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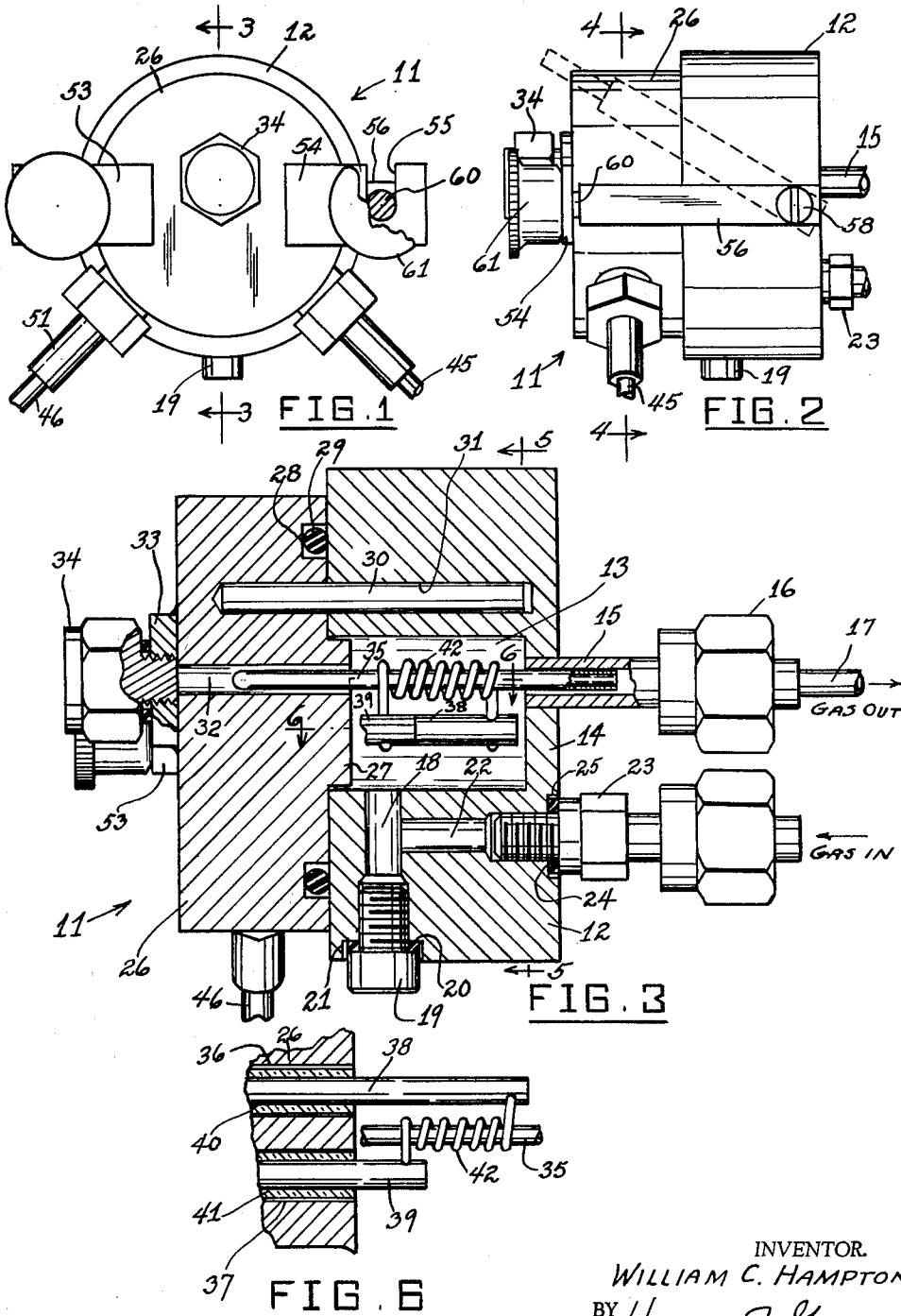
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3,186,801

