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SERIAL NUMBER FILING DATE CLASS GROUP ART UNIT ATTOBNEY DOCKET NO. 09/113,258 07/10/98 208 1764 GLN-1623 LARRY E. CAMPBELL, KNOXVILLE, TN; GREGORY J. WAGNER; KNOXVILLE, TN NONE \*\*CONTINUING DOMESTIC DATA\*\*\*\*\* VERIFIED JUN. NONE \*371 (NAT'L STAGE) DATA TRIFIED jW NONE \*\*FOREIGN APPLICATIONS\* VERIFIED? FOREIGN FILING LICENSE GRANTED 07/27/98 \*\*\*\*\* SMALL ENTITY \*\*\*\*\* □yes ⊠no □yes gino □Met after Allowance Foreign Priority claimed 35 USC 119 (a-d) conditions met STATE OR COUNTRY SHEETS TOTAL CLAIMS INDEP DENT Fw Verific 1 and Acknowledged 16. 40 TN xaminer s Intuels KENNETH H JOHNSON P @ BOX 630708 **ADDRES** HOUSTON TX 77263 SORBER TROCESS AND CATALYST/BOBER FOR TREATING SULFUR COMPOUND CONTAINING EFFLUENT F FILING FEE RECEIVED All Fees FEES: Authority has been given in Paper 1.16 Fees (Filing) 1.17 Fees (Processing Ext. of time) No. to charge/credit DEPOSIT ACCOUNT NO. for the following: \$656 1.18 Fees (Issue) `Other`\_ Credit



# IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Case Docket No.: GLN 1623

THE ASSISTANT COMMISSIONER FOR PATENTS Washington, D.C. 20231

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Transmitted herewith for filing  $\bar{\Psi}s$  the patent application of inventor(s): LARRY E. CAMPBELL and GREGORY J. WAGNER

FOR: PROCESS AND CATALYST/SORBER FOR TREATING SULFUR COMPOUND CONTAINING EFFLUENT

Enclosed are:

[X] 16 sheet(s) of drawings. (in triplicate)

 $\ensuremath{\mathsf{IXJ}}$  An assignment of the invention to: GOAL LINE ENVIRONMENTAL TECHNOLOGIES LLC with cover sheet for recordation.

[X] Inventors Declaration and Power of Attorney

[X] A verified statement to establish small entity status under 37 CFR § 1.0 and 37 CFR § 1.27.

[] Information Disclosure Statement with references.

The filing fee has been calculated as shown below

FOR:	(Col. 1) (Col. 2) NO.FILED NO. EXTRA	SMALL ENTITY RATE FEE OR	
BASIC FEE		\$395	\$790
TOTAL CLAINS*	40 - 20 = 20	x 11 ≂ 220	x 22 =
INDEP CLAIMS*	4 - 3 = 1	x 41 = 41	x 82 = \$

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[X] Any patent application processing fees under 37 CFR § 1.17

Respectfull Date: 07/10/ Ini

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# PROCESS AND CATALYST/SORBER FOR TREATING SULFUR COMPOUND CONTAINING EFFLUENT

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#### BACKGROUND OF THE INVENTION

Field of the Invention 5

> The present invention relates to removing gaseous sulfur compounds from gaseous streams, particularly the removal of SO2 and H2S from combustion and industrial process effluents, by using a regenerable catalyst/sorber.

Related Art 10

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of concern with air As one result pollution, environmental regulators have severely reduced and are continuing to reduce allowable emissions of sulfur oxides As a result, a variety of and hydrogen sulfide. technologies have been developed and are continuing to be developed for use in flue gas desulfurization (FGD).

FGD techniques can generally be classified as wet and dry scrubbing. Dry scrubbing contacts the effluent with a solid material which chemically reacts with the sulfur component and forms a compound. The system can be a fixed bed such as zinc oxide pellets that are used to react with H2S to form zinc sulfide. The zinc sulfide pellets must be removed and replaced after saturation. The dry scrubber can also be in powder or particulate injected into the stream followed by a baghouse or electrostatic filtration to remove the reacted product. An example of powder would be limestone, which would react after injection into the exhaust stream to form calcium sulfate and/or calcium The material is typically sulfite hemihydrate sludge. delivered as a wet slurry, which dries in the exhaust and reacts with the sulfur oxides. This material is then removed and typically disposed of in a landfill. There are also regenerable dry sorubber materials such as copper oxide on aluminum oxide pellets or spheres. The regenerable copper systems must be heated to and or reduced This technique requires that the at high temperatures. sorption temperature be less than the regeneration temperature.

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Wet scrubbing techniques use wet slurries or amine solutions and require that the exhaust temperature be reduced to below the boiling point of the solutions to be These techniques give rise to losses by evaporation used. and entrainment and produce products which are contaminated and unusable and must be disposed of /or purified before reuse.

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The present invention introduces a new technology for the removal of  $H_2S$ ,  $SO_2$  and oxidation of CO. This technology utilizes a catalytic oxidative sorption process for the removal of the sulfur component where the sulfur component is first oxidized and preconcentrated and then released in a concentrated stream of much smaller volume which is delivered to processes for recovery as sulfur, This technology has sulfur dioxide, or sulfuric acid. tremendous advantages over the scrubbing techniques. It is an advantage that the present process operates in the exhaust stream with oxidative capture mode and reductive regeneration occurring at the same temperature. It is a further advantage that it is a dry process, which is selective for sulfur components and will produce sulfur oxide off gases with high concentration and purity. It is a feature of the present invention that it reduces the volume of sulfur containing gases thereby reducing the costs for further processing. It is a further feature that the process also operates over a wide range of temperatures (200°F to, 800°F). It is another advantage of the process of the present invention that it has high capture efficiencies of over 99.75% and also has very low pressure 30 · drop.

## SUMMARY OF THE INVENTION

One aspect of the present invention is a process for removing gaseous sulfur compounds, particularly SO2 and/or H<sub>2</sub>S from gaseous streams, such as effluent combustion and industrial process streams, comprising contacting a gaseous stream, containing gaseous sulfur compounds with a catalyst/sorber under sulfur sorbing condition, said catalyst/sorber comprising a noble catalyst metal

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component, a metal oxide sorber component, and optionally modifiers consisting of oxides of Ag, Cu, Bi, Sb, Sn, As, In, Pb, Au or mixtures thereof to remove said gaseous sulfur compound from said stream to said catalyst/sorber. In a preferred embodiment of the present process the gaseous sulfur compound containing stream is terminated and the sorbed sulfur is desorbed from said catalyst/sorber by contacting the catalyst/sorber with a regenerating gas stream under sulfur compound desorbing conditions whereby the catalyst/sorber is regenerated for reuse in the sorbing. A most preferred embodiment comprises alternating said sorbing and regeneration steps.

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Preferred regenerating gases provide a reducing The reducing agents include hydrogen and environment. The hydrocarbon hydrocarbons or mixtures thereof. preferably comprises of  $C_1-C_{12}$  hydrocarbons, which may be used as one compound or mixtures of compounds. Usually the reducing agent will comprise methane and/or a mixture of The principal source of methane is natural hydrocarbons. The principal component of the gaseous stream is an qas. inert carrier gas such as nitrogen, helium, argon or steam. The term "principal component" is used to mean over 50%. The regeneration may also be carried out with the inert Air may also carried gases alone or with oxygen present. be used for regeneration.

Another aspect of the present invention is the catalyst/sorber. The noble metal component may comprise Pt, Pd, Rh, Ru or mixtures thereof, preferably Pt. The metal oxide sorber component is an oxide of Ti, Zr, Hf, Ce, Al, Si or mixtures thereof. In addition to these components, the catalyst/sorber optionally contains a modifier comprising an oxide of Ag, Cu, Bi, Sb, Sn, As In, Pb, Au or mixtures thereof, preferably Cu, Ag, Bi and mixtures thereof. The purpose of the modifier is to inhibit the formation of  $H_2S$  during regeneration.

The catalyst/sorber can be used in pellet, spheres, particulate, or extruded form. Preferably the catalyst/sorber can be coated onto a carrier with the

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catalyst/sorber comprising 1 to 50 wt% of the total weight. The noble metal component is preferably present as from 0.005 to 20.0 wt% of the catalyst/sorber, the sorber component is preferably present as from 70 to 99 wt% of the catalyst/sorber, and the modifier is preferably present as from 1 to 10 wt% of the catalyst/sorber.

Although the sulfur compounds are removed from a gaseous stream on and/or onto the catalyst/sorber according to the present invention, it is not known in what form or by what sulfur is associated with the mechanism the It is the invention that the sulfur catalyst/sorber. compounds in the gaseous stream are in some manner releasably associated with the catalyst/sorber in an It is believed that the sulfur is oxidizing atmosphere. associated with the catalyst/sorber as a compound, most likely as an oxide, but not in the elemental form. Elemental sulfur has not been observed. Preferably the sulfur is removed from the catalyst/sorber as a more concentrated stream of sulfur compounds. Unless otherwise stated percentages and ratios of compounds expressed herein are by weight.

# BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 shows outlet SO2 values during 300°F - 30 minute sorption runs with 30 ppm SO2 and the conditions given in Table 2 with different catalyst/sorber compositions.

Figure 2 shows outlet SO2 values during a 500 ppm SO2 sorption run and a 60 ppm SO2 sorption run at 500°F.

Figure 3 shows sulfur components released during partially saturated TiO2/Pt regeneration of catalyst/sorber.

Figure 4 shows the amount of SO2 captured and released during a 20 minute run and a subsequent reductive regeneration of the TiO2/Pt catalyst/sorber at 500°F.

Figure 5 shows the amount of SO2 captured and released during a 20 minute run and a subsequent regeneration of the TiO2/Pt/Cu catalyst/sorber at 500°F.

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Figure 6 shows the effect of Cu loading reported as precursor  $Cu(NO_3)_2$  on the proportion of  $H_2S$  and  $SO_2$  released during 500°F regenerations (Table 4) with the  $TiO_2/Pt/Cu$  system.

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Figure 7 shows 300°F, 60 ppm SO<sub>2</sub> sorption runs with TiO<sub>2</sub>/Pt catalyst/sorbers modified with Bi, Ag or Cu.

Figure 8 shows the SO<sub>2</sub> release during regeneration of TiO<sub>2</sub>/Pt catalyst/sorbers modified with Bi, Ag or Cu.

Figure 9 shows consecutive 60 ppm  $SO_2$  cycles with  $TiO_2/Cu$  sorber/modifier with no noble metal catalyst component at 500°F.

Figure 10 shows the effect of Pt loading reported as wt% of sorber/carrier on the  $SO_2$  breakthrough during 60 ppm  $SO_2$  run cycles at 500°F.

Figure 11 shows the effect of oxygen on the  $SO_2$  sorption for the TiO<sub>2</sub>/Pt catalyst/sorber system.

Figure 12 shows capture efficiencies obtained during 75 minute 60 ppm sorption cycles with the  $TiO_2/Pt$  system at 520, 620, and 810°F and comparatively the  $TiO_2/Pt/Cu$  system at 620°F.

Figure 13 shows the  $H_2S$  sorption with  $TiO_2/Pt$  and  $TiO_2/Pt/Cu$  catalyst sorbers with 30 ppm inlet  $H_2S$  at 500°F.

Figure 14 shows desorption of captured  $H_2S$  after the sorption run with the TiO<sub>2</sub>/Pt system illustrated in Figure 13.

Figure 15 shows  $SO_2$  released during a 900°F desorption with inert gas, which followed a 100 ppm  $SO_2$  sorption at 500°F.

Figure 16 shows 250 ppm SO<sub>2</sub> sorption runs following a standard regeneration and following a thermal regeneration with air.

#### PREFERRED EMBODIMENTS

The sorption stage may be carried out at 100 to  $1000^{\circ}$ F, preferably 300 to  $600^{\circ}$ F at GHSV 200 to 200,000 hr<sup>-1</sup> preferably 1,000 to 120,000 hr<sup>-1</sup>. The pressures may range from subatmospheric to 500 psig.

