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2,833,371

FILTER APPARATUS FOR FLAME PHOTOMETERS

Filed July 26, 1956

2 Sheets-Sheet 1

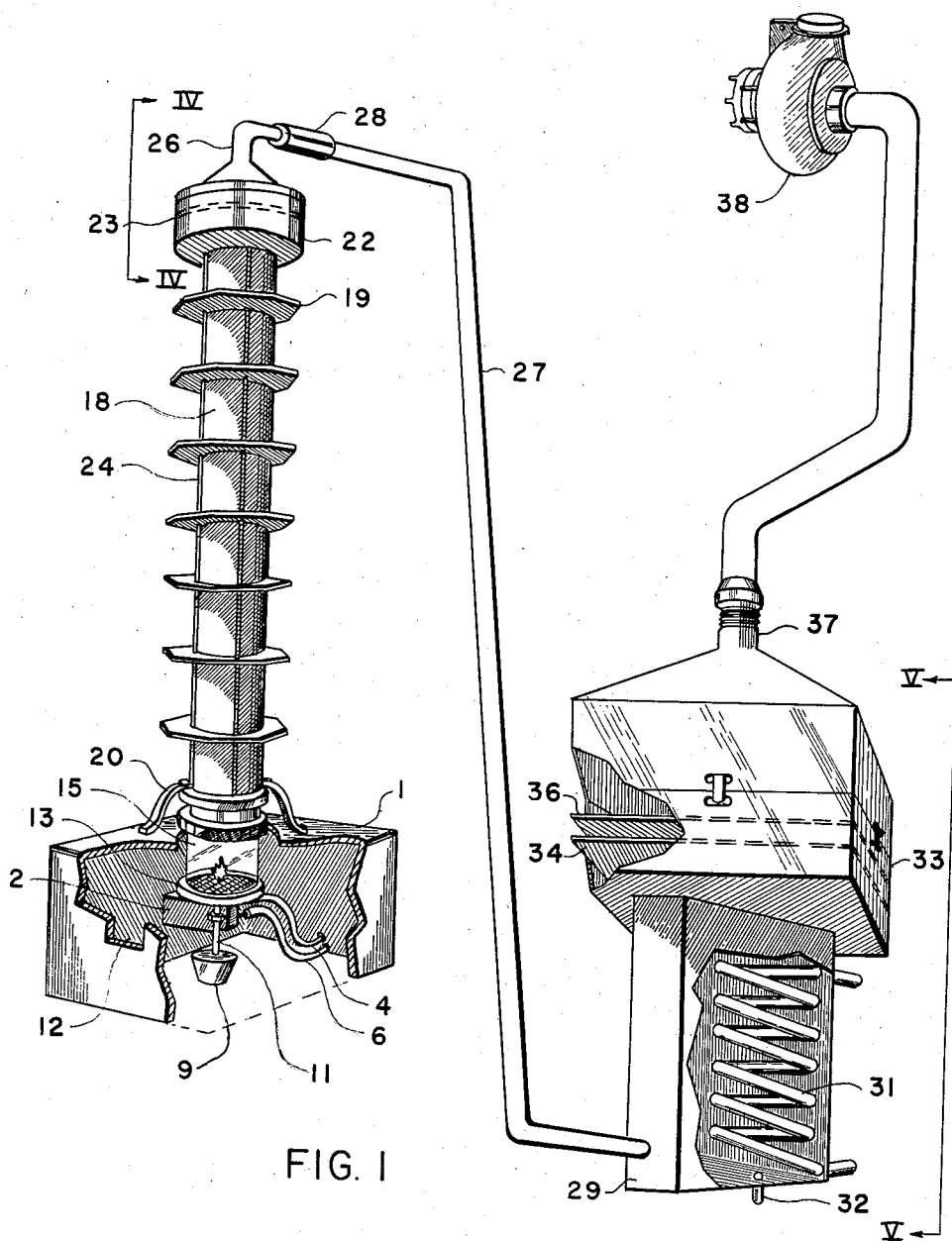


FIG. 1

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2 Sheets-Sheet 2

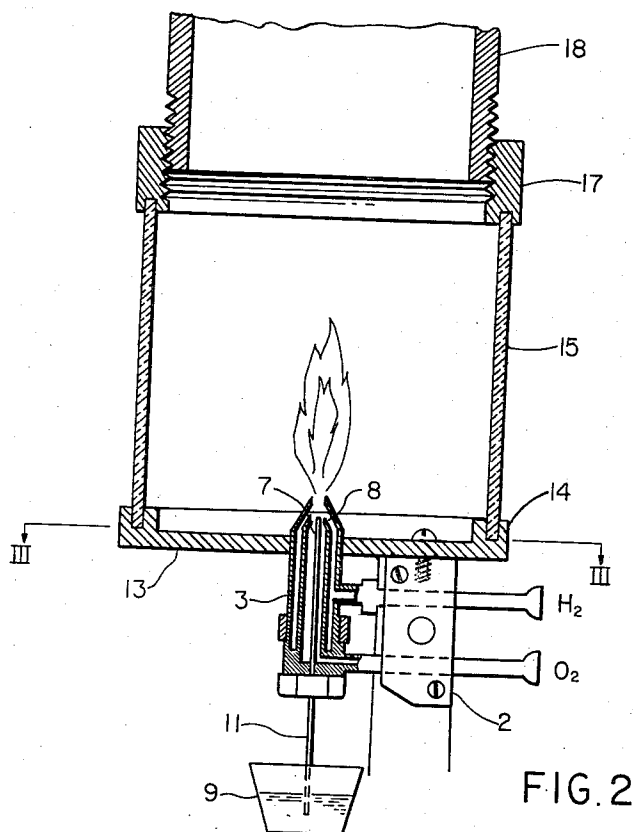


FIG. 2

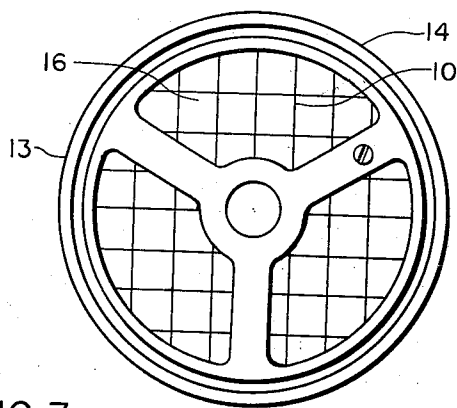


FIG. 3

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FILTER APPARATUS FOR FLAME PHOTOMETERS

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9 Claims. (Cl. 183—32)

(Granted under Title 35, U. S. Code (1952), sec. 266)

This invention may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

This invention relates to flame photometry and more in particular to apparatus for purifying the combustion gases evolved during flame photometric analyses.

Flame photometric analyses conventionally are carried out by spraying or atomizing a solution to be tested in the flame of a burner head which may be a Bunson burner or a hydrocarbon, oxyhydrogen, oxy-acetylene or some other type of burner. The radiation emitted by the flame is isolated and measured by an electronic detection unit, such as a Beckman spectrophotometer and, since the amount of the substance present in the solution is proportional to the radiation emitted, quantitative measurement of the substance is rendered possible. Although there have been numerous developments and improvements in the art of flame photometry, principally in improved burner designs, detection means, or in sample aspiration, the particular problem with which the present development is concerned apparently has received little or no attention.

