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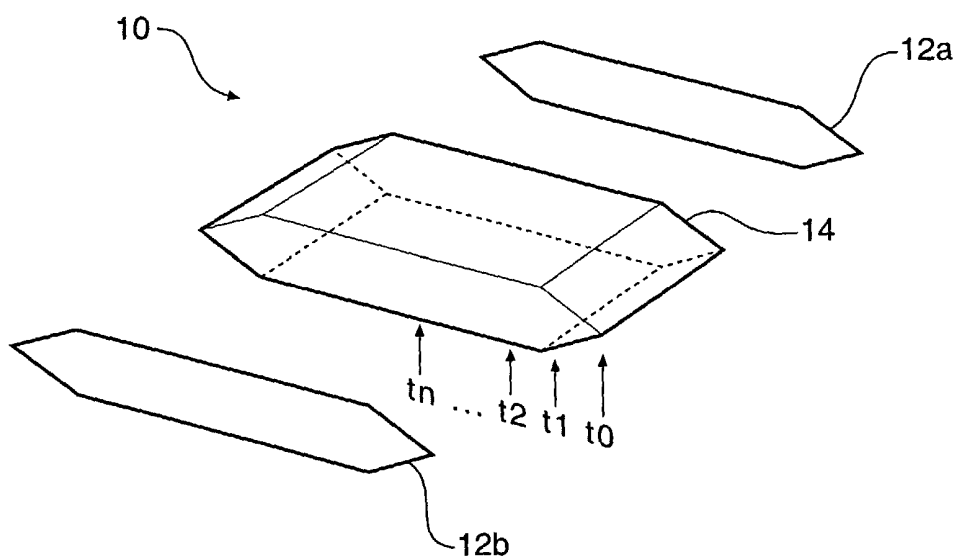
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(54) Title: TAMPER EVIDENT PACKAGING



(57) Abstract: Atamper evident packaging system including a tag (10) attachable to a package comprising a substantially oxygen impermeable upper barrier layer (12a), a reactive intermediate layer (14) including a reactive material changeable between an un-reacted condition in the absence of oxygen and a reacted condition in the presence of oxygen to produce a visible colour change, and a lower layer (12b) which can be a substantially oxygen impermeable lower barrier layer or an integral part of the package.



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## Tamper Evident Packaging

### Field Of The Invention

The present invention relates to a method and device used to create tamper evident packaging.

### 5 Background Of The Invention

Traditionally packaging of products such as food items and drug products likely to be ingested by consumers has involved considerations such as preventing physical damage to the product or maintaining the product free of bacterial, chemical or microbial contamination. More recently, manufacturers of packaged products have also had to  
10 take into account the possibility that their product may be deliberately interfered with by a third party. Tampering with products is a concern for all goods manufacturers because of the effect such tampering may have on product performance and therefore the reputation and good name of the manufacturer as well as possibly injuring those using tampered products. The problems faced by those in the food and drug industries,  
15 where the products are ingested are particularly acute. Contamination of food and drug products with dangerous materials can have severe consequences.

It is therefore desirable for the retailer, the consumer and the manufacturer to have a method of determining whether or not a package has been altered. In such cases the product can be rejected to thereby reduce the likelihood of consumption of a product  
20 that has been tampered with.

To date concern in this area has concentrated on producing wrappings for product containers that are difficult to remove or difficult to re-close following opening. U.S. Pat. No. 4,721,638, issued to Matsuguchi et al, relates to materials that prevent the re-closing of a package. The materials have an adhesive layer, a second layer, a peeling  
25 agent layer laminated on the second layer and a first layer laminated on the peeling agent layer. The second and first layers are adhered via the peeling agent layer. Once the first layer and the second layer are separated they cannot be re-stuck together. These materials have uses as labels, such as price tags.

U.S. Pat. No. 4,746,556, issued to Matsuguchi et al, relates to easily breakable sticking  
30 material. The material has a surface layer, an easily breakable layer laminated to the

surface layer, a first peel-off layer formed on the surface of the easily breakable layer, a second peel-off layer formed on a part of the main surface of the easily breakable layer, a covering layer and an adhesive layer. The surface layer is peeled off of the easily breakable layer and cannot be re-adhered again. These materials have use in labels, such as price tags.

U.S. Pat. No. 5,042,842, issued to Green et al, relates to labels, such as those used for preventing or detecting tampering and counterfeiting. The label is a multilayer security label which has a base layer with a permanent adhesive coated on a lower surface. Indicia or markings are applied to the bottom surface of the top layer and the top layer is laminated to the upper surface of the base layer. The top layer is laminated to the upper surface of the base layer by permanent patterned adhesive. A portion of each of the upper surface of the base layer and the bottom surface of the top layer are free of adhesive.

US5876816 issued to Freedman discloses a multi layer tamper-evident label comprising: first and second layers of polymeric film, a heat-sealable layer there between, and an adhesive layer bonded to lower surface of the second layer.

Notwithstanding all of the packaging methods disclosed above, the fact remains that the malicious interference with products still occurs and can result in huge losses of income resulting from withdrawal of products from sale and from loss of consumer confidence as a result of the interference.

The present invention concerns itself with this issue. The invention is therefore directed at a packaging system that provides a visual indication of product opening and, therefore of product tampering. The packaging system of the invention is adaptable for use in a variety of environments and may be incorporated into a number of existing packaging apparatuses with minimum effort.

### **Summary Of The Invention**

Therefore, according to a first aspect of the invention there is provided a tag attachable to a package so as to produce a tamper evident packaging system, said tag including a substantially oxygen impermeable upper barrier layer, a reactive intermediate layer including a reactive material changeable between an un-reacted condition in the

absence of oxygen and a reacted condition in the presence of oxygen, wherein said change takes the form of a colour change.

5 Preferably, the intermediate reactive layer is secured to a lower substantially oxygen impermeable barrier layer. In some forms of the invention, the lower barrier layer may be an integral part of a product package or container. Thus, the tag of the invention is adapted to be created directly on a product package, or form a part thereof. In a preferred form of the invention the lower barrier layer is of a colour selected to enhance the visibility of the colour change that occurs on exposure of the reactive layer to oxygen. In addition the lower barrier layer and tag dimensions can be selected such that a colour change reaction exposes or obscures a logo or other indicia present on the packaged product or on a surface of the tag.

10 The oxygen reactive layer is thereby secured between two substantially oxygen impermeable layers. In this manner the only direction for oxygen to penetrate the tag is through lateral diffusion into exposed edges of the tag. Thus, the reactive intermediate layer undergoes a visible colour change that commences at the periphery of the tag and that gradually moves towards the centre of the tag. The colour change can thus provide secondary information concerning a period for which the packaged product has been exposed to oxygen, or, a measurement of elapsed time since the breach of the package.

20 Preferably, the tag of the invention is affixed to a product package in an oxygen-depleted atmosphere such that the tag will undergo the colour change reaction on opening of the package. Such an atmosphere can be achieved in a number of ways, for example, the tag can be placed on the package and subsequently covering in an air excluding covering such as a shrink wrap covering. For most effective results a shrink-wrap package should be placed around the tag either in a quick time to avoid any colour change reaction, or in an oxygen free environment. For purposes of description, a suitable environment for affixation of the tag of the invention to a package will hereinafter be described as inert. However, it should be understood that the atmosphere need not be truly chemically inert and that a reducing atmosphere, a vacuum or an environment that excludes oxidising gases would fall within the description inert.

In an alternative form of the invention a tag is located on a product packaging in an inert gas atmosphere, such as a carbon dioxide environment or a nitrogen atmosphere. In

such a manner a highly oxygen sensitive tag could be placed on a product and would, when exposed to atmospheric oxygen on opening, be highly reactive. The use of a highly sensitive tag in an inert atmosphere is a particularly preferred form of the invention. The use of such a tag enables consumers to identify rapidly on visual  
5 inspection those products that have been subjected to tampering.

It is also possible for the tag to be created using upper and lower barrier layers composed of oxygen scavenging plastics. Additionally, the upper and lower barrier layers can comprise multi-layer laminates of a number of plastics materials. By incorporating a number of plastics materials, the desirable properties of a number of  
10 materials can be incorporated into the final product, for example strength, ultraviolet light resistance and so on.

Other strategies that might be adopted to ensure the integrity of the tag include the use of positive or negative pressure within the product packaging and also the use of protective gas shielding.

15 The reactive material layer of the present invention preferably includes a highly oxygen sensitive material such as a reduced vat dyes, for example indigo. Such materials are known to be sensitive to oxidation by atmospheric oxygen and undergo a transition from a largely colourless condition to a coloured condition on oxidation. A reduced vat dye such as indigo is particularly preferred for the purposes of the invention as it is already  
20 approved by the United States Food and Drug Administration for use in various suture products. Accordingly there are likely to be no issues concerning possible toxicity should tags of the invention be accidentally ingested.

