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[54] **PROCESS FOR REDUCING THE ARSENIC CONTENT OF GASEOUS HYDROCARBON STREAMS BY THE USE OF SELECTIVE ACTIVATED CARBON**

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[51] **Int. Cl.** **C10g 17/00,** C10g 17/02

[58] **Field of Search** 208/88, 251, 307, 253; 423/438

[56] **References Cited**

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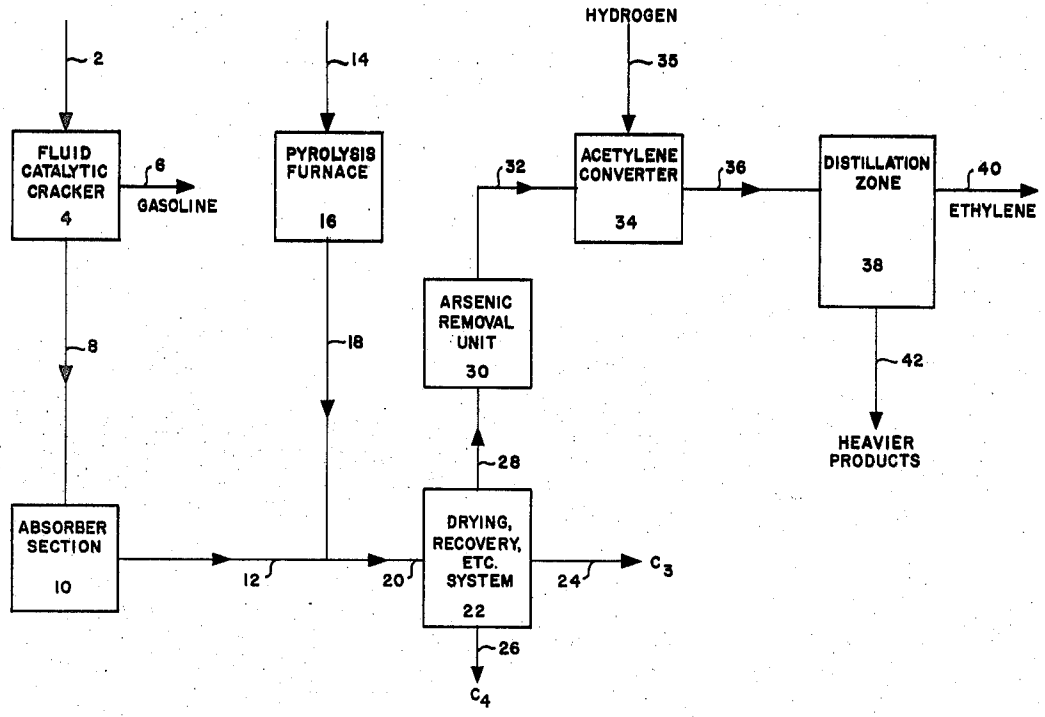
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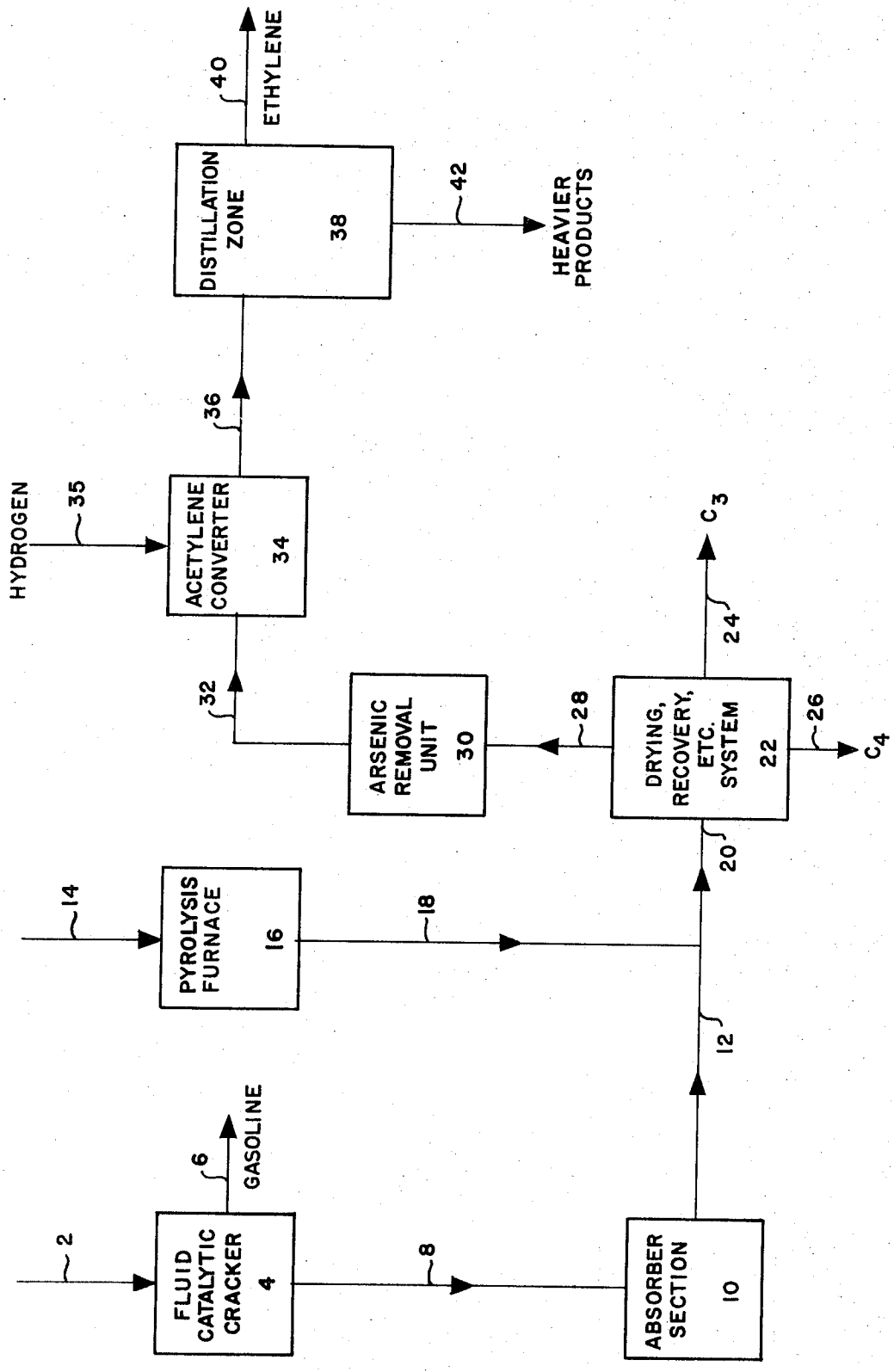
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[57] **ABSTRACT**

