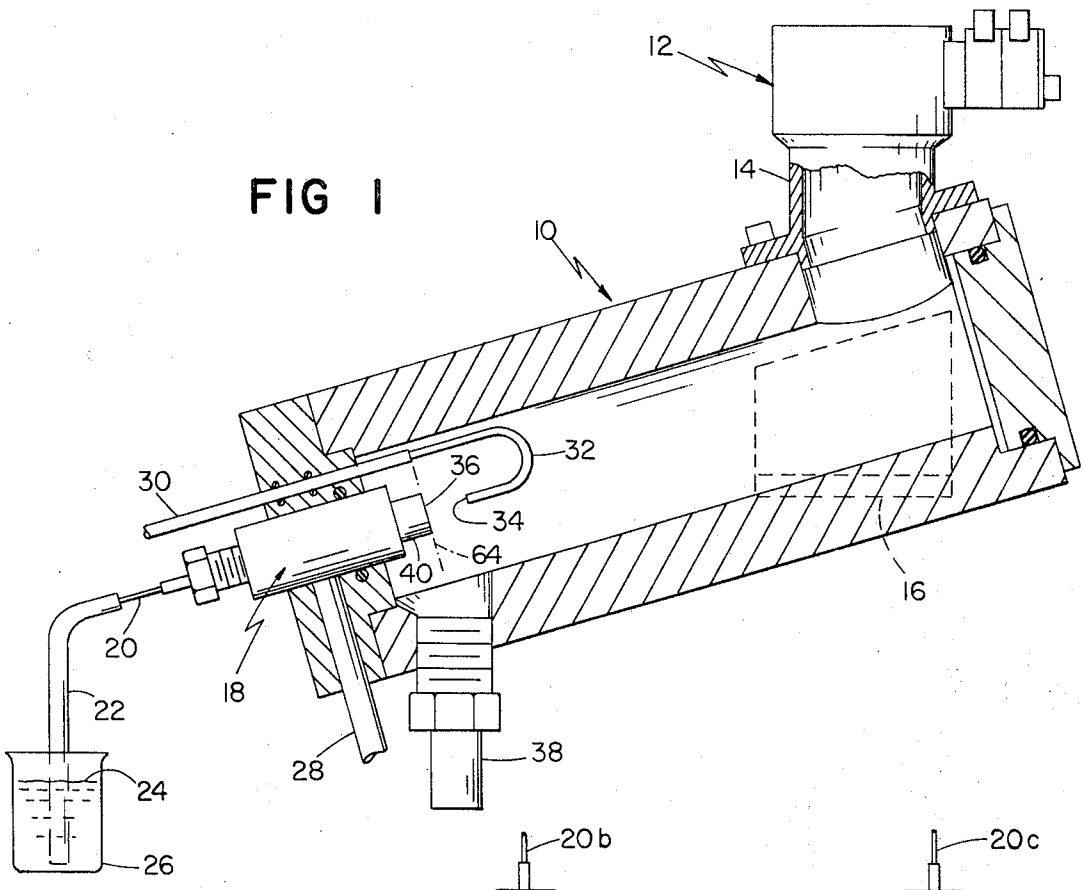
Aug. 25, 1970 E. A. BOLING ET AL 3,525,476
FLUID DIFFUSER WITH FLUID PRESSURE DISCHARGE MEANS
AND ATOMIZING OF MATERIAL IN HOLDER
Filed March 27, 1968 2 Sheets-Sheet 1

