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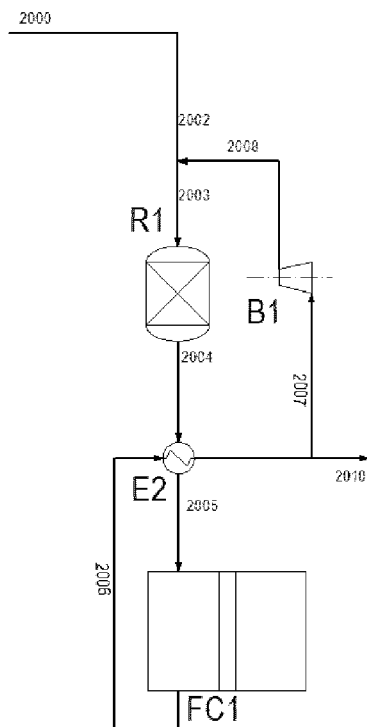
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[Continued on next page]

(54) Title: PRE-REFORMING OF SULFUR-CONTAINING FUELS TO PRODUCE SYNGAS FOR USE IN FUEL CELL SYSTEMS

Fig. 1



(57) Abstract: A method for the processing of hydrocarbon fuels by pre-reforming to produce an anode feed gas for use in connection with a fuel cell system comprises the steps of treating the hydrocarbon fuel with steam, with hydrogen or with syngas, or with combinations thereof in a pre-reformer (R1) to convert the fuel to syngas and removing at least a portion of the sulfur species from the fuel, feeding the syngas to the anode inlet of a fuel cell system (FC1) and optionally recirculating a split of the anode off-gas from the fuel cell system, to the inlet of the pre-reformer (R1), suitably via a heat exchanger (E2) and a recycle blower (B1).

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**Pre-reforming of sulfur-containing fuels to produce syngas
for use in fuel cell systems**

5 The present invention concerns pre-reforming of sulfur-
containing fuels, such as hydrocarbon fuels, to produce
syngas for use in fuel cell systems. More specifically, the
invention concerns a method for the processing of hydrocar-
bon fuels by pre-reforming to produce an anode feed gas for
use in connection with a fuel cell system. The invention
10 also concerns a solid oxide fuel cell assembly supplied
with fuel obtained by the method.

Previously, high pressure steam reforming of diesel has
successfully been tested by the applicant in combination
15 with an upstream hydrodesulfurization (HDS)- ZnO bed. This
process requires pressurized hydrogen. The presence of wa-
ter within the HDS reactor and ZnO bed is most undesirable.

The basic idea underlying the present invention, which will
20 be described in more detail below, consists in using a pre-
reformer also as a sulfur trap. The off-gas from the solid
oxide fuel cell downstream of the pre-reformer, which typi-
cally contains mainly H₂, N₂, H₂O, CO and CO₂, can be used
directly in a recycle loop for mixing with the incoming hy-
25 drocarbon fuel. To avoid carbon formation, a suitable
amount of H₂ is supplied. The method according to the in-
vention requires neither a separate hydrodesulfurization
unit nor a ZnO bed.

30 The idea of mixing the anode recycle gas with incoming fuel
upstream the HDS and an adiabatic reforming process is de-
scribed in WO 2010/044772 A1. However, according to this

prior art document a separate hydrodesulfurization unit is mandatory, and the described system is strictly related to a desulfurization-reformer configuration.

5 A fuel cell system, wherein the fuel is made by adiabatic pre-reforming of a higher carbon (C₂₊) hydrocarbon fuel with steam, is disclosed in US 6.841.279 B1. This patent is however silent as to how the problems with sulfur are dealt with.

10

Other fuel cell systems are described in the following documents: US 2010/0178574 A1 disclosing a system with partial external reforming and direct internal reforming, JP 11273703 A describing the use of an additional adiabatic reformer in the anode recycle stream before mixing with the reformate, which is supplied by the main reformer, and US 15 5.147.735 B1 describing a method of operating a solid electrolyte fuel cell, in which the use of a PSA (pressure swing adsorption) system is considered for the air side 20 (the cathode) to increase the oxygen partial pressure.

