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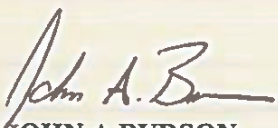
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1		1540-20400-Application.pdf	2602702	yes	23
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	Multipart Description/PDF files in .zip description				
	Document Description		Start		End
	Specification		1		18
	Claims		19		22
	Abstract		23		23
Warnings:					
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2	Drawings-only black and white line drawings	1540-20400-Drawings.pdf	113476	no	3
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3	Application Data Sheet	1540-20400-ADS.pdf	1172867	no	8
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Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

[0001] Not applicable.

BACKGROUND OF THE INVENTION

Field of the Invention

[0002] This invention relates to the field of industrial facility cleanup and more specifically to the disaggregation and subsequent removal of coke deposits on catalyst and to reduce vapors below the lower explosive limit (LEL) to allow for safe vessel entry into industrial equipment.

Background of the Invention

[0003] During the refinement process of crude oil and natural gas, reactors may be used to produce various products from a hydrocarbon feed stream. Reactors may comprise a catalyst which may promote conversion from a reactant to a product. Catalysts may allow for reactions to occur at lower severity (e.g. lower temperature and pressure) and with greater selectivity to desired products by providing an alternative reaction mechanism with a lower activation energy than a non-catalyzed mechanism. Catalysts are a critical component of hydrocarbon processing and catalyst performance may determine quality of the products and profitability of a hydrocarbon processing operation. As such, catalyst performance is often a closely monitored metric in the operation of a reactor. Some common hydrocarbon processing operations that use catalyst may include cracking, such as fluidized catalytic cracking and thermal cracking, hydro processing such as hydro treating and hydrocracking, catalytic reformation, dehydrogenation, and other operations well known in the art.

[0004] Carbon deposits, often referred to as coke, may foul reactors and catalyst therein that process hydrocarbon streams. Coke forming reactions are generally undesirable as the coke that forms may collect on the surface of catalyst and reduce catalytic activity. While process design and process operation may reduce the amount of coke formed, the complete elimination of coke forming reactions may not be possible in all processes. Once a catalyst has been fouled by coke deposits, the catalyst may require regeneration to regain catalytic activity. In addition to coke,

additional contaminants may be deposited on the reactor vessel and catalyst. Contaminants including coke may be referred to collectively as "deposits." The additional contaminants are generally process specific and may include hydrocarbons such as saturated and unsaturated hydrocarbon, aromatic hydrocarbon such as benzene, as well as gums, resins, heavy oil deposits, oligomers, and porphyrins containing nickel and vanadium, for example. In some systems that process sour hydrocarbons, hydrogen sulfide may also be a contaminant.

[0005] Due to the reduction in catalytic activity induced by coke and other deposits, catalysts may be periodically regenerated to regain catalytic activity as part of the reactors operation. In addition to on stream regeneration, most units may be periodically shut down during a plant turnaround to remove fouling and regenerate catalyst. During routine maintenance of equipment such as during a turnaround event, removal of catalyst from equipment and/or entry into the equipment may be necessary. There may be many challenges to removing fouled catalyst such as the catalyst becoming immobile from coke deposition and agglomeration of the catalyst into larger pieces which may impair removal efforts. Coke and other deposits described above may be agglomerated on internals of reactors which may cause the internals to become difficult to remove.

[0006] Furthermore, residual deposits such as coke, H_2S , and other hydrocarbons previously described present in the equipment may pose a fire hazard when the equipment is opened to remove catalyst or for entry. The lower explosive limit (LEL) may be the lowest concentration of a vapor in air capable of producing a fire when exposed to an ignition source. Controlling vapor concentrations within reactors to below the LEL to allow for safe removal of catalyst and vessel entry may be a concern for operators as regulations and safety requirements dictate that LEL must be controlled before a vessel can be opened.

[0007] Various techniques have been developed that enable removal of deposits from catalyst that also reduce vapor concentrations below the LEL. The particular techniques used to remove deposits and reduce hydrocarbon vapor concentration to below LEL may depend upon the particular catalyst and equipment in which the catalyst is present. For example, the catalysts used in hydroprocessing applications may be particularly sensitive and may readily convert to inactive states when exposed to certain regeneration treatments. Furthermore, the de-coking and vapor removal treatment may inadvertently introduce catalyst poison which may reduce the activity of the catalyst after treatment. In some equipment, simple steaming out of units may be performed to remove coke and hydrocarbon vapor. Steaming out may be a generally slow process that typically

may include a unit to be shut down for an extended period of time. Additionally, the excess temperature associated with the steam for decontamination may compact and condense coke from “soft” coke to “hard” coke resulting in tougher deposits than were originally present. The tough hydrocarbon deposits may be removed by mechanical action that may result in longer downtimes or equipment and catalyst damage.

[0008] Other techniques may include a hydrogen sweep followed by a nitrogen purge. The hydrogen sweep method may remove a portion of hydrocarbon vapors and H₂S but may not bring the hydrocarbon vapors and H₂S down to acceptable levels for vessel entry. Another method may comprise a hot hydrogen strip followed by nitrogen. The hot hydrogen may be introduced into the equipment at an elevated temperature, e.g. 700 °F (341 °C) or greater. The high heat and gas flow of hydrogen may break up hydrocarbon deposits and allow the hydrocarbons to flow out of the equipment. A nitrogen purge may then be used to push out any residual hydrogen and hydrocarbons and reduce the hydrocarbon vapors to below the LEL and temperature to an acceptable entry level. Again, the hot hydrogen sweep may not always be effective in reducing LEL and H₂S to below acceptable levels. Furthermore, the hydrogen sweep and hot hydrogen strip is a relatively slow process and may involve several days to complete, thereby increasing the cost associated with the treatment.

[0009] Consequently, there is a need in the art for new methods of deposit removal that also reduce hydrocarbon vapors to below the LEL and allow for de-inventorying of catalyst and safe entry into equipment in a shorter period of time.

BRIEF SUMMARY OF SOME OF THE PREFERRED EMBODIMENTS

[0010] These and other needs in the art are addressed in an embodiment whereby a hot hydrogen stream comprising hydrogen, a fatty acid methyl ester, and an oxygenated solvent are circulated through equipment. The hot hydrogen stream may dissolve and disaggregate at least a portion of deposits collected in the equipment.

[0011] In embodiments, a method of equipment decontamination includes introducing a cleaning stream comprising hydrogen and a solvent comprising a fatty acid methyl ester and an oxygenated solvent into the equipment. The method also includes introducing a stream comprising nitrogen into the equipment, wherein the equipment comprises deposits.

[0012] In other embodiments, a method includes introducing a stream comprising hydrogen into a reactor. The reactor includes a catalyst, deposits on the catalyst, and other contaminants. The method also includes introducing into the stream comprising hydrogen a solvent stream comprising a fatty acid methyl ester and an oxygenated solvent and vaporizing the solvent stream into the stream comprising hydrogen to produce a cleaning stream. The method further includes contacting the deposits with the cleaning stream and removing at least a portion of the deposits and other contaminants with the cleaning stream. In addition, the method includes introducing a nitrogen stream into the reactor.

[0013] The foregoing has outlined rather broadly the features and technical advantages of the present invention in order that the detailed description of the invention that follows may be better understood. Additional features and advantages of the invention will be described hereinafter that form the subject of the claims of the invention. It should be appreciated by those skilled in the art that the conception and the specific embodiments disclosed may be readily utilized as a basis for modifying or designing other embodiments for carrying out the same purposes of the present invention. It should also be realized by those skilled in the art that such equivalent embodiments do not depart from the spirit and scope of the invention as set forth in the appended claims.

BRIEF DESCRIPTION OF THE DRAWINGS

[0014] For a detailed description of the preferred embodiments of the invention, reference will now be made to the accompanying drawings in which:

[0015] FIG. 1 illustrates an embodiment of a system where a solvent composition may be used to remove contaminants from equipment;

[0016] FIG. 2 illustrates an embodiment of the system of FIG. 1 in which the cooling equipment is the heat exchanger.

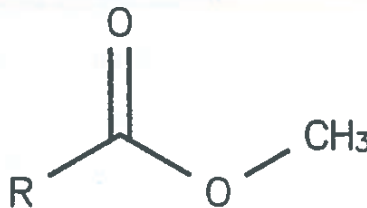
[0017] FIG. 3 is an embodiment of a flow chart detailing a process for removing contaminants from equipment.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0018] As discussed above, in some equipment cleaning techniques including a hot hydrogen sweep and nitrogen purge may be used to clean and regenerate catalysts. In an embodiment of the present application, a solvent composition may be injected into the hot hydrogen stream such that

the solvent composition is vaporized and carried into a reactor alongside the hot hydrogen. In some embodiments, the solvent composition is vaporized and/or dispersed. The addition of the solvent composition may increase the effectiveness of the treatment to remove deposits such as hydrocarbons and H_2S , and lower the concentration of hydrocarbon vapors in the head space. In embodiments, the solvent composition may be used to remove a contaminant material from any industrial equipment or vessel including, but not limited to, reactors, vessels, tanks, vacuum towers, heat exchangers, piping, distillation columns, and the like. Some specific applications may include, but are not limited to, olefins processing, fluid catalytic cracking, hydrotreating, ammonia processing, and other processes that use a catalyst. Further, without limitation, the solvent composition may remove a sufficient amount of contaminant material from the industrial equipment or vessel to allow manned entry in a safe manner. In embodiments, contaminant materials to be removed may include any contaminant material produced, stored, transported, or the like during the process of crude oil refinement, natural gas processing, hydrocarbon transport, hydrocarbon processing, hydrocarbon cleanup, and the like. In embodiments, examples of contaminant materials or deposits may include residual oil, hydrogen sulfide, combustible gas, coke, oligomers, the like, or any combinations thereof. In embodiments, the contaminant materials are contacted with the solvent composition and hot hydrogen, such that the deposits are disaggregated and/or dissolved and may then be subsequently removed from the industrial equipment by flowing the deposits out of the equipment with the hydrogen and/or nitrogen gas.

