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(54) Title: PACKING MATERIAL

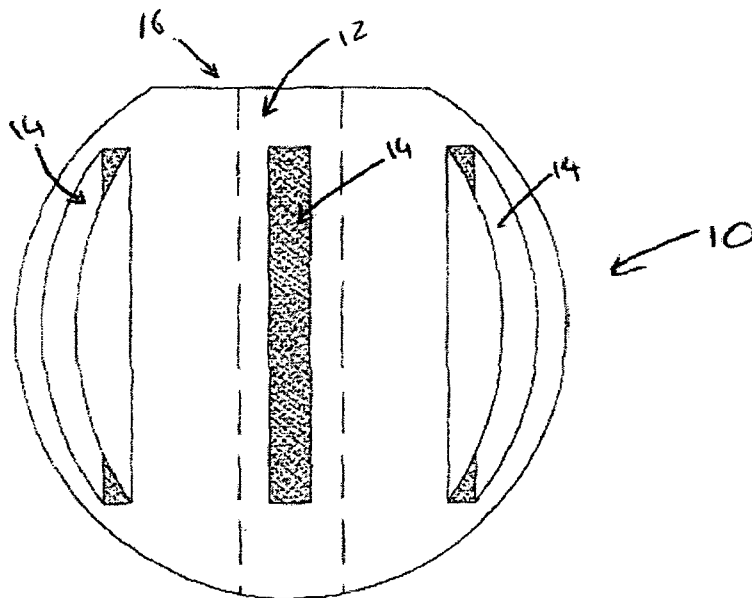


Figure 1

(57) Abstract: The invention provides a packing material unit for use in high temperature fluid flow applications. The unit is generally spherically shaped with at least one formation defined in the volume of the sphere, and which is manufactured from ceramic material which is selected from material which retains its structural integrity in temperatures of at least 800°C with maximum temperatures ranging to about 1700 °C. The formation may be selected from a channel or an open ended hollow which extends through the volume of the sphere, or both.



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Title: Packing Material**Technical field of the invention**

5

This invention relates to packing material for use in high temperature fluid flow applications.

Background to the invention

10

The applicant is aware that solid ceramic spheres and other monolithic shapes are being used as packing material in high temperature gas flow applications such as catalyst support and structural elements and filling material in gas reformers, gas converters, kilns, heat exchangers, columns, stacks, smelters and reactors where gas flow is required. Packing material includes support and filler material. The functions of the packing material are generally to provide support for catalysts used in various chemical conversion reactions wherein the reagents are in a gaseous state and to increase reagent contact, mixing and it is also used for catalyst dilution. With regard to catalyst support, a high surface area may be important and structural strength is a requirement. With regard to gas mixing and chemical reagent contact, the number, direction changes and diameter of the tortuous flow paths are important factors.

25

The traditional solid spheres, while relatively simple to manufacture and structurally sound, only provide tortuous flow paths between the spaces formed between packed spheres and the surface area is limited to the surfaces of the spheres. In certain applications the tortuous flow paths get clogged with solid material deposits and naturally it will take a longer time to block a bigger volume of flow paths and improved mixing will lead to a lower rate of solid material being deposited.

30

It is an object of this invention to provide packing material for high temperature fluid flow applications, with a high contact area, and improved

tortuous flow paths. It will be appreciated that any reference to fluids includes material in a gas and/ or liquid phase and that gas-liquid phase changes often occur during reactions. Fluid flow would be understood to include gaseous flow.

5

General description of the invention

According to a first aspect of the invention there is provided a packing material unit for use in high temperature fluid flow applications, which unit is generally spherically shaped with at least one formation defined in the volume of the sphere, and which is manufactured from ceramic material which is selected from material which retains its structural integrity in temperatures of at least 800°C with maximum temperatures ranging to about 1700 °C.

10 The formation may be selected from a channel or an open ended hollow which extends through the volume of the sphere, or both. It will be appreciated that the formations increase the surface area of the unit and provides additional flow paths for flowing fluids, in use.

15 The unit may include a plurality of formations defined therein of which one is an open ended hollow which extends through the volume of the sphere with the remainder of the formations being at least two channels.