The reactive material may in addition to the reduced vat dye include further materials that enhance the performance of the product. For example, the vat dye may be  
25 supplied in the form of a matrix comprising an adduct of an adhesive material and the dye. In such a manner an oxygen sensitive adhesive tag that can be applied as a liquid or sprayable coating is created.

The tag of the invention is adapted to change colour from the un-reacted to the reacted condition in a time period from a number of seconds to up to several months and  
30 possibly longer. Where extended viability of the product is a consideration the incorporation of UV resistant materials and materials that reduce the effects of photo-oxidation into the product is preferable. Such materials can be included either in the

reactive layer or in an upper layer of the product. Appropriate material might include the use of UV stabilizing plastics such as polycarbonate or treated polycarbonate and butylated hydroxytoluene (BHT), which is known to counter the effects of photo-oxidation.

## 5 **Description Of Drawings**

The above and other objects, features, and advantages of the present invention will be apparent from the following detailed description of a preferred embodiment in conjunction with the accompanying drawings. In the drawings:

10 FIG 1a and FIG 1b illustrate schematically a tamper evident packaging system in accordance with a first aspect of the present invention;

FIG 2 represents a tag as shown in figure 1 in arrangement with product packaging;

FIG 3 illustrates the packaging of figure 1 as used;

FIG 4 illustrates the development of a colour change reaction in the tamper evident package of figures 1-3;

15 FIG5 illustrates schematically a tamper evident packaging system in accordance with a second embodiment of the present invention;

FIG 6a and FIG 6b illustrates schematically a tamper evident packaging system in accordance with a third embodiment of the present invention;

20 FIG 7 represents the key steps in the production process of a tamper evident packaging system in accordance with the invention;

FIG 8 shows a carbon dioxide cycling system for use in the packaging system of figure 1;

FIG 9 illustrates schematically and in cross-sectional view a film used in producing a tamper evident package in accordance with the invention;

25 FIG 10 illustrates a tamper evident packaging system in accordance with a fourth embodiment of the invention;

FIG 11 illustrates a tamper evident packaging system in accordance with a fifth embodiment of the invention;

FIG 12 represents the key steps in the production process of a liquid tamper evident packaging system in accordance with the invention;

- 5 FIG 13 illustrates schematically and in cross-sectional view a film used in producing a tamper evident package in accordance with the invention.

### Description Of The Preferred Embodiment

10 Figures 1a and 1b illustrate schematically a tag 10 formed in accordance with the invention. The tag 10 includes upper and lower layers of transparent gas impermeable plastic sheeting 12a, 12b. Between the layers of plastic sheeting is an oxygen sensitive matrix reactive layer 14 containing a vat dye or a vat dye adduct in a reduced form. Typically the vat dye used would be one suitable for use in foodstuffs such as indigo. Vat dyes are known to be changeable from a reduced state to an oxidised state in which  
15 the dye assumes an intense colour. For example, indigo in the reduced state is pale in colour and produces a rich blue colour in the oxidised state.

The tag 10 is used in tamper evident packaging as will be described herein below.

The upper plastic impermeable layer 12a is formed of colourless transparent material and the base layer 12b is formed of colourless or whiter material. The thickness of the  
20 vat dye matrix is approximately 0.46mm.

The upper and lower layers 12a 12b are made of any one of a number of commercially available thin film products. For example, a co-polymer of ethylene and polyvinyl alcohol can be manufactured to have a low rate of oxygen and carbon dioxide diffusion therethrough of the order of  
25  $O_2$  0.2  $cm^3/m^2/24hr$  for a 20 micron film thickness at 0% relative humidity and atmospheric pressure; and  
 $CO_2$  0.8  $cm^3/m^2/24hr$  for a 20 micron film thickness at 0% relative humidity and atmospheric pressure

Similarly, biaxially oriented ethyl vinyl alcohol, polyvinyl alcohol and polypropylene films can be used to form a gas barrier film.

The oxygen permeability of a polyvinyl alcohol film suitable for use in a tag formed in accordance with the structure described has been measured as  $0.25\text{cm}^3/\text{m}^2/24\text{hr}$  for a  
5 14nm film at a relative humidity of 35%.

A suitable co-polymer of ethylene and vinyl alcohol (ethylene content 32 mol%) has a gas permeability of:

$\text{O}_2$   $0.2\text{ cm}^3/20\text{ }\mu\text{m}^2\text{ day atm }20^\circ\text{C }0\%$  relative humidity

$\text{CO}_2$   $0.8\text{ cm}^3/20\text{ }\mu\text{m}^2\text{ day atm }20^\circ\text{C }0\%$  relative humidity

10 Alternatively the layers 12a and 12b may be constituted as laminate films at least one layer of which is a gas impermeable barrier film as described above that may be formed as mono or co-extruded films. Figure 13 provides an indication of a structure of a film that may be used in the formation of the tag 10.

The reactive layer 14 comprises a matrix of a vat dye in a base material, which may be  
15 selected from any suitable material such as a protein matrix, an agar-agar matrix, a polyvinyl acetate/alcohol matrix, a methyl-cellulose matrix, or a cellulose acetate matrix.

The reactive material layer includes a reduced vat dye composition in the proportion 0.001%-50 % wt/wt and a base material as defined forming the balance. The reduced vat dye composition would be constituted as follows.

20 Indigo 3.8mM  
Sodium hydroxide 0.3M  
Sodium dithionite 0.24M  
pH within the range 4-10

The base material is, in the embodiment under consideration, a casein hydrochloride  
25 material used to form a reduced vat dye adduct with the reduced vat dye composition. The modified casein material is used to promote the adhesion of the reactive material layer to the upper and lower gas-impermeable layers 12a and 12b. Casein adhesive in the composition enhances the ability of the composition to bind to cellulose fibres. The addition of cellulose material to the upper and lower layers of adhesive coated plastics  
30 material can enhance the adhesion of a casein vat dye adduct to the plastic upper and

lower layers. The adhesion of the casein hydrochloride reduced vat dye adduct to a plastics surface such as a polyethylene surface can also be enhanced by the flame treatment of the polyethylene material.

Alternatively, the base material may be constituted by a polyvinyl acetate material hydrolysed and converted in situ to a polyvinyl alcohol vat dye adduct. While not wishing to be bound by any theory it is suggested that the repeating polymer chain units of the poly vinyl alcohol form hydrogen bonds with the reduced vat dye to form the adduct.

The adduct is a product that can be drawn and which changes from a white colour to a royal blue colour on being exposed to air. The colour change process commences at the periphery of the tag 10 at a time ( $t_0$ ) when the tag is first exposed to air, the area changed to the blue oxidised colour of the dye migrating inwardly as time progresses as shown in  $t_1 - t_n$ . The colour change process is therefore

not restricted to the outer surface of the adduct but permeates the entire volume of the adduct thereby increasing the intensity of the colour change. The plastic material can also be converted to a white emulsion product prior to use that also changes to a royal blue on exposure to atmospheric oxygen. The conversion is achieved by washing the plastic material in a series of increasingly non-polar solvents while being stored under carbon dioxide. A final wash is conducted using n-heptane.

The polyvinyl alcohol reduced vat dye adduct can also be incorporated into a woven product where necessary, or alternatively, the adduct can be adhered to a plastic lower layer with or without the addition of cellulose to the plastics layer or, importantly, applied as a spray on material to any surface.

Figure 2 illustrates how the tag 10 of figure 1 may be incorporated into a packaged product 20. The product 20 is a generally rectangular cardboard carton that is over-wrapped with a layer of gas impermeable plastics material 22.

The tag 10 is either a stand-alone tag having a structure as shown in figure 1, or, alternatively, can be a spray on tag formed of a polymer vat dye adduct as described. A second outer wrapper 24, also formed of a gas impermeable plastics material is then located over the tag 10. The tag 10 is thus surrounded on two sides by gas impermeable materials. The inter-wrapper space indicated at 26 comprises an inert

(that is non-oxidising) gas such as carbon dioxide or nitrogen, or the inter-wrapper space is formed of a vacuum. To further ensure the absence of oxygen in the inter-wrapper space 26 it is possible for the inner and outer wrap layers 22 and 24 to contain an oxygen scavenging material. Alternatively, the inter-wrap space 26 may be subject to vacuum conditions such that air is excluded, or may include an inert gas generating system.

In any event, to access the interior of the package, and therefore the contents of the package, it is necessary for the outer wrapper to be breached thereby exposing the tag 10 to atmospheric oxygen. Thus whatever the sealing mechanism used in the construction of the package shown in figure 2 the opening of the package, either for use or for nefarious purposes the opening will be apparent from a colour change in the tag 10. As can be seen from the drawing, the colour change in the tag has a related logo. The choice of logo is, of course a matter of the discretion of the user, however, either a company logo – which might serve as an indicator of a genuine product or as a warning to highlight the fact that a package has been opened.

Figure 3 illustrates in three dimensions how a package bearing a tag in accordance with the invention might appear. The package in figure 3 is a small pharmaceutical package and gives an approximate indication of the size of the tag 10.