A process for reducing the arsenic content of a gaseous hydrocarbon stream by contacting the stream with a sorbent comprising a specially defined metals content activated carbon prepared from a bituminous coal.

10 Claims, 1 Drawing Figure




FLUID CATALYTIC CRACKER 4
PYROLYSIS FURNACE 16
ACETYLENE CONVERTER 34
DISTILLATION ZONE 38
ARSENIC REMOVAL UNIT 30
DRYING, RECOVERY, ETC. SYSTEM 22
ABSORBER SECTION 10
HYDROGEN
ETHYLENE
GASOLINE
HEAVIER PRODUCTS
C_3
C_4
2
6
8
12
14
18
20
24
26
28
32
35
36
40
42

PROCESS FOR REDUCING THE ARSENIC CONTENT OF GASEOUS HYDROCARBON STREAMS BY THE USE OF SELECTIVE ACTIVATED CARBON

This invention relates to the removal of arsenic from gaseous streams, and more particularly to a process for reducing the arsenic content of a gaseous hydrocarbon stream, by the use of a specially defined metals content activated carbon derived from bituminous coal.

BACKGROUND OF THE INVENTION

Catalytic cracking is one of the principal methods for refining petroleum fractions to recover more valuable hydrocarbon products such as gasoline. The unit in which the cracking operation takes place generally employs a fluidized bed and thus is termed a fluid catalytic cracking (FCC) unit. A variety of lower boiling products in gaseous form are discharged from the FCC unit and these are usually further treated to recover separate hydrocarbon fractions, e.g. ethylene. This further treatment of FCC vapors may, as in the case of the hydrogenation of acetylene, involve the use of a noble metal catalyst. As is well known, noble metal catalysts are rapidly deactivated by feedstock impurities such as arsenic. It thus becomes desirable to reduce the arsenic content of the FCC gases to the lowest possible level before subjecting them to further treatment.

It should be noted that the exact form in which arsenic is present in FCC gases is difficult to determine. It is known, however, that FCC gases in which arsenic can be detected cause the aforesaid deleterious effects upon a noble metal catalyst. Although it is believed that a major portion of the arsenic contained in the gases is present as arsine (ASH_3), the term "arsenic" as used herein is intended to include arsenic in any combined gaseous form.

DESCRIPTION OF THE PRIOR ART

It is well known that arsenic in gaseous form is a highly toxic substance. Workers in the gas mask art have suggested the use of activated charcoal impregnated with a metal or metal oxide such as copper or copper oxide as a material through which air (or other oxygen-containing gases) may be passed for the removal of arsenic. Exemplary of these proposals are U.S. Pat. Nos. 1,520,437; 2,511,288; and 2,513,508.

It has been found more recently that the presence of arsenic in gasolines which are treated by contact with a noble metal containing catalyst causes a permanent deactivation of the catalyst. Suggestions have been made in the art to pretreat petroleum fractions to remove arsenic by use of a wide range of materials such as a lignite-based activated carbon (U.S. Pat. No. 3,542,669); silica gel impregnated with sulfuric acid (U.S. Pat. No. 3,093,574); aluminum silicate (U.S. Pat. No. 2,939,833); or a salt of a metal not higher than copper in the electromotive series of metals (U.S. Pat. No. 2,781,297).

SUMMARY OF THE INVENTION

It has now been discovered that a specially defined activated carbon derived from a bituminous coal will directly remove arsenic from gaseous hydrocarbon streams. For purposes of this application, the activated carbon material will be termed a "sorbent," although that term is not intended to suggest that the arsenic removal is accomplished solely by physical adsorption. While not wishing to be bound by any particular theory, it is believed that some chemical reaction is involved between the arsenic and the sorbent, but the exact nature of the reactions are not known. At a minimum, it is believed that the removal of arsenic is accomplished, at least in part, by chemisorption; that is, the arsenic forms bonds with the surface atoms of the sorbent that are of comparable strength with ordinary chemical bonds and stronger than the bonds formed in physical adsorption.

Particularly surprising is the fact that other forms of activated carbon, such as those derived from lignite, are very inferior in their activity for the removal of arsenic from gaseous hydrocarbon streams. It should be noted that while many materials will function to remove arsenic from admixture with inert gases such as argon, and such materials remain active for reasonable loadings of arsenic, most of these materials fail quickly in the removal of arsenic from light hydrocarbon gases such as ethane-ethylene streams. In this context, the term "breakthrough" means the passage of arsenic beyond or downstream of the substance intended to remove it and is usually expressed as a percentage of the arsenic not removed in relation to the arsenic content of the charge stock.

