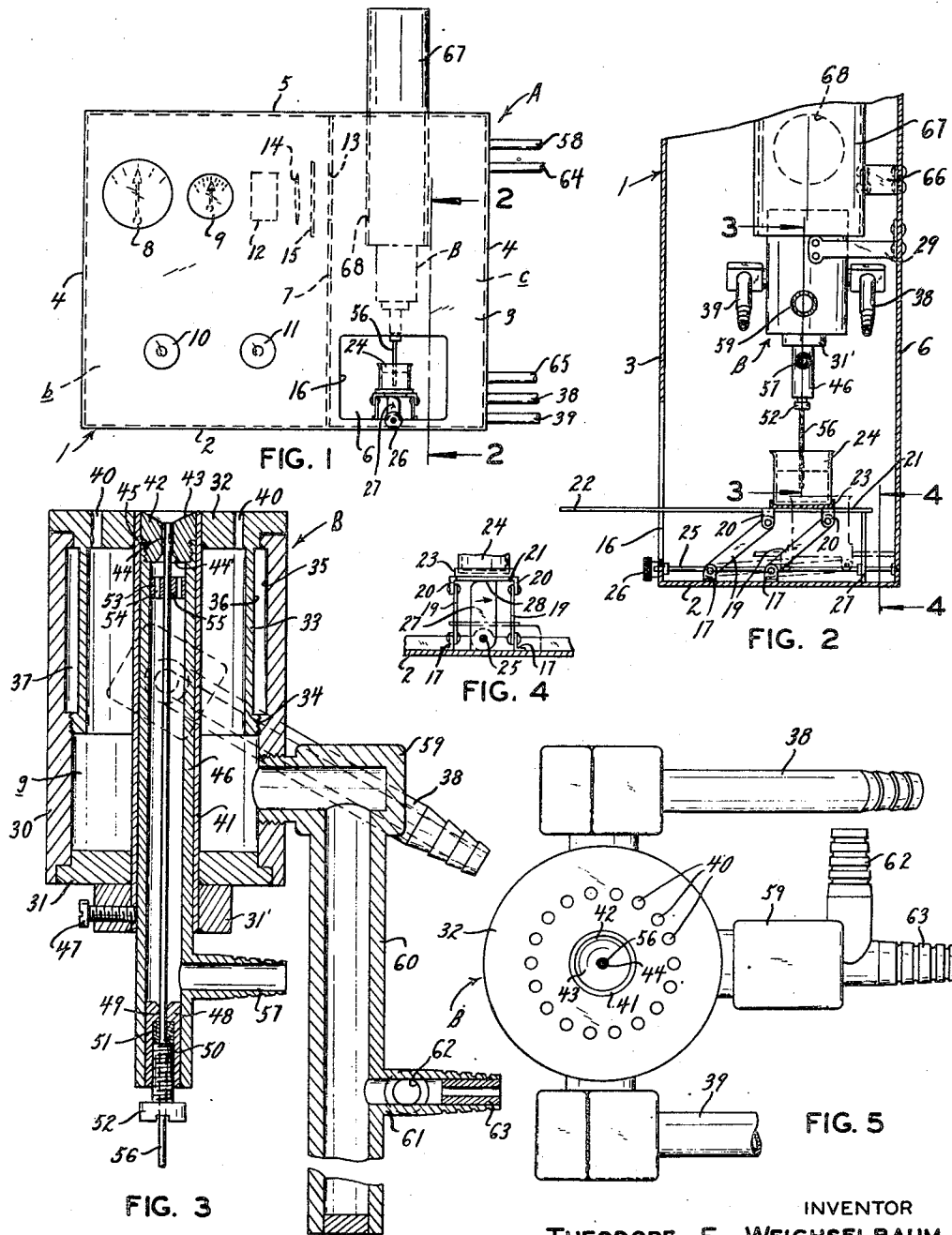


Dec. 5, 1950

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METHOD AND FLAME GENERATING APPARATUS FOR
SPECTROPHOTOMETRIC QUANTITATIVE ANALYSIS
Filed Oct. 21, 1948

2,532,687



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2,532,687

METHODS AND FLAME GENERATING APPARATUS FOR SPECTROPHOTOMETRIC QUANTITATIVE ANALYSIS

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Application October 21, 1948, Serial No. 55,754

11 Claims. (Cl. 88-14)

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This invention relates in general to methods and apparatus for spectro-photometric quantitative analysis and, more particularly, to a new and novel flame injection burner for use in conjunction with spectro-photometers.