PYROLYZER ASSEMBLY

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2 Sheets-Sheet 1



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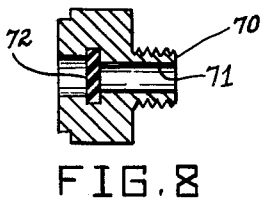
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PYROLYZER ASSEMBLY

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7 Claims. (Cl. 23-253)

This invention relates to apparatus for analyzing materials, and more particularly to a pyrolyzer chamber for vaporizing a sample of material and for introducing the vaporized sample into the gas flow stream of a gas chromatography apparatus or a mass spectrometer.

A main object of the invention is to provide a novel and improved pyrolyzer chamber into which a sample of liquid or solid may be easily introduced, in which the heater element will not be contaminated by residue, and in which the residue can be further analyzed, if so desired.

A further object of the invention is to provide an improved pyrolyzer chamber assembly which is very simple in construction, which is easy to load, and which is very compact in size.

A still further object of the invention is to provide an improved pyrolyzer chamber which is easy to assemble, which is adequately sealed, and which is mechanically rugged.

A still further object of the invention is to provide an improved pyrolyzer chamber wherein the heating element can be quickly elevated in temperature, wherein the heating element has high mechanical strength, even at high temperatures, and wherein liquid or solid samples may be efficiently vaporized to introduce them into the gas flow stream of a gas chromatography or similar apparatus without the risk of contaminating the heating element or other parts of the chamber with residue.

A still further object of the invention is to provide an improved pyrolyzer chamber wherein a sample may be loaded into the chamber either in a supporting container, or, alternatively, by syringe injection, without the necessity of disassembling the chamber or of disconnecting the chamber from the gas chromatography or similar apparatus with which it is used.

A still further object of the invention is to provide an improved pyrolyzer chamber assembly which can be quickly and easily opened up for internal examination or cleaning, or for replacement of its sealing gasket.

Further objects and advantages of the invention will become apparent from the following description and claims, and from the accompanying drawings, wherein:

FIGURE 1 is a front elevational view, with a part broken away, of an improved pyrolyzer chamber assembly constructed in accordance with the present invention.

FIGURE 2 is a side elevational view of the pyrolyzer chamber assembly of FIGURE 1.

FIGURE 3 is an enlarged longitudinal vertical cross-sectional view of the assembly, taken substantially on the line 3-3 of FIGURE 1.

FIGURE 4 is an enlarged transverse vertical cross-sectional view taken substantially on the line 4-4 of FIGURE 2.

FIGURE 5 is a transverse vertical cross-sectional view taken on the line 5-5 of FIGURE 3.

FIGURE 6 is a fragmentary horizontal cross-sectional view taken on the line 6-6 of FIGURE 3.

FIGURE 7 is an enlarged fragmentary horizontal cross-sectional view taken substantially on the line 7-7 of FIGURE 5.

FIGURE 8 is a longitudinal vertical cross-sectional view taken through a modified form of closure plug which may be used with the pyrolyzer chamber of FIGURES 1 to 7 for syringe injection of a sample to be vaporized.

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Referring to the drawings, 11 generally designates an improved pyrolyzer assembly constructed according to the present invention. The assembly 11 comprises a generally cylindrical rear block 12 formed with an axial cylindrical recess or cavity 13. Secured in the rear wall 14 of said cavity and communicating therewith is the gas outlet conduit 15, which extends parallel to the axis of the block 12. Connected to conduit 15 by a conventional detachable fitting 16 is the gas outlet tube 17, which leads to a gas chromatography column, or the like.