The desorption stage may be carried out at 100 to 1000°F, preferably 300 to 900°F at GHSV 204 to 20,000  $\rm hr^{-1},$ 

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preferably 50 to 10,000  $hr^{-1}$ . The pressures may range from subatmospheric to 500 psig.

By carrying out the present sorption/desorption the sulfur compounds in the feed may be concentrated from 5 to 100 times.

a carrier is utilized, the carrier Τf may be characterized as cetamic or metal monolith having a honeycomb structure. Preferably a carrier is used to distribute the catalyst/sorber and any modifier. The components are deposited onto the carrier in the proportions desired. The composition of the ceramic carrier can be any oxide or combination of oxides. Suitable oxide carriers include the oxides of Al, Zr, Ca, Mg, Hf, Ti, and various mixtures of the oxides, such as cordierite and mullite.

The structure and composition of the carrier is of great importance. The structure of the carrier affects the flow patterns through the catalyst/sorber system which in turn affect the transport to and from the catalyst surface. The ability of the structure to effectively transport the compounds to be catalyzed to the catalyst surface influences the effectiveness of the catalyst/sorber. The carrier is preferably macroporous with 64 to 600 cells (pores) per square inch (cpsi) which is about 25 to 80 pores per linear inch (ppi), although carriers having 5 to 90 ppi are suitable

The catalyst/sorber may be conditioned by repeated sorption/desorption cycling. The amount of sulfur components released during this conditioning increases with each regeneration until it equals the amount sorbed. It was found that by pretreating the catalyst/sorber with dilute H2SO4, that the conditioning time was dramatically reduced. For example, instead of 12 hours, conditioning after H<sub>2</sub>SO<sub>4</sub> treatment only required several hours at 300°F. The conditioning time was also found to be dependent on the temperature at which the sorption and regenerations were conducted. By increasing the temperature one can increase the amount released, hence, decreasing the conditioning

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time. A special conditioning may not be necessary, since the catalyst/sorber will become conditioned after several cycles of sorption/regeneration and remain so thereafter.

The amount of SO<sub>2</sub> capable of being consumed by the catalyst/sorber will be referred to as the capacity of the catalyst/sorber. From a short capacity study it was concluded:

1. When the sorption/desorption temperature increases, the capacity increases.

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2. When the catalyst/sorber loading is increased, the capacity increases.

3. When the noble metal content in the catalyst/sorber is increased, the capacity increases.

The catalyst/sorber used during this study was coated onto a ceramic honeycomb carrier. The catalyst/sorber/carrier contained 3.48 grams/in<sup>3</sup> TiO<sub>2</sub>/Pt with a platinum content of 0.5%. The capacity of this sample was determined to be 20 cc  $SO_2/in^3$ . This value was determined with an  $SO_2$  breakthrough less than 10%, hence, 20 cc is a minimum value.

Experimental data gave a  $SO_2/Pt$  mole ratio of 10 during catalyst/sorber saturation. One theory which is not intended to reduce the scope of the present invention is that this large excess of sorbed  $SO_2$  suggests that the role of the platinum is not of a sorber. The platinum under this theory promotes the sorption of  $SO_2$  through the catalytic oxidation of  $SO_2$  and the sulfur subsequently sorbs onto the conditioned TiO<sub>2</sub>/Pt surface.

The capacity of a  $TiO_2/Pt/Cu$  system was examined in a manner similar to that conducted with the  $TiO_2/Pt$  system. The results were almost identical. The conclusions were:

 When the sorption/desorption temperature increases, the capacity increases.

2. When the TiO<sub>2</sub>/Pt loading is increased, the capacity increases.

3. When the platinum content in the  $TiO_2/Pt$  is increased, the capacity increases. The copper modifier had little effect on sorber capacity.

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According to the present invention sulfur can be captured and released by the catalyst/sorber, for example, a TiO<sub>2</sub>/Pt system at temperatures as low as 300°F, however, the desorbed components released during regeneration consisted mainly of a mixture of  $H_2S$  and  $SO_2$ . The ratio of  $H_2S/SO_2$  depended on residence time and/or the presence of modifiers. In some applications the  $H_2S$  would not be desirable; hence it would be preferably to release the sulfur components as  $SO_2$ .

The nature of the desorbed sulfur components can be changed by adding a modifier to the catalyst/sorber. A variety of modifiers have been utilized but to date the most efficient have been copper, silver and bismuth. It was found that the addition of modifiers to the catalyst sorber increased the release of sulfur as  $SO_2$  and diminish or inhibit the formation of  $H_2S$ . The modifiers may be deposited directly onto the catalyst/sorber or deposited within. A variety of modifies were determined to work; however, the most efficient were found to be copper, bismuth, and silver.

Modifiers were also examined without a noble metal, e.g., Pt, i.e., sorber only in the washcoat, for example  $TiO_2/Cu$ . Although many were examined and found to capture the sulfur, the release of the  $SO_2$  or  $H_2S$  during regenerations was not accomplished and the sorbers soon became saturated. This is illustrated in Figure 9 where consecutive 60 ppm  $SO_2$  sorption cycles were carried out with a  $TiO_2/Cu$  sample. Intermediate regenerations followed each sorption cycle. The results show decreasing removal efficiency with each run.

Ву adding the modifier, e.g., Cu to the catalyst/sorber, the SO<sub>2</sub> captured during the sorption cycles was found to be released during the standard regeneration cycles. This is illustrated in Figure 5 where both the sorption and desorption cycles reached 7.7 cc SO2. The sulfur released during the regeneration was SO2. No 1125 was detected during this regeneration.

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The Cu(NO3)3 precursor loading on the catalyst/sorber used in Figure 5 was 0.3 g/in<sup>3</sup>. Figure 6 illustrates the effect of the  $Cu(NO_3)_3$  loading on the proportion of sulfur released as  $SO_2$  and  $H_2S$ . This figure demonstrates that in the vicinity of 0.3 g/in $^3$  and greater, all the sulfur compounds released during the regenerations was released as SO<sub>2</sub>; however, below  $^{4}$ 0.3 g/in<sup>3</sup> H<sub>2</sub>S is released and the amount of  $H_2S$  released increases with decreasing  $Cu(NO_3)_3$ This effect was also observed with the other loading. TiO2/Pt/X (where X is the metal modifier as defined) modifiers such as the bismuth and silver. The ability to change the  $H_2$ S/SO<sub>2</sub> ratio with modifier loading is a tremendous asset of this new technology. The ability to adsorb sulfur from a dilute stream and then release it in a concentrated stream with appropriate  $H_2S/SO_2$  ratio for use in the Claus process has not been heretofore disclosed. Figure 6 clearly illustrates that the Cu(NO3)2 loading can be used for this purpose.

Figure 12 shows the  $SO_2$  removal efficiency of the  $TiO_2/Pt$  system at various temperatures and the removal efficiency of the  $TiO_2/Pt/Cu$  system at 620°F. From this figure, it is evident that the capacity of the  $TiO_2/Pt$  system increases with temperature. The role of the copper and other modifiers appears to reside only in changing the regeneration of the catalyst to diminish  $H_2S$  formation.

By increasing the  $SO_2$  inlet concentration or lengthening the sorption time, the  $TiO_2/Pt$  catalyst/sorber will eventually become saturated; hence, producing  $SO_2$ breakthrough. This is illustrated in Figure 2 where both a 500 ppm and a 60 ppm sorption run were conducted for 10 minutes.

Figure 2 shows that there is no evidence of  $SO_2$  breakthrough during the 60 ppm run. The 500 ppm run, in contrast, does show breakthrough. During this sorption run, the  $SO_2$  breakthrough is relatively low (<40 ppm) for the first 3 minutes, however, after 3 minutes the  $SO_2$  breakthrough increases dramatically. This breakthrough

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results when the majority of sorption sites have been consumed.

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## EXAMPLES

The catalyst/sorbers used in the examples were prepared on 200-cell-per-square-inch cordierite square-cell honeycomb. The TiO<sub>2</sub>/Pt washcoat was prepared by incipient wetness. Pt content ranged from 0.1 to 2.2%. After drying and calcining at 500°C, the solids were then dispersed in 7% acetic acid and ball milled overnight. The ceramic honeycombs were then dipped into the slurry of washcoat/Pt, removed, blown off, and then dried at 150°C.

The  $TiO_2/Pt/X$  samples were prepared by immersing the  $TiO_2/Pt$  honeycomb sample into a modifier solution. The sample was then removed, blown off, and finally dried at 150°C. The catalyst/sorbers had a nominal composition as shown in Table 1.

Table 1: Catalyst/sorber Composition (except as otherwise noted):

 $\begin{array}{ccccccc} \text{TiO}_2 & 0.4 - 3.5 \text{ g/in}^3 \\ \text{Pt} & 0 - .08 \text{ g/in}^3 \\ \text{X} & 0 - 0.3 \text{ g/in}^3 \end{array}$ 

Gas Component

 $SO_2$ 

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H<sub>2</sub>ō

 $N_2$ 

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For testing, the samples were placed into a 304 stainless steel tubular reactor and placed inside a threezone furnace. The reactor was connected to a gas delivery system which delivered mixed gases simulating a gas turbine exhaust. The gases were measured and controlled by Matheson mass flow transducers. Water was injected into a preheat furnace using a Cole Palmer instrument number 74900 precision syringe pump. Unless stated otherwise, the test gas compositions are given in Table 2. All sorption runs were conducted with a space velocity of 30,000 hr<sup>-1</sup>. Table 2: Test Gas Compositions

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Concentration 30~500 ppm 14-52% 3.05% 10.20% Balance

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Before passing processed gas through the analytical instruments, the water was removed with a chiller. The dried exhaust was then analyzed with the instruments shown in Table 3. To measure  $H_2S$ , the exhaust was first directed through a  $H_2S$  to  $SO_2$  converter. This converter consisted of a heated (900°F) stainless steel tube. During regenerations, oxygen was added to the exhaust before entering the converter. Laboratory tests with calibration gas showed virtually 100%  $H_2S$  conversion to  $SO_2$ . During some experiments the  $H_2S$  was measured with a BOVAR model 922 analyzer.

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Table 3: Analytical Instruments Used During TestingGas ConstituentInstrumentSO2BOVAR model 721-MCOTECO model 48NO,NO2TECO model 10SNH3TECO model 300/10SCO2Horiba model DIA 510H2SBOVAR model 922

0.02%

1.00% 57.14%

40.84%

4.00%

Concentration

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Table 4: Regeneration Gas Composition

Catalyst Preparation

Gas Component

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CO2

The catalyst for the following examples was prepared as follows:

- Ceramic honeycomb carrier.

precursor  $(0.00 \text{ to } 0.30 \text{ g/in}^3)$ .

- Sorber component of 2.2 g/in<sup>3</sup>.

- Platinum loading from 0.25 to 1.1 wt % of the sorber component.

Metal modifier prepared with the metal nitrate

# Example 1

Example 1 shows 30 minute, 30 ppm SO<sub>2</sub> sorption runs with the TiO<sub>2</sub>, TiO<sub>2</sub>/Pt, ZrO<sub>2</sub>/Pt and CeO<sub>2</sub>/Pt systems at 300°F. With the TiO<sub>2</sub> sample there is little SO<sub>2</sub> capture, however, the TiO<sub>2</sub>/Pt, ZrO<sub>2</sub>/Pt, and CeO<sub>2</sub>/Pt catalyst/sorbers gave significant SO<sub>2</sub> captured as shown in Figure 1. This illustrates the effectiveness of the Ti, Zr, and Ce and the importance of the noble metal component. TiO<sub>2</sub> loading 2.18g/in<sup>3</sup>; XO<sub>2</sub>/Pt loading 2.18g/in<sup>3</sup>; 1.1% Pt where X = Ti, Zr, or Ce.

#### Example 2

Example 2 illustrates the effect of inlet  $SO_2$  concentration (60 ppm vs 500 ppm) during 10 minute, 500°F sorption runs with a TiO<sub>2</sub>/Pt catalyst/sorber (2.18g/in<sup>3</sup>; 0.5% Pt). Figure 2 shows  $SO_2$  breakthrough with the higher (500 ppm)  $SO_2$  inlet. This breakthrough results when the catalyst/sorber becomes saturated. After or during saturation, the catalyst/sorber can be revitalized with a reducing gas or a thermal desorption.