Figure 4 gives an illustration of the development of colour in the tag 10 on exposure to atmospheric oxygen. As discussed the tag 10 has upper and lower layers of gas impermeable material. The tag 10 has, as can be seen from the expanded view in figure 1, a definite thickness. The area exposed around sides of the tag is therefore available for reaction with oxygen to undergo the colour change reaction. Figure 4 illustrates how this change develops at three time intervals from initial exposure of the tag to air. The development of the colour change may be viewed as the development of an oxidation contour.

Figure 5 illustrates a packaging system in accordance with a second embodiment of the invention. The package 30 includes a container of a size used for pharmaceutical products for consumer sales. The container 30 has a tag in accordance with the invention attached thereto. The package itself, therefore serves as a point of attachment of the tag 10. The package is surrounded by a space 32, similar to the inter-wrap space 26 of the device of figure 2. In this case, the space 32 and the interior of the package are occupied by carbon dioxide as well as carbon dioxide absorbing

material to effect a partial vacuum therein. Thus, the tag 10 is maintained in a reduced state until the package is breached thereby exposing the tag to atmospheric oxygen, at which point the colour change reaction will commence.

5 Figures 6a and 6b illustrate a further embodiment of the invention in the form of a package 40 being a container having a window portion 42 formed therein. The window portion is transparent and allows viewing of a tag 10 positioned directly below the window space. The tag 10 is, as with previous devices, surrounded by a space occupied by an inert gas, vacuum or other means as described above. In the case of the device illustrated in figure 6 the gas shield is provided within the package as indicated by the  
10 dotted lines inside of the package boundaries.

Figure 7 illustrates in schematic form how the tag 10 of figure 1 might be incorporated into a package manufacturing process for the manufacturing of a package in accordance with figure 2. A blister pack 50 of pharmaceutical products is packed in an outer container 52 in chamber D that is maintained in an inert atmosphere as defined  
15 herein above. The container and blister pack travel into chamber E that is also maintained under inert atmosphere conditions. Tags 10 are delivered onto each container through feed wheel P operated under the control of pulley R. The container with an attached tag 10 is then delivered to chamber F wherein the final over-wrapping layer is attached to the carton in an inert atmosphere. Thus, the package incorporates a  
20 tag sensitive to the presence of atmospheric oxygen, which will remain unaltered until the package is opened.

Figure 8 indicates, schematically, a carbon dioxide cycling system used in the production of packages as detailed in figure 7. The arrowed line 43 indicates the flow of carbon dioxide through the system and the dotted line 44 indicates the passage of  
25 packages to which a tag has to be applied. Thus, the production line described above passes through a carbon dioxide holding chamber 45, or a number of carbon dioxide chambers 45 in series. The chambers 45 have sensors 46a and 46b to indicate high and low levels of the gas to ensure adequate levels are maintained and introduced into any packages passing through the system.

30 Thus, the operation depicted in figure 7 would be conducted in a carbon dioxide chamber 45. In addition, the carbon dioxide in the system is recycled to a holding tank 47 through the overflow line 46c. To ensure an adequate condition of the gas in the

system, including maintenance of level and quality, the holding tank 47 may be equipped with sterilising equipment, for example an ultra violet lamp.

The carbon dioxide is recirculated through the system by a fan 48.

5 The structure of an over-wrapping material is depicted in figure 9. A multi-layer laminate film is shown including an oxygen impermeable outer layer, for example a bi-axially oriented polypropylene, an oxygen impermeable inner layer of similar material and a lower layer of oxygen scavenging material. This material may also serve as the gas impermeable layers 12a and 12b in the tag 10.

10 The apparatus of figure 7 may also be operated to produce a blister pack within a package in an inert atmosphere. By using such equipment in conjunction with the equipment used for application of the tags of the invention it is possible to produce a blister package incorporating a tamper evident tag either on a surface of the package, or on the blister pack itself.

15 As discussed earlier, figure 9 illustrates a structure used in the formation of a tamper evident package in accordance with the invention. The wrapping, typically used on either side of a tag containing a reduced vat dye in a matrix, includes both oxygen impermeable barrier layers and also oxygen scavenging materials to ensure that a substantially oxygen free environment is maintained around the tag 10 in the package following manufacture.

20 Figures 10 and 11 illustrate fourth and fifth embodiments respectively of the invention. The drawings show how a tag formed in accordance with the invention can be incorporated into a blister pack itself rather than the outer container thereof. Again, the tag 10 is located underneath an outer covering of a gas impermeable material manufactured in a process using equipment similar to that illustrated in Figure 7. A  
25 tamper evident tag 10 can, depending on the packaging size and requirements, be inserted over each separate component of the blister pack or simply as a single unit that will be exposed to air wherever the pack is breached. The development of a colour change product in the tag 10 is assessable visually through different transparent portions in the packaging as shown in the different positions of the tag depicted in  
30 figures 10 and 11.

The invention has been described with reference to a solid tag. However, it is also possible for the invention to be applied as a spray on liquid tag. A process for achieving a spray on tag is depicted in figure 12. The spray on tag is formulated with suitable liquid matrix material such as polyvinyl alcohol as previously described. A gas impermeable inner layer is wrapped over the outer carton of the package and the wrapped package enters a carbon dioxide or other inert gas chamber, such as the chamber illustrated in figure 7. Within the carbon dioxide chamber, depicted schematically by dotted lines in figure 12, a spray on liquid tag is applied to a predetermined outline in step 60 and allowed to dry in step 62. A second gas impermeable outer wrapper is applied to the carton in step 64, followed by a second drying stage 66. Where applicable, a logo outline can also be applied, in step 68 to the product prior leaving the carbon dioxide gas chamber. The use of a liquid spray on tag clearly allows a great deal of freedom in selection of tag outline and geometry, being determined by a stencil rather than a specific geometry.

As mentioned previously it is also possible to construct a tag in accordance with the invention by application of the tag to a carton in substantially oxygen free conditions and covering the tag in an impermeable outer wrapper. Figure 13 illustrates a structure of a tag used in such circumstances.

The wrapper of figure 13 includes a tag enclosed on all sides by gas impermeable layers. Where the laminate material is breached, eg by tearing upon opening, or by piercing for example by a malicious party using a syringe needle, the oxygen sensitive layer will be exposed and will undergo the colour change reaction. A colour sensitive packaging comprising a single layer covering the entire package could therefore be used.

In the drawing layer a represents an outer layer of oxygen impermeable plastics material, for example, a bi-axially oriented polypropylene; layer b represents an oxygen impermeable plastics material inner layer, for example, a bi-axially oriented polyvinyl alcohol material; layer c represents an oxygen scavenging layer; and layer d represents an enclosed central layer of reduced vat dye adduct. The edges of the film, as shown schematically, protect the central layer d from exposure to oxygen and exposure only occurs when the film is breached. The overall height of the film is 0.5mm or less.

It will be readily apparent that the form of the invention may be varied considerably without departing from the scope of the invention. The examples have been provided

for purposes of illustration only and should not be taken as limiting the form of the invention. In particular the invention has been described with reference to its suitability for use in connection with packaging of various pharmaceutical products. There are however countless other products that are packaged in a variety of ways and in which  
5 the invention would find use. For example, many food products would benefit from a tamper evident system as envisaged by the invention. Modifications and variations of the present invention such as would be apparent to a skilled addressee are deemed to be within the scope of the invention.

## CLAIMS

1. A tag attachable to a package so as to produce a tamper evident packaging system, characterised in that said tag includes a substantially oxygen  
5 impermeable upper barrier layer, a reactive intermediate layer including a reactive material changeable between an un-reacted condition in the absence of oxygen and a reacted condition in the presence of oxygen, wherein said change takes the form of a colour change.
2. A tag according to claim 1, characterised in that said substantially oxygen  
10 impermeable upper barrier layer is formed of colourless transparent material.
3. A tag according to claim 1, characterised in that said tag additionally includes a substantially oxygen impermeable lower barrier layer.
4. A tag according to claim 3, characterised in that, the lower barrier layer may be an integral part of a product package or container.
- 15 5. A tag according to claim 3, characterised in that said lower barrier layer is of a colour selected to enhance the visibility of the colour change that occurs on exposure of the reactive layer to oxygen.
6. A tag according to claim 3, characterised in that said lower barrier layer and tag  
20 dimensions are selected such that a colour change reaction exposes or obscures a logo or other indicia present on the packaged product or on a surface of the tag.
7. A tag according to claim 3, characterised in that said lower barrier layer is formed of colourless or white material.
8. A tag according to claim 3, characterised in that said upper and lower barrier  
25 layers are formed of adhesive coated transparent material with the addition of cellulose material said cellulose material serving to promote adhesion of the reactive material to the upper and lower barrier layers.

