

March 9, 1971

A. DRAVNIKS ET AL

3,568,411

CHEMOSENSOR BOMB DETECTION DEVICE

Filed March 18, 1969

3 Sheets-Sheet 1

FIG. 1

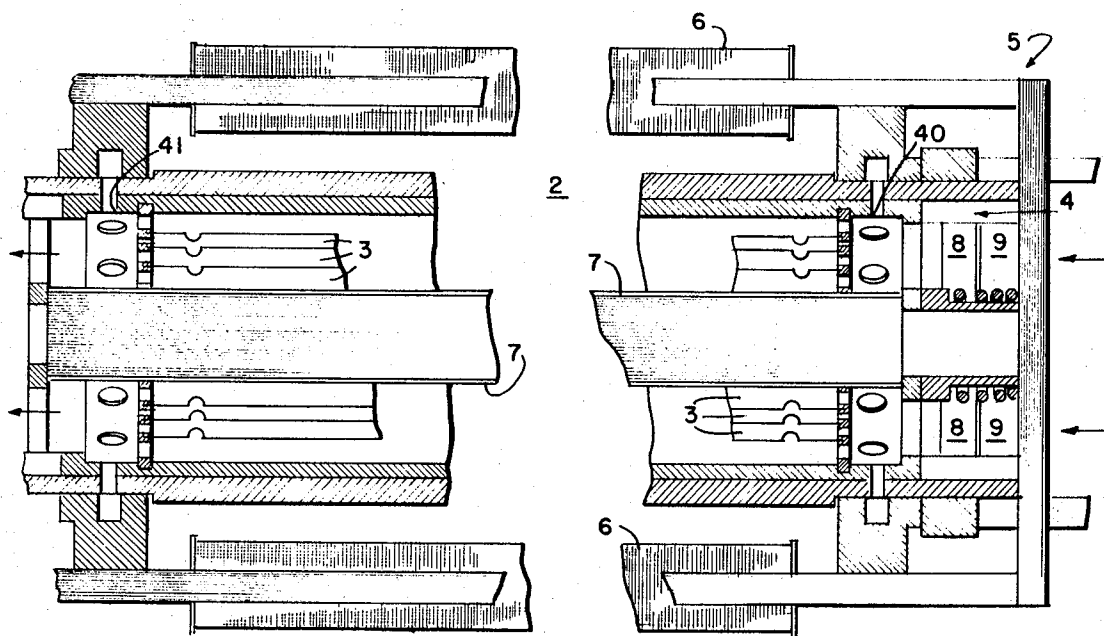
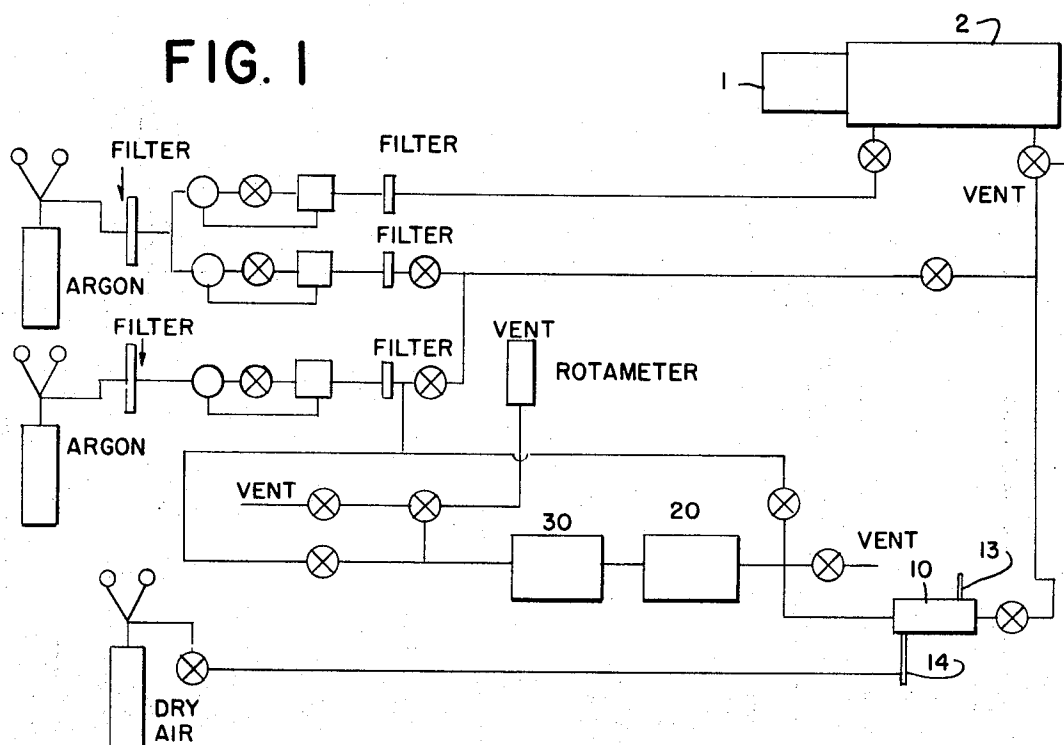


