United States Patent [19]

Kaiser

[11] B **3,925,023**

[45] **Dec. 9, 1975**

[54] **FLAME IONIZATION DETECTOR**

[76] Inventor: **Rudolf Kaiser**, Dr.-Dahlem-Str. 9, 6702 Bad Durkheim, Germany

[22] Filed: **Nov. 21, 1973**

[21] Appl. No.: **418,121**

[44] Published under the Trial Voluntary Protest Program on January 28, 1975 as document no. B 418,121.

[30] **Foreign Application Priority Data**

Nov. 21, 1972 Germany 2257099

[52] **U.S. Cl.** ..... **23/254 EF**

[51] **Int. Cl.**[2] **G01N 31/12**

[58] **Field of Search** ..... 23/254 EF, 254 E, 232 C

[56] **References Cited**

UNITED STATES PATENTS

3,372,994	3/1968	Giuffrida	23/254 EF
3,425,806	2/1969	Karmen	23/254 EF X
3,504,976	4/1970	Gilbert, Jr.	23/254 EF
3,692,492	9/1972	Poli, Jr. et al.	23/254 E

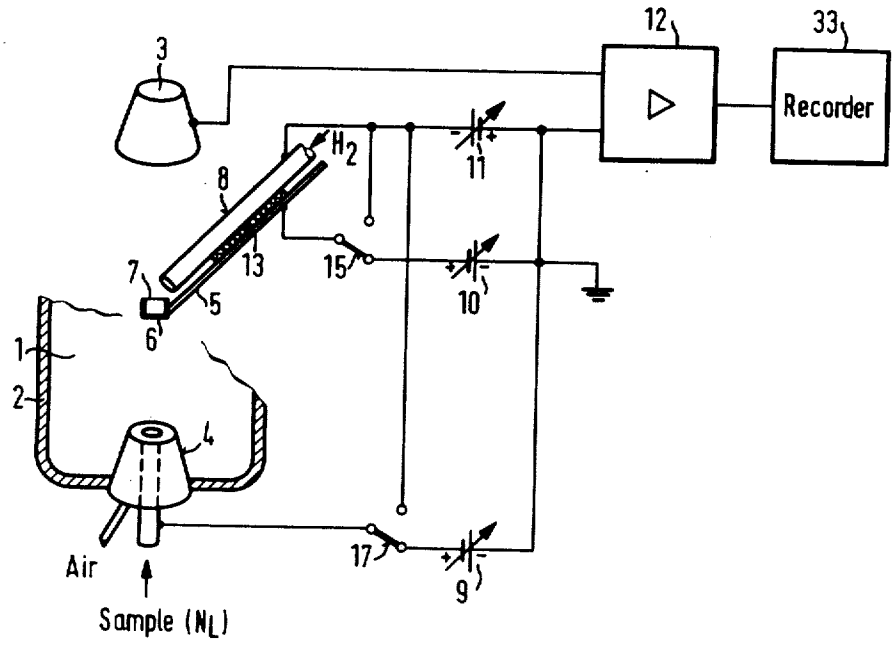
Primary Examiner—Robert M. Reese

Attorney, Agent, or Firm—Kenyon & Kenyon Reilly Carr & Chapin

[57] **ABSTRACT**

A flame ionization detector which is capable of being selectively switched to a mode of operation for the detection of hydrocarbon compounds and another mode of operation for the detection of nitrogen compounds, in which two burner nozzles are arranged in the same chamber below a collector electrode, with the flame of the first burner nozzle directed upward and that of the second burner nozzle downward onto a salt source. In both modes, the substance to be detected is fed to the first burner nozzle, with hydrogen as the fuel gas being fed to the first nozzle when detecting hydrocarbon compounds and to the second nozzle when detecting nitrogen compounds. When detecting hydrocarbon compounds, both nozzles and the salt source are kept at a negative potential and when detecting nitrogen compounds, the second nozzle is at a negative potential and the first nozzle and salt source at a positive potential or ground.

