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GAS-LIQUID CONTACT APPARATUS

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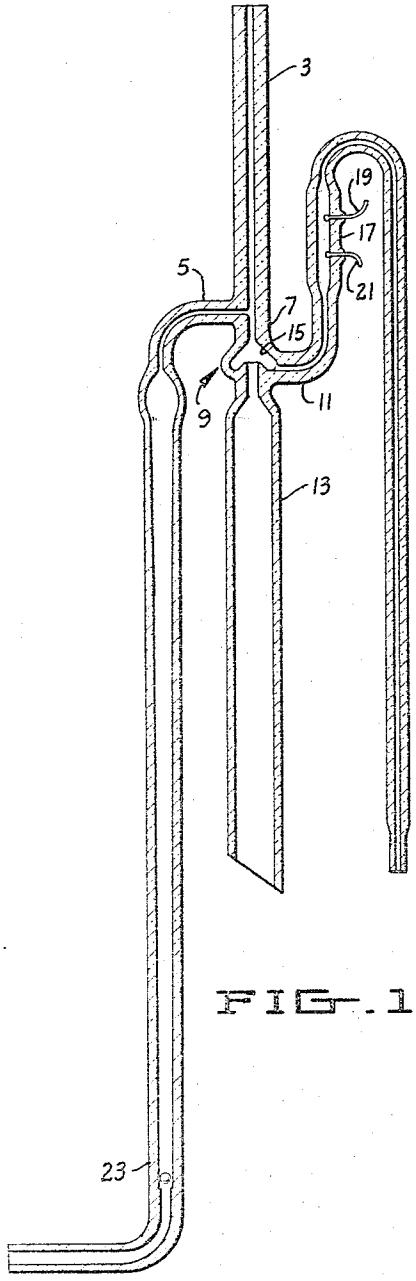



FIG. 1.

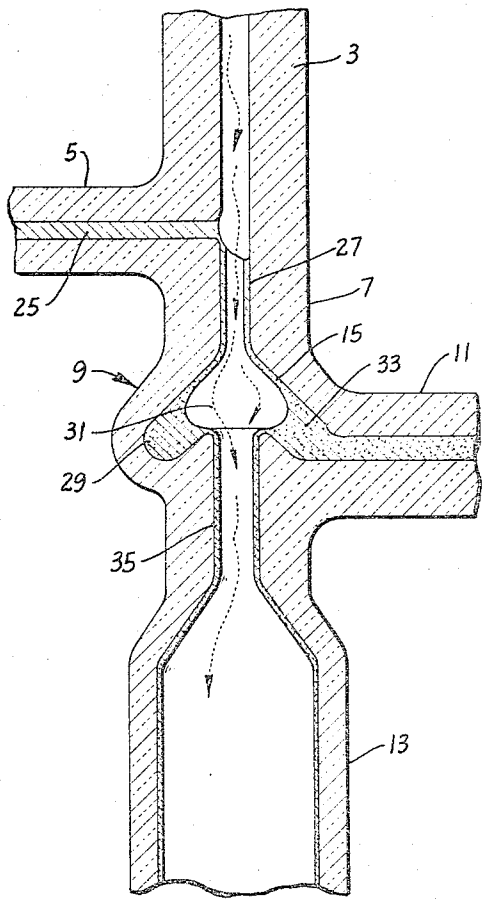



FIG. 2.

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# GAS-LIQUID CONTACT APPARATUS

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2 Claims. (Cl. 55—228)

This invention relates to a gas-liquid contactor wherein a gas is dissolved in a liquid and which has a separating section for the removal of any undissolved gas.

It has previously been proposed to provide a detector for use in gas chromatography based on the principle of determining the conductivity of a solution produced by dissolving a portion of the eluted gas in a liquid. Although this is a very simple and effective procedure, particularly in the analysis of pesticide residues containing chlorine or sulfur, it has heretofore been difficult to provide an efficient device for dissolving the eluted gas in the liquid. The devices hertofore employed have had relatively large chambers, and particularly large separating sections so that there was a large dead volume or holdup in the dissolving apparatus.

In gas chromatography, it is important that the holdup be very slight in order to obtain the highest possible resolution. This is particularly true when one considers that the quantities involved may be as little as 10 nanograms and that successive peaks of the eluted material can occur within a few seconds of each other. It is thus apparent that if there is any substantial holdup in the contact apparatus and particularly in the separating section thereof, the height of the peaks will be lessened and a false tail reading will be obtained.

It is therefore an object of the present invention to provide a gas-liquid contact apparatus including a separator section wherein there is little holdup of liquid.

Another object of this invention is to provide a gas-liquid contact apparatus which will operate over long periods of time without surges.

Still another object of this invention is to provide a gas-liquid contact apparatus which is simple and inexpensive to construct.

Other objects will be apparent in the balance of the specification which follows.