FIG. 2

INVENTORS

ANDREW DRAVNIKS
JAY FISCHMAN

BY

Alan H. Randy

ATTORNEY

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A. DRAVNIKS ET AL

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FIG. 4

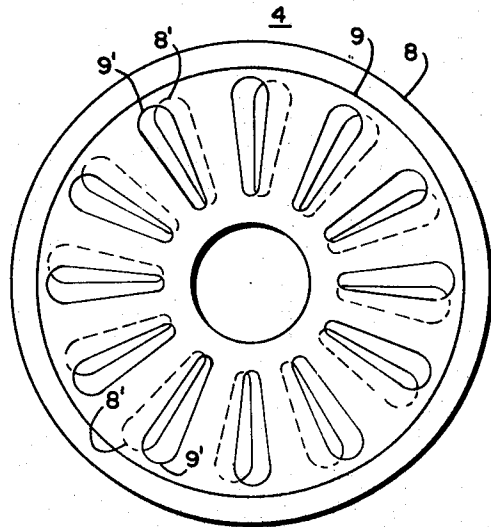
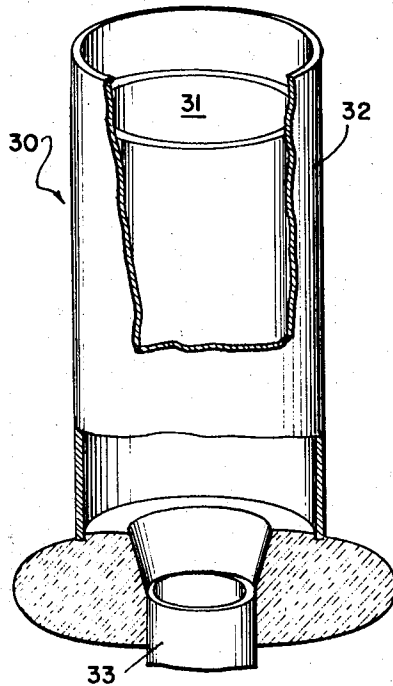