20 The channels may intersect with the open ended hollow to provide fluid flow access to the inside of the sphere, an intersecting channel.

25 The channels may be configured not to intersect with the open ended hollow to provide fluid flow access through the generally outer perimeter of the sphere, a non intersecting channel.

30

The unit may include a plurality of formations defined therein of which one is an open ended hollow which extends through the volume of the sphere with the remainder of the formations being at least one intersecting channel

and one being a non intersecting channel. The unit may include respective pairs of intersecting channels and non intersecting channels.

One or more, preferably all, channels may be arranged in parallel.

5

The ceramic material may be selected from ceramics which can retain its structural integrity in temperatures of up to 1700°C.

10 The ceramic material may be selected from ceramics which is inert up to temperatures of up to 1700°C.

The ceramic material may be selected from ceramics from which minerals do not leach into its surroundings, in use, up to temperatures of 1700°C.

15

20 The ceramic material may a high alumina (Al_2O_3) content, preferably above 70%, more preferably above 99% and which is sintered above a temperature of 1600°C. Other high melting, stable oxide ceramics such as Zirconia (ZrO_2), Magnesia (MgO) and Spinell will also find application within the invention. Non-oxide ceramics such as certain variations of recrystallized Silicon Carbide (SiC) may be used depending on the atmosphere in the reformer / catalyst stack as well as the chemical stability thereof.

25 According to a further aspect of the invention there is provided a method of manufacturing a packing material unit for use in high temperature fluid flow applications.

30 LINE-OX[®] Hollow spheres made from LINE-OX[®] 72 to LINE-OX[®] 99 materials, are manufactured by means of a centreless casting technique, developed in-house by the applicant and utilizing different materials ranging from polished brass and aluminium to plaster of paris.

This technique may include the steps:

- 1.) The manufacture of an over-sized pattern of the required shape

- 2.) Subsequent manufacture of plaster of paris; metal composite; or plastics material such as Poly Urethane, rubber or silicone moulds.
- 3.) Preparation of casting slip in accordance with internal works procedures for different grades of Alumina. Water and electrolytes are added to the dry alumina powders to create a castable slip in controlled environments and measured against internal required flow and density properties.
- 4.) Timed casting process where casting slip is gravity fed or pressure fed into the composite mould.
- 5.) Depressurizing and opening of mould cavity.
- 6.) Demoulding LINE-OX[®] hollow sphere.
- 7.) Fettling and further subsequent value addition.
- 8.) Pre-sintering.
- 9.) Final sintering at > 1500 °C.

LINE-OX[®] Hollow spheres material composition:

Materials range from 72 % Alumina to >99.7 % Alumina. The alumina being a high temperature oxide ceramic material. Other materials covered by the scope of the invention are LINE-OX[®] Mullite, Corundum, Zirconium and composites thereof.

LINE-OX[®] Semi-solid spheres, made in coarse – grained materials such as LINE-OX[®] SAS 1600 , are manufactured by means of a vibratory casting technique utilizing different materials ranging from polished brass and aluminium to plaster of paris, or a semi-wet or dry pressing technique, utilizing steel tooling / moulds and hydraulic or mechanical presses. There are variations of the semi-solid sphere, depending on the application. Larger spheres with slots are used at the exit side of the stack, giving support. Smaller diameter semi-solid spheres are dispersed.

This technique may include the steps:

- 1.) The manufacture of an over-sized pattern or press tool cavity for the required shape.

- 2.) Subsequent manufacture of plaster of paris; metal composite; or plastics material such as Poly Urethane, rubber or silicone moulds.
- 3.) Preparation of vibratory ceramic body in accordance with internal works procedures for different grades of Alumina.
- 5 4.) Vibratory casting process where casting slip is gravity fed or pressure fed into composite mould. Alternatively the compaction of coarse grained raw material blends in conjunction with binder systems in hydraulic or mechanical presses.
- 5.) Demoulding LINE-OX[®] hollow sphere.
- 10 6.) Fettling and further subsequent value addition.
- 7.) Pre-sintering.
- 8.) Final sintering at > 1500 °C.

