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PARTICULADO PARA RESANADO.

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71. SOLICITANTE

BP EXPLORATION OPERATING COMPANY LIMITED

DOMICILIO

MIDDLESEX, GRAN BRETAÑA

74. APODERADO

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Capítulo II

2

**SOLICITANTE
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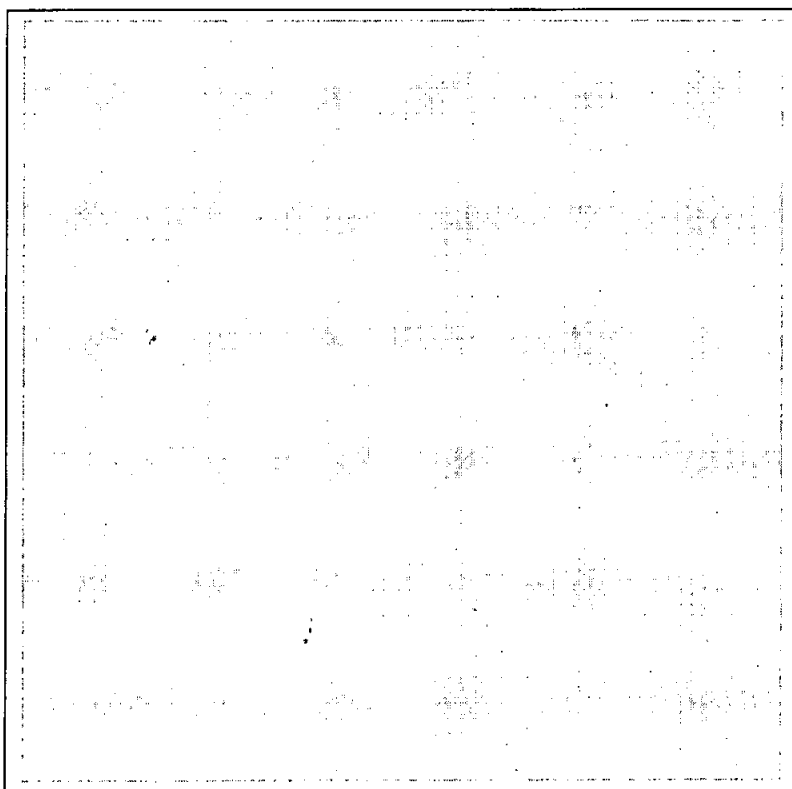
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Fecha 23 de julio de 2008

- ☒ Comprobante de pago de la tasa de presentación de la solicitud
- ☐ Documento que acredita la existencia y representación legal cuando el solicitante sea persona jurídica
- ☐ Poderes si fuese el caso
- ☐ Documento de cesión del inventor al solicitante o a su causante
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- ☒ Copia de la solicitud internacional (Resumen, descripción, reivindicaciones, dibujos)
- ☒ Traducción de la solicitud internacional al castellano. (Resumen, descripción, reivindicaciones, dibujos)
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- ☐ De ser el caso, modificaciones efectuadas a la solicitud internacional
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RESUMEN DE LA INVENCION

Un fluido para perforación que comprende un fluido base y un agente para resanado particulado
5 constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina (2,4,5-triamino-1,3,5-triazina), carbonato de litio, fosfato de litio (Li_3PO_4), y sulfito de magnesio.

FLUIDO PARA PERFORACION QUE COMPRENDE UN FLUIDO BASE Y UN

AGENTE PARTICULADO PARA RESANADO

CAMPO DE LA INVENCION

5

La presente invención se refiere a fluidos para perforación utilizados en la construcción, reparación o tratamiento de una perforación y a la remoción de la torta de filtro depositada por los fluidos para perforación sobre
10 o en formaciones de roca penetradas por la perforación.

ANTECEDENTES DE LA INVENCION

Los fluidos para perforación incluyen fluidos
15 para barrenado, fluidos para circulación perdida, fluidos para terminación (tales como píldoras para perforación y fluidos para ensanchamiento), y fluidos para mantenimiento (tales como fluidos para reoperación, fluidos para molienda, fluidos para fracturación, solventes, fluidos
20 acuosos que contienen agentes para disolución no ácidos, y fluidos que contienen agentes de desviación particulados).

Los fluidos para barrenado se utilizan cuando se barrena una perforación a través de una formación de roca porosa y permeable, por ejemplo, una formación de roca que
25 contiene hidrocarburos. Es bastante deseable que el fluido

para barrenado reduzca al mínimo el daño a la permeabilidad de la formación de roca. Por ejemplo, el daño a la permeabilidad de una formación de roca que contiene hidrocarburos puede dar como resultado pérdidas de producción o una capacidad reducida de la formación para aceptar fluidos inyectados (por ejemplo, agua o fluidos para tratamiento).

Los fluidos para terminación se utilizan durante operaciones que se presentan en la denominada fase de terminación de una perforación (después de barrenar la perforación y antes de comenzar la producción de fluidos desde una formación de roca hacia la perforación o la inyección de fluidos desde la perforación hacia una formación de roca). De nuevo, es bastante deseable que los fluidos para terminación reduzcan al mínimo el daño a la permeabilidad de las formaciones rocosas.

Los fluidos para mantenimiento se pueden utilizar de manera intermitente durante el tiempo de vida productiva de una perforación, por ejemplo, cuando se efectúan operaciones de reoperación, estimulación o corrección en una formación de roca penetrada por la perforación. Por ejemplo, en casos en los que el fluido para mantenimiento sea un fluido para fracturación, es bastante deseable que se reduzca al mínimo el escurrimiento de fluido desde las fracturas que son inducidas en las paredes de la

perforación.

Los fluidos para barrenado, terminación o mantenimiento por lo general comprenden un agente para resanado sólido particulado de un tamaño de partícula que es lo suficientemente grande para resanar las gargantas de poro de una formación de roca porosa y permeable y un aditivo para control de filtración (con frecuencia denominado un "aditivo para control de pérdida de fluido"). Los fluidos para barrenado, terminación o mantenimiento depositan una capa de partículas conocida como una "torta de filtro" sobre las paredes de las perforaciones. En casos en los que la perforación penetra una formación de roca porosa y permeable, esta torta de filtro de baja permeabilidad evita que se pierdan cantidades grandes de fluidos ("filtrado") desde el fluido para barrenado, terminación o mantenimiento hacia el interior de la formación de roca y también evita que los sólidos entren a los poros de la formación. El fluido que se pierde desde un fluido para barrenado, terminación o mantenimiento hacia el interior de una formación de roca porosa y permeable se denomina "filtrado". La torta de filtro está constituida por el agente para resanado particulado y el agente para control de pérdida de fluido y también puede incluir otros sólidos que estén presentes en el fluido para perforación y que se puedan depositar sobre las paredes de la

perforación. Después del barrenado, terminación o mantenimiento de la perforación, es conveniente que se retire tanto como sea posible de la torta de filtro antes de comenzar la producción de fluidos a partir de una
5 formación de roca porosa y permeable al interior de la perforación o antes de inyectar fluido en una formación de roca permeable y porosa desde la perforación. Sin embargo, con frecuencia es difícil tener acceso a, y eliminar, cantidades sustanciales de la torta de filtro.

10 En caso que se esté perdiendo un volumen grande de fluido para perforación a través de conductos de alta conductividad en las paredes de una perforación hacia el interior de una formación de roca porosa y permeable, se bombea al interior de la perforación un fluido para
15 circulación perdida que comprende un material para circulación perdida (LCM por sus siglas en inglés) suspendido en un fluido base. Los conductos de alta conductividad típicamente son fisuras, fracturas o oquedades en las paredes de la perforación (en donde una
20 oquedad es una cavidad, espacio vacío o poro grande en una formación rocosa). Los fluidos para circulación perdida con frecuencia comprenden sólidos particulados más gruesos (LCM) que los agentes para resanado particulados de los fluidos para barrenado, mantenimiento o terminación con el
25 fin de resanar y sellar los conductos de alta conductividad

dentro de los cuales se está perdiendo el fluido para perforación. Por lo tanto, se deposita un tapón de permeabilidad relativamente baja que comprende al LCM particulado y opcionalmente otros sólidos, a partir del fluido para circulación perdida en los conductos de conductividad elevada. Estos tapones particulados pueden ser difíciles de remover de los conductos de alta conductividad cuando se desea comenzar la producción de fluidos desde una formación de roca al interior de una perforación de producción o la inyección de fluidos al interior de una formación de roca desde una perforación de inyección.

Convencionalmente, las tortas de filtro se remueven de las paredes de la perforación poniendo en contacto las tortas de filtro con uno o más fluidos para limpieza. Un agente para resanado común para resanar las gargantas de los poros de una formación porosa y para obstruir cualesquiera conductos de alta conductividad (por ejemplo fisuras) en la misma es carbonato de calcio en polvo. La torta de filtro se puede eliminar utilizando un fluido para limpieza que comprende enzimas y oxidantes para degradar el aditivo para control de pérdida de fluido antes de poner en contacto la torta de filtro con una solución para limpieza fuertemente ácida durante un periodo suficiente de tiempo para disolver el agente para resanado

de carbonato de calcio particulado. Sin embargo, a pesar de los pasos anti-corrosión actuales, la solución fuertemente ácidas con frecuencia corroe las superficies metálicas y el equipo para terminación tal como tamices para control de arena lo que ocasiona la falla temprana de dicho equipo. La solución para limpieza ácida también puede ser incompatible con la formación productora y puede ocasionar daño a la formación. Además, se presenta la disolución ineficiente de la torta de filtro cuando una solución para limpieza ácida reacciona rápidamente con una porción de la torta de filtro, abriendo la comunicación de fluido entre el fluido para limpieza y la perforación y la formación permeable, después de lo cual el fluido para limpieza entra a la formación sin hacer contacto con el remanente de la torta de filtro. Otro problema surge cuando una malla para arena extensible se coloca en una perforación de agujero abierto en el intervalo de la perforación adyacente a una formación que contiene hidrocarburos. Después de la colocación de la malla de arena, ésta se expande para que se ajuste al diámetro de la perforación con lo cual se provee soporte excelente a la perforación y la exclusión de producción de arena. Por desgracia, esto da como resultado que la torta de filtro quede atrapada entre la malla de arena expandida y la formación de modo tal que es muy difícil tener acceso a la torta de filtro con una solución para limpieza. Bajo

tales circunstancias, podría ser conveniente si la torta de filtro fuera soluble en fluidos menos corrosivos y menos dañinos, por ejemplo fluidos de perforación normalmente presentes. De esta manera finalmente se puede llegar a la
5 torta de filtro no tratada o atrapada y es disuelta en los fluidos.

En casos en los que la torta de filtro se deposita sobre y/o en las paredes de una perforación para producción de hidrocarburos, la formación que contiene
10 hidrocarburos por lo general produce una proporción significativa de agua. En casos en que la torta de filtro se deposita sobre y/o en las paredes de un pozo para inyección de agua o un pozo productor de agua, de nuevo la torta de filtro queda expuesta a volúmenes grandes de agua
15 a través de un periodo prolongado de tiempo. En casos en los que la torta de filtro se deposita sobre y/o en las paredes de una perforación geotérmica, la torta de filtro queda expuesta al agua caliente y vapor. Por consiguiente, se han utilizado o propuesto agentes para resanado sólidos
20 particulados formados a partir de una sal soluble en agua (por ejemplo, halogenuros de metal alcalino) o una sal poco soluble en agua (por ejemplo borato de magnesio y sales de magnesio de ácidos carboxílicos) en los fluidos para barrenado o mantenimiento. Por lo tanto, las tortas de
25 filtro que contienen el agente para resanado soluble en

agua o poco soluble en agua han sido eliminadas poniendo en contacto la torta de filtro con una solución salina acuosa la cual está sub-saturada con respecto a la sal soluble en agua o poco soluble en agua. Estos agentes para resanado
5 solubles en agua o poco solubles en agua se pueden utilizar en fluidos para tratamiento ya sea de base acuosa o de base oleosa con la condición que el fluido de base acuosa esté saturado con respecto a la sal soluble en agua o poco soluble en agua. Sin embargo, sigue existiendo la necesidad
10 respecto a fluidos para perforación adicionales en los cuales el agente para resanado esté constituido por un material poco soluble en agua.

SUMARIO DE LA INVENCION

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Por consiguiente, la presente invención se refiere a un fluido para perforación que comprende un fluido base y un agente para resanado particulado comprendido por un material poco soluble en agua que se
20 selecciona a partir del grupo que consiste de melamina (2,4,5-triamino-1,3,5-triazina), carbonato de litio, fosfato de litio (Li_3PO_4), y sulfito de magnesio, de preferencia, melamina y carbonato de litio.

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DESCRIPCION DETALLADA DE LA INVENCION

El término "fluido para perforación" tal como se utiliza en la presente invención abarca fluidos para
5 barrenado, fluidos para circulación perdida, fluidos para terminación tales como píldoras para perforación y fluidos para ensanchamiento, y fluidos para mantenimiento tales como fluidos para neutralización, fluidos para reoperación, fluidos para molienda, fluidos para fracturación,
10 solventes, agentes para disolución acuosos no ácidos, y fluidos que contienen agentes de desviación particulados.

El fluido para perforación de la presente invención es apropiado para ser utilizado en una variedad de perforaciones incluyendo perforaciones productoras de
15 petróleo y/o gas, pozos de inyección de agua o gas, pozos productores de agua y pozos geotérmicos.

Los materiales poco solubles en agua que han sido seleccionados para uso como el agente para resanado
20 particulado tienen una solubilidad en agua a una temperatura de 25°C menor de 7% en peso, de preferencia menor de 2% en peso. Además, estos materiales tienen una solubilidad en agua a una temperatura de 80°C menor de 7% en peso, de preferencia menor de 3.5% en peso.

De manera opcional, se incluye un aditivo para
25 control de pérdida de fluido en el fluido para perforación

de la presente invención.

La presente invención también provee un método para formar una torta de filtro removible sobre las paredes de una perforación que penetra una formación de roca porosa y permeable que comprende los pasos de:

(a) colocar un fluido para perforación en la perforación en el cual el fluido para perforación comprende un fluido base y un agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio (Li_3PO_4), y sulfito de magnesio, de preferencia, melamina y carbonato de litio; y

(b) permitir que el agente particulado para resanado se deposite desde el fluido para perforación sobre y/o en las paredes de la perforación para formar de esta manera la torta de filtro removible, con lo cual se reduce la pérdida de fluido hacia la formación a través de la torta de filtro.

Idóneamente, el agente para resanado particulado puede resanar las gargantas de los poros de las formaciones rocosas penetradas por la perforación y/o puede entrar en cualesquiera grietas, fisuras, fracturas, o huecos en la pared de la perforación.

Opcionalmente, se incluye un aditivo para control de pérdida de fluido en el fluido para perforación.

Con "removable" se quiere decir que la torta de filtro se puede remover sin bombear un fluido de limpieza especializado al interior de la perforación. En otras palabras, la torta de filtro puede ser auto-removable.

5 Se permite que la torta de filtro se acumule en las paredes de la perforación debido a que la presión de fluido para perforación en la perforación se mantiene por encima de la presión de poro de la formación porosa y permeable que es penetrada por la perforación. De
10 preferencia, la presión diferencial entre la presión del fluido para perforación en la perforación y la presión de poro es de por lo menos 14.06 kg/cm^2 .

En casos en los que la perforación es una perforación para producción de hidrocarburos, el agente
15 para resanado se puede eliminar poniendo el pozo en producción debido a que se produce agua conjuntamente con el hidrocarburo la cual disuelve el material poco soluble en agua. En casos en los que la perforación es un pozo para producción de agua o un pozo geotérmico, el agente para
20 resanado se puede eliminar poniendo el pozo en producción debido a que el material poco soluble en agua se disuelve en el agua que se produce. En casos en los que la perforación es un pozo para inyección de agua, la torta de filtro se puede eliminar iniciando la inyección de agua
25 debido a que el agua inyectada disuelve al material poco

soluble en agua. Por lo tanto, tanto el agua producida como el agua inyectada están sub-saturadas con respecto al material poco soluble en agua. El agente para resanado se puede solubilizar completamente con el tiempo en el agua o
5 de manera alternativa se solubiliza hasta el grado en el que las partículas tienen un tamaño lo suficientemente reducido para permitir su remoción de la formación con el agua producida o inyectada. El tiempo requerido para solubilizar las partículas depende de un número de factores
10 incluyendo, la temperatura en la perforación, el tamaño y la forma de las partículas del agente para resanado, y la cantidad de agua a la que la torta de filtro esté expuesta. Se espera que la torta de filtro subsista durante menos de 200 horas cuando un pozo para producción se pone en
15 producción o cuando se inyecta agua en un pozo de inyección.

De manera opcional, si se requiere la disolución rápida, se puede bombear un fluido para limpieza al interior de la perforación. El fluido para limpieza puede
20 ser un fluido acuoso que esté sub-saturado con respecto al agente para resanado. De preferencia, el fluido para limpieza es una solución acuosa de un ácido, de preferencia, una solución acuosa de un ácido débil o de un precursor de un ácido débil. De preferencia, el ácido débil
25 se selecciona a partir del grupo que consiste de ácido

fórmico, ácido cítrico, ácido acético, ácido láctico, ácido glicólico, ácido succínico, y secuestrantes ácidos tales como aquellos basados en ácido etilendiamintetra-acético (EDTA) parcialmente neutralizado. De preferencia, el precursor del ácido débil se selecciona a partir de materiales que pueden hidrolizarse para producir ácidos débiles tales como los homopoliésteres de ácido poliglicólico o poliláctico y ortoésteres tales como los ésteres de ortoformiato. De preferencia, el ácido débil o precursor de ácido está presente en el fluido de limpieza en una cantidad entre 1% y 20% peso. Una ventaja de utilizar una solución acuosa de un ácido débil o una solución acuosa de un precursor de un ácido débil es que el fluido para limpieza es menos corrosivo para las superficies y equipo metálicos que los ácidos fuertes que se utilizan para disolver agentes para resanado inorgánicos convencionales tales como carbonato de calcio. Una ventaja adicional de los materiales poco solubles en agua utilizados en la presente invención es que incluso la reacción parcial con un ácido forma productos que tienen una mayor solubilidad en agua. Por lo tanto, el carbonato de litio se convierte a bicarbonato de litio, el fosfato de litio (Li_3PO_4) se convierte en hidrogenofosfatos de litio, y el sulfito de magnesio se convierte en bisulfito de magnesio después de reaccionar parcialmente con un ácido.

Todos estos productos son de una solubilidad mucho más alta que sus precursores poco solubles en agua. Además, los grupos amina que están presentes en la melamina se protonan bajo condiciones ligeramente ácidas, incrementando en gran
5 manera la solubilidad en agua del agente para resanado.

En casos en los que el agente para resanado particulado poco soluble en agua esté constituido por sulfito de magnesio, el fluido para limpieza puede comprender una solución acuosa de un agente oxidante que
10 pueda convertir el sulfito de magnesio en sulfato de magnesio soluble en agua. Por lo tanto, el sulfato de magnesio tiene una solubilidad mucho más alta en agua que el sulfito de magnesio. Los agentes oxidantes apropiados incluyen peróxido de hidrógeno, sales de persulfato, y
15 perácidos tales como ácido peracético. De preferencia, el agente oxidante está presente en el fluido para limpieza en una cantidad de 1 a 20% peso. De manera opcional, el fluido para limpieza también comprende un ácido débil o un precursor de un ácido débil.

20 En casos en los que el agente para resanado particulado poco soluble en agua esté constituido por melamina, se contempla que la torta de filtro removible se puede retirar de las paredes de una perforación colocando un fluido para lavado acuoso agujero abajo y dejando que el
25 fluido para lavado acuoso remoje el intervalo de la

perforación en donde se desea eliminar la torta de filtro. El tiempo de remojo debe ser suficiente para que el fluido de lavado acuoso se caliente hasta una temperatura de por lo menos 60°C, de preferencia por lo menos 75°C, por ejemplo, por lo menos 90°C. Debido a que la solubilidad de la melamina se incrementa en forma relativamente rápida al incrementarse la temperatura, el agente para resanado particulado se puede disolver completamente en el fluido para lavado acuoso o éste se solubiliza hasta el grado en el que las partículas son de un tamaño lo suficientemente reducido para permitir su remoción desde la formación. El fluido para lavado acuoso se calienta hasta la temperatura deseada debido a la transferencia de calor geotérmico proveniente de la formación. Típicamente, podría tomar por lo menos varias horas, por ejemplo, aproximadamente 1 día para que el fluido de lavado acuoso se caliente hasta la temperatura deseada. En general, el operador podrá determinar que ha transcurrido un periodo de tiempo suficiente cuando se incremente la velocidad de pérdida de fluido desde la perforación hacia la formación.

La velocidad de disolución en agua de los agentes para resanado también se ve incrementada por la presencia de dióxido de carbono. Por consiguiente, se puede esperar que las presiones parciales altas del dióxido de carbono que con frecuencia están presentes en los fluidos

producidos a partir de formaciones que contienen hidrocarburo aceleren la disolución del agente para resanado.

De manera opcional se coloca un fluido para
5 limpieza agujero abajo y se deja que remoje a través del intervalo de la perforación en donde se desea eliminar la torta de filtro durante un periodo de tiempo suficiente ya sea para disolver completamente el agente para resanado o para solubilizar el agente para resanado hasta el grado en
10 el que las partículas sean de un tamaño lo suficientemente reducido para permitir su remoción de la formación. El fluido para limpieza puede contener enzimas o agentes oxidantes para degradar los polímeros para control de pérdida de fluido y viscosificantes que se acumulan en la
15 torta de filtro, y puede contener ácidos o precursores de ácido para acelerar la disolución de los sólidos para resanado. De preferencia, se deja que la solución de limpieza opcional remoje durante aproximadamente 2 a 24 horas. En general, el operador podrá determinar que ha
20 transcurrido un periodo de tiempo suficiente cuando se incremente la velocidad de pérdida de fluido desde la perforación hacia la formación. Después de esto, se puede bombear un fluido para lavado (por ejemplo, un fluido acuoso tal como agua o agua de mar o una salmuera diluida)
25 a una velocidad elevada al interior de la perforación con

el fin de crear condiciones de limpieza turbulentas con lo cual se elimina la torta de filtro remanente de las paredes de la perforación. De manera alternativa, la torta de filtro remanente se puede eliminar produciendo agua a partir de la formación o inyectando agua dentro de la formación.

De preferencia, el fluido para perforación se selecciona a partir de (a) un fluido para barrenado; (b) un fluido utilizado para controlar la circulación pérdida (denominado "fluido para circulación pérdida"); (c) un fluido para terminación utilizado durante operaciones de terminación; y (d) un fluido para mantenimiento de pozo utilizado cuando se efectúan operaciones de reoperación, estimulación o corrección.

Por lo tanto, en una modalidad preferida de la presente invención se provee un método para barrenar una perforación a través de una formación de roca porosa y permeable utilizando un fluido para barrenado que comprende un fluido base, un aditivo para control de pérdida de fluido, y un agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio (Li_3PO_4), y sulfito de magnesio en el cual la presión del fluido para barrenado se mantiene por encima de la presión en la formación de roca porosa y

permeable de modo tal que se deposite una torta de filtro sobre y/o en las paredes de la perforación y reduzca la pérdida de fluido desde el fluido para perforación hacia la formación de roca.

5 Con la frase que se deposita "en las paredes de la perforación" se quiere decir que la torta de filtro se puede depositar en cualesquiera grietas, fracturas, fisuras u oquedades que estén presentes en las paredes de la perforación.

10 De manera apropiada, la perforación que se barrena utilizando esta modalidad preferida de la presente invención es una perforación para producción de hidrocarburos (un pozo de petróleo o gas), una perforación para inyección (por ejemplo, un pozo para inyección de agua
15 o gas), una perforación productora de agua o una perforación geotérmica.

 En otra modalidad preferida de la presente invención se provee un método para controlar la pérdida de fluido desde una perforación hacia una formación de roca
20 porosa y permeable a través de un conducto de alta conductividad que se extiende desde la perforación al interior de la formación de roca que comprende los pasos de:

 (a) colocar un fluido para circulación pérdida en
25 la perforación en el cual el fluido para circulación

pérdida comprende una suspensión de material particulado para circulación pérdida (LCM) en un fluido base en el cual el LCM está suspendido en el fluido base en una cantidad de por lo menos 14.175 kg/m³, de preferencia por lo menos 5 28.35 kg/m³, más preferido por lo menos 56.7 kg/m³, y más preferido aún por lo menos 85.05 kg/m³, y está constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio (Li₃PO₄), y sulfito de magnesio; y

10 (b) permitir que el LCM se acumule en, o a la entrada, del conducto de alta conductividad con lo cual se forma un tapón removible de baja permeabilidad que resana el conducto con lo cual se reduce la pérdida de fluido hacia la formación a través del conducto.

15 Con "removible" se quiere decir que el tapón se puede retirar sin la ayuda de un fluido para limpieza diseñado específicamente.

La suspensión se bombea dentro del intervalo de la perforación en donde está presente un conducto de alta 20 conductividad (por ejemplo una fisura) en la pared de la perforación y a través de la cual el fluido se pierde hacia la formación de roca porosa y permeable, por ejemplo, una formación de roca que contiene hidrocarburo. La filtración de la suspensión da como resultado la deposición del LCM 25 particulado en el conducto de alta conductividad de modo

tal que el conducto se llena con un paquete sólido de partículas de LCM. De manera opcional, puede estar presente un agente para control de pérdida de fluido en la suspensión con lo cual se ayuda a sellar el conducto de alta conductividad. De preferencia el sellado del conducto se hace más completo cuando un fluido para perforación subsiguiente tal como un fluido para barrenado, en particular, un fluido para barrenado con baja pérdida de fluido, forma una torta de filtro impermeable después del tapón de LCM particulado.

Incluso en otra modalidad preferida de la presente invención se provee un método para controlar la pérdida de fluido desde un fluido para terminación hacia una formación de roca permeable y porosa penetrada por una perforación:

(a) colocando un fluido para terminación en la perforación en el cual el fluido para terminación comprende un fluido base, un aditivo para control de pérdida de fluido, y un agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio, y sulfito de magnesio; y

(b) manteniendo la presión del fluido para terminación en la perforación por encima de la presión de poro de la formación de roca de modo tal que se deposite

una torta de filtro sobre o en las paredes de la perforación.