FIG. 3

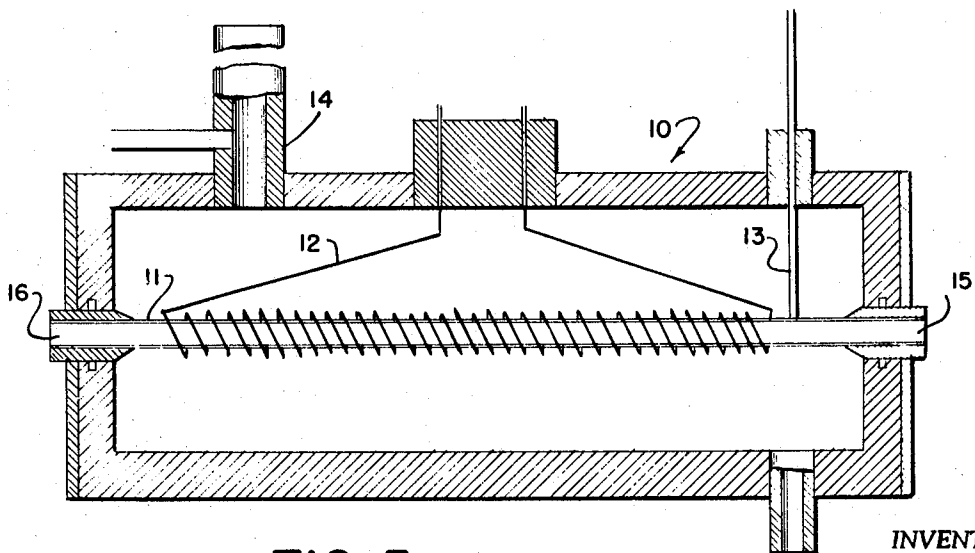


FIG. 5

INVENTORS

ANDREW DRAVNIKS

JAY FISCHMAN

BY

Alva H. Bandy

ATTORNEY

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A. DRAVNIKS ET AL

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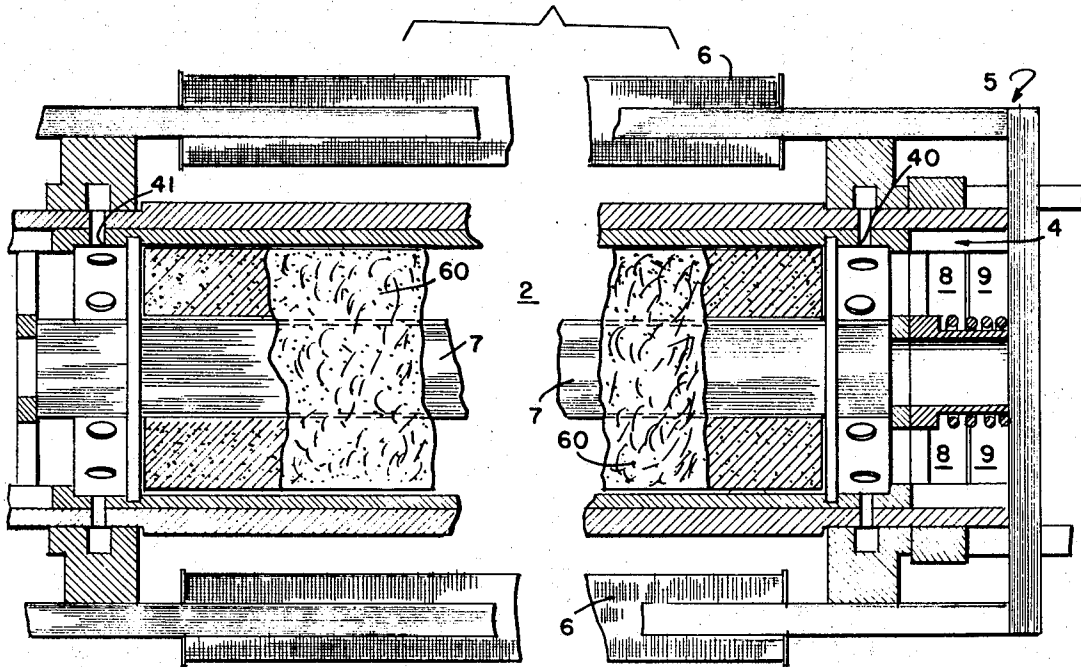


