[Contribution from the Chemical Laboratory of the Johns Hopkins University]

THE REMOVAL OF GASES FROM LIQUIDS

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Received June 24, 1930   Published September 5, 1930

The complete removal of gases from liquids is essential in some research problems, and is a matter presenting considerable difficulty. Although the distribution coefficient of many gases between the vapor and liquid phases of the solvent is of such magnitude as to indicate that practically all of the gas should be removable by sweeping off a comparatively small amount of the solvent, the establishment of equilibrium is usually very slow; the rate of liberation of dissolved gas is dependent on the extent, and particularly on the rate of renewal, of the liquid–vapor interface. Periodic removal of the vapor above a liquid standing in a bulb is a very tedious method of accomplishing gas removal, since diffusion of the gas to the liquid–vapor interface is slow. Stirring the liquid, especially if it can be spread out in a large thin film, accelerates the process considerably. This can be accomplished in an apparatus previously described.[1]

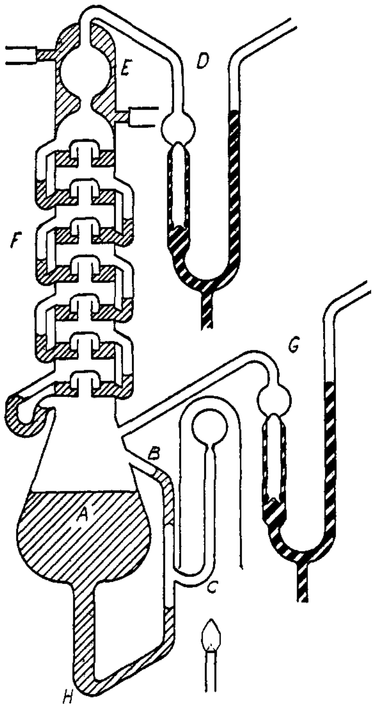

Fig. 1.

In another apparatus devised by the author, and shown in Fig. 1, the removal of air or other gas from any liquid of appreciable vapor pressure may be readily accomplished. The liquid is contained in A; most of the gas in the apparatus is pumped out through D. Then, with D closed, a small flame is placed under the side tube C, and cold water is circulated through the jacket E. The vaporization of the liquid in C carries a succession of slugs of the liquid up the tube B, splashing it against the opposite wall and permitting it to flow in a thin film down the wall, back to the bulk of the liquid. This action gives constant circulation and stirring of the liquid and active motion of the liquid–vapor interface.

C is sealed in somewhat above the center of B, so that even if B is for a moment completely filled with liquid, no opportunity is offered for vapor to push back to or past H. The liquid level in A must of course be somewhat above the junction of B and C. The constriction and bulb at the top of C, and jacket around C, are to keep this tube ever from filling completely with liquid, either by distillation or splashing; the heating of a nearly gas-free liquid at any considerable distance from a liquid–vapor interface leads to irregular, practically explosive, boiling and possibly disastrous bumping.

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[1] Taylor, This Journal, 50, 2937 (1928).

When sufficient condensation has taken place in E to give overflow from all of the sections of the column F, the latter functions like any fractionating column. The mixture of gas and vapor passes up the column, bubbling through the liquid on each plate, and the only way for gas to pass from any given section to the one below is to return in solution in the overflow. The partial pressure of gas (and hence its solubility) is low; the volume of condensate, compared with that of the vapor forming it, is very small. Accordingly a state of equilibrium will finally be reached at which virtually all of the gas in the system is in the top of the column, since the ascending vapor in each section will be relatively richer in gas than the descending liquid; the slowly descending liquid is stripped of its gas content by meeting in counter-current the ascending practically gas-free vapor. It is evident that when equilibrium as to gas content is reached, the liquid in A will be nearly gas-free even when the partial pressure of gas at the top of the column is quite appreciable. Incidentally, a rough estimate of the amount of gas present may be made by comparing temperatures at A and at the top of F; for obvious reasons the presence of gas will lead to a higher temperature at A, while when the gas is nearly all gone the slight difference due to the head of liquid through which the vapor must bubble is not appreciable to the touch.