Fuel cells are electrochemical systems which generate electric current by chemically reacting a fuel gas and an oxidant gas on the electrode surfaces. Conventionally the oxidant gas is oxygen or air, and the fuel gas is hydrogen or 25 a mixture of hydrogen, carbon oxides and traces of hydrocarbons. The specific fuel gas composition requirements depend on the type of fuel cell. Low temperature fuel cells, such as proton exchange membrane (PEM) cells and alkaline fuel cells (AFCs), can only utilize hydrogen as fuel, and 30 they contain precious metal catalysts that become poisoned by carbon monoxide. High temperature fuel cells, such as

solid oxide fuel cells (SOFCs) and molten carbonate fuel cells (MCFCs), do not usually contain precious metal catalysts, and can utilize hydrogen containing carbon monoxide and methane as fuel. Most fuel cell types are adversely affected by sulfur compounds.

Pure hydrogen is the ideal fuel for lots of fuel cell types, but it is not widely available. Moreover, storage and transportation involves large, heavy and costly means, such as compressed gas bottles. In practice fuel cells must therefore utilize commonly available and easily transported fuels including natural gas, methanol, ethanol, diesel fuel and hydrocarbon fuel. These hydrocarbons and alcohols must be reformed to a fuel gas that is suitable for the particular fuel cell application. In addition, these fuels often contain sulfur that has to be removed.

The conversion of common hydrocarbon fuels, such as gasoline, into a gas rich in hydrogen for use in electricity-producing fuel cell systems has so far only been demonstrated within a few prototypes. Besides, oil resources are becoming increasingly less available and also less attractive for environmental reasons. Consequently the hydrocarbon processing industry has developed technologies for converting low value feedstocks to hydrogen and syngas. Common approaches include reforming, partial oxidation and auto-thermal reforming.

In order to support the widespread use of fuel cells in many areas of transportation it is necessary to develop methods of processing liquid fuels to generate hydrogen, which can be utilized in fuel cells. The present invention

utilises hydrocarbon fuels, which contain both H and C in various ratios. Examples of hydrocarbon fuels include saturated hydrocarbons (e.g. methane, ethane, propane and butane), natural gas, biogas, gasoline, gasified coal or biomass, diesel, synthetic fuels, marine fuel and jet fuels. The term "hydrocarbon fuels" also includes alcohols commonly used as fuels, e.g. methanol, ethanol and butanol.

However, the fuels with the highest energy density, such as diesel or jet fuels, contain large amounts of heavy hydrocarbons as well as more than 0.5 wt% sulfur. Catalysts for high temperature reforming of these fuels are very susceptible to carbon formation from these higher hydrocarbon species, as well as from sulfur poisoning, and thus it is very difficult to develop a fuel processing unit that can operate directly on these fuels.

It is possible to convert the heavy hydrocarbons to methane through the process of pre-reforming, whereby the fuel is contacted with steam over a catalyst at temperatures around 300 to 600°C below the typical reforming temperatures to produce an equilibrium mix of methane, hydrogen, carbon oxides and water. In practice, traces of heavier hydrocarbons (C₂₊) will also be present. This lower pre-reforming temperature can reduce, but not completely eliminate the formation of carbon during the pre-reforming process. The output stream from the pre-reforming process can then be reformed at high temperatures without concerns for carbon formation with methane as essentially the only hydrocarbon present over well-known catalysts.

Sulfur is generally removed from fuel for pre-reforming by either hydrodesulfurization (HDS) with a downstream adsorption of H_2S ; this process requires H_2 which can be supplied by an external hydrogen source or via an anode recycle:

5

diesel + H_2 → HDS → ZnO → diesel + H_2O → pre-reforming

or liquid desulfurization:

10

diesel → liq. desulph. → diesel + H_2O → pre-reforming.