At the present time, extensive efforts have been made to perform quantitative analyses upon solutions containing heat-ionizable cations by photometric methods by introducing a measured quantity of such solution into an oxy-hydrogen, oxygen-gas or gas-air flame and determining, by spectro-photometric means, the intensity of color thereby imparted to the flame. Appreciable inaccuracies and inconclusive results have been encountered and difficulties in reducing such methods to reliable routines have resulted due to lack of precision in the methods and apparatus by which the solution being analyzed is introduced into the flame.

It is, hence, the primary object of the present invention to provide methods and apparatus for spectro-photometric quantitative analysis which are simple, accurate and precise.

It is a further object of the present invention to provide for use with a photometer a simple, highly efficient flame producing apparatus or burner which is capable of accurate, reliable and uniform operation.

It is also an object of the present invention to provide a photometric burner having injection means capable of introducing a solution to be analyzed into the flame at a steady uniform rate and in an accurately controlled amount so as to produce a photometrically measurable flame-effect which bears a precise quantitative relation to the chemical constituency of such solution.

It is an additional object of the invention to provide apparatus of the type stated which is economical in construction and simple in operation.

In the drawing,

Figure 1 is a front elevational view of photometric quantitative analytical apparatus constructed in accordance with and embodying the present invention.

Figure 2 is a fragmental vertical sectional view of the photometric quantitative analytical apparatus taken along line 2-2 of Figure 1.

Figures 3 and 4 are fragmentary vertical sectional view taken along lines 3-3 and 4-4 respectively of Figure 2; and

Figure 5 is a top plan view of the burner forming a part of the present invention.

Broadly speaking the present invention resides

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in the use of a color-analyzing photometer in combination with an oxy-hydrogen or oxygen-gas burner or so-called lamp having means for introducing a precisely constant flow of the liquid or solution to be analyzed directly into the interior portion of the flame.

Referring now in more detail and by reference characters to the drawing, which illustrates a preferred embodiment of the present invention, A designates a photometric quantitative analysis apparatus comprising a cabinet 1 having a bottom wall 2, a front wall 3, side walls 4, a top wall 5, a back wall 6, and a vertical partition 7 sub-dividing the cabinet transversely into an instrument compartment b and a burner compartment c. Suitably mounted in the instrument compartment b is a switch-back multivoltmeter comprising a potentiometric indicator 8, galvanometer 9, and variable-resistance control knobs 10, 11, conventionally connected in circuit with each other and with one or more photocells or photo-tubes 12 in such a manner that the voltage in the circuit, as shown in the galvanometer 9, may be controlled by the knobs 10, 11, to balance the voltage output of the photocells 12 as shown on the indicator 8. The photometric circuit, in itself, may be of any well known type and hence has not been illustrated.

The photocell 12 is located adjacent the partition 7 opposite a window or aperture 13 therein and interposed between the photocell 12 and a window 13 are a lens 14 and color filter 15, as schematically indicated in Figure 1 and for purposes presently more fully appearing.

The portion of the front wall 3 extending across the burner compartment c is provided with a large rectangular opening 16 and welded or otherwise fixed upon the bottom wall 2 inwardly of and substantially centered, with respect to, the opening 16, are longitudinally spaced pairs of laterally aligned pivot-lugs 17. Swingably mounted at their lower ends upon the pivot-lugs 17 are pairs of parallel links 19, respectively, which are, in turn, swingably connected at their upper ends to depending ears 20 formed integrally with a tray-bar 21 extending at its forward end outwardly through the opening 16 in the formation of a handle 22 and being provided adjacent its interior end with a tray 23 adapted to receive and retain a sample-holding beaker 24. Journaled in and extending horizontally between the front wall 3 and back wall 6, preferably midway between the pivot-lugs 17, is a shaft 25 provided on its outer end with a knurled operating

knob 26 and on its inner end with a locking blade 27 having a flat transverse end face 28 for optional engagement with the interior end of the tray-bar 21, as shown in Figures 2 and 4. It should be noted in this connection that the links 19 are of such length, with respect to each other, and are so arranged that the tray-bar 21 will always remain horizontal as it is shifted up and down and the locking blade 27 is of such width that when in upright position, as shown in full lines in Figure 4, the tray-bar 21 will rest on the flat end face 28 and be supported stationarily in upwardly shifted position. On the other hand, when the locking blade 27 is swung down to the horizontal position, shown in dotted lines in Figure 4, the tray-bar 21 may be manually lowered to the position shown in dotted lines in Figure 2.