The charge stock for treatment in accordance with the invention is a gaseous hydrocarbon feedstream wherein the hydrocarbons preferably have from one to four carbon atoms per molecule and which feedstream contains arsenic as an impurity in an amount from about 20 ppb to about 20 ppm or more. In this application the term "ppb" means parts per billion, and "ppm" means parts per million, and such parts are parts by volume unless otherwise indicated. Particularly preferred for treatment by the process of this invention are those light unsaturated hydrocarbon gases obtained by the catalytic cracking of heavier petroleum hydrocarbons such as gas oils or the thermal cracking of propane streams to produce ethylene-ethane streams. The light gases from an FCC unit have been found to contain small concentrations of arsenic, even though arsine, for example, is known to decompose at about 450°F. (232°C.) and the temperatures in an FCC unit are known to reach over 900°F. (482°C.). There is probably insufficient contact time in an FCC unit to decompose the arsine, or perhaps the arsine decomposes and reforms on cooling. For whatever reason, the present invention is applicable to those light hydrocarbon streams, especially olefinic streams, containing more than 20 ppb of arsenic, regardless of the source of the arsenic.

Preferably the charge stock of this invention is substantially dry, and by this is meant that the charge stock contains less than 10 ppm of water. While the use of a dry charge stock is not essential, the absence of water permits the operation to continue for a much longer time before breakthrough of the arsine. If water is present in the gaseous hydrocarbon stream, such as the gaseous hydrocarbon stream obtained as a product from an FCC unit, the water can be removed by any suitable procedure well known in the art such as by the use of molecular sieves or other drying agents. Similarly, the charge stock is preferably free of sulfur compounds such as H_2S since sulfur compounds may interfere with the removal of arsines from gaseous hydrocarbon charge stocks and reduce the loading of the sorbent be-

fore breakthrough. Again the manner of removing sulfur compounds from the charge stock in the preferred embodiment may be by any of the methods well known in the art, and such methods include, for example, the use of liquid solutions of amines or the use of caustic solutions, e.g., sodium hydroxide solution.

The process of the invention will now be further described by reference to the attached FIGURE. Referring to the FIGURE, the petroleum charge for catalytic cracking enters through line **2** into FCC unit **4** where it is converted under usual catalytic cracking conditions to a variety of lower boiling products, including gasoline type products. Gasoline is removed from FCC unit **4** through line **6.** The other gaseous products of the cracking process, which products are of primary concern here, are removed from FCC unit **4** through line **8** and enter an absorber section **10.** Absorber section **10** normally consists of several component units (not shown) such as an amine absorber, a knock-out drum to remove any entrained liquids from the gaseous products, and a heater to insure that the gases remain in the vapor phase. The FCC gases exiting from the heater unit of absorber section **10** have the typical composition shown in the following Table I:

TABLE I

Component	Vol. %
Nitrogen	9.5
Hydrogen	9.8
Methane	29.7
Ethylene	9.7
Ethane	12.6
Propylene	15.5
Propane	6.8
Butenes	0.5
Butanes	1.9
Pentenes	0.4
Pentanes	0.5
Hexanes	0.1
Carbon Monoxide	2.9

The FCC absorber gases are usually at a temperature from 80° to 150°F. (27° to 65°C.), more usually from 100° to 125°F. (38° to 51°C.) and at a pressure from 250 to 400 psig (17 to 27 atmospheres), more usually at a pressure from 290 to 360 psig (20 to 24.5 atmospheres). The increased pressures are those normally employed in the FCC unit and are used to propel the gases through the various units in the recovery train.

Light hydrocarbon gases such as ethane and propane are fed through line **14** into pyrolysis furnace **16** for the purpose of cracking the ethane and propane to produce ethylene. After recovery of liquid product (not shown) from pyrolysis furnace **16,** the gaseous products are passed through line **18** where they are combined with the products in line **12** from absorber section **10** and are passed through line **20** into system **22** which consists of a number of units, not individually shown, for the purpose of drying and recovering various hydrocarbon fractions. A C_3 fraction, for example, can be removed through line **24,** and a C_4 fraction through line **26.** The stream of most present interest and of greatest volume is the C_2 stream containing small amounts of acetylene, which stream is shown in the FIGURE as being removed from systems **22** through line **28.** A typical composition of this C_2 stream is shown on Table II below.