[0019] In an embodiment, the solvent composition may comprise a fatty acid methyl ester and an oxygenated solvent. The fatty acid methyl ester may be the product of transesterification of soybean oil with methanol, for example. The fatty acid methyl ester may also be a biodiesel or a biodiesel equivalent blend. In some embodiments, the fatty acid methyl ester may comprise structure (1) where R is a C_{14} - C_{18} alkyl group.



(1)

In embodiments, the oxygenated solvent may comprise glycol ethers such as di-propylene glycol, alcohols such as benzyl alcohol, esters such as ethyl lactate, ethoxylated alcohols, glycol ether acetates or combinations thereof. In embodiments, the fatty acid methyl ester and oxygenated solvent may be present in any ratio in the solvent composition. Without limitation, the amount of fatty acid methyl ester and oxygenated solvent may depend on many factors including the identity of the fatty acid methyl ester and oxygenated solvent. In embodiments, the fatty acid methyl ester may be present in an amount ranging between about 70% to about 100% by volume of the solvent composition with the balance volume being the oxygenated solvent or combination of aforementioned oxygenated solvents. Alternatively, the fatty acid methyl ester may be present at a point in a range of about 70% to about 75% by volume, about 75% to about 80% by volume, about 85% to about 90% by volume, about 90% to about 95% by volume, about 95% to about 99.5%, or about 99.5% to about 100% by volume of the solvent composition, or any value in between the explicitly stated ranges. One of ordinary skill in the art with the benefit of this disclosure should be able to select an appropriate identity and amount of fatty acid methyl ester and oxygenated solvent for a particular application.

[0020] The solvent composition may have a boiling point in a range of about 125 °C to about 300 °C depending on the identity and volumetric ratio of the chemical species in the solvent composition. There may be advantages to relatively higher boiling point solvent compositions, namely that vapor phase chemistries have higher performance at relatively higher temperatures. A hot hydrogen strip process using the solvent composition described herein may be operable at relatively higher temperatures of about 260 °C to about 400 °C.

[0021] In an embodiment of the present application, deposits may be removed from equipment by introducing a hot hydrogen stream comprising the solvent composition into the equipment. The solvent composition may be present in any amount in the hot hydrogen stream, depending on various factors such as vessel size, volume of catalyst in the vessel, type and amount of fouling, among many other factors. The deposit removal method may comprise injecting hot hydrogen into the equipment to heat to and/or maintain the equipment at a high temperature, which alongside the solvent composition and hydrogen, may disaggregate and dissolve deposits present in the equipment. The solvent composition may be introduced into the hot hydrogen stream causing the solvent composition to vaporize and be carried into the equipment in a gaseous phase. The gaseous solvent composition may contact deposits in the equipment and cause the deposits to be loosened or become solvated by the solvent. Deposits may become mobile from contact with the solvent composition and may flow out from the equipment. The stream exiting the equipment may be passed to a collection vessel such that the deposits may be collected for disposal. The hydrogen gas stream may be collected, recycled, and heated again so that additional solvent composition may be added. The recycled hydrogen may be passed to the equipment to further remove more deposits. The hot hydrogen may be introduced into the equipment at a temperature of between about 200 °C to about 430 °C, or any temperature in-between, depending on the particular application. The hydrogen stream may be heated by any method including, for example, an electric or a fired heater. One of ordinary skill in the art, with the benefit of this disclosure, should be able to select an appropriate temperature for a particular application.

[0022] Reference will now be made to FIG.1 which illustrates an embodiment of a system 100 where the solvent composition may be used. System 100 may comprise fouled equipment 102, heater 104, heat exchanger 106, separation equipment 108, and additive pump 112. Fouled equipment 102 may comprise industrial equipment or vessels including, but not limited to, reactors, vessels, tanks, vacuum towers, heat exchangers, piping, distillation columns, and the like. Fouled equipment 102 may comprise any of the fouling previously discussed including coke, H₂S, unsaturated hydrocarbons, aromatic hydrocarbons such as benzene, as well as gums, resins, heavy oil deposits, and oligomers, for example. Hydrogen may be introduced through stream 113 into additive pump 112. An output steam from additive pump 112 is input stream 114, which is introduced to heat exchanger 106. Heat exchanger 106 may heat input stream 114 to a desired temperature by when input stream 114 exits as stream 117. Stream 117 from the outlet of heat

exchanger 106 may be passed to heater 104 whereby heater 104 may increase the temperature of the stream 117 to between about 200 °C to about 430 °C, alternatively between about 260°C and about 430°C.

[0023] As previously discussed, a solvent composition may be injected into the hydrogen stream at one or more points prior to the hydrogen stream reaching fouled equipment 102. For example, the solvent composition may be introduced at any of the injection points 121. The solvent composition may be introduced by any suitable means such as by additive pumps 112 as illustrated. As illustrated in FIG. 1, injection points 121 may be before heat exchanger 106, after heat exchanger 106, or after heater 104. In some embodiments, the solvent composition may be introduced into more than one injection point 121. Heated stream 118 comprising hydrogen and solvent composition may be introduced into fouled equipment 102 whereby the hydrogen and solvent may act to loosen and/or dissolve deposits within fouled equipment 102. Equipment output stream 116 comprising hydrogen, solvent composition, dissolved deposits, disaggregated deposits, LELs, benzene, and H₂S may exit fouled equipment 102 and be introduced to cooling equipment 124. Cooling equipment 124 may comprise any equipment configured to cool equipment output stream 116 such that separation of components in equipment output stream 116 may be facilitated. In some embodiments, cooling equipment 124 may include refrigerated heat exchangers, air cooled heat exchangers, or integrated heat exchangers. Cooled stream 119 may exit cooling equipment 124 and be introduced to separation equipment 108. Cooled stream 119 is at a temperature between about 30°C and about 430°C, alternatively between about 80°C and about 280°C.

[0024] Separation equipment 108 may comprise equipment configured to separate the components of cooled stream 119 and separate the hydrogen from the solvent and deposits present in cooled stream 119. Separation equipment 108 may comprise any equipment such as, without limitation, tanks, vessels, coalescers, knock out drums, demister systems, hot separators, cold separators, de-oilers, and processes such as amine columns and caustic towers, which may treat sour components or H₂S present in cooled stream 119. In an embodiment, separation equipment 108 separates hydrogen, hydrogen sulfide, and LEL's from the solvents and deposits. In some embodiments, hydrogen stream 150 including hydrogen, hydrogen sulfide, LEL's or an combinations thereof is directed to flare 155. In embodiments, solvent stream 160 including the solvents and deposits is introduced to collection vessel 165.

[0025] In some embodiments as shown in FIG. 1, after the concentration of deposits in the equipment output stream 116 has reached the desired level, nitrogen stream 150 may be introduced into system 100 through heated stream 118 or be introduced directly into equipment 102 (not illustrated). Nitrogen stream 150 may be introduced by any suitable means. In an embodiment, nitrogen stream 150 is not introduced by a pump. Without limitation, nitrogen stream 150 may push H₂, H₂S, solvent, LELs, benzene, and the like out of equipment 102. In some embodiments, nitrogen stream 150 is not heated. For instance, without limitation, nitrogen stream 150 is not heated in some embodiments because ammonia may be produced at a temperature above 500°F. The nitrogen stream 150 may flow into equipment 102 and displace hydrogen and solvent composition, and may further reduce hydrocarbon concentration in the vapor space in equipment 102 through displacement. Nitrogen stream 150 may be introduced to system 100 until the concentration of hydrocarbons leaving equipment 102 in equipment output stream 116 is at a desired level, generally below the LEL of the hydrocarbons or at a particular ppm measurement for the hydrocarbons. In embodiments, a desired target for H₂S is below about 10 ppm. In an embodiment, a desired target is at about 10% or less LEL.

[0026] In an embodiment of system 100 as shown in FIG. 2, equipment output stream 116 is introduced to heat exchanger 106. In such an embodiment, input stream 114 is introduced to heat exchanger 106 as a cross-flow with equipment output stream 116 with heat exchanger 106 configured to allow heat from equipment output stream 116 to be passed to input stream 114 to heat input stream 114 and cool equipment output stream 116. In such an embodiment, input stream 114 is heated to a desired temperature by when input stream 114 exits heat exchanger 106 as stream 117. Equipment output stream 116 exits heat exchanger 106 as cooled stream 119 and is then introduced to separation equipment 108. Cooled stream 119 is at a temperature between about 30°C and about 430°C, alternatively between about 80°C and about 280°C.