Supported, by means of a bracket 29, within the burner compartment c, in upwardly aligned relation to the beaker-tray 23, is a lamp or burner B comprising an outer cylindrical shell 30 provided in its lower end with a transverse closure plug 31 peripherally welded or brazed thereto to form a gas-tight permanent seal and being provided upon its under face with a concentric collar 31'. Peripherally brazed or otherwise secured within and across the upper end of the shell 30 is a burner-plate 32 integrally provided upon its under face with a depending cylindrical skirt 33 threadedly engaged, around its lower peripheral margin, as at 34, with the inner face of the shell 31. Both the shell 31 and the skirt 33 are complementarily under-cut, as at 35, 36, respectively, to provide a chamber 37 which communicates through the walls of the shell 31 with a water inlet pipe 38 and a water outlet pipe 39 in the provision of a water jacket for cooling the lamp or burner B. Extending axially through the shell 30 and being brazed at its ends to the plug 31 and burner plate 32 is a steel tube 41 in the formation of a gas chamber g within the shell 30. The burner plate 32 is provided just inwardly of the skirt 33 with a uniformly spaced annular series of apertures or burner-ports 40 opening into the gas chamber g.

Rigidly mounted and tightly sealed within the upper end of the tube 41 is an inserted nozzle 42 having a downwardly depressed conical upper face 43 and a central or axial bore 44 which flares out at its lower end as at 44'. The nozzle 42 is turned down along its lower or interior end in the provision of an externally threaded neck-portion 45 for engagement with the upper end of a nozzle-tube 46 which extends snugly through the steel tube 41 being releasably locked in place by a set screw 47 threadedly mounted in and extending radially through the collar 31'. Welded or otherwise tightly secured within the lower end of the nozzle tube 46 is a gland sleeve 48 internally bored as at 49 and counterbored as at 50 for receiving a packing ring 51 held in place by an axially bored packing screw 52. Similarly welded in the upper end of the nozzle tube 46, in downwardly spaced relation to the nozzle 42, is a collar 53 having an annular series of apertures 54 and a central bore 55 axially aligned with the bores of the gland-sleeve 48 and the packing screw 52 for supporting a fine-bore liquid-tube or capillary 56 which extends axially there-through and projects downwardly at its lower end for a substantial distance below the packing screw 52 so as to dip down into the liquid sample held within the beaker 24 when the tray bar 21 is in elevated position. It should be noted in this connection that the lower or intake end of

the capillary 56 is so positioned in relation to the arc of movement of the beaker 24, as it moves from upper to lower position, that it will clear the edge of the beaker 24 and will be located close to the bottom of the beaker 24 when the latter is in elevated position. The upper end of the capillary 56 extends through and is of substantially smaller diameter than the bore 44 of the nozzle 42 so as to provide an annular space between the nozzle-bore 44 and the liquid tube or capillary 56 and thereby create a Venturi-effect at the mouth of the nozzle 42. It should be noted in this connection that the upper end of the capillary 56 should preferably be precisely concentric with the bore 44 to insure maximum uniformity and accuracy of analytical results. The capillary 56, furthermore, may be adjusted vertically up or down so that its upper or discharge end will be located at an optimum position in relation to the upper or discharge end of the bore 44, since it has been found that this adjustment is somewhat critical for the different groups of unknown chemical elements, as to which the analytical determination is being made. For example, if the solution is to be analyzed for sodium content the adjustment can be made empirically using known standard solutions and then left at that setting for subsequent sodium determinations on unknown solutions. It is, of course, obvious that such adjustment can be made at the time the instrument is being built initially and fixed permanently as by soldering or welding, in which case the instrument is thereafter permanently a sodium analysis device, or the adjustment can be left unfixed so that the user may make appropriate adjustments from time to time as necessity requires, in which case the instrument is more or less universal in its applicability.