Block 12 is formed with a radially extending passage 18 communicating with cavity 13, the outer end of passage 18 being normally sealed by a removable threaded, headed plug 19, with an annular sealing gasket 20 of resilient deformable material provided between the head of the plug and a counterbore 21 receiving said head. The block 12 is further formed with a longitudinal passage 22 communicating with passage 18. A gas inlet fitting 23 is threadedly engaged in the block in communication with the passage 22, said inlet fitting being adapted to be connected to a suitable source of carrier gas. A resilient deformable sealing gasket 24 is interposed between the head portion of fitting 23 and a counterbore 25 shaped to receive said gasket, as shown in FIGURE 3.

The fitting 23 and the plug 19 are mechanically interchangeable, so that, if so desired, the gas inlet fitting 23 may be connected radially to the block in place of plug 19, and said plug may be threaded into the end of the block to seal off the end of passage 22.

Designated at 26 is generally cylindrical front block formed with a central circular boss 27 engageable in the end of cavity 13. Block 26 is formed with an annular groove 28 in which is positioned an annular resilient deformable sealing ring 29 sealingly engageable with the front surface of rear block 12. Secured to front block 26 is a guide pin 30 extending parallel to the axis of the block and being slidably engageable in a similarly directed longitudinal bore 31 formed in block 12. Front block 26 is formed with a longitudinal loading bore 32, and the pin 30 and bore 31 are located so that bore 32 will be accurately aligned with the bore of gas outlet conduit 15 when the pyrolyzer chamber is assembled.

Rigidly secured to the front wall of block 26 coaxially with bore 32 is an internally threaded nut 33 in which is threadedly engaged a removable closure plug 34. Closure plug 34 may be removed to allow an elongated slender tubular glass sample container 35 to be inserted in the chamber with the open end of the container received in the gas outlet conduit 15, as shown in FIGURE 3.

Block 26 is formed with the parallel spaced longitudinal bores 36 and 37 spaced below and symmetrically located on opposite sides of bore 32. Secured in said bores 36 and 37 are respective longitudinally extending tubular metal electrodes 38 and 39, said electrodes being provided with ceramic insulating sleeves 40 and 41, of porcelain, or the like. A helically wound heating coil 42 of platinum tubing, or other suitable metal tubing, has its ends electrically connected and supportingly secured to the inner ends of the respective tubular electrodes 38 and 39, the heating coil being supported in axial alignment with bore 32 and the bore of conduit 15, as shown in FIGURE 3, so as to receive the sample tube 35 therein.

It has been found that by using a heating coil of tubular rather than solid material, the mechanical strength of the heating coil is relatively high, as compared with its mass, and the temperature of the coil may be raised much more rapidly, with a given applied heating current, than with a coil of solid material having the same cross-sectional metallic conducting area.

Block 26 is formed with the respective outwardly di-

rected bores 43 and 44 communicating with the bores 37 and 36 through which electrical connections are made between current supply cables 45 and 46 and the electrode members 38 and 39. Thus, the conductor 47 of cable 46 is connected to the end of the axial conductor 48 having a flanged insulating collar 49 received in bore 43 and clamped therein by a threaded bushing member 50 engaged in block 26, as shown in FIGURE 4, the insulating sleeving 51 of cable 46 extending up to the flange 52 of collar 49. The inner end of conductor 48 is secured to the end of electrode member 39, providing an electrical connection between conductor 47 and said member 49. Similar means are provided in bore 44 to electrically connect the conductor of cable 45 to the remaining electrode member 38.

Respective diametrically opposed outwardly projecting locking plates 53 and 54 are rigidly secured to the front face of block 26, the outer portion of each of said plates being formed with an open-ended vertical slot 55, as shown in FIGURE 1. Respective fastening bars 56 and 57 are pivotally secured at 58 and 59 to the rear side portions of block 12. The fastening bars are provided at their forward ends with threaded stud portions 60 engageable in the slots 55 and receiving thereon internally threaded locking caps 61, which, when tightened, clamp front block 26 securely to rear block 12, with gasket 29 compressed in groove 28 to seal the connection between the blocks.

