Alberdingk and Boley)

³ polyurethane / polycarbonate dispersion (Alberdingk and Boley)

[0036] The thermosensitive recording sheets S1 to S16 are suitable to be printed on conventional thermal printers. The color of the printing image is violet-black.

Example 2:

[0037] The coating color A2 was manufactured in a dispersing unit by adding nitrocellulose E400, EDS-W(N), zinc stearate and lauryl gallate to acetone. The mixture was dispersed until a homogeneous dispersion was obtained. The composition of A2 is given in table 6.

Table 6:

Compound	A2 [wt-/00]
nitrocellulose E400 ¹	8,2
EDS-W(N) ²	2,2
zinc stearate	3,8
lauryl gallate	13,4
acetone	72,4

¹ nitrocellulose (Wolff Cellulosics)

² ethylene bissteramide (Faci)

[0038] The coating composition A2 was applied to a PE film. The coating weight is 6 g/m².

Example 3:

[0039] The coating colors B2 to B4 were manufactured in a dispersing unit. The mixture was dispersed until a homogeneous dispersion was obtained. The composition of B2 to B4 is given in table 7.

Table 7:

Compound	B2 [wt-%]	B3 [wt-%]	B4 [wt-%]
Mowital B30T ¹		4,9	
nitrocellulose E375 ²	5,3		
Vinapas UW10 ³			6,2

¹ polyvinylacetal (Clariant)

² nitrocellulose in epoxidized soy oil

³ polyvinylacetate (Wacker)

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Table 7: (continued)

Compound	B2 [wt-%]	B3 [wt-%]	B4 [wt-%]
Martinal OLQ 107 ⁴	18,9	18,9	
Sipernat P820 ⁵			10
EDS-W (N) ⁶	5,3	5,3	2,7
zinc stearate			0,3
iron (III) stearate	4,9	4,9	4,2
ethanol		65,8	
acetone	65,6		76,6

⁴ aluminium hydroxide (Alusuisse)

⁵ aluminium silicate (Degussa)

⁶ ethylene bissteramide (Faci)

[0040] Either the coating composition B2, B3 or B4 were applied to the coated side of the substrate according example 2. The coating weight is 6 g/m². The obtained thermosensitive recording sheets are suitable to be printed on conventional thermal printers. The color of the printing image is violet-black.

Example 4:

[0041] The coating color A3 was manufactured in a dispersing unit by adding nitrocellulose E400, EDS-W(N), zinc stearate and octyl gallate to acetone. The mixture was dispersed until a homogeneous dispersion was obtained. The composition of A3 is given in table 8.

Table 8:

Compound	A3 [wt-%]
nitrocellulose E400 ¹	8,2
EDS-W(N) ²	2,2
zinc stearate	3,8
octyl gallate	13,4
acetone	72,4

¹ nitrocellulose (Wolff Cellulosics)

² ethylene bissteramide (Faci)

[0042] The coating color Z1 was manufactured in a dispersing unit by adding nitrocellulose E400, EDS-W(N), zinc stearate and octyl gallate to acetone. The mixture was dispersed until a homogeneous dispersion was obtained. The composition of Z1 is given in table 9.

Table 9:

Compound	Z1 [wt-%]
nitrocellulose E400 ¹	16,3
EDS-W(N) ²	3,2
zinc stearate	0,4

¹ nitro cellulose (Wolff Celulosics)

² ethylene bissteramide (Faci)

Table 9: (continued)

Compound	Z1 [wt-%]
Sipernat P820 ³	14,7
acetone	65,5

³ aluminium silicate (Degussa)

[0043] The coating composition A3 is applied to a PE film having a coating weight of 5 g/m². The coating composition Z1 is applied to the first coating having as an intermediate coating a coating weight of 2 g/m². The coating composition B3 (example 2) is applied to said intermediate coating having a coating weight of 6 g/m².

The obtained thermosensitive recording sheet is suitable to be printed on conventional thermal printers. The color of the printing image is violet-black.

Example 5:

[0044] The coating composition A3 (example 4) is applied to a PE film having a coating weight of 5 g/m². Setalux 6774 (polyacrylate dispersion, Akzo Resins) is applied as intermediate coating to the first coating having a coating weight of 3 g/m². The coating composition B2 (example 3) is applied to said intermediate coating having a coating weight of 7 g/m². The obtained thermosensitive recording sheet is suitable to be printed on conventional thermal printers. The color of the printing image is violet-black.

[0045] The thermosensitive recording sheet of the present invention is conforming to § 35 of the German "Lebensmittel- und Bedarfsgegenständengesetz", EN 1186 and EN 13130.

The amount of compounds migrating from said recording sheet to foodstuff is admissible for articles having direct contact with foodstuff. Furthermore, the amount of volatile isomers, isocyanates, butadiene and remaining solvent in the recording sheet of the present invention is conforming to said regulation.

Claims

1. Thermosensitive recording sheet for labeling foodstuff having direct contact with said foodstuff comprising a substrate and on one surface of said substrate at least one thermosensitive layer comprising a ferric salt and a water-insoluble gallic acid ester, no reaction inhibitor being present in said layer.
2. Thermosensitive recording sheet for labeling foodstuff having direct contact with said foodstuff comprising a substrate and on one surface of said substrate at least two layers comprising a ferric salt in one layer and a gallic acid ester in the other layer.
3. Thermosensitive recording sheet according to claim 2, **characterized in that** at least one intermediate layer is present between the layer comprising a ferric salt and the layer comprising the gallic acid ester.
4. Thermosensitive recording sheet according to any of the preceding claims, **characterized in that** an adhesive is present on the side of the substrate opposite to the side of the substrate having thereon the layer comprising the ferric salt and/or the gallic acid ester.
5. Thermosensitive recording sheet according to any of the preceding claims, **characterized in that** at least one thermal insulating layer is present between the substrate and the layer comprising the ferric salt and/or the gallic acid ester.
6. Thermosensitive recording sheet according to any of the preceding claims, **characterized in that** a cover layer is present on the coating comprising the ferric salt and/or the gallic acid ester.
7. Thermosensitive recording sheet according to any of the preceding claims, **characterized in that** the ferric salt is ferric stearate.
8. Thermosensitive recording sheet according to any of the preceding claims, **characterized in that** the gallic acid

ester is selected from the group consisting of lauryl gallate, stearyl gallate, cetyl gallate, octyl gallate, methyl gallate, ethyl gallate, propyl gallate and mixtures thereof.

5 9. Thermosensitive recording sheet according to any of the preceding claims, **characterized in that** the amount of the ferric salt in the layer is in the range of 2 to 10 percent by weight based on the total weight of said layer.

10 10. Thermosensitive recording sheet according to any of the preceding claims, **characterized in that** the amount of the gallic acid ester in the layer is in the range of 5 to 20 percent by weight based on the total weight of said layer.

15 11. Thermosensitive recording sheet according to any of the preceding claims, **characterized in that** the substrate is selected from paper, board, nonwoven material and synthetic polymeric film.

20 12. Use of the thermosensitive recording sheet according to any of the preceding claims for labeling foodstuff by directly affixing the thermosensitive recording sheet to the foodstuff.

25 13. Use of the thermosensitive recording sheet according to claim 12, **characterized in that** the foodstuff is selected from meat, sausages and similar products, fish, seafood, cheese, vegetables, fruit and baker's ware.



European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 02 01 5885

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The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 16 December 2002	Examiner Martins Lopes, L
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

EPO FORM 1503 03.82 (P/4C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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