Brazed into and extending through the nozzle tube 46 is a tubular lateral off-take 57 for connection to an oxygen line 58 (see Figure 1) and threadedly mounted in the shell 31 is an elbow 59 having an integral vertical leg 60 which is in turn integrally provided with a Venturi-type T connection 61 having a gas off-take 62 and an oxygen off-take 63 for connection respectively through a hydrogen or gas supply line 64 and oxygen line 65 to sources of hydrogen or other suitable inflammable gas and oxygen (not shown). As the high pressure oxygen rushes through the off-take 63 past the orifice of the off-take 62, suction is exerted on the hydrogen line 64 which draws hydrogen or other suitable inflammable gas into the oxygen stream.

Supported within the chamber c for upward and downward shifting movement in vertically aligned relation to the burner B, by means of a sliding bracket 66, is a chimney 67 extending upwardly through the top wall 5 of the cabinet 1. The chimney 67 is provided with a circular window 68 located opposite and in horizontal alignment with the filter 15 and the lens 14.

In use the burner B is set in operation by first turning on the water supply so that cooling water will circulate through the water-jacket chamber 37. Next in order the hydrogen or other suitable inflammable gas is turned on to the line 64 and the gas which fills the chamber g exits through the burner ports 40. The burner B may then be lit by lifting the chimney 67 and applying a match or other conventional pyrophoric means to the stream of gas issuing from the burner-ports 40. The oxygen is then turned on to the line 65 and the gas pressures

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in the lines 64, 65, are adjusted by conventional pressure regulators (not shown) until the gases burn steadily and evenly in a light blue practically colorless flame. Under these conditions the flame will burn "straight up," so to speak, in the form of a vertical cylinder.

The oxygen is then turned on to the oxygen supply line 58 and pressure carefully regulated by a conventional regulating valve (not shown). This stream of oxygen will flow upwardly through the nozzle tube 46 and issue in a high-pressure blast through the constricted orifice formed between the nozzle-bore 44 and the upper end of the capillary 56 causing the flame to taper down to an intensely hot conical blast and also creating a Venturi-effect directly upon the capillary 56. Thereupon, a suitable quantity of the liquid sample is placed in a carefully cleaned beaker 24 and the latter set into the tray 23. Thereupon the tray-bar 21 is pulled upwardly and forwardly elevating the beaker 24 so that the intake end of the capillary 56 will dip down into the liquid. By rotating the shaft 25 and turning the locking blade 27 into upright position the tray-bar 21 is held in elevated position as shown in Figure 2. The suction capillary 56 draws the liquid sample upwardly in a constant flow. As the liquid leaves the upper end of the capillary 56 it is immediately atomized or volatilized and projected into the interior of the flame for immediate ionization. It should be noted that the volatilization and ionization of the solution are practically simultaneous.

As the sample is ionized in the flame, a characteristic color will be imparted thereto, such as yellow for sodium, red for lithium and so on. Furthermore, the degree of intensity of coloration is directly proportional to the percentage of such cation in the sample. This degree of color intensity produces a voltage emission by the photo-cells 12 and the circuit balanced to measure the output voltage emitted responsive to such color intensity. The knobs 10, 11, are turned to increase or decrease the balancing voltage until the indicator 8 shows a condition of exact circuit balance whereupon the voltage may be read on the galvanometer 9. Where, as is usually the case, the solution contains more than one cation suitable filters 15 may be employed to screen out all other wave length of light other than the one associated with the particular cation for which the analysis is being made. If results as to other cations are desired the filters 15 may be changed successively to transmit, and permit analysis for, one characteristic wave length at a time. During analytical procedure the pressures of entering gases must be held constant within very close limits.

A minute amount of a non-ionic wetting agent, such as polyalkylene ether of a partial lauric acid, should preferably be added to the sample to facilitate passage up the capillary 56. It has been found that such substances when added to all samples in precisely the same amount do not adversely effect the results of the herein described spectro-photometric method for quantitative analysis but aid in the maintenance of even flow of the sample upwardly through the liquid tube or capillary 56 at a uniform rate.

With apparatus of the present invention variations in ambient temperatures and atmospheric conditions have no adverse effect upon the analytical results. The burner of the present invention furthermore obviates all side-effect due to condensation of volatilized liquid, inasmuch as a

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constant amount of liquid per unit of time is delivered directly into the flame.