LINE-OX[®] Semi-solid spheres material composition:

- 15 Materials used are blended from a wide range of particle sizes. Highly calcined alumina types such as Tabular Alumina, in grain sizes ranging from 6 mm down to 2 micron are utilized the relative proportions of each particle size being dependent on the required density, flow properties and final application requirements (e.g. improvement of hardness, thermal shock resistance,
- 20 chemical corrosion resistance etc.)

According to a further aspect of the invention there is provided the use of packing material, in accordance with the invention to aerate the catalyst bed itself. Hollow spheres are placed in the catalyst area to ensure

25 that sedimentation of debris from upstream processes do not block off gas flow through the catalyst completely. Differently orientated slots and holes also ensure proper mixing and agitation of gases to optimise contact area with surrounding catalyst.

30 LINE-OX[®] Semi-solid spheres have the following functions or advantages:

- 1.) Optimising reaction area in reformer / catalyst stack.
- 2.) Avoid drop in pressure over the entire depth of the catalytic bed.
- 3.) Give structural support, carrying the catalyst bed.

- 4.) allow for unrestricted gas flow.
- 5.) maintain temperature stability and chemical inertness of the complete stack.

5 Detailed description of the invention

The invention is now described by way of example with reference to the accompanying drawings.

10 In the drawings:

Figure 1 shows a side view of a first embodiment of packing material unit, in accordance with the invention;

Figure 2 shows a sectional plan view of the first embodiment of packing material unit;

15 Figure 3 shows a side view of a second embodiment of packing material unit, in accordance with the invention;

Figure 4 shows a sectional plan view through A-B of the second embodiment of packing material unit;

20 Figure 5 shows a plan view of a third embodiment of packing material unit, in accordance with the invention;

Figure 6 shows a perspective view of the third embodiment of packing material unit.

25 Referring now to the drawings, the packing material unit, in accordance with the invention, is generally indicated by reference numeral 10.

The unit of packing material shown in figures 1 and 2 for use in high temperature fluid flow applications is manufactured from 90% alumina (Al_2O_3) ceramic material which retains its structural integrity at temperatures up to 30 1600 °C. The unit 10 which is generally spherically shaped with one open ended hollow 12 formation with a constant bore defined in the volume of the sphere and six parallel channels 14, which do not intersect with the hollow formation. The radius of the sphere in this example is 40mm, the radius of the bore of the hollow 12 is 7.5mm with the width of the channel being 6mm. The

sphere is further provided with a flat surface 16 perpendicular to the hollow with a diameter of 40mm. In a preferred example, not shown, the sides of the channels are rounded to help resist attrition.

5 The unit of packing material shown in figures 3 and 4 for use in high temperature fluid flow applications is manufactured from 99% alumina (Al_2O_3) ceramic material which retains its structural integrity at temperatures up to 1700 °C. The unit 10 which is generally spherically shaped with a generally constant wall thickness of 5mm and with one open ended hollow 12 formation
10 defined in the volume of the sphere and eight parallel channels 14., of which four channels 14.1 intersect with the hollow 12 and four channels 14.2 which do not intersect with the hollow formation. The width of the non intersecting channels is 5mm and the inner width of the non intersecting channels is 5mm with the outer width being 7.95.

15

These hollow sphere units 10 are made from LINE-OX[®] 99 materials, are manufactured by means of a centreless casting technique, developed in-house by the applicant and utilizing different materials ranging from polished brass and aluminium to plaster of paris.

20

This technique includes the steps:

- 1.) The manufacture of an over-sized pattern of the required shape.
- 2.) Subsequent manufacture of plaster of paris, metal composite moulds.
- 25 3.) Preparation of casting slip in accordance with internal works procedures for different grades of Alumina. Water and electrolytes are added to the dry alumina powders to create a castable slip in controlled environments and measured against internal required flow and density properties.
- 30 4.) Timed casting process where casting slip is gravity fed or pressure fed into the composite mould.
- 5.) Depressurizing and opening of mould cavity.
- 6.) Demoulding LINE-OX[®] hollow sphere.
- 7.) Fettling and further subsequent value addition.

- 8.) Pre-sintering.
- 9.) Final sintering at > 1500 °C.

LINE-OX[®] Hollow spheres material composition comprises 99%
5 Alumina, the alumina being a high temperature oxide ceramic material.