## Example 3

Example 3 shows the regeneration of a partially saturated  $TiO_2/Pt$  catalyst/sorber at 500°F. Regeneration of the catalyst/sorber revitalizes the sorber and produces a concentrated stream of sulfur compounds with the primary compound being  $H_2S$ . The regeneration cycle was about 10 minutes as shown in Figure 3.

#### Example 4

Example 4 shows the amount of sulfur captured and released with a  $TiO_2/Pt$  catalyst/sorber during a 30 minute, 30 ppm  $SO_2$  sorption cycle at 500°F and a standard desorption cycle at 500°F. The results in Figure 4 show that the amount of sulfur compounds desorbed was equivalent to the amount of  $SO_2$  sorbed during the previous sorption cycle and that the desorbed gas was composed of a mixture of  $H_2S$  and  $SO_2$ .

#### Example 5

Example 5 shows the amount of sulfur sorbed and desorbed with a  $TiO_2/Pt/Cu$  sample during  $\frac{4}{5}$  20 minute, 60

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ppm SO<sub>2</sub> sorption cycle at 500°F and a standard desorption cycle at 500°F. The results in Figure 5 demonstrates that the amount of sulfur components desorbed was equivalent to the amount of 502 sorbed during the previous sorption cycle and that the components desorbed was primarily SO2.

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#### Example 6

Example 6 shows the effect of copper loading (metal modifier) on the proportion of sulfur released as SO2 or H<sub>2</sub>S during regeneration cycles of partially saturated TiO2/Pt catalyst/sorber. This example shows in Figure 6 that with 0.3 g/in<sup>3</sup> of  $Cu(NO_3)_2$  and higher, all the sulfur is released as  $SO_2$ . With no  $Cu(NO_3)_2$ , 90% of the sulfur is released as H2S and 10% is releases as SO2. This effect was also obtained with other modifiers.

#### Example 7

Example 7 shows the SO2 capture during 20 minute, 60 ppm sorption cycles using TiO2/Pt with Cu, Ag and Bi modifiers (TiO2/Pt/Cu 2.18 g/in3; 1.1% Pt; 0.3 g/in3  $X(NO_3)_V$ ; where X=Cu, Ag, Bi). All exhibit similar characteristics with initially 100% capture efficiency until reaching a point where SO2 breakthrough begins. The amount of breakthrough then steadily increases with time as can be seen in Figure 7.

#### Example 8

Example 8 demonstrate the SO2 released during the regeneration of TiO2/Pt catalyst/sorber with Bi, Ag, and Cu

These regenerations followed 20 minute,

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metal modifiers.

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#### Example 9

30 ppm SO<sub>2</sub> sorption cycles and appear to be almost identical with liberation of SO2 as shown in Figure 8.

Example 9 illustrates consecutive 60 ppm SO<sub>2</sub> sorption cycles with a  $TiO_2/Cu$  modifier/sorber (2.18g/in<sup>3</sup>  $TiO_2$ ;  $0.3g/in^3 Cu(NO_3)_2$ ) with no noble metal component at 500°F. This result shown in Figure 9 illustrates that the TiO2/metal modifier will capture the sulfur, however, the catalyst/sorber is not regenerated during the regeneration cycle.

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## Example 10

Example 10 illustrates the effect of Pt loading on the  $SO_2$  breakthrough during 60 ppm  $SO_2$  sorption cycles with a  $TiO_2/Pt/Cu$  catalyst/sorber (2.18 g/in<sup>3</sup>  $TiO_2/Pt$ : 0.3 g/in<sup>3</sup>  $Cu(NO_3)_2$ ) at 500°F. Figure 10 shows that the efficiency of the  $TiO_2/Pt/Cu$  is directly related to the Pt loading, such that the higher the loading the higher the sulfur capture capacity.

#### Example 11

Example 11 illustrates the effect of oxygen in the flue gas during 25 minute, 250 ppm  $SO_2$  sorption cycles with a  $TiO_2/Pt/Cu$  catalyst sorber at 500°F. Figure 11 shows that sorption of the sulfur components is significantly reduced in the absence of oxygen.

#### Example 12

Example 12 shows the sulfur removal efficiency obtained during 75 minute, 60 ppm  $SO_2$  sorption runs with a  $TiO_2/Pt$ catalyst/sorber at various temperatures and a  $TiO_2/Pt/Cu$ catalyst/sorber at 620°F is shown. Figure 12 shows that the removal efficiency increases with temperature and that the removal efficiency is slightly decreased by the addition of the metal modifier.

#### Example 13

Example 13 shows that  $H_2S$  is removed during sorption cycles with both the TiO<sub>2</sub>/Pt and TiO<sub>2</sub>/Pt/Cu

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# catalyst/sorbers at 500°F. Figure 13 shows the results and demonstrates that $H_2S$ is also captured by the catalyst/sorbers.

#### Example 14

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Example 14 shows that sulfur is released primarily as  $H_2S$  during a desorption run of a partially saturated  $TiO_2/Pt$  catalyst sorber at 500°F. The preceding sorption run utilized a 20 minute, 20 ppm  $H_2S$  cycles at 500°F. This result, shown in Figure 14, demonstrates that the catalyst/sorbers can be regenerated after  $H_2S$  capture.

#### Example 15

In this Temperature Programmed Desorption (TPD) experiment a 4-minute sorption cycle with<sup>4</sup> 100 ppm of SO<sub>2</sub>

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was conducted at 300°F (13 cc TiO<sub>2</sub>/Pt 3.0 g/in<sup>3</sup> TiO<sub>2</sub>:1.1% Pt), space velocity = 30,000 hr<sup>-1</sup>). After the temperature was increased to 900°F, nitrogen was then passed over the catalyst as a space velocity of 30,000 hr<sup>-1</sup>. From the TPD experiment it was determined that all the sulfur captured during the 4 minute run cycle was released during the high temperature regeneration; however, no  $H_2S$  was detected, hence all the sulfur released was in the form of SO<sub>2</sub>. This illustrates that the H2S formation occurs only in the presence of the  $H_2$ . Figure 15 shows that the sulfur is released in two pulses. The first being much sharper than the second.

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#### Example 16

In this Temperature Programmed Desorption (TPD) experiment a 10 minute sorption cycle with 250 ppm of SO<sub>2</sub> was conducted at 500°F (13 cc TiO<sub>2</sub>/Pt 3.0 g/in<sup>3</sup> TiO2:1.1% Pt), space velocity = 20,000 hr<sup>-1</sup>). After the temperature was increased to 900°F, air (approximately 20.9% O<sub>2</sub>) was then passed over the catalyst for 10 minutes at a space velocity of 10,000 hr<sup>-1</sup>. During the desorption no SO<sub>2</sub> or H<sub>2</sub>S was detected, however; continuous cycling showed that the TiO<sub>2</sub>/Pt catalyst/sorber was revitalized during each 900°F air regeneration. This is illustrated in Figure 16. The regeneration gas was also bubbled through isopropanol and analyzed for sulfur. A significant amount of dissolved sulfur was found suggesting that SO<sub>3</sub> was released during regenerations.

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The invention claimed is:

1. A process for removing gaseous sulfur compounds from gaseous streams, comprising contacting a gaseous stream, containing gaseous sulfur compounds with a catalyst/sorber under sulfur sorbing conditions, said catalyst/sorber comprising a noble metal component and a metal oxide sorber component selected from the group consisting of Ti, Zr, Hf, Ce, Al, Si and mixtures thereof and removing a portion of said gaseous sulfur compounds from said stream.

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2. The process according to claim 1 wherein said noble metal component comprises Pt, Pd, Rh, Ru or mixtures thereof.

3. The process according to claim 2 wherein noble metal comprises Pt.

4. The process according to claim 1 wherein the metal oxide sorber component is an oxide of Ti, Zr, Hf, Ce or mixtures thereof.

5. The process according to claim 2 wherein the metal oxide sorber component is an oxide of Ti, Zr, Hf, Ce or mixtures thereof.

6. The process according to claim 1 wherein a modifier consisting of an oxide Ag, Cu, Bi, Sb, Sn, As In, Pb, Au or mixtures thereof is present as a component of said catalyst/sorber.

7. The process according to claim 6 wherein the modifier is Cu, Ag, B or mixtures thereof.

8. The process according to claim 2 wherein a modifier consisting of an oxide of Ag, Cu, Bi, Sb, Sn, As In, Pb, Au or mixtures thereof is present as a component of said catalyst/sorber.

9. The process according to claim 8 wherein the modifier is an oxide Cu, Ag, Bi or mixtures thereof.

10. The process according to claim 5 wherein a modifier consisting of an oxide Ag, Cu, Bi, Sb, Sn, As In, Pb, Au or mixtures thereof is present as a component of said catalyst/sorber.

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11. The process according to claim 10 wherein the modifier is an oxide of Cu, Ag, Bi or mixtures thereof.
12. The process according to claim 9 wherein the modifier is cu oxide.

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13. The process according to claim 9 wherein the modifier is Ag oxide.

14. The process according to claim 9 wherein the modifier is Bi oxide.

15. The process according to claim 1 wherein the gaseous sulfur compound containing stream is terminated and the sorbed sulfur components is desorbed from said catalyst/sorber by contacting the catalyst/sorber with a regenerating gas stream under sulfur compound desorbing conditions whereby the catalyst/sorber is regenerated for reuse in the sorbing.

16. The process according to claim 15 where the regeneration gas comprises an inert carrier and a reducing component.

17. The process according to claim 16 where the inert gas is nitrogen, helium, argon, or steam.

18. The process according to claim 16 wherein the reducing component is hydrogen, methane, hydrocarbons from  $C_1$  to  $C_{12}$  or mixtures thereof.

19. The process according to claim 15 wherein the regeneration gas is nitrogen, helium, argon, or steam.

20. The process according to claim 15 wherein the regeneration gas comprises oxygen.

21. The process according to claim 15 wherein the regeneration gas is nitrogen, helium, argon, or steam with oxygen present.

22. The process according to claim 15 wherein residual gaseous sulfur containing stream and reducing agent is added without carrier a gas.

23. The process according to claim 15 wherein an amount of combustible gas is added during said regeneration to raise the desorbing temperature to facilitate thermal desorption.

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24. A catalyst/sorber comprising a noble metal catalyst component, a metal oxide sorber component selected from Ti, Zr, Hf, Ce, Al, Si or mixtures thereof and a modifier consisting of an oxide of Ag, Cu, Bi, Sb, Sn, As In, Pb, Au or mixtures thereof.

 $\mathcal{T}_{\mathcal{F}_{2}}$ . The catalyst/sorber according to claim  $\mathcal{J}_{4}$  wherein said noble metal component is Pt, Pd, Rh, Ru or mixtures thereof.

26. The catalyst/sorber according to claim 25 wherein noble metal is Pt.

1/21. The catalyst/sorber according to claim 24 wherein the metal oxide sorber component is an oxide of Ti, Zr, Hf, Ce or mixtures thereof.

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5%. The catalyst/sorber according to claim 28 wherein the metal oxide sorber component is an oxide of Ti, Zr, Hf, Ce or mixtures thereof.

(22). The catalyst/sorber according to claim 24 wherein component
the modifier, is an oxide of Cu, Ag, Bi or mixtures thereof.
730. The catalyst/sorber according to claim 25 wherein the modifier, is an oxide of Cu, Ag, Bi or mixtures thereof.
541. The catalyst/sorber according to claim 24 wherein component is
the modifier Cu oxide.