These known technologies are encumbered with a number of drawbacks. In the hydrodesulfurization process the fuel is contacted with hydrogen at pressures around 5-20 atmospheres and temperatures of 350-500°C, whereby most of the sulfur is converted to components (e.g. H_2S , COS) which are suitable for easy downstream removal. The sulfur components will still need to be removed prior to sending the sulfur-free fuel to a reformer. Sulfur can also be removed by an adsorption bed, usually based on zinc oxide. While this bed will capture not only H_2S , but also sulfur-containing hydrocarbons, it will have a limited adsorption capacity for sulfur uptake.

25 Neither of these options is ideal for a fuel processing unit. The zinc oxide bed requires frequent maintenance, and a hydrodesulfurization system will be rather energy intensive and also difficult to design for a small fuel processing unit. It would therefore be desirable to provide a novel method for pre-reforming of sulfur containing fuels to produce syngas for solid oxide fuel cell applications,

30

in which method neither a hydrodesulfurization nor a zinc oxide bed will be required.

5 Solid oxide fuel cells (SOFCs) provide promising improvements with regard to efficiency and emissions. The choice of fuel processing method, e.g. catalytic partial oxidation, autothermal reforming or steam reforming, strongly affects the efficiency and power density of the system. Pre-reforming of hydrocarbon fuels is one of the most attractive solutions for SOFCs and MCFCs, both making it possible to obtain high electrical system effectivities and also allowing more compact SOFC systems than hitherto possible.

10 It has now been found that it is possible to remove the sulfur from the fuel (particularly the heavy hydrocarbons thereof) via the reforming process instead of having to make use of an upstream HDS-ZnO process or an upstream liquid desulfurizer.

20 The invention therefore concerns a method for the processing of hydrocarbon fuels by pre-reforming to produce an anode feed gas for use in connection with a fuel cell system, said method comprising the following steps:

25 (a) treatment of the hydrocarbon fuel with steam, with steam and hydrogen or with syngas, or with combinations thereof in a pre-reformer to convert the fuel to syngas and to remove at least a portion of the sulfur species from the fuel,

30

(b) feeding the syngas obtained in step (a) to the anode inlet of a fuel cell system.

5 Within the meaning of the present invention, syngas is a gas containing mainly H_2 , CO , CO_2 , CH_4 , H_2O and in some instances also N_2 . Syngas is typically obtained at the outlet of a pre-reformer, or anode outlet of a fuel cell such as an SOFC or MCFC.

10 The method may optionally include the step of: (c) recirculating part of the anode off-gas from the fuel cell system to the inlet of the pre-reformer. Another optional step is: treating the hydrocarbon fuel in one or more reforming beds after step (a) and before step (b), above.

15 The invention further concerns a solid oxide fuel cell system supplied with fuel obtained by the above method. One embodiment of this system is shown schematically in figure 1.

20 In the present invention, the terms "pre-reforming" and "pre-reformer" are considered synonymous with "reforming" and "reformer".

25 Examples of "sulfur species" removed from the hydrocarbon fuel include H_2S , and COS but also organic sulfur compounds, including thiols, thiophenes, organic sulfides and disulfides.

30 The pre-reforming step (step a.) of the method may be carried out adiabatically, or with heating or with cooling. Suitable operating temperatures of the pre-reforming step

lie between 250 and 950°C, preferably between 350 and 650°C. Suitably, in the method according to the invention, pre-reforming is carried out adiabatically. Accordingly, an adiabatic pre-reformer is preferably used. The terms "adiabatic" and "adiabatically", when used in connection with the system or method of the invention, are used to describe a thermally insulated state, without input or removal of heat to/from the pre-reformer.

Figure 1 schematically illustrates a particular solid oxide fuel cell assembly according to the invention.

In general terms, the solid oxide fuel cell assembly according to the invention comprises:

a pre-reformer (R1), wherein the hydrocarbon fuel is treated with steam, with steam and hydrogen or with syngas, or with combinations thereof, to remove at least a portion of the sulfur species from the fuel,

a solid oxide fuel cell system (FC1) in the form of a single solid oxide fuel cell or at least one solid oxide fuel cell stack, to the anode inlet of which the syngas from the pre-reformer (R1) is fed, and

optionally, a recycle loop from the anode outlet of the solid oxide fuel cell system (FC1), through which a part of the anode off-gas from the solid oxide fuel cell system is recirculated to the inlet of the pre-reformer (R1).