The gas is removed as it accumulates, together with some of the vapor, by opening periodically the trap D for an instant to a pump or to an evacuated space of some five or ten times the volume from the top plate to the trap. Active bubbling up the column, persisting for some time, follows this operation if gas has accumulated to any extent.

Trap G communicates with the apparatus in which the gas-free liquid is to be used; at the proper time the receiver is chilled, and when G is opened the liquid distils over. Or, of course, the unit itself may be employed as a reservoir for the gas-free liquid.

The advantages of this over the apparatus previously described[1] are that with the same amount of stirring the gas–vapor mixture need not be removed so frequently; if equilibrium is established before each removal, the same number of removals carries off a far larger fraction of the gas; hence, though the loss of liquid in complete degassing is small in either case, it is less with the present device; and the slight chance of carrying air in mechanically with the flowing mercury of the former apparatus is obviated with this one. Furthermore, even if there should be a minute leak admitting air into the system, it would still be possible to withdraw a practically air-free vapor through G, as the air would be carried out of A up the column as it leaked in.

On the other hand, the present apparatus cannot be used as well as the former if it is necessary to maintain the liquid at a constant temperature during the degasification.

A device of the present sort has a number of uses. (1) At this University it has served for the preparation of air-free water, benzene and toluene, used in several researches.

(2) The author has employed it in a study of the retention *in vacuo* of gases by soluble salts. In this instance air-free water was admitted, by breaking a septum with a magnetically operated hammer, to an evacuated tube sealed on at H containing the salt under investigation; after the salt had dissolved, the solution was degassed as described above; the gas liber-

ated, together with some water vapor, was pumped off at D for measurement and analysis.

(3) It could evidently be used to determine the solubility of gases in liquids, by admitting the saturated liquid into the evacuated apparatus and pumping off and measuring the gas liberated.

(4) Adsorption of gases by soluble materials could be determined by a method similar to that used in (2).

A modified Töpler pump, embodying the features of the McLeod gage, has been found convenient in handling small amounts of gases, as in (2). Such an apparatus as shown in Fig. 2 may well be used in any case, such as (2), (3) and (4), when the amount and nature of the gas evolved are of interest.

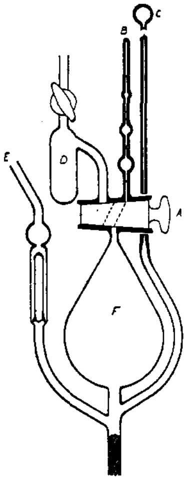

Fig. 2.

By raising the mercury, gas that has entered at E is compressed into the calibrated tube B up to one of the graduations, and the pressure is determined by comparing mercury levels in B and C. (Condensation of solvent vapor occurs in both B and C, so that vapor pressure is canceled in making this measurement.) If more than one stroke of the pump is to be made, condensation of vapor in F must be avoided, as otherwise re-vaporization on the down-stroke will leave practically no difference in pressure between E and F. This is easily accomplished by warming the mercury; most of the condensation then takes place in B instead of in F, and when A is closed the down stroke leaves a vacuum in F.

If a mixture of gases is present, it may be analyzed as follows. The total amount of gas is first determined as above; then by suitable manipulation of A and the mercury level, the gas is transferred to D, which is evacuated and contains an absorbent for one component of the mixture. When absorption is complete, the residual gas is pumped back into B and re-measured. D may then be opened, cleaned out, charged with another absorbent, and pumped out; the next constituent is then determined in a similar manner, and so on.

Summary

Description is given of apparatus and method for removal of gases from appreciably volatile liquids.

Some uses, actual and potential, for this device are listed.

Description is given of apparatus and method for pumping off, measuring and analyzing small amounts of gas, in absence or presence of condensable vapors.

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