[0027] FIG. 3 is a flowchart illustrating an embodiment of a method 200 of removing deposits from equipment using the previously described solvent composition. In embodiments, method 200 begins at bubble 202. At box 204, hot hydrogen begins to circulate through the equipment comprising the deposits (fouled equipment 102 of Figures 1 and 2) and other auxiliary equipment connected to the equipment comprising the deposits. The hot hydrogen may be at any suitable temperature, including about 200 °C to about 430 °C, or any temperature in-between. The hot hydrogen injection may start out at a relatively higher temperature of about 260 °C to about 430 °C

and the temperature may be stair-stepped or gradually lowered over a period of time to a temperature below about 230 °C. Without limitation, stair-stepped or a gradually lower temperature may facilitate not producing ammonia when switching to nitrogen.

[0028] At box 206, the solvent composition is injected into the hot hydrogen stream to vaporize the solvent composition. The solvent composition may be any solvent composition previously described. The hot hydrogen stream including the solvent composition may enter the equipment (fouled equipment 102 of FIG. 1 and FIG. 2) and auxiliary equipment whereby the hydrogen and solvent composition may contact the deposits and dissolve and/or disaggregate the deposits. The equipment used to introduce the solvent composition into the hot hydrogen stream may comprise pumps, nozzles, and associated control systems to accurately meter the solvent composition into the hot hydrogen stream. The ratio of hydrogen to solvent composition may depend on the chemical identity of the species present in the solvent composition and the equipment the hot hydrogen stream is being introduced into as previously described.

[0029] At box 208, the location of the equipment where the hot hydrogen stream flows out may be monitored for H₂S and/or benzene with a monitoring device to track the progress of deposit removal. The monitoring device may be any suitable monitoring device such as a gas meter and/or a bag sample and a gas chromatograph. In embodiments, the measurement may be done in real time using in line equipment, or aliquots of the stream leaving the equipment may be drawn for later analysis. Analysis may include determining a concentration of H₂S and/or benzene in the aliquot. Although not shown in FIG. 3, the concentration of solvent composition, or the volumetric flow rate of solvent composition into the hot hydrogen stream, may be adjusted to achieve more or less removal of deposits. Temperature of the hot hydrogen stream may also be adjusted in box 208.

[0030] At decision 210, the current concentration of H₂S and/or benzene in the hydrogen stream is compared to the desired concentration. In an embodiment where the solvent composition and hot hydrogen are circulated through the equipment without removing the deposits, a concentration of the deposits may continuously increase until the deposits reach an equilibrium with the solvent composition, or the solvent composition removes all deposits within the equipment. In an embodiment where the solvent composition has deposits removed before re-introduction into the equipment, the concentration of deposits within an outlet stream generally may decrease over time. Whichever method is performed, the concentration of H₂S and/or benzene in an outlet stream may be monitored such that the progress of deposit removal may be observed as the presence of H₂S

and benzene is related to the deposits. In some embodiments, the hot hydrogen and solvent composition may be circulated until the H₂S and/or benzene concentration reaches a steady state where no additional deposit is being removed. In the instance where the H₂S and/or benzene concentration has not reached the desired, or steady state level, the method may proceed to step 214. At step 214, the hydrogen in the outlet stream may be flared off. The method may continue with step 204 to inject more hot hydrogen into the equipment. At decision 210, when the H₂S and/or benzene concentration reaches a desired level, the method may proceed to block 216, wherein the injection of hydrogen gas is discontinued, and the gas stream switches to nitrogen instead.

[0031] At block 218, nitrogen may be metered into the equipment and auxiliary equipment and circulated to displace the hot hydrogen, solvent composition, and dissolved and disaggregated deposits. As previously discussed, the concentration of hydrocarbon vapors in the headspace in an enclosed vessel may exceed the LEL for the hydrocarbon vapors thereby presenting a fire hazard if the vessel is exposed to atmosphere. The supply of nitrogen through the equipment and auxiliary equipment may cause hydrocarbons present to be displaced from the headspace. At decision 220, the LEL for the hydrocarbon vapors may be monitored. If the LEL is not below the allowable limit, step 222 may commence wherein the present nitrogen is flared off. The method may proceed to step 218 to inject more nitrogen gas into the equipment, and nitrogen may be continuously supplied until the LEL for the hydrocarbon vapors falls below a desired value. If the LEL is below the allowable limit, the process may terminate at end step 224.

[0032] To facilitate a better understanding of the present embodiments, the following examples of certain aspects of some embodiments are given. In no way should the following examples be read to limit, or define, the entire scope of the embodiments.

EXAMPLE 1

[0033] Twelve formulations were prepared and tested against a d-Limonene control and another proprietary commercial solvent control to determine each formulation's ability to dissolve asphalt. Blown asphalt from a Texas refinery was chosen for dissolution testing. The asphalt was determined to soften around 175 °C (79.4 °C) and melt around 320 °F (160 °C). Densities of each component are tabulated in Table 1.

Table 1

Species	density (g/ml)
d-Limonene	0.84
Benzyl alcohol	1.04
Dipropylene Glycol	1.02
Ethyl Lactate	1.03

[0034] The samples were prepared according to Table 2. Liquid was prepared in a vial and sonicated for 5 minutes to ensure the mixture was homogeneous. Each liquid was then individually added to a vial containing the blown asphalt. The samples were allowed to sit at room temperature undisturbed. The commercial solvent is a proprietary solvent blend formulated to dissolve hydrogen-deficient hydrocarbon deposits such as asphalts.

Table 2

Vial	Blown Asphalt (g)	Benzyl Alcohol (mL)	Dipropylene Glycol (mL)	Ethyl Lactate (mL)	d-Limonene (mL)	Commercial Solvent (mL)
A	1.26	3.33	3.33	3.33	-	-
B	1.15	6	2	2	-	-
C	1.17	5	5	-	-	-
D	1.11	5	-	5	-	-
E	1.23	-	5	5	-	-
F	1.16	2	2	6	-	-
G	1.14	10	-	-	-	-
H	1.12	-	-	10	-	-
Control	1.28	-	-	-	10	-
I	1.29	-	-	-	-	10

[0035] After five minutes, it was observed that the d-Limonene sample had dissolved the most asphalt followed by the commercial solvent and thereafter samples G>B>C=D>A>F>E=H. Samples E and H did not show any signs of discoloration, and only sample G showed signs of spalling and discoloration. After one hour, sample C had begun to stick to the vial bottom. After two hours, it was observed that the commercial solvent was running, and samples A-H had not progressed. After three hours, the vials were inverted to determine progression. Samples A-H did not show notable improvement over hour two. Sample G was observed to have dark discoloration, and the d-Limonene sample was observed to be opaque. After twenty-two hours, it was observed

that samples A-H had not shown significant improvement. The d-Limonene sample had dissolved approximately 85% of the asphalt, and the commercial solvent had dissolved approximately 30% of the asphalt. After twenty-six hours, the samples were placed into a warm water bath to speed dissolution of the asphalt. After about one hour in the warm water bath, the d-Limonene sample was completely dissolved. The commercial sample (vial I) continued to dissolve, and samples B, D, and G showed signs of darkening and spalling. After twenty-four hours in the hot water bath, samples A through H were observed to not have significantly improved. The samples were removed from the warm water bath and allowed to sit at ambient temperature for a period of six days. It was observed that samples A through H did not show much more dissolution after six days.

EXAMPLE 2

[0036] A sample was prepared according to Table 3.

Table 3

Vial	Blown Asphalt (g)	Dipropylene Glycol (mL)	Soy methyl ester (mL)
J	1.21	0.4	9.4

[0037] The liquids were sonicated to mix, and the blown asphalt was added to vial J. It was observed that the solution became opaque within minutes. After about ten minutes, it was observed that about 60% of the asphalt had dissolved. After about five hours, it was observed that about 20% of the asphalt remained. Sample J was left overnight to allow further dissolution. It was observed that after about 20.5 hours, sample J was about 95% dissolved. After about forty-eight hours it was observed that sample J was completely dissolved.

EXAMPLE 3

[0038] Samples were prepared according to Table 4.

Table 4

Vial	Blown Asphalt (g)	Benzyl Alcohol (mL)	Ethyl Lactate (mL)	Soy methyl ester (mL)
K	1.19	2	-	8

L	1.29	-	2	8
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[0039] The liquids were sonicated to mix, and the blown asphalt was added to each sample. It was observed that after about five minutes, samples K and L were over 50% dissolved. After forty-five minutes, K and L both appeared to have about 70-75% dissolved. After about twenty-six hours, it was observed that sample L was completely dissolved, and sample K had dissolved approximately 95%.

EXAMPLE 4

[0040] Zeolite samples were prepared by filling four vials 3/5 full of 8 x 14 mesh zeolite, 2.5 full of 6 x 8 mesh zeolite, and 1/5 full of 1/4 x 6 mesh zeolite. The Zeolite was natural zeolite (clinoptilolite). Each vial was poured into a separate beaker and mixed thoroughly. Blown asphalt was added to each vial and the vials were heated to 85 °C. The temperature was increased up to 215 °C with periodic stirring until it was observed that the asphalt uniformly coated the zeolite mixture.

[0041] While the coated zeolite sample mixture was still hot, sample aliquots were taken and added to vials. Solvent compositions were prepared according to Table 5. All vials were sonicated for 5 minutes after preparation to ensure homogeneous mixing.