It should be understood that changes and modifications both in the methods as well as in the form, construction, arrangement, and combination of the several parts of the apparatus for spectro-photometric quantitative analysis may be made and substituted for those herein shown and described without departing from the nature and principle of the present invention.

Having thus described my invention, what I claim and desire to secure by Letters Patent is:

1. A method of spectro-photometric analysis of liquid which comprises generating a substantially colorless intensely hot flame, and injecting the liquid to be analyzed directly into the interior portion of the flame and at the base thereof in a high-pressure blast of oxygen to impart color thereto, imposing the light from the colored flame upon an electronic device capable of emitting a measurable electric impulse directly proportional to the light stimulus and measuring said electric impulse.

2. A method of spectro-photometric analysis of liquid which comprises generating a substantially colorless intensely hot flame, and injecting the liquid to be analyzed directly into the interior portion of the flame and at the base thereof in a high pressure blast of oxygen and atomizing the liquid at the point of introduction into the flame to impart color thereto, imposing the light from the colored flame upon an electronic device capable of emitting a measurable electric impulse directly proportional to the light stimulus and measuring said electric impulse.

3. In a spectro-photometric analytical apparatus, a flame generating burner comprising a chamber for receiving a combustible mixture of gases, said chamber being provided with a plurality of spaced burner-ports adapted to produce a hollow flame, injector means extending through said chamber and having a discharge nozzle opening into the interior portion of the hollow flame, said injector means being provided with a connection for receiving a high-pressure combustion-supporting gas, and a liquid intake tube disposed within and extending coaxially through the injector means, said tube terminating at one end adjacent to the discharge nozzle and at its other end projecting outwardly from the burner for insertion into a body of liquid to be analyzed.

4. In a spectro-photometric analytical apparatus, a flame generating burner comprising a water-jacketed chamber for receiving a combustible mixture of gases, said chamber being provided with a plurality of spaced burner-ports adapted to produce a hollow flame, Venturi-type injector means extending through said chamber and having a discharge nozzle opening into the interior portion of the hollow flame, said injector means being provided with a connection for receiving a high-pressure combustion-supporting gas, and a liquid intake tube disposed within and extending coaxially through the injector means, said tube terminating at one end adjacent to the discharge nozzle and at its other end projecting outwardly from the burner for insertion into a body of liquid to be analyzed.

5. In a spectro-photometric analytical apparatus, a flame generating burner comprising a chamber for receiving a combustible mixture of gases, said chamber being provided with a plurality of spaced burner-ports adapted to produce a hollow flame, injector means extending through said chamber and having a discharge nozzle

opening into the interior portion of the hollow flame, said injector means being provided with a connection for receiving a high-pressure combustion-supporting gas, and a vertical liquid intake tube disposed within and extending coaxially through the injector means, said tube terminating at one end adjacent to the discharge nozzle and at its other end projecting outwardly from the burner for insertion into a body of liquid to be analyzed.

6. In a spectro-photometric analytical apparatus, a flame generating burner comprising a chamber for receiving a combustible mixture of gases, said chamber being provided with a plurality of spaced burner-ports adapted to produce a hollow flame, injector means extending through said chamber and having a discharge nozzle opening into the interior portion of the hollow flame, said injector means being provided with a connection for receiving a high-pressure combustion-supporting gas, and a straight liquid intake tube disposed within and extending coaxially through the injector means, said tube terminating at one end adjacent to the discharge nozzle and at its other end projecting outwardly from the burner for insertion into a body of liquid to be analyzed.

7. In a spectro-photometric analytical apparatus, a flame generating burner comprising a chamber for receiving a combustible mixture of gases, said chamber being provided with a plurality of spaced burner-ports adapted to produce a hollow flame, injector means extending through said chamber and having a discharge nozzle opening into the interior portion of the hollow flame, said injector means being provided with a connection for receiving a high-pressure combustion-supporting gas, a straight liquid intake tube disposed within and extending coaxially through the injector means, said tube terminating at one end adjacent to the discharge nozzle and at its other end projecting outwardly from the lower end of the burner for insertion into a body of liquid to be analyzed, and elevator means located in downwardly spaced relation to the intake tube for shifting a sample-container toward and away from the intake tube.