FIG. 6

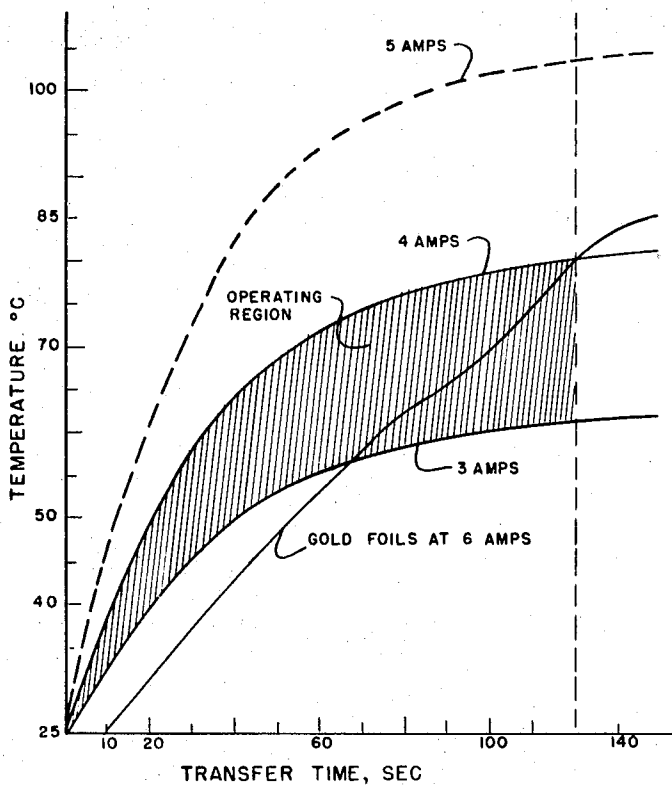


FIG. 7

INVENTORS
ANDREW DRAVNIKS
JAY FISCHMAN

BY

Alva H. Bandy
ATTORNEY

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3,568,411

CHEMOSENSOR BOMB DETECTION DEVICE
Andrew Dravnieks, Park Forest, Ill., and Jay Fischman,
Houston, Tex., assignors to the United States of America
as represented by the Secretary of the Army and/or
the Administrator of the Federal Aviation Administration

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Int. Cl. B01d 53/04

U.S. Cl. 55—208

2 Claims

ABSTRACT OF THE DISCLOSURE

This invention relates to an improved bomb detector for identifying the presence of dynamite, for example, in an airplane. The basic method detects the presence of ethylene glycol dinitrate vapor in air. The essential steps are selective adsorption of the vapor on a surface, desorption of the vapor and a timed passage through a short, chromatographic partition column, followed by passage through a vapor detector which emits an identifying signal. The improvement comprises the use of gold-plated, copper foametal as a primary adsorber surface for ethylene glycol dinitrate vapor.

CROSS REFERENCE TO A RELATED APPLICATION

This is an improvement in the bomb detector of patent application Ser. No. 655,267, filed July 20, 1967, now Pat. No. 3,430,482, issued Mar. 4, 1969.

BACKGROUND OF THE INVENTION

(1) Field of the invention

The invention describes a particular method and apparatus for qualitative gas analysis utilizing the properties of a particular vapor to identify minute quantities thereof in air. The method may be modified depending on the particular vapor to be identified. The vapor is characteristically evolved from a particular organic explosive material. For instance, ethylene glycol dinitrate (hereinafter referred to as EGDN) vapor evolves from dynamite and self-made nitroglycerine. Nitrobenzene, which can originate from various solvents, is separable from EGDN vapor and is detectable from plastic explosives by a similar process. No method is herein disclosed to detect the presence of trinitrotoluene and dinitrotoluene containing explosives or black powder.

(2) Description of the prior art

The prior art contains no rapid means for detecting the presence of a bomb in an airplane short of a physical search.

SUMMARY

It has been discovered that trace quantities of EGDN vapor will be readily adsorbed on a gold surface, and desorbed with an increase in temperature. This property is utilized together with a partition column to identify the vapor. The process and apparatus of this invention separate the EGDN vapor from air and pass it through a partition column for subsequent identification based upon the time taken for the vapor to travel through the column and its subsequent ability to change the electric current flow in a tritium containing electron-capture detector.

