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Cohen

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[54] **APPARATUS FOR DETERMINING ORGANIC CARBON CONTENT OF POLLUTED LIQUIDS**

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[52] **U.S. Cl.**..... **23/253 PC,** 23/230 PC, 23/284

[51] **Int. Cl.**..... **B01j 9/02,** G01n 31/12, G01n 33/18

[58] **Field of Search** ..... 23/230 PG, 253 PC, 284, 23/277 R

[56] **References Cited**

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Primary Examiner—Robert M. Reese

[57] **ABSTRACT**

Apparatus for determining the organic carbon content of a liquid sample whereby the sample is acidified and scrubbed to remove its inorganic carbon content, and then vaporized in a reaction chamber having an air atmosphere which oxidizes to carbon dioxide the total organic carbon content of the sample. Measurement of the amount of carbon dioxide formed from the organic carbon is effected by an infrared analyzer.

5 Claims, 3 Drawing Figures

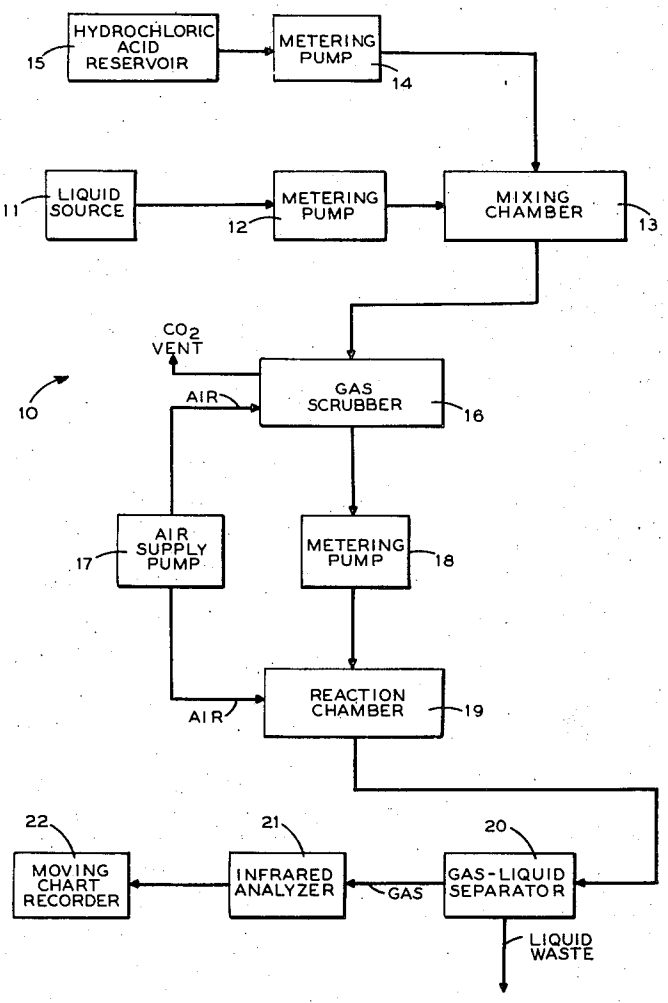

FIG. 1

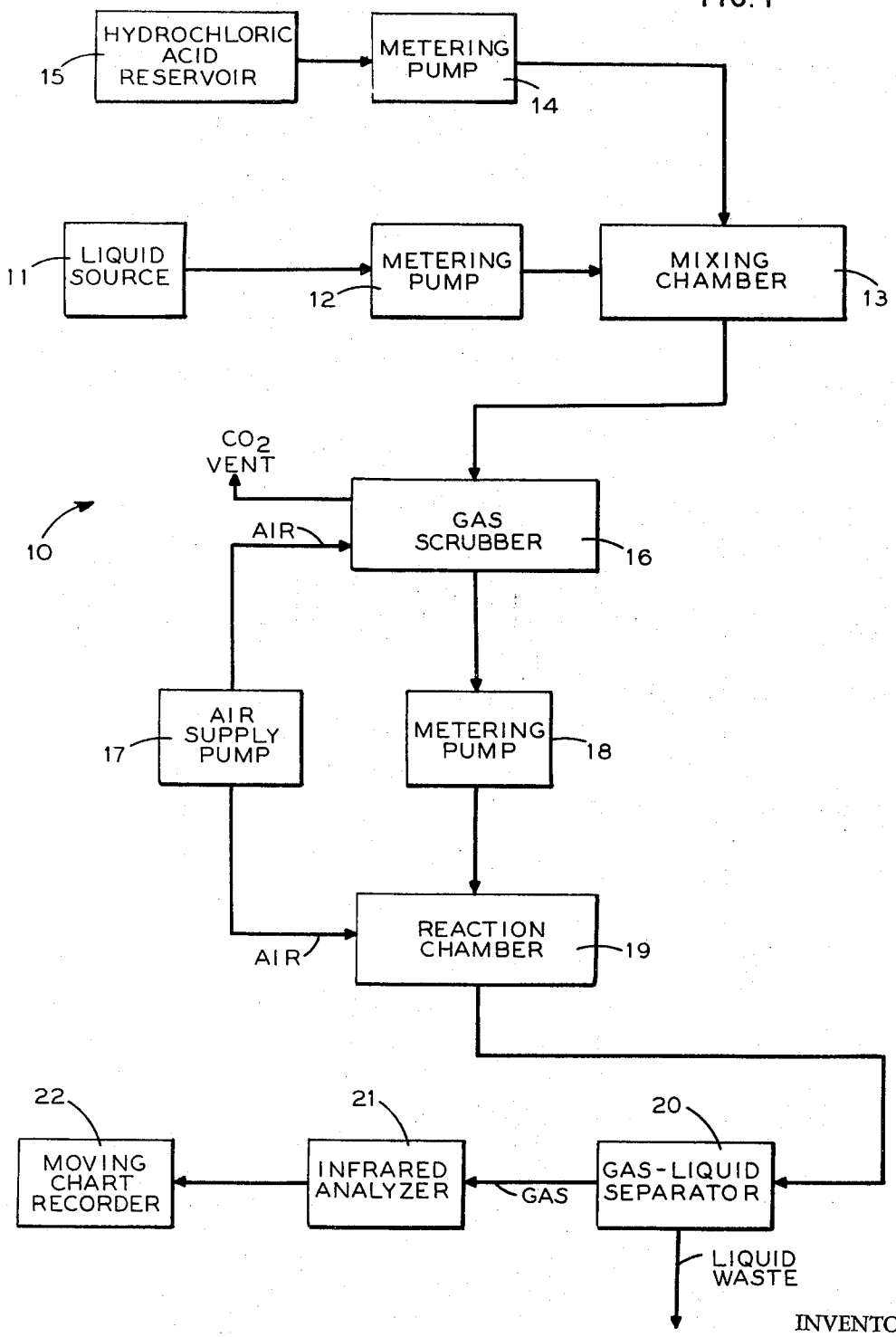

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FIG. 3

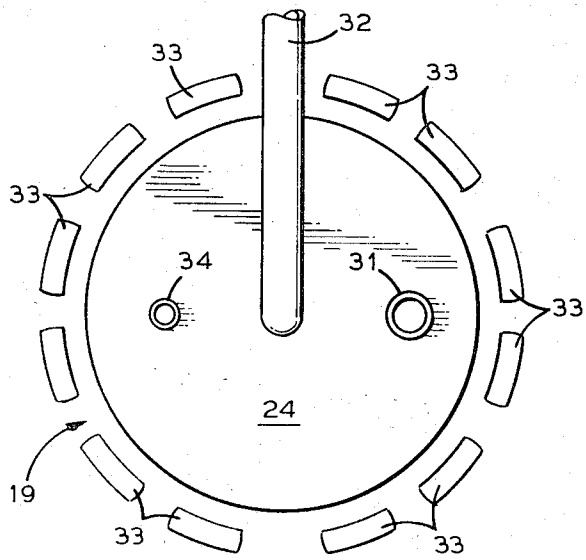
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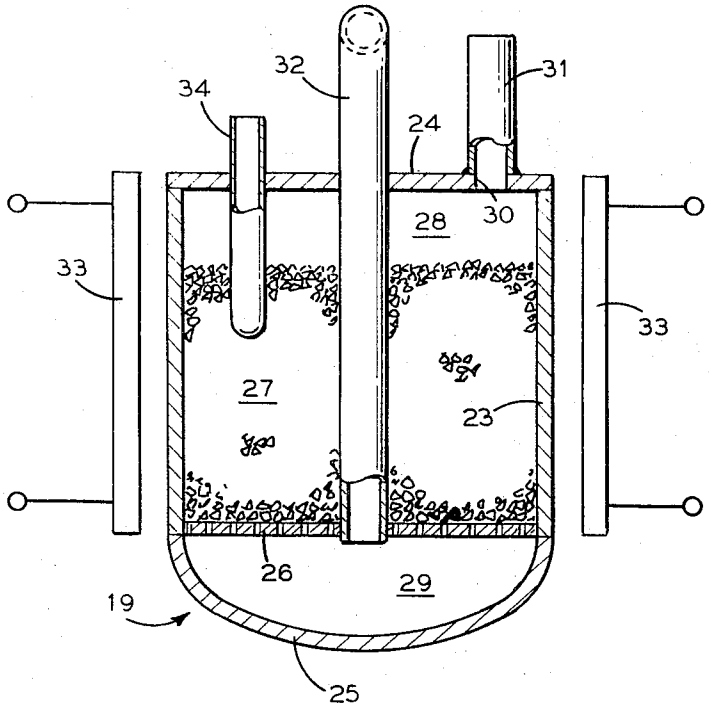
FIG. 2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APPARATUS FOR DETERMINING ORGANIC CARBON CONTENT OF POLLUTED LIQUIDS

