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Sitek et al.

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[54] **COMBUSTION APPARATUS FOR ANALYTICAL INSTRUMENTS**

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[52] **U.S. Cl.** ..... **23/253 PC**; 23/292; 138/40

[51] **Int. Cl.**[2] **G01N 31/12**; F15D 1/00

[58] **Field of Search** ..... 23/230 PC, 253 PC, 292; 219/10.43; 55/386; 73/61.1 C; 210/198 C; 161/125, 139, 206; 138/40, 89, 109, 174

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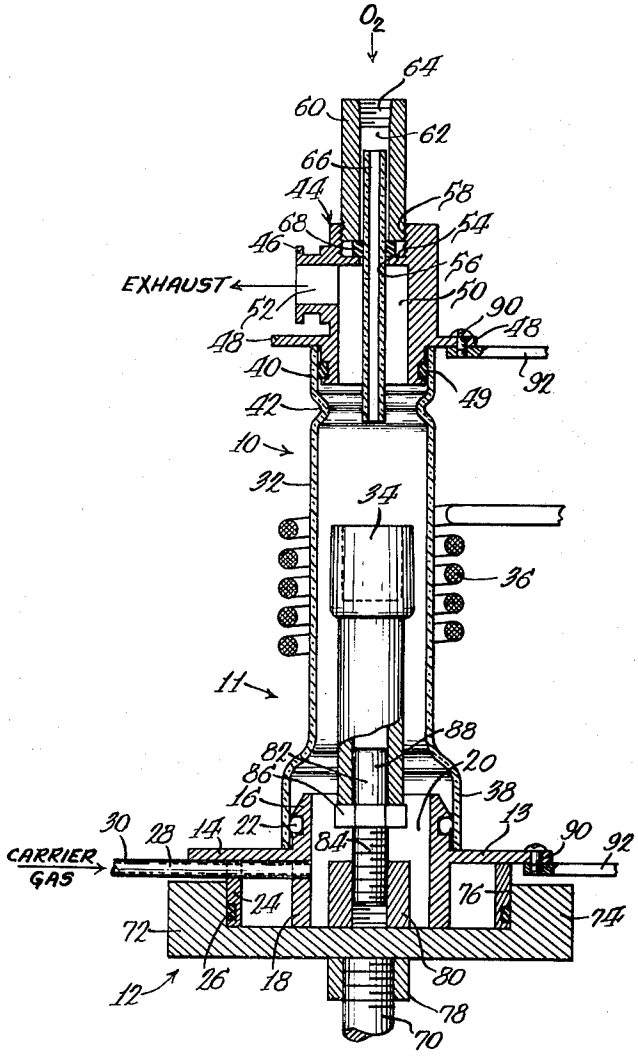
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[57] **ABSTRACT**

A combustion furnace for analytical apparatus in which the products of combustion of samples are to be transported in a gas stream for analysis wherein the admission of carrier gas is separate from the admission of combustion gas, resulting in a reduction of eddy currents of the gases within the furnace, so leading to a faster transfer of the component to be measured and to reduced purge and combustion time.