Accordingly, it is an object of this invention to detect the presence of explosive materials in a closed area such as an airplane rapidly and with a minimum of passenger discomfort.

It is another object to provide a compact vapor analysis apparatus capable of installation on an airplane which will reliably indicate the presence of explosives in a few minutes of operation.

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It is a further object to provide a vapor analysis apparatus which will analyze the air exhausted from an airplane to detect minute quantities of EGDN vapor.

It is an additional object to provide an efficient, compact, adsorber having a maximum surface area to selectively adsorb EGDN vapor with a minimum amount of power expense.

These and other objects will become readily apparent with reference to the drawings and following description.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a block diagram of the analysis apparatus of this invention.

FIG. 2 shows the primary adsorber in longitudinal cross-section.

FIG. 3 shows an end view of the rotary disk valve used at either end of the primary adsorber of FIG. 2.

FIG. 4 shows the electron-capture detector in partial section.

FIG. 5 shows the secondary adsorber.

FIG. 6 shows the improved primary adsorber in partial cross-section.

FIG. 7 is a graph of the temperature of the adsorber plotted in relation to elapsed time with amperes of current as a parameter.

DESCRIPTION OF THE PREFERRED EMBODIMENT

To detect the presence of EGDN vapor the analyzer of this invention may be miniaturized for installation on board an airplane or it may be installed on the same truck used to heat and air-condition aircraft on the ramp. Cabin air at ambient temperature is circulated by a blower 1, through the primary adsorber 2.

The adsorber 2 consists of three concentric metal cylinders 3. The metal internal to the adsorber is the only exposed metal internal to the system. Stainless steel, copper, polytetrafluoroethylene (hereinafter referred to as Teflon), platinum, and gold were used in experimentation. Since EGDN is an electron-accepting, polar molecule it may be held on a metal surface by a partial transfer of electrons to the adsorbed molecule. This process would then be partially inhibited in metals having the usual oxide layer from reaction with air and adsorbed moisture films. It was therefore found that gold, a relatively chemically inactive metal, displayed a great affinity for EGDN molecules, and the cylinders of one embodiment are constructed of gold foil. The adsorber of this improved embodiment is a cylinder of gold-plated copper foametal used in place of the three concentric, gold foil cylinders.

As the cabin air passes longitudinally through the primary adsorber, FIG. 2, the EGDN vapors present are adsorbed on the gold surfaces. When the air has passed through adsorber 2, rotary disk valves 4, at either end are closed.

The cylinders 3 are in reality one turn shorts in a transformer 5, having primary coils 6 and a laminated, insulated core 7.

In the improved embodiment, foametal cylinder 60 is substituted for cylinders 3 and forms a one turn short in transformer 5.

The rotary disk valve 4 comprises a stationary disk 8 and a rotating disk 9, having ports 8' and 9' respectively. These disks may be Teflon coated aluminum.

When the valves are closed current is applied to the system and cylinder 60 is heated to 80° C. At this temperature, EGDN molecules are desorbed. An inert carrier gas such as argon is admitted during the heating at manifold 40, to carry the vapor from exit manifold 41, to the secondary adsorber 10.

The secondary adsorber 10 consists of a gold tube 11, wrapped with an asbestos covered Nichrome wire heating

element 12. A thermo couple 13 is used to regulate the internal temperature, and a cooling means 14, such as a Vortex tube, is used to cool the system.

When the vapor containing argon is admitted through the inlet valve of the gold tube 15, the cooling means 14 has precooled the tube to 0° C. At this temperature EGDN molecules are adsorbed on the gold surface. The tube 11 is then heated by wire 12, to 80° C. at which point the EGDN molecules are desorbed.

The outlet valve 16 is then opened and argon gas is injected to carry the vapors to the partition column 20.