De manera apropiada, el fluido para terminación (y también el fluido para barrenado referido anteriormente) contiene de manera adicional un agente o agentes para viscosidad tales como goma xantano, hidroxietilcelulosa, goma welan (por ejemplo, BiozanTM; de Kelco) o goma "diutan" (por ejemplo, Geovis XTTM; de Kelco). El fluido para terminación que se coloca en la perforación puede llenar la perforación completa. De manera alternativa, el fluido para terminación se puede utilizar como una píldora de volumen suficiente para llenar el intervalo de la perforación que se va a "terminar" llenando el remanente de la perforación con un segundo fluido que tiene una densidad apropiada para propósitos de control de pozo. Por lo tanto, la densidad del segundo fluido se elige de manera tal que el fluido no fluya desde una formación de roca hacia el interior de la perforación. Se contempla que el segundo fluido puede ser una salmuera que esté sustancialmente libre de sólidos suspendidos.

También se contempla que la perforación puede ser una perforación entubada que se perfora en un intervalo de la perforación que yace a través de una formación de roca porosa y permeable, por ejemplo, una formación de roca que contiene hidrocarburos. Por consiguiente, la torta de

filtro se deposita desde del fluido de terminación en los túneles de la perforación formados en la perforación entubada con lo cual se reduce la pérdida de fluido desde el fluido de terminación hacia la formación.

5 Incluso en una modalidad adicional de la presente invención, se provee un método para controlar la pérdida de fluido desde un fluido para reoperación hacia un intervalo de una perforación que yace a través de una formación de roca permeable y porosa en la cual el método comprende los
10 pasos de:

 (a) bombear un volumen suficiente de un primer fluido de reoperación para llenar el intervalo de la perforación que yace a través de la formación de roca porosa y permeable en el cual el primer fluido para
15 reoperación comprende un fluido base, un aditivo para control de pérdida de fluido, y un agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio, y sulfito
20 de magnesio de modo tal que se deposite una torta de filtro removible a partir del primer fluido para reoperación en dicho intervalo de la perforación sobre las paredes de la perforación y dentro de cualesquiera grietas, fracturas o fisuras en la misma;

25 (b) bombear un segundo fluido de reoperación

dentro de la perforación en el cual el segundo fluido de reoperación es de una densidad suficiente para por lo menos contrarrestar la presión de la formación de roca porosa y permeable;

5 y en la cual la torta de filtro depositada en el paso (a) reduce la pérdida de fluido desde los fluidos de reoperación hacia la formación de roca porosa y permeable.

El segundo fluido de reoperación puede ser de la misma composición que el primer fluido de reoperación o
10 puede ser de una composición diferente, por ejemplo una salmuera o aceite libre de sólidos. En el caso de fluidos de reoperación acuosos, se prefiere que el segundo fluido de reoperación esté sustancialmente saturado con respecto al material poco soluble en agua que constituye el agente
15 particulado para resanado del primer fluido de reoperación. El primer fluido de reoperación se utiliza para sellar la formación para evitar pérdidas de fluido desde el segundo fluido de reoperación mientras que el segundo fluido de reoperación se utiliza para efectuar funciones tales como
20 mantenimiento de control de pozos (cabeza hidrostática), restos en circulación tales como el equipo agujero abajo "molido" fuera de la perforación (por ejemplo empacadoras o tamices "molidos"), para proveer un fluido de baja viscosidad para permitir el fácil desplazamiento de las
25 herramientas dentro y fuera de la perforación y para que

actúe como un fluido de "re-terminación".

En casos en los que la perforación sea una perforación entubada que se perfora en el intervalo de la perforación a través de una formación de roca porosa y permeable, la torta de filtro se deposita a partir del primer fluido de reoperación en los túneles de la perforación en el entubamiento de la perforación con lo cual se reduce la pérdida de fluido desde el segundo fluido de reoperación hacia la formación.

Los pozos que requieren de una "reoperación" con frecuencia son pozos de producción de hidrocarburo agotados en los cuales la formación de roca que contiene hidrocarburos tiene una baja presión de poro. Por consiguiente, la cabeza hidrostática del segundo fluido de reoperación en el intervalo de la perforación a través de la formación de roca que contiene hidrocarburos puede estar sobre-excedida con respecto a la presión de poro en la formación de roca que contiene hidrocarburos agotada incluso cuando el segundo fluido de reoperación es un fluido de baja densidad simple tal como agua (por ejemplo, agua de mar), o un aceite. Por consiguiente, la capacidad de controlar la pérdida de fluido utilizando el método antes descrito es más importante a presiones diferenciales altas (en las cuales la presión del segundo fluido de reoperación en la perforación es significativamente más

alta que la presión de poro de la formación rocosa).

Los fluidos para fracturación por lo general comprenden un agente de soporte (por ejemplo, partículas de arena o glóbulos de cerámica) suspendido en un fluido base
5 acuoso que normalmente se hace viscoso utilizando un polímero o un agente tensoactivo viscoelástico de modo tal que el agente de soporte que se utiliza para mantener abiertas las fracturas es transportado en forma eficiente al interior de las fracturas que son creadas cuando el
10 fluido para fracturación se bombea a presión elevada al interior de una formación de roca porosa y permeable. Sin embargo, si el fluido para fracturación se escurre muy rápidamente al interior de la formación la presión elevada se disipa y la fractura deja de crecer. El control de
15 escurrimiento normalmente se logra dispersando partículas molidas tales como harina de sílice en el fluido para fracturación para bloquear/resanar los poros expuestos en la fractura que están aceptando el "escurrimiento". Por desgracia, los materiales tales como la sílice pueden
20 ocasionar por lo menos alguna obstrucción permanente de los poros de la formación.

Por consiguiente, incluso en otra modalidad preferida de la presente invención se provee un método para fracturar una formación de roca porosa y permeable que
25 comprende:

inyectar un fluido para fracturación dentro de un intervalo de una perforación a través de la formación de roca que se va a fracturar en el cual el fluido para fracturación comprende un fluido base, agente de soporte, un agente para viscosidad, y un agente particulado para control de escurrimiento constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio, y sulfito de magnesio; y

mantener la presión del fluido para fracturación en el intervalo de la perforación a través de la formación de roca por encima de la presión de fractura de la formación con lo cual el agente de soporte entra y mantiene abiertas las fracturas que se forman en la pared de la perforación y el agente particulado para control de escurrimiento sella las gargantas de poro expuestas en las paredes de la fractura.

Una ventaja de esta modalidad preferida de la presente invención es que la presión del fluido para fracturación en la fractura en crecimiento se mantiene tanto como sea posible por encima de la presión de fracturación de la formación de roca reduciendo el escurrimiento de fluido hacia la formación y por lo tanto reduciendo la disipación de presión hacia la formación. En casos en los que las fracturas se forman en una formación

de roca que contiene hidrocarburos penetrada por un pozo de producción, el material particulado para resanado se disuelve en el agua que se produce conjuntamente después de retornar la perforación a producción con lo cual se mejora el flujo de fluido desde la formación que contiene hidrocarburos. En casos en que las fracturas se formen en una formación de roca porosa y permeable penetrada por un pozo para inyección de agua, el material particulado para resanado se disuelve en el agua que se inyecta dentro de la formación de roca, con lo cual se mejora el flujo de fluido desde el pozo de inyección al interior de la formación.

En otra modalidad de la presente invención, se provee un método para desviar fluidos de tratamiento no ácidos lejos de las formaciones de roca de alta permeabilidad o de los conductos de alta conductividad y hacia formaciones de roca de baja permeabilidad y/o parcialmente obstruidas o conductos de conductividad más baja utilizando un fluido de tratamiento que comprende un fluido no ácido y un agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio (Li_3PO_4), y sulfito de magnesio. Por ejemplo, en casos en los que el fluido no ácido es un solvente aromático, el fluido de tratamiento se puede utilizar para disolver depósitos de cera y/o

asfalteno que obstruyen los canales de flujo en los pozos petroleros (y por lo tanto reducen la producción de petróleo). El método comprende bombear una suspensión que comprende al agente particulado para resanado suspendido en un solvente aromático al interior de una perforación de producción de hidrocarburo de modo tal que se forme una torta de filtro en o sobre una formación de roca de permeabilidad alta o el agente particulado para resanado entre y selle los conductos de alta conductividad (o canales de flujo) en las paredes de la perforación con lo cual se limita la pérdida de solvente aromático desde la perforación. Por consiguiente, el solvente aromático se desvía hacia los conductos de baja conductividad (o canales de flujo) que pudieran estar dañados por depósitos de asfalteno y/o cera, con lo cual se mejora la disolución de los depósitos por parte del solvente aromático.

A continuación se describen las características preferidas del fluido para perforación de la presente invención.

El fluido base del fluido para perforación puede ser agua, un aceite (por ejemplo, un aceite mineral), un solvente (por ejemplo, un solvente aromático), o una mezcla de los mismos (por ejemplo, una emulsión aceite en agua). En términos generales, el fluido base está presente en el fluido para perforación en una cantidad en el intervalo de

aproximadamente 30 a 99% en peso del fluido, de preferencia, aproximadamente 70 a 97% en peso.

En casos en los que el fluido base es agua, se prefiere que el fluido base sea una solución acuosa de una sal soluble en agua que incremente la densidad. La sal soluble en agua que incrementa la densidad se puede seleccionar a partir del grupo que consiste de halogenuros de metal alcalino (por ejemplo, cloruro de sodio, bromuro de sodio, cloruro de potasio y bromuro de potasio), carboxilatos de metal alcalino (por ejemplo, formiato de sodio, formiato de potasio, formiato de cesio, acetato de sodio, acetato de potasio o acetato de cesio), carbonato de sodio, carbonato de potasio, halogenuros de metal alcalinotérreo (por ejemplo, cloruro de calcio y bromuro de calcio), y sales de halogenuro de zinc.

De manera alternativa, se puede proveer control de densidad al fluido para perforación basado en agua utilizando agentes para ponderación insolubles. Los agentes para ponderación apropiados incluyen partículas minerales suspendidas tales como baritas molidas, óxidos de hierro, (por ejemplo, hematita), ilmenita, calcita, magnesita (MgCO_3), dolomita, olivina, siderita, hausmannita o partículas de metal suspendidas.

En casos en los que el fluido es un aceite, se prefiere que el aceite se seleccione a partir del grupo que

consiste de aceites minerales, aceites sintéticos, ésteres, queroseno y diesel.

El fluido base también puede ser una emulsión aceite en agua que comprenda gotas minúsculas de una fase acuosa dispersas en una fase oleosa continua. De manera apropiada, la fase acuosa de la emulsión comprende una solución acuosa de una sal soluble en agua que incrementa la densidad con lo cual se incrementa la densidad del fluido para perforación. Las sales solubles en agua incrementadoras de densidad apropiadas se indicaron anteriormente. De preferencia, la concentración de sal en las gotas minúsculas dispersas de la fase acuosa se ajusta para proveer una actividad de agua similar a la de la formación subterránea que entra en contacto con el fluido para perforación. La fase oleosa continua puede ser cualquier aceite en el cual se pueda emulsificar una solución acuosa de sales. Los aceites apropiados se indicaron anteriormente. Una ventaja de una emulsión aceite en agua es que ésta incrementa tanto el control de filtración (debido a que las gotas minúsculas de la emulsión bloquean el flujo de fluido a través de la torta de filtro) como las propiedades viscosas del fluido. El término fluido para perforación de base acuosa tal como se utiliza en la presente invención abarca fluidos para perforación en los cuales el fluido base es una emulsión

aceite en agua.

También se puede proveer control de densidad al fluido para perforación de base oleosa utilizando agentes para ponderación. Los agentes para ponderación apropiados
5 son como los indicados anteriormente para fluidos para perforación de base acuosa.

En casos en los que el fluido base es agua, el agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir
10 del grupo que consiste de melamina, carbonato de litio, fosfato de litio, y sulfito de magnesio (de aquí en adelante "agente particulado para resanado poco soluble en agua") se dosifica en el fluido para perforación a una concentración que sea significativamente más alta que su
15 solubilidad en agua a la temperatura encontrada agujero abajo con lo cual se asegura que por lo menos una porción de los sólidos para resanado permanezcan suspendidos en el fluido para perforación. De manera alternativa, el agente
20 particulado para resanado poco soluble en agua se puede proteger con un revestimiento hidrofóbico que pueda disolverse en un hidrocarburo líquido producido, por ejemplo, un petróleo producido o un condensado de gas producido. Sin embargo, dichos agentes para resanado
25 particulados revestidos no se deben emplear cuando se barrenan o se terminan pozos de inyección de agua o pozos

de gas que estén libres de condensado de gas.

En términos generales, el agente para resanado
particulado poco soluble en agua está presente en el fluido
para perforación en una cantidad suficiente para crear una
5 torta de filtro eficiente que provea el nivel deseado de
control de pérdida de fluido. Típicamente, el agente para
resanado particulado poco soluble en agua está presente en
el fluido para perforación en una cantidad en el intervalo
de 1 a 70% en peso, de preferencia 2 a 50% en peso, más
10 preferido, 3 a 30% en peso, en particular 3 a 15% en peso.
Se prefieren dosis altas para fluidos para circulación
pérdida, por ejemplo 10 a 60% en peso.

La distribución de tamaño de partícula deseada
del material para resanado particulado poco soluble en agua
15 se determina mediante el tamaño de cualesquiera fracturas y
similares dentro de las cuales el fluido para perforación
se está perdiendo o mediante el tamaño de la garganta del
poro de la formación que se va a barrenar o a tratar.
Típicamente, para uso como un material de circulación
20 pérdida, el agente para resanado particulado poco soluble
en agua tiene una distribución de tamaño de partícula en el
intervalo de aproximadamente 50 micras hasta
aproximadamente 10 mm, de preferencia 50 micras hasta 2 mm
aproximadamente. Para uso como un sólido para resanado en
25 un fluido para barrenado, mantenimiento o terminación, el

agente particulado para resanado poco soluble en agua tiene una distribución de tamaño de partícula en el intervalo de aproximadamente 0.1 micras hasta 600 micras, de preferencia 0.1 hasta 200 micras, y más preferido 0.1 a 100 micras. De preferencia, el material para resanado particulado poco soluble en agua tiene una distribución de tamaño polidisperso amplia. Los materiales (carbonato de litio, fosfato de litio, sulfito de magnesio y melamina) se pueden conseguir como materiales cristalinos del tamaño deseado o como cristales o gránulos que se pueden moler hasta el tamaño deseado. El material para resanado particulado poco soluble en agua puede estar en forma de partículas sustancialmente esféricas o puede ser de una forma irregular.

Se puede utilizar más de un agente para resanado particulado poco soluble en agua en el fluido para perforación.

Los fluidos para perforación pueden comprender de manera adicional uno o más de los siguientes materiales: un agente para resanado particulado o para ponderación convencional, por ejemplo barita; materiales solubles en ácido tales como carbonato de calcio; materiales solubles en agua tales como halogenuros de metal alcalino; y, otros materiales poco solubles en agua tales como borato de magnesio y sales de magnesio de ácidos carboxílicos. Dichos

agentes para resanado particulados convencionales se pueden emplear ya sea en un fluido para perforación de base oleosa o en un fluido para perforación de base acuosa. En casos en los que el agente para resanado particulado convencional
5 esté comprendido por un material soluble en agua o poco soluble en agua, éste se utiliza en un fluido de base acuosa en cantidades superiores a la concentración de saturación del material soluble en agua o poco soluble en agua en agua a las condiciones encontradas agujero abajo
10 para proveer partículas suspendidas del agente para resanado particulado convencional. Los fluidos para perforación de base acuosa pueden comprender de manera adicional agentes para resanado sólidos particulados constituidos por materiales solubles en aceite tales como
15 resinas. Las resinas apropiadas incluyen resinas termoplásticas obtenidas a partir de la polimerización de hidrocarburos, que tengan una estructura amorfa o cristalina que permita que éstas puedan ser trituradas y molidas a temperatura ambiente y al mismo tiempo conserven
20 su resistencia de modo tal que permanezcan no deformables cuando se someten a la presión en los poros y fisuras de una formación de roca. Estas resinas tienen un punto de fusión por encima de la temperatura encontrada agujero abajo y son insolubles en fluidos para tratamiento de base
25 acuosa pero son solubles en los petróleos crudos y

condensados de gas producidos. Los ejemplos de resinas preferidas incluyen resinas de cumarona-indeno, y resinas aromáticas alquiladas.

De preferencia, el agente para resanado
5 particulado poco soluble en agua utilizado en la presente invención comprende una porción significativa de los sólidos suspendidos contenidos en el fluido para perforación y por lo tanto en la torta de filtro. De manera idónea, el agente particulado para resanado poco soluble en
10 agua constituye por lo menos 15% en volumen, de preferencia por lo menos 30% en volumen, más preferido por lo menos 60% en volumen de los sólidos suspendidos del fluido para perforación (siendo el resto agentes particulados para resanado convencionales, agentes para ponderación, sólidos
15 barrenados, y arcillas). Sin desear estar limitado a ninguna teoría, se cree que la disolución del agente particulado para resanado poco soluble en agua crea huecos en la torta de filtro con lo cual dicha torta se vuelve permeable. En casos en los que la torta de filtro se forma
20 en un pozo de producción, la torta de filtro es fácilmente degradada cuando el pozo se pone en producción debido a que los fluidos producidos fluyen más libremente a través de la torta de filtro permeable. Por consiguiente, otros sólidos que se depositan en la torta de filtro son arrastrados en
25 el fluido producido de modo tal que la torta de filtro se

elimina de la pared de la perforación.

En casos en los que el fluido para perforación es un fluido de base acuosa, el fluido para perforación puede comprender aditivos adicionales para mejorar el desempeño
5 del fluido para perforación con respecto a una o más propiedades. Los ejemplos de aditivos que se pueden agregar a fluidos para perforación de base acuosa incluyen viscosificantes, agentes para ponderación, sales solubles en agua incrementadoras de densidad, agentes para control
10 de pérdida de fluido (también conocidos como aditivos para control de filtración), agentes para control de pH, inhibidores de hidratación de arcilla o esquistos (tales como polialquilenglicoles), bactericidas, agentes tensoactivos, lubricantes sólidos y líquidos, inhibidores
15 de hidrato de gas, inhibidores de corrosión, desespumantes, inhibidores de incrustación, líquidos hidrofóbicos emulsificados tales como aceites, depuradores de gas ácido (tales como los depuradores de sulfuro de hidrógeno), adelgazadores (tales como lignosulfonatos),
20 desemulsificantes y agentes tensoactivos diseñados para ayudar en la limpieza de fluido invadido proveniente de formaciones productoras.

Se pueden agregar polímeros solubles en agua a un fluido para perforación de base acuosa para impartir al
25 fluido propiedades viscosas, de dispersión de sólidos y de

control de filtración. Se puede utilizar una amplia gama de polímeros solubles en agua para un fluido para perforación de base acuosa incluyendo derivados de celulosa tales como carboximetilcelulosa, hidroxietilcelulosa, carboximetil-
5 hidroxietilcelulosa, sulfoetilcelulosa; derivados de almidón (los cuales pueden estar entrelazados) incluyendo carboximetil-almidón, hidroxietil-almidón, hidroxipropil-almidón; gomas de origen bacteriano incluyendo xantano, welan, diutan, succinoglucano, escleroglucano, dextrano,
10 pululano; gomas obtenidas a partir de plantas tales como goma guar y gomas de algarrobo y sus derivados; polímeros y copolímeros sintéticos obtenidos a partir de cualesquiera monómeros apropiados incluyendo ácido acrílico o ácido metacrílico y sus ésteres hidroxílicos (por ejemplo ácido
15 hidroxietilmetacrílico), anhídrido o ácido maléico, monómeros sulfonados tales como ácido estirensulfónico y AMPS, acrilamida y acrilamidas sustituidas, N-vinilformamida y N-vinilacetamida, N-vinilpirrolidona, acetato de vinilo, N-vinilpiridina y otros monómeros
20 vinílicos catiónicos (por ejemplo, cloruro de dialil-dimetilamonio, DADMAC); y cualesquiera otros polímeros solubles en agua o que se puedan expandir en agua conocidos por los expertos en la técnica. En términos generales, los polímeros solubles en agua viscosificantes están presentes
25 en el fluido para perforación de la presente invención en

una cantidad suficiente para mantener los sólidos de resanado y de ponderación en suspensión y para proveer una limpieza eficiente del pozo de restos tales como los cortes de barrenado. El polímero viscosificante puede estar
5 presente en el fluido para perforación en una cantidad en el intervalo de 0.567 a 14.175 kg de viscosificante por metro cúbico de fluido para perforación, de preferencia 1.4175 a 8.505 kg por metro cúbico de fluido para perforación.

10 También se puede proveer control reológico (por ejemplo, propiedades de gelificación) al fluido para perforación de base acuosa agregando arcillas y/u otras partículas finas inorgánicas. Los ejemplos incluyen bentonita, montmorilonita, hectorita, atapulguita,
15 sepiolita, LaponiteTM (de Laporte) e hidróxidos de metal mixtos.

Se puede utilizar un aditivo para control de pérdida de fluido para llenar los espacios vacíos entre el agente particulado para resanado. Además de los polímeros
20 solubles en agua indicados anteriormente, los ejemplos de aditivos para control de pérdida de fluido para fluidos de perforación basados en agua incluyen lignita tratada con cáusticos, lignitas modificadas, lignosulfonatos entrelazados y similares. Por lo tanto, estos aditivos para
25 control de pérdida de fluido son macromoléculas disueltas

que se pueden adsorber sobre los sólidos para resanado o son macromoléculas que están en dispersión coloidal en el fluido de base acuosa, por ejemplo, un polímero hidratado que adopta una conformación helicoidal cuando se dispersa en el fluido con base acuosa haciendo que el polímero hidratado pueda ocluir poros de tamaño micrométrico o nanométrico en la torta de filtro.

Los agentes para control de pH apropiados para fluidos para perforación con base acuosa incluyen hidróxido de calcio, hidróxido de magnesio, óxido de magnesio, hidróxido de potasio, hidróxido de sodio, y similares.

En casos en los que el fluido para perforación es un fluido de base oleosa, el fluido para perforación puede comprender aditivos adicionales para mejorar el desempeño del fluido para perforación con respecto a una o más propiedades. Los ejemplos de aditivos que se pueden agregar a fluidos para perforación con base oleosa incluyen viscosificantes, agentes tensoactivos (para formar emulsiones aceite en agua estables y para impregnar con aceite la superficie de agentes de ponderación minerales), aditivos para control de pérdida de fluido (también conocidos como aditivos para control de filtración), lubricantes (sólidos y líquidos), y depuradores de gas ácido (por ejemplo, depuradores de sulfuro de hidrógeno).

Se puede agregar un viscosificante al fluido para

perforación de base oleosa para impartir al fluido propiedades de viscosidad, suspensión de sólidos y propiedades de limpieza de agujeros. Normalmente el viscosificante es una arcilla montmorilonita o hectorita
5 que ha sido tratada con sales de amonio cuaternario de ácido graso para hacer que la arcilla se pueda dispersar y exfoliar en el fluido para perforación de base oleosa. Se pueden utilizar polímeros y oligómeros solubles en aceite como modificadores reológicos.

10 Los agentes tensoactivos que se pueden agregar al fluido de base oleosa para formar emulsiones agua en aceite estables y para impregnar con aceite la superficie de los agentes de ponderación minerales incluyen ácidos grasos tales como ácido graso de aceite de madera (TOFA por sus
15 siglas en inglés) y productos de condensación de TOFA con polialquilenaminas tales como trietilentetramina. La amidoamina de ácido graso resultante y los productos de imidazolina se pueden utilizar como tal o estos se pueden hacer reaccionar adicionalmente con, por ejemplo, anhídrido
20 maléico para mejorar su desempeño. En casos en los que el agente tensoactivo contiene un grupo funcional ácido carboxílico, dichos grupos por lo general se convierten en las sales de calcio correspondiente mediante adición de cal.

25 Los aditivos para control de pérdida de fluido

apropiados que se pueden agregar al fluido para perforación de base oleosa incluyen asfalto, asfalto soplado, asfalto sulfonado, gilsonita, lignita modificada con amina de ácido graso, y polímeros sintéticos solubles/expandibles en
5 aceite.

A continuación la presente invención se ilustra con respecto a los siguientes ejemplos.

Pruebas de solubilidad

10 Las siguientes pruebas muestran la solubilidad en agua y en soluciones ácidas acuosas de los materiales poco solubles en agua.

EJEMPLO 1

15 Solubilidad de melamina

En la siguiente tabla 1 se provee la solubilidad de melamina en agua a través de un intervalo de temperaturas. El experto en la técnica entenderá que todo
20 lo que se requiere es una cantidad suficiente de agua para disolver la melamina particulada depositada en un pozo, en especial si se deja que el pozo se caliente hasta su temperatura natural (predominante) después del enfriamiento experimentado durante el barrenado de una perforación o
25 durante la inyección de agua fría desde la superficie. Por

consiguiente, la melamina particulada se puede limpiar (disolver) automáticamente en el agua que pudiera ser producida a partir de un pozo junto con hidrocarburos, o en el agua que se bombee al interior de un pozo de inyección para mantener la presión del yacimiento.

TABLA 1

Temperatura (°C)	Solubilidad de melamina (% en peso)
20	0.3
35	0.6
50	1.05
60	1.5
80	3.0
100	5
120	8

La melamina también es fácilmente soluble en ácidos tibios o calientes tales como ácido acético y ácido clorhídrico. Por lo tanto, una mezcla de melamina (25.2 g, 0.2 M), 250 ml de agua y 24 g de ácido acético (0.4 M) produce una solución clara cuando se calienta hasta una temperatura de 80°C. De manera similar, una mezcla de melamina (126 g, 1 M) y 1985 ml de ácido clorhídrico 1.0075 M produce una solución clara cuando se calienta hasta una temperatura de 83°C.

La solubilidad de melamina en soluciones acuosas ácidas es conveniente en casos en los que se contempla la estimulación del pozo mediante inyección de ácido, o en casos en los que se colocan cantidades grandes de melamina
5 particulada en el pozo, por ejemplo como tapones de material de circulación perdida en formaciones fracturadas.

EJEMPLO 2

Solubilidad de carbonato de litio

10

En la siguiente tabla 2 se provee la solubilidad de carbonato de litio en agua a través de un intervalo de temperaturas. Al igual que para el ejemplo 1, el experto en la técnica entenderá que todo lo que se requiere es una
15 cantidad suficiente de agua para disolver las partículas de carbonato de litio que se depositan en un pozo. Como se indicó anteriormente, ésta puede ser agua producida o agua que se bombee al interior de un pozo de inyección o un fluido acuoso colocado dentro del pozo con el propósito de
20 disolver las partículas de carbonato de litio. La reducción en solubilidad al incrementarse la temperatura es una ventaja en casos en los que se utiliza carbonato de litio particulado en pozos de temperaturas más altas (por ejemplo pozos que tienen una temperatura de fondo de agujero (BHT
25 por sus siglas en inglés) de 100°C o mayor) en el sentido

de que se evita más fácilmente la disolución prematura de las partículas sólidas.