FIG 1



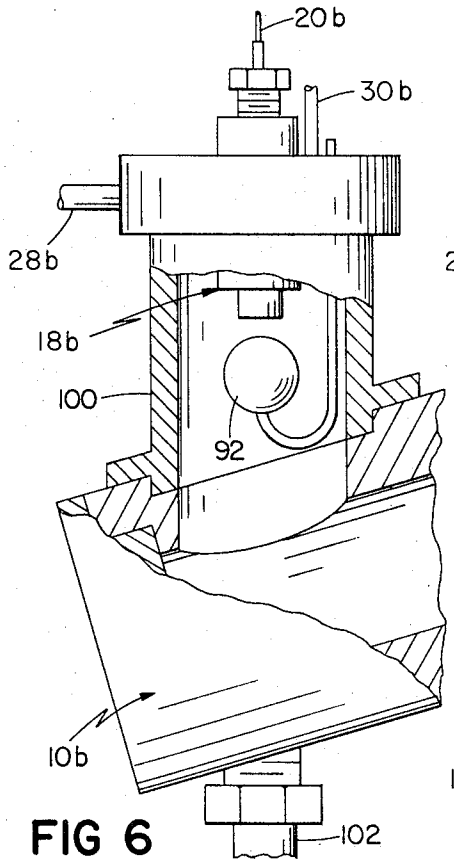


FIG 6

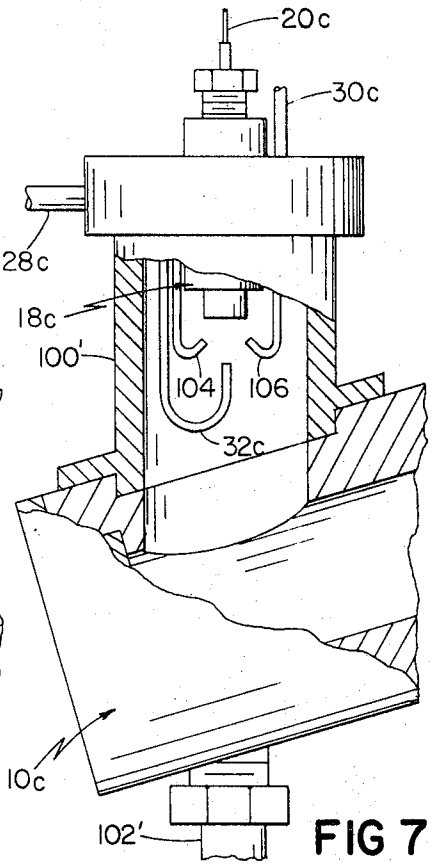
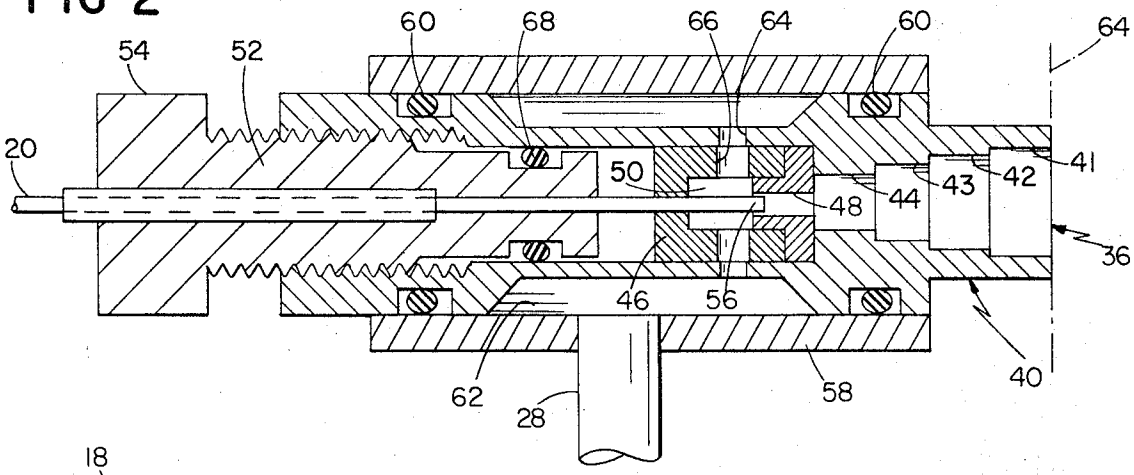


FIG 7

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FLUID DIFFUSER WITH FLUID PRESSURE DISCHARGE MEANS AND ATOMIZING OF MATERIAL IN HOLDER
Filed March 27, 1968 2 Sheets-Sheet 2

