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Hubbard et al.

[11] **3,773,468**

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[54] **CENTRIFUGABLE EXTRACTION AND HOHOGENIZATION VESSEL**

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[52] **U.S. Cl.** **23/259,** 23/292

[51] **Int. Cl.** **G01n 1/04,** G01n 9/30

[58] **Field of Search** 23/259, 292, 253; 73/425, 425.2

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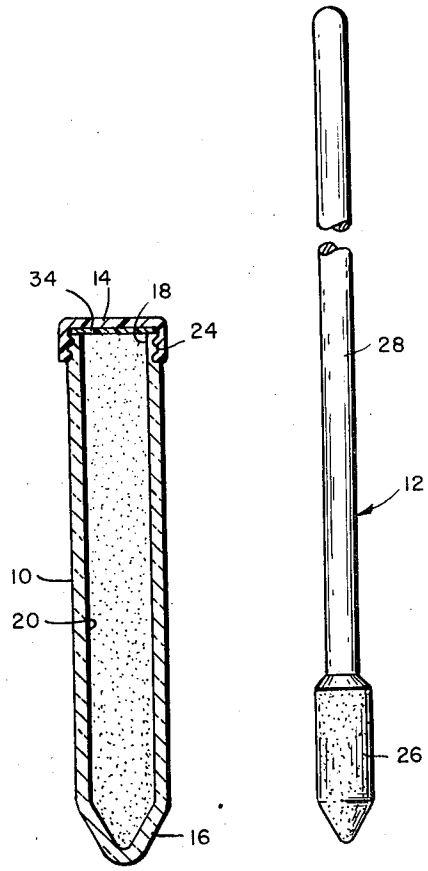
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Attorney—Robert T. Gammons et al.

[57] **ABSTRACT**

A glass tube closed at one end and open at the other, said open end being threaded, said tube being adapted, on the one hand, to receive a pestle slidingly within it, the interior surface of the tube and the exterior surface of the pestle being roughened so as to effect grinding of the material within the tube and, on the other hand, to receive a cap for closing the tube for shaking its content together with a solvent and for sealing the contents within the tube during centrifugation.