TABLA 2

5	Temperatura (°C)	Solubilidad de carbonato de litio (gramos por 100 g de agua)
	20	1.33
	40	1.17
	60	1.01
	80	0.85
10	100	0.72

El carbonato de litio también se disuelve rápidamente en ácidos. Por ejemplo, el ácido acético reacciona con el carbonato de litio para generar acetato de litio el cual es muy soluble en soluciones acuosas mientras que el ácido clorhídrico reacciona con el carbonato de litio para generar la sal cloruro de litio altamente soluble. La capacidad para remover el carbonato de litio particulado bombeando un ácido al interior de un pozo es una ventaja en casos en los que se colocan cantidades grandes de carbonato de litio particulado en el pozo, por ejemplo como tapones de material de circulación perdida en formaciones fracturadas.

El carbonato de litio también muestra una solubilidad incrementada en agua en presencia de dióxido de

carbono, el cual frecuentemente se encuentra en los fluidos producidos a partir de pozos de petróleo o pozos de gas (debido a la formación de bicarbonato de litio, LiHCO_3). Por ejemplo, a una temperatura de 60°C y a una presión de 5 50 atmósferas de CO_2 , 100 gramos de solución saturada contienen 9.61 gramos de LiHCO_3 .

La alta solubilidad de LiHCO_3 es particularmente conveniente en pozos de gas debido a que el gas en la formación inevitablemente está casi saturada con vapor de 10 agua y por lo general contiene concentraciones altas de CO_2 . A medida que el gas fluye a través de la formación portadora de gas hacia un pozo de producción de gas, la presión se reduce ocasionando enfriamiento adiabático y condensación de agua. El agua condensada junto con la 15 concentración elevada de CO_2 disuelve por lo tanto los residuos de carbonato de litio particulado sin la necesidad de bombear fluidos para disolución desde la superficie.

EJEMPLO 3

20

Solubilidad de sulfito de magnesio

En la siguiente tabla 3 se provee la solubilidad de sulfato de magnesio en agua a través de un intervalo de temperaturas. Al igual que para los ejemplos 1 y 2, todo lo 25 que se necesita es una cantidad suficiente de agua para

disolver el sulfito de magnesio particulado depositado en un pozo. Por lo tanto, los residuos de sulfito de magnesio particulado se limpian automáticamente (disuelven) en el agua que se pudiera producir junto con los hidrocarburos, o en el agua que se bombee dentro de un pozo de inyección para mantener la presión del yacimiento. De manera alternativa se pueden bombear agua o mezclas acuosas al interior del pozo para disolver los residuos de sulfito de magnesio particulado.

10

TABLA 3

Temperatura (°C)	Solubilidad de sulfito de magnesio (% en peso)
25	0.65
42	0.94
50	0.84
85	0.62
98	0.61

15

El sulfito de magnesio también se disuelve fácilmente en soluciones acuosas de ácidos tales como ácido acético o ácido clorhídrico para producir dióxido de azufre y el acetato de magnesio o cloruro de magnesio muy solubles respectivamente. Incluso la acidulación parcial hasta bisulfito de magnesio es efectiva para disolver el sulfito de magnesio particulado ya que el bisulfito de magnesio es

20

25

muy soluble en agua. Por ejemplo, el bisulfito de magnesio se puede conseguir comercialmente como una solución acuosa al 30% en peso de Sigma Aldrich.

De manera alternativa los agentes oxidantes tales como peróxido de hidrógeno pueden ocasionar la disolución del sulfito de magnesio al convertirlo en el sulfato de magnesio soluble (62.9 g de sulfato de magnesio se disuelven en 100 gramos de agua a una temperatura de 20°C).

10

EJEMPLO 4

Solubilidad de fosfato de litio

El fosfato de litio (Li_3PO_4) tiene una solubilidad relativamente baja en agua (0.038 gramos por 100 gramos de agua a una temperatura de 20°C). Por lo tanto es menos preferido para aplicaciones en las cuales se utiliza agua (agua producida, agua de inyección, o fluido para limpieza acuoso) para disolver los residuos particulados.

20

Sin embargo la acidulación ligera, por ejemplo, con ácido acético o ácido clorhídrico incrementa la solubilidad en gran manera. Por ejemplo, LiH_2PO_4 es muy soluble en agua a 55% en peso.

25

EJEMPLO 5Formulaciones de fluido para perforación de base acuosa

Las siguientes pruebas se refieren a
5 formulaciones de fluido para perforación basadas en agua.

Las formulaciones de fluido 1-4 (véase la
siguiente tabla 4) son apropiadas para uso como fluidos
para barrenado, fluidos para terminación tal como una
píldora de perforación o un fluido para ensanchamiento, un
10 fluido de reoperación tal como un fluido de neutralización.
La formulación de fluido 4 representa un fluido típico para
perforación de la técnica antecedente que actualmente se
utiliza en la industria como, por ejemplo, un fluido para
barrenado de yacimiento. Este fluido de la técnica
15 antecedente contiene sólidos de carbonato de calcio
insoluble en agua para resanado y se incluye para
propósitos comparativos. Las propiedades de las
formulaciones de fluido 1-4 se proveen en la siguiente
tabla 5.

20

Materiales

Melamina en polvo, carbonato de litio, fosfato de
litio y cloruro de potasio fueron tal como son
suministrados por Aldrich UK (proveedor de productos
25 químicos de laboratorio). DuoVis™ (viscosificante de goma

xantano), DualFlo™ (reductor de pérdida de fluido a base de derivado de almidón) y Starcarb™ (carbonato de calcio en polvo) fueron suministrados por M-I Swaco llc.

Las formulaciones de fluido se analizan de conformidad con ISO 10416: 2002 (API RP 131 7ª edición). Los resultados de la pérdida de fluido también se presentan en la siguiente tabla 5 más adelante.

TABLA 4

Formulaciones de fluido

Componente (g)	Fluido 1	Fluido 2	Fluido 3	Fluido 4
Agua desionizada	330	330	330	330
DuoVis	1.0	1.0	1.0	1.0
DualFlo	4.0	4.0	4.0	4.0
Cloruro de potasio	10	10	10	10
Melamina	16	-	-	-
Carbonato de litio	-	20	-	-
Fosfato de litio	-	-	16	-
Starcarb	-	-	-	25
Sosa cáustica	A pH 10.0	Ninguna (pH 11.0)	A pH 10.0	A pH 10.0

La variación en las dosis gravimétricas de los polvos es para proveer aproximadamente la misma carga en volumen que la del fluido comparativo Starcarb (Fluido 4).

TABLA 5

Propiedades de las formulaciones de fluido

Propiedades	Fluido 1	Fluido 2	Fluido 3	Fluido 4
Viscosidad plástica cP	12	12	12	13
Punto de relajación (Pa)	7.6608	8.1396	8.1396	8.1396
Geles (10 seg/10min)	2.873/3.352	3.352/3.83	3.352/3.83	3.83/4.31
Pérdida de fluido API (mls) determinada de conformidad con ISO 10416	6.8	6.5	9.8	6.4

5

Después de la prueba de pérdida de fluido API, se
10 decanta el exceso de fluido para perforación de la celda
utilizada en la prueba y se reemplaza con agua desionizada.
La celda se vuelve a sellar, se presuriza hasta 7.03 kg/cm²
con nitrógeno, y se mide la velocidad de permeación a
través de la torta de filtro durante 30 minutos.

15

Se efectúa una prueba similar repitiendo la
prueba de pérdida de fluido API para proveer nuevas tortas
de filtro provenientes de los fluidos 2 y 4, seguido por
permeación de agua desionizada que se presuriza con dióxido
de carbono hasta 7.03 kg/cm².

20

Se efectúa una prueba similar repitiendo la
prueba de pérdida de fluido API para proveer una torta de
filtro con los fluidos 1 a 4, seguido por permeación de
ácido acético al 5% durante 30 minutos, o hasta que todo el
líquido en la celda pase a través de la torta de filtro.

25

Los resultados de estas pruebas adicionales se

proveen en la siguiente tabla 6, la cual muestra las velocidades de permeación promedio (mls/min).

TABLA 6

5 **Resultados de la prueba de pérdida de fluido**

Fluido permeante	Fluido 1 (melamina)	Fluido 2 (carbonato de litio)	Fluido 3 (fosfato de litio)	Fluido 4 (carbonato de calcio)
Agua desionizada	7.4	0.52	0.32	0.18
Agua desionizada + presión superior de 7.03 kg/cm ² de CO ₂	-	1.8	-	0.37
Acido acético al 5%	10.6	24.0	9.0	1.56

10

La velocidad de flujo de agua desionizada a través de las tortas de filtro que contienen fosfato de litio y carbonato de litio está claramente mejorada en
 15 comparación con la torta de filtro de referencia que contiene carbonato de calcio (fluido 4). Las velocidades siguen siendo bastante lentas debido a que los polímeros Duovis y DualFlo concentrados en la torta de filtro reducen la velocidad de flujo y por lo tanto la disolución de las
 20 partículas poco solubles en agua durante la breve duración (30 minutos) de la prueba.

La torta de filtro que contiene melamina rápidamente desarrolla una permeabilidad mucho más alta hacia el agua desionizada.

25 La presencia de dióxido de carbono incrementa la

velocidad de flujo de agua a través de la torta de filtro que contiene carbonato de litio en más de tres veces.

Los sólidos poco solubles en agua de la presente invención reaccionan con ácido acético al 5% mucho más rápido que el carbonato de calcio particulado (la norma de la industria).

EJEMPLO 6

Material para circulación perdida y fluido de control de circulación perdida de base acuosa

10

Se retira un tamiz base de una celda para pérdida de fluido API y se coloca un lecho de aproximadamente 2.54 cm de arena de malla 20-30 en la celda. Este lecho de arena representa una formación de roca de permeabilidad extremadamente alta. Después se vierte agua a través del lecho para mojar la arena.

15

Se mezcla un fluido para barrenado simple de conformidad con la siguiente formulación:

20

Agua desionizada	330 g
Duovis TM	1.5 g
DualFlo TM	3.5 g
Barita	63 g

25

Una porción de este fluido para barrenado se vierte suavemente en la parte superior del lecho de arena. Al presurizar la celda hasta una presión de 3.515 kg/cm^2 , el fluido para barrenado completo fluye inmediatamente a través del lecho de arena en menos de 3 segundos. Esto representa un problema de circulación perdida tal como el que se podría encontrar en el campo.

Se obtiene una suspensión mezclando 100 gramos de melamina (de Aldrich) en 290 g de agua, y se vierten 100 ml de la suspensión dentro de la celda. Al presurizar a 3.515 kg/cm^2 la fase acuosa de la suspensión inmediatamente se filtra a través del lecho de arena. Al abrir la celda se observa una capa de torta de filtro blanca de partículas de melamina encima del lecho de arena. Se vierte una porción del fluido para barrenado "simple" dentro de la celda la cual se vuelve a presurizar a 3.515 kg/cm^2 . Una corriente mucho más lenta del fluido para barrenado pasa través del lecho de arena, pero todo (aproximadamente 100 ml) se sigue perdiendo de la celda a través de un periodo de aproximadamente 30 segundos. Al abrir la celda se observa que el fluido para barrenado ha fluido través de una discontinuidad pequeña en el lecho de las partículas de melamina.

Las partículas de melamina se agregan después al fluido para barrenado remanente a una dosis de

aproximadamente 35.44 kg/m^3 . Al colocar este fluido dentro de la celda y volver a presurizar a 3.515 kg/cm^2 , el fluido para barrenado comienza a fluir a través del empaque de arena pero se desacelera hasta una detención virtual en
 5 aproximadamente 5-10 segundos. La presión se incrementa hasta 7.03 kg/cm^2 . La velocidad del efluente proveniente de la celda se estabiliza después a una velocidad de filtración normal, lenta.

Este experimento ilustra el uso de partículas
 10 sólidas poco solubles en agua como un material para circulación perdida, ya sea en un fluido especialmente diseñado que se bombea hasta su lugar en una perforación para controlar pérdidas de fluido, o como un aditivo para un fluido de perforación tal como un fluido para barrenado.
 15 Se puede utilizar la adición del material poco soluble en agua a los fluidos para barrenado para detener las pérdidas de fluido, pero ésta también se puede utilizar de manera preventiva para evitar la ocurrencia de dichas pérdidas.

El tamaño de partícula de la melamina obtenida a
 20 partir de Aldrich se mide mediante tamizado en seco utilizando un agitador de malla vibratoria. Los resultados en por ciento en peso son los siguientes:

	> 500 micras	0.22%
25	<500 > 300 micras	1.60%

<300 > 150 micras	74.0%
<150 micras	24.2%

Dichas partículas dimensionadas son bastante
5 adecuadas para resanar los poros en formaciones de arena
con permeabilidad extrema, y también para que se acumulen
en las fracturas con una anchura menor de aproximadamente 1
mm mediante filtración rápida de una suspensión con alto
contenido de sólidos de las partículas que está fluyendo al
10 interior de la fractura.

EJEMPLO 7

Fluido para barrenado de base oleosa que contiene
partículas de melamina, y tratamiento de la torta de filtro
15 de la misma para establecer el flujo pasante de agua de mar

Se prepara un fluido para barrenado de base
oleosa, basado en el producto FazePro™ de M-I Swaco LLC
(véase la siguiente tabla 7). La emulsión invertida de este
20 fluido para barrenado de base oleosa está diseñada para que
se desestabilice después de la aplicación de un ácido con
lo cual se permite una limpieza mejorada en comparación con
los fluidos para barrenado de base oleosa convencionales.
La adición de polvo de melamina provee material para
25 resanar y para la torta de filtro para sellar la superficie

de arena de formaciones permeables. Después de barrenar, la torta de filtro se puede tratar con una solución ácida para romper la emulsión dentro de la torta de filtro con el fin de incrementar la permeabilidad de la torta. El ácido también comienza a disolver algo de la melamina particulada. En el caso de un pozo de inyección de agua de mar, el ácido puede ser seguido por inyección de agua de mar la cual continúa disolviendo la melamina remanente hasta que se remuevan los residuos completamente.

10 Este ejemplo muestra que se puede formular un fluido para barrenado de base oleosa con propiedades apropiadas para propósitos de barrenado con sólidos para resanado con melamina. Posteriormente, la torta de filtro se trata con una solución ácida seguido por el flujo de
15 agua de mar inyectada, de los cuales ambos fluidos son activos para remover el sello que fue provisto por la torta de filtro.

La formulación para barrenado de base oleosa se mezcla utilizando una mezcladora Silverson L4RT equipada
20 con una cabeza de esfuerzo cortante alto. Los tiempos de mezclado por componente se muestran en la siguiente tabla 7. La velocidad de la mezcladora es de aproximadamente 6000 rpm. La temperatura se monitorea minuciosamente y se mantiene a 65.56°C o menos mediante el uso de un baño de
25 agua para enfriamiento.

TABLA 7

Producto	Concentración (kg/m³)	Orden	Tiempo
DF-1 BASE OIL™ (a)	381.3	1 =	
FAZEMUL™ (b)	34.02	1 =	5 minutos
FAZEWET™ (b)	17.01	1 =	
TRUVIS™ (b)	2.835	4	2 minutos
Cal	11.34	5	2 minutos
ECOTROL RD™ (b)	1.42	6	16 minutos
Salmuera de CaBr₂ a 0.162 kg/l	467.49	7	20 minutos
Polvo de melamina	85.05	8	15 minutos

(a) de TotalFinaElf UK Limited

(b) de Trademark M-I Swaco llc

Después de mezclar, el fluido para barrenado de base oleosa se lamina en caliente a una temperatura de 65.56 °C durante 16 horas para simular el calentamiento agujero bajo en el campo. Después se miden las propiedades viscosas y la pérdida de fluido a temperatura elevada/presión elevada (HTHP FL) y se proveen en la siguiente tabla 8.

TABLA 8

Propiedades reológicas a 48.8°C	
Viscosidad plástica (cP)	15
Punto de relajación (Pa)	6.703

TABLA 8 (cont.)

Propiedades reológicas a 48.8°C	
Gel a los 10 segundos (Pa)	3.352
Gel a los 10 minutos (Pa)	3.352
Pérdida de fluido HTHP (mls) a 93.33°C y 35.15 kg/cm ²	3.1

Los resultados presentados en la tabla 8 muestran
 10 que se obtiene rendimiento reológico y de filtración
 satisfactorio.

Después de la prueba de pérdida de fluido HTHP,
 se drena el exceso de fluido para barrenado de la celda y
 se remplaza con una solución de ácido acético glacial al 5%
 15 en queroseno. La celda se cierra y calienta a una
 temperatura de 45°C. La solución de ácido se presuriza
 después hasta 7.03 kg/cm² para que la solución permee a
 través de la torta de filtro, se mide el peso del flujo
 pasante contra el tiempo, como se registra más adelante en
 20 la Tabla 9.

TABLA 9**Permeación de la solución de ácido a través de la torta de
filtro**

Tiempo (minutos)	Solución ácida permeada (gramos)
1	7.604
5	15.878
10	25.14
15	39.548
15 minutos 45 segundos	Desprendimiento de gas

La celda se llena después de nuevo con agua de mar y se calienta hasta una temperatura de 45°C. Al presurizar a 7.03 kg/cm², el agua de mar rápidamente pasa a través de la torta de filtro (66.5 g en 17 segundos). La evaluación de la torta de filtro muestra que se han lixiviado áreas irregulares dejando algunos residuos de melamina blancos. Los residuos de la torta de filtro en el papel filtro se colocan en 500 ml de agua de mar y se mantienen a una temperatura de 45°C durante 72 horas. Después de este tiempo no permanecen partículas de melamina visibles.

Esto es muy conveniente para pozos de inyección de agua de mar en los cuales la inyección de agua de mar normalmente se continúa durante años, dejando poca oportunidad de que cualquier torta de filtro de melamina

NOVEDAD DE LA INVENCION

Habiendo descrito el presente invento se considera como novedad y por lo tanto se reclama como
5 propiedad lo contenido en las siguientes:

REIVINDICACIONES

1.- Un fluido para perforación que comprende un
10 fluido base y un agente particulado para resanado
constituido por un material poco soluble en agua que se
selecciona a partir del grupo que consiste de melamina
(2,4,5-triamino-1,3,5-triazina), carbonato de litio,
fosfato de litio (Li_3PO_4), y sulfito de magnesio.

15 2.- Un fluido para perforación de conformidad con
la reivindicación 1 caracterizado porque el fluido base
está presente en el fluido para perforación en una cantidad
en el intervalo de aproximadamente 30 a 99% en peso del
fluido.

20 3.- Un fluido para perforación de conformidad con
las reivindicaciones 1 o 2, caracterizado porque el agente
particulado para resanado poco soluble en agua está
presente en el fluido para perforación en una cantidad en
el intervalo de 1 a 70% en peso.

25 4.- Un fluido para perforación de conformidad con

cualquiera de las reivindicaciones precedentes, caracterizado porque el fluido para perforación es un fluido para barrenado, mantenimiento o terminación y el agente particulado para resanado poco soluble en agua tiene
5 una distribución de tamaño de partícula en el intervalo de aproximadamente 0.1 micras a 600 micras.

5.- Un fluido para perforación de conformidad con cualquiera de las reivindicaciones precedentes, caracterizado porque el fluido para perforación es un
10 fluido de base acuosa y el fluido para perforación comprende por lo menos un aditivo adicional que se selecciona a partir del grupo que consiste de viscosificantes, agentes de ponderación, sales solubles en agua que incrementen la densidad, agentes para control de
15 filtración o pérdida de fluido, agentes para control de pH, inhibidores de hidratación de arcilla o esquisto, bactericidas, agentes tensoactivos, lubricantes sólidos y líquidos, inhibidores de hidrato de gas, inhibidores de corrosión, desespumantes, inhibidores de incrustación,
20 líquidos hidrofóbicos emulsificados tales como aceites, depuradores de gas ácido (tales como depuradores de sulfuro de hidrógeno), adelgazadores (tales como lignosulfonatos), y desemulsificantes.

6.- Un fluido para perforación de conformidad con
25 la reivindicación 5, caracterizado porque el fluido para

perforación de base acuosa comprende un agente para control de pérdida de fluido que se selecciona a partir del grupo que consiste de polímeros solubles en agua, lignitas, lignitas modificadas, y lignosulfonatos entrelazados.

5 7.- Un fluido para perforación de conformidad con cualquiera de las reivindicaciones 1 a 4, caracterizado porque el fluido para perforación es un fluido de base oleosa que comprende por lo menos un aditivo adicional que se selecciona a partir del grupo que consiste de
10 viscosificantes, agentes tensoactivos (para formar emulsiones agua en aceite estables y para impregnar con aceite la superficie de agentes para ponderación minerales), aditivos para control de pérdida de fluido, lubricantes (sólidos y líquidos), y depuradores de gas
15 ácido (por ejemplo, depuradores de sulfuro de hidrógeno).

8.- Un método para formar una torta de filtro removible sobre las paredes de una perforación que penetra una formación de roca porosa y permeable que comprende los pasos de:

20 (a) colocar un fluido para perforación en la perforación en el cual el fluido para perforación comprende un fluido base y un agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina,
25 carbonato de litio, fosfato de litio (Li_3PO_4), y sulfito de

magnesio, de preferencia, melamina y carbonato de litio; y

(b) permitir que el agente particulado para resanado se deposite desde el fluido para perforación sobre y/o en las paredes de la perforación para que forme la
5 torta de filtro removible, con lo cual se reduce la pérdida de fluido hacia la formación a través de la torta de filtro.

9.- Un método de conformidad con la reivindicación 8, caracterizado porque se incluye un
10 aditivo para control de pérdida de fluido en el fluido para perforación.

10.- Un método de conformidad con las reivindicaciones 8 o 9, caracterizado porque el agente particulado para resanado poco soluble en agua se elimina
15 poniendo el pozo en producción.

11.- Un método de conformidad con las reivindicaciones 8 o 9, caracterizado porque el agente particulado para resanado poco soluble en agua se elimina de la perforación de un pozo de inyección mediante
20 disolución en el agua que se inyecta al interior del pozo de inyección.

12.- Un método de conformidad con las reivindicaciones 8 o 9, caracterizado porque el agente particulado para resanado poco soluble en agua se elimina
25 (a) colocando un fluido para limpieza agujero abajo y (b)

dejando que el fluido para limpieza remoje a través del intervalo de la perforación en donde se desea eliminar la torta de filtro durante un periodo de tiempo suficiente ya sea para disolver completamente el agente para resanado o para solubilizar el agente para resanado hasta el grado en el que las partículas sean de un tamaño lo suficientemente reducido para permitir su remoción desde la formación.

13.- Un método de conformidad con la reivindicación 12, caracterizado porque el fluido de limpieza es un fluido acuoso que está sub-saturado con respecto al agente para resanado o es una solución acuosa de un ácido o un precursor de un ácido débil.

14.- Un método de conformidad con la reivindicación 12, caracterizado porque el agente particulado para resanado poco soluble en agua está constituido por sulfito de magnesio, y el fluido para limpieza comprende una solución acuosa de un agente oxidante que pueda convertir el sulfito de magnesio en sulfato de magnesio soluble en agua.

15.- Un método de conformidad con las reivindicaciones 12 o 13, caracterizado porque el agente particulado para resanado poco soluble en agua está constituido por melamina, y la torta de filtro removible se elimina de las paredes de una perforación colocando un fluido acuoso para lavado agujero abajo y permitiendo que

el fluido de lavado remoje el intervalo de la perforación en donde se desea eliminar la torta de filtro durante un periodo de tiempo suficiente para que el fluido de lavado se caliente hasta una temperatura de por lo menos 60°C y
5 dejando que el fluido de lavado caliente remoje el intervalo ya sea hasta que el agente particulado para resanado se disuelva completamente en el fluido de lavado o se solubilize hasta el grado en el que las partículas sean de un tamaño lo suficientemente reducido para permitir su
10 remoción de la formación.

16.- Un método para barrenar una perforación a través de una formación de roca porosa y permeable utilizando un fluido para barrenado que comprende un fluido base, un aditivo para control de pérdida de fluido, y un
15 agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio (Li_3PO_4), y sulfito de magnesio caracterizado porque se mantiene la presión del fluido para
20 barrenado en la perforación por encima de la presión en la formación de roca porosa y permeable de modo tal que se deposite una torta de filtro sobre y/o en las paredes de la perforación y reduzca la pérdida de fluido desde el fluido para barrenado hacia la formación de roca.

25 17.- Un método de barrenado de conformidad con la

reivindicación 16, caracterizado porque el fluido para barrenado es como define en cualquiera de las reivindicaciones 4 a 7.

18.- Un método para controlar la pérdida de fluido desde una perforación hacia el interior de una formación de roca porosa y permeable a través de un conducto de alta conductividad que se extiende desde la perforación al interior de la formación de roca que comprende los pasos de:

10 (a) colocar un fluido para circulación perdida en la perforación caracterizado porque el fluido para circulación perdida comprende una suspensión de material para circulación perdida particulado (LCM) en un fluido base en el cual el LCM está suspendido en el fluido base en
15 una cantidad de por lo menos 14.175 kg/m^3 , de preferencia por lo menos 28.35 kg/m^3 , más preferido, por lo menos 56.7 kg/m^3 , y más preferido aún por lo menos 85.05 kg/m^3 , y está constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina,
20 carbonato de litio, fosfato de litio (Li_3PO_4), y sulfito de magnesio; y

(b) permitir que el LCM se acumule en el conducto de alta conductividad con lo cual se forma un tapón de baja permeabilidad removible que resana el conducto con lo cual
25 se reduce la pérdida de fluido hacia la formación a través

del conducto.

19.- Un método de conformidad con la reivindicación 18, caracterizado porque en la suspensión está presente un agente para control de pérdida de fluido con lo cual se ayuda a sellar el conducto de alta conductividad.

20.- Un método de conformidad con las reivindicaciones 18 o 19, caracterizado porque el fluido base es un fluido para barrenado.

21.- Un método para controlar la pérdida de fluido desde un fluido de terminación al interior de una formación de roca porosa y permeable penetrada por una perforación:

(a) colocando un fluido de terminación en la perforación caracterizado porque el fluido de terminación comprende un fluido base, un aditivo para control de pérdida de fluido, y un agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio, y sulfito de magnesio; y

(b) manteniendo la presión del fluido de terminación en la perforación por encima de la presión de poro de la formación de roca de modo tal que se deposite una torta de filtro sobre o en las paredes de la

perforación.

22.- Un método de conformidad con la reivindicación 21, caracterizado porque el fluido de terminación es como se define en cualquiera de las 5 reivindicaciones 4 a 7.

23.- Un método de conformidad con las reivindicaciones 21 o 22, caracterizado porque el fluido de terminación contiene de manera adicional un agente o agentes viscosificantes poliméricos tales como goma 10 xantano, hidroxietilcelulosa, goma "welan" (por ejemplo, BiozanTM; de Kelco) o goma diutan (por ejemplo, Geovis XTTM; de Kelco).