10 Claims, 2 Drawing Figures



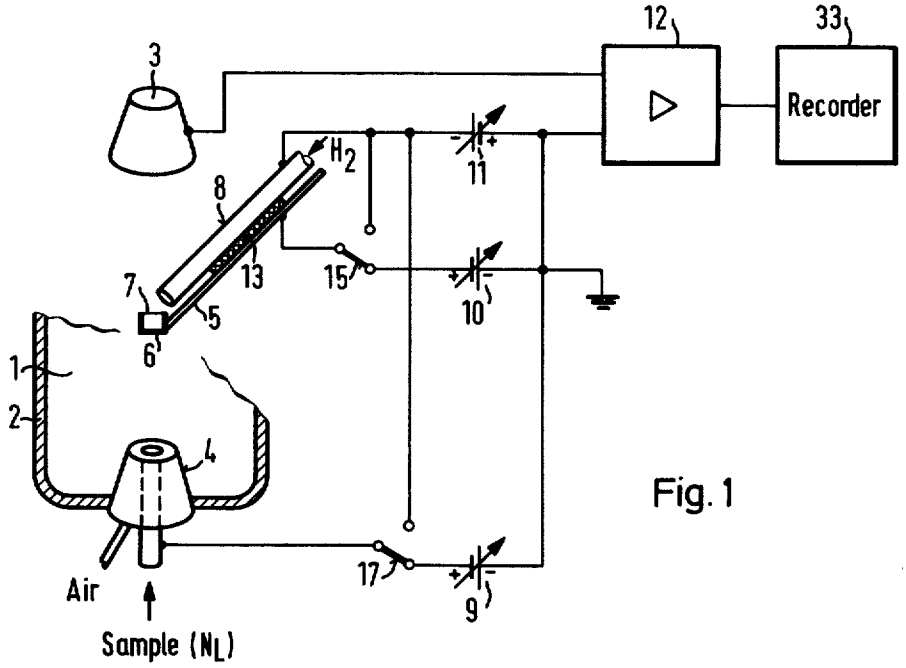

12
33
3
Recorder
H_2
8
11
13
15
7
5
10
6
1
2
4
17
9
Air
Sample (N_L)

Fig. 1

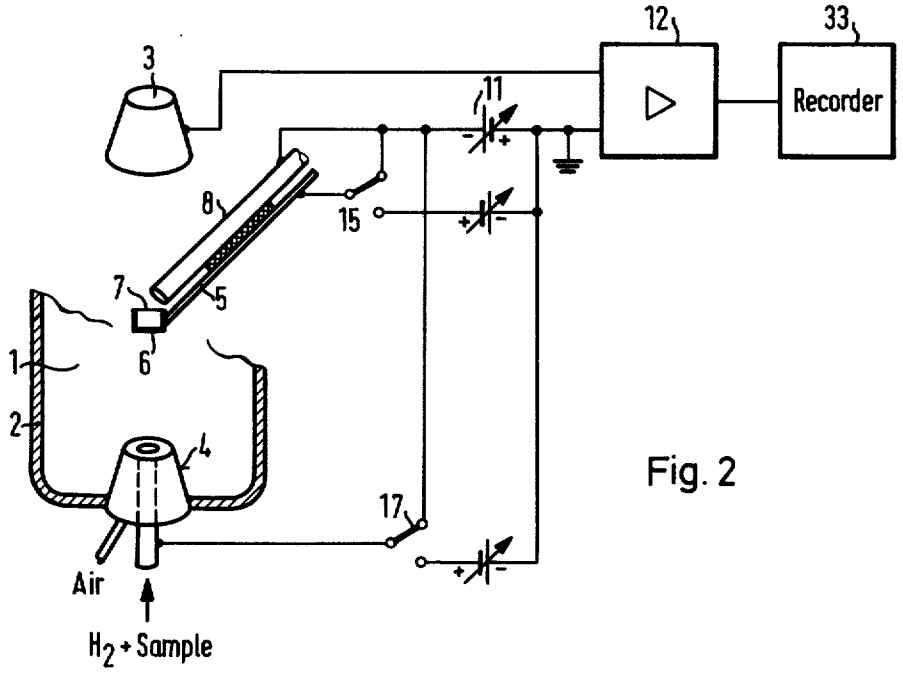

12
33
3
11
Recorder
8
15
7
5
6
1
2
4
17
Air
H_2 + Sample

Fig. 2

FLAME IONIZATION DETECTOR

BACKGROUND OF THE INVENTION

Flame ionization detectors [FIDs] are used for detecting hydrocarbons and, in conjunction with alkali salts, for selectively detecting phosphorus, nitrogen and other organic compounds containing hetero atoms. These organic compounds, which contain hetero atoms, will be referred to herein as hetero compounds for short. In such devices, the alkali salts are arranged so that they are heated by a flame fed with hydrogen from a burner nozzle in order to give off ions, which form compounds with the ions of the substances to be detected. This general mode of operation is described in U.S. Pat. No. 3,372,994. The ion current to the collector is selectively increased to a large degree if phosphorus, nitrogen, arsenic or other hetero compounds are present as long as a suitable salt source is chosen.

It is known that the sensitivity of such arrangements can be increased through the use of several annular electrodes at different potentials, interposed between the flame and the collector. In addition, systems have been proposed in which a common detection chamber contains two systems having two flames and which can be operated simultaneously or in an alternating manner. Other than these dual system arrangements, no existing FIDs have the capability of being operated for the detection of both hydrocarbons and nitrogen compounds or other hetero compounds.