The unit of packing material shown in figures 5 and 6 for use in high
temperature fluid flow applications is manufactured from 99% alumina (Al₂O₃)
ceramic material which retains its structural integrity at temperatures up to
10 1700 °C. The unit 10 which is generally spherically shaped with three open
ended constant bore hollow 12 formations defined in the volume of the
sphere. The diameter of the sphere is 45mm. The diameter of the respective
bores is 4, 10 and 20 mm. Each hollow is perpendicular to the other two
hollows and intersects each other at the centre point of the sphere.

15

The semi-solid sphere units shown in Figure 1, 2 5 and 6 are
manufactured from a coarse grained materials such as LINE-OX[®] SAS 1600,
by means of a vibratory casting technique utilizing different materials ranging
from polished brass and aluminium to plaster of paris.

20

This technique includes the steps:

- 1.) The manufacture of an over-sized pattern or press tool cavity for the
required shape.
- 2.) Subsequent manufacture of plaster of paris, metal composite
25 moulds.
- 3.) Preparation of vibratory ceramic body in accordance with internal
works procedures for different grades of Alumina.
- 4.) Vibratory casting process where casting slip is gravity fed or
pressure fed into composite mould. Alternatively the compaction of
30 coarse grained raw material blends in conjunction with binder
systems in hydraulic or mechanical presses.
- 5.) Demoulding LINE-OX[®] hollow sphere.
- 6.) Fettling and further subsequent value addition.
- 7.) Pre-sintering.

8.) Final sintering at > 1500 °C.

5 LINE-OX[®] Semi-solid spheres material composition comprises a blend from a wide range of particle sizes. Highly calcined alumina, Tabular Alumina, in grain sizes ranging from 6 mm down to 2 micron are utilized the relative proportions of each particle size being dependent on the required density, flow properties and final application requirements.

10 It shall be understood that the examples are provided for illustrating the invention further and to assist a person skilled in the art with understanding the invention and are not meant to be construed as unduly limiting the reasonable scope of the invention.

CLAIMS

1. A packing material unit for use in high temperature fluid flow applications, which unit is generally spherically shaped with at least one formation defined in the volume of the sphere, and which is manufactured from ceramic material which is selected from material which retains its structural integrity in temperatures of at least 800°C with maximum temperatures ranging to about 1700 °C.
2. A packing material unit as claimed in Claim 1, wherein the formation is selected from a channel or an open ended hollow which extends through the volume of the sphere, or both.
3. A packing material unit as claimed in Claim 1 or Claim 2, which includes a plurality of formations defined therein of which one is an open ended hollow which extends through the volume of the sphere with the remainder of the formations being at least two channels.
4. A packing material unit as claimed in Claim 3, wherein at least some of the channels are intersecting channels, which intersect with the open ended hollow to provide fluid flow access to the inside of the sphere.
5. A packing material unit as claimed in Claim 3 or Claim 4, wherein at least some of the channels are non-intersecting channels configured not to intersect with the open ended hollow to provide fluid flow access through the generally outer perimeter of the sphere.
6. A packing material unit as claimed in Claim 4 or Claim 5, which includes a plurality of formations defined therein of which one is an open ended hollow which extends through the volume of the sphere with the remainder of the formations being at least one intersecting channel and one being a non-intersecting channel.

7. A packing material unit as claimed in Claim 6, which includes respective pairs of intersecting channels and non intersecting channels.

8. A packing material unit as claimed in any one of Claim 3 to 7,
5 wherein the channels are arranged in parallel.

9. A packing material unit as claimed in any one of the previous claims, wherein the ceramic material is selected from ceramics which can retain its structural integrity in temperatures of up to 1700°C, which is inert up
10 to temperatures of up to 1700°C and from which minerals do not leach into its surroundings, in use, up to temperatures of 1700°C.

10. A packing material unit as claimed in any one of the previous claims, wherein the ceramic material comprises an alumina (Al_2O_3) content
15 above 70%.

11. A method of manufacturing a packing material unit for use in high temperature fluid flow applications, which method includes the steps of:
20 manufacturing an over-sized pattern of the required shape;
manufacture a mould;
preparing a casting slip;
casting wherein the casting slip is gravity fed or pressure fed into the composite mould;
depressurizing and opening of the mould cavity;
25 demoulding the formed hollow sphere; and
sintering.