972. The catalyst/sorber according to claim 24 wherein Component the modifier, is Ag oxide.

33. The catalyst/sorber according to claim 24 wherein the modifier is Bi oxide.

34. A catalytic structure comprising a monolith carrier and a catalyst/sorber comprising a noble metal catalyst component, a metal oxide sorber component selected from Ti, Zr, Hf, Ce, Al, Si or mixtures thereof and a modifier consisting of an oxide of Ag, Cu, Bi, Sb, Sn, As In, Pb, Au or mixtures thereof deposited on said carrier. // 35. The catalytic structure according to claim 34 wherein said catalyst/sorber comprises 1 to 50 wt% of the total weight of carrier and catalyst/sorber. /2 36. The catalytic structure according to claim 38

wherein said noble metal component comprises 0.005 to 20.0 wt% of the catalyst/sorber. 3/. The catalytic structure according to claim 26 wherein said sorber component comprises from 70 to 99 wt of the catalyst/sorber.

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Wherein said modifier, comprises from 1 to 10 wt% of said catalyst/sorber.

contacting a first gaseous sulfur compound containing stream in a first concentration of sulfur compounds in a first concentration with a catalyst/sorber comprising 0.005 to 20 wt% noble metal catalyst component, 70 to 99 wt% metal oxide sorber component selected from Ti, Zr, Hf, Ce, Al, Si or mixtures thereof and 0 to 10 wt% modifier consisting of an oxide of Ag, Cu, Bi, Sb, Sn, As, In, Pb, Au or mixtures thereof under conditions to remove a portion said sulfur compounds from said gaseous stream onto said catalyst/sorber;

removing a portion of said sulfur compounds from said gaseous sulfur containing stream;

terminating said gaseous sulfur compound containing stream;

contacting said catalyst/sorber with a regenerating gas to remove sulfur compounds from said catalyst/sorber into said regenerating at a second concentration to form a second gaseous sulfur compound containing stream comprising said regenerating gas and gaseous sulfur compound component comprising SO<sub>2</sub> or a mixture of SO<sub>2</sub> and  $H_2S$ ;

wherein the composition of the sulfur compound component is adjustable by the amount of modifier present in said catalyst/sorber.

40. The method according to claim 39 wherein the amount of  $H_2S$  is decreased 90 to 0 % and the  $SO_2$  increases from 10 to 100 % as the modifier present increases from 0 to 10 wt%

# ABSTRACT OF THE DISCLOSURE

system for treating the exhaust of an industrial process or combustion source to capture gaseous sulfur compounds which includes contacting the exhaust containing gaseous sulfur compounds with a system of: (1)а structured support coated with for example a monolith (2) a ceramid oxide containing for example TiO<sub>2</sub> (3) a precious metal component for example Pt and optionally (4) a modifier such as Cu. The system captures the gaseous sulfur compounds. Caseous sulfur compounds are then driven off in higher concentrations in a separate isolated lower flow stream in higher concentrations which are easier to process to less noxious or useful sulfur materials, and the catalyst/sorber degenerated. The system also removes carbon dioxide by the oxidation of the carbon monoxide to carbon dioxide.

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Figure 3















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2: Figure









































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# 1 Line Env Tech LLC Kenneth H. Johnson & Hs

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DECLARATION FOR PATENT APPLICATION

## Docket Number: GLN 1623

As a below named inventor, 1 hereby declare that:

Hy residence, post office address and citizenship are as stated below next to may name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled <u>PROCESS AND CATALYST/BORBER FOR TREATING BUTFUR COMPOUND CONTAINING EFFLUENT</u>, the specification of which is attached hereto unless the following box is checked:

[] was filed on \_\_\_\_\_\_ as United States Application number surface \_\_\_\_\_\_ and was amended on \_\_\_\_\_\_ (if applicable \_ or PCY International Application (if applicable).

) hereby state that 1 have reviewed and sinderstand the contents of the above [dentified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in title 37, code of Federal Regulation, §1.56.

1 hereby claim foreign priority benefits under Title 35, United States Dode \$119 (a)-(d) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below my foreign priority is claimed.

(Hunber)	(Country)	(Day/Honth/Tear Filed)	Priority Claimed
(Number)	(Country)	(Day/Honth/Year Filed)	

i hereby claim the benefit under Title 35, United States Code, \$ 199(a) of any United States provisional application(s) listed below.

(Application Number)

(Filing Date)

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I hereby claim the benefit under title 35. United States Code, § 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the marner provided by the first paragraph of fille 35. United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in file 37. Code of federal Regulations, § 1.56 which became available between the filing date of the prior application and the mational or PCI international filing date of this application.

(Application Humber)	(Filing Date)	(Status-patented, pending, abandoned)
(Application Hunder)	(Filing Date)	(Contribute on Landard - 4

#### (Filing Date) (Status-patented, pending, abandoned)

I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith: KENNETH N. JOHNSOW REG. # 22,966

Address ail telephone calls to XENNETH R. JOHNSON at telephone number (713) 780-7047

Address all correspondence to KENNETH H. JOHNSON, P.O. BOX 630708, HOUSTON, TEXAS 77263

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 wiliful false statements and the like so made are punishable by fine imprisonment, or both, under Section 1001 of fitte 18 of the United States Code and that such willful false statements may impordize the validity of the application or any patent issued thereon.

Full name of first joint inventor, if any (given name, family name): LARRY E. CAMPBELL Inventor's signature <u>Lange Campbell</u> Date Residence: KNOWLLE, TENNESSE Citizenship: USA Pout office Address: 11141 CUTLET DRIVE, KNOWLLE, TENNESSEE 37932 July 9, 1998 Date \_

Full name of second joing inventor, is any (given name, family name): GRECORY J. WACKER

TERMESSEE Wagne Inventor's signature Date July 9,  $\boldsymbol{\gamma}$ Residence: DADAVILLE, TERMESSEE CItizenship: USA Post Office Address: 11161 OUTLET DRIVE, KNOXVILL, TERMESSEE 37932 1998

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#### ATTY FILE: OLN 1623

APOLICANT OF PATENTANE LANKY R. CAMPBELL and CRESORY J. WAGNER Serial of Parent Noi filed of Issued:

FOR1 PROCESS AND CATALYST/SORBER FOR TREATING SULFUR COMPOUND CONTAINING EFFLUENT

# VERIFIED TIATEMENT (DECLARATION) CLAIMIND SMALL ENTITY STATUS (37 CFR 1.9(1) Mgd 1.27(C) - Small Business Concern

I hereby declare that I am []-the duner of the ampli business concern identified belows [X] am official of the small pusiness concern ampowered to act on behalf of the concern identified below:

NAME OF CONCERN: GOAL LINE ENVIRONMENTAL TECHNOLOGIEN LLC ADDRE33 OF CONCERN: 2065 %, vernam Avenum, Lon Angelos, California 900%

I mereby decises that the above identified small business concern qualifies as a small business concern as defined in 13 CFR 121.3-8, and reproduced in 37 CFR 1.9(27, for purposes of paying request fees under section 61(2) and (b) of Title 35, united States Code, in that the number of employees of the soncern, including those of jtt affiliates, does not exceed 300 persons. For purposes of this scatement, (1) the number of apployees of the business concern is the average over the previous fiscal year of the concern of the persons employee on a full-time, part-time or temporary basis during each of the pay periods of the fiscal year, and (2) concerns are affiliates of each other when dismer, directly or indirectly, one concern controls or has the power to control the other, or a third party or parties controls or has power to control both.

I hereby declure that rights under contract or law have been conveyed to and remain with the small business concern identified above with regard to invention, cutities <u>process and</u> <u>catalystanges for refailing swifus compound containing effluent</u> by inventor(s) <u>LARRY £. CAMPBFLL</u> and <u>oregony J. where</u> were thed in:

CAl the specification filed perowith. I application works no.\_\_\_\_\_\_ filed \_\_\_\_\_\_ I parent no.\_\_\_\_\_\_\_ issued \_\_\_\_\_\_

If the rights held by the above identified small business concern are not exclusive, each individual, concern or organization having rights to the invention is listed below and no rights to the invention are held by any person, other than the inventor, who could not qualify as a small business soncern under 37 CFR 1.9(d) or by any concern which would not qualify as a small business concern under 37 CFR 1.9(d) or a nonprofix organization under 37 CFR 1.9(a). (NOTE: Separate verified assessments are required from each named person, concern or organization naving rights to the invention overring to their status as small entities. (37 CFR 1.27)?

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#### [] INDIVIDUAL [] SMALL BUSINESS CONCERN [] NONPROFIT ORDANIZATION

I acknowledge the duty to file, in this application or patent, notification of any change in MIRLUS resulting in less of entitiement to small entity status prior to paying, or at the time of paying, the carifest of the issue for or ony maintenance for due store the date on which status as a small entity is nother appropriate. (37 GPR 1.28(b))

I hereby declare that all statements mode herein or my own knowledge are true and that all statements made on information and belief are believed to be true; and turther that these statements were made with the knowledge that willful taise statements and the like so made are punishable by fine or imprisonment, or both, where section 1001 of fitte 15 of the united States Code, and that such witiful false statements may desparaize the wijdity of the application, any patent issuing thereon, or ony patent to which this verified statement is directed.

NAME OF PERSON SIGNING: MICHAEL B. MARTIX Title of person Signing: Sectusery Address of person signing: 2045 E. Vernon Avanue, Los Angeles, California - 90058

SIGNATURE

7-10-98 DATE

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## EXPRESS MAIL CERTIFICATE

I hereby certify that the attached papers in re application of CAMPBELL ET AL, (Docket: GLN 1623) for: PROCESS AND CATALYST/SORBER FOR TREATING SULFUR CO MPOUND CONTAINING EFFLUENT, contains an application, 16 drawings in triplicate, small entity statement, assignment and cover sheet for recording, declaration and Power of Attorney, check for \$696, and postcard were deposited in the "EXPRESS MAIL POST OFFICE TO ADDRESSEE" service of the U.S. Postal Service on July 10, 1998, addressed to Box PATENT APPLICATION, Assistant Commissioner for Patents, Express Mail mailing label Number EM354532497US-

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Kenneth H. Johnson Reg. #22,966 P.O. Box 630708 Houston, Texas 77263 Phone: (713) 780-7047

Fax No.: (713) 780-7671

IN THE UNITED STATE PATENT AND TRADEMARK OFFICE

In re Application of CAMPBELL ET AL	. S	Attorney File No.: GI	N 1623
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Serial No.: 09/113,258	5 5	Examiner:	#2
Filed: 07/10/98	. <b>.</b> §		CS 9-2-98

For: PROCESS AND CATALYST/SORBER FOR TREATING SULFUR COMPOUND CONTAINING EFFLUENT

#### INFORMATION DISCLOSURE STATEMENT

Assistant Commissioner for Patents Washington, D.C. 20231 Sir:

This document and the attachments hereto are presented in accordance with the requirements of 37 CFR § 1.56. Below is listed information which may be material to the cited application and, which the Examiner may deem important and wish to consider in conjunction with the examination of the cited application. The relevance of the information is a subjective test and its submission here may be based on the fact the information provides background or in some manner relates to subject matter disclosed or claimed in the cited application. The submission of information herewith is not intended to be, nor is it an admission either directly or indirectly that all or any portion of said information is prior art as to any aspect of the invention(s) disclosed or claimed in the cited application. A copy of each reference is enclosed herewith. (See attached Form 1449).

Respectfully Submit

Kenneth H. Johnson ATTORNEY FOR APPLICANT Reg. No. 22,966 P.O. Box 630708 Houston, Texas 77263 TEL: (713) 780-7047 FAX: (713) 780 7671

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Date 08/12



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## CERTIFICATE OF MAILING

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Assistant Commissioner for Patents Washington, D.C. 20231 Ime

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Responsive to communication(s) filed on	·····				
□ This action is FINAL.					
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Part of Paper No. 3

Art Unit: 1754

## DETAILED ACTION

Page 2

### Election/Restriction

Restriction to one of the following inventions is required under 35 U.S.C. 121:

 Claims 1-23 and 39-40 (group I), drawn to a process for removing gaseous sulfur compounds out of gases, classified in class 423, subclass 242, 1+.

II. Claims 24-38 (group II), drawn to a catalyst, classified in class 502, subclass 300+.

The inventions are distinct, each from the other because of the following reasons: The inventions set forth in claims 24-38 (group II) and claims 1-23 and 39-40 (group II) are related as product and process of use. The inventions can be shown to be distinct if either or both of the following can be shown: (1) the process for using the product as claimed can be practiced with another materially different product or (2) the product as claimed can be used in a materially different process of using that product (MPEP § 806.05(h)). In the instant case the product as claimed can be used in another and materially different process such as catalytically promoting the purification of internal combustion engine exhaust gas.