9. A tag according to claim 3, characterised in that said upper and lower barrier layers are made of a thin film product.
10. A tag according to claim 9, characterised in that said thin film product is a co-polymer of ethylene and polyvinyl alcohol and adducts thereof having a maximum rate of oxygen and carbon dioxide diffusion therethrough of the order of  $0.5\text{cm}^3/\text{m}^2/24\text{hr}$  for a 20 micron film thickness at 0 % relative humidity and atmospheric pressure.
11. A tag according to claim 9, characterised in that said thin film product is selected from a, biaxially oriented ethyl vinyl alcohol, polyvinyl alcohol, polypropylene and adducts thereof.
12. A tag according to claim 9, characterised in that said thin film product is a polyvinyl alcohol film or a film of adducts thereof having a maximum oxygen permeability of  $0.25\text{cm}^3/\text{m}^2/24\text{hr}$  for a 14 nanometre film thickness at a relative humidity of 35% and atmospheric pressure.
13. A tag according to claim 9, characterised in that said thin film product is a co-polymer of ethylene and vinyl alcohol and adducts thereof having a maximum gas permeability of:  
 $\text{O}_2$   $0.2\text{cm}^3/\text{m}^2/24\text{hr}$  for a 20 micron film thickness at 0% relative humidity and atmospheric pressure; and  
 $\text{CO}_2$   $0.8\text{cm}^3/\text{m}^2/24\text{hr}$  for a 20 micron film thickness at 0% relative humidity and atmospheric pressure.
14. A tag according to claim 3, characterised in that said upper and lower barrier layers are constituted as laminate films, at least one layer of said laminate film being a substantially gas impermeable barrier film formed as mono or co-extruded film.
15. A tag according to claim 3, characterised in that said upper and lower barrier layers additionally include oxygen scavenging materials, or ultra violet resistant materials or wherein the upper and lower barrier layers incorporate ultra violet stabilized plastics materials such as polycarbonate, treated polycarbonate and adducts thereof with anti-oxidant materials such as butylated hydroxy-toluene.

16. A tag according to claim 1, characterised in that said reactive material is a vat dye matrix changeable between an un-oxidised condition in the absence of oxygen and an oxidised, colour changed, condition in the presence of oxygen.
- 5 17. A tag according to claim 16, characterised in that said reactive material comprises a matrix of a vat dye in a base material which may be selected from any suitable material such as a protein matrix, an agar-agar matrix, a polyvinyl acetate/alcohol matrix, a methyl-cellulose matrix, a cellulose acetate matrix and adducts thereof.
- 10 18. A tag according to claim 17, characterised in that said reactive material includes an un-oxidised vat dye composition in the proportion 0.001%-50 % wt/wt the base material forming the balance.
19. A tag according to claim 16, characterised in that said un-oxidised vat dye composition contains indigo 3.8mM, sodium hydroxide 0.3M, sodium dithionite 0.24M and has a pH within the range 4-10.
- 15 20. A tag according to claim 17, characterised in that said base material is a casein hydrochloride adhesive material which acts as a matrix material containing the vat dye.
21. A tag according to claim 17, characterised in that said base material is constituted by a polyvinyl acetate material hydrolysed and converted in situ to a polyvinyl alcohol vat dye adduct.
- 20 22. A tag according to any one of the preceding claims, characterised in that the oxygen reactive layer is secured between two substantially oxygen impermeable layers such that oxygen penetrates the tag through lateral diffusion into exposed edges of the tag, whereby, the reactive intermediate layer undergoes a colour change that commences at the periphery of the tag and that gradually moves  
25 towards a centre of the tag.
23. A method of manufacturing a tag according to any one of the preceding claims, characterised in that the tag is affixed to a product package in an oxygen-depleted atmosphere such that the tag will undergo the colour change reaction on opening of the package in an oxygen containing atmosphere.

24. A method according to claim 23, characterised in that the tag is placed on the package and subsequently covered in an air excluding covering such as a shrink wrap covering.
25. A method according to claim 24, characterised in that a shrink-wrap package is placed around the tag in an oxygen free atmosphere.
26. A method according to claim 25, characterised in that the tag is located on a product packaging in a non oxidising atmosphere, such as a carbon dioxide environment, a nitrogen environment or a vacuum or combinations thereof and subsequently wrapped in a gas impermeable covering.
27. A method according to claim 25, characterised in that the tag is placed in a package having a positive pressure and a non-oxidising gas within the package.
28. A method according to claim 25, characterised in that the tag is placed in a package having a negative pressure and a non-oxidising gas within the package.
29. A tag according to any one of claims 1 to 22, characterised in that the tag is visualized using an electronic optical detection device.

