

tains 100 parts of acetone to 36.3 parts of nitrotoluene by vol. or a total volume for the two constituents of 136.3. The solution itself has a volume of 139.6. Hence in this case, though the specific gravity of the acetone is increased by addition of the solid, the volume of the solution is greater than the combined volumes of its constituents.

A mixture of acetic acid and water (sp. gr. at 20° —1.0680) was saturated at 20° with *p*-nitrotoluene. This solution contained 3.40% of nitrotoluene and had a specific gravity at 20° of 1.0708. This solution contains 2.9 volumes of nitrotoluene per 100 volumes of the acetic acid water mixture, but its actual volume calculated from its specific gravity is 103.2 volumes, a gain of 0.3 volume. It appears from these tests that, though *p*-nitrotoluene does not always lower the specific gravity of the liquids in which it dissolves, yet the tendency is for the solution to occupy a greater volume than the combined volumes of its constituents.

o-Nitrotoluene, a liquid having a specific gravity at 20° of 1.1650, was dissolved in carbon bisulfide and the specific gravity of the solution determined. Its specific gravity was 1.2060 and it contained 99.3 parts by weight of *o*-nitrotoluene to 100 parts of carbon bisulfide. This is 107.8 volumes of *o*-nitrotoluene to 100 volumes of carbon bisulfide, a total volume of 207.8. The corresponding volume of the solution calculated from its specific gravity is 209.1, an increase of 1.3 volumes.

From the determinations of the specific gravity of solutions of sulfur in carbon bisulfide made by Macagno and given in the tables in the Chemiker Kalender, it appears that a solution containing 37.2% sulfur in carbon bisulfide (sp. gr. at 15°—1.271), has a specific gravity of 1.391. Assuming that the sulfur used had a specific gravity of 2.07 (the value given in the Chemiker Kalender for octahedral sulfur) this would be 22.8 volumes of sulfur in 100 volumes of carbon bisulfide. The volume of the corresponding solution calculated from its specific gravity would be 125.4, an increase of 2.6 volumes.

Thus it appears that the tendency of both carbon bisulfide and *p*-nitrotoluene is to increase the volume of solutions in which they are constituents, though the specific gravity of the solution is not always less than that of the pure solvent. In the particular case where the solution contains both of these substances, this tendency results in an actual lowering of specific gravity.

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SOME NEW FORMS OF LABORATORY APPARATUS.¹

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In the following paragraphs are described several new forms of apparatus

¹ Paper read before the Cornell Section of the American Chemical Society.

tus that have been designed in connection with various research problems during the past year. These devices include: (1) a gas washing bottle with circulating device; (2) a modified Soxhlet extraction apparatus with accessory distilling apparatus; (3) a modified Schiff nitrometer; (4) a yellow glass phosphorus pipet; (5) a telescope burner.

1. *A Gas Washing Bottle with Circulating Device.*—The wash bottle shown in the accompanying sketches (Figs. 1 and 2) differs from those of the usual form in that it has a greater capacity for the absorbent, and is provided with a bell fused over the inlet tube.

The gas enters in the usual manner through the inlet tube, but is unable to escape directly because the inner tube, by capillary attraction, has become filled with liquid. The pressure then rises several millimeters

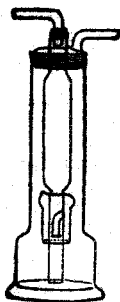


Fig. 1.

until the capillarity is overcome, and the liquid in the inner tube is thrown out into the bell with considerable force. The gas is now free to pass from the inlet tube into the bell, until the level of the liquid has risen and again sealed the inner tube. The operation just described is then repeated. Since the inlet tube reaches to the bottom of the vessel, the liquid that enters the bell through the inner tube is drawn up from the bottom. In this way a continuous circulation of the entire mass of absorbent is obtained.

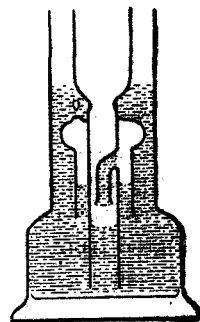


Fig. 2.