12. The use of a packing material unit as claimed in any one of claims 1 to 10 to aerate a catalyst bed.

30

13. A packing material unit for use in high temperature fluid flow applications, substantially as described herein with reference to the accompanying drawings.

14. A method of manufacturing a packing material unit, substantially as described herein with reference to the accompanying drawings.

15. The use of a packing material unit, substantially as described
5 herein with reference to the accompanying drawings.

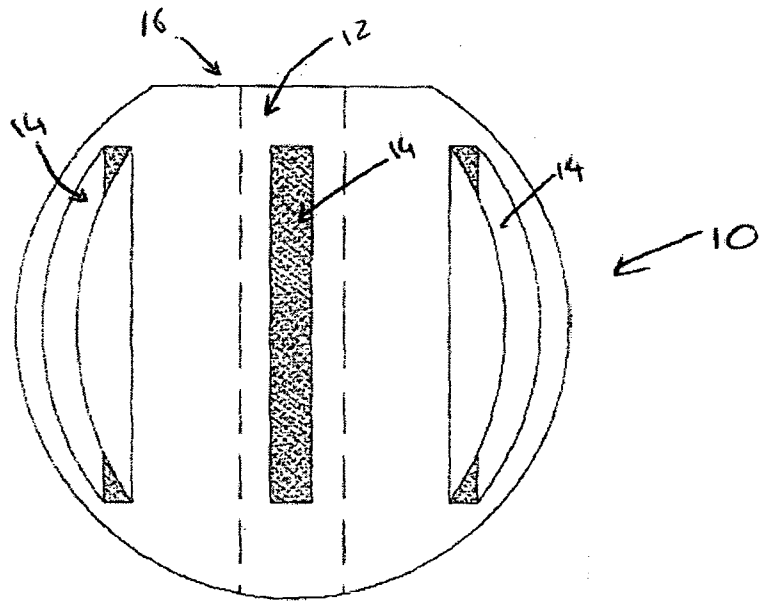


Figure 1

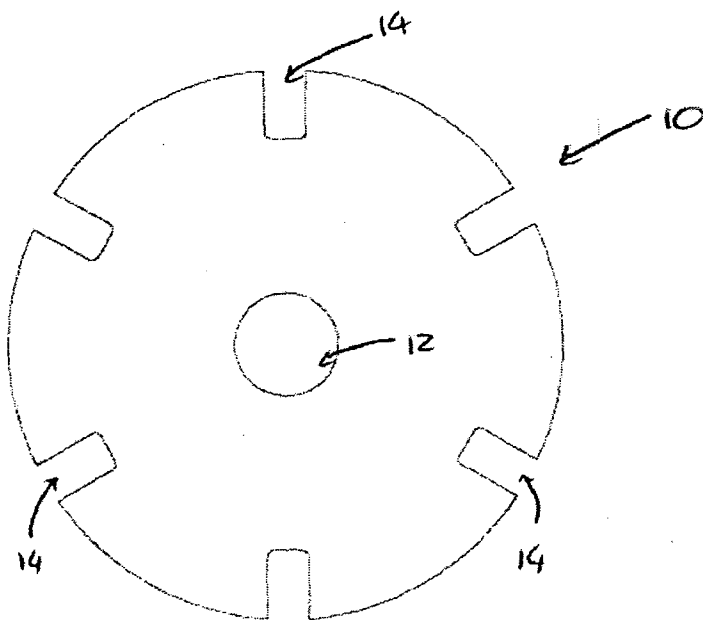


Figure 2

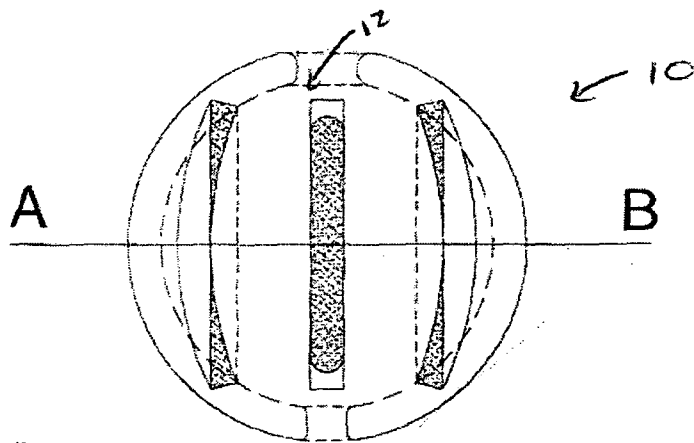


Figure 3

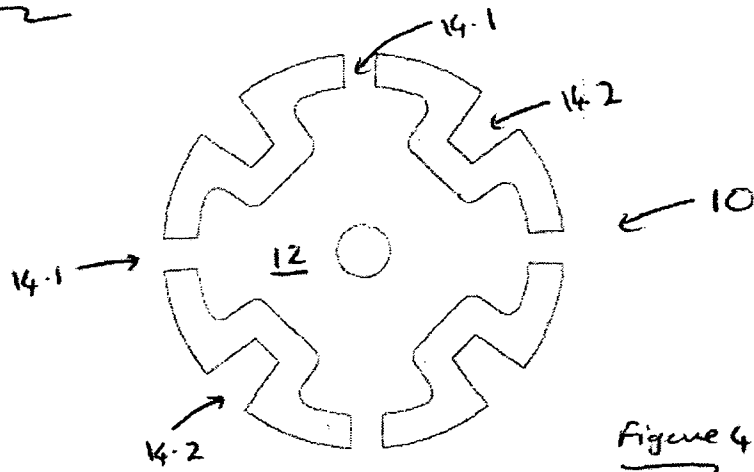


Figure 4

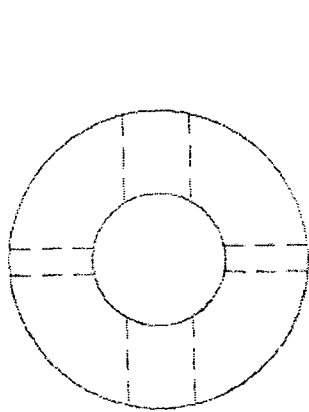


Figure 5

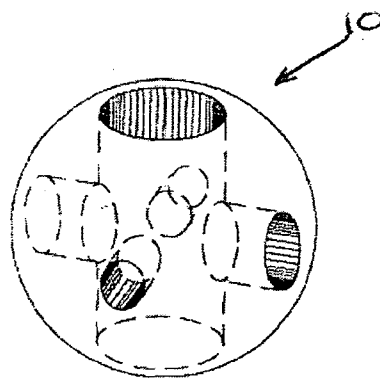


Figure 6

INTERNATIONAL SEARCH REPORT

International application No.
PCT/ZA 2009/000014

A. CLASSIFICATION OF SUBJECT MATTER IPC ⁸ : B01J 19/30 (2006.01); B01D 53/18 (2006.01) 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 ⁸ : B01D; B01J		
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) EPODOC, WPI		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2007/0182035 A1 (KAVOLIK, JR.) 9 August 2007 (09.08.2007) <i>The whole document, especially fig. 1-4; claims 1-3, 8.</i>	1-15
	--	
A	US 6547222 B2 (BLISCHAK et al.) 15 April 2003 (15.04.2003) <i>The whole document; especially fig. 1-9; claims 1&2.</i>	1-15
	--	
A	US 4113810 A (IKAWA) 12 September 1978 (12.09.1978) <i>The whole document.</i>	1-13
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<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 "E" earlier application or patent 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 "&" document member of the same patent family		
Date of the actual completion of the international search 25 January 2010 (25.01.2010)		Date of mailing of the international search report 19 February 2010 (19.02.2010)
Name and mailing address of the ISA/ AT Austrian Patent Office Dresdner Straße 87, A-1200 Vienna Facsimile No. +43 / 1 / 534 24 / 535		Authorized officer PUSTERER F. Telephone No. +43 / 1 / 534 24 / 311

INTERNATIONAL SEARCH REPORT

International application No.
PCT/ZA 2009/000014

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4581299 A (JÄGER) 8 April 1986 (08.04.1986) <i>The whole document.</i>	1-13
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/ZA 2009/000014

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