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M. HONMA

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APPARATUS FOR SPECTROGRAPHIC ANALYSIS

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Fig. 1.

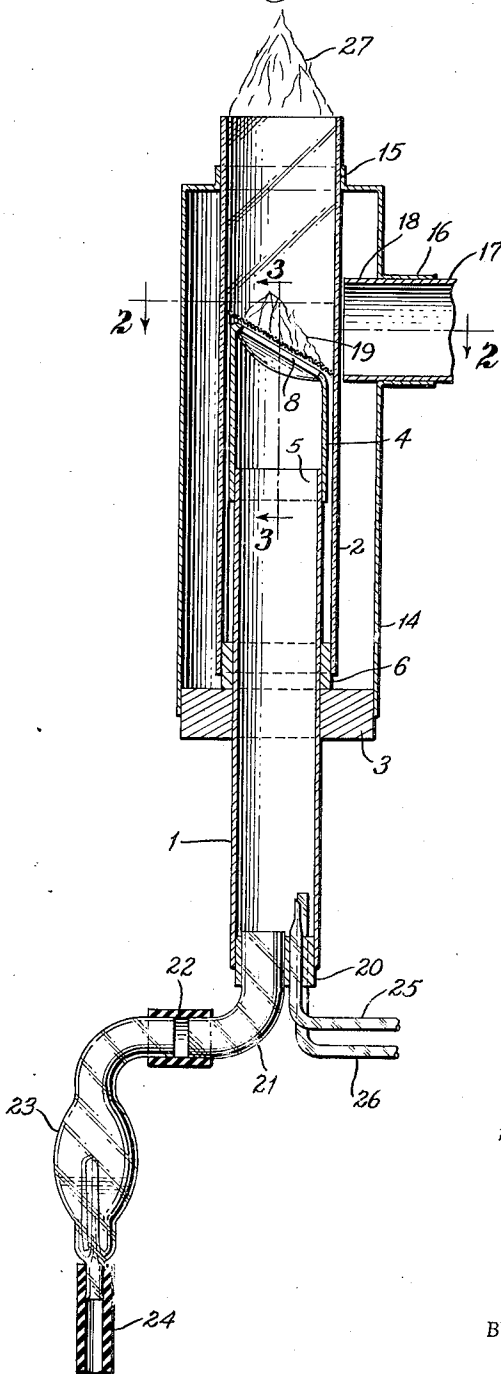


Fig. 2.

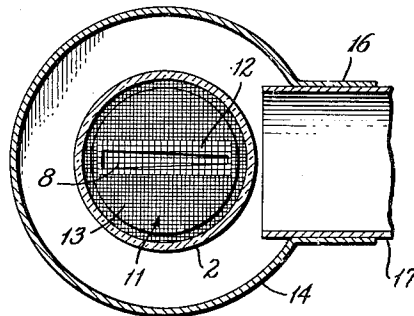


Fig. 3.

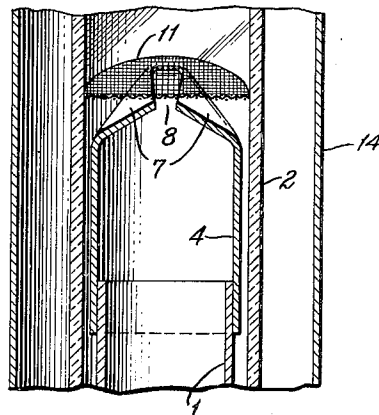
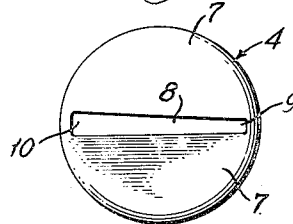


Fig. 4.



INVENTOR

Minoru Honma

BY *Swett, Mead, Brown & Schuyler*

ATTORNEYS

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APPARATUS FOR SPECTROGRAPHIC ANALYSIS

Minoru Honma, Iowa City, Iowa, assignor to The State of Iowa, for the benefit of the State University of Iowa, Iowa City, Iowa, an educational institution of Iowa

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8 Claims. (Cl. 88—14)

This invention relates to apparatus for spectrographic analysis, and more particularly to a burner useful for performing qualitative and quantitative analysis by flame spectroscopy.