FIG 2



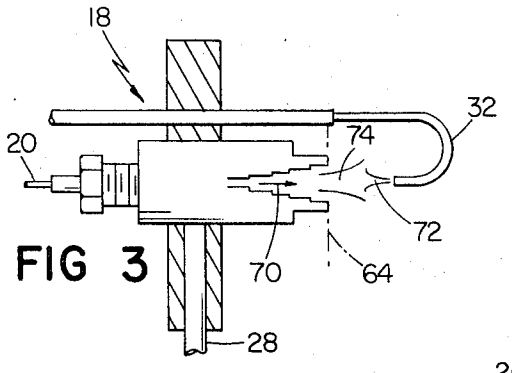


FIG 3

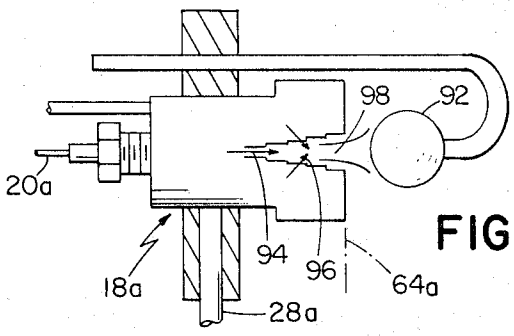


FIG 5

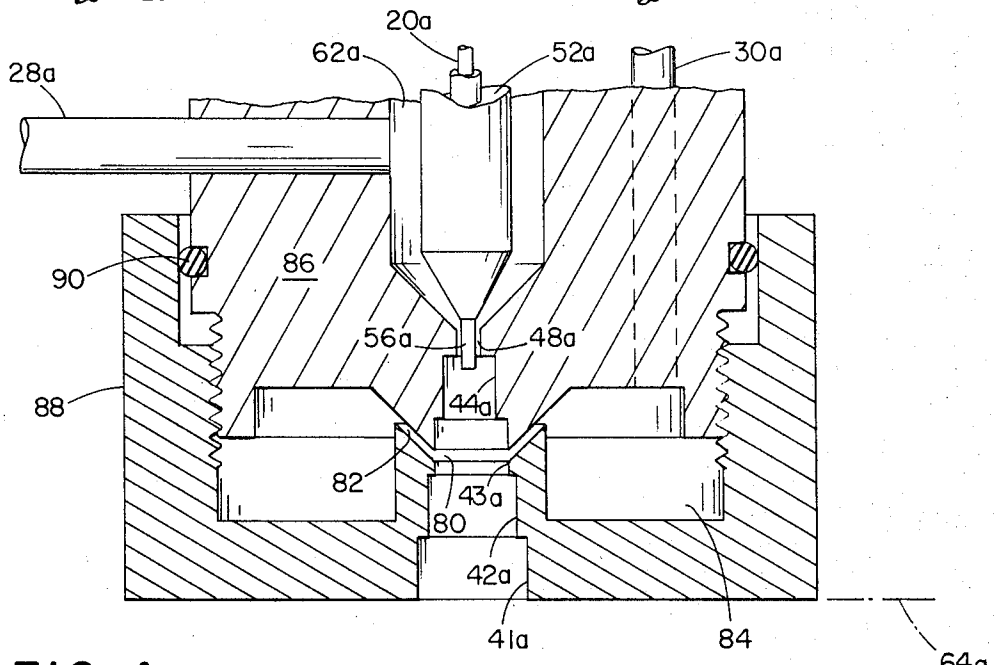


FIG 4



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3,525,476

FLUID DIFFUSER WITH FLUID PRESSURE DISCHARGE MEANS AND ATOMIZING OF MATERIAL IN HOLDER

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U.S. Cl. 239—338 8 Claims

ABSTRACT OF THE DISCLOSURE

A pneumatic nebulizer includes a plenum chamber, a capillary tube that extends through the plenum chamber and a nozzle section that has a series of five coaxial steps. The end of the capillary tube is disposed in the smallest step section of the nozzle section and air at a pressure of 40 p.s.i.g. aspirates a sample through the capillary tube into the nozzle to produce a primary stream of nebulized sample. In one embodiment a secondary stream of fuel and air is supplied in a counter stream from an 0.050 inch orifice spaced 0.375 inch from the outlet plane of the nozzle. This secondary stream impinges on the primary sample stream and further reduces the size of the nebulized sample particles. In a second embodiment, a secondary stream is provided from an annular orifice 0.020 inch in width which impinges on the primary stream from a 45° angle within the nozzle section. A baffle in the form of a 0.375 inch diameter ball is placed in front of the nozzle and the mixed streams impinge on that surface.