TABLE II

Component	Vol. %	Vol. % Range
Acetylene (C_2H_2)	0.50	0.1 - 1.0
Ethylene (C_2H_4)	65.0	55 - 75
Ethane (C_2H_6)	34.5	45 - 25

The ethane-ethylene stream in line **28** is of the greatest interest commercially, and it is this stream which is passed into arsenic removal unit **30.**

The function of arsenic removal unit **30** is to reduce the concentration of arsenic in the ethane-ethylene gases from a concentration in excess of 20 ppb to a concentration at the outlet of less than 20 ppb. The concentration of arsenic in the ethane-ethylene gases is usually on the order of 100 to 2,000 ppb but can be as high as 20 ppm or more. Preferably, the arsenic content of the gases leaving arsenic removal unit **30** is less than 10 ppb and is more preferably less than 2 ppb.

The type of sorbent material employed in arsenic removal unit **30** is an important feature of this invention and will be discussed in detail hereinbelow. Suffice it to say here that the sorbent material comprises a special metals content activated carbon derived from a bituminous coal.

The temperatures to be employed in the arsenic removal unit **30** can suitably be from 50° to 200°F. (10° to 93°C.), are usually from 80° to 150°F. (27° to 55°C.), and are preferably from 90° to 120°F. (32° to 49°C.). Temperatures below 50°F. (10°C.) are undesirable because of increased costs. Similarly temperatures above the stated range are undesirable due to the increased expense of operating the process.

The pressure to be employed in arsenic removal unit **30** is suitably atmospheric pressure or below to 1,000 psig (69 atmospheres) or more. As noted above, FCC units typically operate to produce product gases at pressures of about 250 to 350 psig (18 to 25 atmospheres). The process of the invention will operate satisfactorily at atmospheric pressure, but since it is expensive to depressure the FCC absorber gases and repressure the final products for transport through pipelines, it is desirable to operate the arsenic removal unit **30** at increased pressures of, say, 250 to 350 psig for transportation purposes.

Gaseous volume hourly space velocity (GVHSV) at standard conditions of temperature and pressure can suitably be from 1,000 to 20,000 v/v/hour, and is usually from 2,000 to 10,000 v/v/hour. The product is removed from the arsenic removal unit **30** through line **32,** and these gases, free of arsine but containing acetylene, are passed into an acetylene converter **34.** The acetylene content is produced in the pyrolysis furnace **16.** Hydrogen enters acetylene converter **34** by means of line **35.**

Acetylene converter **34** may contain a catalyst which is sensitive to poisoning by even minute quantities of arsenic, and thus it is one of the main objectives of the present invention to protect the catalyst in the acetylene converter **34** from permanent deactivation by arsenic. Catalysts which are particularly susceptible to arsenic poisoning are those containing the noble metals such as platinum and palladium.

Hydrogenation conditions are, of course, employed in acetylene converter **34,** and such conditions are well

known to workers skilled in the art. The C_2 stream, substantially free of acetylene, is then taken from acetylene converter **34** through line **36** to a distillation zone **38** where ethylene is removed through line **40** and heavier products may be suitably removed through line **42.** The heavier products may be recycled as feed to pyrolysis furnace **16,** if desired.

It should be noted here that the same benefits would accrue for any arsenic susceptible catalysts used in the hydrogenation of the propadiene in the C_3 stream in line **24** or the butadiene in the C_4 stream in line **26.** Thus the olefinic streams in line **24** or in line **26** could be passed into an arsenic removal unit similar to unit **30.**

The sorbent material for use in the process of this invention is a specially defined metal containing activated carbon derived from a bituminous coal.