Thus, in the conventional flame photometer, it has been almost universal practice to permit the exhaust gases, along with such particulate matter as may evolve with these gases, to diffuse freely into the surrounding area, although frequently some attempt is made to control these gases by operating the flame photometer in a ventilated hood. For this reason, the use of flame photometers has been limited mostly to determinations of test samples which are known to be safe to the extent that they do not contain noxious or harmful element such as radioactive elements or perhaps toxic substances such as beryllium compounds, fluorides or concentrated copper solutions.

Although it would seem quite possible that many others have recognized the fact that such noxious elements could be analyzed by flame photometry, no suitable apparatus for this purpose has been forthcoming. In fact, so far as has been learned from the available literature, there has been only one prior attempt along this line, this attempt being concerned with the problem of safely conducting away the gases evolved during sulphur determinations. However, even in this earlier work, the solution of the problem required a special burner head which was equipped with a forced air injection and, in general, the development failed to provide apparatus that could be easily and quickly adapted for use in making determinations of different noxious or radioactive substances.

It is, therefore, the primary object of the present invention to provide a flame photometric apparatus in which radioactive or otherwise noxious substances can be safely analyzed.

A more specific object is to provide a means for effectively purifying the burned gases of such a flame photometer to the extent that radioactive or noxious combustion products are removed before the burned gases are discharged into atmosphere.