24.- Un método para controlar la pérdida de fluido desde un fluido de reoperación hacia un intervalo de 15 una perforación que yace a través de una formación de roca porosa y permeable caracterizado porque el método comprende los pasos de:

(a) bombear un volumen suficiente de un primer fluido de reoperación para llenar el intervalo de la 20 perforación que yace a través de la formación de roca porosa y permeable en el cual el primer fluido de reoperación comprende un fluido base, un aditivo para control de pérdida de fluido, y un agente particulado para resanado constituido por un material poco soluble en agua 25 que se selecciona a partir del grupo que consiste de

melamina, carbonato de litio, fosfato de litio, y sulfito de magnesio de modo tal que se deposite una torta de filtro removible desde el primer fluido de reoperación en dicho intervalo de la perforación sobre las paredes de la perforación y al interior de cualesquiera grietas, fracturas o fisuras en la misma;

(b) bombear un segundo fluido de reoperación al interior de la perforación en el cual el segundo fluido de reoperación está sustancialmente saturado con el material poco soluble en agua y tiene una densidad suficiente para contrarrestar por lo menos la presión de la formación de roca porosa y permeable; y en el cual la torta de filtro depositada en el paso (a) reduce la pérdida de fluido desde los fluidos de reoperación hacia la formación de roca porosa y permeable.

25.- Un método para fracturar una formación de roca porosa y permeable que comprende:

inyectar un fluido de fracturación dentro de un intervalo de una perforación a través de la formación de roca que se va a fracturar caracterizado porque el fluido de fracturación comprende un fluido base, agente de soporte, un viscosificante, y un agente para control de escurrimiento particulado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio,

y sulfito de magnesio; y

mantener la presión del fluido de fracturación en el intervalo de la perforación a través de la formación de roca por encima de la presión de fractura de la formación con lo cual el agente de soporte entra y mantiene abiertas las fracturas que se forman en la pared de la perforación y el agente para control escurrimiento particulado sella las gargantas de poro expuestas en las paredes de la fractura.

26.- Un método para desviar fluidos de tratamiento no ácidos lejos de formaciones de roca de alta permeabilidad o conductos de alta conductividad y al interior de formaciones de roca de permeabilidad más baja y/o parcialmente obstruidas o conductos de conductividad más baja utilizando un fluido de tratamiento que comprende un fluido no ácido y un agente particulado para resanado constituido por un material poco soluble en agua que se selecciona a partir del grupo que consiste de melamina, carbonato de litio, fosfato de litio (Li_3PO_4), y sulfito de magnesio.

27.- Un método de conformidad con la reivindicación 26, caracterizado porque el fluido no ácido es un solvente aromático y el fluido de tratamiento se bombean al interior de una perforación para producción hidrocarburos de modo tal que se forme una torta de filtro sobre y/o en una formación de roca de alta permeabilidad

y/o el agente particulado para resanado entre y selle los conductos de alta conductividad (o canales de flujo) en las paredes de la perforación con lo cual se limita la pérdida de solvente aromático desde la perforación con lo cual se
5 desvía el solvente aromático hacia los conductos de baja conductividad (o canales de flujo) que están dañados por depósitos de asfaleno y/o cera, con lo cual se mejora la disolución de los depósitos por parte del solvente aromático.

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(54) Title: WELLBORE FLUID COMPRISING A BASE FLUID AND A PARTICULATE BRIDGING AGENT

(57) Abstract: A wellbore fluid comprising a base fluid and a particulate bridging agent comprised of a sparingly water-soluble ma-
terial selected from the group consisting of melamine (2,4,5- triamino-1,3,5-triazine), lithium carbonate, lithium phosphate (Li₃PO₄),
and magnesium sulfite.

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WELLBORE FLUID COMPRISING A BASE FLUID AND A PARTICULATE BRIDGING AGENT

The present invention relates to wellbore fluids utilized in the construction, repair or treatment of a wellbore and to the removal of filter cake deposited by the wellbore fluids
5 on or in rock formations penetrated by the wellbore.

Wellbore fluids include drilling fluids, lost circulation fluids, completion fluids (such as perforating pills and under-reaming fluids), and servicing fluids (such as workover fluids, milling fluids, fracturing fluids, solvents, aqueous fluids containing non-acidic dissolving agents, and fluids containing particulate diverting agents).

10 Drilling fluids are utilized when drilling a wellbore through a porous and permeable rock formation, for example, a hydrocarbon-bearing rock formation. It is highly desirable that the drilling fluid minimizes damage to the permeability of the rock formation. For example, damage to the permeability of a hydrocarbon-bearing rock formation may result in production losses or a reduced ability of the formation to accept injected fluids (for
15 example, water or treatment fluids).

Completion fluids are utilised during operations that take place in the so-called completion phase of a wellbore (after drilling of the wellbore and before commencement of production of fluids from a rock formation into the wellbore or injection of fluids from the wellbore into a rock formation). Again, it is highly desirable that completion fluids
20 minimize damage to the permeability of rock formations.

Servicing fluids may be utilized intermittently during the life of a wellbore, for example, when conducting work-over, stimulation or remedial operations in a rock formation penetrated by the wellbore. For example, where the servicing fluid is a fracturing fluid, it is highly desirable that leak-off of fluid from the fractures that are
25 induced in the walls of the wellbore is minimized.

Drilling, completion or servicing fluids usually comprise a particulate solid bridging agent of a particle size that is large enough for bridging the pore throats of a porous and permeable rock formation and a filtration control additive (often termed a "fluid loss control additive"). The drilling, completion or servicing fluids deposit a layer
30 of particles known as a "filter cake" on the walls of the wellbores. Where the wellbore penetrates a porous and permeable rock formation, this low-permeability filter cake prevents large amounts of fluids ("filtrate") from being lost from the drilling, completion

or servicing fluid into the rock formation and also prevents solids from entering the pores of the formation. Fluid that is lost from a drilling, completion or servicing fluid into a porous and permeable rock formation is termed "filtrate". The filter cake is comprised of the particulate bridging agent and the fluid loss control agent and will also include other
5 solids that are present in the wellbore fluid and are capable of depositing onto the walls of the wellbore. After the drilling, completion or servicing of the wellbore, it is advantageous that as much as possible of the filter cake is removed before commencing production of fluids from a porous and permeable rock formation into the wellbore or before fluid is injected into a porous and permeable rock formation from the wellbore. However, it is
10 often difficult to access and remove substantial amounts of the filter cake.

In the event that a large volume of wellbore fluid is being lost through high conductivity conduits in the walls of a wellbore into a porous and permeable rock formation, a lost circulation fluid comprising a Lost Circulation Material (LCM) suspended in a base fluid is pumped into the wellbore. The high conductivity conduits are typically
15 fissures, fractures or vugs in the walls of the wellbore (where a vug is a cavity, void or large pore in a rock formation). Lost circulation fluids frequently comprise coarser particulate solids (LCM) than the particulate bridging agents of drilling, servicing or completion fluids in order to bridge and seal the high conductivity conduits into which the wellbore fluid is being lost. Thus, a relatively low permeability plug comprising the
20 particulate LCM and optionally other solids is deposited from the lost circulation fluid in the high conductivity conduits. These particulate plugs can be difficult to remove from the high conductivity conduits when it is desired to commence production of fluids from a rock formation into a production wellbore or injection of fluids into a rock formation from an injection wellbore.

25 Conventionally, filter cakes are removed from wellbore walls by contacting the filter cakes with one or more clean-up fluids. One common bridging agent for bridging the pore throats of a porous formation and for plugging any high conductivity conduits (e.g. fissures) therein is powdered calcium carbonate. The filter cake may be removed by using a clean-up fluid comprising enzymes and oxidizers to degrade the fluid loss control
30 additive prior to contacting the filter cake with a strongly acidic clean-up solution for a sufficient period of time to dissolve the particulate calcium carbonate bridging agent. However, despite current anti-corrosion steps, the strongly acidic solution often corrodes

metallic surfaces and completion equipment such as sand control screens causing early failure of such equipment. The acidic clean-up solution may also be incompatible with the producing formation and may cause formation damage. In addition, inefficient dissolution of filter cake occurs when an acidic clean-up solution reacts rapidly with a portion of the filter cake, opening up fluid communication between the clean-up fluid in the wellbore and the permeable formation, whereupon the clean-up fluid enters the formation without contacting the remaining filter cake. Another problem arises when an expandable sandscreen is placed in an open hole wellbore in the interval of the wellbore adjacent to a hydrocarbon-bearing formation. After placement of the sandscreen, it is expanded to fit the diameter of the wellbore thereby providing excellent support to the wellbore and exclusion of sand production. Unfortunately, this results in the filter cake becoming trapped between the expanded sandscreen and the formation so that it is very difficult to access the filter cake with a clean-up solution. Under such circumstances it would be advantageous if the filter cake were soluble in less-corrosive and less-damaging fluids, for example, naturally occurring wellbore fluids. In this way untreated or trapped filter cake should ultimately be reached by and become dissolved in the fluids.

Where the filter cake is deposited on and/or in the walls of a hydrocarbon production wellbore, the hydrocarbon-bearing formation will generally produce a significant proportion of water. Where the filter cake is deposited on and/or in the walls of a water injection well or a water producing well, the filter cake will again be exposed to large volumes of water over a long period of time. Where the filter cake is deposited on and/or in the walls of a geothermal wellbore, the filter cake will be exposed to hot water and steam. Accordingly, particulate solid bridging agents formed of a water-soluble salt (for example, alkali metal halides) or a sparingly water-soluble salt (for example, magnesium borate and magnesium salts of carboxylic acids) have been utilized or proposed in drilling or servicing fluids. Thus, filter cakes containing the water-soluble or sparingly water-soluble bridging agent have been removed by contacting the filter cake with an aqueous salt solution which is undersaturated with respect to the water-soluble or sparingly water-soluble salt. These water-soluble or sparingly water-soluble bridging agents may be employed in either an oil-based treatment fluid or in an aqueous based treatment fluid provided that the aqueous base fluid is saturated with respect to the water-soluble or sparingly water-soluble salt. However, there remains a need for further wellbore

fluids where the bridging agent is comprised of a sparingly water-soluble material.

Accordingly, the present invention relates to a wellbore fluid comprising a base fluid and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine (2,4,5-triamino-1,3,5-triazine), lithium carbonate, lithium phosphate (Li_3PO_4), and magnesium sulfite, preferably, melamine and lithium carbonate.

The term "wellbore fluid" as used herein encompasses drilling fluids, lost circulation fluids, completion fluids such as perforating pills and under-reaming fluids, and servicing fluids such as kill fluids, workover fluids, milling fluids, fracturing fluids, solvents, non-acidic aqueous dissolving agents, and fluids containing particulate diverting agents.

The wellbore fluid of the present invention is suitable for use in a variety of wellbores including oil and/or gas producing wellbores, water or gas injection wellbores, water producing wellbores and geothermal wellbores.

The sparingly water-soluble materials that have been selected for use as the particulate bridging agent have a solubility in water at a temperature of 25°C of less than 7% by weight, preferably less than 2% by weight. In addition, these materials have a solubility in water at a temperature of 80°C of less than 7% by weight, preferably less than 3.5% by weight.

Optionally, a fluid loss control additive is included in the wellbore fluid of the present invention.

The present invention also provides a method of forming a removable filter cake on the walls of a wellbore that penetrates a porous and permeable rock formation comprising the steps of:

- (a) placing a wellbore fluid in the wellbore wherein the wellbore fluid comprises a base fluid and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate (Li_3PO_4), and magnesium sulfite, preferably, melamine and lithium carbonate; and
- (b) permitting the particulate bridging agent to deposit from the wellbore fluid onto and/or into the walls of the wellbore thereby forming the removable filter cake, whereby fluid loss to the formation through the filter cake is reduced.

Suitably, the particulate bridging agent may bridge the pore throats of the rock

formations penetrated by the wellbore and/or may enter any cracks, fissures, fractures, or vugs in the wellbore wall.

Optionally, a fluid loss control additive is included in the wellbore fluid.

By "removable" is meant that the filter cake may be removed without pumping a specialised clean-up fluid into the wellbore. In other words, the filter cake may be self-removing.

The filter cake is permitted to build up on the walls of the wellbore owing to the pressure of the wellbore fluid in the wellbore being maintained at above the pore-pressure of the porous and permeable formation that is penetrated by the wellbore. Preferably, the differential pressure between the pressure of the wellbore fluid in the wellbore and the pore-pressure is at least 200 psi.

Where the wellbore is a hydrocarbon production wellbore, the bridging agent may be removed by putting the well into production owing to the water that is co-produced with the hydrocarbon dissolving the sparingly water-soluble material. Where the wellbore is a water production well or a geothermal well, the bridging agent may be removed by putting the well into production owing to the sparingly water-soluble material dissolving in the produced water. Where the wellbore is a water injection well, the filter cake may be removed by commencing water injection owing to the injected water dissolving the sparingly water-soluble material. Thus, both the produced water and injected water are undersaturated with respect to the sparingly water-soluble material. The bridging agent can be eventually completely solubilised in water or alternatively solubilised to the extent that the particles are sufficiently reduced in size to permit their removal from the formation with the produced or injected water. The time required to solubilise the particles depends on a number of factors including, the temperature in the wellbore, the size and shape of the bridging agent particles, and the amount of water that the filter cake is exposed to. The filter cake is expected to subsist for less than 200 hours when a production well is put into production or when water is injected into an injection well.

Optionally, if rapid dissolution is required, a clean-up fluid may be pumped into the wellbore. The clean-up fluid may be an aqueous fluid that is under-saturated with respect to the bridging agent. Preferably, the clean-up fluid is an aqueous solution of an acid, preferably, an aqueous solution of a weak acid or a precursor of a weak acid. Preferably, the weak acid is selected from the group consisting of formic acid, citric acid, acetic acid,

lactic acid, glycolic acid, succinic acid, and acidic sequestrants such as those based upon partially neutralised ethylenediaminetetracetic acid (EDTA). Preferably, the precursor of the weak acid is selected from materials that are capable of hydrolysing to produce weak acids such as the polyglycolic or polylactic homopolyesters and orthoesters such as orthoformate esters. Preferably, the weak acid or acid precursor is present in the clean-up fluid in an amount of between 1% and 20% by weight. An advantage of using an aqueous solution of a weak acid or an aqueous solution of a precursor of a weak acid is that the clean-up fluid is less corrosive to metal surfaces and equipment than the strong acids that are used to dissolve conventional inorganic bridging agents such as calcium carbonate. A further advantage of the sparingly water-soluble materials employed in the present invention is that even partial reaction with an acid forms products having a higher solubility in water. Thus, lithium carbonate is converted to lithium bicarbonate, lithium phosphate (Li_3PO_4) is converted to lithium hydrogen phosphates, and magnesium sulfite is converted to magnesium bisulfite upon partial reaction with an acid. All of these products are of much higher solubility than their sparingly water-soluble precursors. In addition, the amine groups that are present in melamine will protonate under mildly acidic conditions, greatly increasing the water-solubility of the bridging agent.

Where the sparingly water-soluble particulate bridging agent is comprised of magnesium sulfite, the clean-up fluid may comprise an aqueous solution of an oxidizing agent that is capable of converting magnesium sulfite to water-soluble magnesium sulfate. Thus, magnesium sulfate has a much higher solubility in water than magnesium sulfite. Suitable oxidizing agents include hydrogen peroxide, persulfate salts, and per-acids such as peracetic acid. Preferably, the oxidizing agent is present in the clean-up fluid in an amount of 1 to 20% by weight. Optionally, the clean-up fluid also-comprises a weak acid or a precursor of a weak acid.

Where the sparingly water-soluble particulate bridging agent is comprised of melamine, it is envisaged that the removable filter cake may be removed from the walls of a wellbore by placing an aqueous wash fluid downhole and leaving the aqueous wash fluid to soak in the interval of the wellbore where it is desired to remove the filter cake. The soak time should be sufficient for the aqueous wash fluid to heat up to a temperature of at least 60°C, preferably at least 75°C, for example, at least 90°C. Because the solubility of melamine increases relatively rapidly with increasing temperature, the particulate bridging

agent is either completely dissolved in the aqueous wash fluid or is solubilised to the extent that the particles are sufficiently reduced in size to permit their removal from the formation. The aqueous wash fluid is heated to the desired temperature owing to transfer of geothermal heat from the formation. Typically, it may take at least several hours, for example, about 1 day for the aqueous wash fluid to be heated to the desired temperature. In general, the operator will be able to determine that a sufficient period of time has elapsed when the fluid loss rate from the wellbore to the formation increases.

The rate of dissolution of the bridging agents in water is also enhanced by the presence of carbon dioxide. Accordingly, the high partial pressures of carbon dioxide that are often present in fluids produced from hydrocarbon-bearing formations can be expected to accelerate dissolution of the bridging agent.

Optionally, a clean-up fluid is placed downhole and left to soak across the interval of the wellbore where it is desired to remove the filter cake for a sufficient period of time to either completely dissolve the bridging agent or to solubilise the bridging agent to the extent that the particles are sufficiently reduced in size to permit their removal from the formation. The clean-up fluid may contain enzymes or oxidising agents to degrade the fluid-loss control and viscosifying polymers that accumulate in the filter-cake, and may contain acids or acid precursors to speed the dissolution of the bridging solids. Preferably, the optional clean-up solution is left to soak for about 2 to 24 hours. In general, the operator will be able to determine that a sufficient period of time has elapsed when the fluid loss rate from the wellbore to the formation increases. Thereafter, a wash fluid (for example, an aqueous fluid such as water or seawater or a dilute brine) may be pumped at a high rate into the wellbore in order to create turbulent cleaning conditions thereby removing the remaining filter cake from the walls of the wellbore. Alternatively, the remaining filter cake may be removed by producing water from the formation or by injecting water into the formation.

Preferably, the wellbore fluid is selected from (a) a drilling fluid; (b) a fluid used to control lost circulation (termed "loss circulation fluid"); (c) a completion fluid used during completion operations; and (d) a well-servicing fluid used when conducting work-over, stimulation or remediation operations.

Thus, in a preferred embodiment of the present invention there is provided a method of drilling a wellbore through a porous and permeable rock formation using a

drilling fluid comprising a base fluid, a fluid loss control additive, and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate (Li_3PO_4), and magnesium sulfite wherein the pressure of the drilling fluid in the wellbore is maintained at above the
5 pressure in the porous and permeable rock formation such that a filter cake deposits on and/or in the walls of the wellbore and reduces fluid loss from the drilling fluid to the rock formation.

By being deposited "in the walls of the wellbore" is meant that filter cake may be deposited in any cracks, fractures, fissures or vugs that are present in the walls of the
10 wellbore.

Suitably, the wellbore that is drilled using this preferred embodiment of the present invention is a hydrocarbon production wellbore (an oil or gas well), an injection wellbore (for example, a water or gas injection well), a water-producing wellbore or a geothermal wellbore.

15 In another preferred embodiment of the present invention there is provided a method of controlling loss of fluid from a wellbore into a porous and permeable rock formation through a high conductivity conduit that extends from the wellbore into the rock formation comprising the steps of:

- (a) placing a lost circulation fluid in the wellbore wherein the lost circulation fluid
20 comprises a slurry of particulate lost circulation material (LCM) in a base fluid wherein the LCM is suspended in the base fluid in an amount of at least 5 pounds per barrel, preferably at least 10 pounds per barrel, more preferably at least 20 pounds per barrel, and most preferably at least 30 pounds per barrel, and is comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium
25 phosphate (Li_3PO_4), and magnesium sulfite; and
(b) permitting the LCM to accumulate in, or at the entrance of, the high conductivity conduit thereby forming a removable low-permeability plug that bridges the conduit whereby fluid loss to the formation through the conduit is reduced.

By "removable" is meant that the plug may be removed without the assistance of a
30 specifically designed clean-up fluid.

The slurry is pumped into the interval of the wellbore where a high conductivity conduit (for example a fissure) is present in the wall of the wellbore and through which

fluid is being lost into the porous and permeable rock formation, for example, a hydrocarbon-bearing rock formation. Filtration of the slurry results in deposition of the particulate LCM in the high conductivity conduit such that the conduit becomes filled with a solid pack of LCM particles. Optionally, a fluid loss control agent may be present in the slurry thereby assisting in sealing the high conductivity conduit. Preferably the sealing of the conduit is made more complete when a subsequent wellbore fluid such as drilling fluid, in particular, a low fluid loss drilling fluid, forms an impermeable filter cake upon the plug of particulate LCM.

In yet another preferred embodiment of the present invention there is provided a method of controlling loss of fluid from a completion fluid into a porous and permeable rock formation penetrated by a wellbore by:

- (a) placing a completion fluid in the wellbore wherein the completion fluid comprises a base fluid, a fluid loss control additive, and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate, and magnesium sulfite; and
- (b) maintaining the pressure of the completion fluid in the wellbore at above the pore-pressure of the rock formation such that a filter cake deposits on or in the walls of the wellbore.

Suitably, the completion fluid (and also the drilling fluid referred to above) additionally contains a polymeric viscosifier(s) such as xanthan gum, hydroxyethylcellulose, welan gum (for example, Biozan™; ex Kelco) or diutan gum (for example, Geovis XT™; ex Kelco). The completion fluid that is placed in the wellbore may fill the entire wellbore. Alternatively, the completion fluid may be employed as a pill of sufficient volume to fill the interval of the wellbore that is to be "completed" with the remainder of the wellbore being filled with a second fluid having an appropriate density for well control purposes. Thus, the density of the second fluid is chosen such that fluid does not flow from a rock formation into the wellbore. It is envisaged that the second fluid may be a brine that is substantially free of suspended solids.

It is also envisaged that the wellbore may be a cased wellbore that is perforated in an interval of the wellbore that lies across a porous and permeable rock formation, for example, a hydrocarbon-bearing rock formation. Accordingly, the filter cake will deposit from the completion fluid in the perforation tunnels formed in the cased wellbore thereby

reducing fluid loss from the completion fluid to the formation.

In yet a further preferred embodiment of the present invention, there is provided a method of controlling loss of fluid from a workover fluid to an interval of a wellbore that lies across a porous and permeable rock formation wherein the method comprises the steps of:

- (a) pumping a sufficient volume of a first workover fluid to fill the interval of the wellbore that lies across the porous and permeable rock formation wherein the first workover fluid comprises a base fluid, a fluid loss control additive, and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate, and magnesium sulfite such that a removable filter cake deposits from the first workover fluid in said interval of the wellbore onto the walls of the wellbore and into any cracks, fractures or fissures therein;
 - (b) pumping a second workover fluid into the wellbore wherein the second workover fluid is of sufficient density to at least counterbalance the pressure of the porous and permeable rock formation;
- and wherein the filter cake deposited in step (a) reduces fluid loss from the workover fluids to the porous and permeable rock formation.

The second workover fluid may be of the same composition as the first workover fluid or may be of a different composition, for example a solids free brine or oil. In the case of aqueous workover fluids, it is preferred that the second workover fluid is substantially saturated with respect to the sparingly water-soluble material that comprises the particulate bridging agent of the first workover fluid. The first workover fluid is used to seal the formation to prevent fluid losses from the second workover fluid while the second workover fluid is used to perform functions such as maintaining well control (hydrostatic head), circulating debris such as "milled out" downhole equipment out of the wellbore (for example, "milled out" packers or screens), providing a low viscosity fluid to allow the easy running of tools in and out of the wellbore and acting as a "re-completion" fluid.

Where the wellbore is a cased wellbore that is perforated in the interval of the wellbore across a porous and permeable rock formation, the filter cake will deposit from the first workover fluid in the perforation tunnels in the casing of the wellbore thereby reducing fluid loss from the second workover fluid to the formation.

Wells that require a "workover" are often depleted hydrocarbon production wells where the hydrocarbon-bearing rock formation has a low pore pressure. Accordingly, the hydrostatic head of the second workover fluid in the interval of the wellbore across the hydrocarbon-bearing rock formation may be greatly in excess of the pore pressure in the depleted hydrocarbon-bearing rock formation even when the second workover fluid is a simple low-density fluid such as water (for example, seawater), or an oil. Accordingly, the ability to control fluid loss by the method described above is more important at high differential pressures (where the pressure of the second workover fluid in the wellbore is significantly higher than the pore pressure of the rock formation).

Fracturing fluids generally comprise a proppant (for example, sand particles or ceramic beads) suspended in an aqueous base fluid that is normally viscosified by a polymer or a viscoelastic surfactant such that the proppant that is used to prop open the fractures is efficiently transported into the fractures that are created when the fracturing fluid is pumped at high pressure into a porous and permeable rock formation. However, if the fracturing fluid leaks off too quickly into the formation the high pressure dissipates and the fractures cease to grow. Leak-off control is normally achieved by dispersing ground particles such as silica flour in the fracturing fluid to block / bridge the exposed pores in the fracture that are accepting the "leak-off". Unfortunately, materials like silica may cause at least some permanent plugging of the pores of the formation.

Accordingly, in yet another preferred embodiment of the present invention there is provided a method of fracturing a porous and permeable rock formation comprising: injecting a fracturing fluid into an interval of a wellbore across the rock formation that is to be fractured wherein the fracturing fluid comprises a base fluid, proppant, a viscosifier, and a particulate leak-off control agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate, and magnesium sulfite; and maintaining the pressure of the fracturing fluid in the interval of the wellbore across the rock formation at above the fracture pressure of the formation whereby proppant enters and props open the fractures that are formed in the wellbore wall and the particulate leak-off control agent seals exposed pore throats on the walls of the fracture.

An advantage of this preferred embodiment of the present invention is that the pressure of the fracturing fluid in the growing fracture is maintained for as long as possible

at above the fracturing pressure of the rock formation by reducing leak-off of fluid to the formation and hence reducing pressure dissipation to the formation. Where the fractures are formed in a hydrocarbon-bearing rock formation penetrated by a production wellbore, the particulate bridging material will dissolve in co-produced water upon returning the wellbore to production thereby improving the flow of fluid from the hydrocarbon-bearing formation. Where the fractures are formed in a porous and permeable rock formation penetrated by a water injection well, the particulate bridging material will dissolve in water that is injected into the rock formation, thereby improving the flow of fluid from the injection well into the formation.

In another embodiment of the present invention, there is provided a method of diverting non-acidic treatment fluids away from high permeability rock formations or high conductivity conduits and into lower permeability and/or partially plugged rock formations or lower conductivity conduits using a treatment fluid comprising a non-acidic fluid and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate (Li_3PO_4), and magnesium sulfite. For example, where the non-acidic fluid is an aromatic solvent, the treatment fluid may be used to dissolve wax and/or asphaltene deposits that plug flow channels in oil wells (and hence reduce oil production). The method comprises pumping a suspension comprising the particulate bridging agent suspended in an aromatic solvent into a hydrocarbon production wellbore such that a filter cake forms on or in a high permeability rock formation or the particulate bridging agent enters and seals high conductivity conduits (or flow channels) in the walls of the wellbore thereby limiting the loss of aromatic solvent from the wellbore. Accordingly, the aromatic solvent is diverted towards low conductivity conduits (or flow channels) that may be damaged by asphaltene and/or wax deposits, thereby improving the dissolution of the deposits by the aromatic solvent.