SUMMARY OF THE INVENTION

It is the primary object of this invention to provide an FID of particularly simple design, having two burner nozzles in a common detection chamber, which can be used selectively for the detection of hydrocarbons and nitrogen compounds or other hetero compounds. This is accomplished by having a first burner nozzle which is directed along the central axes of the detection chamber, vertically upward, in the manner of typical prior art FID systems. Along therewith, there is provided a second burner nozzle having an opening which is directed near the flame of the first burner nozzle in a downward direction onto the salt source. In the operation of the present device, the substance to be detected is fed, whether detecting hydrocarbons or nitrogen or other hetero compounds, to the first burner nozzle and the fuel gas fed to the first burner nozzle when detecting hydrocarbons and to the second burner nozzle when detecting nitrogen or other hetero compounds. When detecting hydrocarbons, the first and second burner nozzles are connected to a negative voltage and when detecting nitrogen or other hetero compounds, the second burner nozzle is connected to a negative voltage and the first burner nozzle connected to a ground or positive voltage. The detector of the present invention can be rapidly switched without requiring mechanical changes in the system to obtain maximum selectivity for hydrocarbons or maximum selectivity for nitrogen compounds. In each mode of operation, the system retains high sensitivity to the compound which is being detected.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic illustration of the system of the present invention configured for the detection of nitrogen or other hetero compounds.

FIG. 2 is a similar schematic diagram illustrating the configuration of the present system for the detection of hydrocarbon compounds.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

FIG. 1 is a schematic illustration of the system of the present invention, configured for the detection of nitrogen or other hetero compounds. In typical fashion, the components of the system will be contained within a housing **2**, made of upper and lower parts. The detection chamber itself is designated by the reference numeral **1**. Within the chamber **1**, is provided a first burner nozzle **4**. This burner nozzle has a feed line inserted into the central axes of its housing below, preferably through an insulating feed through made of a material such as Teflon (tetra-fluor ethylene). A funnel-shaped collector electrode is mounted in the top of the chamber above the burner nozzle **4** and similarly may have its connections brought through insulating feedthroughs in typical fashion. Arranged above the burner **4**, i.e., between the burner **4** and the collector **3**, is a holder **5** into which there is placed a salt source **7**. The salt source comprises a dish **6** of platinum, into which is fused rubidium bromide or other suitable salt. Structurally attached to the holder **5** is a second burner nozzle **8** and its associated feed line. Coupling between the holder **5** and the burner **8** is through an adjustable coupling **13** which permits relative motion therebetween to adjust the nozzle of the second burner **8** from the salt source **7**. When detecting a nitrogen or other hetero compound, the burner **4** is fed with the sample along its central axes as indicated. Burner **4**, additionally has a second inlet through which air for combustion is fed as indicated. The burner **8** in this arrangement is fed with the fuel gas, H_2, as indicated. The burner nozzle **8** is electrically connected to a negative voltage source **11**. The holder **5** for the salt source is connected to an additional positive voltage source **10**. The burner nozzle **4** is also connected to a positive voltage source **9**. The other side of each of the voltage sources is connected to ground and to the one input of an amplifier **12**. The other input to amplifier **12** is taken from the collector electrode **3** in conventional fashion. In a well known manner, the amplifier output is then provided to a recorder **33** or the like. The nozzles **4** and **8** along with their feed lines will preferably be made of steel in conventional fashion.

In operation, the hydrogen fed to the burner nozzle **8** as the fuel device will be ignited in well known fashion by an ignition device (not shown) which is provided within the chamber **1**. The tip of the flame produced therefrom will touch the salt source **7**. Air from combustion is provided in well known fashion by the air inlet at the burner **4**. The sample to be examined for traces of nitrogen is fed-in through the nozzle **4**. The preferred voltages at each of the points will be as follows: a negative potential of approximately 200 to 300 V, being provided by the voltage source **11** to the burner nozzle **8**; a voltage which is at ground or slightly positive being provided to the holder **5**; and similarly, a ground or positive voltage being provided by the voltage source **9** to the burner nozzle **4**. Each of the voltage supplies **9**, **10** and **11** will preferably be adjustable for voltages between 0 and 300 volts. Preferably, the combination of the holder **5** and nozzle **8** will be axially moveable so that the spacing of these parts from the nozzle **4** can be adjusted.