BACKGROUND AND SUMMARY OF THE INVENTION

In the measurement of pollution levels in aqueous systems, the amount of organic carbon in a sample taken from the liquid source to be monitored is an important index of the existing pollution level.

Accordingly, there is a need for equipment and methods that enable rapid and accurate determination of the organic carbon content of a liquid on a continuous or quasi-continuous sampling basis. One rather elaborate apparatus for total carbon analysis is described in U.S. Pat. No. 3,296,435 issued on Jan. 3, 1967 to J. L. Teal et al.

The invention provides an improved apparatus and a method for performing total organic carbon analyses more efficiently and rapidly than can be done with either the standard laboratory five-day biochemical oxygen demand test (B O D), or the two-hour chemical oxygen demand test, (C O D).

In the analytical apparatus of the invention, a sample stream from the aqueous liquid source to be monitored is continuously fed into a mixing chamber together with sufficient acid to react with and remove as carbon dioxide all of the inorganic carbon content of the sample, i.e. the carbonates in the sample. The acidified sample is scrubbed to remove all carbon dioxide produced by the carbonate-acid reaction, and the carbonate-free sample is metered into a reaction chamber. The reaction chamber has an inlet through which the sample and air for oxidizing the organic carbon of the sample are introduced; an outlet for the exit of gaseous and vapor products formed from the sample; and a granular packing material inside that is heated by electric resistor elements disposed outside the chamber in surrounding relation thereto. This packing material is chemically inert, as for example, crushed quartz, and is heated to a temperature at which the organic carbon in the sample is oxidized to carbon dioxide by the air atmosphere in the chamber.

Clogging of the reaction chamber by solids deposited upon the packing material is minimized through a special arrangement of the packing material with respect to the chamber boundaries. In accordance with the invention, the packing material is disposed to define within the reaction chamber an inlet plenum zone communicating with the inlet to accommodate preheating of the sample by the packing material without contact therewith; and an outlet plenum zone. The outlet plenum zone is separated from the inlet plenum zone and communicates with the outlet to collect and deliver thereto the gaseous and vapor products of the sample.

The size of the chamber, its inlet plenum zone and the thermal mass of the packing material are such that the sample is vaporized within the inlet plenum zone, before contact with the packing material itself.

Carbonaceous matter in the sample is quickly oxidized by the hot air atmosphere of the chamber so that the effluent therefrom is a mixture of water vapor and other gaseous products including carbon dioxide, the amount of carbon dioxide corresponding to the amount of organic carbon originally present in the sample. The water vapor portion of the reaction chamber effluent is expediently removed by a conventional gas-liquid separator before delivery to an infrared analyzer for measurement thereby of the carbon dioxide component.

For a better understanding of the invention and its various advantages, reference should be had to the following detailed description and accompanying drawing which together exemplify a preferred embodiment of the invention.

BRIEF DESCRIPTION OF THE DRAWING

In the drawing:

FIG. 1 is a schematic diagram of an organic carbon content analyzer apparatus according to a preferred embodiment of the invention.