Preferred features of the wellbore fluid of the present invention will now be described below.

The base fluid of the wellbore fluid may be water, an oil (for example, a mineral oil), a solvent (for example, an aromatic solvent), or a mixture thereof (for example, a water-in-oil emulsion). Generally, the base fluid is present in the wellbore fluid in an amount in the range of from about 30 to 99% by weight of the fluid, preferably, about 70 to

97% by weight.

Where the base fluid is water, it is preferred that the base fluid is an aqueous solution of a density increasing water-soluble salt. The density increasing water-soluble salt may be selected from the group consisting of alkali metal halides (for example, sodium chloride, sodium bromide, potassium chloride and potassium bromide) alkali metal carboxylates (for example, sodium formate, potassium formate, caesium formate, sodium acetate, potassium acetate or caesium acetate), sodium carbonate, potassium carbonate, alkaline earth metal halides (for example, calcium chloride and calcium bromide), and zinc halide salts.

Alternatively, density control may be provided to the water-based wellbore fluid using insoluble weighting agents. Suitable weighting agents include suspended mineral particles such as ground barites, iron oxides, (for example, haematite), ilmenite, calcite, magnesite (MgCO_3), dolomite, olivine, siderite, hausmannite or suspended metal particles.

Where the base fluid is an oil, it is preferred that the oil is selected from the group consisting of mineral oils, synthetic oils, esters, kerosene, and diesel.

The base fluid may also be a water-in-oil emulsion comprising droplets of an aqueous phase dispersed in a continuous oil phase. Suitably, the aqueous phase of the emulsion comprises an aqueous solution of a density increasing water-soluble salt thereby increasing the density of the wellbore fluid. Suitable density-increasing water-soluble salts are listed above. Preferably, the concentration of salt in the dispersed droplets of aqueous phase is adjusted to provide a Water Activity similar to that of the underground formation being contacted by the wellbore fluid. The continuous oil phase may be any oil in which an aqueous solution of salts can be emulsified. Suitable oils are listed above. An advantage of a water-in-oil emulsion is that this enhances both filtration control (owing to the emulsion droplets blocking the flow of fluid through the filter cake) and the viscous properties of the fluid. The term oil-based wellbore fluid as used herein encompasses wellbore fluids where the base fluid is a water-in-oil emulsion.

Density control may also be provided to the oil-based wellbore fluid using weighting agents. Suitable weighting agents are as listed above for aqueous-based wellbore fluids.

Where the base fluid is water, the particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium

carbonate, lithium phosphate, and magnesium sulfite (hereinafter "sparingly water-soluble particulate bridging agent") is dosed into the wellbore fluid at a concentration that is significantly higher than its solubility in water at the temperature encountered downhole thereby ensuring at least a portion of the bridging solids remain suspended in the wellbore fluid. Alternatively, the sparingly water-soluble particulate bridging agent may be protected with a hydrophobic coating that is capable of dissolving in a produced liquid hydrocarbon, for example, a produced oil or produced gas condensate. However, such coated particulate bridging agents should not be employed when drilling or completing water injection wells or gas wells that are free of gas condensate.

Generally the sparingly water-soluble particulate bridging agent is present in the wellbore fluid in an amount sufficient to create an efficient filter cake that provides the desired level of fluid loss control. Typically, the sparingly water-soluble particulate bridging agent is present in the wellbore fluid in an amount in the range of 1 to 70% by weight, preferably 2 to 50% by weight, more preferably, 3 to 30% by weight, in particular 3 to 15% by weight. High doses are preferred for lost circulation fluids, for example 10 to 60% by weight.

The desired particle size distribution of the sparingly water-soluble particulate bridging material is determined by the size of any fractures and the like into which the wellbore fluid is being lost or by the pore throat size of the formation that is to be drilled or treated. Typically, for use as a Lost Circulation material the sparingly water-soluble particulate bridging agent has a particle size distribution in the range of from about 50 microns to about 10 mm, preferably 50 microns to about 2mm. For use as a bridging solid in a drilling, servicing or completion fluid the sparingly water-soluble particulate bridging agent has a particle size distribution in the range of from about 0.1 micron to 600 microns, preferably 0.1 to 200 microns, and more preferably 0.1 to 100 microns. Preferably, the sparingly water-soluble particulate bridging material has a broad polydisperse size distribution. The materials (lithium carbonate, lithium phosphate, magnesium sulfite and melamine) are available as crystalline materials of the desired size or as crystals or granules that can be ground to the desired size. The sparingly water-soluble particulate bridging material may be in the form of substantially spherical particles or may be of an irregular shape.

More than one sparingly water-soluble particulate bridging agent may be employed

in the wellbore fluid.

The wellbore fluids may additionally comprise one or more of the following materials: a conventional particulate bridging or weighting agent, for example barite; acid-soluble materials such as calcium carbonate; water-soluble materials such as alkali metal halides; and, other sparingly water-soluble materials such as magnesium borate and magnesium salts of carboxylic acids. These conventional particulate bridging agents may be employed in either an oil-based wellbore fluid or in an aqueous based wellbore fluid. Where the conventional particulate bridging agent is comprised of a water-soluble or sparingly water-soluble material, it is employed in an aqueous based fluid in amounts above the saturation concentration of the water-soluble or sparingly water-soluble material in water at the conditions encountered downhole so as to provide suspended particles of the conventional particulate bridging agent. Water-based wellbore fluids may additionally comprise particulate solid bridging agents comprised of oil-soluble materials such as resins. Suitable resins include thermoplastic resins derived from the polymerization of hydrocarbons, having an amorphous or crystalline structure which allows it to be crushed and ground at room temperature while retaining its strength so that it remains non-deformable when subjected to pressure in the pores and fissures of a rock formation. These resins have a melting point above the temperature encountered downhole and are insoluble in aqueous based treatment fluids but are soluble in produced crude oils and gas condensates. Examples of preferred resins include coumarone-indene resins, and alkylated aromatic resins.

Preferably, the sparingly water-soluble particulate bridging agent employed in the present invention comprises a significant portion of the suspended solids contained in the wellbore fluid and hence in the filter cake. Suitably, the sparingly water-soluble particulate bridging agent comprises at least 15% by volume, preferably at least 30% by volume, more preferably at least 60% by volume of the suspended solids of the wellbore fluid (the remainder being conventional particulate bridging agents, weighting agents, drilled solids, and clays). Without wishing to be bound by any theory it is believed that dissolution of the sparingly water-soluble particulate bridging agent creates voids in the filter cake thereby rendering it permeable. Where the filter cake is formed in a production well, the filter cake is readily degraded when the well is put into production owing to produced fluids flowing more freely through the permeable filter cake. Accordingly, other

solids that are deposited in the filter cake become entrained in the produced fluid such that the filter cake is removed from the wellbore wall.

Where the wellbore fluid is an aqueous based fluid, the wellbore fluid may comprise additional additives for improving the performance of the wellbore fluid with respect to one or more properties. Examples of additives that may be added to aqueous based wellbore fluids include viscosifiers, weighting agents, density increasing water-soluble salts, fluid loss control agents (also known as filtration control additives), pH control agents, clay or shale hydration inhibitors (such as polyalkylene glycols), bactericides, surfactants, solid and liquid lubricants, gas-hydrate inhibitors, corrosion inhibitors, defoamers, scale inhibitors, emulsified hydrophobic liquids such as oils, acid gas-scavengers (such as hydrogen sulfide scavengers), thinners (such as lignosulfonates), demulsifiers and surfactants designed to assist the clean-up of invaded fluid from producing formations.

Water-soluble polymers may be added to an aqueous based wellbore fluid to impart viscous properties, solids-dispersion and filtration control to the fluid. A wide range of water-soluble polymers may be used for an aqueous based wellbore fluid including cellulose derivatives such as carboxymethyl cellulose, hydroxyethylcellulose, carboxymethylhydroxyethyl cellulose, sulphoethylcellulose; starch derivatives (which may be cross-linked) including carboxymethyl starch, hydroxyethylstarch, hydroxypropyl starch; bacterial gums including xanthan, welan, diutan, succinoglycan, scleroglucan, dextran, pullulan; plant derived gums such as guar and locust-bean gums and their derivatives; synthetic polymers and copolymers derived from any suitable monomers including acrylic acid or methacrylic acid and their hydroxylic esters (for example, hydroxyethylmethacrylic acid), maleic anhydride or acid, sulphonated monomers such as styrenesulphonic acid and AMPS, acrylamide and substituted acrylamides, N-vinylformamide and N-vinylacetamide, N-vinylpyrrolidone, vinyl acetate, N-vinylpyridine and other cationic vinylic monomers (for example, diallyldimethylammonium chloride, DADMAC); and any other water-soluble or water-swellaable polymers known to those skilled in the art. Generally, viscosifying water-soluble polymers are present in the wellbore fluid of the present invention in an amount sufficient to maintain the bridging and weighting solids in suspension and provide efficient clean out from the well of debris such as drilled cuttings. The viscosifying polymer may be present in the wellbore fluid in an

amount in the range of 0.2 to 5 pounds of viscosifier per barrel of wellbore fluid, preferably 0.5 to 3 pounds per barrel of wellbore fluid.

Rheological control (for example, gelling properties) can also be provided to the aqueous based wellbore fluid by adding clays and/or other inorganic fine particles.

- 5 Examples include bentonite, montmorillonite, hectorite, attapulgite, sepiolite, LaponiteTM (ex Laporte) and mixed metal hydroxides.

- A fluid loss control additive may be used to fill the voids between the particulate bridging agent. Besides the water-soluble polymers listed above, examples of fluid loss control additives for water-based wellbore fluids include causticised lignite, modified
10 lignites, cross-linked lignosulphonates and the like. Thus, these fluid loss control additives are dissolved macromolecules that are capable of adsorbing onto the bridging solids or are macromolecules that are in colloidal dispersion in the aqueous base fluid, for example, a hydrated polymer that adopts a coiled conformation when dispersed in the aqueous base fluid rendering the hydrated polymer capable of plugging micro- or nano-sized pores in the
15 filter cake.

Suitable pH control agents for aqueous based wellbore fluids include calcium hydroxide, magnesium hydroxide, magnesium oxide, potassium hydroxide, sodium hydroxide and the like.

- Where the wellbore fluid is an oil based fluid, the wellbore fluid may comprise
20 additional additives for improving the performance of the wellbore fluid with respect to one or more properties. Examples of additives that may be added to oil-based wellbore fluids include viscosifiers, surfactants (for forming stable water-in-oil emulsions and to oil-wet the surface of mineral weighting agents), fluid loss control additives (also known as filtration control additives), lubricants (solid and liquid), and acid gas scavengers (for
25 example, hydrogen sulfide scavengers).

- A viscosifier may be added to the oil-based wellbore fluid to impart viscous properties, solids suspension and hole cleaning properties to the fluid. Normally the viscosifier is a montmorillonite or hectorite clay that has been treated with fatty quaternary ammonium salts to render the clay dispersible and exfoliatable in the oil-based wellbore
30 fluid. Oil-soluble polymers and oligomers may be used as rheological modifiers.

Surfactants that may be added to the oil-based fluid to form stable water-in-oil emulsions and to oil-wet the surface of mineral weighting agents include fatty acids such

as Tall Oil Fatty Acid (TOFA) and condensation products of TOFA with polyalkylene amines such as triethylenetetramine. The resulting fatty amidoamine and imidazoline products may be used as is or they may be further reacted with, for example, maleic anhydride to improve their performance. Where the surfactant contains a carboxylic acid functional group, such groups are generally converted to the corresponding calcium salt by addition of lime.

Suitable fluid loss control additives that may be added to the oil-based wellbore fluid include asphalt, blown asphalt, sulphonated asphalt, gilsonite, fatty amine-modified lignite, and synthetic oil-soluble/swellable polymers.

The present invention will now be illustrated with respect to the following examples

Solubility Tests

The following tests show the solubility in water and in aqueous acidic solutions of the sparingly water-soluble materials.

Example 1 – Solubility of Melamine

The solubility of Melamine in water over a range of temperatures is given below in Table 1. The person skilled in the art would understand that a sufficient quantity of water is all that is required to dissolve particulate melamine deposited in a well, especially if the well is allowed to warm up towards its natural (prevailing) temperature after the cooling experienced during drilling of a wellbore or during injection of cold water from the surface. Accordingly, particulate melamine may automatically clean up (dissolve) in water that may be produced from a well along with hydrocarbons, or in water that is pumped into an injection well to maintain reservoir pressure.

Table 1

Temperature (°C)	Solubility of Melamine (% by weight)
20	0.3
35	0.6
50	1.05
60	1.5
80	3.0
100	5
120	8

Melamine is also readily soluble in warm or hot acids such as acetic acid and hydrochloric acid. Thus, a mixture of melamine (25.2 g, 0.2M), 250 ml of water and 24 g acetic acid (0.4M) gives a clear solution when heated to a temperature of 80°C. Similarly a mixture of melamine (126 g, 1 M) and 1985 ml of 1.0075M hydrochloric acid produces a clear solution when heated to a temperature of 83°C.

The solubility of melamine in aqueous acidic solutions is advantageous where stimulation of the well by acid injection is contemplated, or where large amounts of particulate melamine are placed in the well, for instance as lost circulation material plugs in fractured formations.

Example 2 - Solubility of Lithium Carbonate

The solubility of lithium carbonate in water over a range of temperatures is given below in Table 2. As for Example 1, the person skilled in the art would understand that a sufficient quantity of water is all that is required to dissolve lithium carbonate particles that are deposited in a well. As before, this could be produced water or water pumped into an injection well, or an aqueous fluid placed into the well for the purpose of dissolving the lithium carbonate particles. The reduction in solubility with increasing temperature is an advantage where particulate lithium carbonate is used in higher temperature wells (for example wells having a bottom hole temperature (BHT) of 100°C or more) in that premature dissolution of the solid particles is more easily avoided.

Table 2

Temperature (°C)	Solubility of Lithium Carbonate (g per 100 g of water)
20	1.33
40	1.17
60	1.01
80	0.85
100	0.72

Lithium carbonate also rapidly dissolves in acids. For instance, acetic acid reacts with lithium carbonate to generate lithium acetate which is very soluble in aqueous solutions while hydrochloric acid reacts with lithium carbonate to generate the highly soluble lithium chloride salt. The ability to remove particulate lithium carbonate by pumping an acid into a well is an advantage where large amounts of particulate lithium carbonate are placed in the well, for instance as lost circulation material plugs in fractured formations.

Lithium carbonate also shows an enhanced solubility in water in the presence of carbon dioxide, which is frequently found in fluids produced from oil-wells or gas-wells (owing to the formation of lithium bicarbonate, LiHCO_3). For instance, at a temperature of 60°C and at a pressure of 50 atmospheres of CO_2 , 100g of saturated solution contains 9.61g of LiHCO_3 .

The high solubility of LiHCO_3 is particularly advantageous in gas wells since the gas in the formation is almost inevitably saturated with water vapour and generally contains high concentrations of CO_2 . As gas flows through the gas-bearing formation towards a producing gas well, the pressure reduces causing adiabatic cooling and condensation of water. The condensed water together with the high concentration of CO_2 will therefore dissolve particulate lithium carbonate residues without any need to pump dissolving fluids from the surface.

Example 3 – Solubility of Magnesium Sulfite

The solubility of magnesium sulfate in water over a range of temperatures is given below in Table 3. As for Examples 1 and 2, a sufficient quantity of water is all that is

required to dissolve particulate magnesium sulfite deposited in a well. Thus, the particulate magnesium sulfite residues may automatically clean up (dissolve) in the water that may be produced along with hydrocarbons, or in the water that is pumped into an injection well to maintain reservoir pressure. Alternatively water or aqueous mixtures may be pumped into the well to dissolve the particulate magnesium sulfite residues.

Table 3

Temperature (°C)	Solubility of Magnesium Sulfite (% by weight)
25	0.65
42	0.94
50	0.84
85	0.62
98	0.61

Magnesium sulfite is also readily dissolved in aqueous solutions of acids such as acetic or hydrochloric acid to produce sulphur dioxide and the very soluble magnesium acetate or magnesium chloride respectively. Even partial acidification to magnesium bisulfite is effective in dissolving particulate magnesium sulfite in that magnesium bisulfite is very water-soluble. For example, magnesium bisulfite is available commercially as a 30% by weight aqueous solution from Sigma Aldrich.

Alternatively oxidising agents such as hydrogen peroxide will cause the dissolution of magnesium sulfite by converting it to the soluble magnesium sulfate (62.9g of magnesium sulfate dissolves in 100g of water at a temperature of 20°C).

Example 4 – Solubility of Lithium Phosphate

Lithium phosphate (Li₃PO₄) has a relatively low solubility in water (0.038g per 100g water at a temperature of 20°C). It is therefore less preferred for applications where water (produced water, injection water, or aqueous clean-up fluid) is used to dissolve the particulate residues.

However mild acidification with, for example, acetic acid or hydrochloric acid increases the solubility greatly. For instance, LiH₂PO₄ is very soluble in water at 55% by weight.

Example 5 -Water-based Wellbore Fluid Formulations

The following tests relate to water-based wellbore fluid formulations.

Fluid Formulations 1-4 (see Table 4 below) are suitable for use as drilling fluids, completion fluids such as a perforating pill or an under-reaming fluid, or workover fluids such as a kill fluid. Fluid Formulation 4 represents a typical prior art wellbore fluid that is currently used in the industry as, for example, a reservoir drilling fluid. This prior art fluid contains water-insoluble calcium carbonate bridging solids and is included for comparative purposes. The properties of Fluid Formulations 1-4 are given in Table 5 below.

Materials:

10 Powdered melamine, lithium carbonate, lithium phosphate and potassium chloride were all as supplied by Aldrich UK (laboratory chemical supplier). DuoVis™ (xanthan gum viscosifier), DualFlo™ (starch derivative Fluid loss Reducer) and Starcarb™ (calcium carbonate powder) were supplied by M-I Swaco llc.

15 The Fluid Formulations were tested in accordance with ISO 10416: 2002 (API RP 13I 7th edition). The Fluid Loss results are also presented in Table 5 below.

Table 4 – Fluid Formulations

Component (g)	Fluid 1	Fluid 2	Fluid 3	Fluid 4
Deionised water	330	330	330	330
DuoVis	1.0	1.0	1.0	1.0
DualFlo	4.0	4.0	4.0	4.0
Potassium Chloride	10	10	10	10
Melamine	16	--	--	--
Lithium carbonate	--	20	--	--
Lithium phosphate	--	--	16	--
Starcarb	--	--	--	25
Caustic soda	To pH 10.0	None (pH 11.0)	To pH 10.0	To pH 10.0

The varying gravimetric dose of the powders is to provide approximately the same loading by volume as the comparative Starcarb fluid (Fluid 4).

Table 5 – Properties of the Fluid Formulations

PROPERTIES	Fluid 1	Fluid 2	Fluid 3	Fluid 4
Plastic Viscosity cP	12	12	12	13
Yield Point lb/100ft ²	16	17	17	17
Gels(10sec/10min)	6/7	7/8	7/8	8/9
API Fluid Loss (mls) (determined according to ISO 10416)	6.8	6.5	9.8	6.4

After the API Fluid Loss test, the excess wellbore fluid was decanted from the cell employed in the test and was replaced with deionised water. The cell was resealed, pressurised to 100 psi with nitrogen, and the permeation rate through the filter cake was measured for 30 minutes.

A similar test was performed by repeating the API Fluid Loss test to provide new filter cakes from Fluids 2 and 4, followed by permeating deionised water that was pressurised with carbon dioxide to 100 psi.

A similar test was performed by repeating the API Fluid Loss test to provide a filter cake with Fluids 1 to 4, followed by permeating 5% Acetic acid for 30 minutes, or until the liquid in the cell was all passed through the filter cake.

The results of these additional tests are given in Table 6 below, which shows average permeation rates (mls/min).

Table 6 – Fluid Loss Test Results

Permeant fluid	Fluid 1 (melamine)	Fluid 2 (lithium carbonate)	Fluid 3 (lithium phosphate)	Fluid 4 (calcium carbonate)
Deionised water	7.4	0.52	0.32	0.18
Deionised water + 100 psi CO ₂ top pressure	—	1.8	—	0.37
5% acetic acid	10.6	24.0	9.0	1.56

The rate of flow of deionised water through the lithium phosphate and lithium carbonate-containing filter cakes is clearly improved compared to the benchmark calcium carbonate-containing filter cake (Fluid 4). The rates are still quite slow because the Duovis and DualFlo polymers concentrated in the filter cake reduce the flow rate and hence the dissolution of the sparingly water-soluble particles during the short (30 minutes) duration of the test.

The melamine-containing filter cake rapidly developed a much higher permeability to deionised water.

The presence of carbon dioxide increased the flow rate of water through the lithium carbonate containing filter cake more than three-fold.

The sparingly water-soluble solids of the present invention react to 5% acetic acid much more rapidly than particulate calcium carbonate (the industry norm).

Example 6 –Lost-Circulation Material and Water-Based Lost-Circulation Control Fluid

A base screen was removed from an API Fluid Loss cell and a bed of about one inch of 20-30 mesh sand was placed in the cell. This sand bed represents an extremely high permeability rock formation. Water was then poured through the bed to water-wet the sand.

A simple drilling fluid was mixed according to the following formulation:

Deionised water	330 g
Duovis™	1.5g
DualFlo™	3.5g
Barite	63g

5 A portion of this drilling fluid was gently poured on top of the sand bed. On pressurising the cell to a pressure of 50 psi, the complete drilling fluid immediately flowed through the sand bed in less than 3 seconds. This represents a lost circulation problem such as may be encountered in the field.

10 A slurry was obtained by mixing 100g of melamine (ex Aldrich) in 290g of water, and 100 mls of the slurry was poured into the cell. On pressurising to 50 psi the aqueous phase of the slurry immediately filtered through the sand bed. On opening the cell a white filter cake layer of melamine particles was observed on top of the sand bed. A portion of the "simple" drilling fluid was poured into the cell which was re-pressurised to 50 psi. A much slower stream of drilling fluid passed through the sand bed, but all (about 100 mls) 15 was still lost from the cell over a period of about 30 seconds. On opening the cell it was observed that the drilling fluid had all flowed through a small discontinuity in the bed of melamine particles.

Melamine particles were then added to the remaining drilling fluid at a dose of approximately 12.5 lbs/bbl. On placing this fluid into the cell and re-pressurising to 50 psi, 20 the drilling fluid started to flow through the sand pack but slowed to a virtual stop in about 5-10 seconds. The pressure was increased to 100 psi. The effluent rate from the cell was then stabilised at a normal, slow filtration rate.

This Experiment illustrates the use of sparingly water-soluble solid particles as Lost Circulation Material, either in a specially designed fluid pumped into place in a wellbore to 25 control fluid losses, or as an additive to a wellbore fluid such as a drilling fluid. The addition of the sparingly water-soluble material to drilling fluids may be used to stop fluid losses, but it may also be used pre-emptively to avoid the occurrence of such losses.

The particle size of the melamine obtained from Aldrich was measured by dry screening using a vibratory screen shaker. Results in weight percent are as follows:

30 > 500 microns	0.22%
<500 > 300 microns	1.60%
<300 > 150 microns	74.0%

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<150 microns 24.2%

Such sized particles are well-suited to bridging the pores in extreme permeability sand formations, and also for accumulating in fractures of width less than about 1mm by rapid filtration of a high-solids slurry of the particles which is flowing into the fracture.

5 Example 7 - Oil-based drilling fluid containing melamine particles, and treatment of the filter cake therefrom to establish the flow-through of seawater.

10 An oil-based drilling fluid was prepared, based upon the FazePro™ product of M-I Swaco LLC (see Table 7 below). The invert emulsion of this oil-based drilling fluid is designed to de-stabilise upon the application of an acid thereby enabling improved clean-up compared to conventional oil-based drilling fluids. The addition of melamine powder provides bridging and filter cake material to seal the sand-face of permeable formations. After drilling, the filter cake may be treated with an acid solution to disrupt the emulsion within the filter cake in order to increase the cake permeability. The acid also starts to dissolve some of the particulate melamine. In the case of a seawater injection well, the acid can be followed by injection of seawater which continues to dissolve the remaining melamine until the residues are completely removed.

20 This Example shows that an oil-based drilling fluid with suitable properties for drilling purposes can be formulated with melamine bridging solids. Subsequently the filter cake is treated with an acid solution followed by the flow of injected seawater, both of which fluids are active in removing the seal that was provided by the filter cake.

The oil-based drilling formulation was mixed using a Silverson L4RT Mixer fitted with a high shear head. The mixing times per component are shown in Table 7 below. The mixer speed was at about 6000 RPM. The temperature was monitored throughout and maintained at 150°F or less by the use of a cooling water bath.

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Table 7

Product	Concentration (ppb)	Order	Time
DF-1 BASE OIL™ (a)	134.5	1=	
FAZEMUL™ (b)	12.0	1=	5 mins
FAZEWET™ (b)	6.0	1=	
TRUVIS™ (b)	1.0	4	2 mins
Lime	4.0	5	2 mins
ECOTROL RD™ (b)	0.5	6	16 mins
1.35 ppg CaBr2 Brine	164.9	7	20 mins
Melamine powder	30	8	15 mins

(a) ex TotalFinaElf UK Limited

(b) ex Trademark M-I Swaco llc

- 5 After mixing, the oil-based drilling fluid was hot rolled at a temperature of 150°F for 16 hours to simulate heating downhole in the field. The viscous properties and the High Temperature/ High Pressure Fluid Loss (HTHP FL) were then measured and are given in Table 8 below.

Table 8

Rheological Properties at 120°F/ 48.8°C	
Plastic Viscosity (cP)	15
Yield Point (lb/100ft ²)	14
10 sec gel (lb/100ft ²)	7
10 min gel (lb/100ft ²)	7
HTHP Fluid Loss (mls) At 200°F and 500 psi	3.1

The results presented in Table 8 show that satisfactory rheological and filtration performance was obtained.

After the HTHP Fluid loss test the excess drilling fluid was drained from the cell and replaced by a solution of 5% glacial acetic acid in kerosene. The cell was closed and heated to a temperature of 45°C. The acid solution was then pressurised to 100 psi so that the solution permeated through the filter cake, the flow-through weight being measured vs. time, as recorded below in Table 9.