SUMMARY OF INVENTION

This invention relates to pneumatic nebulizers and more particularly to nebulizer systems especially although not exclusively useful with spectroanalytical systems of the atomic absorption and emission types.

In an analysis of a sample, uniform distribution of the sample in a carrier is desirable as output signal variation arises if the distribution is non-uniform. It is also desirable to increase the magnitude of output signal which may be increased by increasing the proportion of sample in the carrier.

In an atomic absorption type of spectroanalytical instrument, for example, a light beam is passed through a population of atoms representative of the particular sample being analyzed, the wavelength of the light beam corresponding to an atomic resonance line of the particular element under analysis. The atomic population for use in this analysis is normally produced by nebulizing a liquid containing sample into a flame where the molecules are broken down into atoms. Atoms of the element of interest absorb light of its corresponding wavelength, and the ratio of the intensity of the resonance line light beam passed through the flame to that of an equivalent light beam not passed through the flame under ideal circumstances closely follows Beer's law. However, atomic absorption analyses frequently deviate from Beer's law as the concentration of atoms in the flame is a function not only of the concentration of the element in the liquid sample but also of a variety of other factors including: (1) the rate at which the nebulizer converts sample containing liquid into droplets small enough to be swept through a spray chamber and into the flame (termed hereafter the "fog rate"); (2) the mean size of droplets and the range of droplets sizes in the fog; (3) characteristics of the fuel and oxidant employed; (4) the fuel oxidant ratio; (5) the ratio of fog to fuel and oxidant; (6) the chemical matrix of the sample. While the present

invention was developed primarily for nebulization of samples for spectroanalysis, the invention also has utility in other fields that involve atomization of materials.

It is a particular object of this invention to provide an improved nebulizer for use with spectroanalytical instruments.

Another object of the invention is to provide a novel and improved nebulizer that has an increased fog rate.

Still another object of the invention is to provide a nebulizer which increases the efficiency of conversion of aspirated sample to fog.

A further object of the invention is to provide a nebulizer which substantially increases the rate at which fog can be generated and concurrently reduces the size of fog droplets to a degree that the fog stream will not wet plane surfaces placed in the stream path.

A further object of the invention is to reduce the variations in the flame employed in a spectroanalytical instrument.

Another object of the invention is to provide a novel and improved nebulizer-spray chamber burner combination in which the instrument variations (noise) due to variations in the flame are significantly reduced.

Another object of the invention is to provide an improved source of fuel oxidant and sample which provides improved mixing of the constituents supplied to the burner structure.

Still another object of the invention is to materially increase the sensitivity of and stability of spectroanalytical instruments that employ flames.

In accordance with the invention there is provided a nebulizer that includes a nozzle structure having an aspirator orifice and a sample orifice disposed adjacent the aspirator orifice so that the sample may be aspirated into the nozzle structure by fluid flow through the aspirator orifice to form a primary stream. The nebulizer further includes an auxiliary orifice for directing a second fluid stream to impinge on the primary stream in an interaction zone in the immediate vicinity, that is within approximately one inch of the nebulizer nozzle. The interaction of the primary and secondary streams produces a fog having droplets of smaller dimension than the droplets in the aspirated primary stream. In nebulizers constructed in accordance with the invention, fog droplets of sufficiently small size such that the fog does not wet a plane surface placed in the flow path of the fog may be produced. The second fluid stream should impinge on the primary stream at a substantial angle, i.e., greater than about 15°, to the direction of flow of the primary stream; in one embodiment an angle of 45° is employed and in another particular embodiment an angle of 180° is employed. In a particular embodiment, the secondary stream is produced from an annular orifice located within the nebulizer nozzle so that the interaction zone is at least partly within the nebulizer nozzle itself. Further, it is preferred to employ a nebulizer nozzle formed in a series of steps of increasing cross-sectional area that are coaxial with one another and with the primary stream. In particular embodiments the use of a supplemental baffle structure fixedly positioned in front of the nebulizer nozzle directly in the path of the primary stream is desirable.