As will be appreciated by those skilled in the art, methods of analysis by means of emission spectra have been widely developed and have been used extensively in the analysis of metals, alloys, and inorganic salts in mixtures and solutions. Generally, electrical excitation has been used to produce the source unit. In recent years, the use of the flame photometer for the rapid and selective estimation of materials, particularly alkalis, is finding increasing acceptance and is gradually replacing more troublesome methods of analysis.

In flame spectroscopy or photometry, the material to be analyzed, usually as an analysis solution, is sprayed into the air supply of the flame. The light from the flame is then analyzed photographically or photometrically for the constituent in question. These methods require fairly rigid standardization of all controllable variables but are capable of high accuracy in routine work. They have the advantage that the standards with which the samples are compared may be made up easily and accurately from pure materials. Many of the difficulties which attend other spectrographic methods such as segregation, grain size, and physical state of the sample, are avoided.

The present invention is directed broadly to the problem of providing an improved flame source for use in flame spectroscopy, and more particularly to the problem of providing an intense flame which is substantially free from background and interference and which is substantially stable during the time when it is being photographed. I have used conventional burners, such as Meker burners, Bunsen burners or microburners, in an effort to obtain an intense stable flame source but discarded them because I found that even the inner cone of flame produced by them is not intense enough and it is not sufficiently stable for test purposes. This inner cone of flame is produced by flame separation but these known types of burners are very sensitive to fluctuations in gas and air pressures and hence it is difficult to obtain flame separation with them. I have found that by definitely maintaining separation of the inner cone from the outer cone of the flame, several advantages are obtained. One advantage is that interfering bands and continuum from the outer cone are eliminated, and another is that it is possible to increase the intensity of the inner cone without interference from the outer cone.

It is therefore an object of my invention to provide a new and improved apparatus for performing analysis by flame spectroscopy and whereby it is possible to obtain a very intense flame thus providing greater light for spectrographic analysis.

It is another object of my invention to provide such apparatus whereby it is possible to separate a flame source into an inner cone and an outer cone and maintain said inner cone substantially stable despite normal fluctuations of air or gas pressure.

It is a further object of my invention to provide such apparatus whereby it is possible to substantially prevent interfering bands and continuum from appearing in the inner cone of a separated flame.

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Broadly described, the method of using the apparatus of my invention comprises separating the flame source into an inner cone and an outer cone and concentrating and stabilizing said inner cone to produce a very intense flame source. Material to be analyzed is then introduced into this intense flame and the light therefrom is analyzed spectrographically, as by taking a photograph by means of a spectroscopic apparatus. In further detail, gas and primary air are burned to produce a flame. This flame is then separated into an inner cone and an outer cone by feeding auxiliary air to the flame. The inner cone is concentrated and stabilized to provide an intense sheet of flame. When the auxiliary air pressure and gas pressure have been regulated to provide satisfactory flame separation, it is possible to leave these pressures set and introduce the material to be analyzed into the primary air supply for the tests.

A preferred embodiment of the apparatus of my invention is a burner having an inner hollow tubular mixing chamber with an inclined tapered outlet slot at its upper end. Inlets for the primary air, auxiliary air and gas are provided in the mixing chamber. A hollow tubular chimney-like housing surrounds the mixing chamber and extends beyond the outlet slot thereof. A screen baffle is located within said housing and overlies said outlet slot for a purpose later described. Means are provided to view the sheet of flame produced at said outlet slot, said means enabling a photographic record to be made.

Other objects and advantages of my invention will become more apparent from the following detailed description taken in conjunction with the attached drawing in which:

Fig. 1 is a vertical sectional view of an embodiment of the apparatus of my invention;

Fig. 2 is a horizontal sectional view of said embodiment taken on line 2—2 of Fig. 1;

Fig. 3 is an enlarged sectional view of said embodiment taken on line 3—3 of Fig. 1; and

Fig. 4 is a plan view of a portion of the apparatus shown in Fig. 1.

The apparatus shown on the drawing is a burner which includes, among its principal components, an inner hollow tubular mixing chamber 1 and an outer hollow tubular chimney-like member 2 which forms a housing for the upper portion of mixing chamber 1. Mixing chamber 1 extends through a mounting 3, which may be a cork ring, and has an extension formed by an adapter 4 fitted over the upper end 5 of the mixing chamber as shown in Fig. 1. Housing 2 extends beyond the upper portion of mixing chamber 1 and also surrounds and extends beyond adapter 4 as is shown in Fig. 1. In the embodiment shown on the drawing, tubular members 1 and 2, and also adapter 4, are cylindrical in shape. Furthermore, housing 2 is concentric with and spaced from mixing chamber 1 and adapter 4. The lower end of housing 2 is mounted on mounting member 6 which may be a cork ring similar to ring 3, and, in fact, mounting member 6 may be an extension of mounting member 3.