Table 5

Vial	Blown Asphalt (g)	Benzyl Alcohol (mL)	Dipropylene Glycol (mL)	Ethyl Lactate (mL)	d-Limonene (mL)	Soy Methyl Ester (mL)
1	2.69	-	-	-	10	-
2	2.54	-	0.4	-	-	9.6
3	2.55	2	-	-	-	8
4	2.53	-	-	2	-	8

[0042] All vials were placed in a hot water bath at 55 °C. After about thirty minutes, it was observed that vial 3 did not contain a single large agglomeration while samples 1, 2, and 3 contained a single large agglomeration. At that point, all vials were relocated to a hot mineral oil bath. After about thirty minutes, the vials were observed to be at about 93 °C. It was observed that no agglomerations in any of the vials were large enough to breach the liquid level line when the vials were turned sideways, and each vial now contained smaller agglomerations and fines. The

zeolite appeared to still be coated in each vial. The set point of the hot mineral oil bath was increased.

[0043] After about forty-five minutes, it was observed that vials 1 and 3 started boiling when the hot mineral oil bath reached about 126 °C. It was observed that the zeolite of vials 1, 2, and 4 tended to clump and slide as a large mass with some residual fines while vial 3 had more spalling and larger fines than the other vials. After about three hours, the experiment was paused and the vials were removed from the hot mineral oil bath and allowed to cool. Once cooled, the zeolite from each vial was poured onto a separate watch glass for further observation. It was found that the zeolite from vials 3 and 4 had similar feel regarding ease of separability and residual solvent cohesive properties. Zeolite from vials 1 and 2 were found to have similar ease of separability and were observed to be easier to separate than zeolite from vials 3 and 4.

[0044] Each of the four samples of zeolite was allowed to dry for four days on a watch glass. Each of the samples were replaced in their respective vials and placed into a hot mineral oil bath with a set point of 170 °C. After about five hours, the samples were placed back onto their respective watch glasses, and it was observed that the cohesion of zeolite from vial 3 was less than the zeolite from vial 2, which was about equal to the cohesion of zeolite from vial 4 and which was less than the cohesion of the zeolite from vial 1.

EXAMPLE 5

[0045] Coated zeolite samples were prepared by allowing zeolite to soak in a container of Australian crude oil for a week at room temperature. The crude oil was drained from the zeolite samples and five vials were prepared according to Table 6.

Table 6

Vial	Coated Zeolite (g)	Benzyl Alcohol (mL)	Dipropylene Glycol (mL)	Ethyl Lactate (mL)	d-Limonene (mL)	Soy Methyl Ester (mL)	Ethoxylated Alcohol (mL)
1	2.03	-	-	-	10	-	-
2	2.04	-	0.4	-	-	9.6	-
3	1.98	2	-	-	-	8	-
4	1.97	-	-	2	-	8	-
5	1.99	-	-	-	-	9.6	0.4

[0046] Each of the five vials was placed in a hot mineral oil bath at 120 °C. After about an hour, each vial was removed from the bath and swirled. After about four hours, the vials were removed from the bath and allowed to cool. Each sample was observed for whiteness and by extension, how much oil had been removed from the zeolite. It was observed that the most oil was removed from vial 2 and 4, which were approximately equal. It was further observed that vial 5 performed better than vial 3, which performed better than vial 1.

EXAMPLE 6

[0047] The remaining coated zeolite sample from Example 4 was heated to 163 °C and 28.5 grams of 6 x 8 mesh clinoptilolite natural zeolite and 8.03 grams of blown asphalt was added. The zeolite was stirred until a uniform coating was observed. Samples were prepared according to Table 7.

Table 7

Vial	Zeolite (g)	Benzyl Alcohol (mL)	Dipropylene Glycol (mL)	Ethyl Lactate (mL)	d-Limonene (mL)	Soy Methyl Ester (mL)	Ethoxylated Alcohol (mL)
1	2.01	-	-	-	10	-	-
2	2.00	-	0.4	-	-	9.6	-
3	2.02	-	-	-	-	9.6	0.4
4	1.99	2	-	-	-	8	-
5	2.02	-	-	2	-	8	-
6	2.02	1	-	1	-	8	-
7	2.04	0.5	-	-	-	9.5	-
8	2.03	-	-	0.5	-	9.5	-

[0048] After preparation, each vial was placed in a sonicator for ten minutes. The vials were then placed in an oven at 110 °C. The vials were removed from the oven after about forty-five minutes. It was observed that vials 1, 4, 5, and 7 flowed the easiest while vials 2, 3, and 8 flowed slightly less. Only vial 6 was observed to have packed and settled fines at the bottom. The vials were placed back into the oven. After a total of four hours in the oven, the vials were taken out, observed, and allowed to cool overnight. While hot, the vials did not show a perceptible difference. After about fifteen hours, the zeolite from each vial was removed and placed on a paper towel to remove excess solution and thereafter transferred to individual watch glasses. Although difficult to determine for every sample, the overall amount of residue removed was found to be least for vial 4,

and second least for vials 1 and 2. Due to the difficulty in determining the remaining vials by visual inspection, two alternate rankings were performed. The first ranked the vial solutions' effectiveness in order based on flow of residue, from most to least flow, and the ranking was as such: vial $4 > 5 > 3 = 7 > 6 > 1 \geq 2 > 8$. The second ranking examined how the treated catalyst behaved in solution. For this, each sample of zeolite was placed back into the respective vial, and the cold cohesive behaviors were found to be in order of least cohesive to most cohesive to be $4 \leq 1 \leq 2 < 5 \approx 6 < 3 \approx 7 < 8$. The vials were allowed to sit undisturbed for approximately 2.5 weeks after which the vials were each carefully tilted to about 75° , and the zeolite behavior was observed. It was found that vials 1, 2, and 3 easily flowed off the vial bottom; vials 4, 6, and 8 had about 1/3 of the zeolite stick to the vial bottom; and vials 5 and 7 had a majority of the zeolite stay on the vial bottom and remain in place.

[0049] It should be understood that the compositions and methods are described in terms of "comprising," "containing," or "including" various components or steps, the compositions and methods can also "consist essentially of" or "consist of" the various components and steps. Moreover, the indefinite articles "a" or "an," as used in the claims, are defined herein to mean one or more than one of the element that it introduces.

[0050] For the sake of brevity, only certain ranges are explicitly disclosed herein. However, ranges from any lower limit may be combined with any upper limit to recite a range not explicitly recited, as well as, ranges from any lower limit may be combined with any other lower limit to recite a range not explicitly recited, in the same way, ranges from any upper limit may be combined with any other upper limit to recite a range not explicitly recited. Additionally, whenever a numerical range with a lower limit and an upper limit is disclosed, any number and any included range falling within the range are specifically disclosed. In particular, every range of values (of the form, "from about a to about b," or, equivalently, "from approximately a to b," or, equivalently, "from approximately a-b") disclosed herein is to be understood to set forth every number and range encompassed within the broader range of values even if not explicitly recited. Thus, every point or individual value may serve as its own lower or upper limit combined with any other point or individual value or any other lower or upper limit, to recite a range not explicitly recited.

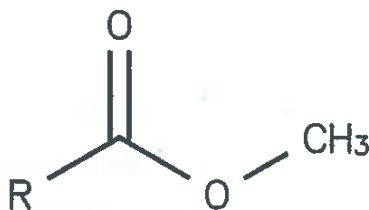
[0051] Therefore, the present invention is well adapted to attain the ends and advantages mentioned as well as those that are inherent therein. The particular embodiments disclosed above are illustrative only, as the present invention may be modified and practiced in different but

equivalent manners apparent to those skilled in the art having the benefit of the teachings herein. Although individual embodiments are discussed, the invention covers all combinations of all those embodiments. Furthermore, no limitations are intended to the details of construction or design herein shown, other than as described in the claims below. Also, the terms in the claims have their plain, ordinary meaning unless otherwise explicitly and clearly defined by the patentee. It is therefore evident that the particular illustrative embodiments disclosed above may be altered or modified and all such variations are considered within the scope and spirit of the present invention. If there is any conflict in the usages of a word or term in this specification and one or more patent(s) or other documents that may be incorporated herein by reference, the definitions that are consistent with this specification should be adopted.

CLAIMS

What is claimed is:

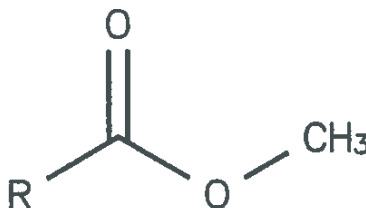
1. A method of equipment decontamination comprising:
introducing a cleaning stream comprising hydrogen and a solvent comprising a fatty acid methyl ester and an oxygenated solvent into the equipment; and
introducing a stream comprising nitrogen into the equipment, wherein the equipment comprises deposits.
2. The method of claim 1 further comprising introducing a solvent-free hydrogen before the step of introducing the cleaning stream, wherein the solvent-free hydrogen stream is at a temperature of about 260 °C to about 430 °C.
3. The method of claim 1 wherein the step of introducing the cleaning stream comprises:
injecting the solvent into a hydrogen stream; and
vaporizing and/or dispersing the solvent with the hydrogen stream.
4. The method of claim 1 wherein the fatty acid methyl ester is a product of transesterification of soy oil with methanol.
5. The method of claim 1 wherein the fatty acid methyl ester comprises a fatty acid methyl ester with the following structure:



wherein R is a C₁₄-C₁₈ alkyl group.

