Dec. 10, 1968 J. H. ELLIS 3,415,380

SAMPLE PREPARATION APPARATUS AND UNITARY FILTER-PIPETTE FOR USE THEREIN

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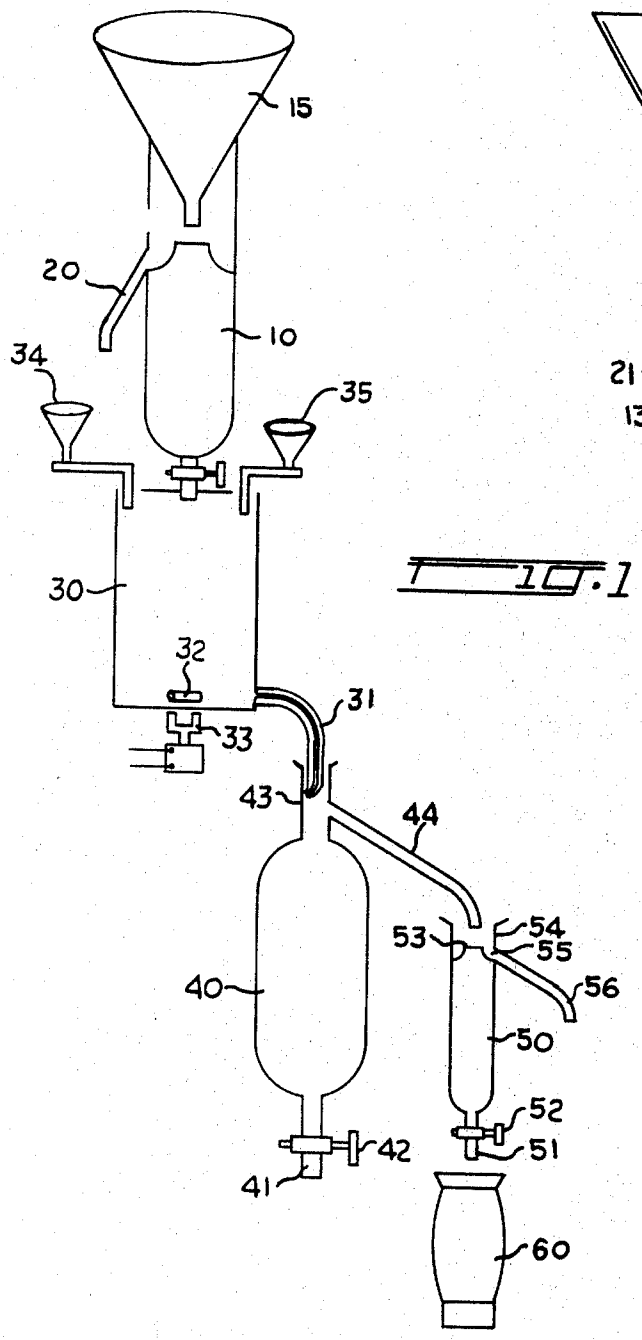

FIG. 2

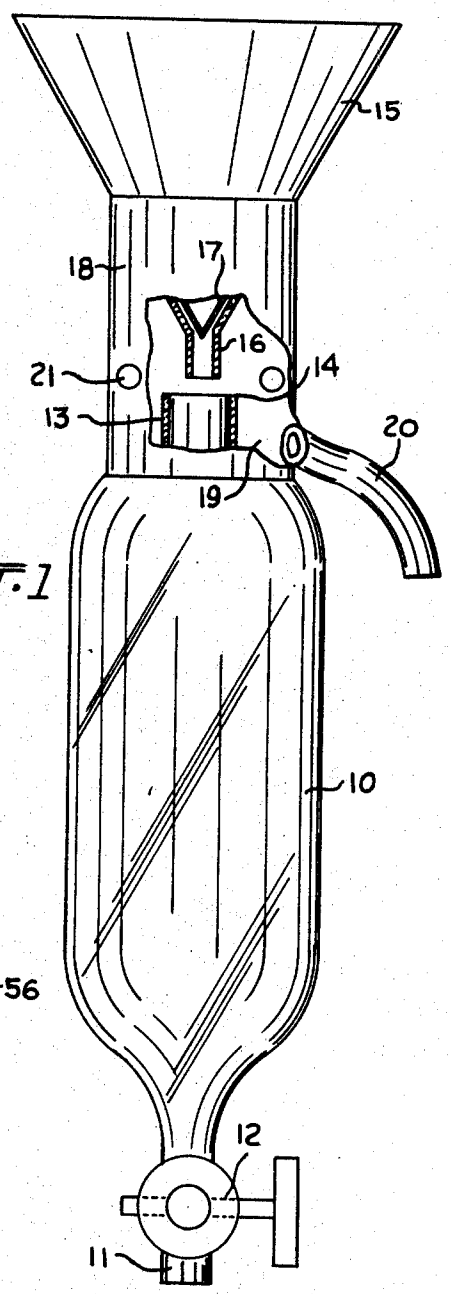

FIG. 1

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SAMPLE PREPARATION APPARATUS AND UNITARY FILTER-PIPETTE FOR USE THEREIN

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ABSTRACT OF THE DISCLOSURE

A unitary filter-pipette which dispenses an accurate aliquot of filtrate is constructed to insure easy washing and rapid dispensing. An assembly of laboratory apparatus employing the filter-pipette as the leading section is disclosed.