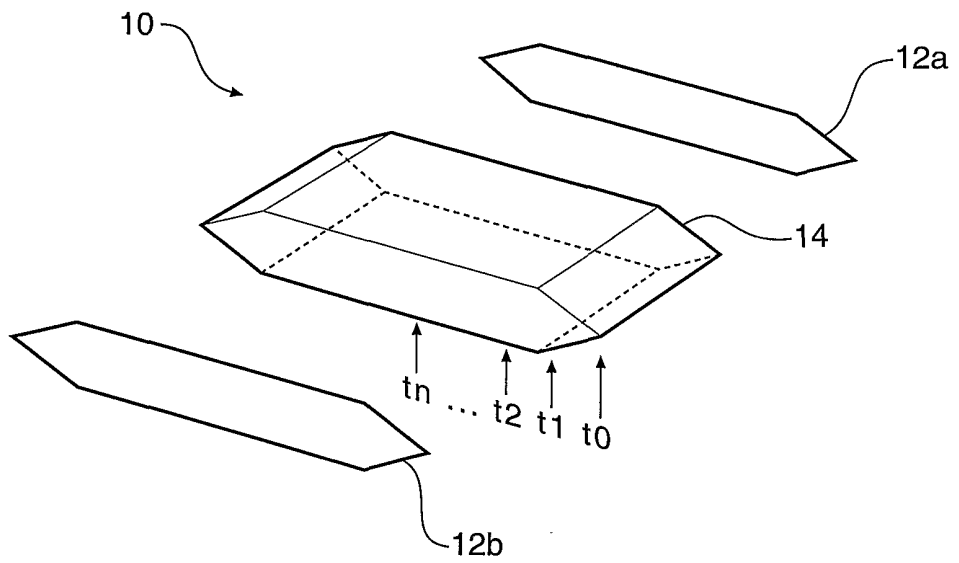
Dated: 5 December 2001

Gareth Lewis

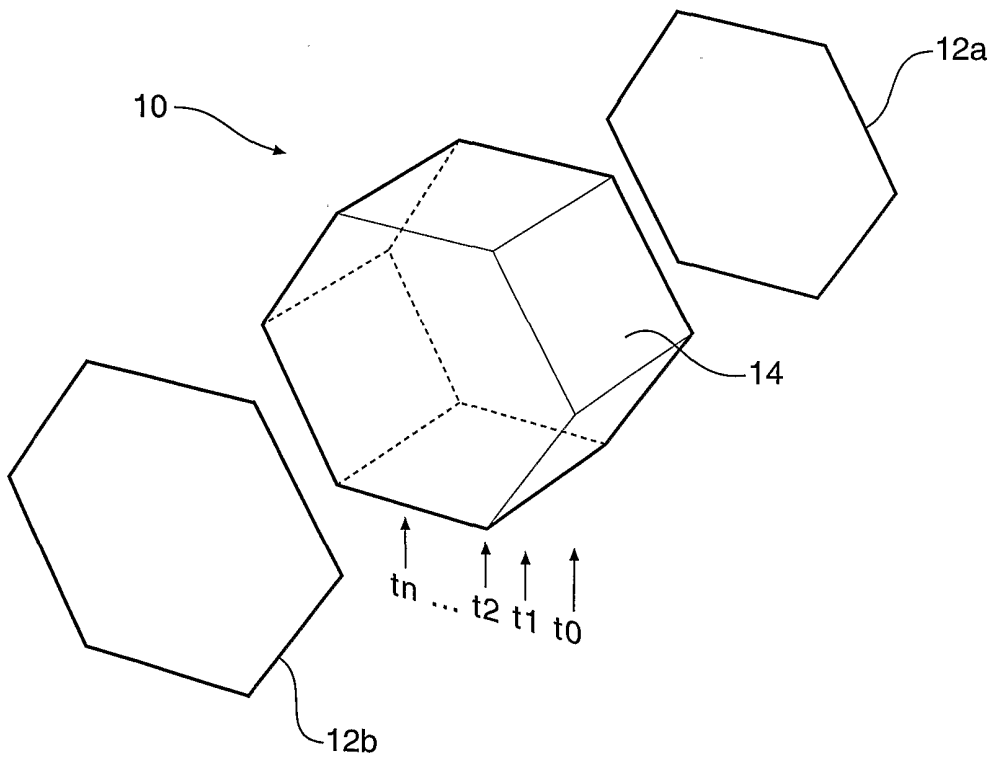
By his Patent Attorneys

Lesicar Perrin

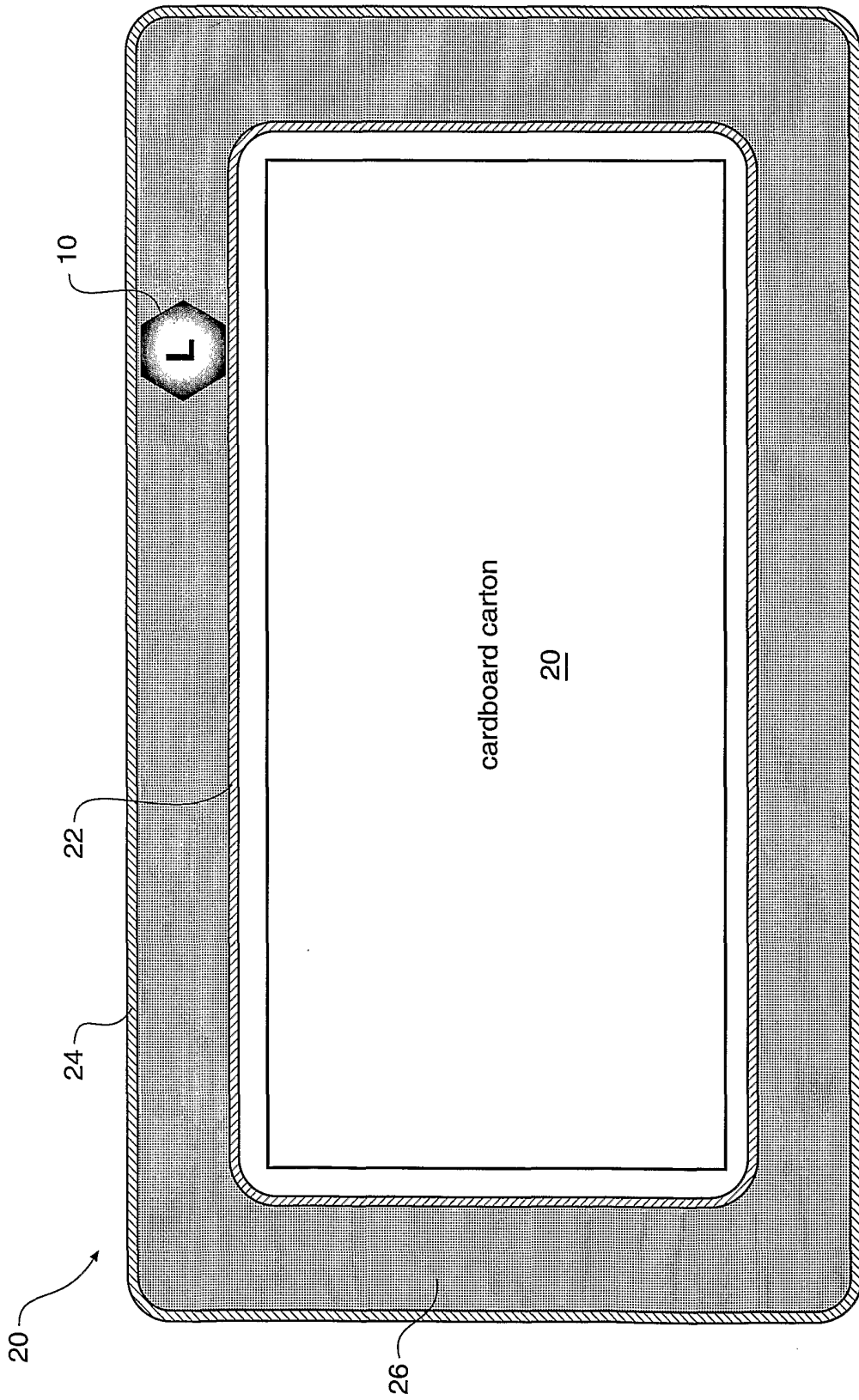
1/11



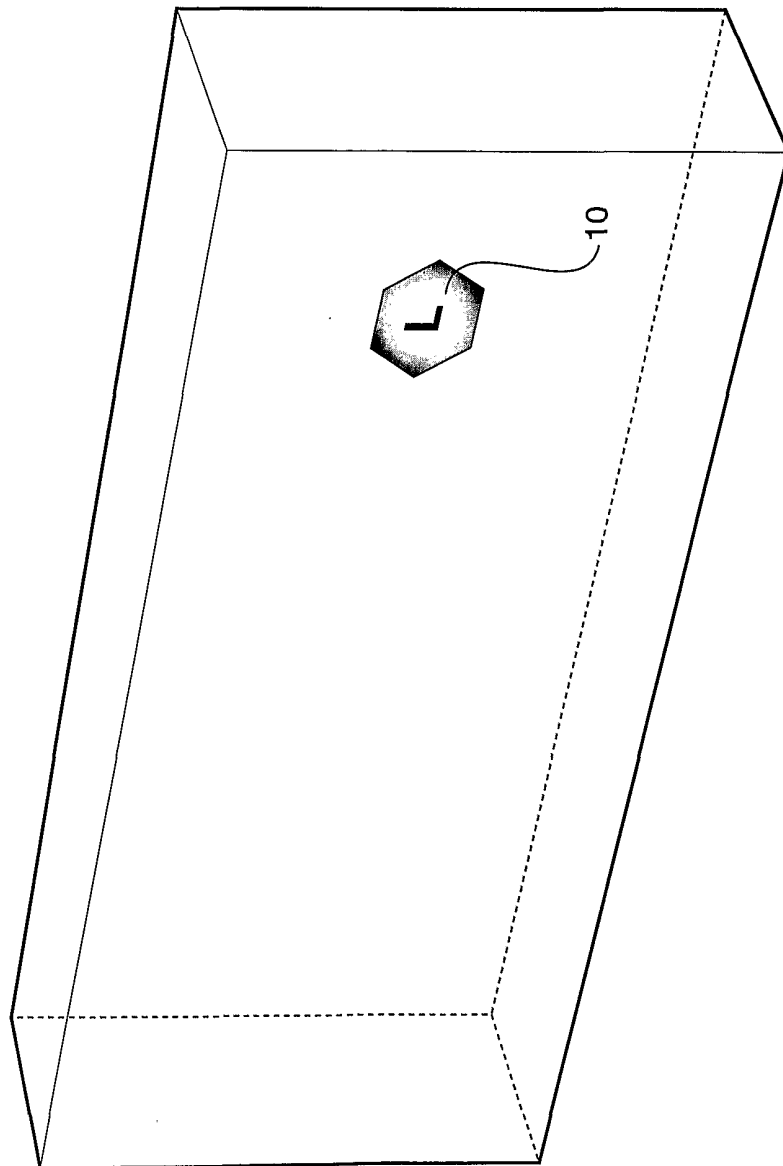