Table 9 - Permeation of Acid Solution through Filter cake

Time (minutes)	Permeated acid solution (grams)
1	7.604
5	15.878
10	25.14
15	39.548
15 minutes 45 seconds	Gas breakthrough

The cell was then refilled with seawater and heated to a temperature of 45 °C. On pressurising to 100 psi, the seawater rapidly passed through the filter cake (66.5g in 17 seconds). Examination of the filter cake showed that irregular areas had been etched away leaving some white melamine residues. The filter cake residues on the filter paper were placed in 500 mls of seawater and held at a temperature of 45°C for 72 hours. After this time no visible melamine particles remained.

This is very advantageous for seawater injection wells where seawater injection is usually continued for years, leaving little chance that any residual melamine filter cake remains undissolved. Thus the injectivity of the seawater is maximised.

Claims

1. A wellbore fluid comprising a base fluid and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine
5 (2,4,5-triamino-1,3,5-triazine), lithium carbonate, lithium phosphate (Li_3PO_4), and magnesium sulfite.
2. A wellbore fluid as claimed in Claim 1 wherein the base fluid is present in the wellbore fluid in an amount in the range of from about 30 to 99% by weight of the fluid.
3. A wellbore fluid as claimed in Claims 1 or 2 wherein the sparingly water-soluble
10 particulate bridging agent is present in the wellbore fluid in an amount in the range of 1 to 70% by weight.
4. A wellbore fluid as claimed in any one of the preceding claims wherein the wellbore fluid is a drilling, servicing or completion fluid and the sparingly water-soluble
15 particulate bridging agent has a particle size distribution in the range of from about 0.1 micron to 600 microns.
5. A wellbore fluid as claimed in any one of the preceding claims wherein the wellbore fluid is an aqueous based fluid and the wellbore fluid comprises at least one
20 additional additive selected from the group consisting of viscosifiers, weighting agents, density increasing water-soluble salts, filtration or fluid loss control agents, pH control agents, clay or shale hydration inhibitors, bactericides, surfactants, solid and liquid
lubricants, gas-hydrate inhibitors, corrosion inhibitors, defoamers, scale inhibitors, emulsified hydrophobic liquids such as oils, acid gas-scavengers (such as hydrogen sulfide
scavengers), thinners (such as lignosulfonates), and demulsifiers.
6. A wellbore fluid as claimed in Claim 5 wherein the aqueous based wellbore fluid
25 comprises a fluid loss control agent selected from the group consisting of water-soluble polymers, lignites, modified lignites, and cross-linked lignosulphonates
7. A wellbore fluid as claimed in any one of Claims 1 to 4 wherein the wellbore fluid is an oil based fluid comprising at least one additional additive selected from the group
30 consisting of viscosifiers, surfactants (for forming stable water-in-oil emulsions and to oil-wet the surface of mineral weighting agents), fluid loss control additives, lubricants (solid and liquid), and acid gas scavengers (for example, hydrogen sulfide scavengers).
8. A method of forming a removable filter cake on the walls of a wellbore that

penetrates a porous and permeable rock formation comprising the steps of:

(a) placing a wellbore fluid in the wellbore wherein the wellbore fluid comprises a base fluid and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate

5 (Li₃PO₄), and magnesium sulfite; and

(b) permitting the particulate bridging agent to deposit from the wellbore fluid onto and/or into the walls of the wellbore thereby forming the filter cake, whereby fluid loss to the formation through the removable filter cake is reduced.

9. A method as claimed in Claim 8 wherein a fluid loss control additive is included in
10 the wellbore fluid.

10. A method as claimed in Claims 8 or 9 wherein the sparingly water-soluble particulate bridging agent is removed by putting the well into production.

11. A method as claimed in Claims 8 or 9 wherein the sparingly water-soluble particulate bridging agent is removed from the wellbore of an injection well by dissolution
15 in water that is injected into the injection well.

12. A method as claimed in Claims 8 or 9 wherein the sparingly water-soluble particulate bridging agent is removed by (a) placing a clean-up fluid downhole and (b) leaving the clean-up fluid to soak across the interval of the wellbore where it is desired to remove the filter cake for a sufficient period of time to either completely dissolve the
20 bridging agent or to solubilise the bridging agent to the extent that the particles are sufficiently reduced in size to permit their removal from the formation.

13. A method as claimed in Claim 12 wherein the clean-up fluid is an aqueous fluid that is under-saturated with respect to the bridging agent or is an aqueous solution of an acid or a precursor of a weak acid.

25 14. A method as claimed in Claim 12 wherein the sparingly water-soluble particulate bridging agent is comprised of magnesium sulfite, and the clean-up fluid comprises an aqueous solution of an oxidizing agent that is capable of converting magnesium sulfite to water-soluble magnesium sulfate.

15. A method as claimed in Claims 12 or 13 wherein the sparingly water-soluble
30 particulate bridging agent is comprised of melamine, and the removable filter cake is removed from the walls of a wellbore by placing an aqueous wash fluid downhole and leaving the wash fluid to soak in the interval of the wellbore where it is desired to remove

the filter cake for a sufficient period of time for the wash fluid to heat up to a temperature of at least 60°C and leaving the heated wash fluid soaking in the interval until either the particulate bridging agent is completely dissolved in the wash fluid or is solubilised to the extent that the particles are sufficiently reduced in size to permit their removal from the formation.

16. A method of drilling a wellbore through a porous and permeable rock formation using a drilling fluid comprising a base fluid, a fluid loss control additive, and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate (Li_3PO_4), and magnesium sulfite wherein the pressure of the drilling fluid in the wellbore is maintained at above the pressure in the porous and permeable rock formation such that a filter cake deposits on and/or in the walls of the wellbore and reduces fluid loss from the drilling fluid to the rock formation.

17. A drilling method as claimed in Claim 16 wherein the drilling fluid is as defined in any one of claims 4 to 7.

18. A method of controlling loss of fluid from a wellbore into a porous and permeable rock formation through a high conductivity conduit that extends from the wellbore into the rock formation comprising the steps of:

(a) placing a lost circulation fluid in the wellbore wherein the lost circulation fluid comprises a slurry of particulate lost circulation material (LCM) in a base fluid wherein the LCM is suspended in the base fluid in an amount of at least 5 pounds per barrel, preferably at least 10 pounds per barrel, more preferably, at least 20 pounds per barrel, and most preferably at least 30 pounds per barrel, and is comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate (Li_3PO_4), and magnesium sulfite; and

(b) permitting the LCM to accumulate in the high conductivity conduit thereby forming a removable low-permeability plug that bridges the conduit whereby fluid loss to the formation through the conduit is reduced.

19. A method as claimed in Claim 18 wherein a fluid loss control agent is present in the slurry thereby assisting in sealing the high conductivity conduit.

20. A method as claimed in Claims 18 or 19 wherein the base fluid is a drilling fluid.

21. A method of controlling loss of fluid from a completion fluid into a porous and permeable rock formation penetrated by a wellbore by:
- (a) placing a completion fluid in the wellbore wherein the completion fluid comprises a base fluid, a fluid loss control additive, and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate, and magnesium sulfite; and
 - (b) maintaining the pressure of the completion fluid in the wellbore at above the pore-pressure of the rock formation such that a filter cake deposits on or in the walls of the wellbore.
22. A method as claimed in Claim 21 wherein the completion fluid is as defined in any one of claims 4 to 7.
23. A method as claimed in Claims 21 or 22 wherein the completion fluid additionally contains a polymeric viscosifier(s) such as xanthan gum, hydroxyethylcellulose, welan gum (for example, Biozan™; ex Kelco) or diutan gum (for example, Geovis XT™; ex Kelco).
24. A method of controlling loss of fluid from a workover fluid to an interval of a wellbore that lies across a porous and permeable rock formation wherein the method comprises the steps of:
- (a) pumping a sufficient volume of a first workover fluid to fill the interval of the wellbore that lies across the porous and permeable rock formation wherein the first workover fluid comprises a base fluid, a fluid loss control additive, and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate, and magnesium sulfite such that a removable filter cake deposits from the first workover fluid in said interval of the wellbore onto the walls of the wellbore and into any cracks, fractures or fissures therein;
 - (b) pumping a second workover fluid into the wellbore wherein the second workover fluid is substantially saturated with the sparingly water-soluble material and is of sufficient density to at least counterbalance the pressure of the porous and permeable rock formation; and wherein the filter cake deposited in step (a) reduces fluid loss from the workover fluids to the porous and permeable rock formation.
25. A method of fracturing a porous and permeable rock formation comprising:

injecting a fracturing fluid into an interval of a wellbore across the rock formation that is to be fractured wherein the fracturing fluid comprises a base fluid, proppant, a viscosifier, and a particulate leak-off control agent comprised of a sparingly water-soluble material

selected from the group consisting of melamine, lithium carbonate, lithium phosphate, and magnesium sulfite; and

maintaining the pressure of the fracturing fluid in the interval of the wellbore across the rock formation at above the fracture pressure of the formation whereby proppant enters and props open the fractures that are formed in the wellbore wall and the particulate leak-off control agent seals exposed pore throats on the walls of the fracture.

26. A method of diverting non-acidic treatment fluids away from high permeability rock formations or high conductivity conduits and into lower permeability and/or partially plugged rock formations or lower conductivity conduits using a treatment fluid comprising a non-acidic fluid and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate (Li_3PO_4), and magnesium sulfite.

27. A method as claimed in Claim 26 wherein the non-acidic fluid is an aromatic solvent and the treatment fluid is pumped into a hydrocarbon production wellbore such that a filter cake forms on and/or in a high permeability rock formation and/or the particulate bridging agent enters and seals high conductivity conduits (or flow channels) in the walls of the wellbore thereby limiting the loss of aromatic solvent from the wellbore thereby diverting the aromatic solvent towards low conductivity conduits (or flow channels) that are damaged by asphaltene and/or wax deposits, thereby improving the dissolution of the deposits by the aromatic solvent.

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2007/000055

A. CLASSIFICATION OF SUBJECT MATTER

INV. C09K8/03 C09K8/16 C09K8/26 C09K8/32 C09K8/502
C09K8/504 C09K8/52 C09K8/66

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)

C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4 369 843 A (MONDSHINE THOMAS C) 25 January 1983 (1983-01-25) column 1, line 18 - line 65 column 2, line 13 - line 35 column 3, line 21 - line 42	1-6, 16-23
X	column 3, line 65 - column 4, line 39 column 5, line 50 - line 62; claims 1,3	8-11
Y	EP 0 845 520 A1 (TEXAS UNITED CHEMICAL CORP [US]) 3 June 1998 (1998-06-03) page 2, line 5 - line 18 page 3, line 5 - line 13 page 3, line 25 - page 4, line 28 page 4, line 58 - page 5, line 19 page 5, line 53 - line 58 page 6, line 27 - line 32	1-6, 8, 9, 16-23

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☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

* Special categories of cited documents:

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
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			US 6300286 B1	09-10-2001
			US 6391830 B1	21-05-2002
US 2006009364	A1	12-01-2006	NONE	
WO 2006054058	A	26-05-2006	US 2006105918 A1	18-05-2006

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2007/000055

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 616 660 A1 (BAKER HUGHES INC. [US]) 28 September 1994 (1994-09-28) paragraph [0001] - paragraph [0002]	1-6, 8, 9, 18, 19
X	paragraph [0017] - paragraph [0018] paragraph [0019] - paragraph [0026] paragraph [0030]	21-26
Y	EP 1 074 598 A1 (TEXAS UNITED CHEMICAL CORP [US]) 7 February 2001 (2001-02-07) paragraph [0002] - paragraph [0003] paragraph [0011] paragraph [0029] - paragraph [0032] paragraph [0038] - paragraph [0040]	1-6, 8, 9, 16-23
Y	US 2006/009364 A1 (DOBSON JAMES W JR [US] ET AL) 12 January 2006 (2006-01-12) paragraph [0002] paragraph [0033] - paragraph [0037]	1, 4-9, 12, 13, 16-23, 25
Y, P	WO 2006/054058 A (HALLIBURTON ENERGY SERV INC [US]; WAIN CHRISTOPHER PAUL [GB]; MUNOZ TR) 26 May 2006 (2006-05-26) page 2, line 5 - page 3, line 16 page 3, line 27 - page 6, line 27 page 10, line 19 - page 11, line 9	1-3, 5-9, 12, 13, 16-23, 25

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2007/000055

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4369843	A	25-01-1983	NONE
EP 0845520	A1	03-06-1998	AU 723115 B2 17-08-2000
		AU 3988297 A	04-06-1998
		CA 2218214 A1	02-06-1998
		DE 69712855 D1	04-07-2002
		DE 69712855 T2	28-11-2002
		DK 845520 T3	09-09-2002
		NO 975494 A	03-06-1998
EP 0616660	A1	28-09-1994	AU 5353394 A 09-05-1994
		NO 933774 A	22-04-1994
		WO 9409254 A1	28-04-1994
		US 5504062 A	02-04-1996
		US 5325921 A	05-07-1994
EP 1074598	A1	07-02-2001	AU 771831 B2 01-04-2004
		AU 4894800 A	08-02-2001
		BR 0012955 A	18-06-2002
		CA 2314806 A1	05-02-2001
		DE 60018857 D1	28-04-2005
		DE 60018857 T2	06-04-2006
		ID 26742 A	08-02-2001
		NO 20003972 A	06-02-2001
		WO 0110976 A1	15-02-2001
		US 6300286 B1	09-10-2001
		US 6391830 B1	21-05-2002
US 2006009364	A1	12-01-2006	NONE
WO 2006054058	A	26-05-2006	US 2006105918 A1 18-05-2006

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference
(if desired) (12 characters maximum) BPX 10314

Box No. I TITLE OF INVENTION
WELLBORE FLUID

Box No. II APPLICANT

☐ This person is also inventor

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

BP EXPLORATION OPERATING COMPANY LIMITED
Chertsey Road
Sunbury-on-Thames
Middlesex, TW16 7BP
United Kingdom

Telephone No.

Facsimile No.

Teleprinter No.

Applicant's registration No. with the Office

State (that is, country) of nationality:
GB

State (that is, country) of residence:
GB

This person is applicant
for the purposes of:

☐ all designated
States

☒ all designated States except
the United States of America

☐ the United States
of America only

☐ the States indicated in
the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

DUNCUM, Simon Neil
9 Wilstrode Avenue
Binfield
Bracknell, Berkshire, RG42 4UW
United Kingdom

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box is
marked, do not fill in below.)

Applicant's registration No. with the Office

State (that is, country) of nationality:
GB

State (that is, country) of residence:
GB

This person is applicant
for the purposes of:

☐ all designated
States

☐ all designated States except
the United States of America

☒ the United States
of America only

☐ the States indicated in
the Supplemental Box

☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf
of the applicant(s) before the competent International Authorities as:

☒ agent

☐ common
representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

COLLINS, Frances Mary
BP INTERNATIONAL LIMITED
Patents & Agreements
Chertsey Road, Sunbury-on-Thames,
Middlesex, TW16 7LN
United Kingdom

Telephone No.

01932 763206

Facsimile No.

01932 762388

Teleprinter No.

Agent's registration No. with the Office

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

SAWDON, Christopher Alan
6 Southview Road
Par
Cornwall, PL24 2HJ
United Kingdom

This person is:

- ☐ applicant only
- ☒ applicant and inventor
- ☐ inventor only (If this check-box is marked, do not fill in below.)

Applicant's registration No. with the Office

State (that is, country) of nationality:

GB

State (that is, country) of residence:

GB

This person is applicant for the purposes of:

☐ all designated States☐ all designated States except the United States of America☒ the United States of America only☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
- ☐ applicant and inventor
- ☐ inventor only (If this check-box is marked, do not fill in below.)

Applicant's registration No. with the Office

State (that is, country) of nationality:

State (that is, country) of residence:

This person is applicant for the purposes of:

☐ all designated States☐ all designated States except the United States of America☐ the United States of America only☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
- ☐ applicant and inventor
- ☐ inventor only (If this check-box is marked, do not fill in below.)

Applicant's registration No. with the Office

State (that is, country) of nationality:

State (that is, country) of residence:

This person is applicant for the purposes of:

☐ all designated States☐ all designated States except the United States of America☐ the United States of America only☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
- ☐ applicant and inventor
- ☐ inventor only (If this check-box is marked, do not fill in below.)

Applicant's registration No. with the Office

State (that is, country) of nationality:

State (that is, country) of residence:

This person is applicant for the purposes of:

☐ all designated States☐ all designated States except the United States of America☐ the United States of America only☐ the States indicated in the Supplemental Box☐ Further applicants and/or (further) inventors are indicated on another continuation sheet.

Box No. V DESIGNATIONS

The filing of this request constitutes under Rule 4.9(a), the designation of all Contracting States bound by the PCT on the international filing date, for the grant of every kind of protection available and, where applicable, for the grant of both regional and national patents. However,

- ☐ DE Germany is not designated for any kind of national protection
☐ JP Japan is not designated for any kind of national protection
☐ KR Republic of Korea is not designated for any kind of national protection
☐ RU Russian Federation is not designated for any kind of national protection

(The check-boxes above may only be used to exclude (irrevocably) the designations concerned if, at the time of filing, the international application contains in Box No. VI a priority claim to an earlier national application filed in the particular State concerned, in order to avoid the ceasing of the effect, under the national law, of this earlier national application. See the Notes to Box No. V as to the consequences of such national law provisions in these States).

Box No. VI PRIORITY CLAIM

The priority of the following earlier application(s) is hereby claimed:

Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country or Member of WTO	regional application:* regional Office	international application: receiving Office
item (1) 31 January 2006	0601961.6	GB		
item (2)				
item (3)				

☐ Further priority claims are indicated in the Supplemental Box.

The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of this international application is the receiving Office) identified above as:

☐ all items ☒ item (1) ☐ item (2) ☐ item (3) ☐ other, see Supplemental Box

* Where the earlier application is an ARIPO application, indicate at least one country party to the Paris Convention for the Protection of Industrial Property or one Member of the World Trade Organization for which that earlier application was filed (Rule 4.10(b)(ii)):

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):

ISA / EP

Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority):

Date (day/month/year) Number Country (or regional Office)

Box No. VIII DECLARATIONS

The following declarations are contained in Boxes Nos. VIII (i) to (v) (mark the applicable check-boxes below and indicate in the right column the number of each type of declaration):

Number of
declarations

- | | | | |
|---|--|---|---|
| ☐ Box No. VIII (i) | Declaration as to the identity of the inventor | : | |
| ☒ Box No. VIII (ii) | Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent | : | 1 |
| ☐ Box No. VIII (iii) | Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application | : | |
| ☒ Box No. VIII (iv) | Declaration of inventorship (only for the purposes of the designation of the United States of America) | : | 1 |
| ☐ Box No. VIII (v) | Declaration as to non-prejudicial disclosures or exceptions to lack of novelty | : | |

Box No. VIII (ii) DECLARATION: ENTITLEMENT TO APPLY FOR AND BE GRANTED A PATENT

The declaration must conform to the standardized wording provided for in Section 212; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (ii). If this Box is not used, this sheet should not be included in the request.

Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent (Rules 4.17(ii) and 51bis.1(a)(ii)), in a case where the declaration under Rule 4.17(iv) is not appropriate:

In relation to this International Application, BP EXPLORATION OPERATING COMPANY LIMITED is entitled to apply for and be granted a patent by virtue of the following:

BP EXPLORATION OPERATING COMPANY LIMITED is entitled as employer of the inventor, DUNCUM, Simon Neil

An Agreement between SAWDON, Christopher Alan and BP EXPLORATION OPERATING COMPANY LIMITED

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (ii)".

Box No. VIII (iv) DECLARATION: INVENTORSHIP (only for the purposes of the designation of the United States of America)
The declaration must conform to the following standardized wording provided for in Section 214; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (iv). If this Box is not used, this sheet should not be included in the request.

**Declaration of inventorship (Rules 4.17(iv) and 51bis.1(a)(iv))
for the purposes of the designation of the United States of America:**

I hereby declare that I believe I am the original, first and sole (if only one inventor is listed below) or joint (if more than one inventor is listed below) inventor of the subject matter which is claimed and for which a patent is sought.

This declaration is directed to the international application of which it forms a part (if filing declaration with application).

This declaration is directed to international application No. PCT/GB2007/000055 (if furnishing declaration pursuant to Rule 26ter).

I hereby declare that my residence, mailing address, and citizenship are as stated next to my name.

I hereby state that I have reviewed and understand the contents of the above-identified international application, including the claims of said application. I have identified in the request of said application, in compliance with PCT Rule 4.10, any claim to foreign priority, and I have identified below, under the heading "Prior Applications," by application number, country or Member of the World Trade Organization, day, month and year of filing, any application for a patent or inventor's certificate filed in a country other than the United States of America, including any PCT international application designating at least one country other than the United States of America, having a filing date before that of the application on which foreign priority is claimed.

Prior Applications:

I hereby acknowledge the duty to disclose information that is known by me to be material to patentability as defined by 37 C.F.R. § 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the PCT international filing date of the continuation-in-part application.

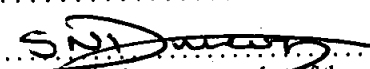
I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name: DUNCUM, Simon Neil

Residence: Bracknell, Berkshire, United Kingdom
(city and either US state, if applicable, or country)

Mailing Address: 9 Wilstrobe Avenue, Binfield, Bracknell, Berkshire, RG42.4UW, United Kingdom.

Citizenship: British

Inventor's Signature: 
(The signature must be that of the inventor, not that of the agent)

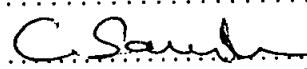
Date: 30th JANUARY 2007

Name: SAWDON, Christopher Alan

Residence: Par, Cornwall, United Kingdom
(city and either US state, if applicable, or country)

Mailing Address: 6 Southview Road, Par, Cornwall, PL24 2HJ, United Kingdom.

Citizenship: British

Inventor's Signature: 
(The signature must be that of the inventor, not that of the agent)

Date: 23rd JANUARY 2007

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (iv)".

Box No. IX CHECK LIST; LANGUAGE OF FILING

This international application contains: (a) on paper, the following number of sheets: request (including declaration sheets) : 6 description (excluding sequence listing and/or tables related thereto) : 28 claims : 5 abstract : 1 drawings : _____ Sub-total number of sheets : 40 sequence listing : _____ tables related thereto : _____ <i>(for both, actual number of sheets if filed on paper, whether or not also filed in electronic form; see (c) below)</i> Total number of sheets : 40 (b) ☐ only in electronic form (Section 801(a)(i)) (i) ☐ sequence listing (ii) ☐ tables related thereto (c) ☐ also in electronic form (Section 801(a)(ii)) (i) ☐ sequence listing (ii) ☐ tables related thereto Type and number of carriers (diskette, CD-ROM, CD-R or other) on which are contained the ☐ sequence listing: _____ ☐ tables related thereto: _____ <i>(additional copies to be indicated under items 9(ii) and/or 10(ii), in right column)</i>	This international application is accompanied by the following item(s) (mark the applicable check-boxes below and indicate in right column the number of each item): <table style="width:100%;"> <tr> <td>1. ☒ fee calculation sheet</td> <td align="right">1</td> </tr> <tr> <td>2. ☒ original separate power of attorney</td> <td align="right">1</td> </tr> <tr> <td>3. ☐ original general power of attorney</td> <td align="right"></td> </tr> <tr> <td>4. ☒ copy of general power of attorney; reference number, if any: _____</td> <td align="right">1</td> </tr> <tr> <td>5. ☐ statement explaining lack of signature</td> <td align="right"></td> </tr> <tr> <td>6. ☐ priority document(s) identified in Box No. VI as item(s): _____</td> <td align="right"></td> </tr> <tr> <td>7. ☐ translation of international application into (language): _____</td> <td align="right"></td> </tr> <tr> <td>8. ☐ separate indications concerning deposited microorganism or other biological material</td> <td align="right"></td> </tr> <tr> <td>9. ☐ sequence listing in electronic form (indicate type and number of carriers)</td> <td align="right"></td> </tr> <tr> <td> (i) ☐ copy submitted for the purposes of international search under Rule 13ter only (and not as part of the international application)</td> <td align="right"></td> </tr> <tr> <td> (ii) ☐ (only where check-box (b)(i) or (c)(i) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Rule 13ter</td> <td align="right"></td> </tr> <tr> <td> (iii) ☐ together with relevant statement as to the identity of the copy or copies with the sequence listing mentioned in left column</td> <td align="right"></td> </tr> <tr> <td>10. ☐ tables in electronic form related to sequence listing (indicate type and number of carriers)</td> <td align="right"></td> </tr> <tr> <td> (i) ☐ copy submitted for the purposes of international search under Section 802(b-quater) only (and not as part of the international application)</td> <td align="right"></td> </tr> <tr> <td> (ii) ☐ (only where check-box (b)(ii) or (c)(ii) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Section 802(b-quater)</td> <td align="right"></td> </tr> <tr> <td> (iii) ☐ together with relevant statement as to the identity of the copy or copies with the tables mentioned in left column</td> <td align="right"></td> </tr> <tr> <td>11. ☒ other (specify): Form 23/77</td> <td align="right">1</td> </tr> </table>	1. ☒ fee calculation sheet	1	2. ☒ original separate power of attorney	1	3. ☐ original general power of attorney		4. ☒ copy of general power of attorney; reference number, if any: _____	1	5. ☐ statement explaining lack of signature		6. ☐ priority document(s) identified in Box No. VI as item(s): _____		7. ☐ translation of international application into (language): _____		8. ☐ separate indications concerning deposited microorganism or other biological material		9. ☐ sequence listing in electronic form (indicate type and number of carriers)		(i) ☐ copy submitted for the purposes of international search under Rule 13ter only (and not as part of the international application)		(ii) ☐ (only where check-box (b)(i) or (c)(i) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Rule 13ter		(iii) ☐ together with relevant statement as to the identity of the copy or copies with the sequence listing mentioned in left column		10. ☐ tables in electronic form related to sequence listing (indicate type and number of carriers)		(i) ☐ copy submitted for the purposes of international search under Section 802(b-quater) only (and not as part of the international application)		(ii) ☐ (only where check-box (b)(ii) or (c)(ii) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Section 802(b-quater)		(iii) ☐ together with relevant statement as to the identity of the copy or copies with the tables mentioned in left column		11. ☒ other (specify): Form 23/77	1
1. ☒ fee calculation sheet	1																																		
2. ☒ original separate power of attorney	1																																		
3. ☐ original general power of attorney																																			
4. ☒ copy of general power of attorney; reference number, if any: _____	1																																		
5. ☐ statement explaining lack of signature																																			
6. ☐ priority document(s) identified in Box No. VI as item(s): _____																																			
7. ☐ translation of international application into (language): _____																																			
8. ☐ separate indications concerning deposited microorganism or other biological material																																			
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(iii) ☐ together with relevant statement as to the identity of the copy or copies with the tables mentioned in left column																																			
11. ☒ other (specify): Form 23/77	1																																		
Figure of the drawings which should accompany the abstract:	Language of filing of the international application: English																																		

Box No. X SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

for BP EXPLORATION OPERATING COMPANY LIMITED

F. M. Collins
COLLINS, Frances Mary

For receiving Office use only	
1. Date of actual receipt of the purported international application: _____ 3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application: _____ 4. Date of timely receipt of the required corrections under PCT Article 11(2): _____ 5. International Searching Authority (if two or more are competent): ISA /	2. Drawings: ☐ received: ☐ not received: 6. ☐ Transmittal of search copy delayed until search fee is paid

For International Bureau use only

Date of receipt of the record copy by the International Bureau: _____

PATENT COOPERATION TREATY

AMP
Fmc

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

To: BP INTERNATIONAL LIMITED Attn. Collins, Frances Mary Patents & Agreements Chertsey Road, Sunbury-on-Thames Middlesex, TW16 7LN GRANDE BRETAGNE	Received in PAT - 5 APR 2007	NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL SEARCH REPORT AND THE WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY, OR THE DECLARATION
(PCT Rule 44.1)		
Applicant's or agent's file reference BPX 10314		Date of mailing (day/month/year) 05/04/2007
International application No. PCT/GB2007/000055		International filing date (day/month/year) 10/01/2007
Applicant BP EXPLORATION OPERATING COMPANY LIMITED		

1. ☒ The applicant is hereby notified that the international search report and the written opinion of the International Searching Authority have been established and are transmitted herewith.
Filing of amendments and statement under Article 19:
 The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46):
When? The time limit for filing such amendments is normally two months from the date of transmittal of the International Search Report.
Where? Directly to the International Bureau of WIPO, 34 chemin des Colombettes
 1211 Geneva 20, Switzerland, Facsimile No.: (41-22) 338.82.70
For more detailed instructions, see the notes on the accompanying sheet.
2. ☐ The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect and the written opinion of the International Searching Authority are transmitted herewith.
3. ☐ **With regard to the protest** against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:
 - ☐ the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.
 - ☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.
4. **Reminders**
 Shortly after the expiration of **18 months** from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in Rules 90bis.1 and 90bis.3, respectively, before the completion of the technical preparations for international publication.
 The applicant may submit comments on an informal basis on the written opinion of the International Searching Authority to the International Bureau. The International Bureau will send a copy of such comments to all designated Offices unless an international preliminary examination report has been or is to be established. These comments would also be made available to the public but not before the expiration of 30 months from the priority date.
 Within **19 months** from the priority date, but only in respect of some designated Offices, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until **30 months** from the priority date (in some Offices even later); otherwise, the applicant must, **within 20 months** from the priority date, perform the prescribed acts for entry into the national phase before those designated Offices.
 In respect of other designated Offices, the time limit of **30 months** (or later) will apply even if no demand is filed within 19 months.
 See the Annex to Form PCT/IB/301 and, for details about the applicable time limits, Office by Office, see the *PCT Applicant's Guide*, Volume II, National Chapters and the WIPO Internet site.