Because these inventions are distinct for the reasons given above and the claims set forth in groups I and II have acquired a separate status in the art as shown by their different classification; the search required for the claims of group I is not required for the claims of group II and the claims of groups I and II have acquired a separate status in the art because of their recognized divergent subject matter, restriction for examination purposes as indicated is proper.

Art Unit: 1754

During a telephone conversation with Mr. Kenneth Johnson, applicants' attorney, on 14 May 1999 a provisional election was made with traverse to prosecute the invention of the catalyst, claims 24-38 (group II). Affirmation of this election must be made by applicant in replying to this Office action. Claims 1-23 and 39-40 (group I) are withdrawn from further consideration by the examiner, 37 CAR I 142(b), as being drawn to a non-elected invention.

Applicant is reminded that upon the cancellation of claims to a non-elected invention, the inventorship must be amended in compliance with 37 CAR 1.48(b) if one or more of the currently named inventors is no longer an inventor of at least one claim remaining in the application. Any amendment of inventorship must be accompanied by a petition under 37 CAR 1.48(b) and by the fee required under 37 CAR 1.17(i).

## Specification

sta) The abstract is objected to because it does not particularly point out what the "gaseous sulfur compounds" are that are removed from the gas.

(it b) The abstract does not particularly point out if the gaseous sulfur compounds driven off of the composition are the same as the gaseous sulfur compounds sorbed by the composition.

 $j^{L}c$ ) The abstract does not point out if "precious metal component" is elemental Pt or a Pt compound. If the precious metal component is a Pt compound, the abstract should be amended to describe what this Pt compound is.

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The abstract does not point out if "modifier" is elemental Cu or a Cu compound. If the modifier is a Cu compound, the abstract should be amended to describe what this Cu compound is.

 $d^{\infty}e$ ) In the abstract, it appears that the monolith *is* the support (rather than the support being coated with a monolith). Note that solutions of catalytic metals are typically wash-coated onto monoliths, but monoliths are not normally washcoated onto supports as the abstract suggests.

(*V*f) The title of the invention should be amended to reflect the claims of the elected invention.

## Claim Objection's

In claim 26, --the-- should be inserted between "wherein" and "noble".

a(b)

In claim 31, -- is-- should be inserted between "modifier" and "Cu".

## Claim Rejections - 35 USC § 102

The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the

basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless --

(c) the invention was described in a patent granted on an application for patent by another filed in the United States before the invention thereof by the applicant for patent, or on an international application by another who has fulfilled the requirements of paragraphs (1), (2), and (4) of section 371(c) of this title before the invention thereof by the applicant for patent.

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## Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness

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rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

The factual inquiries set forth in Graham v. John Deere Co., 383 U.S. 1, 148 USPQ 459

(1966), that are applied for establishing a background for determining obviousness under 35

U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.

2. Ascertaining the differences between the prior art and the claims at issue.

3. Resolving the level of ordinary skill in the pertinent art.

 Considering objective evidence present in the application indicating obviousness or nonobviousness.

The person having "ordinary skill in the art" has the capability of understanding the

scientific and engineering principles applicable to the claimed invention. The references of record in this application reasonably reflect this level of skill.

This application currently names joint inventors. In considering patentability of the claims under 35 U.S.C. 103(a), the examiner presumes that the subject matter of the various claims was commonly owned at the time any inventions covered therein were made absent any evidence to the contrary. Applicant is advised of the obligation under 37 CAR 1.56 to point out the inventor and invention dates of each claim that was not commonly owned at the time a later invention was

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made in order for the examiner to consider the applicability of 35 U.S.C. 103(c) and potential 35 U.S.C. 102(f) or (g) prior art under 35 U.S.C. 103(a).

Claims 24-31 and 34-38 are rejected under 35 U.S.C. 102(e) as anticipated by or, in the alternative, under 35 U.S.C. 103(a) as obvious over U.S. Pat. No. 5,792,436 to Feeley et al.

The Feeley pat. discloses a sorbent/catalyst composition (please see col. 1 lns. 59-63) comprising:

a platinum group metal-component, including platinum, rhodium, palladium, ruthenium, iridium and osmium (please see col. 2 ln. 65 to col. 3 ln. 1 and col. 4 lns. 6-8);

a sorbent material that may include oxides of cerium, titanium and copper (please see col. 3 lns, 19-23), and

a support, such as alumina (please see col. 10 lns. 10-18).

Additionally, note that col. 6 Ins. 19-26 and fig. 1 fairly suggests that a monolith is used to support the composition, as required in applicants' claim 34.

The difference between the applicants' claims and the Feeley et al. pat. is that applicants' claims 36-38 describe the weight percent of the catalyst/sorber present in the composition (1 to 50 wt. percent is reported in applicants' claim 35); the weight percent of noble metal component present in the composition (0.005 to 20 wt. percent is reported in applicants' claim 36); the weight percent of the sorber in the catalyst/sorber (70 to 99 wt. percent is reported in applicants' claim 37) and the weight percent of the modifier in the catalyst/sorber (1 to 10 wt. percent is reported in applicants' claim 38) whereas col. 3 lns. 10-35 in the Feeley et al. pat. describes that

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the "Group A" compounds (i. e. compounds of magnesium, calcium, barium, strontium and lanthanum) comprises from 50 to 95 percent by weight of the "Group B" compounds (i. e. oxides of ceria, praseodymia and elements with atomic numbers ranging from 22 through 29), however this is a difference that is submitted to have been obvious to one of ordinary skill in the art at the time the invention was made because a comparison of the applicants' examples to Feeleys' examples show that there is no perceptible difference between the actual quantities of components present in the applicants' and Feeleys' compositions (compare the composition containing from 0.4 to 3.5 g/cubic inch of titania; 0 to 0.08 g/cubic inch of Pt and 0 to 0.3 g/cubic inch of "X" (the modifier: please see pg. 9 ln. 10 in the applicants' specification) set forth on applicants' pg. 10 in their specification to the composition set forth in Example 1 in the Feeley et al. patent which is taught to include 2.0 g/cubic inch of a Pt and Ce washcoat after calcination supported on what appears to be a monolith carrier).

Claims 24-38 are rejected under 35 U.S.C. 103(a) as being unpatentable over U. S. Pat. No. 5,792,436 to Feeley et al. as applied to claims 24-31 and 34-38 above, and further in view of Japan patent document no. 63-264,137 A and U. S. Pat. 4,977,129 to Ernest.

The difference between the applicants' claims and the Feeley et al. pat. is that applicants' claim 32 calls for the presence of Ag oxide in the catalyst/sorber.

The English abstract in Japan pat. doc. no. 63-264,137 A discloses a catalyst/sorbent composition comprising silver oxides which is useful for removing hydrogen sulfide out of gases.

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It would have been obvious to one of ordinary skill in the art at the time the invention was made to include the silver oxides taught in the English abstract of Japan pat. doc. no. 63-264,137 A into the Feeley catalyst/sorbent composition, in the manner required by at least applicants' claim 32, because of the expected advantage of being able to remove any hydrogen sulfide present in the gas being treated.

The difference between the applicants' claims and the Feeley et al. pat. is that the applicants' claim 33 calls for the presence of bismuth oxide in the catalyst/sorber.

Col. 2 lns. 14-23 and lns. 47-52 in the Ernest pat. fairly teaches the use of bismuth oxide as a stabilizer for refractory oxides (such as alumina).

It would have been obvious to one of ordinary skill in the art at the time the invention was made to include the bismuth oxide taught in col. 2 lns. 14-23 and lns. 47-52 in the Ernest pat, into the Feeley catalyst/sorbent composition, as required in at least applicants' claim 33, because of the expected advantage of the bismuth oxide to stabilize the alumina taught in col. 6 lns. 47-50 in the Feeley et al. pat.

The following references are made of record:

U. S. Pat. No. 5,883,037 disclosing a catalyst composition comprising oxides of cerium, zirconium, bismuth and a Group VIII element;

U. S. Pat. No. 5,128,305 disclosing a catalyst comprising oxides of bismuth, silicon, etc. . . and oxides of Pt, Au, Ag, etc. . ., and
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U. S. Pat. No. 5,061,464 disclosing a catalyst comprising sulfur tolerant Pt catalysts for the oxidative removal of CO out of gas.

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Any inquiry concerning this communication should be directed to Timothy Vanoy at

telephone number (703) 308-2540.

Timothy Vanoy/tv

18 May 1999

Tomothy Vanoy TIMOTHY VANOY PATENTEXAMINER ARTUNIT 1400 1754

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PTO-892 (Rev. 9-95)

Part of Paper No.

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Top 2.5 cm Left 2.5 cm Right 1.5 cm Bottom 1.0 cm	Figure legends are poor. Fig.(s)
SIZE: 87/2 x 11 Margins not acceptable. Fig(s)	Numbers and reference characters not oriented in the same
Top (1) Left (L)	direction as the view. 37 CFR 1:84(p)(3) Fig.(s)
Right (R) Bottom (B)	Engligh alphabet not used, 37 CFR 1.84(p)(3) Fig.(s)
5. VIEWS. CFR 1.84(h) REMINDER: Specification may require revision to	Numbers, letters and reference characters must be at least .32 cm (1/8 inch) in height. 37 CFR 1.84(n)(3) Fig (c)
correspond to drawing changes.	.32 cm (1/8 inch) in height. 37 CFR 1.84(p)(3) Fig.(s) 13. LEAD TINES 37 CFR 1.84(q)
Views connected by projection lines or lead lines.	Lead lines cross each other Fig.(s)
Partial views, 37 CFR 1,84(h)(2)	14. NUMBERING OF SHEETS OF DRAWINGS, 37 CFR 1,48(t)
Brackets needed to show figure as one entity.	
Fig.(s) Views not labeled separately or properly.	beginning with number 1. Fig.(s)
Fig.(s)	15. NUMBERING OF VIEWS. 37 CFR 1.84(u)
Enlarged view not labeled separately or properly.	Views not numbered consecutively, and in Abrabic numerals,     beginning with number 1. Fig.(s)
Fig.(s)	16. CORRECTIONS. 37 CFR 1.84(w)
	Corrections not made from PTO-948 dated
	17. DESIGN DRAWINGS, 37 CFR 1,152Surface shading shown not appropriate, Fig.(s)
	Surface shading shown not appropriate, Fig.(s)     Solid black shading not used for color contrast.
· · ·	Fig.(s)
OMMENTS	
	<b>`</b>

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S PATENT AND TRADEMARK OFFICE IN THE U

In Re Application of: CAMPBELL ET AL.

Atty File: GLN 1623

GAU17;

Serial No.: 09/113,258

Filed: 07/10/98

OCT 1 9 1999

Group Art Unit: 1754 Examiner: T. Vanoy

For: PROCESS AND CATALYST/SORBER FOR TREATING SULFUR COMPOUND CONTAINING EFFLUENT

### PETITION FOR EXTENSION OF TIME

Assistant Commissioner for Patents Washington, D.C. 20231

Sir:

In accordance with 37 CFR 1.136(a) applicant requests an extension of TWO (2) months to 10/19/99 to file a response to the OFFICE ACTION mailed on 05/19/99, which was originally due 08/19/99. This petition is made within the second month. A check for 5. \$190,00 (small entity) to cover the fee is enclosed.