FIG. 2 is an elevation view, partly in section, of the reaction chamber used in the apparatus represented by FIG. 1, as seen in a typical installation arranged for external heating of the reaction chamber and packing material therein.

FIG. 3 is a plan view of the reaction chamber and heating installation shown in FIG. 2.

DESCRIPTION OF A PREFERRED EMBODIMENT OF THE INVENTION

In FIG. 1 there is schematically exemplified an analyzer apparatus **10** constructed in accordance with the invention and which is operable to measure, on a continuous flow basis, the total organic carbon content in a sample stream taken from an aqueous liquid source **11**, such as a body of water to be monitored for pollution level.

A continuous sample stream from source **11** is delivered by a metering pump **12** into a mixing chamber **13**. Another metering pump **14** delivers hydrochloric acid from a reservoir **15** into mixing chamber **13** for the purpose of acidifying the liquid sample therein to a pH of approximately 2.0. The inorganic carbon present in the sample as carbonates is converted to carbon dioxide by reaction with the acid, and the organic carbon content of the sample is substantially unchanged.

The acidified sample stream is expediently fed into a conventional gas scrubber **16** that is supplied with air from a pump **17** to purge from the sample whatever carbon dioxide was formed by the acidification reaction. This carbon dioxide, which corresponds quantitatively to the amount of carbonates in the sample, is by choice vented to a waste zone, but could be delivered to a measuring means (not shown) should it be desired to measure the amount of inorganic carbon in the sample.

The carbonate-free sample is fed by a metering pump **18** from scrubber **16** into a heated reaction chamber **19**, which is also supplied with air from pump **17** to establish an oxidizing atmosphere within chamber **19**. This hot oxidizing atmosphere rapidly vaporizes the sample and oxidizes the organic carbon content thereof to carbon dioxide.

The vapor and gaseous mixture effluent of reaction chamber **19** is delivered to a conventional gas-liquid separator **20** that removes by condensation the vapor component of the mixture and passes the gaseous portion containing the carbon dioxide to an infrared analyzer **21**.

Infrared analyzer **21** is of conventional construction and serves to measure the amount of carbon dioxide formed by oxidation of the organic carbon in the sample. The total quantity of organic carbon originally present in the sample can be readily established by ap-

plying a known proportionality factor to the carbon dioxide measurement. Analyzer **21** is preferrably of a type which has an electrical analog signal presentation of the carbon dioxide measurement data. In such case, the carbon dioxide data signal is applied to the input of a compatible moving chart recorder **22** for direct display of the analysis results, either in terms of carbon dioxide quantity or, in terms of equivalent organic carbon, which can be done by suitable calibration of recorder **22** using techniques well known to those skilled in the art.

For calibration purposes, the inlet of metering pump **12** can be diverted temporarily from source **11** to another source, (not shown), of liquid having a known concentration of organic carbon sufficiently constant as to be useable as a standard. The gas mixture output of separator **20** can be applied to an oxygen demand analyzer, (not shown), instead of to the infra-red analyzer **21** where it is desired to measure the oxygen demand level of the sample.

While other means could be employed for effecting oxidation of the organic carbon content to carbon dioxide, the reaction chamber **19,** shown in specific detail by FIGS. **2** and **3** offers definite improvements and advantages over prior art counterparts. The Chamber **19** has a peripheral wall **23** joined to a top cover **24** and a bottom cover **25** so as to form a sealed enclosure. Within chamber **19** is an apertured plate **26** which serves to retain a quantity of crushed quartz material **27.**

Packing material **27** is so disposed as to define within chamber **19** an inlet plenum zone **28** and an outlet plenum zone **29** which is separated from inlet plenum zone **28.** Plenum zone **28** communicates with an inlet opening **30** provided in cover **24** and a conduit **31** extending outside chamber **19.** Conduit **31** in turn communicates with both the air supply pump **17** and the outlet of metering pump **18** to accommodate the introduction of oxidizing air and the sample into chamber **19.** The outlet of chamber **19** is defined by a conduit **32** that extends through cover **24,** inlet plenum zone **28,** packing **27** and plate **26** to communicate with the outlet plenum zone **29.** Plenum zone **29** thus serves to collect and deliver to outlet conduit **32** the gaseous and vapor products from the sample for exit from chamber **19.** Plenum zone **28** is used for preheating the sample by packing **27** without contact therewith.