The liquid thrown out from the inner tube by each pulsation strikes the wall of the bell, and moistens it. As the gas impinges against this surface, which is thus continuously moistened with fresh absorbent, a very efficient absorption is effected. Since the gas is required to escape slowly through the small opening at the upper end of the bell, a part of the gas always remains until the next quantity is introduced. In this way, ample time is allowed for complete settling of the spray produced by the injection. After escaping from the bell the gas rises in small bubbles through a layer of absorbent in the outer compartment of the apparatus, and leaves the wash bottle through the usual outlet tube.

Among the chief advantages of the new gas washing bottle are its simple and convenient construction, its low resistance to the current of gas, and the possibility of employing a comparatively large volume of absorbent without increase of the pressure. The automatic circulation, moreover, insures complete utilization of the absorbent.

2. *A Modified Soxhlet Extraction Apparatus with Accessory Distilling Apparatus.*—The chief disadvantage of the usual stationary extraction apparatus lies in the fact that the condenser is so connected with the ex-

traction vessel that it must be removed after each extraction, in consequence of which it is not possible to connect the condenser rigidly with the water supply. A further disadvantage is the necessity of employing a special distillation apparatus for the recovery of the solvent.

Both of these disadvantages are avoided in the apparatus shown in the subjoined sketch (Fig. 3). The new apparatus differs from the usual form chiefly in that the condenser is eccentrically connected with the extraction vessel. With this arrangement it is possible to provide the extraction vessel with a separate stopper and with an accessory distillation apparatus. The lower end of the perforated stopper *A* is somewhat tapered and is ground obliquely in order that the solvent cannot fall outside of the extraction thimble.

The manipulation of the apparatus is as follows: After removal of the perforated stopper, the apparatus is charged with the material to be extracted, and the stopper is set in place so as to establish direct communication between the extraction vessel and the condenser. When heat is applied to the flask *B*, the vapor of the solvent rises in the usual way into the condenser, from which it returns, after condensation, through the perforated stopper to the extraction vessel, from which it is then intermittently siphoned into *B*.

After the process of extraction is complete, the stopper *A* is turned so as to cut off communication between *C* and *K*, and the stopcock *D* is opened. The solvent now distils from *B* into *E*, which is ground the same as *B* so as to fit the extraction vessel. The siphon *D* is of such diameter as to remain sealed with a drop of liquid as the result of capillarity and thus to afford passage for the liquid, but not for the vapor. After the flask *B*, which contains the extract, has been replaced by the flask *E*, containing the solvent, the apparatus is ready for a second determination.

The chief advantages of the apparatus are as follows:

1. The condenser may be rigidly connected with the water supply, thus avoiding the use of long pieces of rubber tubing.

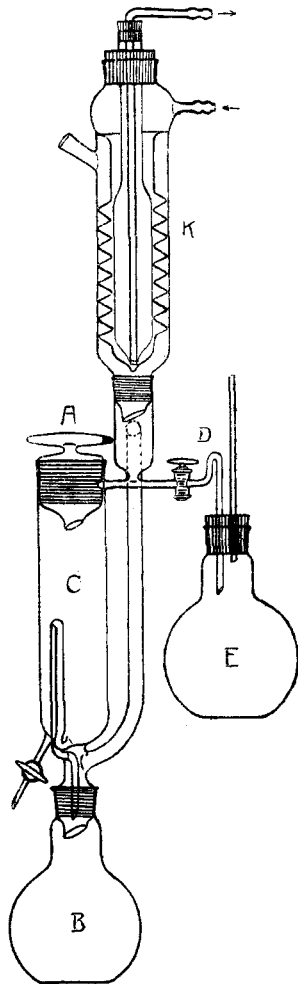


Fig. 3.

2. The extraction, and the recovery of the solvent by distillation, may be accomplished in the same apparatus.

3. The manipulation is rapid and convenient.

4. The danger of breaking is minimized, since the entire apparatus may be securely installed so that only the perforated stopper and the flask need be moved during recharging.