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These and other objects of the invention generally are achieved by providing a closed, combustion product-containing circuit through which the combustion gases produced at the burner head are constrained to flow prior to their discharge to atmosphere. At this point, however, it might be noted that, although the circuit is defined as being "closed," this is true only in a limited sense. Thus, the circuit is closed to the extent that noxious combustion products are contained and cannot escape to atmosphere through either end. On the other hand, as already indicated, purified combustion gases can escape at the discharge end of the circuit and, in addition, atmosphere is admitted into the circuit at the other end. The end of the circuit nearest the burner head is closed in the above-defined sense by means of a base filter member in which the burner head is sealably mounted while, at the other or discharge end of the circuit, there is mounted a pump that is capable of producing an air current for drawing atmosphere in through the base filter member and discharging the atmosphere, along with the purified combustion gases directly into a room or perhaps into a ventilated hood. As will be appreciated, any such closed system requires a flow of air around the burner head and, according to the present invention, this flow is created by means of the pump that draws air through the filter member that surrounds the burner head.

Within the closed circuit itself there preferably are provided at least two other filter members and a condenser. One of these members, the primary filter, is disposed between the condenser and the burner head and, most suitably, it is located at such a position that the gases reaching it are above their condensation point. The secondary filter member is disposed between the condenser and the pump and its purpose is to filter any gases which may not be condensed. Also, in determinations involving radioactive materials it has been found preferable to use a tertiary filter between the secondary filter and the pump to assure complete purification.

The invention further provides a number of structural refinements that will become apparent in the ensuing detailed description. Notable among these refinements is the fact that the apparatus forming the closed circuit is readily and easily detachable for permitting replacement of individual filter members as the occasion arises. Also, the apparatus preferably includes a rigid, finned chimney member mounted directly above the flame and the primary filter is disposed at the top of this member. It has been found that such chimney must be changed with about every eight determinations so that ease of detachment is an important factor. It also has been found that the creation of air flow around the burner, while permitting sufficient heat for the optical requirements of the spectrophotometer, nevertheless causes the maximum temperature zone of the chimney to be a substantial distance above the connection between the chimney and the flame photometer housing; this feature permitting the connection to be easily broken because the area surrounding it remains sufficiently cool to be unscrewed or otherwise detached by hand. Vacuum gauges also may be used to constantly determine the pressure differentials of the various filters and, of course, when radioactive substances are being tested, a beta-gamma survey meter can be attached.

The preferred embodiment of the invention is illustrated in the accompanying drawing of which Fig. 1 is a somewhat diagrammatic perspective of the assembled apparatus, the apparatus being partially broken away to show underlying elements; Fig. 2 a vertical section through the burner head and its surrounding elements, and Fig. 3 a horizontal section taken along lines III—III of Fig. 2.

Referring to the drawings, the apparatus is conventional to the extent that it includes a flame photometer housing

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1 provided interiorly with a burner mounting block 2 that carries a burner head 3 (Fig. 2) into which hydrogen and oxygen are supplied by separate tubes 4 and 6. As may be noted in Fig. 2, this burner admits the oxygen into an interior chamber 7 and the hydrogen into an exterior chamber 8, both of these chambers having converging outlets at their upper ends to permit effective mixing of the gases. The test solution to be burned is carried in a closed vessel 9 that has fitted into it a slim pipette 11 extending axially through the center of the burner into converging proximity with the junction of the hydrogen and oxygen outlet openings. As will be appreciated, supply of the test solution then is accomplished by aspiration. Also, when the burner is lit and the test material aspirated into the flame, the analysis is made by means of a spectrophotometer which samples the flame radiation through a window 12 provided at one side of housing 1.

The foregoing apparatus is mostly conventional and can be varied in a number of different respects. The novel structure is, as stated, more concerned with the provision of the so-called closed circuit in which the burned gases evolving from the flame are effectively purified prior to their discharge to atmosphere. As also stated, one end of the closed circuit is formed by a base filter member 13 in the central portion of which burner head 3 is sealably mounted. Base member 13, most suitably, is formed of a base ring 14 (Fig. 3) supported on burner block 2 and a disc-shaped piece of filter material 16 formed of a high porosity, high retention filter paper of fire-proof material, the paper being backed by a circular screen mesh 10. A particular filter that has been found entirely suitable is one known as 1106 B glass filter paper containing 7 percent binder.