This invention relates to chemical laboratory apparatus for preparation of liquid samples for analysis. More particularly it relates to an improved unitary filter-pipette and to an assembly of apparatus in which the filter-pipette serves as the leading section.

The output of work of a laboratory worker usually is related to the number of separate manipulations which he is required to perform and when one or more of these manipulations can be omitted, simplified, or shortened in duration, a more efficient use of the worker's time can be accomplished, particularly when repetitive preparation of samples is undertaken. Traditionally, the steps of filtering and pipetting an aliquot of filtrate have been time consuming and although various forms of laboratory equipment have been proposed for automatic filling of a pipette, as, for example, the measuring device disclosed in Markwood Patent 2,149,303, to the best of my knowledge none have been entirely satisfactory in accomplishing the purposes of the present invention.

An object of the invention is to provide an improved unitary filter-pipette which will provide an accurate aliquot of filtrate, which will drain rapidly, and which can be easily washed.

Another object is to provide an improved assembly of laboratory apparatus employing a unitary filter-pipette as the leading section of the assembly.

These and other objects of the invention will become more apparent as the description proceeds and when considered in conjunction with the accompanying drawings in which

FIG. 1 is an elevation view with parts broken away and showing a unitary filter-pipette constructed in accordance with the invention, and

FIG. 2 is a diagrammatic view of an assembly of apparatus in accordance with the invention.

Referring now to FIG. 1 the pipette element, or lower portion, of the filter-pipette preferably includes a glass tube **10** having a narrowed outlet pipe **11** controlled by a conventional valve **12**. At its upper end the tube **10** is formed in the shape of an inlet nozzle **13** having a generally circular edge **14** and of a diameter providing a generous sized opening through which drops of filtrate may fall without touching the nozzle and through which a large volume of air may rush when the pipette is drained.

The filter element, or upper portion, of the article includes a funnel **15** preferably made of glass and having a centrally disposed lower pipe **16** whose axis preferably coincides with the axis of the nozzle and in which funnel a sheet of conventional filtering medium **17** may be detachably supported. A tubular collar **18** preferably made of glass is rigidly joined at its lower periphery to the exposed surface of the pipette tube below the level of rim **14** and is rigidly joined at its upper periphery to the exposed surface of the funnel above the level of the rim **14**.

Accordingly, an overflow space **19** is provided within the collar in surrounding relation to the inlet nozzle and into which liquid spilling over rim **14** following the filling of the pipette will enter. Attached to one side of the collar and communicating with the overflow space at the bottom thereof is an open drain tube **20**. Moreover, one or more generous sized apertures **21** are formed in the wall of the collar above the level of rim **14**.

As will now be evident, when a liquid to be filtered is pourned into the funnel and with valve **12** in its closed position, the pipette fills to its predetermined volume reaching to the edge of rim **14** and any additional filtrate is vented automatically through drain **20**. Then when the valve **12** is opened, air rushes in through aperture **21** and a fast dispensing of the pipette contents will result. The article may be detachably mounted in any suitable manner, as by means of a laboratory stand and when washing in preparation for another sample is required, all that is needed is to remove the spent filter sheet **17**, close valve **12**, pour in washing liquid until rim **14** overflows and then drain the pipette by opening valve **12**. When washing of the article detached from its support is preferred, as by immersion in a bath, the aperture **21** provides a means for easy ingress of a large volume of wash liquid quickly into the interior of the article.

As thus far described, the filter-pipette article is of general application to laboratory work and it will be understood that in its broader aspects the present invention is not necessarily limited to the remaining apparatus with which the filter-pipette article is to be employed. I now have discovered, however, that when the described article constitutes the leading section of an improved assembly of apparatus in which partitioning of a liquid is involved, a significant saving of the time of the laboratory worker results.

Referring now to FIG. 2 and considering the invention in its relation to the conduct of a typical sample preparation for determining the pesticide residue in a dairy feed by column chromotography technique, the pipette **10** when emptied of a predetermined aliquot rapidly dispenses into a non-metallic mixing tank **30** maintained at atmospheric pressure and having a constantly open discharge drain **31** embodying a passage of capillary dimension sufficient to pass the contents of tank **30** in about seven minutes when an aliquot of about 75 milliliters of sample material is dispensed from the pipette.

Disposed within tank **30** is a conventional magnetic stirrer **32** actuated by a motor-driven magnet **33**. During washing of the apparatus this stirrer does not require removal from the tank. Adjacent the top of the tank is a first inlet means **34** for an aqueous solvent and a second inlet means **35** for a relatively light weight partitioning carrier, such as n-hexene. As will be understood, simultaneously with the discharge of the pipette into the tank a predetermined quantity of the aqueous solvent and of the liquid carrier also are discharged into the tank and the three liquids are simultaneously mixed by the mixer whereupon the ingredient of the filtrate to be analyzed is bound to the light weight carrier liquid.