8. In a spectro-photometric analytical apparatus, a flame generating burner comprising a chamber for receiving a combustible mixture of gases, said chamber being provided with a plurality of spaced burner-ports adapted to produce a hollow flame, injector means extending through said chamber and having a discharge nozzle opening into the interior portion of the hollow flame, said injector means being provided with a connection for receiving a high-pressure combustion-supporting gas, a straight liquid intake tube disposed within and extending coaxially through the injector means, said tube terminating at one end adjacent to the discharge nozzle and at its other end projecting outwardly from the lower end of the burner for insertion into a body of liquid to be analyzed, elevator means located in downwardly spaced relation to the intake tube for shifting a sample-container toward and away from the intake tube, and locking means for optional engagement with the elevator means to hold the latter in position wherein the intake tube dips into the liquid to be analyzed as it is held in the sample-container.

9. In a spectro-photometric analytical apparatus, a flame generating burner comprising a shell having opposite end walls providing a cylindrical chamber, one of said end walls being

provided with a plurality of spaced burner-ports adapted to produce a hollow flame concentric with said chamber, a nozzle tube extending axially through said chamber and having a discharge nozzle opening into the interior portion of the hollow flame, said nozzle tube being provided with a connection for receiving a high-pressure combustion-supporting gas, a liquid intake tube disposed within and extending coaxially through the injector means, said tube terminating at one end adjacent to the discharge nozzle and at its other end projecting outwardly from the burner for insertion into a body of liquid to be analyzed, and oxygen intake means provided at the lower end of said nozzle tube, whereby upon discharge through the nozzle the oxygen will cause the hollow flame to taper downwardly to form a conical blast.

10. In a spectro-photometric analytical apparatus, a flame generating burner comprising a cylindrical chamber for receiving a combustible mixture of gases, said chamber being provided in its upper end face adjacent its periphery with a plurality of annularly spaced burner-ports adapted to produce a hollow flame upon combustion of the gaseous mixture, a nozzle tube extending through said chamber in concentricity therewith and having a discharge nozzle opening into the interior portion of the hollow flame, said discharge nozzle having a downwardly depressed conical upper face and a central bore which flares outwardly at its lower end, said nozzle tube being provided with a liquid intake tube in axial alignment therewith which projects at its lower end outwardly from the burner for insertion into a body of liquid to be analyzed, said liquid intake tube being disposed at its upper end within the bore of the discharge nozzle spacedly from the walls of said bore, and a source of oxygen under pressure, said nozzle tube being provided at its lower end with intake means from said source of oxygen, whereby upon upward flow of the oxygen through the nozzle tube an area of decreased pressure will be developed adjacent the upper end of the liquid intake tube by flow of the oxygen through the discharge nozzle causing the liquid to be ejected upwardly into the flame, said hollow flame tapering downwardly by discharge of the oxygen to form an intensely hot conical blast for volatilization and ionization of the ejected liquid.

11. In a spectro-photometric analytical apparatus, a flame generating burner comprising a cylindrical chamber for receiving a combustible mixture of gases, said chamber being provided in its upper face adjacent its periphery with a plurality of spaced burner-ports in concentricity with said chamber and adapted to produce a hollow flame, a nozzle tube extending through said chamber in axial alignment therewith and having a discharge nozzle opening into the interior portion of the hollow flame, said discharge nozzle having a downwardly depressed conical upper face and a central bore which flares outwardly at its lower end, said nozzle tube being provided with a liquid intake tube in concentricity therewith the upper end of which is disposed within the bore of the discharge nozzle in spaced relation to the walls thereof, the lower end of said liquid intake tube projecting outwardly from the burner for insertion into a body of liquid to be analyzed, elevator means for shifting a sample container toward and away from the intake tube, locking means for optional engagement with the elevator means to hold the latter in position

wherein the intake tube dips into the liquid to be analyzed as it is held in the sample container, a source of oxygen under high pressure, said nozzle tube being provided at its lower end with intake means connected to said source of oxygen, whereby upon upward flow of the oxygen through said nozzle tube and through said discharge nozzle an area of decreased pressure will be caused adjacent the upper end of the liquid intake tube for ejection of liquid into the flame for volatilization and ionization therein, said hollow flame tapering downwardly by attraction to the discharged oxygen to form a hot conical blast.

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