6. The method of claim 1 wherein the oxygenated solvent comprises a solvent selected from the group consisting of di-propylene glycol, benzyl alcohol, ethyl lactate, ethoxylated alcohols, glycol ether acetates, and combinations thereof.
7. The method of claim 1 wherein the fatty acid methyl ester is present in an amount of about 70% to about 100% by volume of the solvent.
8. The method of claim 1 further comprising:
 - absorbing the deposits, disaggregating the deposits, or any combinations thereof;
 - and
 - generating a bottoms stream comprising the hydrogen, the solvent, and the absorbed and/or disaggregated deposits.
9. The method of claim 8 further comprising:
 - removing at least a portion of the solvent and the absorbed and/or disaggregated deposits.
10. The method of claim 1 wherein the equipment is a reactor comprising a catalyst and wherein the method further comprises removing at least a portion of the catalyst after the step of introducing the stream comprising the nitrogen.
11. A method comprising:
 - introducing a stream comprising hydrogen into a reactor, wherein the reactor comprises a catalyst, deposits on the catalyst, and other contaminants;
 - introducing into the stream comprising hydrogen a solvent stream comprising a fatty acid methyl ester and an oxygenated solvent and vaporizing the solvent stream into the stream comprising hydrogen to produce a cleaning stream;
 - contacting the deposits with the cleaning stream and removing at least a portion of the deposits and/or other contaminants with the cleaning stream; and
 - introducing a nitrogen stream into the reactor.

12. The method of claim 11 wherein the hydrogen stream is at a temperature of about 260 °C to about 430 °C.
13. The method of claim 11 wherein the deposits comprise at least one of an unsaturated hydrocarbon, an aromatic hydrocarbon, a hydrocarbon based gum, a hydrocarbon based resin, a heavy oil, or oligomers.
14. The method of claim 11 wherein the other contaminants are comprised of, but not limited to, hydrogen sulfide, benzene, LELs, or combinations thereof.
15. The method of claim 11 wherein the fatty acid methyl ester comprises a fatty acid methyl ester with the following structure:



wherein R is a C₁₄-C₁₈ alkyl group.

16. The method of claim 11 wherein the fatty acid methyl ester is a product of transesterification of soy oil with methanol.
17. The method of claim 11 wherein the oxygenated solvent comprises a solvent selected from the group consisting of di-propylene glycol, benzyl alcohol, ethyl lactate, ethoxylated alcohols, glycol ether acetates, and combinations thereof.

18. The method of claim 11 further comprising:
generating a bottoms stream from the reactor comprising the cleaning stream and the removed portion of the deposits; and
removing the deposits and at least a portion of the solvent from the bottoms stream to produce a hydrogen waste stream.
19. The method of claim 11 further comprising flaring nitrogen and hydrogen.
20. The method of claim 11 further comprises removing at least a portion of the catalyst and any residual contaminants after the step of introducing the stream comprising the nitrogen.

ABSTRACT

A method of equipment decontamination may include: introducing a cleaning stream comprising hydrogen and a solvent comprising a fatty acid methyl ester and an oxygenated solvent into the equipment; and introducing a stream comprising nitrogen into the equipment, wherein the equipment comprises deposits and other contaminants.

Figs. 1

100

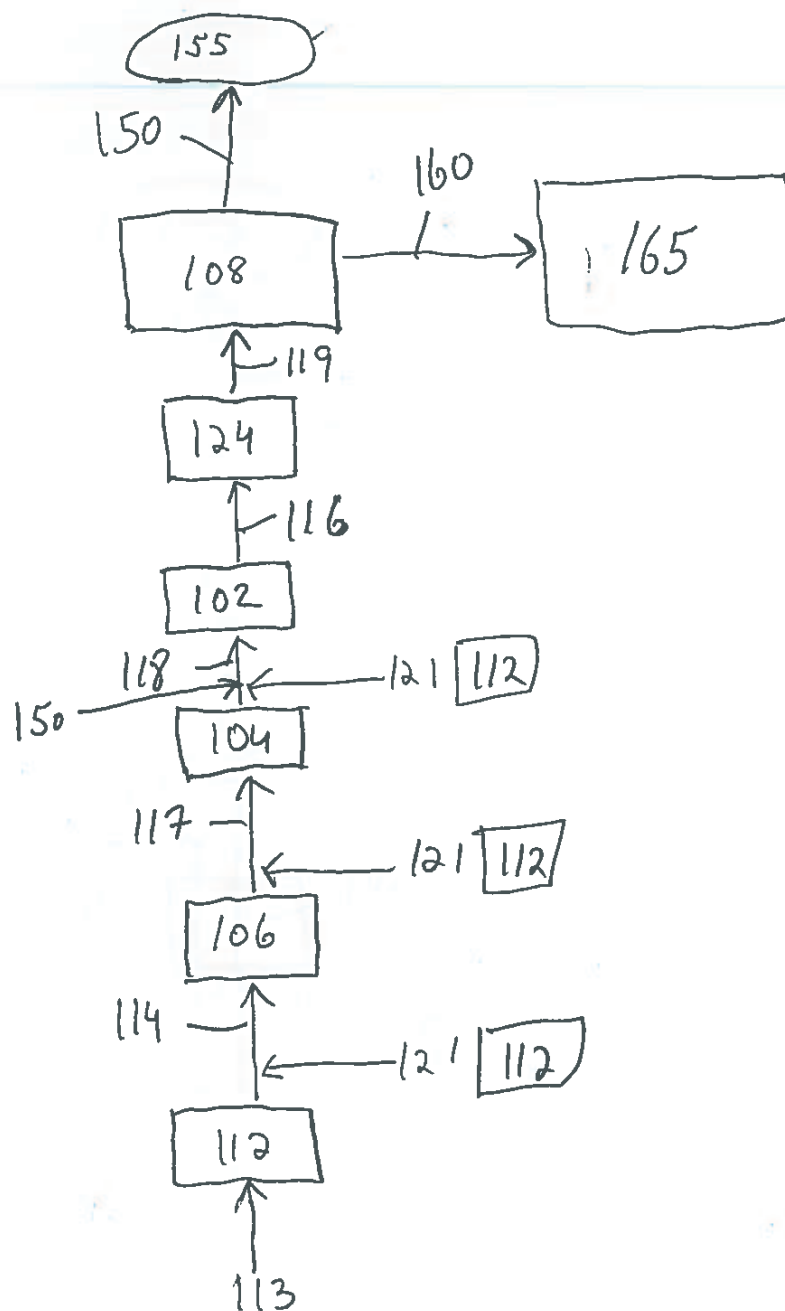
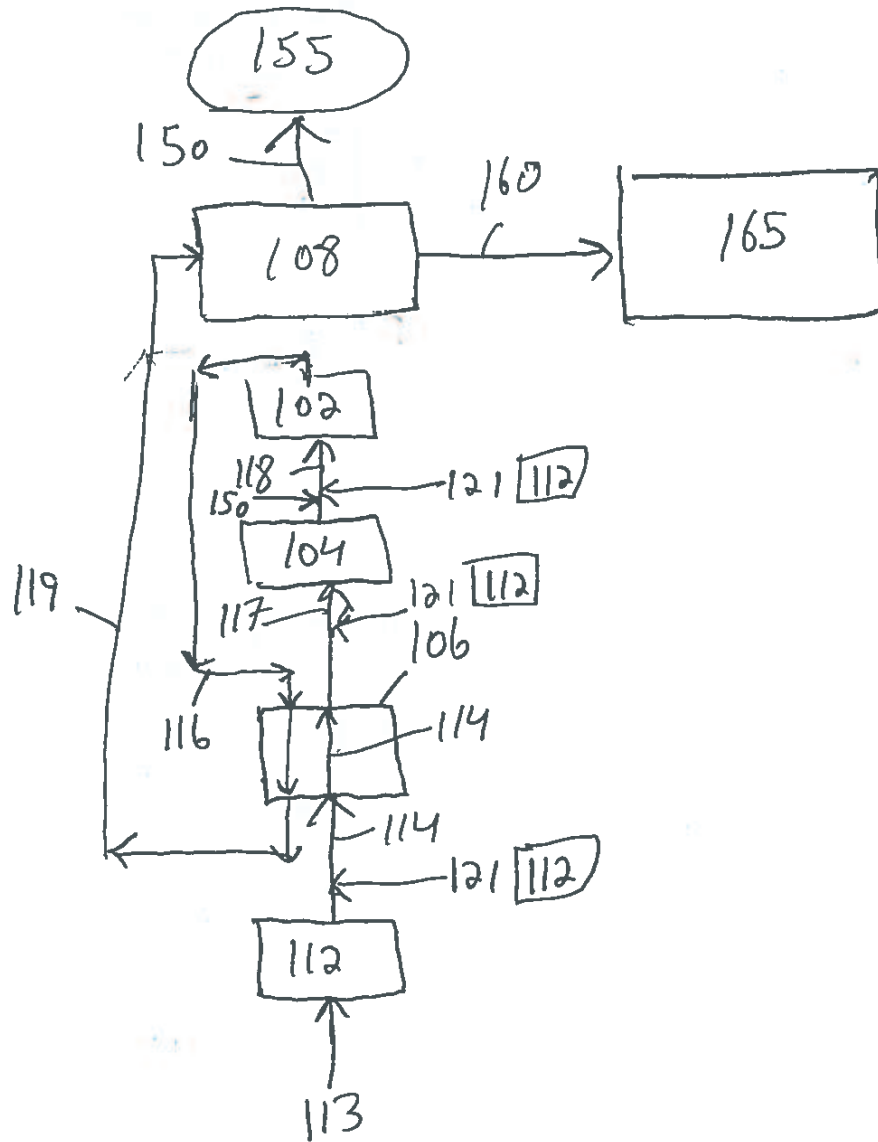