Pump **17** is selected and operated so as to deliver into chamber **19** several times the stoichiometric volume of air required for complete oxidation of the maximum anticipated carbon content of the sample.

Disposed outside chamber **19** in surrounding spaced relation thereto are a plurality of electric heater elements **33** that are used to heat the chamber **19** and quartz packing **27** to a temperature of approximately 850°C. At such temperature, and with a mass of packing **27** that occupies at least **50** percent of the volume of chamber **19,** the sample is preheated to vaporization almost immediately upon entering plenum zone **28,** for sample feed rates up to about **2** percent per minute of the plenum zone **28** volume.

For example, at a packing **27** temperature of 850°C, plenum zone **28** vaporization of an aqueous sample was achieved at a sample feed rate of 4cc/minute into chamber **19** having a plenum zone **28** volume of 500 cc and a total volume of 2,660 cc of which 1,660 cc was occupied by quartz packing **27.**

With the plenum zone vaporization afforded by the invention, there is no cold liquid impingement upon the packing, and therefore the packing is not subject to further pulverization by thermal stress action. Consequently, the flow area presented by the voids between the pieces of packing **27** stays relatively constant and is not reduced significantly by clogging due to pulverization compaction of the packing **27.**

In plenum zone **28,** the sample is converted to a mixture of vapor and suspended solids which then pass the inert packing **27** in heat exchange flow contact therewith, and the organic carbon content of the mixture is completely oxidized to carbon dioxide before passing through plate **26** into the outlet plenum zone **29** at the opposite end of chamber **19.**

To aid in controlling the temperature of the packing **27** there is provided in chamber **19** a thermocouple well **34,** into which can be inserted a standard thermocouple, (not shown).

From the foregoing description it will become apparent to the artisan that the invention is adaptable to many specific applications where the carbon content of liquids must be monitored, and that obvious changes in the exemplified apparatus can be made to satisfy particular requirements.

What is claimed is:

1. In an analytical apparatus for determining the total quantity of organic carbon as a pollutant in an aqueous stream by vaporizing a sample taken from the stream and oxidizing to carbon dioxide the total organic carbon in the sample, the determination being on the basis of the measure of carbon dioxide formed per unit measure of the sample, the improvement which comprises a mixing chamber, a first metering pump disposed to deliver into said mixing chamber a continuous flow sample taken from the stream, a second metering pump disposed to deliver into said mixing chamber a continuous flow of liquid reagent for reaction therein with the sample to remove the inorganic carbron content thereof as carbon dioxide; a reaction chamber, means connected to said mixing chamber and to said reaction chamber to deliver from said mixing chamber into said reaction chamber a continuous flow aqueous sample to be analyzed and which is substantially free of inorganic carbon and carbon dioxide, said reaction chamber having an inlet for introducing the aqueous sample to be analyzed and an outlet for the exit of gaseous and vapor products formed from such sample, means for introducing air into said reaction chamber to establish an oxidizing atmosphere therein, a packing material disposed within said reaction chamber for heating the sample introduced, and means for heating said packing material to a temperature at which the organic carbon in the sample is oxidized to carbon dioxide by said atmosphere, said packing material being disposed to define within the reaction chamber an inlet plenum zone communicating with said inlet to accommodate preheating of the sample by the packing material without contact therewith, and an outlet plenum zone separated from said inlet plenum zone and communicating with the outlet to collect and deliver thereto the gaseous and vapor products from the sample, said outlet plenum zone being communicated with said outlet by a conduit extending through said packing material.

2. The improvement according to claim **1** including a conduit extending outside the reaction chamber and communicating with an air supply means and said inlet

for introducing air therethrough along with the sample.

3. The improvement according to claim 1 wherein the means for heating said packing material is disposed outside the reaction chamber.

4. The improvement according to claim 1 wherein said packing material is crushed quartz and occupies at least 50 percent of the reaction chamber volume.

5. The improvement according to claim 1 wherein said inlet plenum zone is at one end of the reaction chamber and said outlet plenum zone is at the opposite end of the reaction chamber, and said packing material contains voids accommodating the flow therethrough to the outlet plenum zone of the gaseous and vapor products of the sample.

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