The preparation of activated carbon from various coal sources has been known for extended periods of time. Exemplary methods are described in the Kirk-Othmer Encyclopedia, Second Edition, Vol. 4, pages 149 to 156, and this description is incorporated herein by reference. Bituminous coals are similarly well known and are described, for example, in "The Production of Active Carbon from Bituminous Coal," J. G. King, D. MacDougall and H. Gilmour: Technical Paper No. 47, Dept. Scientific and Industrial Research, Fuel Research, His Majesty's Stationery Office, London, England (1938); and this description is incorporated herein by reference.

Coals naturally contain various metals in differing concentrations. It has now been found that a certain activated carbon prepared from a bituminous coal has unusually superior ability for the removal of arsenic from light olefinic gas streams such as the combined C_2 stream from an FCC unit and an ethylene pyrolysis furnace.

Various activated carbons were tried for this purpose, and it was found from a comparison of the properties of the activated carbons that those activated carbons derived from bituminous coal and containing certain concentrations of cobalt, molybdenum, nickel and vanadium were surprisingly active for arsenic removal. The amounts of these metals per 100 cubic feet of activated carbon are suitably from 0.1 to 1.0 pound of cobalt, preferably 0.1 to 0.3 pound; 0.5 to 2.0 pounds of molybdenum, preferably 1.25 to 1.75 pounds; 0.1 to 1.0 pound of nickel, preferably from 0.2 to 0.6 pound; and 0.5 to 5 pounds of vanadium, preferably from 1.5 to 2.5 pounds.

Furthermore, it is preferred that the activated carbon have less than 1 pound of magnesium and 2 pounds of titanium per 100 cubic feet of the activated carbon.

The surface areas of the activated carbons are high, usually on the order of 500 to 1,500 m^2/g.

The invention will be further described with reference to the following experimental work.

A series of runs was made with several different activated carbons to determine the effectiveness of these activated carbons for the removal of arsine from a given charge stock. In all of the runs, the experimental procedure was as follows:

Gaseous charge stocks were prepared by mixing a sufficient amount of a blend of 2,000 ppm AsH_3 and nitrogen (supplied by Matheson Gas Company) with added pure nitrogen to obtain a charge stock having 50 ppm of AsH_3.

The reactor containing the activated carbon sorbent consisted of a ⅜ inch I.D. stainless steel cylinder with a ⅛ inch O.D. thermowell extending along its axis. The reactor was suitably heated. The temperature at the center of the activated carbon was measured by means of an iron-constantan thermocouple inserted into the thermowell. The test gas was introduced at the bottom of the reactor, passing through an approximately 6-inch-long bed of quartz chips, which served to preheat the gas stream.

The bed of activated carbon within the reactor was approximately 4 to 8 inches in length, and consisted of 5 to 10 cc's of material sized to 20/40 mesh.

The arsine not removed by the passage through the bed of supported sorbent was scrubbed from the effluent gas stream by a pyridine solution containing 0.50 g. silver diethyldithiocarbamate (Fisher Certified Reagent) per 100 ml. pyridine. This silver salt combines with the arsine to form a highly colored complex, permitting colorimetric monitoring of the total arsine breakthrough accumulation. Small samples were periodically drawn from the arsine scrubber, and the optical transmittance at 540 mm. wavelength was measured with a Bausch and Lomb Spectronic 70 spectrophotometer. This optical transmittance was then plotted as a function of time. The numerical derivative of this curve was calculated to determine the rate of arsine breakthrough. The percent breakthrough figures given in the tables below represent the percentage of the arsenic not removed in relation to the arsenic content of the charge stock.

The results of a first series of runs is shown in Table III below:

TABLE III

Removing Arsine with Activated Carbon
Using 50 ppm by Weight of AsH_3 in N_2

Conditions:	80°F. (27°C.); atmospheric pressure; and 3,000 GHSV			
Ex. No.	1	2	3	4
Type of Carbon	Bituminous[a]	Lignite[b]	Ash[c]	Bituminous[d]
Minutes to Breakthrough:				
Initial	310	142	32	5
10%	342	154	36	—
33%	410	171	43	—

[a]Purchased from Westvaco - known as Nuchar-WV-H (Tradename).
[b]Purchased from Atlas Chemical Co., known as Darco (Tradename).
[c]Experimental high-ash carbon.
[d]Purchased from Calgon Corp. - known as BPL (Tradename).