The invention is particularly useful in spectroanalytical apparatus in combination with a spray chamber in which a fog of sample material is transmitted to an analysis station. In an atomic absorption type of spectroanalytical instrument, the invention increases the amount of sample converted to fog, and reduces the size of the particles of the fog; thus increasing the sensitivity of the analysis both from the standpoint of increasing the amount of material available per unit time for analysis and reducing

instrument output signal noise due to variations in the flame.

Other objects, features and advantages of the invention will be seen as the following description of particular embodiments thereof progresses, in conjunction with the drawings, in which:

FIG. 1 is a diagrammatic sectional view of an atomizer-burner structure constructed in accordance with the invention for use in an atomic absorption spectrometer;

FIG. 2 is a sectional view of the nebulizer structure employed in the structure shown in FIG. 1;

FIG. 3 is a diagrammatic view of the flow patterns employed in the nebulizer shown in FIG. 2;

FIG. 4 is a sectional view of a second form of nebulizer constructed in accordance with the invention;

FIG. 5 is a diagrammatic view of the flow patterns produced by the nebulizer shown in FIG. 4;

FIG. 6 is a diagrammatic view of a second form of nebulizer-spray chamber system constructed in accordance with the invention; and

FIG. 7 is a diagrammatic view of still another form of nebulizer-spray chamber system constructed in accordance with the invention.

DESCRIPTION OF PARTICULAR EMBODIMENTS

With reference to FIG. 1 spray chamber **10** has mounted on it a burner head **12** which is connected to the spray (premix) chamber **10** by means of conduit **14**. The chamber is supported by brackets **16**. At the end of the spray chamber **10** remote from the burner head **12** is secured an end cap that supports a nebulizer structure generally indicated at **18**. This nebulizer structure includes a capillary **20** to which is connected **22**, the end of which is disposed in a liquid sample **24** held in container **26**. Coupled to the nebulizer structure **18** is oxidant inlet conduit **28** and extending through the end cap is a fuel inlet conduit **30**. Conduit **30** is connected to tube **32** that has an outlet orifice **34** disposed in coaxial opposition to the outlet orifice **36** of the nebulizer. A drain **38** is provided in the base of spray chamber **10**.

Details of the nebulizer structure may be seen with reference to FIG. 2. The nebulizer body **10** includes a nozzle section that has a series of coaxial steps **41–44** dimensioned as indicated in the following table:

Step	Length	Diameter (inch)
41	7/64	0.180
42	7/64	0.156
43	3/32	0.128
44	1/8	0.099

Disposed within nebulizer body **40** is an insert **46** which defines a further coaxial nozzle step **48**, 0.035 inch in diameter and 0.093 inch long, and a plenum chamber **50**; and a sample tube holder **52** which supports the capillary tube **20**. Holder **52** is threadedly connected to body **40** so that rotation of the knurled head **54** adjusts the position of the end **56** of the capillary tube **20** relative to the entrance of the nozzle step **48**. A housing **58**, to which is secured oxidant entrance conduit **28**, is disposed over nebulizer body **40** and O-rings **60**, carried by the nebulized body **40**, provide a seal between the housing and the body to define a chamber **62** into which the oxidant is introduced. The body further includes passages **64** which are aligned with corresponding passages **66** in insert **46** to provide flow of oxidant into plenum chamber **50** and from that chamber through the narrow space between the end of capillary **20** and the entrance to passage **48**, the flow of oxidant past the end of the capillary aspirating sample from container **26** for flow into the nebulizer nozzle section. An O-ring **68** mounted on holder **52** provides a seal between holder **52** and body **40**.

Capillary tube **20** has an O.D. of 0.028 inch and an I.D. of 0.018 inch. The orifice **34** of the fuel supply tube **32** has a diameter of 0.050 inch. In this particular embodiment satisfactory operation has been obtained where orifice **34** is spaced over a range of positions from ¼ inch to 1 inch from the outlet plane **64** of the nebulizer nozzle, a preferred spacing being ⅜ inch.