The counter current reflux condenser¹ furnished with the apparatus insures efficient condensation with minimum consumption of water.

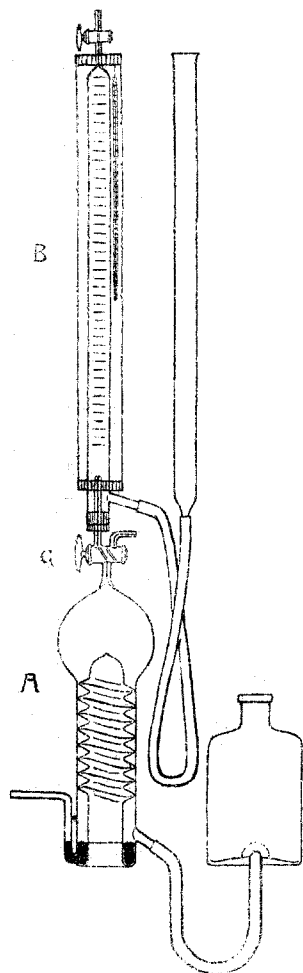


Fig. 4.

3. *A Modified Schiff Nitrometer.*—The Schiff nitrometer, although very convenient in many respects, possesses the disadvantage that the gas is measured over the absorbent, the vapor pressure of which varies with the degree of saturation. This source of error may be avoided in two ways: by drying the gas after it has passed through the absorbent, and measuring it over mercury, or by saturating the gas with water vapor and measuring it above water. The latter method, which is obviously the simpler, has been used by Ferry,² who has designed a nitrometer in which the recently described glass screw³ has been utilized. By using this apparatus, it is possible to avoid a second disadvantage of the Schiff nitrometer, which has a low efficiency of absorption, especially during absorption of large volumes of gas, in which case the absorbent reaches a high degree of saturation toward the end of the experiment.

A more convenient form of this apparatus is shown in Fig. 4. The apparatus consists essentially of two parts: the absorption vessel *A* and the gas buret *B*. The gas enters the absorption vessel through a mercury seal, passes the glass screw in the usual way, and after removal of the soluble constituents collects in the bulb at the upper end of the vessel. The point at which the process is complete may be

¹ *Z. angew. Chem.*, **23**, 2425-26 (1911); *C. A.*, **5**, 1347 (1911); *Chem. Ztg.*, **35**, 1125 (1911); *THIS JOURNAL*, **34**, 285 (1912).

² *Z. anal. Chem.*, **51**, 367 (1912).

³ *Ibid.*, **50**, 175 (1911).

readily observed by simply noting when bubbles no longer rise in the thread of the screw. The two-way stopcock *G* is now turned so as to establish communication with the buret, into which the gas is caused to rise in small bubbles through the water by raising the level bottle of the absorption vessel and lowering the level tube of the buret. The volume of the gas sample, which is by this time saturated with water vapor, is now measured in *B*. It is possible by this procedure to measure the gas with water as the confining liquid, no matter what absorbent may have been employed in *A*. By turning the two-way stopcock so as to connect the absorption vessel with the outside air, and raising the level bottle, it is possible to expel the air that collects at first in *A*, directly from the apparatus, without causing it to pass through the buret.

The apparatus in this modified form has been of considerable use in this laboratory in controlling the air content of gaseous ammonia. For this purpose the absorption vessel is filled with dilute sulfuric acid.