Adapter 4, which fits over and provides an extension of the upper end 5 of tubular member 1, in the manner of a tall cap member, has a converging upper end portion 7 which defines an outlet orifice or slot 8. As can be seen from Figs. 2—4 this outlet slot 8 is tapered or, to put it another way, is of gradually increasing width from its lower end 9 to its upper end 10. Furthermore, as can be seen best from viewing Fig. 1, slot 8 is inclined with respect to the axis of tubular member 1 and the angle of inclination may be in the neighborhood of 30° from the horizontal as seen in Fig. 1.

While I prefer to use an adapter over the upper end

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of tubular member 1, as this facilitates replacement, it is obvious that tubular member 1 and adapter 4 could be made integral, or as one member. In other words, the upper end portion 5 of tubular member 1 could be heightened and converged in the manner of adapter 4 to define slot 8. Accordingly, when I refer in the claims appended hereto, to an outlet slot at one end of the mixing chamber this is to be understood as meaning that the mixing chamber is defined by both tubular member 1 and adapter 4 and can be defined solely by tubular member 1 if the upper end of this member is properly shaped.

A differential screen baffle or barrier 11 is located within tubular housing 2 and overlies the upper end of adapter 4. In fact, this screen baffle or barrier 11 may lie directly against the upper end of adapter 4 and includes a coarse screen portion 12 and a fine screen portion 13. The coarse screen portion 12 is positioned directly over outlet slot 8 and the fine screen portion 13 extends across the remainder of the space defined by housing 2 as shown in Figs. 1-3. While screen baffle 11 may be made of any suitable material, I have found that it is satisfactory to make the coarse portion 12 of brass and the fine portion 13 of copper. The purpose of this screen barrier is described later in the specification.

A light shield 14, which is preferably made of metal, surrounds tubular member 2 as shown in Fig. 1. Thus, this light shield casing 14 has its lower end supported on mounting ring 3 and has a reduced diameter collar portion 15 which fits snugly about the upper end portion of tubular member 2 as is shown in Fig. 1. In order to provide an opening through which flame appearing at slot 8 may be viewed, light shield 14 has an opening therein defined by a hollow sleeve 16 which projects from the light shield as shown in Figs. 1 and 2.

A viewing tube 17, which may be called a light tube, fits snugly within sleeve 16 and projects through the opening in light shield 14 which is defined by sleeve 16. The end portion 18 of viewing tube 17 is closely adjacent to tubular member 2 and in a position such that it can transmit light from the flame 19 which is depicted as coming from tapered slot 8. In fact, the diameter of viewing tube 17 will be at least equal to the height of flame 19 so that the full intense flame ridge produced as the result of the inclined slot and screen baffle arrangement can be viewed. In this connection, reference is again made to the fact that the tapered slot 8 has a narrow end 9 and a wide end 10 as shown more particularly in Fig. 4. The wide end 10 is the upper end of the slot, i. e., the end which is highest with respect to the base of tubular member 1. Moreover, it is also the end which is farthest away from the viewing tube 17. However, because the upper end 10 of slot 8 is wider than the lower end 9, there is more flame at the upper end and hence more light can come from this end of the slot to and through the viewing tube 17 to the spectroscopic apparatus. As is well known, the intensity of light from a given light source varies inversely with the square of the distance from the light source. Therefore, even though the spectroscopic apparatus is not a great distance away from the flame source, the fact that the portion of the flame source which is farthest away from the spectroscopic apparatus produces more light than that portion of the flame source which is nearest to the spectroscopic apparatus is an important factor in achieving a uniform line in a photographic record. The tapered outlet slot 8, or sloping orifice, according to the present invention, is thus a very important feature in achieving a good photographic record. The taper of the slot compensates for the fact that one end of the slot is farther away from the spectroscopic apparatus than the other end of the slot.