Fig. 2

100 \



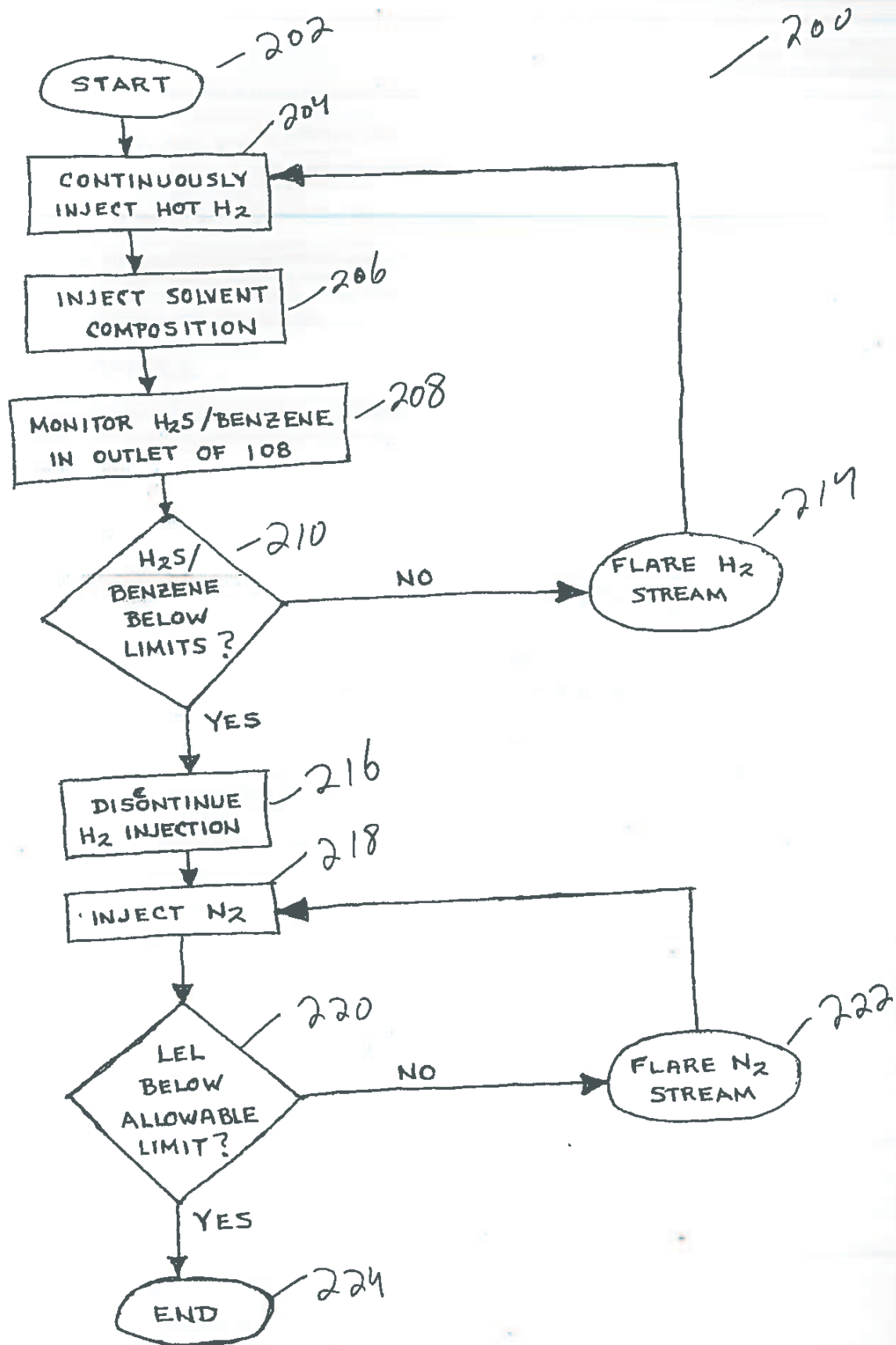


FIG. 3

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	1540-20400
		Application Number	
Title of Invention	Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ		
The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76. This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.			

Secrecy Order 37 CFR 5.2:

☐ Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.)

Inventor Information:

Inventor 1						Remove	
Legal Name							
Prefix	Given Name	Middle Name		Family Name		Suffix	
	Stephen	D.		Matza			
Residence Information (Select One) ☒ US Residency ☐ Non US Residency ☐ Active US Military Service							
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Address 2							
City	Sugar Land	State/Province	TX				
Postal Code	77478	Country	US				
Inventor 2						Remove	
Legal Name							
Prefix	Given Name	Middle Name		Family Name		Suffix	
	Elisa			Rice			
Residence Information (Select One) ☒ US Residency ☐ Non US Residency ☐ Active US Military Service							
City	Houston	State/Province	TX	Country of Residence	US		
Mailing Address of Inventor:							
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Address 2							
City	Houston	State/Province	TX				
Postal Code	77034	Country	US				
All Inventors Must Be Listed - Additional Inventor Information blocks may be generated within this form by selecting the Add button. Add							

Correspondence Information:

Enter either Customer Number or complete the Correspondence Information section below.
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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	1540-20400
	Application Number	
Title of Invention	Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ	

☐ **An Address is being provided for the correspondence information of this application.**
Customer Number 62763

Email Address ttumey@tumeyllp.com

Application Information:

Title of the Invention	Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ		
Attorney Docket Number	1540-20400	Small Entity Status Claimed	☒
Application Type	Nonprovisional		
Subject Matter	Utility		
Total Number of Drawing Sheets (if any)	3	Suggested Figure for Publication (if any)	1

Filing By Reference:

Only complete this section when filing an application by reference under 35 U.S.C. 111(c) and 37 CFR 1.57(a). Do not complete this section if application papers including a specification and any drawings are being filed. Any domestic benefit or foreign priority information must be provided in the appropriate section(s) below (i.e., "Domestic Benefit/National Stage Information" and "Foreign Priority Information").

For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

Application number of the previously filed application	Filing date (YYYY-MM-DD)	Intellectual Property Authority or Country

Publication Information:

☐ **Request Early Publication** (Fee required at time of Request 37 CFR 1.219)

☐ **Request Not to Publish.** I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application has not and will not be the subject of an application filed in another country, or under a multilateral international agreement, that requires publication at eighteen months after filing.

Representative Information:

Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32). Either enter Customer Number or complete the Representative Name section below. If both sections are completed the customer Number will be used for the Representative Information during processing.

Please Select One:	☒ Customer Number	☐ US Patent Practitioner	☐ Limited Recognition (37 CFR 11.9)
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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	1540-20400
	Application Number	
Title of Invention	Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ	

Domestic Benefit/National Stage Information:

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, 365(c), or 386(c) or indicate National Stage entry from a PCT application. Providing benefit claim information in the Application Data Sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78.

When referring to the current application, please leave the "Application Number" field blank.

Prior Application Status			Remove
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)

Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the Add button.

Foreign Priority Information:

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55. When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX)ⁱ the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(i)(1) and (2). Under the PDX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

			Remove
Application Number	Country ⁱ	Filing Date (YYYY-MM-DD)	Access Code ⁱ (if applicable)

Additional Foreign Priority Data may be generated within this form by selecting the Add button.

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

- ☐ This application (1) claims priority to or the benefit of an application filed before March 16, 2013 and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013.
- NOTE: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	1540-20400
		Application Number	
Title of Invention	Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ		

Authorization or Opt-Out of Authorization to Permit Access:

When this Application Data Sheet is properly signed and filed with the application, applicant has provided written authority to permit a participating foreign intellectual property (IP) office access to the instant application-as-filed (see paragraph A in subsection 1 below) and the European Patent Office (EPO) access to any search results from the instant application (see paragraph B in subsection 1 below).

Should applicant choose not to provide an authorization identified in subsection 1 below, applicant **must opt-out** of the authorization by checking the corresponding box A or B or both in subsection 2 below.

NOTE: This section of the Application Data Sheet is **ONLY** reviewed and processed with the **INITIAL** filing of an application. After the initial filing of an application, an Application Data Sheet cannot be used to provide or rescind authorization for access by a foreign IP office(s). Instead, Form PTO/SB/39 or PTO/SB/69 must be used as appropriate.

1. Authorization to Permit Access by a Foreign Intellectual Property Office(s)

A. Priority Document Exchange (PDX) - Unless box A in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the State Intellectual Property Office of the People's Republic of China (SIPO), the World Intellectual Property Organization (WIPO), and any other foreign intellectual property office participating with the USPTO in a bilateral or multilateral priority document exchange agreement in which a foreign application claiming priority to the instant patent application is filed, access to: (1) the instant patent application-as-filed and its related bibliographic data, (2) any foreign or domestic application to which priority or benefit is claimed by the instant application and its related bibliographic data, and (3) the date of filing of this Authorization. See 37 CFR 1.14(h)(1).

B. Search Results from U.S. Application to EPO - Unless box B in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the EPO access to the bibliographic data and search results from the instant patent application when a European patent application claiming priority to the instant patent application is filed. See 37 CFR 1.14(h)(2).

The applicant is reminded that the EPO's Rule 141(1) EPC (European Patent Convention) requires applicants to submit a copy of search results from the instant application without delay in a European patent application that claims priority to the instant application.