The blocks 26 and 12 may be readily separated, for example, to provide access to cavity 13 for cleaning or for removal or replacement of parts therein, such as the heating coil, by loosening the caps 61 and swinging the bars 56 and 57 upwardly, as shown in dotted view in FIGURE 2, whereby the blocks may be moved axially away from each other and separated.

In using the apparatus, the glass sample tube 35, containing a sample of liquid or solid material to be vaporized, is inserted through bore 32 and heater coil 42 into the bore of conduit 15 with its open end facing conduit 15, as shown in FIGURE 3. The closure plug 34 is then screwed into nut 33, and the heating current is applied to the coil 42 through the cables 45 and 46. After the sample material has been vaporized and chromatographically analyzed, the tube 35 may be removed, and, if so desired, the residue therein may be also analyzed.

Instead of employing a container 35 of sample material to be analyzed, the sample material may be injected directly into the pyrolyzer chamber. For this procedure, a cover plug 70, such as shown in FIGURE 8, is employed in substitution for the closure plug 34. Plug 70 is provided with an axial bore 71 in which is secured a silicone rubber disc 72 through which a syringe needle may easily penetrate. The sample material is thus injected into the pyrolyzer chamber by employing a syringe whose needle is forced through the disc 72, allowing the material to be injected directly inside the heating coil 42. The disc 72 is retained with sufficient tightness that the puncture will close and reseal after the syringe needle is withdrawn therefrom.

While certain specific embodiments of an improved pyrolyzer assembly have been disclosed in the foregoing description, it will be understood that various modifications within the spirit of the invention may occur to those skilled in the art. Therefore it is intended that no limitations be placed on the invention except as defined by the scope of the appended claims.

What is claimed is:

1. A pyrolyzer for gas chromatography comprising a chamber, a gas outlet conduit connected to one wall of said chamber, said chamber having an opposite wall formed with a loading passage longitudinally aligned with said gas outlet conduit, a sample tube slidably supported in said loading passage and having an open end portion received in said outlet conduit, closure means sealingly disposed in said loading passage, a gas inlet conduit communicatively connected to said chamber at a location spaced from the loading passage and the outlet conduit connection, and an electrical heating coil mounted in said chamber between and in axial alignment with said loading passage and gas outlet conduit and surrounding the sample tube.

2. A pyrolyzer for gas chromatography comprising a chamber, a gas outlet conduit connected to one wall of said chamber, said chamber having an opposite wall formed with a loading passage longitudinally aligned with said gas outlet conduit, a sample tube slidable supported in said loading passage and having an open end portion received in said outlet conduit, closure means sealingly disposed in said loading passage, a gas inlet conduit communicatively connected to said chamber at a location spaced from the loading passage and the outlet conduit connection, a pair of spaced electrodes mounted in said opposite wall and extending into said chamber, said electrodes being substantially parallel to said loading passage, and an electrical heating coil secured on and electrically connected between said electrodes between and in axial alignment with said loading passage and gas outlet conduit and surrounding the sample tube.

3. The structure of claim 1, and wherein said heating coil comprises a helically wound coil of tubular metal.

4. The structure of claim 2, and wherein said heating coil comprises tubular material formed into a helix with the ends of the helix secured to the respective electrodes.

5. The structure of claim 1, and wherein said closure means comprises a removable plug threadedly engaged in the outer end of the loading passage.

6. The structure of claim 1, and wherein said chamber comprises a front block containing the loading passage and a rear block formed with a cavity and having the gas outlet conduit connected thereto, and means to detachably and sealingly secure said front block to said rear block to cover said cavity.

7. The structure of claim 6, and wherein the means to detachably and sealingly secure the front block to the rear block comprises respectively outwardly extending opposite locking plates secured to said front block, said locking plates being formed with open-ended slots, respective fastening bars pivoted to opposite sides of said rear block and being lockingly engageable in said slots, and a resilient deformable sealing gasket interposed between the front and rear blocks and surrounding said cavity.

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