Burner head 3 projects inwardly through this base member so that the test sample is burned within a transparent glass cylinder 15 mounted in an annular groove formed in base ring 14 and, of course, it is through this transparent glass cylinder that the radiation intensity is observable. The upper portion of the glass cylinder also is securely mounted in an annular groove formed in a screw collar 17 by means of which the glass chimney is detachably secured to a finned metal chimney 18 that forms the first part of the purification circuit. As may be noted in Fig. 1, chimney 18 is supported on the top of photometer housing 1 by means of legs 20 and it may be readily detached simply by unscrewing collar 17. Also, chimney 18 preferably is provided with seven perforated copper heat-radiating plates 19 spaced one from the other by eight cylindrical pipe segments, and it is desirable to provide each of these segments with inwardly extending baffles of any configuration such as will produce a tortuous path through the chimney.

At the upper end of chimney 18 is mounted a filter housing 22 provided with a transverse filter member 23 that is held in place by four tie rods 24 which extend through copper fins 19 and are secured at their lower ends to legs 20. The filter element in filter housing 22 is known as the primary filter and it is formed preferably of a heat-resistant filter paper, such as the paper of filter 16, backed in the usual manner with wire mesh. Primary filter 23, as shown, divides the housing into inlet and outlet chambers, the outlet chamber converging at its upper end for funneling the gases into a short length of tubing 26. Tubing 26, in turn, is connected to another longer section of tubing 27 by a detachable connection 28 which may be of any conventional design capable of preventing escape of the gases. Tubing 27 leads into a condenser unit 29 in which is mounted a condenser coil 31, the function of which is to cool the incoming gases to a temperature below their condensation point. Preferably, the lower portion of this condenser chamber is provided with a suitable stopcock 32 by means of which the condensed fluid can be emptied.

Condenser housing 29 communicates at its upper end with a second filter housing 33 which is divided transversely by two separate filter members designated, for purposes of description, secondary filter member 34 and tertiary filter 36. According to conventional practice, these filter members are mounted in metal frames secured in any suitable manner to the interior of housing 33. Secondary filter member 34 may be formed of the same material as primary filter 23 and base filter 16 which, as stated, is a heat-resistant glass filter paper backed with a wire mesh. However, tertiary filter is of a special type which has a high retentivity for particulate radioactivity and an example of such a filter is that designated as the No. 6 filter by the Army Chemical Corps. Such a filter has a retentivity of approximately 99.9%. As is apparent, the tertiary filter is for the specific purpose of removing radioactivity and, when other noxious substances are being tested, it may not be required.

For purposes of convenience, filter housing 33 is made of two parts so as to permit easy access to the filters and the two parts are held together by four link-lock fasteners. The top part of the filter housing also is funneled into a short length of outlet pipe 37 to which is attached by means of a hose coupling a positive displacement pump 38 (Fig. 1). As has been stated, it is this pump 38 that creates sufficient pressure differential to draw air in through base filter member 13 and move the gases through the various filter members and the condenser. If desired, the pump may be discharged into a ventilated hood although, in most determinations, such a measure is purely precautionary and not absolutely required.

The operation of the apparatus should be quite clear from the foregoing description. In commencing a determination or analysis of a solution, such as a radioactive solution, the first step is to unscrew the glass chimney or cylinder by turning screw collar 17 until the glass chimney can be removed. The chimney then is taken out and, with the oxygen and fuel turned on, the burner is lit. Next, positive displacement pump 38 is turned on and, for most determinations involving the use of hydrogen-oxygen flame, the pump is operated at a sufficient speed to draw approximately 14 cubic feet of air per minute into and through the circuit. Next, the flame is turned as low as possible to permit replacement of the glass chimney. After this chimney has been replaced and tightly secured in position, it usually is desirable to initially introduce into the flame a small amount of distilled water to determine optimum operating conditions of the burner which, of course, may be adjusted to suit the circumstances. Finally, the radioactive solution is introduced by means of pipette 11 and it is analyzed by being burned in the flame.

In the operation of the filtering and condensing elements, it is found that a substantial portion of the heavier particulate material is not capable of being carried to the primary filter by means of the air stream and instead it falls to the base of the circuit where it is caught and held on the base filter paper. The gases and the remaining particulate material rising through chimney 18 come in contact with the copper heat radiators with the result that the gases are sufficiently cooled by the time the primary filtering action takes place at the top of the chimney. The primary filter, which is a glass wool filter mat, acts to retain such particulate matter as is composed essentially of the oxidation products of the solution being aspirated, while the gaseous products from the flame combustion diffuse through and into the condenser assembly.