Respectfully submitter

Kenneth H. Johnson/ ATTORNEY FOR APPLICANT Reg. No. 22,966 P.O. Box 630708 Houston, Texas 77263 TEL: (713)780-7047 FAX: (713) 780-7671

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PETITION EXTENSION

76/<u>9</u>9 IN THE UNITED STATES PATENT AND TRADEMARK OFFICE In re Application of Attorney File No.: GLN 1623 CAMPBELL ET AL § OCT 1 9 1999 Group Art Unit: 1754 Serial No.: 09/113,258 Examiner: T. Vanoy Filed: 07/10/98 按照正常接入,他们还能停止,不可以数 PROCESS AND CATALYST/SORBER FOR TREATING SULFUR COMPOUND For: CONTAINING EFFLUENT . . . . AMENDMENT UNDER 37 CFR 1.111 Assistant Commissioner for Patents Washington, D.C. 20231 Sir: This amendment is presented in response to the office action mailed 05/19/99. Please enter the following amendments: Charge the title to read: CATALYST/SORBER FOR TREATING SULFUR COMPOUND CONTAINING EFFLUENT 10014 IN THE ABSTRACT: Page 20, delote the paragraph and insert the following: A system for treating the exhaust of an industrial process or combustion source to remove MOSTRA gaseous sulfur compounds which includes contacting the exhaust containing gaseous sulfur Loto lytic compounds with a system of (1) a structured support for example a monolith coated with: (2) a B metal oxide sorber component selected from the group consisting of Ti, Zr, Hf, Ce, Al, Si and mixtures thereof, for example  $\text{TiO}_2$ : (2) a precious metal component, for example Pt metal and a modifier consisting of an oxide Ag, Cu, Bi, Sb, Sn, As, In, Pb, Au or mixtures optionally (A) GOAL/1623.AM1

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	· .
	thereof, such as Cu as a compound. The system captures the gaseous sulfur compounds. Gaseous
	sulfur compounds are then driven off as mainly $H_2S$ and $SO_2$ in higher concentrations in a separate
0)	isolated lower flow stream. The higher concentrations are easier to process to less noxious or useful
() B	sulfur materials and the catalyst/sorber is regenerated. The system also removes carbon dioxide by
•	the oxidation of the carbon monoxide to carbon dioxide.
-	IN THE SPECIFICATION:
	Page 8, line 37, delete H2S and insoft therefor $H_2S$
	IN THE CLAIMS:
<u> </u>	CANCEL CLAIMS 1-23 AND 39-40 WITHOUT PREJUDICE.
	A. (AMENDED) A regenerable catalyst/sorber for removing gaseous sulfur compounds
17	from gaseous streams comprising a noble metal catalyst component, a metal oxide sorber component נוף דס
$\mathcal{D}$	selected from Ti, Zr, Hf, Ce, Al, Si or mixtures thereof and $0.3 \text{ g/in}^3$ of a modifier component
Jer Unionagy	consisting of an oxide of Ag, Cu, Bi, Sb, Sn, As, Jn, Pb, Au or mixtures thereof.
	In claim 31, line 2 insert-is- between "modifier" and "Cu".
	1. (AMENDED) A catalytic structure comprising a monolith carrier and a regenerable
m b	catalyst/sorber for removing gaseous sulfur compounds from gaseous streams comprising a noble
$\mathcal{V}_{\alpha}$	metal catalyst component, a metal oxide sorber component selected from Ti, Zr, Hf, Ce, Al, Si or $\mu p + o$
В	mixtures thereof and $0.3 \text{ g/in}^3 \text{ of a modifier } \frac{\text{component}}{\Lambda}$ consisting of an oxide of Ag, Cu, Bi, Sb, Sn,
Acronat	As, In, Pb, Au or mixtures thereof deposited on said carrier.
QU	26. (AMENDED) The catalyst/sorber according to claim 28 wherein the noble metal is Pt.
	ADD NEW CLAIMS:
,	41. A catalyst/sorber comprising a noble metal catalyst component, a metal oxide sorber
$\mathcal{O}$	component selected from Zr, Hf, Si or mixtures thereof and a modifier consisting of an oxide of Ag,
	GOAL/1623.AM1 2
	21.
	$\alpha$

Cu, Bi, Sb, Sn, As In, Pb, Au or mixtures thereof.

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42. A catalyst/sorber comprising a noble metal catalyst component, a metal oxide sorber component selected from Ti, Zr, Hf, Ce Al, Si/or mixtures thereof and a modifier consisting of an oxide of Sb, Sn, As In, Pb, Au or mixtures thereof.

### REMARKS

Claims 24-38 remain in the application.

Applicants acknowledge the telephone restriction requirement and the election of Group II, claims 24-38 with traverse on May 14, 1999.

The title has been changed as suggested by the examiner. Please note that the PTO filing receipt contains a PTO misspelling of the word "SORBER".

The Abstract was objected to because it did not specifically state the gaseous sulfur compounds were removed. The terminology to show removal was "capture" of the gaseous sulfur compounds, however for clarity this has now been changed to "remove". Other changes to the Abstract have been made to better represent the entire scope of the disclosure and to clarify the nature of the system. The examiner also objects that the Abstract does not state that compounds are the same compounds as those sorbed. The gaseous sulfur compounds that may be removed are not limited. Thus H2S, SO2, mercaptans, thiophines, etc may be removed, "however the desorbed components released during regeneration consisted mainly of a mixture of H2S and SO2" Specification, page 8, lines 4-5. The Abstract now recites this. The examiner also objected that the nature of the Pt and Cu, i.e., elemental or compound was not indicated. The preparation of a TiO<sub>2</sub>/Pt washcoat on page 10 of the specification is disclosed by the process of incipient wetness, which is well known to be the process in which a solution of a soluble compound is applied to a solid. In this case the solid is the TiO<sub>2</sub> and the soluble solution is that of a Pt compound. There are many 3

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solutions of the various noble metal compounds, however two preferred procedures for ease of use are disclosed in the enclosed U.S. Pat No.5,451,558 (aqueous system, see col. 5 at lines 26 et seq) and WO 98/05412 (organic system, page 8 under b. Pt Catalyst (GLPM) Washcoat). In either system a platinum compound is applied to a support or carrier from solution and converted to the metal by heating at around 500 °C. The syntax regarding the support has been corrected.

Claims 26 and 31 have been amended as suggested by the examiner to overcome objections. Claims 24-38 were rejected under 35 USC 102(e) or 103(a) over Feeley et al. The examiner has acknowledged certain distinctions between the claimed invention and Feeley et al, however there is a very substantial difference which is now recited in all of the claims. In particular Feeley et al do not disclose a modifier, although the sorber of Feeley et al may include a Cu oxide. Feeley et a. discloses that the sorbent material may be Mg, Ca, Sr, Ba, La, Ti, V, Cr, Mr, Fe, Co, Ni, and Cu, and the oxidation catalyst Pt. Feeley et al gives no guidance as to amounts of components (only the relative amounts of group A and group B components of the sorbent), hence one must turn to the examples to determine at least some definition of the sorbent/oxidation catalyst combination. In Feeley et al. Example 1-3 the typical loading of Pt/CeO2, PtMnO2 or Pt/Al2O3 is 2 g/in3. The preparative process does not give sufficient information to determine the ratio of Pt to sorbent (CeO2, MgO or Al<sup>2</sup>O<sup>3</sup>) but it is clear that the sorbent is present in the washcoat at about 10 times the weight of Pt. Thus in the loading the sorbent is present at about 1.9 g/in<sup>3</sup> and the Pt at about 0.2 g/in<sup>3</sup>. The sorbent as noted above maybe comprised of an A group and a B group. The cu noted by the examiner is in the B group. No example of a composition of groups A and B is given, but as disclosed the B component if present comprises 40-95 wt % of the sorbent. Thus, out of the 1.9 g/in3, (Group A and B), Cu oxide as a sorbent component would be present in the area at from 0.95 g/in<sup>3</sup> (50%) to 1.8 g/in<sup>3</sup> (95%) (col 3, lines 19-25). These are not exact numbers because the

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information available in Feeley et al does not allow exactness but they indicated the general amount of a Cu oxide present.

In the present examples the amount of modifier used was from 0 to 0.3 g/in<sup>3</sup>. See examples general but specifically Example 5, 6, 7, 9, 10 and Specification Page 10 at line 21. Since the present claims recite the presence of a modifier they have been limited to 0.3 g/in<sup>3</sup> which is supported at page 10 line 21 and generally. The Feeley et al., reference would not suggest the use of less than about 0.95 g/in<sup>3</sup> Cu (see also specification at page 9, lines 1-18). Thus the maximum amount of modifier disclosed by Feeley et al, is at least three times the amount routinely recited in the present claims. The claims as amended overcome the anticipation.

Similarly there is no basis in Feeley et al to suggest any of the other recited modifiers and the 103 over Feeley et al alone is overcome.

The rejection of claims 24-38 under 35 USC 103(a) over Feeley et al in view of the Japanese reference and Ernest is respectfully traversed.

The Japanese reference abstract discloses that Ag oxide adsorbs  $H_2S$ , and only a support of carbon, bentonite, zeolite, silica acid gel or alumina and diatomite, activated clay or polymer water absorbent are disclosed as other components. From the abstract alone it is not possible to determine if the Japanese reference is analogous art. Moreover, Feeley et al teaches only sorbing  $SO_2$  and made no mention of  $H_2S$ , whereas the Japanese reference was removing the odorous  $H_2S$  and made no mention of  $SO_x$ . Feeley et al would have no incentive to add Ag oxide to its sorber component and even if it did it would be at 3 times the amount claimed. As reported in the present specification, "The role of the copper and other modifiers appears to reside only in changing the regeneration of the catalyst to diminish  $H_2S$  formation." (Specification page 9). Feeley et al exhibited no interest in the form of the desorbed gas and hence one of ordinary skill would have no motivation to add a

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silver component to the Feeley composition."

Ernest discloses only the removal of  $H_2S$  from emissions. Since Feeley et al as noted above was only interested in So<sub>x</sub> removal, one of ordinary skill in the art would not have any motivation to import any particular element from Ernest into the Feeley et al composition.

"Obviousness cannot be established by combining teachings of the prior art to produce the claimed invention, <u>absent</u> some teaching suggestion or incentive supporting the combination." <u>In</u> <u>re Geiger</u>, 2 USPQ2d 1276 (CAFC 1987). Hence, without the requisite teaching, suggestions or incentives there is no <u>prima facie</u> case and the rejection must fail.

The CCPA and CAFC have tried to look at whether something was obvious to try or obvious at the beginning. This is not rebutting the <u>prima facie</u> case but determining whether a <u>prima facie</u> case has been made out. If a claimed invention was only "obvious to try", even had the art been properly combined, the invention is patentable. See <u>In re Huellmantel</u>, 139 USPQ 496,499 (CCPA) <u>In re Panzer</u>, 144 USPQ 415, 417 (CCPA), <u>In re Tomlinson</u>, 150 USPQ 623, 626 (CCPA), <u>In re <u>Henderson</u>, 146 USPQ 372 (CCPA), and more recently <u>In re Geiger</u>, 2 USPQ2d 1276 (CAFC 1987). Some guidance as to when something was obvious to try was given in <u>re Tomlinson</u>, supra</u>

at 426 where the court held:

Slight reflection suggests, we think, that there is usually an element of 'obvious to try' in any research endeavor, that it is not undertaken with complete blindness but rather with some semblance of a chance of success, and that patentability determinations based on that as the test would not only be contrary to statute but result in a marked deterioration of the entire patent system as an incentive to invest in those efforts and attempts which go by the name of 'research'.

However, the very best discussion of when something is only "obvious to try" is given by the court in <u>re O'Farrell, supra</u>. The present rejection falls into the improper first case of obvious to try. "Obviousness cannot be established by combining teachings of the prior art to produce the claimed

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invention, <u>absent</u> some teaching suggestion or incentive supporting the combination." <u>In re Geiger</u>, <u>supra</u>. Hence, without the requisite teaching suggestions or incentives there is no <u>prima facie</u> case and the rejection must fail. See also <u>In re Fine</u>, 5 USPQ2d 1596 and <u>Ex parte Levengood</u>, 28 USPQ2d 1300 (BdPatApp 1993).

Applicants submit that, at best, it is only "obvious to try" the combinations of components recited in the present claims since the only way to do this would be through routine experimentation. However, because of the disparity of and lack of commonality between Feeley et al and the secondary references, it is submitted that the proposed art combination must fail and that no <u>prima</u> <u>facie</u> case of obviousness has been made out.