10 Claims, 1 Drawing Figure



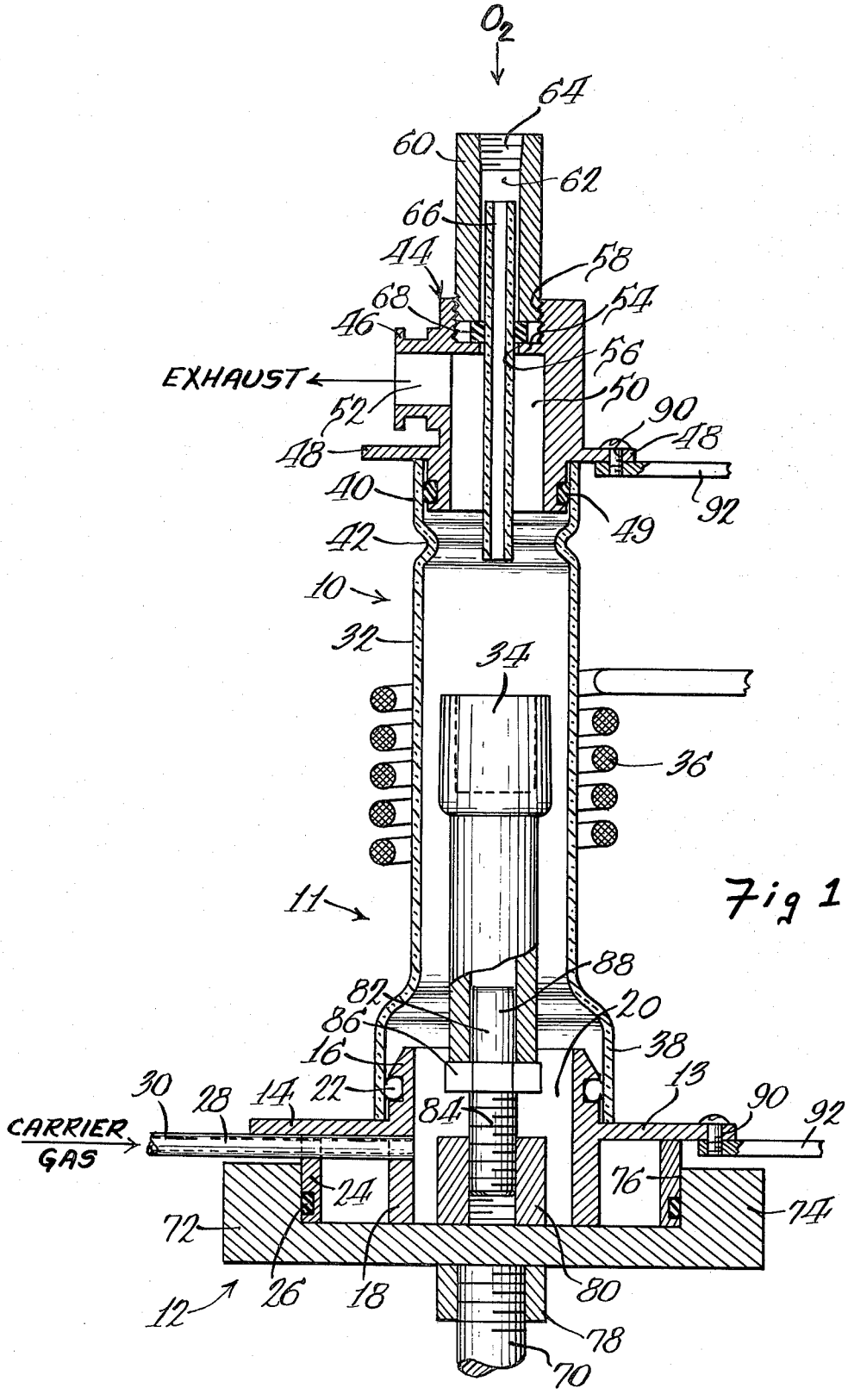

O2
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EXHAUST
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90
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CARRIER GAS
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74
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Fig 1

COMBUSTION APPARATUS FOR ANALYTICAL INSTRUMENTS

BACKGROUND OF THE INVENTION

In U.S. Pat. No. **2,809,100** dated Oct. **8, 1957,** to G. J. Krasl, entitled "Combustion Analyzer", there is shown a glass capsule adapted to fit within an induction coil and to fit over a crucible supported on a post which positions the crucible within the field of the combustion coil. The bottom of the capsule is open for the vertical elevation of the crucible and post thereinto and is closed by a base plate carrying the post brought up into contact with the bottom of the capsule. The base plate has a passage therethrough for the downward flow of exhaust gases from the capsule. A tube extends centrally down through the top of the capsule into a position to direct a jet of oxygen into the crucible when it is in its elevated position. In use, a sample in a crucible is elevated into the capsule and heated by the induction coil. At the same time, oxygen is directed down onto the sample to support its combustion and the products of combustion together with a carrier stream of excess oxygen from the jet are carried out through the bottom of the capsule for electronic analysis.

This apparatus does not function as well as it might for several reasons. There is a dead space above the crucible between the jet tube and the wall of the capsule in which eddy currents will exist, delaying the total transfer of the material to be analyzed and leading to a trailing of the combustion gases which causes a tailing of the analysis curve. Optimum readout is derived from a total transfer of the product to be analyzed within as short a time period as possible, or in other words, an abrupt rise in the transfer curve to a high peak and an abrupt decline or decay thereafter. Eddying of the carrier gas extends the rise time, reduces the peak, and causes the decay curve to tail off gradually. The same eddy currents cause a depositing of metal on the walls of the capsule which, in time, couple to the field of the induction furnace and destroy the capsule.

By virtue of the single inlet port for the gas which serves both a combustion and carrier function which is directed straight into the crucible, the velocity of gas flow is desirably limited to avoid excessive reaction temperatures and excessive turbulence. Under conditions of a high content of combustible material, the combustion process can absorb the whole input of combustion gas and create a below-atmospheric pressure condition within the capsule. This necessarily extends the analysis time because the transportation of the unknown must await the completion of the combustion. Furthermore, in analytical procedures involving such sensors as conductivity cells in bridge configurations, it is necessary to have a constant flow of gas through the cell because these measurements are flow sensitive. The total absorption of the gas during combustion results in a flow change or even a backflow of the gas stream.