In a particular operation, air at a pressure of 40 p.s.i.g. is supplied through inlet **28** to plenum chamber **50** to aspirate a sample from container **26** through capillary **20**. A fuel-air mixture of acetylene at a pressure in the order of 6–8 p.s.i.g. through a 0.036 inch throttling orifice and air at a pressure of 5 p.s.i.g. through a 0.032 inch throttling orifice is supplied through tube **32**, producing a flow interaction as diagrammatically indicated in FIG. 3. The sample-oxidant stream **70** from the primary nebulizer flares out slightly and the fuel-oxidant stream **72** from conduit **32** impinges directly on the sample stream, producing an interaction zone **74** closely adjacent the exit plane **74** of the nebulizer nozzle. (Auxiliary stream **72** should have substantial velocity. In this system, for example, satisfactory results were not obtained with a ⅛ inch conduit orifice **34**.) With this device, fluid samples are converted to fog, at rates up to 2.0 ml./min., which fog does not wet a large plane surface placed in the path of the fog stream. Aqueous samples containing copper in a concentration of 10 parts per million, nebulized with this apparatus, produce atomic absorption instrumental readings up to 1.6 absorbance units while the same copper solution nebulized with a conventional nebulizer structure (without the auxiliary stream from conduit **32**) was converted to fog at a rate 1.0 milliliter per minute or less and produced instrumental readings of 0.7 absorbance units maximum.

A second form of nebulizer structure is shown in FIG. 4. This nebulizer structure also employs a series of nozzle steps *41a–44a*. The base orifice **48***a* and capillary **20***a* are also of the same dimensions as the corresponding elements of the nebulizer shown in FIG. 2. An adjustable annular orifice **80** (a suitable width being 0.020 inch) communicates via conical passage **82** with plenum chamber **84**. Passage **82** is inclined at an angle of 45° to the nebulizer axis and is formed between threadedly connected nebulizer nozzle components **86** and **88** which are sealed by O-ring **90**. In addition, a baffle in the form of a ball **92**, 0.375 inch in diameter is located 0.1875 inch from plane **64***a* of the nebulizer nozzle.

In operation, oxygen supplied through orifice **48***a* aspirates a sample from container **26** through capillary **20***a* and produces a fog stream. Fuel (or a fuel oxidant mixture) is supplied to orifice **80** through passage **82**, producing a total air fuel ratio in the range of 8–1 to 12–1. With reference to the diagrammatic representation in FIG. 5, the primary flow from the nebulizer orifices **20***a* and **48***a* form stream **94** and the secondary flow from orifice **80** forms stream **96**. These two streams impinge on one another and produce an interaction zone **98** inside the nozzle (in an area of sub-atmospheric pressure). The interaction products from zone **98** impinge on baffle surface of ball **92** and the resultant fog produced then flows through the spray chamber to the burner head.

With this nebulizer arrangement, the baffle surface can be placed substantially closer to the exit of the nebulizer than if the secondary stream is not used and the nebulizer efficiency is greater, a larger fraction of the aspirated sample being converted to fog. Further, the mixing of the fuel, oxidant and sample is more thorough and the variation in the flame and its effect on the analysis is thus significantly reduced. For most analyses, the absorbance readings for a given sample are steadier and mode precise, thus enabling the analyses to be completed more quickly. Also instrumental variations (noise) due to variations in the flame are reduced, particularly in the deep ultraviolet. For example, near the resonance line for lead (2170 Angstroms) absorption of light by the flame becomes significant and that absorption is markedly affected by the fuel oxidant ratio in the flame. With the nebulizer shown in FIG. 4, the variation in output signal

base line in this region is one-quarter to one-half that obtained using a conventional nebulizer improving both the precision and the detection limit of elements with resonance lines in this region of the spectrum. A device of the type shown in FIG. 4 can reliably generate fog of reduced droplet size at a rate of 2 milliliters per minute, the fog stream not wetting a large plane surface placed in its path.

