AN ABSORPTION BULB FOR DETECTING VARIOUS GASES

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Measured by modern standards, some of the older tests in analytical chemistry are very low in sensitiveness. For example, the test for CO_3^{--} ions, in which CO_2 is detected by a drop of $Ca(OH)_2$ solution on a stirring rod, is satisfactory only in cases of high concentrations. Even when the CO_2 is evolved in a closed system and the gas bubbled through $Ca(OH)_2$, the sensitiveness varies, depending upon conditions. The capacity of the system, the rate of bubbling, the temperature,—a number of circumstances affect the sensitiveness of the test. In order to provide an apparatus as compact as possible, an absorption trap (Fig. 1) has been devised, using the principle of the Liebig bulb. The speed of the reaction may be increased by direct heating or by placing the test tube in a beaker of hot water. This apparatus has been found to be very efficient, and concentrations of CO_3^{--} as low as 0.03 milligram per milliliter may be detected.

In increasing the sensitiveness of the test, interferences by other anions are magnified. The interference of SO_2 from sulfites and thiosulfates was to be expected, and was obviated by adding $KMnO_4$ to the reaction vessel. But borates were also found to interfere, and even oxalates when present in high concentrations. The complication by borates is due to the volatility of boric acid in steam. Oxalic acid, on the other hand, is generally assumed to be nonvolatile. There is a loss when oxalic acid solutions are evaporated to dryness, but it has been assumed that it is due to sublimation of the solid, and not to distillation of the solution. The present work shows very definitely that solutions of oxalic acid of concentrations greater than 0.75 molar can be made to give off white

TABLE I—SENSITIVITY TESTS WITH ABSORPTION BULB

Ion	Reagent	Gas System	Absorbent	Sensitivity (mg./cc.)
CO_3^{--}	H_2SO_4	$Zn + H_2SO_4$	$Ca(OH)_2$	0.03
$C_2O_4^{--}$	$KMnO_4 + H^+$	$Zn + H_2SO_4$	$Ca(OH)_2$	.01
SO_3^{--}	H_2SO_4	$Zn + H_2SO_4$	$BaCl_2$-$KMnO_4$	.1
S^{--}	HCl	$Zn + HCl$	$Pb(C_2H_3O_2)_2$	.1
I^-	Fe^{+++}	$CaCO_3.MgCO_3$	Starch	.01
Br^-	$KMnO_4 + H^+$	$CaCO_3.MgCO_3$	CCl_4	.1
BO_3^{---}	H_2SO_4	$CaCO_3.MgCO_3$	Turmeric	low
As	$Zn + H_2SO_4$	$Zn + HCl$	$HgCl_2$	.01

fumes of $H_2C_2O_4$ at slightly above 100°, especially if they are boiled vigorously. As an extreme case, arsenious acid may interfere with the carbonate test if the boiling of the solution is prolonged.

These interferences can be obviated by a low temperature distillation of CO_2, since the vapor pressures of boric and oxalic acids are negligible at room temperatures. This low temperature distillation can be accomplished by sweeping the CO_2 out of the reaction mixture by means of an inert gas. For example, CO_2-free air may be passed through the reaction vessel. But a simpler method is to introduce a small piece of metallic zinc, and let the hydrogen formed sweep out the CO_2. This removal may be hastened by placing the tube in a beaker of hot water. As long as the contents of the tube do not boil, none of the interfering acids seem to come over.

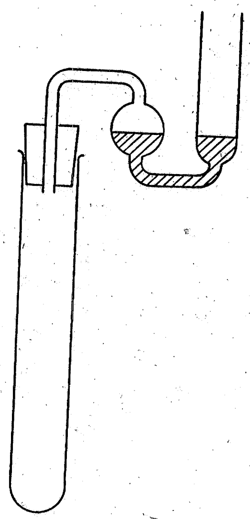

Fig. 1.—Absorption bulb for gases.

In view of the utility of the absorption tube in detecting CO_2, it seemed that its use might be extended to other tests (Table I). The SO_2 from sulfites and thiosulfates may be absorbed in an acid solution containing $KMnO_4$ and $BaCl_2$, bleaching the permanganate and precipitating white $BaSO_4$. The tube may also be used for the detection of sulfides, using $Zn + HCl$ as the solvent for the material and $Pb(C_2H_3O_2)_2$ as an absorbent for the H_2S evolved. This test is sensitive, but does not seem to have any advantage over the usual procedure with $Pb(C_2H_3O_2)_2$ paper. Tests for iodide, bromide and chloride may be made by treating the solution with suitable oxidizing agents and dissolving the liberated halogen in carbon tetrachloride or iodide-starch mixture. Calcite, or dolomite, or other slowly-dissolving carbonate is introduced into the tube to furnish the inert gas required in the distillation. As seen in Table I, these tests have no advantage in sensitiveness over the usual procedures. Borates in HCl solution may be distilled and absorbed in turmeric solution, but again the test has nothing to recommend it. On the other hand, the new apparatus is wonderfully suited for the modified Gutzeit test for arsenic, in which a strip of paper impregnated with murcuric chloride is placed in the open arm.