Referring to Table III it can be seen that the activated carbon used in Example 1 is surprisingly more active than any of the other activated carbons.

Tables IV and V below contain detailed analyses of the various activated carbons used in the experiments shown in Table III above.

TABLE IV

Activated Carbon Ash Content/Analyses

Carbon from Example from Table III	1	2	3	4
Density: lbs/ft³	30	24	32	24.5
Wt % Ash	6.85	14	16.9	6.74
Lbs.Ash/100 ft³ of carbon	206	336	540	165

TABLE IV-Continued

Activated Carbon Ash Content/Analyses

Carbon from Example from Table III	1	2	3	4
Lb.of metal/100 ft³ of carbon				
Aluminum	41.1	33.6	81.1	33.0
Boron	0.083	0.1	0.22	0.066
Calcium	2.06	6.7	3.24	3.3
Chromium	0.083	Trace	0.16	0.066
Cobalt	0.20	—	—	—
Copper	0.185	0.07	0.11	0.066
Iron	12.35	13.5	21.6	9.9
Lead	—	3.36	1.08	—
Magnesium	0.618	3.36	5.4	3.3
Manganese	0.20	0.67	0.22	0.132
Molybdenum	1.45	—	—	—
Nickel	0.41	—	—	0.0495
Potassium	0.20	0.4	0.49	0.0165
Silicon	41.1	67.2	108.0	33.0
Sodium	1.45	13.5	2.7	0.825
Strontium	0.41	1.0	1.08	0.33
Titanium	1.85	6.67	5.4	3.3
Vanadium	2.05	—	—	0.165

ume and pore area occupied by pores having a 7–10 A. pore radius; and from 10 to 25 percent of their pore volume and pore area occupied by pores having a 10–15 A. pore radius.

Example 5

In the run for this example, a slip stream of a C_2 concentrate from a commercial unit was passed through a bed of an activated carbon as used in Example 1 above at ambient temperature, 300 psig (20 atmospheres), and a GHSV of about 8,000. The charge had a composition of about 34.5 percent ethane; 65 percent ethylene; 0.5 percent acetylene; and about 260 ppb AsH_3. Breakthrough of AsH_3 in the product gas occurred after about 120 hours.

Example 6

Example 5 was repeated except the carbon of Example 2 was used and breakthrough occurred after only about 24 hours.

TABLE V

Physical Inspections of Various Activated Carbons

Carbon from Example from Table III	1		2		3		4	
RP: Angstroms[a]	8.5		34.1		10.4		7.1	
Total Pore Vol. cc/g[a]	0.57		0.61		0.80		0.46[c]	
Avg Pore Radius: Angstroms[b]	12.1		22.3		13.5		12.1	
BET Area: m²/g[a]	945.1		550.4		1184.0		764.6[c]	
Pore Radius: Å	Vol. %	Area %	Vol. %	Area %	Vol. %	Area %	Vol. %	Area %
300–250	0.5	0.0	2.5	0.2	0.8	0.0	0.5	0.0
250–200	0.8	0.0	4.7	0.4	1.2	0.1	0.9	0.0
200–150	1.4	0.1	7.7	0.8	1.7	0.1	1.1	0.0
150–100	1.8	0.1	11.2	1.7	3.1	0.3	1.3	0.1
100–90	0.3	0.0	2.4	0.5	0.7	0.1	0.4	0.0
90–80	0.4	0.0	3.4	0.7	1.1	0.1	0.6	0.1
80–70	0.6	0.1	3.0	0.7	1.4	0.2	0.8	0.1
70–60	0.8	0.1	3.7	1.0	1.5	0.3	0.6	0.1
60–50	1.0	0.2	3.6	1.2	1.9	0.4	0.7	0.1
50–45	0.7	0.1	2.2	0.8	1.4	0.3	0.5	0.1
45–40	0.7	0.1	2.5	1.1	1.4	0.4	0.7	0.1
40–35	0.9	0.2	2.8	1.4	1.8	0.5	0.8	0.2
35–30	1.1	0.3	3.0	1.7	2.1	0.7	0.9	0.2
30–25	1.7	0.5	3.8	2.5	3.0	1.2	1.5	0.4
25–20	2.5	1.0	4.9	4.1	4.0	2.0	2.0	0.7
20–15	5.2	2.7	7.0	7.5	6.8	4.3	4.6	2.1
15–10	17.5	13.1	11.1	16.9	18.4	17.1	10.4	6.7
10–7	62.2	81.2	20.6	56.8	47.5	72.0	71.7	89.0