One feature commented upon previously is that the rush of air around the flame causes the zone of maximum heat of the chimney to be substantially above the connection between the finned chimney and the glass tube 15. In fact, it is found that the hottest point of this chimney is about eight inches above housing 1 and this fact permits screw collar 17 to be manually operated.

However, as will be appreciated, the heating or cooling effects of the chimney will be dependent upon a number of factors one of which is the height of glass tube 15. In practice, this chimney is made two and one-half inches high and is cut from three inch diameter borosilicate glass tubing. When such a height is used, the heat of the hydrogen-oxygen flame is effectively dissipated by the radiator assembly, but nevertheless the heat within the glass tube is within the optical requirements of the spectrophotometer. Further, the cooling can be varied in the obvious manner by increasing the length of chimney 18 or by using some positive cooling means such as air or liquid circulation.

The condenser assembly is useful primarily in collecting the gaseous products of flame combustion and, preferably, the condenser is of such capacity that the gases passing from it into housing 23 are substantially at room temperature. In this regard, it may be noted that, when the described hydrogen-oxygen flame is used, the initial temperature of the gases is approximately 3,000° C. but by the time the gases reach the primary filter they have been reduced to the temperature of approximately 150° C. The secondary filter acts much in the manner as the primary filter to remove any remaining particulate matter and also the tertiary filter is useful especially for filtering out radioactive particles.

The principal advantage of the present apparatus is that radioactivity and other toxic particles or gases are effectively removed before release of the combustion gases to atmosphere. Of course, during operation, the filters gradually will become clogged and it usually is desirable to monitor their condition by means of suitable vacuum gauges (not shown). Radioactivity also is monitored by an appropriate beta gamma survey meter (also not shown). The apparatus requires relatively frequent decontamination particularly when it is used in radiation work. In practice, the primary filter, which has been found to trap over 99% of the particulate matter, should be changed after about eight separate determinations. Next to the primary filter, the highest contamination is found in the region of the burner and on the first and second baffle plates of the chimney. Usually only background activity can be found in the secondary and tertiary filters and there is no detectable activity in the pump or its housing couplings. However, all of these filters must be decontaminated after varying periods of use, so that the ease by which access is provided to the filters is of considerable importance. Thus, the two-part nature of housing 33 permits access to its filters while a breaking of connection 28 permits ready access to the primary filter. Preferably, condenser 29 is secured to filter 33 by means of ring-locks or other detachable connections so as to permit ready access.

Obviously many modifications and variations of the present invention are possible in the light of the above teachings. It is, therefore, to be understood that within the scope of the appended claims the invention may be practiced otherwise than specifically described.

I claim:

1. Apparatus for use in flame photometric analysis, said apparatus comprising an air-permeable base filter member, a burner head sealably mounted in said member, a pump, and a containing circuit communicating said pump with said base member, said circuit including a primary filter and a condenser, said primary filter being disposed in advance of said condenser for receiving and purifying uncondensed combustion gases prior to admittance of these gases into said condenser, and said pump being operable for drawing atmosphere into said circuit through said base filter for effecting filtration and for discharging said filtered gases back to atmosphere.

2. Apparatus for use in flame photometric analysis, said apparatus comprising an air-permeable base filter member, a burner head sealably mounted in said member, a pump, and a containing circuit communicating said pump

with said base member, said circuit including a primary filter, a condenser, and a secondary filter, said primary filter being disposed in advance of said condenser for receiving and purifying uncondensed combustion gases prior to admittance of these gases into said condenser, and said pump being operable for drawing atmosphere into said circuit through said base filter for effecting filtration and for discharging said filtered gases back to atmosphere.

3. Apparatus for use in flame photometric analysis, said apparatus comprising an air-permeable base filter member, a burner head sealably mounted in said member, a pump, and a containing circuit communicating said pump with said base member, said circuit including a primary filter, a condenser and a secondary filter, said primary filter being disposed in advance of said condenser for receiving and purifying uncondensed combustion gases prior to admittance of these gases into said condenser, said secondary filter being disposed in said closed circuit subsequent to said condenser, and said pump being operable for drawing atmosphere into said circuit through said base filter for effecting filtration and for discharging said filtered gases back to atmosphere.