The examiner urges that, "The person having 'ordinary skill in the art' has the capability of understanding the scientific and engineering principles applicable to the claimed invention." However, that description defines not only the person of ordinary skill in the art but the most skilled person in that art also. The level of skill in a particular art is the issue. In <u>In re GPAC Inc</u>, 35 USPQ2d 1116 at 1121, (fed. cir. 1996) the court determined the specific level of skill for the relevant art after a consideration of the references, however, the rule as promulgated by the court is "The person of ordinary skill in the art is a hypothetical person who is presumed to know the relevant prior art." The types of problems, the prior art, prior solutions, the rate of innovation and the educational level in the field, are some of the factors for the court to consider in determining the skill level. In the absence of some fact or evidence on the level of ordinary skill in this art, only the prior art is present here. The examiner's assertion that "The references of record in this application reasonably reflect this level of skill", is thus only partially correct. Furthermore, even a high level of skill in the art cannot be relied on to provide the motivation to combine the art references in the manner under the claimed invention obvious, absence some explanation of why such combination was suggested.

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### In Re Rouffet, 47 USPQ2d 1453 (CAFC 1998).

Quite clearly one of ordinary skill in an art must have some familiarity with and experience in the art in question. In other words, in addition to merely understanding scientific and engineering principals, the person of ordinary skill must be able to make practical application thereof in the art in question. The patent references reflect a level of skill much higher than ordinary because they are by law disclosures of inventions which must be shown to be above the ordinary level to become patents, although the disclosure is now part of the prior art. However, if, for example, most of the individuals working in an art had patents, then the patents alone might represent the level of skill in the art. The issue still remains as to what is the "art". There has been no evidence adduced as to what defines the art which embraces the present invention. If the examiner seeks to insert a "level of skill" text then it is the examiner who must furnish evidence of that level of skill. It is settled that an invention may be held to have been obvious without a specific finding of a particular level of skill. <u>Chore-Time Equip., Inc. v. Cumberland Corp.</u>, 713 F.2d 774, 779 n.2, 218 USPQ 673, 676 n.2 (Fed. Cir. 1983).

If the references as suggested by the examiner reasonably reflect the skill in the art, then the examiner agrees that within the four corners of the references there <u>must</u> be found the motivation and the technology to make the <u>prima facie</u> case of obviousness. Since the motivation is lacking or any reason based on the teachings in the secondary references to make the combination proposed, the rejection must fail.

Applicants have addressed the cited art and the examiner's application thereof and by amendment of the claims have clarified that claimed as distinguished from the prior art. It is submitted that all of the amended claims and added claims are now in condition for allowance which is solicited in due course.

#### GOAL/1623.AM1

Respectfully Submitted,

Kenneth H. Johnson ATTORNEY FOR APPLICANT Reg. No. 22,966 P.O. Box 630708 Houston, Texas 77263 TEL: (713) 780-7047 FAX: (713) 780 7671

### CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to:

Assistant Commissioner for Patents Washington, D.C. 20231

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:	IN THE UNITED STATES PA	TENT & TRADEMARK OFFICE
	·	Case Docket No.; GLN 1623
	In Re Application of:	
· •	Serial No: 09/113,258	Art Unit: 1754
	907 1 9 1999 Hr S	
	Filed: 07/10/98	Examiner: T. Venoy
	FOR: PROCESS AND CALENDARY OR BER FOR TR	EATING SULFUR COMPOUND CONTAINING EFFLUENT
	ASSISTANT COMMISSIONER FOR PATENTS	
	Washington, D. C. 20231	
	Sir:	
	Transmitted herewith is an amendment in the above-ic	
	<ul> <li>[X] Petition for Extension of Time (2 months) (fee</li> <li>[] No additional fee is required.</li> <li>[] The fee has been calculated as shown below (Col. 1) Col. 2) (Col. 3) SMAI</li> </ul>	N LL ENTITY OTHER THAN
	CLAIMS HIGHEST NO. PRESENT RAT	E ADDIT. RATE ADDIT.
	REMAINING PREVIOUSLY EXTRA FEE AFTER PAID FOR	
	AMENDMENT	· · · · · · · · · · · · · · · · · · ·
	TOTAL * 17 MINUS ** 40 = 0	x9=\$ x18=\$ 
	INDEP * 4 MINUS ** 4 = 0	
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·	ADDIT. FEE TO	DTAL \$OR_TOTAL \$
	* If the entry in Col. 1 is less than the entry in Col. 2, ** If the "Highest Number Previously Paid For" IN THI	
-	*** If the "Highest Number Previously Paid For" IN TH	IS SPACE is less than 3, write "3" in this space.
	The "Highest Number Previously Paid For" (Total or Inc 1 of a prior amendment or the number of claims origin	tependent) is the highest number found from the equivation tox in Col. hally filed.
	· · · ·	
•		1740 in the amount of \$ A duplicate of this sheet is enclosed. Petition for Extension of Time (2 months) is enclosed.
		arge payment of the following fees associated with this communication nt No, 10-0740. A duplicate copy of this sheet is attached.
	[X] Any filing fees under 37 CFR 1.16 for the pre-	
	[X] Any patent application processing fees under	ar 37 CFR 1.17.
		Respectfully submitted
		Kan III. KAL
		1 miner 1. 11 1
		Kenneth H. Johnson Reg. No. 22,966
		Reg. No. 22,966 Attorney for Applicant
	DATE: 10 15 97	Reg. No. 22,966
	1 1 1 9 3	Reg. No. 22,966 Attorney for Applicant

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ŧį E ጉ OCT 1 9 1999 MAIL CERTIFICATE I here we that this correspondence is being deposited with the United Postal Service as first class mail in an envelope addressed to: Assistant Commissioner for Patents Washington, D.C. 20231 or UM TC 1706 MALL ROOM 五 25 1999

		Address: COMMIS Washing	S DEPARTMENT OF COMMERCE ademark Office SIONER OF PATENTS AND TRADEMARKS Icon, D.C. 20231	
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09/113,258 07/10/98 C	AMPBELL		L GLN-1623	
			EXAMINER	
	IM22/1118	3	VANOY, T	
KENNETH H JOHNSON P O BOX 630708 🐄			ART UNIT PAPER NUMBER	
HOUSTON TX 77263		• • •	1754 7	
			DATE MAILED: 11/18/99	
This is a communication from the examiner in char COMMISSIONER OF PATENTS AND TRADEMAI	ge of your application RKS	i.	· .	
N	IOTICE OF ALL	OWABILITY		
All claims being allowable, PROSECUTION ON THE previously mailed), a Notice of Allowance and Issue I This communication is responsive to <u>THE Amer</u> The allowed claim(s) is/are <u>24-38</u>	MERITS IS (OR RE Fee Due or other ap	SMAINS) CLOSED I propriate communic 19 Dcf. 1999	In this application. If not included herewith (or ation will be mailed in due course. <u>FINTERVIEW</u> of 12 Mor. (999.	
The drawings filed on	are acceptab	le.		
Acknowledgement is made of a claim for foreign				
All Some* None of the CERTIFIED	•		been	
recalved.				
received in Application No. (Series Code/Se	arial Number)		· · · · · · · · · · · · · · · · · · ·	
received in this national stage application from		Bureau (PCT Rule 1	7.2(a)).	
*Certified copies not received:			· · · ·	
Acknowledgement is made of a claim for domes	tic priority under 35	U.S.C. § 119(e).		
A SHORTENED STATUTORY PERIOD FOR REPL FROM THE "DATE MAILED" of this Office action. F time may be obtained under the provisions of 37 CF	Y to comply with the ailure to timely com	requirements noted	below is set to EXPIRE THREE MONTHS INDONMENT of this application. Extensions of	:
Note the attached EXAMINER'S AMENDMENT declaration is deficient. A SUBSTITUTE OATH	OR DECLARATION	ORMAL APPLICATI I IS REQUIRED.	ON, PTO-152, which discloses that the oath or	
Applicant MUST submit NEW FORMAL DRAW	NGS	•		
because the originally filed drawings were de			2	
X including changes required by the Notice of D	Iraitperson's Patent	Drawing Review, P	CO-948, attached hereto or to Paper No. <u>5</u>	•
including changes required by the proposed of by the examiner.	drawing correction fil	led on	, which has been approved	1
including changes required by the attached E				
Identifying indicia such as the application nu The drawings should be filed as a separate p	aper with a transmi	ittal letter addresse	d to the Official Dranperson.	
Note the attached Examiner's comment regard				
Any reply to this notice should include, in the upper applicant has received a Notice of Allowance and I ALLOWANCE should also be included.	r right hand comer, t ssue Fee Due, the it	he'APPLICATION N SSUE BATCH NUM	IUMBER (SERIES CODE/SERIAL NUMBER). BER and DATE of the NOTICE OF	if
Attachment(s)				
Notice of Reference's Cited, PTO-892				
Information Disclosure Statement(s), PTO-1	449, Paper No(s)			
Notice of Draftsperson's Patent Drawing Rev				
Notice of Informal Patent Application, PTO-1		s. N		
Interview Summary, PTO-413				
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Examiner's Amendment/Comment	ant for Deposit of Dia	Jonical Material		
		ological Material		

\*U.S. GPO:1998/444-443

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Art Unit: 1754

Page 2

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#### EXAMINER'S AMENDMENT

An examiner's amendment to the record appears below. Should the changes and/or additions be unacceptable to applicant, an amendment may be filed as provided by 37 CFR 1.312. To ensure consideration of such an amendment, it MUST be submitted no later than the payment of the issue fee.

Authorization for this examiner's amendment was given in a telephone interview with Mr. Kenneth Johnson, Applicants' attorney, on 12 November 1999.

The application has been amended as follows:

In line 3 of the abstract, delete ": (1)".

In line 3 of the abstract, between "a" and "system" insert --catalytic-.. In line 3 of the abstract, substitute --(i)-- in lieu of "(2)". In line 5 of the abstract, substitute --(ii)-- in lieu of "(3)". In line 6 of the abstract, substitute --(iii)-- in lieu of "(4)". In line 10 of the abstract, substitute --monoxide-- in fieu of "dioxide". In claim 24 line 3, between "and" and "0.3" insert --up to--. In claim 26, between "metal" and "is" insert --component--. In claim 29, line 2, between "modifier" and "is" insert --component--. In claim 30 line 2, between "modifier" and "is" insert --component--. In claim 31 line 2, between "modifier" and "is" insert --component--. In claim 32 line 2, between "modifier" and "is" insert --component--. In claim 31 line 2, between "modifier" and "is" insert --component--. In claim 32 line 2, between "modifier" and "is" insert --component--. In claim 32 line 2, between "modifier" and "is" insert --component--.

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Page 3

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In claim 34 line 4, between "and" and "0.3" insert -up to-..

In claim 37 line 2, between "said" and "sorber" insert -- metal oxide --.

In claim 38 line 2, between "modifier" and "comprises" insert --- component---

Capcel claims 41 and 42.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Timothy C. Vanoy whose telephone number is 703-308-2540.

fel

Timothy Vanoy/tv November 12, 1999

A COMMA HUS BEEN INSERTED BETWEEN "As" AND "In" IN THE ABSTRACT AND CLAIMS 24 AND 34.

STEVEN P. GRIFFIN SUPERVISORY PATENT EXAMINER TECHNOLOGY CENTER 1700 11/17/99

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Page 2

Art Unit: 1754

## PROPOSED

### EXAMINER'S AMENDMENT

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In line 3 of the abstract, delete ": (1)".

In line 3 of the abstract, between "a" and "system" insert -catalytic-.

In line 3 of the abstract, substitute --(i)- in lieu of "(2)".

In line 5 of the abstract, substitute --(ii)-- in lieu of "(3)'.

In line 6 of the abstract, substitute --(iii)-- in lieu of "(4)".

In line 10 of the abstract, substitute --monoxide---in lieu of "dioxide".

In claim 26, between "metal" and "is" insert -component-.

In claim 29, line 2, between "modifier" and "is" insert -component ---.

In claim 30 line 2, between "modifier" and "is" insert -component-,

In claim 31 line 2, between "modifier" and "is" insert -component-.

In claim 32 line 2, between "modifier" and "is" insert -component-.