Column 20 is a chromatographic column used to differentially separate gases based upon the time taken to travel the length of the column. The column is a tube having a packing and an inner surface coated with an absorbent material. The gases are dissolved and evaporate until equilibrium is reached. Because each chemical is absorbed and evaporated at a different rate, each has a different rate of travel down the length of the tube so that each emerges at a different time.

The column 20 is of conventional design. The length is important because too great a length will allow the vapors to decompose. Accordingly, a Teflon tube 1/8 inch outside diameter and approximately 12-14 inches long was selected. The column was packed with 0.690 gram of 10% Apiezon-L on Fluoropak 80 which is commercially available shredded Teflon coated with Apiezon-L, a hydrocarbon grease with a molecular weight of about 1400. The internal wall of the column was also coated with the same material.

Various other absorbent materials were experimentally substituted as coatings for the packing, Apiezon-N, a grease with a molecular weight of about 1200



Carotene, a vitamin having the formula $C_{40}H_{56}$; Biliverdin, a bile pigment having a formula of $C_{32}H_{36}O_8N_4$; Carbowax 4000, a polyethylene glycol with a structure of $HOCH_2(CH_2OCH_2)_xCH_2OH$ and a molecular weight of about 4000; Squalene, $C_{30}H_{50}$; Silicon Ester-52, a proprietary silicon rubber; Dinonyl phthalate, $C_{26}H_{42}O_4$; and Kel-F Grease, $Cl \cdot (CF_2 \cdot CFCl)_x \cdot Cl$.

As the vapors exit the column they enter the electron-capture detector 30 of FIG. 4. It is essentially an ionization device in which vapors that have an affinity for electrons decreased the electrical conductivity of gases. Because gases are poor conductors a means 31, to introduce ions, is placed between the cathode 32 and the anode 33. The means selected was a foil of titanium metal that has been reacted with the radio active hydrogen isotope, tritium. The tritium is a source of high energy electrons (beta particles) which interact with the incoming gas atoms to lose energy and produce lower-energy, slow electrons. Under the electrical potential provided by a battery, these electrons are collected at the positive electrode 33, the inlet tube. This causes a current to flow.

When any material that is capable of forming negative ions (anions) enters the detector some of the electrons flowing toward the anode react to form such anions. Since the mobility of an anion is some 10^5 to 10^6 times less than that of free electrons, the anions are carried away by the flowing carrier gas and react with positively charged gas molecules instead of being discharged at the anode.

The process in the detector may be summarized as follows. A tritium atom disintegrates to form a helium-3 positive ion and a beta particle with an energy of about 18000 ev. By collisional interaction with the argon carrier gas, $n+1$ slow electrons are produced. The value of n is about 500 to 1000, since about 15 ev. is required to ionize argon. The slow electrons either recombine with the positive argon ions or are collected by the anode. When a gas that forms stable negative ions as EGDN is admitted to the detector with the argon carrier the slow electron moving toward the anode is "captured" to form the stable negative ions which flow out with the carrier

gas. This "capture" effect causes a decrease in current flow which may be amplified to produce a measurable signal.

The change in the current flow through the detector may be recorded as peaks on a strip chart, or it may be coordinated with a time signal to ring an alarm bell when a peak appears at a preselected time interval. These recording devices are conventional and modifications would be obvious to one of ordinary skill in the art.

Because of the nature of the processes in an electron capture detector, such parameters as the applied voltage, type of carrier gas, temperature, pressure, gas flow rate, and type of recording device affect the instrument performance, but the calibration of such an instrument is within the skill of an ordinary practitioner in the art.

After the sample of air passes through each of the chambers described above, it is necessary to clean the vacated chamber. This can be readily accomplished by heating to a temperature of from 150-200° C., to assure that all residual vapors are released. The chamber is then purged with the inert carrier gas. This decontamination is necessary to assure that explosive material vapors detected in one aircraft do not give a false alarm in subsequent tests.