In the drawings forming a part of this application:

FIGURE 1 is a side sectional view of a device embodying the present invention.

FIGURE 2 is an enlarged partial sectional view showing the contact and separation sections of the device shown in FIGURE 1, illustrating the principle of operation.

Turning now to a description of the drawings, there is shown in FIGURES 1 and 2 a capillary tube 3 for the introduction of a gas and a second capillary tube 5 at right angles thereto for the introduction of a liquid. The two tubes merge into a contact section 7 and then empty into a separation section 9. Leading from the separation section 9 is a side arm 11 for the removal of a portion of the liquid having gas dissolved therein while the bottom of the separating section leads to an enlarged tube 13 for the removal of any gas which did not dissolve in the liquid as well as any portion of the liquid which is not removed through the side arm 11.

It will be noted that the top portion of the separating section 9 is flared as at 15, the purpose of which will be later explained.

Although not necessarily forming a part of the present invention it will be noted that the side arm 11 leads to a measuring cell 17 having electrodes 19 and 21 making conductivity measurements. Further, the entrance section 5 may have a rotameter 23 for measuring the flow of the in-coming liquid.



Referring now specifically to FIGURE 2, it will be seen that the liquid 25 as it enters through the tube 5 to the capillary 7 flows down the walls as at 27 and continues along the flared portion 15 collecting in a small pool 29 within that section. The liquid does not flow into the center as at 31 as the result of the flared entrance so that a single, stationary bubble of gas forms in the separating section. Liquid 29 surrounding this bubble of gas can flow out through the tube 11 as at 33 and any liquid which does not follow this path continues to flow down the walls as at 35; any liquid which is not taken off through the tube 11 flows out of the apparatus through the tub 13 while any gas which has not dissolved in the liquid flows out through the tube 13 through the center thereof.

The capillary tubes employed can be from 0.1 to 1 mm. in inside diameter. Preferably the gas entry tube 3 and the contact tube 7 have a larger diameter than the tube 5 for the introduction of liquid. The length of the co-current contact section 7 should be 10 to $10^4$ times the diameter in order to get good contact between the gas and liquid phases. The desired length depends on the affinity of the impurity in the gas stream for the liquid phase. Hydrochloric acid can be quantitatively transferred into water in a very short contractor section while carbon dioxide requires a very long contactor for equilibration.

In this manner there is provided a gas-liquid contact apparatus wherein the flow is smooth and the holdup very slight.

The combination shown in FIGURES 1 and 2 may be used for the detection of traces of HCl, $SO_2$, $SO_3$, $NO_2$, $CO_2$ and other acids or acid anhydrides as well as bases in a gas stream if water or any other suitable solvent for ionization of acids, bases and salts is used as the liquid fed into the liquid inlet. This detector has been used for the detection of nanogram amounts of certain chlorine—and also certain sulfur—and also certain nitrogen-containing organic chemicals that have been eluted from a gas chromatography column, combusted with oxygen (or air) and fed into the detector as the gas stream entering the gas inlet capillary. With the cell shown in FIGURE 1, with a water flow rate of approximately (but controlled) 1 to 2 ml./min. and a gas flow rate into the detector of 100 to 300 cc./min., successful analyses of mixtures of chlorinated organic pesticides have been demonstrated. The relationship between peak areas and the amounts of chloride represented by the various peaks is linear and directly proportional from as low as $5\times10^{-9}$ g. of chloride or greater.

I claim:

1. A gas-liquid contact apparatus comprising in combination:

(a) a first generally vertical capillary tube for the introduction of a gas into the apparatus;

(b) a second generally horizontal capillary tube entering into the first tube at substantially right angles thereto;

(c) a smooth walled gas-liquid contact capillary tube, the contact tube forming a continuation of the first tube and extending downwardly from the junction of the first and second tubes;

(d) a separator section at the lower terminal end of the contact capillary tube, said separator section comprising an enlarged chamber having a gently flared entrance leading from the contact tube;

(e) a side capillary tube leading at right angles from the separator section;

(f) an enlarged tube leading from said separator section and extending generally downwardly therefrom;

(g) a barrier extending upwardly from the bottom of the separator section between the side capillary tube and the enlarged tube;

(h) said tubes being so proportioned that liquid will flow down the walls of the contact and separation sections with gas flowing downwardly at the center of the hollow, liquid-wall column so formed and said barrier and said separator section being so constructed to cause a pool of liquid to form in the bottom portion of the separator section so that a portion of the liquid having gas dissolved therein will flow out the side capillary tube and the balance of the gas and liquid will flow over said barrier and out the enlarged tube.

**2.** The structure of claim **1** wherein the capillaries have a diameter of 0.1 to 1 mm. and wherein the length of the gas-liquid contact capillary tube is from 10 to 10,000 times the diameter of the contact capillary.




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