2. Opt-Out of Authorizations to Permit Access by a Foreign Intellectual Property Office(s)

☐ A. Applicant **DOES NOT** authorize the USPTO to permit a participating foreign IP office access to the instant application-as-filed. If this box is checked, the USPTO will not be providing a participating foreign IP office with any documents and information identified in subsection 1A above.

☐ B. Applicant **DOES NOT** authorize the USPTO to transmit to the EPO any search results from the instant patent application. If this box is checked, the USPTO will not be providing the EPO with search results from the instant application.

NOTE: Once the application has published or is otherwise publicly available, the USPTO may provide access to the application in accordance with 37 CFR 1.14.

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	1540-20400
		Application Number	
Title of Invention	Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ		

Applicant Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

Applicant 1

If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section.

- ☒ Assignee
 ☐ Legal Representative under 35 U.S.C. 117
 ☐ Joint Inventor
- ☐ Person to whom the inventor is obligated to assign.
 ☐ Person who shows sufficient proprietary interest

If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:

Name of the Deceased or Legally Incapacitated Inventor:

If the Applicant is an Organization check here. ☒

Organization Name United Laboratories International, LLC

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State/Province

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Postal Code

77034

Phone Number

Fax Number

Email Address

Additional Applicant Data may be generated within this form by selecting the Add button.

Assignee Information including Non-Applicant Assignee Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	1540-20400
		Application Number	
Title of Invention	Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ		

Assignee 1

Complete this section if assignee information, including non-applicant assignee information, is desired to be included on the patent application publication. An assignee-applicant identified in the "Applicant Information" section will appear on the patent application publication as an applicant. For an assignee-applicant, complete this section only if identification as an assignee is also desired on the patent application publication.

If the Assignee or Non-Applicant Assignee is an Organization check here. ☒

Organization Name	United Laboratories International, LLC
-------------------	--

Mailing Address Information For Assignee including Non-Applicant Assignee:

Address 1	12600 North Featherwood, Suite 330		
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Address 2			
-----------	--	--	--

City	Houston	State/Province	TX
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Country ⁱ	US	Postal Code	77034
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Phone Number		Fax Number	
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Email Address	
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Additional Assignee or Non-Applicant Assignee Data may be generated within this form by selecting the Add button.

Signature:

NOTE: This Application Data Sheet must be signed in accordance with 37 CFR 1.33(b). However, if this Application Data Sheet is submitted with the **INITIAL** filing of the application and either box A or B is **not** checked in subsection 2 of the "Authorization or Opt-Out of Authorization to Permit Access" section, then this form must also be signed in accordance with 37 CFR 1.14(c).

This Application Data Sheet **must** be signed by a patent practitioner if one or more of the applicants is a **juristic entity** (e.g., corporation or association). If the applicant is two or more joint inventors, this form must be signed by a patent practitioner, **all** joint inventors who are the applicant, or one or more joint inventor-applicants who have been given power of attorney (e.g., see USPTO Form PTO/AIA/81) on behalf of **all** joint inventor-applicants.

See 37 CFR 1.4(d) for the manner of making signatures and certifications.

Signature	/ttumey/		Date (YYYY-MM-DD)	2019-03-01
First Name	Tod	Last Name	Tumey	Registration Number
Additional Signature may be generated within this form by selecting the Add button.				

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	1540-20400
		Application Number	
Title of Invention	Solvent System for Cleaning Fixed Bed Reactor Catalyst In Situ		

This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

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THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office

June 12, 2019

THIS IS TO CERTIFY THAT ANNEXED IS A TRUE COPY FROM THE
RECORDS OF THIS OFFICE OF A DOCUMENT RECORDED ON
MAY 28, 2019.

By Authority of the
Under Secretary of Commerce for Intellectual Property
and Director of the United States Patent and Trademark Office



JOHN A BURSON
Certifying Officer

PATENT ASSIGNMENT COVER SHEET

Electronic Version v1.1
Stylesheet Version v1.2

EPAS ID: PAT5542902

SUBMISSION TYPE:	NEW ASSIGNMENT
NATURE OF CONVEYANCE:	ASSIGNMENT
CONVEYING PARTY DATA	
Name	Execution Date
STEPHEN D. MATZA	05/08/2019
ELISA RICE	05/08/2019
RECEIVING PARTY DATA	
Name:	UNITED LABORATORIES INTERNATIONAL LLC
Street Address:	12600 NORTH FEATHERWOOD
Internal Address:	SUITE 330
City:	HOUSTON
State/Country:	TEXAS
Postal Code:	77034
PROPERTY NUMBERS Total: 1	
Property Type	Number
Application Number:	16290679
CORRESPONDENCE DATA	
Fax Number:	(713)622-0220
<i>Correspondence will be sent to the e-mail address first; if that is unsuccessful, it will be sent using a fax number, if provided; if that is unsuccessful, it will be sent via US Mail.</i>	
Phone:	7136227005
Email:	asalazar@tumeyllp.com
Correspondent Name:	TOD TUMEY
Address Line 1:	P.O. BOX 22188
Address Line 4:	HOUSTON, TEXAS 77227-2188
ATTORNEY DOCKET NUMBER:	1540-20400
NAME OF SUBMITTER:	TOD TUMEY
SIGNATURE:	/ttumey/
DATE SIGNED:	05/28/2019
Total Attachments: 2	
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source=1540-20400_Assignment-executed#page2.tif	

ASSIGNMENT

FOR GOOD AND VALUABLE CONSIDERATION, the receipt and sufficiency of which are hereby acknowledged, we, **Stephen D. Matza and Elisa Rice** ("ASSIGNORS"), having made an invention in **Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ**, while in the employ either as an employee or consultant of **United Laboratories International, LLC**, a limited liability company organized, existing, and doing business at 12600 North Featherwood, Suite 330, Houston, TX 77034 (sometimes hereinafter called "ASSIGNEE"), do hereby ASSIGN, SELL and CONVEY to said **United Laboratories International, LLC**, its successors and assigns, the entire right, title and interest throughout the world in and to:

1. Said invention in **Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ**;
2. United States of America patent application on said invention, Application No. 16/290,679 filed March 1, 2019, entitled **Solvent System for Cleaning Fixed Bed Reactor Catalyst in Situ**;
3. All applications for patent or like protection on said invention that have now been or may in the future be made by us or our legal representatives, including any continuation, continuation-in-part and any other utility applications that may be based on this invention, whether in the United States of America or any other place anywhere in the world;
4. All patents and like protection that have now been or may in the future be granted on said invention to us or our legal representatives, whether in the United States of America or in any other country or place anywhere in the world;
5. All substitutions for and divisions, continuations, continuations-in-part, renewals, reissues, extensions, and the like of said applications and patents and like grants, including without limitation, those obtained or permissible under past, present and future laws and statutes;
6. All rights of action on account of past, present and future unauthorized use of said invention and for infringement of said patents and like protection;

7. The right in ASSIGNEE to file in its name applications for patents and like protection for said invention in any country or countries foreign to the United States; and
8. All international rights of priority associated with said invention, applications, patents and like protection; and we covenant that we, and our heirs, legal representatives, assigns, administrators, and executors, will, at the expense of ASSIGNEE, its successors and assigns, execute all papers and perform such other acts as may be reasonably necessary to give ASSIGNEE, its successors and assigns, the full benefit of this Assignment.

EXECUTED on the date indicated below, opposite our signatures.

ASSIGNORS

Date: MAY 8, 2019

Stephen D. Matza
Stephen D. Matza

Date: May 8, 2019

Elisa Rice
Elisa Rice

P-2019/19633

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
DIRECCIÓN DE NUEVAS CREACIONES

E. S. D.

Ref.: Expediente No. NC2019/0005730

Solicitud de registro de patente de invención a nombre de **UNITED LABORATORIES INTERNATIONAL, LLC**

Yo, CAROLINA DAZA MONTALVO, identificada como aparece al pie de mi firma, domiciliada en la Carrera 11 No. 86-53, piso 3, obrando como apoderada de la solicitante de la patente de invención de la referencia, **UNITED LABORATORIES INTERNATIONAL, LLC**, en respuesta al Oficio No. **4824** del 18 de junio de 2019, atentamente me permito presentar el siguiente documento:

1. **Análisis de prioridad:** Se anexa la copia certificada de la prioridad 16/290,679.
2. **Cesión:** Así mismo, se allega la cesión de derechos de los inventores, con su respectiva traducción a español.

Habiendo dado cumplimiento al requerimiento de este Despacho, solicito respetuosamente se continúe con el trámite de la referencia.

Atentamente,



CAROLINA DAZA MONTALVO

C.C. 66.783.790 de Palmira Valle

TP-141563 CSJ

notificaciones@clarkemodet.com.co

TRADUCCIÓN OFICIAL No. 129/CKM REALIZADA POR DIEGO F. VARGAS ZAMORA, TRADUCTOR E
INTÉRPRETE OFICIAL INGLÉS-ESPAÑOL, RECONOCIDO MEDIANTE RESOLUCIÓN No. 0001 DEL 4 DE
ENERO DE 1999 DEL MINISTERIO DE JUSTICA



LOS ESTADOS UNIDOS DE AMÉRICA

A TODOS AQUELLOS A QUIENES LOS PRESENTES LLEGAREN:

DEPARTAMENTO DE COMERCIO DE LOS ESTADOS UNIDOS
Oficina de Marcas y Patentes de los Estados Unidos

12 de junio de 2019

POR EL PRESENTE SE CERTIFICA QUE LA ADJUNTA ES
UNA ES UNA COPIA FIEL DE LOS ARCHIVOS DE ESTA
OFICINA DE UN DOCUMENTO INSCRITO EL 28 DE MAYO
DE 2019.