Preferably, the inner surface of viewing or light tube 17 will be coated with black optical paint and the tube 17 will lead to a spectroscopic apparatus, not shown, by means of which a photographic record of particular tests

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may be made. In the embodiment shown, the chimney-like tubular member 2 is preferably made of a transparent material such as Pyrex glass. However, it will be obvious to those skilled in the art that this tubular member 2 could be made of any suitable material as long as it is provided with a window portion opposite flame 19 and through which light can pass into light tube 17.

In order to admit fuel and material to be analyzed to the burner, several tubes extend into the lower end of tubular mixing chamber 1 through a mounting member 20, which may be a cork plug. The largest of these tubes is shown as an elbow 21 which has one end opening into the interior of mixing chamber 1 and the other end extending into a flexible sleeve 22, which may be made of rubber. Also extending into flexible sleeve 22 is one end of an atomizer 23, the other end of the atomizer being connected to a source of primary air which is fed to the atomizer through tube 24. The type of atomizer shown on the drawing is a well-known one and since its construction and operation will be apparent to those skilled in the art, further description of it is omitted as being unnecessary.

Gas and auxiliary air are fed to the interior of mixing chamber 1 through feed tubes 25 and 26 respectively.

In operating the burner of the present invention, a preferred procedure is to first turn on the gas which feeds through tube 25 into the interior of mixing chamber 1. Then the gas is regulated to a suitable pressure and the burner lit, at which point the entire flame burns at the top of chimney-like tubular member 2, this flame being depicted at 27 in Fig. 1. The primary air supply is then turned on and regulated to a suitable pressure and it feeds through tube 24, through atomizer 23, and through elbow 21 into the interior of mixing chamber 1 where it is led to the flame 27. By then turning on the auxiliary air, which feeds through tube 26 into mixing chamber 1, and regulating the pressure of this auxiliary air; it is possible to separate flame 27 into an inner cone, which is the flame 19 shown in Fig. 1, and an outer cone which remains at the top of chimney-like tubular member 2. The inner cone can be brought down onto the differential screen 11 and will burn thereon as an intense sheet of flame, or ridge of light, because of the tapered shape of adapter slot 8 which permits an even escape of fuel from the slot.

When the fuel gas pressure and auxiliary air pressure have been satisfactorily regulated, atomizer 23 can be disconnected from flexible coupling 22, filled with material to be analyzed, which will usually be in solution, and then reconnected into the primary air supply whereby the material to be analyzed is fed to the flame. This process can be repeated for successive tests. During the time when the atomizer is disconnected, the inner cone 19 of flame will jump up and rejoin the flame 27 at the top of chimney-like tubular member 2, thus causing an unseparated flame during this stage of the proceedings. However, when the atomizer is reconnected and the primary air is being fed into the mixing chamber 1, flame separation again occurs and the inner cone 19 appears thereby enabling a photographic record to be taken. When the inner cone 19 is down upon the differential screen baffle or barrier 11, the outer cone burns at the top of chimney-like tubular member 2 with a substantially invisible flame. By regulating the auxiliary air supply, it is possible to vary the height of the inner cone flame 19.

As mentioned earlier in this specification, separating the flame into an inner cone and an outer cone has definite advantages from the standpoint of facilitating spectroscopic analysis. By using the slot configuration and screen baffle arrangement of the present invention, it is possible to obtain a much improved inner cone flame source. Taking atomized acetonitrile solution as an example of a material tested, it was found that merely separating the flame into an inner cone and an outer cone, even by previously known methods, produced a flame

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source (the inner cone flame) four times as intense as the unseparated flame. Using the slot and screen baffle arrangement of the present invention, it is possible to obtain a flame source at the inner cone which is twelve times as intense as the unseparated flame and which is not subject to objectionable background and continuum. Thus, separating the flame produces a better flame source and utilizing the slot and screen baffle arrangement in addition to flame separation produces an even better flame source.

In addition to providing a more intense flame source for spectrographic analysis, the slot and screen baffle arrangement of the present invention also provides a more stable flame source and one which is not subject to failure under normal gas and air pressure fluctuations. The screen baffle is found to prevent backfiring of the burner and it permits the atomized vapors to escape only through the orifice or outlet slot of the adapter. In fact, I have found that by using the method and apparatus of my invention, the exposure time for making a photographic record for analysis purposes is reduced from a number of hours to a relatively few minutes. Thus, by use of my method and apparatus, flame excitation becomes comparable to electrical excitation.