Name and mailing address of the International Searching Authority European Patent Office, P.B.-5818 Patentaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Alex Schmidt
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NOTES TO FORM PCT/ISA/220

These Notes are intended to give the basic instructions concerning the filing of amendments under article 19. The Notes are based on the requirements of the Patent Cooperation Treaty, the Regulations and the Administrative Instructions under that Treaty. In case of discrepancy between these Notes and those requirements, the latter are applicable. For more detailed information, see also the *PCT Applicant's Guide*, a publication of WIPO.

In these Notes, "Article", "Rule", and "Section" refer to the provisions of the PCT, the PCT Regulations and the PCT Administrative Instructions, respectively.

INSTRUCTIONS CONCERNING AMENDMENTS UNDER ARTICLE 19

The applicant has, after having received the international search report and the written opinion of the International Searching Authority, one opportunity to amend the claims of the international application. It should however be emphasized that, since all parts of the international application (claims, description and drawings) may be amended during the international preliminary examination procedure, there is usually no need to file amendments of the claims under Article 19 except where, e.g. the applicant wants the latter to be published for the purposes of provisional protection or has another reason for amending the claims before international publication. Furthermore, it should be emphasized that provisional protection is available in some States only (see *PCT Applicant's Guide*, Volume I/A, Annexes B1 and B2).

The attention of the applicant is drawn to the fact that amendments to the claims under Article 19 are not allowed where the International Searching Authority has declared, under Article 17(2), that no international search report would be established (see *PCT Applicant's Guide*, Volume I/A, paragraph 296).

What parts of the international application may be amended?

Under Article 19, only the claims may be amended.

During the international phase, the claims may also be amended (or further amended) under Article 34 before the International Preliminary Examining Authority. The description and drawings may only be amended under Article 34 before the International Examining Authority.

Upon entry into the national phase, all parts of the international application may be amended under Article 28 or, where applicable, Article 41.

When?

Within 2 months from the date of transmittal of the international search report or 16 months from the priority date, whichever time limit expires later. It should be noted, however, that the amendments will be considered as having been received on time if they are received by the International Bureau after the expiration of the applicable time limit but before the completion of the technical preparations for international publication (Rule 46.1).

Where not to file the amendments?

The amendments may only be filed with the International Bureau and not with the receiving Office or the International Searching Authority (Rule 46.2).

Where a demand for international preliminary examination has been/is filed, see below.

How?

Either by cancelling one or more entire claims, by adding one or more new claims or by amending the text of one or more of the claims as filed.

A replacement sheet must be submitted for each sheet of the claims which, on account of an amendment or amendments, differs from the sheet originally filed.

All the claims appearing on a replacement sheet must be numbered in Arabic numerals. Where a claim is cancelled, no renumbering of the other claims is required. In all cases where claims are renumbered, they must be renumbered consecutively (Section 205(b)).

The amendments must be made in the language in which the international application is to be published.

What documents must/may accompany the amendments?

Letter (Section 205(b)):

The amendments must be submitted with a letter.

The letter will not be published with the international application and the amended claims. It should not be confused with the "Statement under Article 19(1)" (see below, under "Statement under Article 19(1)").

The letter must be in English or French, at the choice of the applicant. However, if the language of the international application is English, the letter must be in English; if the language of the international application is French, the letter must be in French.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference BPX 10314	FOR FURTHER ACTION see Form PCT/ISA/220 as well as, where applicable, Item 5 below.	
International application No. PCT/GB2007/000055	International filing date (day/month/year) 10/01/2007	(Earliest) Priority Date (day/month/year)
Applicant BP EXPLORATION OPERATING COMPANY LIMITED		

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 4 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

a. With regard to the language, the international search was carried out on the basis of:

- ☒ the international application in the language in which it was filed
☐ a translation of the international application into _____, which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1(b))

b. ☐ With regard to any nucleotide and/or amino acid sequence disclosed in the international application, see Box No. I.

2. ☐ Certain claims were found unsearchable (See Box No. II)

3. ☐ Unity of invention is lacking (see Box No. III)

4. With regard to the title,

- ☐ the text is approved as submitted by the applicant
☒ the text has been established by this Authority to read as follows:

WELLBORE FLUID COMPRISING A BASE FLUID AND A PARTICULATE BRIDGING AGENT

5. With regard to the abstract,

- ☒ the text is approved as submitted by the applicant
☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority

6. With regard to the drawings,

- a. the figure of the drawings to be published with the abstract is Figure No. _____
☐ as suggested by the applicant
☐ as selected by this Authority, because the applicant failed to suggest a figure
☐ as selected by this Authority, because this figure better characterizes the invention
- b. ☐ none of the figures is to be published with the abstract

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2007/000055

A. CLASSIFICATION OF SUBJECT MATTER

INV. C09K8/03 C09K8/16 C09K8/26 C09K8/32 C09K8/502
C09K8/504 C09K8/52 C09K8/66

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)

C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4 369 843 A (MONDSHINE THOMAS C) 25 January 1983 (1983-01-25) column 1, line 18 - line 65 column 2, line 13 - line 35 column 3, line 21 - line 42	1-6, 16-23
X	column 3, line 65 - column 4, line 39 column 5, line 50 - line 62; claims 1,3	8-11
Y	EP 0 845 520 A1 (TEXAS UNITED CHEMICAL CORP [US]) 3 June 1998 (1998-06-03) page 2, line 5 - line 18 page 3, line 5 - line 13 page 3, line 25 - page 4, line 28 page 4, line 58 - page 5, line 19 page 5, line 53 - line 58 page 6, line 27 - line 32	1-6,8,9, 16-23

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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 document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

& document member of the same patent family

Date of the actual completion of the international search

29 March 2007

Date of mailing of the international search report

05/04/2007

Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Boulon, Annie

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2007/000055

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 616 660 A1 (BAKER HUGHES INC [US]) 28 September 1994 (1994-09-28) paragraph [0001] - paragraph [0002]	1-6, 8, 9, 18, 19
X	paragraph [0017] - paragraph [0018] paragraph [0019] - paragraph [0026] paragraph [0030]	21-26
Y	EP 1 074 598 A1 (TEXAS UNITED CHEMICAL CORP [US]) 7 February 2001 (2001-02-07) paragraph [0002] - paragraph [0003] paragraph [0011] paragraph [0029] - paragraph [0032] paragraph [0038] - paragraph [0040]	1-6, 8, 9, 16-23
Y	US 2006/009364 A1 (DOBSON JAMES W JR [US] ET AL) 12 January 2006 (2006-01-12) paragraph [0002] paragraph [0033] - paragraph [0037]	1, 4-9, 12, 13, 16-23, 25
Y, P	WO 2006/054058 A (HALLIBURTON ENERGY SERV INC [US]; WAIN CHRISTOPHER PAUL [GB]; MUNOZ TR) 26 May 2006 (2006-05-26) page 2, line 5 - page 3, line 16 page 3, line 27 - page 6, line 27 page 10, line 19 - page 11, line 9	1-3, 5-9, 12, 13, 16-23, 25

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2007/000055

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 4369843	A	25-01-1983	NONE	
EP 0845520	A1	03-06-1998	AU 723115 B2	17-08-2000
			AU 3988297 A	04-06-1998
			CA 2218214 A1	02-06-1998
			DE 69712855 D1	04-07-2002
			DE 69712855 T2	28-11-2002
			DK 845520 T3	09-09-2002
			NO 975494 A	03-06-1998
EP 0616660	A1	28-09-1994	AU 5353394 A	09-05-1994
			NO 933774 A	22-04-1994
			WO 9409254 A1	28-04-1994
			US 5504062 A	02-04-1996
			US 5325921 A	05-07-1994
EP 1074598	A1	07-02-2001	AU 771831 B2	01-04-2004
			AU 4894800 A	08-02-2001
			BR 0012955 A	18-06-2002
			CA 2314806 A1	05-02-2001
			DE 60018857 D1	28-04-2005
			DE 60018857 T2	06-04-2006
			ID 26742 A	08-02-2001
			NO 20003972 A	06-02-2001
			WO 0110976 A1	15-02-2001
			US 6300286 B1	09-10-2001
			US 6391830 B1	21-05-2002
US 2006009364	A1	12-01-2006	NONE	
WO 2006054058	A	26-05-2006	US 2006105918 A1	18-05-2006

PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

PCT

To:

see form PCT/ISA/220

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY (PCT Rule 43bis.1)

Date of mailing
(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference
see form PCT/ISA/220

FOR FURTHER ACTION
See paragraph 2 below

International application No.
PCT/GB2007/000055

International filing date (day/month/year)
10.01.2007

Priority date (day/month/year)
31.01.2006

International Patent Classification (IPC) or both national classification and IPC
INV. C09K8/03 C09K8/16 C09K8/26 C09K8/32 C09K8/502 C09K8/504 C09K8/52 C09K8/66

Applicant
BP EXPLORATION OPERATING COMPANY LIMITED

1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☒ Box No. VI Certain documents cited
- ☐ Box No. VII Certain defects in the international application
- ☐ Box No. VIII Certain observations on the international application

2. FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1bis(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

3. For further details, see notes to Form PCT/ISA/220.

Name and mailing address of the ISA:



European Patent Office - P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk - Pays Bas
Tel. +31 70 340 - 2040 Tx: 31 651 epo nl
Fax: +31 70 340 - 3016

Date of completion of
this opinion

see form
PCT/ISA/210

Authorized Officer

Boulon, Annie

Telephone No. +31 70 340-2340



**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY**

International application No.
PCT/GB2007/000055

Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of:

- ☒ the international application in the language in which it was filed
- ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1 (b)).

2. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application and necessary to the claimed invention, this opinion has been established on the basis of:

a. type of material:

- ☐ a sequence listing
- ☐ table(s) related to the sequence listing

b. format of material:

- ☐ on paper
- ☐ in electronic form

c. time of filing/furnishing:

- ☐ contained in the international application as filed.
- ☐ filed together with the international application in electronic form.
- ☐ furnished subsequently to this Authority for the purposes of search.

3. ☐ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

4. Additional comments:

**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY**

International application No.
PCT/GB2007/000055

Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	<u>1-27</u>
	No: Claims	
Inventive step (IS)	Yes: Claims	
	No: Claims	<u>1-27</u>
Industrial applicability (IA)	Yes: Claims	<u>1-27</u>
	No: Claims	

2. Citations and explanations

see separate sheet

Box No. VI Certain documents cited

1. Certain published documents (Rules 43bis.1 and 70.10)

and / or

2. Non-written disclosures (Rules 43bis.1 and 70.9)

see form 210

Re Item V.

1. Preliminary remark

Although claims 8,16,18,21,24,25,26 have been drafted as separate independent claims, they appear to relate effectively to the same subject-matter and to differ from each other only with regard to the definition of the subject-matter for which protection is sought or in respect of the terminology used for the features of that subject-matter, and thus lack conciseness.

Moreover, lack of clarity as a whole arises, since the plurality of independent claims makes it difficult to determine the matter for which protection is sought.

The aforementioned claims therefore do not meet the requirements of Article 6 PCT.

2. Reference is made to the following documents:

D1 : US 4 369 843 A (MONDSHINE THOMAS C) 25 January 1983 (1983-01-25)

D2 : EP 0 845 520 A1 (TEXAS UNITED CHEMICAL CORP [US]) 3 June 1998
(1998-06-03)

D3 : EP 0 616 660 A1 (BAKER HUGHES INC [US]) 28 September 1994 (1994-09-28)

D4 : EP 1 074 598 A1 (TEXAS UNITED CHEMICAL CORP [US]) 7 February 2001
(2001-02-07)

D5 : US 2006/009364 A1 (DOBSON JAMES W JR [US] ET AL) 12 January 2006
(2006-01-12)

3. INDEPENDENT CLAIM 1

3.1 Document D1, which is considered to represent the most relevant state of the art to the subject matter of claim 1, discloses (see the relevant passages indicated in the search report) a well completion workover method wherein a subterranean hydrocarbon-producing formation or formations is contacted with a high density, nondamaging treating fluid for controlling the formation pressure and also for temporarily bridging and sealing off the productive formation or formations in the well bore to minimize fluid loss or damage to such producing formations.

- The treating fluid comprises a saturated aqueous saline solution with at least one water soluble salt which is substantially insoluble in the saturated saline solution, the water soluble salt having a particle size range of about 5 microns to about 800 microns, - Any water soluble salt which is substantially insoluble in the saturated saline solution may be employed. Some which are generally available include potassium chloride, sodium chloride, calcium chloride, sodium sulfate, sodium carbonate, sodium bicarbonate, calcium bromide and potassium carbonate.

The bridging and sealing salt particles used in the well completion and work over method can be dissolved by the flow of produced field brine or by the injection of water or an unsaturated saline solution. This eliminates the undesirable use of oil or acid solutions to remove the bridge from the subterranean hydrocarbon producing formation

3.2 The subject-matter of independent claim 1 differs from the disclosure of D1 in that the bridging agent is melamine, lithium carbonate, lithium phosphate, or magnesium sulfite.

3.3 The technical effect in view of these distinguishing features is, according to the description of present application, that the filter cake which is deposited by this composition on the walls of the well does not need an acid clean up fluid to be removed

3.4 The problem to be solved by the present invention may therefore be regarded as to provide an alternative composition for depositing an easily removable filter cake

3.5 Document D2 discloses ((see the relevant passages indicated in the search report) low solids, high density fluids useful as drilling, work over, and completion fluids. - The fluids comprise a brine, a viscosifier which is a high surface area silica, a biopolymer viscosifier, a fluid loss control additive, an alkaline buffer, a polar additive, and a bridging agent and provide more readily removable filter cakes, easier well clean-up, and better hydrocarbon production.

- Acid soluble salts (calcium carbonate, calcium/magnesium carbonate, iron carbonate, and other carbonates) , as well as water soluble salts (sodium chloride, potassium chloride, calcium chloride, sodium formate, potassium formate, cesium formate, sodium bromide, potassium bromide, calcium bromide, sodium acetate, potassium acetate, and

the like) can be used as bridging agents.

3.6 Considering the teachings of D1 and D2, the skilled person starting from D1 would regard it as a normal option to try out all the various bridging agents having different solubilities known from D2 in order to solve the problem posed.

3.7 Consequently, the solution proposed in claim 1 of the present application cannot be considered as involving an inventive step (Article 33(1) in the sense of Article 33(3) PCT)

4. INDEPENDENT CLAIM 8,18,18,21,24

The reasoning of paragraph 3 above applies mutatis mutandis for the subject-matter of claims 8,18,18,21,24 which cannot be considered as involving an inventive step (Article 33(1) in the sense of Article 33(3) PCT)

5. INDEPENDENT CLAIMS 25,26

5.1 The document D3 is regarded as being the closest prior art to the subject-matter of claim 25 and discloses (see the relevant passages indicated in the search report) hydraulic fracturing methods using fluid systems for controlling fluid losses, and more particularly relates, to multi-fluid methods which form a durable, thin filter cake to assist in intentional screen-out during fracturing, but which is easy to remove and results in little damage to the permeability of the formation or the conductivity of the propped fracture.

The cake-building solid particulate may be selected from, but not limited to, graded salt, silica, clays (e.g. montmorillonite, bentonite, attapulgite, etc.), oil soluble particulates, and where the salt of the graded salt is selected from the group consisting of potassium chloride; sodium chloride; calcium chloride; magnesium chloride; sodium sulfate; sodium carbonate; calcium carbonate; sodium bicarbonate; sodium bromide; potassium bromide; potassium carbonate and mixtures thereof.

5.2 The subject-matter of claim 25, therefore differs from this known D3 in that the leak-off control agent is selected from melamine, lithium carbonate, lithium phosphate or magnesium sulfite.

5.3 No plausible technical effect has been shown for these distinguishing features other than that already known from D3.

5.4 The problem to be solved by the present invention can therefore only be regarded as to provide an alternative composition for easily removable particulate leak-off agent

5.5 D3 clearly indicates that a wide variety of particulate materials can be used, therefore it would be part of the routine work of the skilled person to experiment with other components in order to solve the problem posed, without exercising any inventive activity (Article 33(1) in the sense of article 33(3) PCT).

5.6 The same reasoning applies mutatis mutandis to the subject-matter of claim 28 which therefore cannot be regarded as involving an inventive step (Article 33(1) in the sense of article 33(3) PCT).

6. DEPENDENT CLAIMS 2-7,9-15,17,19,20,22,23,and 27

The features disclosed in dependent claims 2-7,9-15,17,19,20,22,23,and 27 are well-known in the state of the art (see D1-D5 and the corresponding passages in the search report) , and are either not novel or not inventive. Thus they would be patentable only if the subject-matter of claims 1 ,8,16,18,21,24,25,26 to which they refer, were patentable.

The demand must be filed directly with the competent International Preliminary Examining Authority or, if two or more Authorities are competent, with the one chosen by the applicant. The full name or two-letter code of that Authority may be indicated by the applicant on the line below:

IPEA/ _____

PCT

CHAPTER II

DEMAND

under Article 31 of the Patent Cooperation Treaty:

The undersigned requests that the international application specified below be the subject of international preliminary examination according to the Patent Cooperation Treaty.

For International Preliminary Examining Authority use only	
Identification of IPEA	Date of receipt of DEMAND
Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION	
Applicant's or agent's file reference 10314	
International application No. PCT/GB2007/000055	International filing date (day/month/year) 10 January 2007
(Earliest) Priority date (day/month/year) 31 January 2006	
Title of invention WELLBORE FLUID COMPRISING A BASE FLUID AND A PARTICULATE BRIDGING AGENT	
Box No. II APPLICANT(S)	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) BP EXPLORATION OPERATING COMPANY LIMITED Chertsey Road Sunbury-on-Thames Middlesex, TW16 7BP United Kingdom	
Telephone No.	
Facsimile No.	
Applicant's registration No. with the Office	
State (that is, country) of nationality: GB	State (that is, country) of residence: GB
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) DUNCUM, Simon Neil 9 Wilstrode Avenue Binfield, Bracknell Berkshire, RG42 4UW United Kingdom	
State (that is, country) of nationality: GB	State (that is, country) of residence: GB
☒ Further applicants are indicated on a continuation sheet.	

Continuation of Box No. II APPLICANT(S)

If none of the following sub-boxes is used, this sheet should not be included in the demand.

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

SAWDON, Christopher Alan
6 Southview Road
Par
Cornwall, PL24 2HJ
United Kingdom

State (that is, country) of nationality:
GB

State (that is, country) of residence:
GB

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

State (that is, country) of nationality:

State (that is, country) of residence:

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

State (that is, country) of nationality:

State (that is, country) of residence:

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

State (that is, country) of nationality:

State (that is, country) of residence:



Further applicants are indicated on another continuation sheet.

Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCEThe following person is ☒ agent ☐ common representativeand ☒ has been appointed earlier and represents the applicant(s) also for international preliminary examination.☐ is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked.☐ is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*COLLINS, Frances Mary
BP INTERNATIONAL LIMITED
Global Patents and Technology Law (Central)
Chertsey Road, Sunbury-on-Thames,
Middlesex, TW16 7LN, United Kingdom

Telephone No.

01932 763201

Facsimile No.

01932 763206

Agent's registration No. with the Office

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.**Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION****Statement concerning amendments:***

1. The applicant wishes the international preliminary examination to start on the basis of:

☒ the international application as originally filedthe description ☒ as originally filed☐ as amended under Article 34the claims ☒ as originally filed☐ as amended under Article 19 (together with any accompanying statement)☐ as amended under Article 34the drawings ☐ as originally filed☐ as amended under Article 342. ☐ The applicant wishes any amendment to the claims under Article 19 to be considered as reversed.3. ☐ Where the IPEA wishes to start the international preliminary examination at the same time as the international search in accordance with Rule 69.1(b), the applicant requests the IPEA to **postpone** the start of the international preliminary examination until the expiration of the applicable time limit under Rule 69.1(d).4. ☐ The applicant expressly wishes the international preliminary examination to **start earlier** than at the expiration of the applicable time limit under Rule 54bis.1(a).

* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purposes of international preliminary examination: **English**☒ which is the language in which the international application was filed.☐ which is the language of a translation furnished for the purposes of international search.☐ which is the language of publication of the international application.☐ which is the language of the translation (to be) furnished for the purposes of international preliminary examination.**Box No. V ELECTION OF STATES**

The filing of this demand constitutes the election of all Contracting States which are designated and are bound by Chapter II of the PCT.

Box No. VI CHECK LIST

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination:

- | | | |
|--|---|--------|
| 1. translation of international application | : | sheets |
| 2. amendments under Article 34 | : | sheets |
| 3. copy (or, where required, translation) of amendments under Article 19 | : | sheets |
| 4. copy (or, where required, translation) of statement under Article 19 | : | sheets |
| 5. letter | : | sheets |
| 6. other (<i>specify</i>) | : | sheets |

For International Preliminary Examining Authority use only

received	not received
----------	--------------

☐	☐
☐	☐
☐	☐
☐	☐
☐	☐
☐	☐

The demand is also accompanied by the item(s) marked below:

- | | |
|--|---|
| 1. ☒ fee calculation sheet | 5. ☐ statement explaining lack of signature |
| 2. ☐ original separate power of attorney | 6. ☐ sequence listing in electronic form |
| 3. ☐ original general power of attorney | 7. ☐ tables in electronic form related to a sequence listing |
| 4. ☐ copy of general power of attorney; reference number, if any: | 8. ☐ other (<i>specify</i>): |

Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand).

F. M. Collins
COLLINS, Frances Mary
Authorised Representative

For International Preliminary Examining Authority use only

1. Date of actual receipt of DEMAND:

2. Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):

3. ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply.

☐ The applicant has been informed accordingly.

4. ☐ The date of receipt of the demand is WITHIN the time limit of 19 months from the priority date as extended by virtue of Rule 80.5.

5. ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.

6. ☐ The date of receipt of the demand is AFTER the expiration of the time limit under Rule 54bis.1(a) and item 7 or 8, below, does not apply.

7. ☐ The date of receipt of the demand is WITHIN the time limit under Rule 54bis.1(a) as extended by virtue of Rule 80.5.

8. ☐ Although the date of receipt of the demand is after the expiration of the time limit under Rule 54bis.1(a), the delay in arrival is EXCUSED pursuant to Rule 82.

For International Bureau use only

Demand received from IPEA on:

PCT

FEE CALCULATION SHEET

Annex to the Demand

International application No. PCT/GB2007/000055	For International Preliminary Examining Authority use only
Applicant's or agent's file reference 10314	Date stamp of the IPEA
Applicant BP EXPLORATION OPERATING COMPANY LIMITED et al	
CALCULATION OF PRESCRIBED FEES	
1. Preliminary examination fee	EUR 1595-00 P
2. Handling fee (<i>Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.</i>)	EUR 129-00 H
3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box	EUR 1,724-00 TOTAL
MODE OF PAYMENT <i>(Not all modes of payment may be available at all IPEAs)</i>	
☒ authorization to charge deposit account with the IPEA (see below)	☐ cash
☐ cheque	☐ revenue stamps
☐ postal money order	☐ coupons
☐ bank draft	☐ other (specify):
AUTHORIZATION TO CHARGE (OR CREDIT) DEPOSIT ACCOUNT <i>(This mode of payment may not be available at all IPEAs)</i>	
☒ Authorization to charge the total fees indicated above.	IPEA/ <u>EP</u>
☐ (<i>This check-box may be marked only if the conditions for deposit accounts of the IPEA so permit</i>) Authorization to charge any deficiency or credit any overpayment in the total fees indicated above.	Deposit Account No.: <u>28050129 BP Int Ltd</u>
	Date: <u>1st November 2007</u>
	Name: <u>Anne Perry</u>
	Signature: <u>A.M. Perry</u>

PATENT COOPERATION TREATY

AMP
FMC

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

PCT

To:

COLLINS, Frances, Mary
BP INTERNATIONAL LIMITED
Global Patents & Technology Law (Central)
Chertsey Road
Sunbury on Thames
Middlesex TW16 7LN
GRANDE BRETAGNE

Received in PAT

21 NOV 2007

NOTIFICATION OF RECEIPT
DEMAND BY COMPETENT INTERNATIONAL
PRELIMINARY EXAMINING AUTHORITY

(PCT Rules 59.3(e) and 61.1(b), first sentence
and Administrative Instructions, Section 601(a))

Date of mailing
(day/month/year) 19-11-2007

Applicant's or agent's file reference
10314

IMPORTANT NOTIFICATION

International application No.