Por Autoridad del
Subsecretario de Comercio para la Propiedad
Intelectual y Director de la Oficina de Marcas
y Patentes de los Estados Unidos



Firma Ilegible
JOHN A BURSON
Funcionario Certificador

DIEGO F. VARGAS ZAMORA
TRADUCTOR E INTÉRPRETE OFICIAL

505496098 05/28/2019

PORTADA DE CESIÓN DE PATENTE

Versión Electrónica v1.1

EPAS ID: PAT5542902

Versión Hoja de estilo v1.2

TIPO DE PRESENTACIÓN:	NUEVA CESIÓN
NATURALEZA DE ENAJENACIÓN:	CESIÓN
INFORMACIÓN PARTE CEDENTE	
Nombre	Fecha Suscripción
STEPHEN D. MATZA	05/28/2019
ELISA RICE	05/28/2019
INFORMACIÓN PARTE RECEPTORA	
Nombre:	UNITED LABORATORIES INTERNATIONAL, LLC
Dirección Externa:	12600 NORTH FEATHERWOOD
Dirección Interna:	SUITE 330
Ciudad:	HOUSTON
Estado/País	TEXAS
Código Postal	77034
NÚMERO DE BIENES Total: 1	
Tipo de bien	Número
Solicitud Número:	16290679
INFORMACIÓN DE CORRESPONDENCIA:	
Número Fax:	(713) 622-0220
<i>Se enviará la correspondencia primero a la dirección de correo electrónico; si es infructuoso, se enviará empleando un número de fax, si fue suministrado, si esto es infructuoso, se enviará por Correo de los Estados Unidos.</i>	
Teléfono:	7136227005
Correo electrónico:	asalazar@tumeyp.com
Nombre del Corresponsal:	TOD TUMEY
Dirección Línea 1:	P.O. BOX 22188
Dirección Línea 4:	HOUSTON, TEXAS 77227-2188
REFERENCIA DEL APODERADO:	1540-20400
NOMBRE DEL REMITENTE:	TOD TUMEY
Firma	/ttumey/
Fecha:	05/28/2019
Total Adjuntos: 2	
Source:1540-20400_Assignment-executed#page1.tif	
Source:1540-20400_Assignment-executed#page2.tif	

CESIÓN

POR CONTRAPRESTACIÓN LEGAL Y VÁLIDA, el recibo y suficiencia de la cual se reconoce por el presente, nosotros, Stephen D. Matza y Elisa Rice ("CEDENTES"), quienes realizamos una invención relativa a un Sistema de Solventes para Limpieza in Situ de Catalizadores en un Reactor de Lecho Fijo, durante nuestra vinculación, ya sea como trabajadores o consultores de United Laboratories International, LLC, una compañía de responsabilidad limitada constituida, existente y que realiza negocios en la dirección 12600 North Featherwood, Suite 330, Houston, TX 77034 (a veces denominada en el presente como la "CESIONARIA"), por el presente CEDEMOS, VENDEMOS Y ENAJENAMOS a dicha United Laboratories International, LLC, a sus sucesores y cesionarios, todo el derecho, título e interés en todo el mundo en y a:

1. Dicha invención relativa a un Sistema de Solventes para Limpieza in Situ de Catalizadores en un Reactor de Lecho Fijo;
2. La solicitud de patente de los Estados Unidos de América para dicha invención, Solicitud No. 16/290,679, presentada el 1 de marzo de 2019, titulada Sistema de Solventes para Limpieza in Situ de Catalizadores en un Reactor de Lecho Fijo;
3. Todas las solicitudes de patente o de protección similar para dicha invención, que ya se hayan o se lleguen a presentar en el futuro por parte nuestra o de nuestros representantes legales, incluyendo cualquier continuación,

continuación en parte y cualquier otra solicitud de patente de utilidad que pueda estar basada en esta invención, ya sea en los Estados Unidos de América o en cualquier otro lugar del mundo;

4. Todas las patentes y protecciones similares que ya se hayan o se lleguen a otorgar en el futuro para dicha invención a favor nuestro o de nuestros representantes legales, ya sea en los Estados Unidos de América o en cualquier otro país o lugar del mundo;
5. Todas las sustituciones y divisiones, continuaciones, continuaciones en parte, renovaciones, reexpediciones, prórrogas y solicitudes similares y patentes y protecciones similares, incluyendo, sin limitación, las obtenidas o permitidas de conformidad con leyes y regímenes pasados, presentes y futuros.
6. Todos los derechos de acción por cuenta del uso no autorizado anterior, presente y futuro de dicha invención y por la infracción de dichas patentes y protecciones similares;

PATENTE

ROLLO: 049295 CUADRO: 0285

-
7. El derecho para que la CESIONARIA presente a su nombre solicitudes de patente y de protección similar para dicha invención en cualquier país o países en el exterior de los Estados Unidos; y
 8. Todos los derechos internacionales de prioridad asociados con dicha invención, solicitudes, patentes y protecciones similares; y nos comprometemos a que nosotros y nuestros

DIEGO F. VARGAS Z.

TRADUCTOR E INTERPRETE OFICIAL

ESPAÑOL - INGLÉS - INGLÉS - ESPAÑOL

RES. 0001 DE ENERO 4 DE 1994

MINISTERIO DE JUSTICIA

herederos, representantes legales, cesionarios, administradores y albaceas testamentarios suscribirán, por cuenta de la CESIONARIA, de sus sucesores y cesionarios, todos los documentos y realizarán dichos otros actos que puedan ser razonablemente necesarios para concederle a la CESIONARIA, a sus sucesores y cesionarios, el beneficio total de la presente Cesión.

SUSCRITO en la fecha indicada a continuación al frente de nuestras firmas.

CEDENTES

Fecha: 8 de mayo de 2019

Firma Ilegible
Stephen D. Matza

Fecha: 8 de mayo de 2019

Firma Ilegible
Elisa Rice

PATENTE

INSCRITO: 05/28/2019

ROLLO: 049295 CUADRO: 0286

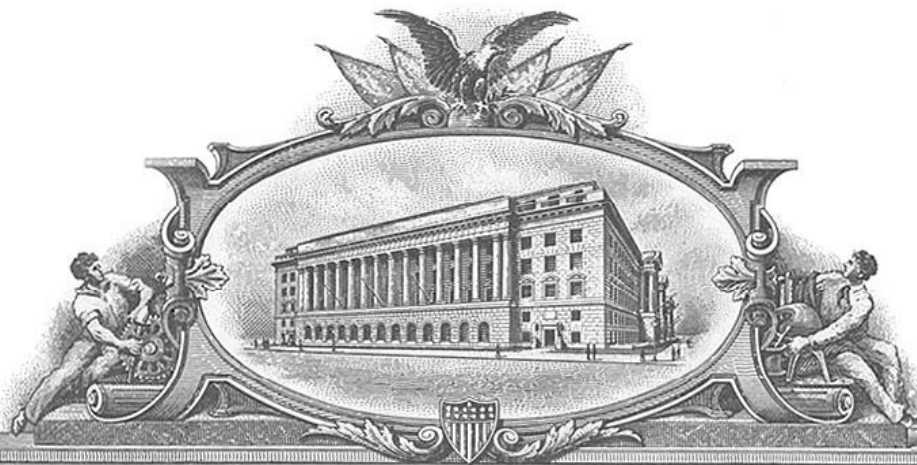
FIN DE LA TRADUCCIÓN

Bogotá D.C., 3 de julio de 2019

Es traducción fiel y completa del documento original realizada por:

DIEGO F. VARGAS ZAMORA, Traductor e Intérprete Oficial, Resolución No. 0001 de 1999
Ministerio de Justicia de Colombia.

DIEGO F. VARGAS Z.
TRADUCTOR E INTERPRETE OFICIAL
ESPAÑOL - INGLÉS - INGLÉS - ESPAÑOL
RES. 0001 DE ENERO 4 DE 1999
MINISTERIO DE JUSTICIA



THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

10 de junio de 2019

POR LA PRESENTE SE CERTIFICA QUE EL DOCUMENTO ADJUNTO ES UNA COPIA FIEL DE LOS REGISTROS DE LA OFICINA DE PATENTES Y MARCAS DE LOS ESTADOS UNIDOS DE DICHS DOCUMENTOS DE LA SOLICITUD DE PATENTE IDENTIFICADA A CONTINUACIÓN QUE CUMPLIÓ CON LOS REQUISITOS PARA QUE SE LE OTORGARA UNA FECHA DE PRESENTACIÓN EN VIRTUD DE 35 USC 111.

NÚMERO DE SOLICITUD: 16/290,679

FECHA DE PRESENTACIÓN: 1 de marzo de 2019

EL CÓDIGO DE PAÍS Y EL NÚMERO DE SU SOLICITUD DE PRIORIDAD, QUE SE USARÁ PARA LA PRESENTACIÓN EN EL EXTRANJERO EN VIRTUD DE LA CONVENCIÓN DE PARÍS, ES US16/290.679

Por autoridad del
Subsecretario de Comercio para la Propiedad Intelectual
y Director de la Oficina de Patentes y Marcas de los Estados Unidos

JOHN A BURSON

Funcionario certificador