I have used my apparatus in qualitative and quantitative determinations of nitrogen, sodium, potassium and chlorine, and it is obvious that it may be applied to other materials, the atoms of which may be excited by flame. Perhaps the greatest spectrographic advantage of my apparatus is that analysis of organic nitrogen may be done in the presence of atmospheric nitrogen.

While I have described and illustrated a preferred embodiment of my invention, I wish it to be understood that I do not intend to be restricted solely thereto but that I do intend to cover all modifications thereof which would be apparent to one skilled in the art and which come within the spirit and scope of my invention as expressed in the appended claims.

What I claim as my invention is:

1. A burner useful for performing analysis by flame spectroscopy comprising a substantially upright inner hollow tubular member having an outlet slot at the upper end thereof, said outlet slot being inclined with respect to the axis of said inner tubular member, an outer hollow tubular member concentric with, spaced from and surrounding said inner tubular member in the vicinity of said outlet slot, and a baffle located within said outer tubular member, said baffle having an angle of inclination with respect to the axis of said inner tubular member corresponding to the angle of inclination of said outlet slot.

2. A burner useful for performing analysis by flame spectroscopy comprising a substantially upright inner hollow tubular mixing chamber, a transparent outer hollow tubular housing surrounding said mixing chamber, said mixing chamber having an outlet slot at the upper end thereof and said housing extending beyond said outlet slot, said outlet slot having an upper end and a lower end and extending between said ends at an inclined angle with respect to the axis of said mixing chamber, and a light shield surrounding said housing, said light shield having an opening therein substantially opposite said outlet slot whereby flame emitted from said outlet slot may be viewed through said light shield.

3. A burner useful for performing analysis by flame spectroscopy comprising a substantially upright hollow tubular mixing chamber having an elongated outlet slot at its upper end; said outlet slot having an upper end and a lower end and extending between said ends at an inclined angle with respect to the axis of said mixing chamber means to admit material to be analyzed to said mixing chamber at its lower end, a hollow tubular chimney-like housing concentric with, spaced from, and surrounding said mixing chamber and extending beyond the upper end thereof, a light shield surrounding said housing, and a light tube projecting from said light shield substantially

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opposite said outlet slot whereby flame emitted from said outlet slot may be viewed through said light shield.

4. A burner useful for performing analysis by flame spectroscopy comprising a substantially upright tubular mixing chamber having an outlet slot at the upper end thereof, said outlet slot having an upper end and a lower end and extending between said ends at an inclined angle with respect to the axis of said mixing chamber, means connected to said mixing chamber to admit material to be analyzed to said mixing chamber below the upper end of said mixing chamber, a tubular housing surrounding said mixing chamber in the vicinity of said outlet slot, a screen baffle located within said housing and over said outlet slot, and means connected to said housing to view flame from said outlet slot through said housing.

5. A burner useful for performing analysis by flame spectroscopy comprising a substantially upright inner hollow tubular member having an outlet slot at the upper end thereof, an outer hollow tubular member concentric with and surrounding said inner tubular member in the vicinity of said outlet slot, and a screen baffle located within said outer tubular member and overlying said outlet slot, said baffle consisting of a coarse screen portion and a fine screen portion, the coarse screen portion being located over said outlet slot.

6. A burner useful for performing analysis by flame spectroscopy comprising a substantially upright hollow tubular mixing chamber having an elongated outlet slot at its upper end, said outlet slot having an upper end and a lower end and extending between said ends at an inclined angle with respect to the axis of said mixing chamber, the upper end of said outlet slot being wider than the lower end of said outlet slot, a light tube having an end, and light tube support means connected to said mixing chamber and supporting said light tube so that said light tube end is located adjacent said outlet slot, the upper wider end of said outlet slot being farther away from said adjacent end of said light tube than the lower narrower end of said outlet slot.

7. A burner useful for performing analysis by flame spectroscopy comprising an elongated tubular mixing chamber having an upper end portion defining a tapered outlet slot which extends across said upper end portion at an inclined angle with respect to the longitudinal axis of said tubular mixing chamber, said outlet slot having an upper end and a lower end, the upper end of said outlet slot being wider than the lower end of said outlet slot, means connected to said mixing chamber to admit material to be analyzed to said mixing chamber below the upper end portion of said mixing chamber, and a housing surrounding said outlet slot, said housing including means to view flame from said outlet slot.

8. A burner according to claim 3 including a screen baffle positioned within said chimney-like housing and overlying said outlet slot.

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