SUMMARY OF THE INVENTION

The present invention is directed to a combustion apparatus lacking blind corners or ends wherein eddy currents might develop. The apparatus is characterized by two gas inlet ports, one intended essentially for combustion and the second intended essentially for transport whereby a continuous flow may be obtained through the apparatus. By operating the device above

ambient pressure, the combustion process only decreases the pressure and a constant flow still exists to satisfy the measurement criteria. The device likewise includes provision for positioning the combustion gas inlet whereby optimum combustion circumstances can be provided. The device may operate continuously above or below ambient pressure if constant flow is not necessary. The device is easily cleaned and therefore less subject to damage from sculling, and the destructible element of the combination, the jacket, is inexpensive and easily replaced. Other advantages of this invention will appear in the course of the description which follows.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a central vertical section through an induction furnace embodying the present invention.

DESCRIPTION OF A PREFERRED EMBODIMENT

The furnace **10** consists of a stationary furnace proper **11** and a vertically reciprocable crucible elevator **12.** The stationary furnace **11** includes a base **13** which consists of a flat, annular plate **14** having upper and lower flanges **16** and **18** about the central aperture **20** thereof. The upper flange is externally grooved for the reception of an O-ring **22.** The under side of the plate also has a downwardly extending outer flange **24** thereabout which likewise is grooved on its exterior surface for the reception of an O-ring **26.** A carrier gas inlet tube **28** opens into and extends from the central aperture **20** through the lower flanges **18** and **24** and terminates in a free end **30** well beyond the plate **14** and the confines of the furnace itself.

A Vycor^{R} or similar high silica glass or fused silica combustion tube **32** is mounted on the base **13.** The combustion tube is of relatively large diameter so as to contain comfortably the crucible **34** leaving ample gas flow space thereabout. The tube fits in spaced relation within the induction coil **36.** The tube is belled out at its lower end **38** to fit over the upper flange **16** of the base **13** to rest on its bottom edge against the annular plate **14** and to engage the O-ring **22** in sealed relation. Adjacent its upper end **40,** the tube has a circumferential restriction **42** therein for purposes to be described later.

The tube mounts at its upper end a top fitting **44.** This top fitting is a generally cylindrical member with a side arm **46** and an integral flange **48** thereabout below the side arm **46.** The fitting is externally grooved below the flange **48** for the reception of an O-ring **49.** A large diameter central bore **50** extends from the bottom of the fitting upwardly to a point adjacent the top of the fitting, and the side arm **46** has a bore **52** therein intersecting the axial bore **50.** The axial bore **50** terminates at an upper transverse wall **54** having a small diameter axial passage **56** therethrough. A threaded hole **58** extends from the top end of the top fitting down to the wall **54.** A combustion gas adaptor **60** is threaded into the hole **58.** The adapter **60** is a simple tubular member having exterior threads at its lower end for engagement with the threaded bore **58** and a central longitudinal passage **62** therethrough and internally threaded as at **64** at its upper end for the reception of an oxygen supply fitting (not shown).

The internal bore **62** loosely receives a length of quartz tubing **66** constituting an oxygen lance. The tubing has an annular rubber bushing **68** fitted on the exte-

rior thereof. The bushing is slidable along the tubing for adjustment of the lance position. The bushing is adjusted on the lance to obtain a proper positioning of the lance for optimum performance with respect to the crucible **34**, and the combustion gas adaptor is screwed down on the bushing **68** to compress it against the wall **54** and bulge it against the lance **66** to effect a seal and hold the lance securely.

The elevator **12** is mounted on the top end of a vertically reciprocable rod **70**. The elevating mechanism may be identical with that described in the above-mentioned patent to Krasl. The elevator consists of a shallow dish **72** having a rim **74** with a vertical inside wall **76** beveled at its top edge proportioned to telescope over the outer flange **24** of the furnace base **13** in sealing relation with the O-ring **26**. The dish may have a central internally threaded downward sleeve **78** whereby the dish is mounted to the post **70** and an internally threaded central sleeve **80** extending upward from the floor of the dish **72**. The sleeve **80** receives a stud **82** having a lower threaded end **84**, a hexhead **86**, and a cylindrical plug **88** upward of the head **86**. A tubular ceramic crucible support is carried on the plug **88** and mounts the crucible **34** at its upper end. The adjustment capability of the stud **82** is, of course, to position the crucible optimally within the induction coil **36**.

The plate **14** of the base **13** and the flange **48** of top fitting **44** may be perforated as at **90** for the attachment of the furnace to brackets **92** whereby the furnace may be mounted to a cabinet or other element of the analytical apparatus.

From the foregoing, it will be appreciated that the assigned advantages of this invention have been well met. The provision for two gas inlets makes possible the continuous purge, avoidance of dead spaces, and avoidance of below-ambient pressure with combustion. The provision of the gas passages in the end fittings makes possible a low cost, simple tube and provides for a direct end-to-end passage of the gas. Gas flow through the apparatus may be continuous, and this immediate transfer, together with reduced purge time, appreciably shortens analysis time.