30

Preferably, syngas is used in the pre-reformer (R1) to remove at least a portion of the sulfur species from the fuel.

5 Optionally, one or more reforming beds (not shown) are arranged between the pre-reformer (R1) and the solid oxide fuel cell system (FC1). One or more heat exchangers may be present before and/or after one or more of said one or more reforming beds, so that the gas entering the reforming beds
10 may be heated or cooled. In addition, the reforming beds may be independently operated adiabatically, heated or cooled.

The pre-reformer (R1) and any reforming beds present between the pre-reformer (R1) and the solid oxide fuel cell
15 system (FC1) may be designed so that the pre-reforming catalyst therein may be easily replaced when necessary. This is especially important for the reforming beds which are present upstream (e.g. the first bed, or the first and second
20 beds) in the system, as these become most rapidly contaminated with sulfur). Suitably, the pre-reformer (R1) and any reforming beds may be connected to the system via quick-release connections, allowing simple and rapid removal and replacement of these elements.

25 The pre-reformer (R1) and any reforming beds may be present in the same vessel, or in separate vessels. If present in a single vessel, this vessel may be designed so that reforming beds can be replaced independently.

30 In the particular embodiment of the solid oxide fuel cell assembly shown, the syngas from the pre-reformer (R1) is

fed to the anode inlet of the solid oxide fuel cell assembly via a heat exchanger (E2).

5 In addition, the part of the anode off-gas from the solid oxide fuel cell system (FC1) may be recirculated to the inlet of the pre-reformer (R1) via a heat exchanger and a recycle blower (B1). This heat exchanger may be the same heat exchanger (E2) as between the pre-reformer (R1) and the solid oxide fuel cell, or may be different.

10

Suitably, the pre-reformer (R1) is an adiabatic pre-reformer.

15 As mentioned previously, the pre-reformer (R1) is also used as a sulfur trap in the concept of the present invention. Thus, the anode off-gas (2006) from the fuel cell (FC1) can be used directly in a recycle loop (2008) for mixing with the incoming fuel (2000). To avoid carbon formation, a sufficient amount of H₂ can be provided. A simulation model
20 for a diesel based solid oxide fuel cell system has shown that an anode recycle ratio around 50% will be sufficient in order to achieve appropriate H₂/C ratios. As mentioned above, this concept will neither require a HDS nor a ZnO bed. Thus there are benefits in terms of improved simplicity
25 of the fuel processing concept, system efficiency and costs.

Using the method and the system according to the invention, hydrocarbon fuels can be directly used within the fuel cell
30 system without any deep desulfurization processes. Hydrocarbon fuels like diesel, gasoline or jet fuel can be desulfurized with a manageable effort down to a sulfur lev-

el similar to that of ultra-low sulfur diesel (ULSD), i.e. approximately 10 ppm by weight. The ability to convert hydrocarbon fuels with a sulfur content of 10 ppm by weight within an pre-reformer is thus a prerequisite to avoid any deep desulfurization technologies, thereby keeping the system simple and efficient.

The following example serves to illustrate the invention further.

10

Example

Various long term tests have been carried out with one of the applicant's own catalysts. The pre-reformer was operated on ULSD obtained from the gas station. This example describes a test with ultra low sulfur diesel (ULSD) and a simulated anode recycle gas, operating close to the conditions of a commercial fuel cell system.

Higher hydrocarbons (HHCs) in the reformat gas were analysed using gas chromatography with a flame ionization detector (GC-FID; Agilent GC 7890) up to C₄. The average HHC contents in the reformat gas for Test no. 2 (Set-1) and Test no. 5 (Set-2) were 2 and 1 ppmv, respectively.

25

For sulfur analysis, gas samples from the reformer outlet were taken in sampling bags to eliminate sulfur adsorption in the long distance sampling line between the test rig and the GC. The samples were analysed for hydrogen sulphide, carbonyl sulphide and light mercaptans using a GC equipped with FPD. No sulfur was detected in the reformat gas in any tests.