In certain applications the arrangement of nebulizer and spray chamber indicated in FIG. 6 is particularly advantageous. In that arrangement a nebulizer of configuration shown and described in connection with FIG. 4 is applied, which neutralizer is mounted vertically in a fog forming chamber **100** that has a drain section **102** in its base. The fog chamber **100** has, in its side wall, a connection to mixing chamber **10***b*. This configuration results in quieter overall operation of the nebulizer-spray chamber system and reduced analytical noise. Still another modification of nebulizer-spray chamber system is indicated in FIG. 7 in which a nebulizer of the type detailed in FIG. 2 is used, with supplemental secondary counter flow jets **104** and **106** directed at an angle of 120° to the primary flow from nebulizer **18***c*, the primary nebulizer stream being directed vertically downwardly in fog forming chamber **100'**, which chamber is connected to mixing chamber **10***c* via an aperture in its side wall.

While particular embodiments of the invention and modification thereof having been shown and described, other modifications will be apparent to those skilled in the art and therefore it is not intended that the invention be limited to the disclosed embodiments or to details thereof and departures may be made therefrom within the spirit and scope of the invention.

What is claimed is:

1. A nebulizer comprising a nozzle structure having an aspirator orifice, a sample orifice disposed adjacent said aspirator orifice so that a sample may be aspirated through said sample orifice by fluid flow through said aspirator orifice to form a primary stream, and an auxiliary orifice for directing a second fluid stream for flow directly counter to the flow of said primary stream to impinge on said primary stream in an interaction zone in the immediate vicinity of the nebulizer nozzle, the interaction of said primary and secondary streams producing a fog having droplets of smaller dimension than the droplets in said primary stream.

2. Apparatus for use in a spectroanalytical instrument comprising a burner structure, a spray chamber for supplying a fuel-oxidant mixture to said burner structure, and a nebulizer for furnishing said fuel-oxidant mixture in fog form to said spray chamber comprising a nozzle structure having an aspirator orifice, a sample orifice disposed adjacent said aspirator orifice so that a sample may be aspirated through said sample orifice by fluid flow through said aspirator orifice to form a primary stream, and an auxiliary orifice for directing a second fluid stream for flow directly counter to the flow of said primary stream to impinge on said primary stream in an interaction zone in the immediate vicinity of the nebulizer nozzle, the interaction of said primary and secondary streams producing a fog having droplets of smaller dimension than the droplets in said primary stream for flow through said spray chamber to said burner structure.

3. The apparatus as claimed in claim **2** and further including additional orifices for directing supplementary secondary fluid streams for impingement on said primary stream at said interaction zone.

4. Apparatus for use in a spectroanalytical instrument comprising a burner structure, a spray chamber for supplying a fuel-oxidant mixture to said burner structure, a fog forming chamber connected to said spray chamber, and a nebulizer for furnishing said fuel-oxidant mixture in fog form to said spray chamber comprising a nozzle structure having an aspirator orifice, a sample orifice disposed adjacent said aspirator orifice so that a sample may be aspirated through said sample orifice by fluid flow through said aspirator orifice to form a primary stream, said nebulizer nozzle being mounted on said fog forming chamber to direct said primary stream downwardly through said fog forming chamber and the fog flow from said fog forming chamber through spray chamber to said burner structure being generally horizontal, and an auxiliary orifice for directing a second fluid stream to impinge and on said primary stream in an interaction zone in the immediate vicinity of the nebulizer nozzle, the interaction of said primary and secondary streams producing a fog having droplets of smaller dimension than the droplets in said primary stream for flow through said spray chamber to said burner structure.

5. The apparatus as claimed in claim **4** wherein said nebulizer nozzle includes a nozzle outlet defining wall that includes a series of steps defining a series of openings of increasing cross-sectional area coaxial with said primary stream, said auxiliary orifice directs said secondary stream for impingement on said primary stream at said interaction zone at an angle greater than 15° to the direction of flow of said primary stream, and said interaction zone is within one inch of said nebulizer nozzle.

6. The apparatus as claimed in claim **5** wherein said auxiliary orifice directs said secondary stream for flow directly counter to the flow of said primary stream.

7. The apparatus as claimed in claim **6** and further including additional orifices for directing supplementary secondary fluid streams for impingement on said primary stream at said interaction zone.

8. The apparatus as claimed in claim **5** wherein said auxiliary orifice is an annular orifice formed within said nebulizer nozzle and further including a convex surface fixed in position in front of said nebulizer nozzle directly in the path of said primary stream.

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