4. Apparatus for use in flame photometric analysis, said apparatus comprising an air-permeable base filter member, a burner head sealably mounted in said member, a pump, and a containing circuit communicating said pump with said base member, said circuit including a primary filter, a condenser, a secondary filter, and a tertiary filter, said primary filter being disposed in advance of said condenser for receiving and purifying uncondensed combustion gases prior to admittance of these gases into said condenser, said secondary and tertiary filters being disposed in said closed circuit subsequent to said condenser, and said pump being operable for drawing atmosphere into said circuit through said base filter for effecting filtration and for discharging said filtered gases back to atmosphere.

5. Apparatus for use in flame photometric analysis, said apparatus comprising an air-permeable base filter member, a burner head sealably mounted in said member, a pipette means for feeding test material into said burner head, a pump, and a containing circuit communicating said pump with said base member, said circuit including a primary filter and a condenser, said filter being disposed in advance of said condenser for receiving and purifying uncondensed combustion gases prior to admittance of these gases into said condenser, and said pump being operable for drawing atmosphere into said circuit through said base filter for effecting filtration and for discharging said filtered gases back to atmosphere.

6. Apparatus for use in flame photometric analysis, said apparatus comprising a cylinder formed of transparent sides and an air-permeable base, a burner head projecting upwardly through the center of said base into said cylinder, means for feeding test material into said burner head, and means to effectively remove the products of the combustion of said test material, said removal means including a rigid cylindrical conduit supported in substantially axial alignment with and having its lower end sealably connected to the upper end of the transparent cylinder, cooling means associated with said conduit, a filter housing sealably carried at the upper end of said cylindrical conduit, a primary filter member dividing said housing into inlet and outlet chambers, a condenser, a conduit sealably interconnecting said condenser with said filter housing outlet chamber, a second filter housing having an inlet chamber communicated with said condenser, a secondary filter member forming inlet and outlet chambers in said second housing, and a pump operatively communicated with the outlet chamber of said second housing, said elements forming a containing circuit for said combustion material between said air-permeable cylinder base and said pump discharge, and said pump being operable for drawing atmosphere into

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said circuit through said base whereby a conductive current through the circuit is created.

7. Apparatus for use in flame photometric analysis, said apparatus comprising a cylinder formed of transparent sides and an air-permeable base filter member, a burner head projecting upwardly through the center of said base filter member into said cylinder, means for feeding test material into said burner head, and means to effectively remove the products of the combustion of said test material, said removable means including a rigid cylindrical conduit supported in substantially axial alignment with and having its lower end sealably connected to the upper end of the transparent cylinder, cooling means associated with said conduit, a filter housing sealably carried at the upper end of said cylindrical conduit, a primary filter member dividing said housing into inlet and outlet chambers, a condenser, a conduit sealably interconnecting said condenser with said filter housing outlet chamber, a second filter housing having an inlet chamber communicated with said condenser, a secondary filter member forming inlet and outlet chambers in said second housing, and a pump operatively communicated with the outlet chamber of said second housing, said elements forming a containing circuit for said combustion material between said air-permeable base filter member and said pump discharge, and said pump being operable for drawing atmosphere into said circuit through said base filter member whereby a conductive current through the circuit is created, said rigid vertical conduit being of such a length and so cooled as to admit combustion gases to said primary filter at a temperature slightly above their condensation temperature whereby combustion products carried by condensed gases are collected in said chamber.

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8. Apparatus for use in flame photometric analysis, said apparatus comprising a vertically-disposed cylinder formed of transparent sides and an air-permeable base filter member, a burner head projecting upwardly through the central portion of said base filter member into said cylinder, means for feeding test material into said burner head, and means to effectively remove the products of combustion of said test material, said removal means including an upwardly-projecting rigid cylindrical conduit supported in substantially axial alignment with and having its lower end sealably connected to the upper end of the transparent cylinder, cooling means associated with said conduit, a filter housing sealably carried at the upper end of said cylindrical conduit, a primary filter member mounted in said housing, a pump, and a circuit connecting said pump with said filter housing whereby said pump is operable for drawing atmosphere into said circuit through said base filter member for creating a conductive current through the apparatus, said products of combustion in part falling downwardly through the column onto said base filter member and in part being carried by said conductive current into said primary filter member.

9. Apparatus according to claim 8, said apparatus further including a condenser disposed in said circuit.

References Cited in the file of this patent

UNITED STATES PATENTS

1,030,028	Stampahar et al.	June 18, 1912
1,508,367	Matlock	Sept. 9, 1924
1,716,481	Bilsky	June 11, 1929
2,714,833	Gilbert	Aug. 9, 1955