30

The ULSD slip was worked out by measuring the oil phase in the condensate. Approximate steam outlet in the reformat gas was calculated and the ULSD slip was estimated by comparing the volumes of aqueous and organic phases. The ULSD slip was detected after more than 400 hours of operation.

A reformat composition with around 32% hydrogen on dry basis was demonstrated without any traces of higher hydrocarbons for more than 400 hours.

Dry composition:

CO₂: 48%
H₂: 32%
CO: 2%
CH₄: 18%

These results reflect the high potential of pre-reforming for SOFC systems (both mobile and stationary) utilizing hydrocarbon fuels.

Claims:

1. A method for the processing of hydrocarbon fuels by pre-reforming to produce an anode feed gas for use in connection with a fuel cell system, said method comprising the following steps:
- 5
- (a) treatment of the hydrocarbon fuel with steam, with steam and hydrogen or with syngas, or with combinations thereof in a pre-reformer to convert the fuel to syngas and to remove at least a portion of the sulfur species from the fuel,
- 10
- (b) feeding the syngas obtained in step (a) to the anode inlet of a fuel cell system.
- 15
2. The method according to any one of claims 1-2, including the step of (c) recirculating part of the anode off-gas from the fuel cell system to the pre-reformer, preferably to the inlet thereof.
- 20
3. The method according to any one of claims 1-3, including the step of recirculating at least a part of the reformer effluent to the pre-reformer, preferably to the inlet thereof.
- 25
4. The method according to any one of the preceding claims, further including the step of: treating the hydrocarbon fuel in one or more reforming beds after step (a) and before step (b).
- 30

5. The method according to any one of the preceding claims, wherein the pre-reformer and any reforming beds present between the pre-reformer and the solid oxide fuel cell system are designed so that the pre-reforming catalyst therein may be easily replaced.
6. The method according to any one of the preceding claims, wherein the hydrocarbon fuel is a fuel that has not been treated by a deep desulfurization process.
7. The method according to any one of the preceding claims, wherein the fuel cell system is a medium to high temperature fuel cell system operating at approximate temperatures above 500°C and below 950°C.
8. The method according to any one of the preceding claims, wherein the fuel cell system is a solid oxide fuel cell system comprising a single solid oxide fuel cell or at least one solid oxide fuel cell stack.
9. The method according to any one of the preceding claims, wherein the pre-reformer is an adiabatic pre-reformer.
10. A solid oxide fuel cell assembly suitable for carrying out the method of any one of the preceding claims, said assembly comprising:
- (a) a pre-reformer (R1), wherein the hydrocarbon fuel is treated with steam, with steam and hy-

drogen or with syngas, or with combinations thereof, to remove at least a portion of the sulfur species from the fuel,

- 5 (b) a solid oxide fuel cell system (FC1) in the form of a single solid oxide fuel cell or at least one solid oxide fuel cell stack, to the anode inlet of which the syngas from the pre-reformer (R1) is fed.

10

11. The solid oxide fuel cell assembly according to claim 10, further comprising a recycle loop from the anode outlet of the solid oxide fuel cell system (FC1), through which a part of the anode off-gas from the solid oxide fuel cell system is recirculated to the pre-reformer (R1), preferably the inlet thereof.

15

12. The solid oxide fuel cell assembly according to any one of claims 10-11, wherein the hydrocarbon fuel is treated with syngas in the pre-reformer (R1) to remove at least a portion of the sulfur species from the fuel.

20

13. The solid oxide fuel cell assembly according to any one of claims 10-12, wherein the syngas from the pre-reformer (R1) is fed to the anode inlet of the solid oxide fuel cell assembly via a heat exchanger (E2).

25

30

14. The solid oxide fuel cell assembly according to any one of claims 10-13, wherein said part of the anode

off-gas from the solid oxide fuel cell system is recirculated to the pre-reformer (R1), preferably the inlet thereof, via a heat exchanger (E2) and a recycle blower (B1).

5

15. The solid oxide fuel cell system according to any one of claims 10-14, wherein the pre-reformer (R1) is an adiabatic pre-reformer.