4. *A Yellow Glass Phosphorus Pipet.*—It is a well known fact that the reactivity of phosphorus contained in Hempel pipets of ordinary colorless glass soon undergoes diminution unless well protected from the action of light. This change is of course due to the formation, through the influence of light, of a protective layer of red phosphorus. So far as can be ascertained, no experiments upon the relationship between velocity of transformation of yellow into red phosphorus and the wave length of the light employed, have been described. In order to ascertain whether any such relationship exists, and, if possible, to find a variety of glass that will absorb the active rays and thus retard the transformation, a large number of different kinds of glass have been investigated from this viewpoint. Freshly prepared phosphorus rods, together with a small amount of water,—in order to reproduce the conditions prevailing in the pipet—were sealed into tubes made of different sorts of glass and were exposed out-of-doors to direct sunlight. At intervals of several days one tube of each kind was opened, and the samples of phosphorus were compared. The red coloration appeared first in tubes made of glass transparent to rays of short wave length, very much later in those which absorb these rays, and not at all in those made of black glass. In so far as dependence may be placed upon these preliminary experiments, phosphorus may be regarded as most sensitive toward light rays of short wave-length, as was to have been expected, although not entirely insensitive to rays at the red end of the spectrum. Leaving out of account the black glasses, which could obviously not be satisfactorily used for pipets, the best results were obtained with a brownish yellow glass manufactured by Greiner and Friedrichs. This glass was consequently employed for the pipets, by use of which the action of the ordinary diffused light of the laboratory is so retarded as to be entirely negligible.

The relationship between velocity of transformation of yellow into red phosphorus and the wave length of light will be made the subject of further investigation.

5. *A Telescope Burner.*—In regulating the supply of heat to an apparatus by means of a gas burner two methods may be employed, either the height of the flame may be changed by opening or closing the gas and air supply, or the distance between apparatus and burner may be suitably adjusted. The first of these methods is applicable only within narrow limits because of the striking back of the burner. Recourse is therefore had in general to the method of regulating the distance of the burner from the apparatus by the use of wooden blocks or of some other support for the burner, since the raising or lowering of the entire apparatus is usually difficult, if not impossible.

One burner of adjustable height has been recently constructed,¹ in which metal tubes of different length are screwed to the base of the burner.

This method possesses the disadvantage (1) that the burner may be adjusted only to the heights corresponding to the length of the tubes, and (2) that a change of the adjustment is not possible without shutting off the flame.

These disadvantages may be most satisfactorily avoided by telescoping the tube of the burner as shown in the subjoined sketch (Fig. 5). By means of this apparatus it is possible to regulate the height of the burner from the usual size up to 25 cm. with a single, or up to 30 cm. with a double telescope tube. In the great majority of cases the single or the double telescope tube will suffice. There is, however, theoretically, at least, no limit to the number of tubes used. Since the end of

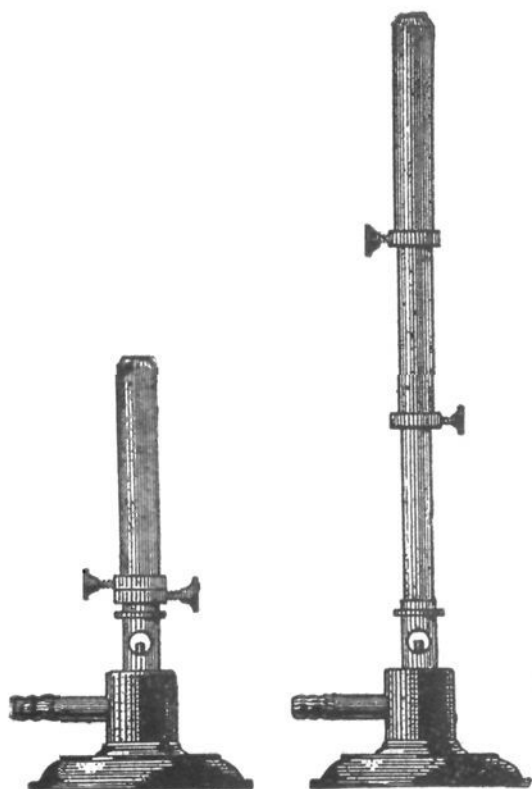


Fig. 5.

the uppermost tube is tapered to the original diameter of the burner, there is no danger that the flame will strike back.

All of the foregoing types of apparatus may be obtained from the firm Greiner & Friedrichs, G. m. b. H., Stützerbach, Germany.

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¹ *Chem. Ztg.*, 36, 78 (1912).