8 Claims, 7 Drawing Figures



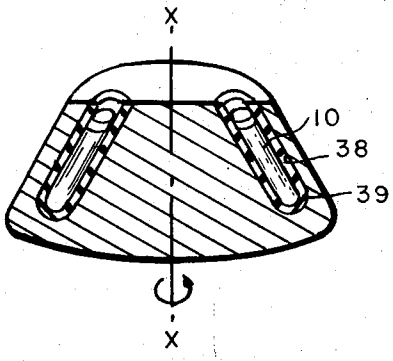


FIG. 1

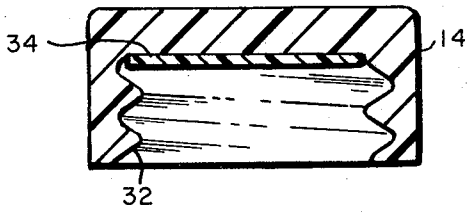


FIG. 6

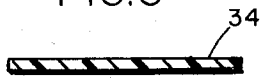


FIG. 7

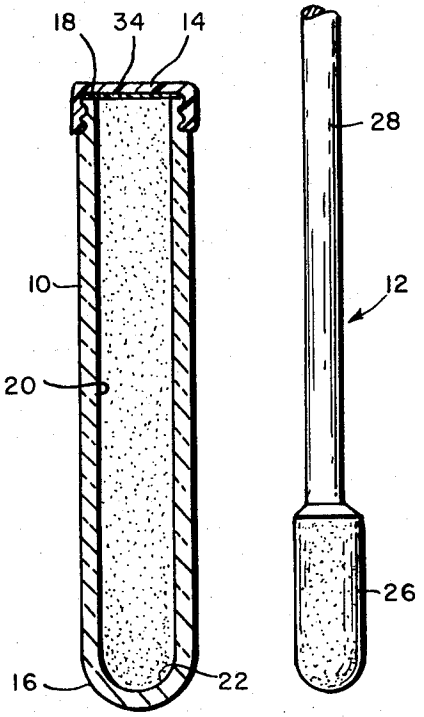


FIG. 2

FIG. 3

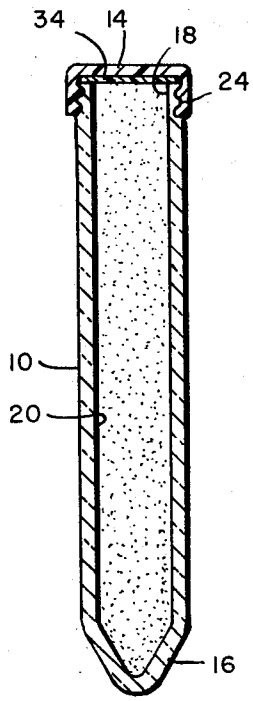


FIG. 4

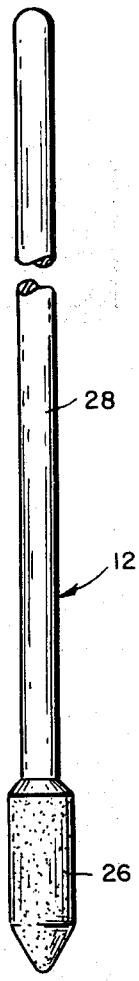


FIG. 5

CENTRIFUGABLE EXTRACTION AND HOHOGENIZATION VESSEL

BACKGROUND OF THE INVENTION

The usual practice in taking samples and extractions is to subject the material to be sampled to comminution with a motar and pestle, transfer it to a test tube where it is shaken up with a solvent and then retransferring it to a centrifuge device for centrifugation. When very small amounts of material are all that is available for testing the several transfers result in substantial loss of material consequently making the tests somewhat unreliable. There is no apparatus presently available in which a very small amount of material can be ground up, extracted and centrifuged in a single piece of equipment. It is accordingly the purpose of this invention to provide an apparatus in which all three operations may be performed without transferring the material from one receptacle to another so that even the smaLlest sample can be processed successfully.

SUMMARY

As herein illustrated, the apparatus comprises a hollow tube closed at one end and open at the other, said tube having a uniform interior cross-section throughout the major portion of its length from end to end, the surface of which is roughened, said tube being designed alternately to receive a pestle provided with a grinding head having a cross-section corresponding to that of the interior of the tube adapted to be slidably moved in the tube by means of a handle attached thereto to effect grinding of the material contained in the tube and to receive a cap for sealing the open top for agitation of the ground material in a solvent and/or sealing the tube during the subsequent centrifugation. The tube and pestle are comprised of glass, the interior surface of the tube and the exterior surface of the pestle being roughened and being slidably fitted with a clearance in the order of 0.004 inches. The tube has a wall thickness in the order of 0.094 inches, its open end is threaded and the cap is comprised of plastic containing threads by means of which it is threaded onto the open end of the tube. The top of the cap at the inside is fitted with a Teflon disc.

The invention will now be described in greater detail with reference to the accompanying drawings wherein:

FIG. 1 diagrammatically illustrates a centrifuge for receiving a capped tube for centrifugation;

FIG. 2 is a diametrical section of one component of the sampling apparatus comprising a hollow tube closed at one end and open at the other;

FIG. 3 is an elevation of another component of the sampling apparatus comprising a pestle adapted to be slidably engaged within the tube;

FIG. 4 is a diametrical section of a modified form of the tube shown in FIG. 2;

FIG. 5 is an elevation of a pestle adapted to be used with the tube shown in FIG. 4;

FIG. 6 is an enlarged diametrical section of another component of the sampling apparatus comprising a cap adapted to be mounted to the open end of the tube; and

FIG. 7 is a section of a sealing disc.