The upward flow of heated gases from the sample combustion, in the absence of the constriction **42** in the tube **32**, exposes the O-ring **49** to excessive temperatures and hot dust due to the direction of flow of the exhaust stream, subjecting it to damage and possible failure, which, of course, cannot be tolerated as leading to erroneous results. It has been found that the constriction **42** effectually shields the O-ring seal from damage by screening the seal from radiation from the burning sample and by deflecting the hot carrier gas stream with entrained products of combustion into the top fitting **44**.

We claim:

1. A combustion furnace for analytical apparatus comprising an induction coil with a vertically extending axis, a combustion tube mounted within said coil, an upper closure member for sealably coupling to an upper end of said combustion tube, said upper closure member including an exhaust gas passage therethrough, and a separate aperture extending through said upper closure member along the vertical axis thereof, a lower closure member for sealably engaging the lower end of said combustion tube, said lower closure member including a passageway extending therethrough to define a carrier gas inlet communicating with the interior of said combustion tube and crucible supporting means associated with said lower closure member, said lower closure member movable between a first position sealing said lower end of said combustion tube and supporting a crucible within the field of said coil and a second position displaced therefrom, a combustion gas lance, and means in said upper closure members for securing said lance in said separate aperture for optimal delivery of combustion gas into said crucible within said field.

2. The combination as claimed in claim **1** wherein said exhaust gas passage of said upper closure member extends vertically through a bottom surface of said upper closure member to communicate with the interior of said combustion tube and includes a radially extending portion extending through a side of said upper closure member for providing an exhaust port for removal of combustion gases from said combustion tube.

3. The combination as claimed in claim **2** wherein said upper closure member includes a sealing member extending around the exterior of the lower periphery thereof to engage an inner surface of said combustion tube for sealing said upper closure member to said combustion tube.

4. The combination as claimed in claim **3** wherein said combustion tube is a cylindrical sleeve having a circumferential constriction formed at its upper end and immediately below said sealing member and extending inwardly sufficiently to shield said sealing member from combustion by-products.

5. A combustion furnace for analytical apparatus comprising an induction coil with a vertical axis, a vertical combustion tube within said coil, a base closure member mounted against the bottom end of said tube and having passage defining means for delivering gas into said tube, a vertically reciprocable elevator movable between a position of sealing against said base member and supporting a crucible within the field of said coil and a position downwardly displaced therefrom, a top fitting closing the top end of said combustion tube, means defining a second gas passage therethrough, and an oxygen lance mounted in said top fitting for delivering combustion gas into said crucible.

6. The combination as claimed in claim **5** wherein said oxygen lance is adjustably mounted in said top fitting for adjustment toward and away from said crucible.

7. The combination as claimed in claim **5** wherein said lance is a high silica tube and said top fitting telescopes within the top end of said combustion tube, said top fitting having a sealing ring thereabout in the telescoped portion thereof to seal against the interior surface of said combustion tube, said tube having a circumferential constriction extending inwardly immediately below said top fitting a distance sufficient for shielding said sealing ring from combustion by-products.

8. For use in a furnace for heating specimens to their combustion temperature wherein the specimens are placed in a crucible positioned in a combustion tube with sealed end closures, and means extending through the end closures to provide a flow of gas through the combustion tube to carry combustion gases from the combustion tube, an improved combustion tube comprising:

a continuously extending cylindrical body having a first open end the interior wall surface of which de-

fines an interior cylindrical sealing surface immediately adjacent said first open end and a circumferential restriction formed inwardly in said cylindrical body and spaced from said first end to shield said sealing surface from combustion by-products, said combustion tube including an enlarged open opposite end defining a cylindrical wall extending substantially parallel with said cylindrical body and defining a cylindrical interior sealing surface having a diameter greater than the diameter of said cylindrical body.

9. The combustion tube as defined in claim **8** wherein said combustion tube is made of high silica glass.

10. An induction furnace comprising:

a vertically extending induction coil;

a cylindrical combustion tube positioned within said induction coil and including an open upper end, an enlarged cylindrical lower open end and a circumferential restriction spaced from said open upper end;

an upper closure member for sealably fitting into said upper end of said combustion tube and including an axial opening through an end thereof for receiving an oxygen lance tube therethrough and further including a relatively large opening formed axially upwardly through a bottom surface of said upper closure member and communicating with a radially extending port for exhausting combustion and carrier gases from said combustion tube, said upper closure member including a peripheral sealing member positioned adjacent said restriction of said combustion tube; and

a lower closure member including means for supporting a crucible within the induction field of said induction coil when said lower closure member is sealably fitted in the enlarged lower end of said combustion tube, said lower closure member including an axially extending aperture opening at the top surface of said lower closure member and means for introducing a carrier gas to said last named aperture to provide a flow of gas upwardly through said combustion tube.

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