In claim 33 line 2, between "modifier" and "is" insert -component-.

In claim 37 line 2, between "said" and "sorber" insert -metal oxide-.

Page 3

Art Unit: 1754

In claim 38 line 2, between "modifier" and "comprises" insert -component-.

Cancel claims 41 and 42.

Ο

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Timothy C. Vanoy whose telephone number is 703-308-2540.

Timothy Vanoy/tv November 12, 1999

Remarks: Claims 41 = 42 are at least obvious from U.S. partents 5,883,037; 5,128,305 and 4,977,129 already of record. A response to this projocted Exmr's Amend Frecewed on or by 17 Nov. 1999 would be greatly appreciated. Respectfully

U.S Department of Commerce Patent & Trademark Office Washington, DC 20231



# Art Unit 1754

To: Mr. Kenneth Johnson Application SN: 091 113,253 Attorney Docket #: GLN 1623 FAX: 713,780,7671 Tel.: 713,780,7047

From: Tim Vanoy Tel.: (703) 308-2540 Fax: (703) 305-6078 Date: (こ パッ、パタイ

Number of Pages: 3

Note: Proposed Examiner's Amendment

Art Unit 1754 2021 South Clark Place Crystal Plaza 3 Arlington, VA 22202 50 NOT REMOVE FRom F

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U.S. Peters and Trademark Office PTO-892 (Rev. 9-96) A copy of this reference is not being funished with this Office action. (See Manual of Patent Examining Procedure, Section 707.05(a).)

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	Application No. 09/113,258	Applicant(s)	CAMPBEL	L et al.	
Interview Summary	Examiner		Group Art Unit		
	Timothy VA	NOY	1754		
All participants (applicant, applicant's representative, PT	O personnel):				
1) Timothy VANOY	(3)				
	(3)				
2) <u>Mr. Kenneth Johnson, Attorney</u>	 ¥				
Date of Interview Nov 12, 1999	— .				
Exhibit shown or demonstration conducted: 🛛 Yes	🕅 No. If yes, brief d	escription:			
Exhibit shown or demonstration conducted: 🗌 Yes	🕅 No. If yes, brief d	escription:		• .	
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E UNITED STATES PATENT AND TRADEMARK OFFICE § Atty File: GLN 1623 In Re Application of: CAMPBELL ET AL § Group Art Unit: 1754 Serial No.: 09/113,258 § Examiner: . T. VANOY δ Filed: 07/10/98 For: CATALYST/SORBER FOR TREATING SULFUR COMPOUND CONTAINING EFFLUENT Publishing Division EFFLUENT DEC - 6 1999 LETTER TO THE OFFICIAL DRAFTSPERSON 05 TECH3 Assistant Commissioner for Patents Washington, D.C. 20231 Sir: Please find enclosed three (3) photocopies of each sheet (8-4A) of the new formal drawings as required for your review and entry in to the above cited application which was allowed 11/18/99 (copy of Notice of Allowance and PTO 948 are enclosed). Respectfully Submitted, Kenneth H. Johnson ATTORNEY FOR APPLICANT Reg. No. 22,966 P.O. Box 630708 Houston, Texas 77263 TEL: (713) 780-7047 FAX: (713) 780 7671 cc: GLN

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### CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to:

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Box ISSUE FEE Assistant Commissioner for Patents Washington, D.C. 20231

on 12

emi KENNETH H. JOHNSON

















DEPARTMENT OF COMMERCE-Patent and Trademark Office. PORM PTO 948 (REV. 11-97) **NOTICE OF DRAFTPERSON'S** PATENT DRAWING REVIEW the Draftpenson under 37 CFR 1.84 or 1.152. Antiobjected to by the Draftperson under 37 CFR 1.84 or 1/152 as indicated below. The Examiner will require submission of new, corrected drawings who necessary. Connected drawings must be submitted according in the instructions on the back of this notice. . . 1. DRAWINGS. 37 CFR 1.84(a): Acceptable categories of drawings: 7, SECTIONAL VIEWS. 37 CFR 1.84(b)(3) Hatching not indicated for sectional portions of an object. Black ink. Color. Color drawing are not acceptable until petition is granted. Fig.(s) Fig.(s)\_ Sectional designation should be noted with Arabic or Pencil and non black ink is not permitted. Fig(s)\_ Roman numbers. Fig.(s)\_ 2. PHOTOGRAPHS. 37 CFR 1.84(b) 8. ARRANGEMENT OF VIEWS. 37 CFR 1.84(i) \_ Photographs are not acceptable until petition is granted; . Words do not appear on a horizontal, left-to-right fashion when 3 full-tone sets are required. Fig(s) page is either upright or turned, so that the top becomes the right Photographs not properly mounted (must brystol board or side, except for graphs. Fig.(s)\_ 4 photographic double-weight paper). Fig(s) Views not on the same plane on drawing sheet. Fig.(s) Poor quailty (half-tone). Fig(s)\_ 9. SCALE 37 CFR 1.84(k) 3. TYPE OF PAPER. 37 CFR 1.84(e) Scale not large enough to show mechansim without crowding 🗧 Paper not fiexible, strong, white and durable. when drawing is reduced in size to two-thirds in reproduction. rig.(s) <u>Ensured</u>, alterations, overwritings, interlineations, folds, copy machine inarts not acceptable. (too thin). Mylar, velluta paper is not acceptable (too thin) Lines, numbers & letters not uniformly thick and well defined, clean, durable and black (poor line quality). ्रते कोंगे जन्म 'Fig(s)\_\_\_\_\_ Fig.(6) -10 4. SIZE OF PAPER. 37 CFR 1.84(F): Acceptable Bizes: 11 States (1) 11 SHA 1 G. H-21.0 cm by 29.7 cm (DIN size A4) Solid black areas pale. Fig.(s)\_ . 21.6 cm by 27.9 cm (8 1/2 x 11 inches) Solid black shading not permitted. Fig.(s) ...... All drawings sheets not the same size, Shade lines, pale, rough and blurred. Fig.(s) Sheet(s) Comments in the discrete state of the light of t Top 2.5 cm Left 2.5 cm Right 1.5 cm Bottom 1.0 cm gine and gint an Numbers and reference characters not plain and legible. mAg (0) [-]6 SIZE: A4 Size Top 25 cm Left 2.5 cm Right 1.5 cm Boltom 1.0 cm Figure legends are poor. Fig.(s)\_ SIZE: 81/2 x 11 Numbers and reference characters not oriented in the same Margina not acceptable. Fig(s) and the market in the direction as the view. 37 CFR 1.84(p)(3) Fig.(s) Left(L) Ton (T) Engligh alphabet not used. 37 CFR 1.84(p)(3) Fig.(s) Right (R)\_ Bottom (B) 6. VIEWS. CFR 1.84(b) 2 . That is in shaceast and the second state ....Numbers, letters and reference characters must be at least REMINDER: Specification may require revision to correspond to drawing changes. - Views connected by projection lines of lead lines. A 1999 (1997) - Han Lead lines cross each other. Fig.(s) Lead lines missing. Fig.(s) Fig.(s)\_ \_\_\_\_\_ Partial views, 37 CFR 1.84(h)(2) 14. NUMBERING OF SHEETS OF DRAWINGS. 37 CFR 1.48(t) Brackets needed to show figure as one entity. Sheets not numbered consecutively, and in Ababic numerals :-- Fig.(s) beginning with number 1. Fig.(s) Views not labeled separately or properly. 15.NUMBERING OF VIEWS: 37 CFR 1.84(u) Views not numbered consecutively, and in Abrabic numerals, Fig.(s) 👌 Enlarged view not labeled separately or properly. beginning with number 1. Fig.(s) Fig.(s) 16. CORRECTIONS, 37 CFR 1.84(w) \_Corrections not made from PTO-948 dated 17. DESIGN DRAWINGS, 37 CFR 1.152 \_\_\_\_Surface shading shown not appropriate. Fig.(s)\_\_\_\_ hid black shuding not used for color contrast. COMMENTS 52.4 r. REVIEWER ELEPHONE NO

UNITED STATES DEPARTMENT OF COMMERCE Patent and Trademark Office NOTICE OF ALLOWANCE AND ISSUE FEE DUE 1118 KENNETH H JOHNSON P 0 BOX 630708 HOUSTON TX 77263 APPLICATION NO. ----- FILING DATE. TOTAL CLAIMS Stand Examiner and group art units and the second DATE MAILED 07/10/99 ▼ VANOV 1754 258 ດ971/13 11/18/99 Find Nar Applicar 0 Days COMPRES 35 USC 154(b) term ext: = TILE OF INVENTIONATALYST/SORBER, FOR TREATING SULFUR COMPOUND CONTAINING EFFLUENT (AS AMENDED) CLASS-SUBCLASS ATTY'S DOCKET NO. BATCH NO. - SMALL ENTERY " CONFEE DUE ?! DATEDUE APPLN: TYPE 502-325.000 GEN-1623 THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. PROSECUTION ON THE MERITS IS CLOSED. THE ISSUE FEE MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED. HOW TO RESPOND TO THIS NOTICE: 2.5 22 <sup>2</sup>8 I. Review the SMALL ENTITY status shown above. If the SMALL ENTITY is shown as YES, verify your current SMALL ENTITY status: If the SMALL ENTITY is shown as NO: a de la compañía de l A. If the status is changed, pay twice the amount of the A. Pay FEE DUE shown above; of FEE DUE shown above and notify the Patent and Trademark Office of the change in status, or B. If the status is the same, pay the FEE DUE shown B. File verified statement of Small Entity Status before, or with; above. payment of 1/2 the FEE DUE shown above. II. Part B-Issue Fee Transmittal should be completed and returned to the Patient and Trademark Office (PTO) with your ISSUE FEE. Even if the ISSUE FEE has already been paid by charge to deposit account. Part B issue Fee Transmittal should be completed and returned. If you are charging the ISSUE FEE to your deposit account, section "4b" of Part B-Issue Fee Transmittal should be completed and an extra copy of the form should be submitted. III. All communications regarding this application must give application number and batch number. Please direct all communications prior to issuance to Box ISSUE FEE unless advised to the contrary. IMPORTANT REMINDER: Utility patents issuing on applications filed on or after Dec. 12, 1980 may require payment of maintenance fees. It is patentee's responsibility to ensure timely payment of maintenance fees when due. ·: .4 YOUR COPY PTOL-85 (REV. 10-96) Approved for use through 06/30/99, (0651-0033) U.S. GPD: 1996-437-639/80022

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PART B (SSUE FEE TR)	INSMITTAL ECT GAN
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First Named       Applicant       CAMPBELL,       35 USC 154 (ft)         TITLE OF       WENTRONCATALYST/SORBER FOR TREATING SULFUR COMP( (AS AMENDED)       CLASS-SUBCLASS       BATCH NO.       APPLALTUR         1       GLN-1623       502-325,000       E38       UTTL         1       GLN-1623       502-325,000       E38       UTTL         1.       Change of correspondence address or indication of * Fee Address' (37 CFR 1.963).       2; For printil (1) the name of the name of the name of the property of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.       2; For printil (1) the name of the name of the name of the name of the property attached.       2; For printil (1) the name of the name of the name of the property attached.         3.       ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print Or type).       PLEASE MOTE: Unless an assignee to learn the obern on assignee data will appear on the petern, inclusion of assignee data is only appropriate when an assignment has been previously, submitted to the PTO or is being submitted under separate cover. Completion of this form is NOT a substitute for filling an assignment.         (A) NAME OF ASSIGNEE GOAL LINE ENVIronTMENTAE       TECHMOL OF MES 1.4C. GOAL LINE ENVIronTMENTAE         (B) RESIDENCE: (CITY & STATE OR COUNTRY)       Please check the appropriate assignee category indicated below (will not be printed on the peternt)         Didividual       Moorporation or other prival in directed below (will not be printed on the peternt)	2) term ext. = 0 Days.         DUND CONTAINING EFFLUENT         E       SMALL ENTITY, FEEDUE       DATE DUE         ITY YES \$605.00 02/18/00         gents OR, Blemathedy, (2)       Accommentation of a control of the con
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