Referring to the drawings, the sampling apparatus comprises essentially three components, to wit, a tube **10**, a pestle **12** and a cap **14**.

The tube **10** has a closed end **16** and an open end **18**. The interior of the tube is of uniform cross-section throughout the major portion of its length from end to end, the interior surface **20** being cylindrical. The closed end of the tube has a surface **22** which is semispherical. The tube is comprised of glass, has a wall thickness of approximately 0.094 inches, is approximately 4 3/16 inches in length and has at its open end at its exterior surface threads **24** having a maximum outside diameter of 0.699 inches and a maximum inside diameter of 0.623 inches.

The pestle **12** has a grinding head **26**, the axial length of which is approximately 1.25 inches, the cross-section of which corresponds to that of the interior cross-section of the tube and which has an outside diameter of approximately 0.508 inches. The head is provided with a handle **28** which measures from one end of the head to its distal end approximately 7 inches.

The pestle is adapted to be slidably interengaged with the interior of the tube and to be manipulated therein by means of a handle **28** to grind material placed in the tube and to this end the entire interior surface of the tube is etched or mechanically roughened and the exterior surface of the head of the pestle is correspondingly etched or roughened.

A plastic cap **14** is provided for sealing the content of the tube within the tube during shaking with a solvent and for the centrifugation. The cap **14** contains threads **32** by means of which it is threaded to the open end of the tube and has within it at the top a Teflon disc **34** which forms a seal between the cap and the open end of the tube. The Teflon disc is approximately 0.079 inches thick.

FIGS. **4** and **5** show an alternative form of the apparatus wherein the closed end of the tube is conical and the end of the head of the pestle is correspondingly shaped.

FIG. **1** shows diagrammatically a centrifuge comprising a rotor **36** rotatable about a vertical axis X—X, the rotor containing pockets **38** for receiving capped tubes. Desirably to protect the tubes and to hold them snugly engaged within the pockets, tubular rubber sleeves **39** are inserted into the pockets to cushion the capped tubes.

The procedure with such a piece of apparatus is to place the sample to be examined or tested in the tube, effect its comminution by reciprocating the pestle therein, remove the pestle, add a solvent, apply the cap and shake the material to dissolve it in the solvent and then mount the capped tube or tubes in the centrifuge shown in FIG. **1** to effect centrifugation.

It should be understood that the present disclosure is for the purpose of illustration only and that this invention includes all modifications and equivalents falling within the scope of the appended claims.

We claim:

1. A sampling assembly comprising a hollow tube closed at one end and open at the other, said tube having a uniform interior cross-section throughout the major portion of its length from end to end, the surface of which is roughened, a cylindrical grinding head having a cross-section corresponding to that of the interior of the tube, the surface of which is roughened like that of the interior of the tube, a handle fixed at one end to one end of the head by means of which the head may be moved in shearing engagement with the interior of the tube to grind material contained in the tube, a cap for closing the open end of the tube following grinding, and a thread circumferentially of the open end of the

tube onto which the cap is adapted to be screwed for sealing the tube incident to centrifugation of the content of the tube.

2. Apparatus according to claim 1, wherein the difference between the inside diameter of the tube and the outside diameter of the head of the pestle is in the order of 0.004 inches.

3. Apparatus according to claim 1, wherein the pestle has a roughened surface which extends axially thereof and is in the order of 1.25 inches.

4. Apparatus according to claim 1, wherein the interior wall of the tube at the closed end is semispherical and the end of the head of the pestle is correspondingly shaped.

5. Apparatus according to claim 1, wherein the interior surface of the tube at the closed end is conical, and the head of the pestle is correspondingly shaped.

6. Apparatus according to claim 1, wherein the wall thickness of the tube is 0.094 inches.

7. Apparatus according to claim 1, wherein the tube is glass and the cap is plastic.

8. A sampling assembly according to claim 1, comprising a Teflon sealing disc fitted between the top of the cap and the open end of the tube.

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