**Fig 1a**



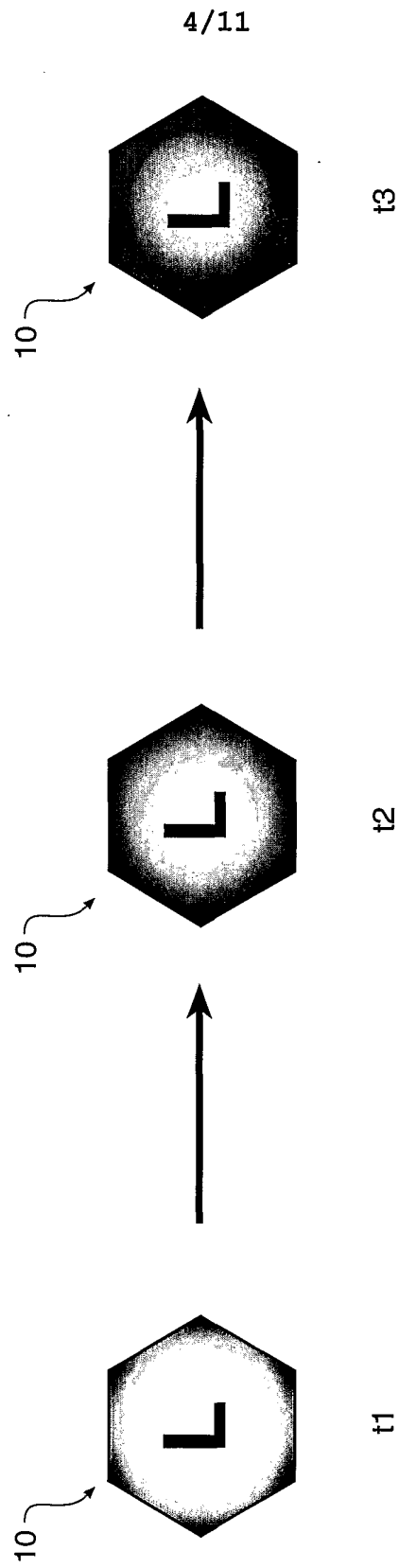
**Fig 1b**



**Fig 2**



**Fig 3**



**Fig 4**

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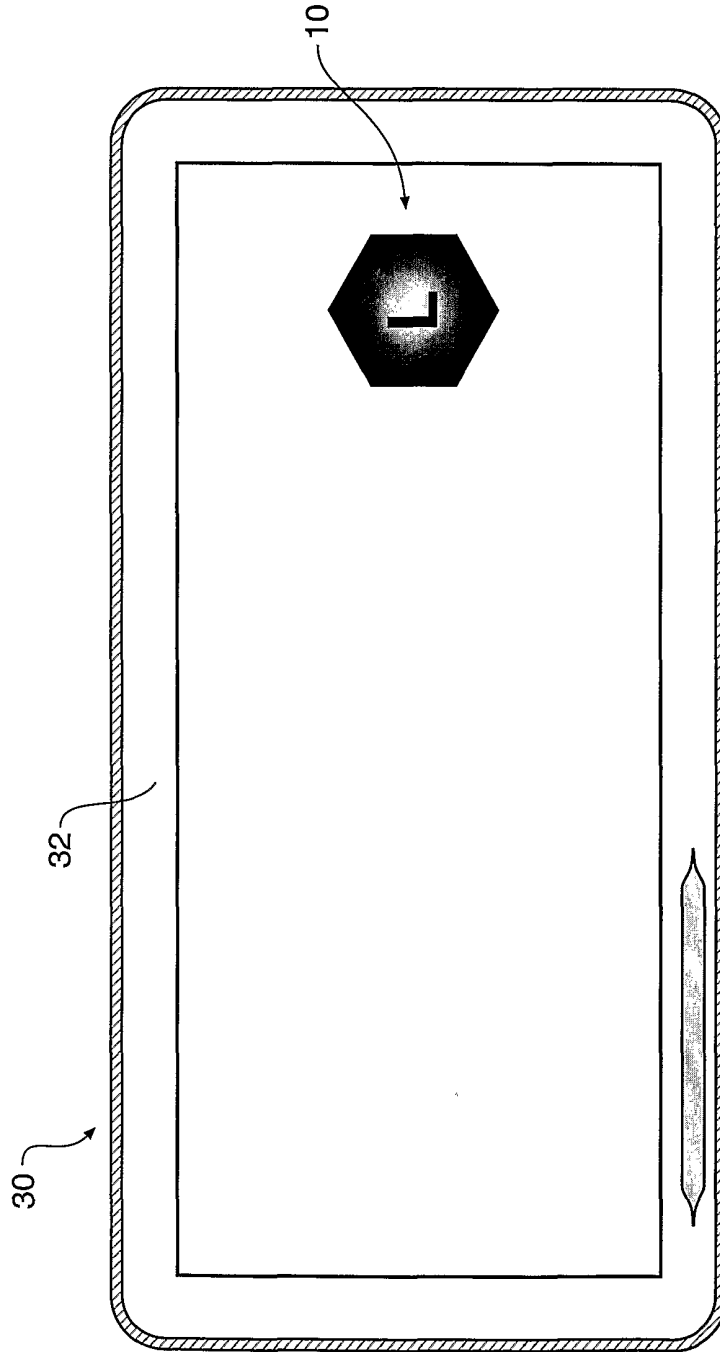
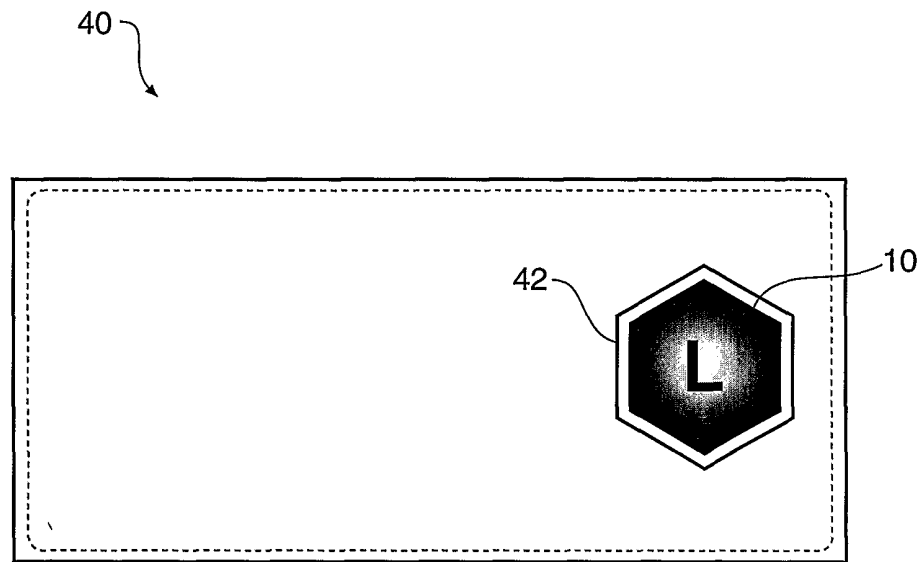
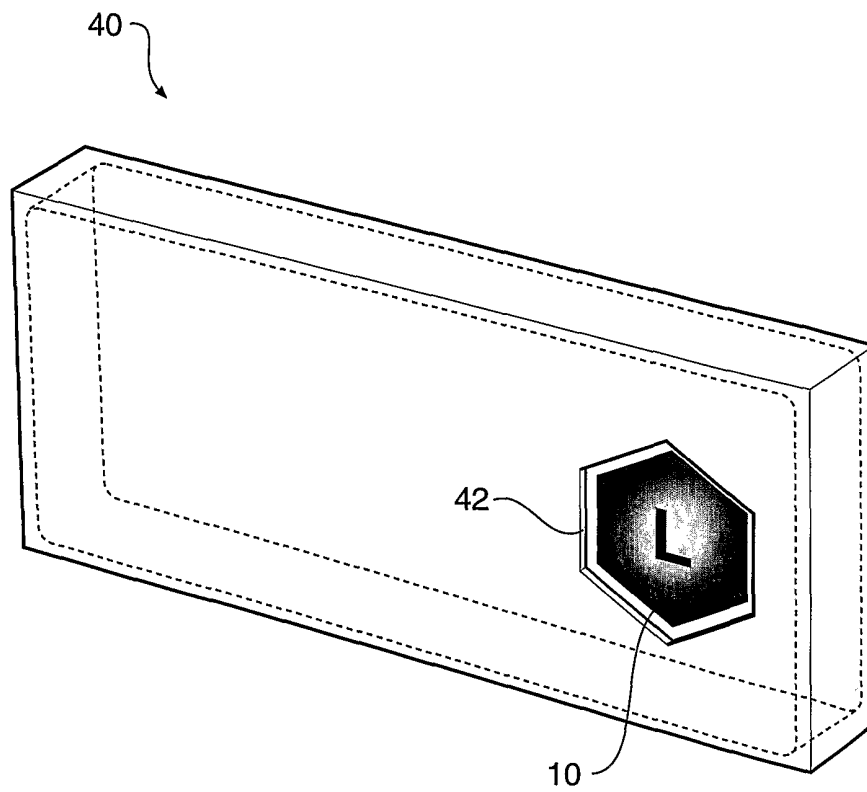