With a positive voltage connected to the nozzle **4**, the background ionization current which interferes in trace analysis is greatly reduced. Keeping the salt source **7** grounded or at a small positive voltage of about 10 V, results in a maximum selectivity with a very small background current and a small flame. This setting gives a medium nitrogen sensitivity. If the flow of hydrogen and therewith, the size of the flame is increased, the nitrogen sensitivity can also be increased. In that case, a voltage of about 90 V should be connected to the salt source.

Sensitivity and selectivity for detecting nitrogen is also influenced by the position of the salt source relative to flame from the burner nozzle **8** and the position of these parts relative to the nozzle **4** and the collector electrode **3**. The hydrogen supply and the ratio of combustion air which is fed into the hydrogen stream must also be adjusted to obtain maximum sensitivity. Furthermore, the adjustment of the positive voltage at the nozzle **4** will have an effect. In the arrangement of FIG. **1**, maximum sensitivities of 3 to 5 Coul/g are attainable and maximum selectivities of 1:90,000 obtainable.

In the example described above, in connection with FIG. **1**, the detection of nitrogen compounds has been described in which fused rubidium bromide is used as the salt source. If another hetero compound, for example, phosphorous, is to be selectively detected, an appropriate alkali metal or earth alkali metal salt is placed in the dish **6**. Suitable for this purpose, in particular are salts of the first and second main groups of the periodic system of elements.

As shown, the negative supply **11** is directly connected to burner nozzle **8**. Supply **10** is connected to holder **5** through a switch **15** and supply **9** connected to burner nozzle **4** through a switch **17**. The other terminals of each of the switches **15** and **17** are connected to supply **11**.

FIG. **2** illustrates the arrangement of the system for the detection of hydrocarbons. Essentially, the same elements are present as were described above, with identical parts given the same reference numerals.

The main difference is the manner in which voltages are supplied to the various elements and through which nozzles the fuel is supplied. As illustrated, both nozzles **4** and **8** and the holder **5** are now all connected to the negative supply **11**. That is, switches **15** and **17** are switched from their respective positive supply connections to the negative supply **11**. As noted above, this supply is adjustable between 0 and 300 V and for use in detecting hydrocarbons, should be adjusted to a voltage of between approximately 200 and 300 V. The fuel is now fed in through the nozzle **4** along with the sample as indicated. Combustion air is supplied in the same manner as noted above. The nozzle **8** no longer acts as a fuel supply with its hydrogen supply being shut off, but only as an additional electrode in the system.

Thus, an improved FID device which may simply be switched to detect either hydrocarbons or nitrogen or other hetero compounds has been shown. Although specific embodiments have been illustrated and described, it will be obvious to those skilled in the art that various modifications may be made, without departing from the spirit of the invention which is intended to be limited solely by the appended claims.

What is claimed is:

1. A flame ionization detector comprising:

a. an enclosed detection chamber having a central axis associated therewith;

b. a collector electrode arranged in the upper portion of said chamber, essentially along said axis;

c. a first burner nozzle in the lower portion of said chamber, arranged with its outlet directed upward along said axis;

d. a salt source supported within said chamber between said collector and said first nozzle;

e. a second nozzle having its opening directed downwardly in the direction of said first burner nozzle and onto said salt source;

f. means for supplying a sample to said first nozzle;

g. means for selectively supplying a fuel gas to one of said first and second nozzles;

h. a negative voltage supply;

i. means for supplying one of a ground and positive potential;

j. means coupling said negative supply to said second nozzle;

k. means for selectively coupling said first burner nozzle to one of said negative supply and said means for supplying one of a positive and ground potential, whereby, by selectively feeding said fuel gas to said second nozzle and coupling said first nozzle to said means for supplying one of a positive and ground potential, a nitrogen or other hetero compound sample may be fed to said first nozzle for detection, and by selectively feeding said fuel gas to said first nozzle and coupling said first nozzle to said negative supply a hydrocarbon may be fed to said first nozzle and detected.

2. The invention according to claim **1** wherein said negative voltage is in the range of 200 to 300 V.

3. The invention according to claim **1** wherein said means for supplying a potential comprise means to supply a ground.

4. The invention according to claim **1** wherein said means to supply a potential comprise a positive voltage source.

5. The invention according to claim **4** and further including a second positive voltage source and means to selectively couple said second positive source to said salt source.

6. The invention according to claim **1** wherein said salt source is contained in a platinum dish.

7. The invention according to claim **6** wherein said platinum dish is mounted to said second burner nozzle such as to be capable of being moved with respect thereto and wherein said second burner nozzle is mounted for relative movement with respect to said first burner nozzle.

8. The invention according to claim **1** and further including an amplifier coupled to said collector electrode.

9. The invention according to claim **8** and further including recording means coupled to the output of said amplifier.

10. The invention according to claim **9** and further including means to selectively apply said negative voltage to said salt source.

* * * * *