[a]Obtained using "absorptomat" instrument manufactured by American Instrument Co., Inc., Silver Spring, Md.
[b]See Absorption of Gases & Vapors, Vol. 1, Princeton University (1943); S. Brunauer, Chap. 11, pp. 365–418.
[c]Appears low compared to manufacturer's specification.

Referring to Tables III, IV and V, it can be seen that carbons derived from bituminous coals per se are not equivalent (compare Examples 1 and 4) and that the superior results shown in Example 1 cannot be attributed to the ash content of the activated carbon. It is observed, however, that only the activated carbon of Example 1 contains cobalt, molybdenum, nickel and vanadium in the concentration ranges defined above. The lignite carbon (Example 2) and the experimental high ash carbon (Example 3) contain none of these metals, while the bituminous carbon of Example 4 has only small amounts of nickel and vanadium and no cobalt or molybdenum. Further, from the data in Table V, it appears the carbons useful in the process of this invention preferably have from 55 to 85 percent of their pore vol-

Resort may be had to such variations and modifications as fall within the spirit of the invention and the scope of the appended claims.

I claim:

1. A process for reducing the arsenic content of a gaseous hydrocarbon charge stock which comprises contacting the charge stock with an activated carbon derived from a bituminous coal and wherein said activated carbon per 100 cubic feet has from 0.1 to 1.0 pound of cobalt; 0.5 to 2.0 pounds of molybdenum; 0.1 to 1.0 pound of nickel; 0.5 to 5 pounds of vanadium.

2. A process in accordance with claim **1** wherein the charge stock comprises a gaseous olefin having from 2 to 4 carbon atoms per molecule and greater than 20 ppb of arsenic.

3. A process in accordance with claim **2** wherein the charge stock is the combined C_2 stream from an FCC cracker and an ethylene pyrolysis furnace.

4. A process in accordance with claim **3** wherein the charge stock contains less than 10 ppm water.

5. A process in accordance with claim **4** wherein the activated carbon in addition has less than 1 pound of magnesium and 2 pounds of titanium per 100 cubic feet of activated carbon.

6. A process in accordance with claim **3** wherein the charge stock comprises from 55 to 75 volume percent ethane; 25 to 45 volume percent ethylene; 0.1 to 1.0 volume percent acetylene.

7. A process according to claim **6** wherein the activated carbon has from 0.1 to 0.3 pound of cobalt; 1.25 to 1.75 pounds of molybdenum; 0.2 to 0.6 pound of nickel; and 1.5 to 2.5 pounds of vanadium.

8. A process in accordance with claim **7** wherein said contacting occurs at a temperature from 50° to 120°F.

9. A process in accordance with claim **8** wherein the charge stock is substantially free of sulfur compounds.

10. A process according to claim **8** wherein the activated carbon has from **55** to **85** percent of its pore volume and pore area occupied by pores having a 7 to 10 A. pore radius and from 10 to 25 percent of its pore volume and pore area occupied by pores having a 10 to 15 A. pore radius.

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