Fig 5



**Fig 6a**



**Fig 6b**

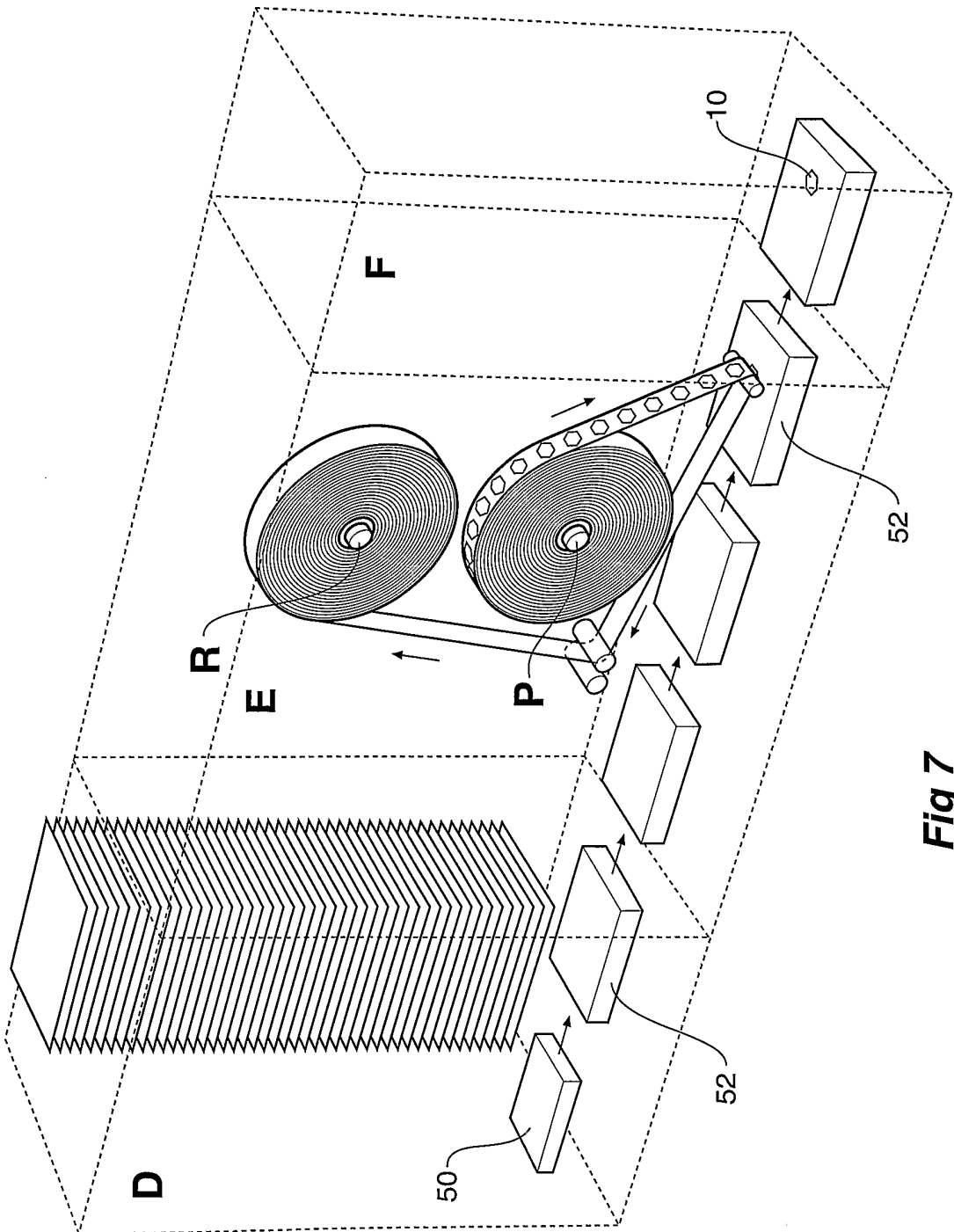


Fig 7

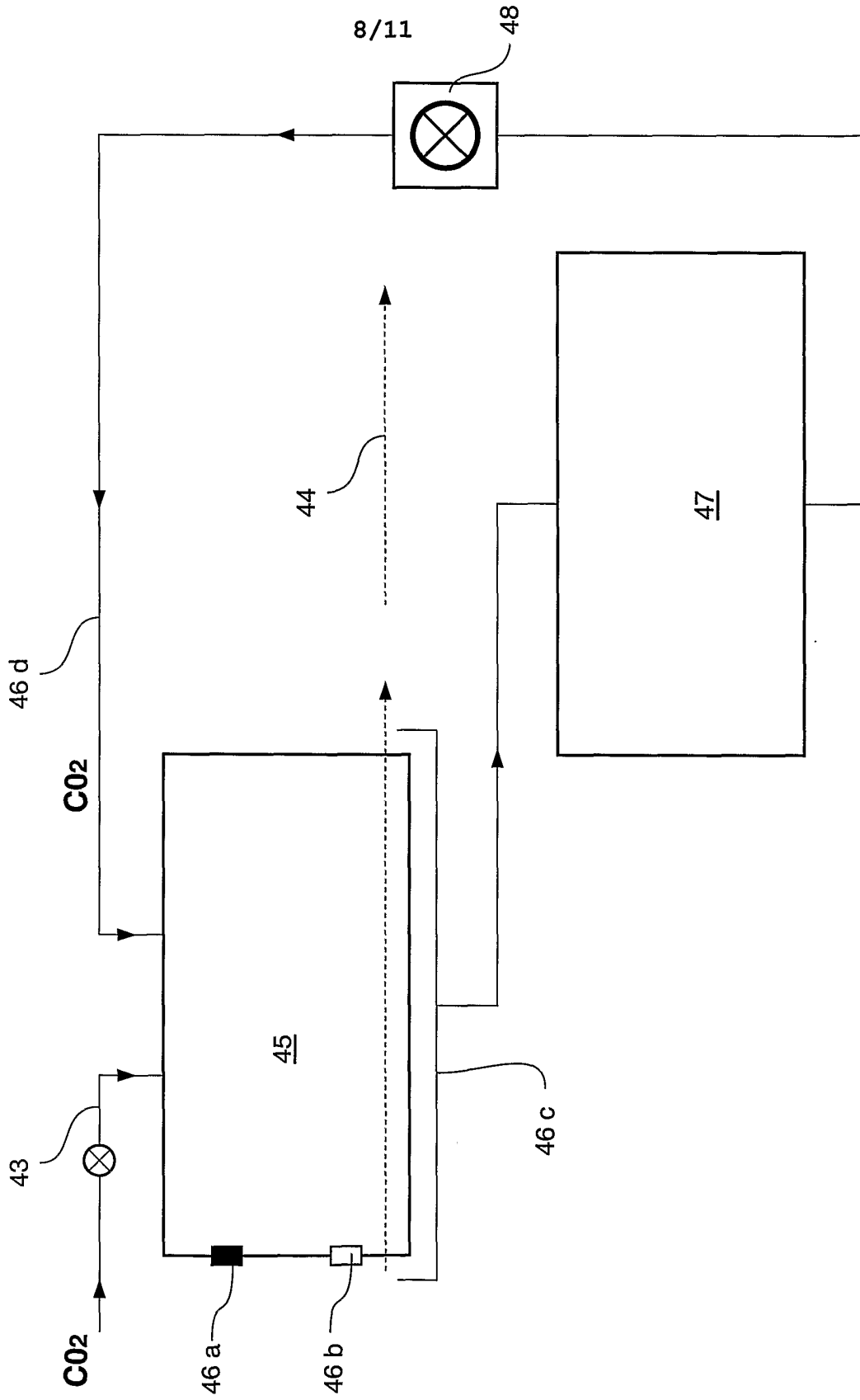
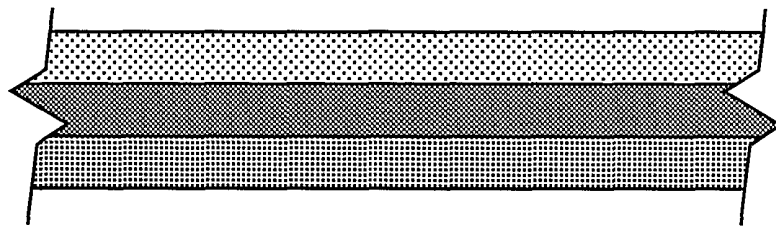
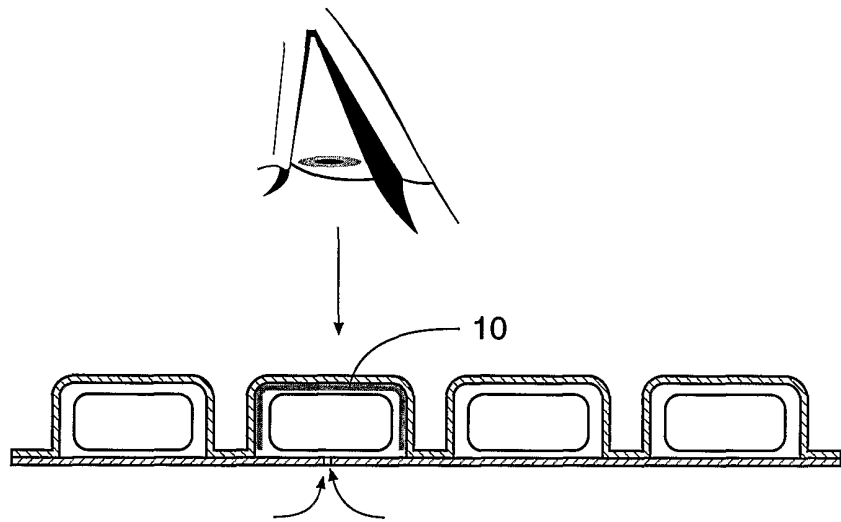


Fig 8

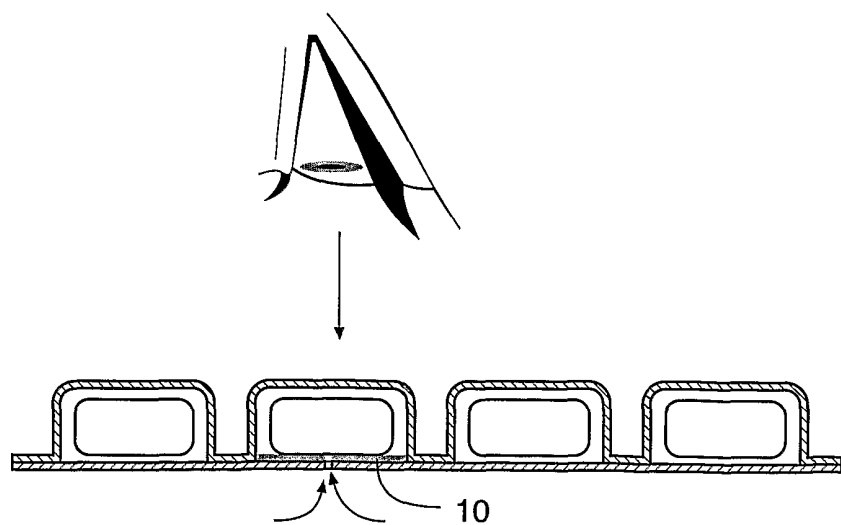
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**Fig 9**