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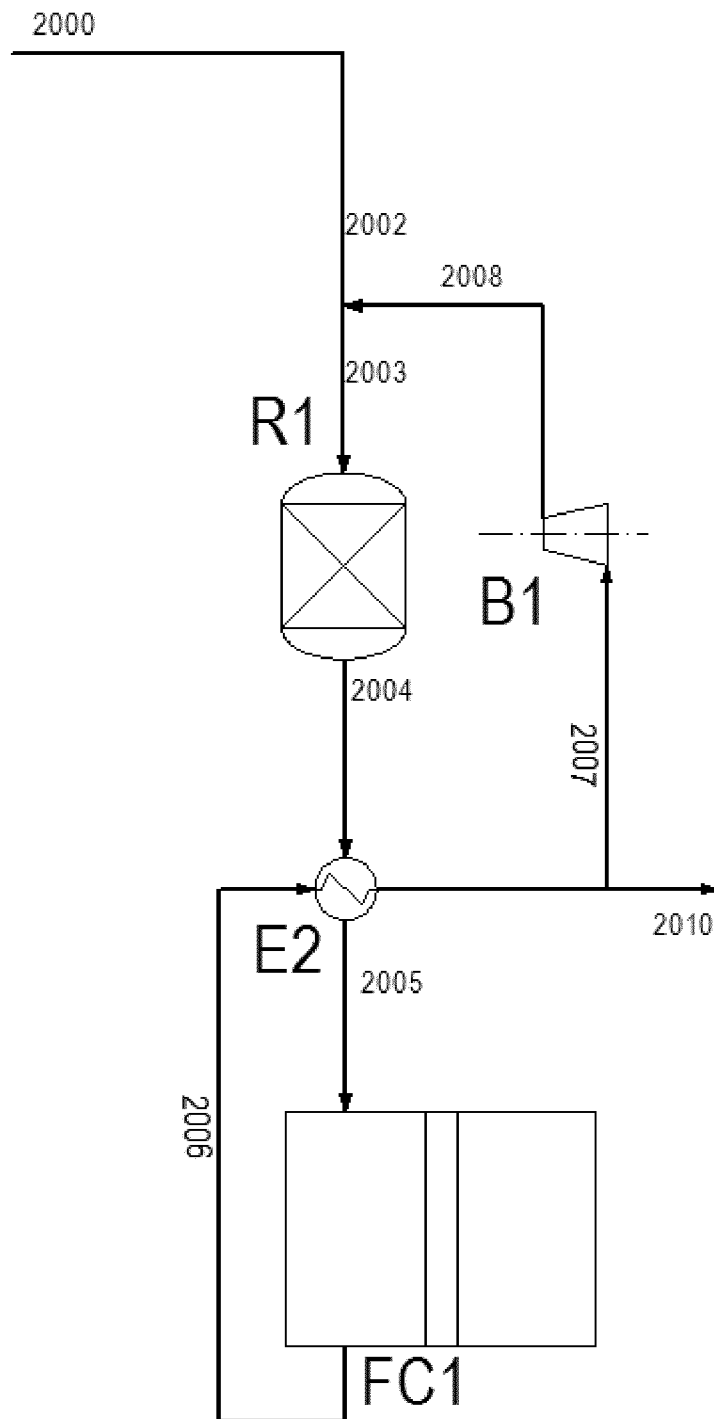
16. The solid oxide fuel cell system according to any one of claims 10-15, further comprising one or more reforming beds arranged between the pre-reformer (R1) and the solid oxide fuel cell system (FC1).

15

17. The solid oxide fuel cell system according to any one of claims 10-16, wherein pre-reformer (R1) and any reforming beds present between the pre-reformer (R1) and the solid oxide fuel cell system (FC1) are designed so that the pre-reforming catalyst therein may be easily replaced.

20

Fig. 1



INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER
 INV. H01M8/06 H01M8/04
 ADD. H01M8/12 C01B3/00

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)
 H01M C01B

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)
 EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Further documents are listed in the continuation of Box C.

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
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Date of the actual completion of the international search 30 August 2013	Date of mailing of the international search report 09/09/2013
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Brune, Markus

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/059356

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Information on patent family members

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