PCT/GB2007/000055

International filing date (day/month/year)

10 January 2007 (10-01-2007)

Priority date (day/month/year)

31 January 2006 (31-01-2006)

Applicant

BP EXPLORATION OPERATING COMPANY LIMITED et al.

1. The applicant is hereby **notified** that this International Preliminary Examining Authority considers the following date as the date of receipt of the demand for international preliminary examination of the international application:

5 November 2007 (05-11-2007)

2. This date of receipt is:

- ☒ the actual date of receipt of the demand by this Authority (Rule 61.1(b)).
☐ the actual date of receipt of the demand on behalf of this Authority (Rule 59.3(e)).
☐ the date on which this Authority has, in response to the invitation to correct defects in the demand (Form PCT/IPEA/404), received the required corrections.

3. ☒ **ATTENTION:** That date of receipt is **after** the expiration of 19 months from the priority date. Consequently, in respect of some Offices, the demand does not have the effect of postponing the entry into the national phase until 30 months from the priority date (or later in some Offices) (Article 39(1)) and the acts for entry into the national phase must therefore be performed within 20 months from the priority date (or later in some Offices). **However**, in respect of some other Offices, the time limit of 30 months (or later) may nevertheless apply. See the Annex to Form PCT/IB/301 and, for details about the applicable time limits, Office by Office, see the *PCT Applicant's Guide*, Volume II, National Chapters and the WIPO Internet site.

- ☐ (If applicable) This notification confirms the information given by telephone, facsimile transmission or in person on:

4. Only where paragraph 3 applies, a copy of this notification has been sent to the International Bureau.

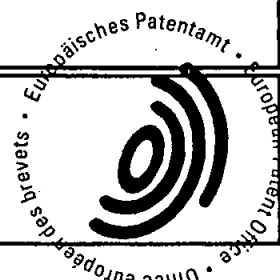
Name and mailing address of the IPEA/



European Patent Office
D-80298 Munich
Tel. (+49-89) 2399-0
Fax: (+49-89) 2399-4465

Authorized officer

Dekker, Monique
Tel: (+31-70)340-4046



PATENT COOPERATION TREATY

AMP
File

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To: BP INTERNATIONAL LIMITED Global Patents & Technology Law (Central) Chertsey Road Sunbury-on-Thames Middlesex TW16 7LN GRANDE BRETAGNE	Received in PAT PCT - 9 MAY 2008 NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY (PCT Rule 71.1)
Applicant's or agent's file reference 10314	IMPORTANT NOTIFICATION
International application No. PCT/GB2007/000055	International filing date (day/month/year) 10.01.2007
Priority date (day/month/year) 31.01.2006	
Applicant BP EXPLORATION OPERATING COMPANY LIMITED	

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary report on patentability and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.
4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary report on patentability. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

The applicant's attention is drawn to Article 33(5), which provides that the criteria of novelty, inventive step and industrial applicability described in Article 33(2) to (4) merely serve the purposes of international preliminary examination and that "any Contracting State may apply additional or different criteria for the purposes of deciding whether, in that State, the claimed inventions is patentable or not" (see also Article 27(5)). Such additional criteria may relate, for example, to exemptions from patentability, requirements for enabling disclosure, clarity and support for the claims.

Name and mailing address of the international preliminary examining authority: European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Authorized Officer Cipolla, Loredana Tel. +31 70 340-3247
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PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 10314		FOR FURTHER ACTION		See Form PCT/PEA/416
International application No. PCT/GB2007/000055		International filing date (day/month/year) 10.01.2007		Priority date (day/month/year) 31.01.2006
International Patent Classification (IPC) or national classification and IPC INV. C09K8/03				
Applicant BP EXPLORATION OPERATING COMPANY LIMITED				
1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36. 2. This REPORT consists of a total of <u>9</u> sheets, including this cover sheet. 3. This report is also accompanied by ANNEXES, comprising: a. ☐ sent to the applicant and to the International Bureau) a total of sheets, as follows: ☐ sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions). ☐ sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box. b. ☐ (sent to the International Bureau only) a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or tables related thereto, in electronic form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).				
4. This report contains indications relating to the following items: ☒ Box No. I Basis of the report ☐ Box No. II Priority ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability ☐ Box No. IV Lack of unity of invention ☒ Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement ☐ Box No. VI Certain documents cited ☐ Box No. VII Certain defects in the international application ☒ Box No. VIII Certain observations on the international application				
Date of submission of the demand 2007-11-05		Date of completion of this report 06.05.2008		
Name and mailing address of the international preliminary examining authority:  European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016		Authorized officer Boulon, Annie Telephone No. +31 70 340-2340 		

**INTERNATIONAL PRELIMINARY REPORT
ON PATENTABILITY**

International application No.
PCT/GB2007/000055

Box No. I Basis of the report

1. With regard to the **language**, this report is based on

- ☒ the international application in the language in which it was filed
- ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of:
 - ☐ international search (under Rules 12.3(a) and 23.1(b))
 - ☐ publication of the international application (under Rule 12.4(a))
 - ☐ international preliminary examination (under Rules 55.2(a) and/or 55.3(a))

2. With regard to the **elements*** of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report):*

Description, Pages

1-28 as originally filed

Claims, Numbers

1-27 as originally filed

- ☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing

3. ☐ The amendments have resulted in the cancellation of:

- ☐ the description, pages
- ☐ the claims, Nos.
- ☐ the drawings, sheets/figs
- ☐ the sequence listing (*specify*):
- ☐ any table(s) related to sequence listing (*specify*):

4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

- ☐ the description, pages
- ☐ the claims, Nos.
- ☐ the drawings, sheets/figs
- ☐ the sequence listing (*specify*):
- ☐ any table(s) related to sequence listing (*specify*):

5. ☐ This opinion has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 70.2 (e)).

**INTERNATIONAL PRELIMINARY REPORT
ON PATENTABILITY**

International application No.
PCT/GB2007/000055

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	<u>1-27</u>
	No: Claims	
Inventive step (IS)	Yes: Claims	
	No: Claims	<u>1-27</u>
Industrial applicability (IA)	Yes: Claims	<u>1-27</u>
	No: Claims	

2. Citations and explanations (Rule 70.7):

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Re Item V.

1. Preliminary remarks

1.1 In claims 1,8,10-12,14-16,18,21,24-26, the term "**sparingly** water-soluble" does not give a clear and unambiguous definition of the required solubility thereby not defining clearly the matter for which protection is sought.

The requirements of Article 6 PCT are thus not fulfilled.

1.2 Although claims 8,16,18,21,24,25,26 have been drafted as separate independent claims, they appear to relate effectively to the same subject-matter and to differ from each other only with regard to the definition of the subject-matter for which protection is sought or in respect of the terminology used for the features of that subject-matter, and thus lack conciseness.

Moreover, lack of clarity as a whole arises, since the plurality of independent claims makes it difficult to determine the matter for which protection is sought.

The aforementioned claims therefore do not meet the requirements of Article 6 PCT.

2. Reference is made to the following documents:

D1 : US 4 369 843 A (MONDSHINE THOMAS C) 25 January 1983 (1983-01-25)

D2 : EP 0 845 520 A1 (TEXAS UNITED CHEMICAL CORP [US]) 3 June 1998
(1998-06-03)

D3 : EP 0 616 660 A1 (BAKER HUGHES INC [US]) 28 September 1994 (1994-09-28)

D4 : EP 1 074 598 A1 (TEXAS UNITED CHEMICAL CORP [US]) 7 February
2001 (2001-02-07)

D5 : US 2006/009364 A1 (DOBSON JAMES W JR [US] ET AL) 12 January 2006
(2006-01-12)

3. NOVELTY (Article 33(2) PCT), independent claims 1,8,16,18,21,24,25,26

None of the cited documents mentions the claimed bridging agents (melamine, lithium carbonate, lithium phosphate, magnesium sulfite).

The subject-matter of claims 1,8,16,18,21,24,25,26 is therefore, notwithstanding the above-mentioned lack of clarity, novel in the sense of Article 33(2)PCT

4 INVENTIVE STEP (Article 33(3) PCT

4.1 Preliminary comments

In his letter dated 31.10.2007 the applicant responded to the arguments set out in the written opinion, submitting that the claims of present application are inventive, the examining division does not share this view for the following reasons :

4.1.1 According to the applicant the fluid defined in claim 1 has the advantage that the **density of the fluid is not limited by the sparingly soluble material**, as the material is only sparingly soluble in water only slightly more material is added to the fluid than needed to form the filter cake which allows the **production of a low density fluid**.

There seem to be a contradiction between these two statements, and there is no indication in the description that the fluid of the invention is a low density fluid (see page 9, line 25 "appropriate density", page 10, line 14 "sufficient density").

Furthermore, there are statements in the description illustrating the contrary :

- page 13, line 2 : addition of a density increasing water soluble salt
- page 13, line 10 and lines 28,29 : "density control provided by insoluble weighting agents"
- page 13, lines 17-19 and page 16, lines 6,7 "Suitably, the aqueous phase of the emulsion comprises an aqueous solution of a density increasing water-soluble salt thereby increasing the density of the wellbore fluid."

The argument on the low density of the fluid of the invention is therefore not regarded as relevant

4.1.2 The applicant also stressed that filter cakes formed according to claim 1 can be removed by simply putting the well into production and that there is no need for a separate procedure supplying a clean-up fluid.

The same effect is clearly indicated in D1 (see below)

4.1.3 The applicant's further argument is that the resultant filter cake can be removed with increased speed using weak acids. (page 5 of present description "if rapid dissolution is

required, a clean-up fluid may be pumped into the formation.."

The method using an acid clean-up fluid is common for removing a filter cake formed with the well-known bridging agents.

4.2 INDEPENDENT CLAIM 1

4.2.1 Document D1, which is considered to represent the most relevant state of the art to the subject matter of claim 1, discloses (see the relevant passages indicated in the search report) a well completion work over method wherein a subterranean hydrocarbon-producing formation or formations is contacted with a high density, non damaging treating fluid for **controlling the formation pressure** and also for temporarily **bridging** and sealing off the productive formation or formations in the wellbore to minimize fluid loss or damage to such producing formations.

- The treating fluid comprises a saturated aqueous saline solution with at least one water-soluble salt which is substantially insoluble in the saturated saline solution, the water-soluble salt having a particle size range of about 5 microns to about 800 microns,
- Any water soluble salt which is **substantially insoluble** in the saturated saline solution may be employed. Some which are generally available include potassium chloride, sodium chloride, calcium chloride, sodium sulfate, **sodium carbonate**, sodium bicarbonate, calcium bromide and **potassium carbonate**.

The bridging and sealing salt particles used in the well completion and work over method can be dissolved by the flow of produced field brine or by the injection of water or an unsaturated saline solution. This eliminates the undesirable use of oil or acid solutions to remove the bridge from the subterranean hydrocarbon producing formation

4.2.2 In view of the clarity objection above (point 1.1) and considering that the difference between **sparingly soluble** and **substantially insoluble** is not clear, the subject-matter of independent claim 1 differs from the disclosure of D1 in that the bridging agent is melamine, lithium carbonate, lithium phosphate, or magnesium sulfite.

4.2.3 The technical effect in view of these distinguishing features is, according to the description of present application, that the filter cake which is deposited by this composition on the walls of the well does not need an acid clean up fluid to be removed
The same effect is mentioned in D1 (see column 2, lines 29-35)

4.2.4 The problem to be solved by the present invention can therefore only be regarded as to provide an alternative composition for depositing an easily removable filter cake .

4.2.5 Document D2 discloses (see the relevant passages indicated in the search report) low solids, high density fluids useful as drilling, work over, and completion fluids.

- The fluids comprise a brine, a viscosifier which is a high surface area silica, a biopolymer viscosifier, a fluid loss control additive, an alkaline buffer, a polar additive, and a bridging agent and **provide more readily removable filter cakes**, easier well clean-up, and better hydrocarbon production (see page 3, lines 9-12).
- The preferred fluids contain a particulate bridging agent(see page 4, line 58) and the bridging agent must not be appreciably soluble in the brine used to prepare the fluid (see page 5, lines 11,12)
- Acid soluble salts (calcium carbonate, calcium/magnesium carbonate, iron carbonate, and other carbonates) , as well as water soluble salts (sodium chloride, potassium chloride, calcium chloride, sodium formate, potassium formate, cesium formate, sodium bromide, potassium bromide, calcium bromide, sodium acetate, potassium acetate, and the like) can be used as bridging agents.

4.2.6 Considering the teachings of D1 and D2, the skilled person starting from D1 would regard it as a normal option to try out all various bridging agents having different solubilities either known from D2 or belonging to similar chemical families, for instance carbonates of different alkali metals, in order to solve the problem posed.

4.2.7 Consequently, the solution proposed in claim 1 of the present application cannot be considered as involving an inventive step (Article 33(1) in the sense of Article 33(3) PCT)

4.3 INDEPENDENT CLAIM 8,18,18,21,24

The reasoning of paragraph 3 above applies mutatis mutandis for the subject-matter of claims 8,18,18,21,24 which, notwithstanding the above-mentioned lack of clarity, cannot be considered as involving an inventive step (Article 33(1) in the sense of Article 33(3) PCT)

4.4 INDEPENDENT CLAIMS 25,26

4.4.1 The document D3 is regarded as being the closest prior art to the subject-matter of claim 25 and discloses (see the relevant passages indicated in the search report) hydraulic fracturing methods using fluid systems for controlling fluid losses, and more particularly relates, to multi-fluid methods which form a durable, **thin filter cake** to assist in intentional screen-out during fracturing, but which is **easy to remove** and results in little damage to the permeability of the formation or the conductivity of the propped fracture.

The cake-building solid particulate may be selected from, but not limited to, graded salt, silica, clays (e.g. montmorillonite, bentonite, attapulgite, etc.), oil soluble particulates, and where the salt of the graded salt is selected from the group consisting of potassium chloride; sodium chloride; calcium chloride; magnesium chloride; sodium sulfate; sodium carbonate; calcium carbonate; sodium bicarbonate; sodium bromide; potassium bromide; potassium carbonate and mixtures thereof.

4.4.2 The subject-matter of claim 25, therefore differs from this known D3 in that the leak-off control agent is selected from melamine, lithium carbonate, lithium phosphate or magnesium sulfite.

4.4.3 No plausible technical effect has been shown for these distinguishing features other than that already known from D3.

4.4.4 The problem to be solved by the present invention can therefore only be regarded as to provide an alternative composition for easily removable particulate leak-off agent

4.4.5 D3 clearly indicates that a wide variety of particulate materials can be used, therefore it would be part of the routine work of the skilled person to experiment with other components in order to solve the problem posed, without exercising any inventive activity.

Thus, the subject-matter of claim 25, notwithstanding the above mentioned lack of clarity, cannot be regarded as involving an inventive step (Article 33(1) in the sense of article 33(3) PCT).

4.4.6 The same reasoning applies mutatis mutandis to the subject-matter of claim 26 which, notwithstanding the above mentioned lack of clarity, cannot be regarded as involving an inventive step (Article 33(1) in the sense of article 33(3) PCT).

5. DEPENDENT CLAIMS 2-7,9-15,17,19,20,22,23, and 27

The features disclosed in dependent claims 2-7,9-15,17,19,20,22,23, and 27 are well-known in the state of the art (see D1-D5 and the corresponding passages in the search report) , and are either not novel or not inventive. Thus they would be patentable only if the subject-matter of claims 1 ,8,16,18,21,24,25,26 to which they refer, were patentable.

Re Item VIII

- The wording of claims 1,8,10-12,14-16,18,21,24-26 lacks clarity see point 1.1 above
- Claims 8,16,18,21,24-26 lack conciseness and clarity see point 1.2 above

bp

Louise Ford

European Patent Attorney



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International Preliminary Examining Authority
European Patent Office
Directorate General 2
Erhardtstrasse 27
D-80298 München 2
Germany

31st October 2007

BY FACSIMILE

Dear Sirs,

International Application No. PCT/GB2007/000055
Applicant: BP EXPLORATION OPERATING COMPANY LIMITED et al
Our Ref: BP Case 10314

This letter responds to the Examiner's observations set out in the Written Opinion of the International Searching Authority, dated 5th April 2007. We respectfully request that the following comments be taken into account in advance of the preparation of the International Preliminary Examination Report.

In the written opinion, the Examiner alleges that each of the independent claims of this application lack an inventive step in light of the prior art cited in the ISR. Contrary to this view, we submit that the claims of this application are, in fact, inventive.

Claim 1 defines a wellbore fluid comprising a base fluid and a particulate bridging agent comprised of a sparingly water-soluble material selected from the group consisting of melamine, lithium carbonate, lithium phosphate and magnesium sulfite.

The fluid as defined in this claim has the advantage that the density of the fluid is not limited by the sparingly water-soluble material. Since the material is only sparingly soluble in water, only slightly more material is added to the fluid than is needed to form the filter cake. This allows the production of relatively low density fluids which can be used in low-pressure

contd/.....

wells which would otherwise be damaged if subjected to the pressures caused by higher density fluids. This also reduces the amount of raw material needed to prepare the fluids, compared with other known fluids discussed below.

Additionally, filter cakes formed as a result of the use of the fluid of claim 1 can be removed when desired, simply by putting the well into production by commencing water injection or the like, since the water naturally present in the formation or in the injection fluid dissolves the filter cake. There is no need for a separate procedure supplying a clean-up fluid. This not only simplifies procedures and materials involved, but avoids one of the problems associated with known fluids comprising water-insoluble bridging agents, whereby a clean-up fluid comprising a strong acid is needed.

A further advantage of the present invention is that the resultant filter cake can optionally be removed with increased speed using weak acids. Even if the weak acid only dissolves a part of the filter cake, the rest of the filter cake will still be removed by the presence of water from the formation or in an injection fluid. This is in contrast to known fluids comprising water-insoluble bridging agents. With these fluids, if only part of the resultant filter cake is removed, the acidic clean-up fluid can break through the filter cake into the formation without acting on the rest of the filter cake.

In the Written Opinion, the Examiner considers that US 4369843 (D1) forms the closest prior art with respect to claim 1, and that claim 1 is obvious in light of this document. D1 teaches a well completion and workover method wherein a subterranean formation is contacted with a high density, nondamaging treating fluid comprising a saturated, aqueous saline solution with at least one water soluble salt that is substantially insoluble in the saturated saline solution.

The saturated saline solution can comprise water with potassium chloride, sodium chloride, calcium chloride, sodium sulphate, sodium carbonate, sodium bicarbonate, calcium bromide, potassium carbonate or a mixture thereof dissolved therein. The water soluble salt to be suspended in the saline solution can be any of the salts mentioned above. The salt is added in a sufficient quantity so that even if some of it dissolves due to the higher temperatures encountered in well bores, the amount dissolved will not materially affect the action of the sized salt particles suspended in the saturated saline solution in functioning as a water soluble bridging agent (column 3 lines 4-36). In other words, the salt is dissolved into water to the saturation point, and then more salt is added to ensure that there will still be solid particles suspended in the fluid even if higher temperatures cause more material to be dissolved.

Consequently, the density of the fluid depends on how much salt must be added to the fluid to ensure at least some salt remains suspended, and so fluids with densities below a threshold limit cannot be produced. There are

many wells that cannot tolerate high density fluids because the rock formation would be fractured by the hydrostatic pressure, and fluid would be lost into the fractures. The fluids of D1 are therefore not suitable for use in such wells.

Conversely, as discussed above, the fluids according to claim 1 of the present application do not suffer from this problem, and so can be used in a greater variety of wells.

Other advantages of fluids comprising the specific salts recited in claim 1, as compared with the fluids of D1, are discussed later.

In the Written Opinion, the Examiner alleges that D1 discloses the fluid of claim 1 with the exception of the use of a sparingly water soluble material selected from melamine, lithium carbonate, lithium phosphate and magnesium sulfite. The Examiner further alleges that it would be obvious to the skilled person to modify the teaching in D1 in view of the disclosure in EP 0845520 (D2), thereby arriving at the invention defined in claim 1 of this application. D2 teaches a method of enhancing the low shear rate viscosity of brine-based, polysaccharide-containing fluids containing a source of soluble zinc upon aging the fluids at elevated temperatures (page 2, lines 6-8). The fluids comprise a brine having a water soluble zinc salt such as zinc bromide dissolved therein. The brine also includes an amorphous silica viscosifier, a biopolymer viscosifier, a fluid loss control additive and zinc carbonate. Optionally, the fluids also can contain a low molecular weight polar additive, a bridging agent and an alkaline buffer, preferably zinc oxide. (page 3, lines 5-8). Other water soluble salts may be included in the brine.

If included, the bridging agent can be an acid soluble one including calcium carbonate, dolomite, iron carbonate, other carbonates, or water insoluble metal oxides or hydroxides. The bridging agent can also be a water soluble salt such as sodium chloride, potassium chloride, calcium chloride, sodium formate, potassium formate, cesium formate, sodium bromide, potassium bromide, calcium bromide, sodium acetate, potassium acetate and the like (page 5, lines 13-16).

Thus, D2 is directed towards enhancing the low shear rate viscosity of a brine-based, polysaccharide fluid containing dissolved zinc. The enhanced low shear rate viscosity provides excellent suspension and hole cleaning properties. D2 does not teach that the choice of, or indeed use of a bridging agent can affect how easily a filter cake can be removed. The choice of bridging agent is not disclosed as having any particular importance and indeed, a bridging agent need not even be included. The three options (a water soluble bridging agent, a water insoluble bridging agent or no bridging agent at all) are presented as equal options with no advantages or disadvantages. The skilled person is therefore not motivated by D2 to carry out any investigation into the nature of the bridging agent. There is

absolutely no reason why the skilled person would be lead in an obvious manner in view of D2 to consider different types of bridging agent (i.e. sparingly water soluble materials), and further no reason why the skilled person would select melamine, lithium carbonate, lithium phosphate and magnesium sulfite as the bridging agent. Consequently, we submit that the Examiner's allegation that the skilled person starting from D1 would regard it as a normal option to try out all the various bridging agents having different solubilities known from D2 is based on hindsight.

However, we submit that if the skilled person reviewed D2, he would learn that one option for the bridging agent is to use acid soluble agents. The materials listed in D2 as acid soluble agents are very insoluble in water. The skilled person would immediately understand that such bridging agents have been used in the past (page 2, line 25 to page 3 line 7 of present application and line 62 column 1 to line 2 column 2 of D1) and that the associated disadvantages of using acids to remove the filter cake are undesirable. Another option presented in D2 is to use water soluble agents, however this is no different to the fluids of D1.

Accordingly, even if the skilled person did look to D2 for alternative bridging agents, he would find only a discussion of known bridging agents, and nothing to suggest the possibility of another type of bridging agent. Further, there is no suggestion in the prior art documents that the compounds specifically selected and recited in claim 1 can result in a fluid which can be used in a wider variety of well bores and which forms a filter cake which is easily removable, as discussed above.

It is therefore submitted that the skilled person, without the benefit of hindsight, is not lead in an obvious manner to the subject matter of claim 1 in view of D1 and D2. This view is supported by the fact that D1 dates back to 1983 and D2 dates back to 1998. The need to seal off well bores, including low pressure well bores, from the surrounding formations whilst not restricting the future operation of the well bore is of great importance and so the field has been actively researched. Despite the active research, it has taken nearly 25 years from the publication of D1 and nearly a decade from the publication of D2 to develop the present invention as claimed in claim 1. It is submitted that this highlights the non-obvious nature of the invention.

The present invention has further advantages over the fluids of D1 where the fluids encounter an increase in temperature after deployment. As the fluids of D1 progress down a well bore, the temperature increases and so additional salt material is dissolved. Not only does this mean that significant amounts of salt must be mixed into the fluid, but also, when the time comes to remove the filter cake and fluid is returned to cooler environment, salt crystallises out of solution. The crystals can be larger than the original salt particles and can cause problems in the flow.

In the present invention, surprisingly lithium carbonate does not suffer this problem since it is actually less soluble at high temperatures, the solubility of magnesium sulphite is virtually unaffected by temperature and the dissolution of melamine at high temperatures can be easily inhibited. The fact that the selected compounds avoid this problem suffered by the fluids of D1 is not suggested in and cannot be appreciated from D1 or D2.

The other independent claims of the application also recite the sparingly water soluble material defined in claim 1. Since claim 1 is novel and inventive in light of D1 and D2, so too are the remaining claims in the application.

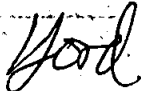
In the Written Opinion, the Examiner also alleged that independent claims 25 and 26 lack an inventive step in light of WO 94/09254 (D3). D3 discloses a method of propagating a hydraulic fracture which can be used across a wide range of permeabilities and heterogeneities in the formation. The method uses two fluids, a fluid pad which comprises a fluid loss control particulate and a proppant transport fluid. The fluid loss control particulate can be selected from a wide variety of materials such as those listed in the Written Opinion. The fluid pad is used to create a fracture and then form a filter cake on the surface of the fracture. Subsequently, this filter cake must be removed.

The materials disclosed in D3 for the fluid loss control particulates (i.e. the materials which form the filter cake) include water soluble salts like those disclosed in D1 and D2 as well as acid soluble/water insoluble salts such as calcium carbonate. Accordingly, the skilled person faces the same problems as discussed above with respect to D1 and D2, for example a lack of control over the fluid density or a need for strong acids to remove the filter cake. Accordingly, the methods of claims 25 and 26 have advantages over the method of D3. In particular, the methods of claims 25 and 26 recite the use of a fluid which permits a greater control over its density and which can leave an easily removable deposit on the formation.

As with D2, D3 teaches the use of both water soluble and water insoluble materials, and provides no discussion of the advantages or disadvantages associated with the different kinds of materials. There is no motivation in D3 or indeed in any other document to conduct investigations into alternative types of material and specifically nothing to lead the skilled reader in an obvious manner to consider sparingly water soluble materials, let alone the materials specifically recited in claims 25 and 26.

Accordingly, for the same reasons as set out above in connection with D1 and D2, we submit that claims 25 and 26 are inventive in light of D3.

Yours faithfully



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Priority date	Priority application No.	Country or regional Office or PCT receiving Office	Date of receipt of priority document
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