**Fig 10**



**Fig 11**

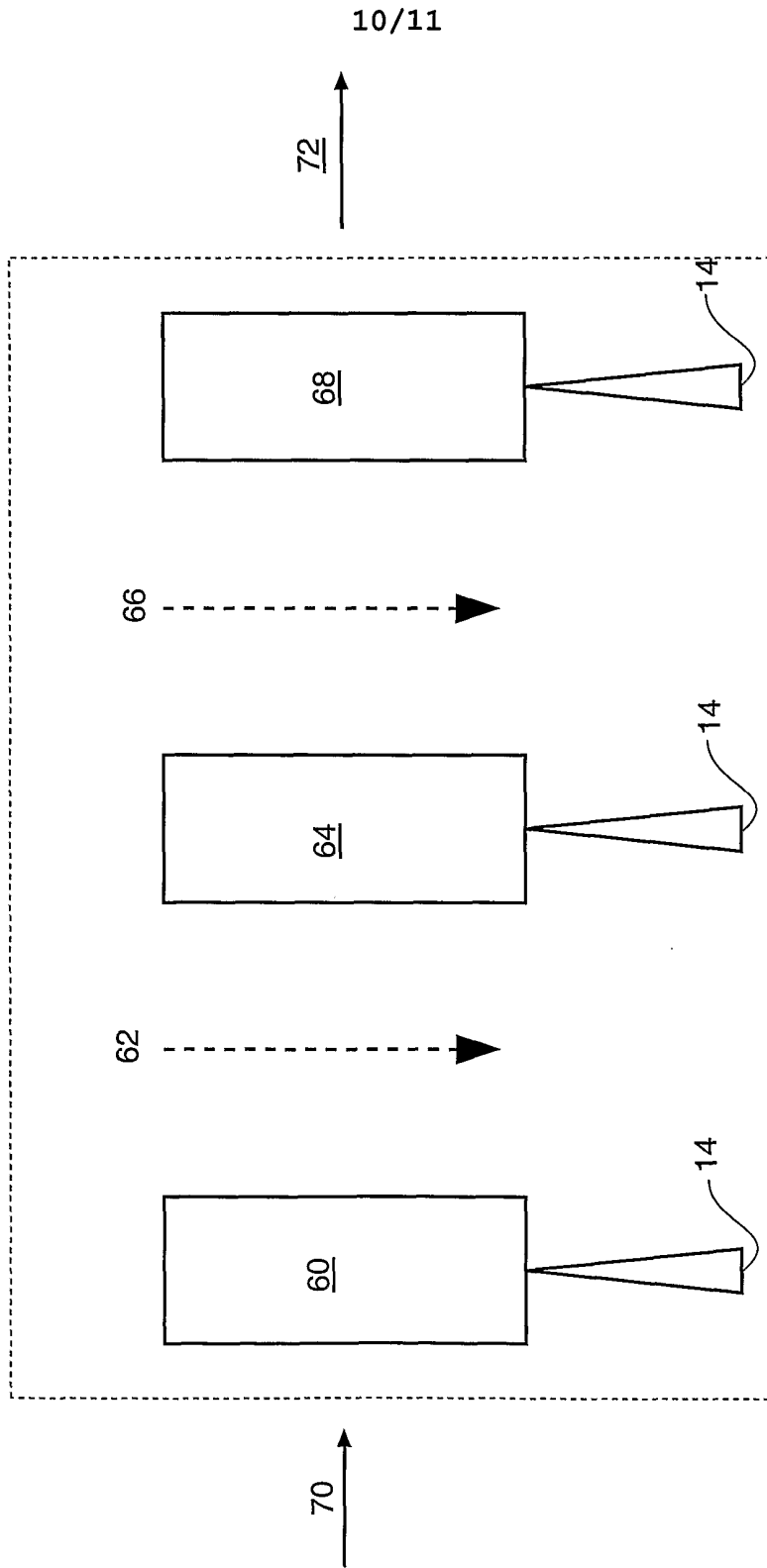
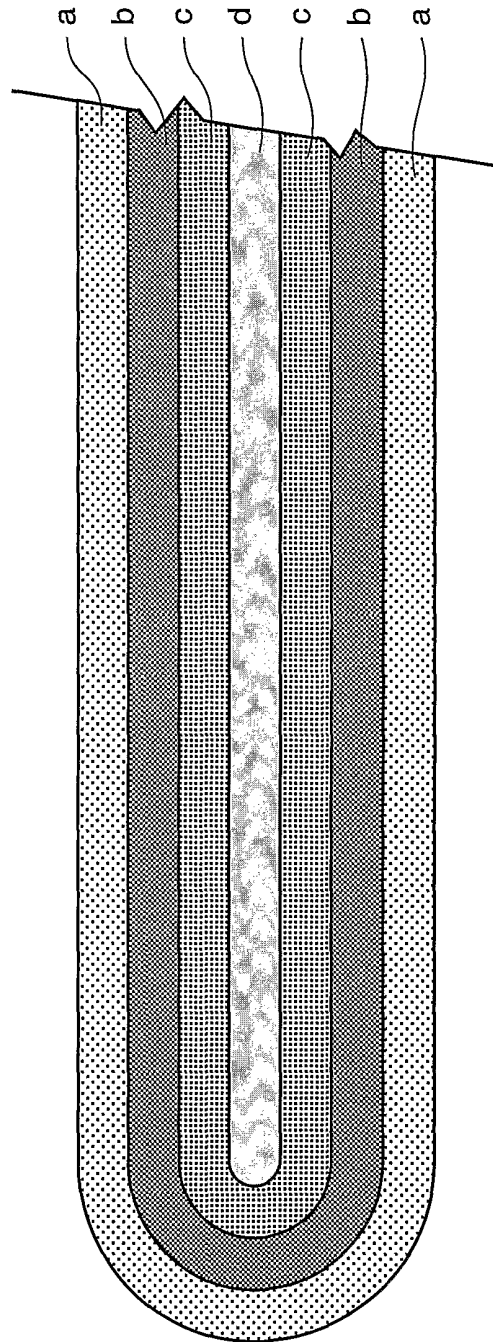


Fig 12

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**Fig 13**

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU01/01567

<b>A. CLASSIFICATION OF SUBJECT MATTER</b>		
Int. Cl. <sup>7</sup> : B65D 55/02, 75/54 // B65D 101:00, G01N 21/78, 31/22, G09F 3/02		
According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b>		
Minimum documentation searched (classification system followed by classification symbols)		
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) DWPI, JAPIO: Int. Cl. <sup>7</sup> G01N 15/08, 21/-, 31/22, G09F 3/-, B65D, A61J 1/-, B65B 25/-, 31/-, A23L 3/34+, 3/35+, tamper, oxygen, colour change.		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	WO 01/53106 A1 (SCIENTIFIC GENERICS LIMITED) 26 July 2001 Whole document	1-29
X	US 4505399 A (WEINER) 19 March 1985 Whole document	1-29
X	GB 2243825 A (ROBERT PETER SUNMAN) 13 November 1991 Whole document	1-29
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C <input checked="" type="checkbox"/> See patent family annex		
* Special categories of cited documents:		
"A"	document defining the general state of the art which is not considered to be of particular relevance	"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
"E"	earlier application or patent but published on or after the international filing date	"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
"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)	"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
"O"	document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P"	document published prior to the international filing date but later than the priority date claimed	
Date of the actual completion of the international search 18 January 2002	Date of mailing of the international search report - 2 FEB 2002	
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized officer  <b>ZOE BRADY</b> Telephone No : (02) 6283 7947	

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU01/01567

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5096813 A (KRUMHAR et al.) 17 March 1992 Whole document	1-29
X	GB 2251608 A (BOWES DARBY DESIGN ASSOCIATES LIMITED) 15 July 1992 Whole document	1-29
X	DE 19840269 A1 (SELECTRONIC GESELLSCHAFT FUR SICHERHEITSTECHNIK & SONDERELEKTRONIK) 9 March 2000 Abstract, Figure 1	1
X	Patent Abstracts of Japan, JP 04-151554 A (DAINIPPON PRINTING CO LTD) 25 May 1992 Abstract	1

**INTERNATIONAL SEARCH REPORT**  
Information on patent family members

International application No.  
**PCT/AU01/01567**

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report	Patent Family Member
WO 200153106	AU 200125381
US 4505399	NONE
GB 2243825	NONE
US 5096813	NONE
GB 2251608	AU 11607/92      WO 9212068
DE 19840269	NONE
JP 4151554	NONE
END OF ANNEX	