Mounted at a lower elevation and receiving all of the contents of the tank from the capillary drain **31** is a separatory member having a lower tubular, body portion **40** equipped with a waste outlet **41** controlled by a valve **42**. This member includes an open upper pipe **43** of substantially less diameter than the body portion **40** and in surrounding relation to the lower end of the tank drain **31**. Extending laterally from pipe **43** is an open overflow pipe **44** communicating with pipe **43** adjacent the lower end of capillary drain **31** and which drain is arranged to trickle liquid down the side of pipe **43** away from the entrance into overflow pipe **44**. Accordingly, as separatory member **40** fills, the light weight liquid containing the ingredient to

be analyzed floats above the heavier liquid and in due time spills into overflow pipe **44**.

Mounted at a lower elevation and receiving the overflow from pipe **44** is a relatively small overflow pipette which for example, may hold a volume of 15 milliliters. This pipette comprises a tubular body portion **50** equipped with an outlet pipe **51** controlled by valve **52**. At its upper part the body portion is formed in the shape of an inlet nozzle **53**, similar to that of nozzle **13** shown in FIG. 1, having a generally circular edge and of a diameter greater than that of the pipe **44** whereby drops of liquid may fall into the pipette without touching that inlet nozzle. An upstanding collar **54** is rigidly attached to the body portion **50** and provides an overflow space **55** surrounding the nozzle and into which space excess liquid will flow by spilling over the nozzle edge after the pipette is filled. Extending from this space is a drain **56** for such excess liquid.

The described apparatus, which normally will be mounted upon laboratory stands at differing elevations to insure gravity flow, preferably also is mounted so that discharge from overflow pipette **50** is directly into the inlet **60** of the associated analyzing apparatus, as, for example, a column chromotograph. It will, of course, be understood that the volume of heavier liquids supplied by the separatory member is so calculated that one of the same will enter overflow pipe **44**.

As will now be apparent, after the laboratory worker has prepared a material for its filtration and with the supplementary liquids ready for introduction into the mixer tank, a rapid and accurate operation may be conducted.

Having thus described the invention and its attendant advantages, it is intended that the appended claims are to cover such changes and modifications of the described invention as come within the true spirit and scope of the same.

What is claimed is:

1. A unitary filter-pipette comprising a lower portion including a pipette tube having a valve-controlled dispensing means at its lower end and an inlet nozzle with a generally circular overflow rim at its upper end, said nozzle being unobstructed so that air may rush quickly therethrough during emptying of the filter-pipette, the diameter of said nozzle being less than the diameter of said tube, an upper portion comprising a funnel adapted to support a filter medium and to direct filtered liquid into said tube within the confines of said rim and a tubular collar portion rigidly attached to said upper and lower portions and spacing the same from each other to define an overflow space surrounding said nozzle and for receiving liquid spilling over said rim following the complete filling of said pipette tube, said collar portion including means for draining to the exterior of said filter-pipette liquid collecting in said overflow space and also including an aperture in its wall, said aperture being located above said rim and of a size sufficient to pass air rapidly into said collar portion during the draining of said pipette tube and to permit passage of washing liquid quickly into or out of said collar portion during cleaning of said filter-pipette.

2. Apparatus for preparing samples of lquid for analysis and including in combination, a unitary filter-pipette for accumulating a predetermined volume of filtered liquid and having an overflow drain for dispensing of liquid supplied thereto in excess of said predetermined volume; a partitioning mixer tank for receiving at its upper end filtered liquid by gravity flow from said filter-pipette and having at its lower end an open drain of capillary dimensions; means for supplying an aqueous solvent and a relatively light weight carrier liquid to said tank; means for mixing said filtered liquid, aqueous solvent and carrier liquid in said tank while simultaneously flowing the mixed liquids through said capillary drain, an overflow separatory member receiving by gravity flow the mixed liquids from said tank; said member including an overflow drain at its upper end for dispensing the light weight carrier liquid following filling of said member, and valve-controlled drainage means at its lower end for venting residual liquids; and an overflow-pipette receiving the light-weight carrier liquid from said member by gravity flow through the overflow drain of said member, said overflow-pipette including means at its lower end for dispensing the liquid collected therein, and an overflow drain at its upper end for dispensing of liquid supplied thereto in excess of the predetermined volume of said overflow-pipette.

3. Apparatus as defined in claim **2** wherein said means for mixing liquids in said tank comprises a magnetic stirrer adapted to remain in said tank during the washing of the apparatus.

4. Apparatus as defined in claim **2**, wherein each of said filter-pipette and said overflow-pipette includes an upwardly directed nozzle portion having a rim over which excess liquid is adapted to spill into the respective drain.

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210—205, 262; 23—259; 73—426