Because EGDN is a strong electron acceptor readily absorbed on a metal surface the internal parts of the system, vapor sampling device, transport lines, valves (denoted by an $\otimes$ in FIG. 1), connections, and chromatographic column are constructed of a material which adsorbs as little as possible. Accordingly, Teflon was used, and the gold tube and foametal cylinder are the only exposed metal surfaces internal to the system.

The embodiment employing gold cylinders had the following dimensional characteristics. The gold cylinders 3, in the primary adsorber 2, have an exposed area of 364 square inches and were 12 inches in length. The air flow rate through the primary adsorber was up to 200 c.f.m. Since desorption must be uniform and complete, the thickness of the cylinders 3 should be adjusted so that each has approximately the same electrical resistance. It was determined that the middle cylinder should be 6.6 mils thick, the outside cylinder 8.4, and the inner tube 5.0 mils thick. The argon is fed into the adsorber 2, at distribution collar 40, at a flow rate of 8 liters per minute, the secondary adsorber used a 3 inch long by 1/16 inch inside diameter gold tube and had an effective adsorbing area of approximately 3.7 cm.².

With the above approximate dimensions, a flow rate to the column of 50 cc./min., and a column temperature of 90° C. the retention time in the column is 1 minute and 30 seconds.

It was also found that Teflon at room temperature adsorbed a measurable amount of EGDN. Accordingly, the overall system temperature was raised to 74-80° C.

The argon used for the detector step was certified to be less than .5 p.p.m. hydrocarbon, and that used in the adsorbers 2 and 10 was 99.996% pure.

On actual tests with the gold cylinders as described above, a test could be completely run and the system purged for another test in as little as 6 minutes 50 seconds. This model was designed so that a peak in current change would read out between 1 and 2 minutes after sample injection into the column.

In the improved embodiment using gold-plated, copper foametal cylinder 60, in place of cylinders 3, the foametal cylinder had an inside diameter of one and one-half inches and an outside diameter of five inches, and was four and one-half inches shorter than the gold foil cylinder. It was discovered that these dimensions provided an adsorptive capacity in the improved unit equal to the gold cylinders. The economics of space, weight, and cost, in the improvement, however, were substantial. An ideal high surface area to volume ratio was achieved in substituting foametal for the foil cylinders. The power input to the transformer was also substantially less.

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FIG. 7 shows a comparison of surface temperature and time for both the foametal and the cylinders. As shown a current of three to four amperes gives the required 80° temperature in two minutes of transfer. By contrast the gold foil cylinders required six amperes to reach the required temperature in two minutes with a poorer temperature rise.

The foametal cylinder may also be heated by applying a voltage directly to the fiber structure, as in a resistance heater, in place of the transformer. A small wedge would be sliced from the foametal to expose two complete cross sections, and a conductor such as a copper sheet affixed at each cross section face. When a direct current is applied the mass of copper foametal would be heated proportional to the voltage applied.

It will be obvious that the system design may be varied greatly within the basic inventive concept. The invention is not intended to be limited by the apparatus described as a preferred embodiment.

We claim:

1. An adsorber for selectively adsorbing electron-accepting, polar, gaseous molecules from air, comprising:
 - (a) an inlet for receiving the air and gaseous molecules;
 - (b) an outlet; and

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(c) a foametal adsorption surface disposed between the inlet and the outlet, for selectively adsorbing the gaseous molecules, wherein the foametal adsorption surface is gold-plated copper foametal.

2. An adsorber for selectively adsorbing electron-accepting, polar, gaseous molecules from air, comprising:

(a) an inlet for receiving the air and gaseous molecules;

(b) an outlet;

(c) a foametal adsorption surface disposed between the inlet and the outlet, for selectively adsorbing the gaseous molecules; and

(d) an internal heating element to heat the foametal surface; wherein the foametal adsorption surface is gold-plated copper foametal.

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JOHN ADEE, Primary Examiner

U.S. Cl. X.R.

55—387; 73—23.1