NOTES

A Condenser for Low Temperature Evaporation of Water.—In concentrating serums, gland extracts or any water solution containing protein, it is often advisable to evaporate the water at as low a temperature as possible. The ordinary vacuum distillation does not permit a distilling temperature below 35°. Even at 20° there may be denaturing, precipitation or destruction of biological activity due to combined exposure to temperature and mechanical agitation of boiling.

We have two condensers each containing, as inner tube, a glass tube $1^5/_8$ inch in diameter and 12 feet long (standard 2 inch iron pipe jacket), and these may be cooled by ice water; 12-liter pyrex flasks serve as retorts. While this is very effective, the distilling temperature can hardly be below 15°. The cooling water cannot be used below 0°, by adding salt to the ice, because an ice plug will block the condenser. Moreover, the heat absorption of the 40-gallon ice tank, the circulating pump, etc., make the system uneconomical for small quantities.

We find a 12-liter flask filled with crushed ice a very effective condenser. This eliminates necessity for transmitting heat through a glass or metal condenser surface; moreover, salt may be added to the ice, giving an effective condensing temperature below −10°. Theoretically, to condense 1 g. of water vapor at 0° requires melting 7.5 g. of ice, and in practice more is required. The 12-liter condenser may be insulated with rags or shavings, and there is little heat absorption from the atmosphere, because of its compactness. A specially long rubber stopper prevents danger of sucking in. The stopper has a large inlet for water vapor and a small inlet for vacuum; by making the tubes concentric, one reduces the tendency of the stopper to leak. We have used iron pipe for the tubes. A 2-liter flask with not more than 1 liter to be evaporated serves as "retort," but if the liquid foams badly a larger flask may be used. The connection to the "retort" should be 0.5 inch or preferably more inside diameter, and as short as possible.

The distilling rate is slow. Thus with outside water-bath at 20° and 700 cc. of water in the "retort," about 2 cc. per minute distils (temp. inside "retort," 7°). When first connected the distilling rate is faster and a layer of ice quickly freezes over the surface in the "retort," but this ice melts after the dissolved air is removed. If a slow stream of air bubbles into the "retort" through the usual capillary tube made by drawing out a broken thermometer, the retort temperature is lowered with an increase to about 3 cc. of water distilled per minute, and it is easier to keep ice in the "retort." In any case the distillation slows down near the end, due to decrease in heat transmitting surface, so that about ten hours may be required to reduce 700 cc. to 50 cc. (If a bath temperature of 25 or 30° is permissible the time is much reduced.)

A slow stream of tap water suffices for "heating" the bath of the "retort." We use a Nelson two-stage vacuum oil pump of a capacity of 5 cu. ft. of free air per minute for vacuum, for the high capacity saves many minutes in starting and eliminates the necessity for bothering about leaks around rubber stoppers. If the bath is heated by a flame, one must watch for a rise in temperature near the end of the evaporation, when the rate has slowed down.

For a capacity of, say, two 12-liter flasks in parallel as "retorts," each containing, say, 7 liters of serum, a condenser of a capacity of 25 to 30 gallons is required. Such steel tanks are used commercially for hot water storage, and may be insulated with 6 inches or more of shavings. If used only occasionally one may use a commercial insulated hot water storage tank, as the insulation dries out when not in use, but an insulation which does not easily rot should be selected. On this scale a vacuum pump of several cubic feet per minute capacity of free air should be used. Tubing for carrying the water vapor should be 1.25 inch or preferably more in diameter. The vacuum pump must naturally give better than the vapor pressure of water at $-10°$, under *service* conditions. Naturally one will select a condenser giving a large surface above the ice and salt, to increase the condensing rate. The operator should guard against possible serious injury due to collapse of a 12-liter flask under vacuum.

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Inorganic Lubricants. III. Mixtures of Aqueous Liquids with Non-Reacting Solids.—When water is mixed with a finely divided insoluble powder in the correct proportions a cream is produced which has the power of forming a thin, continuous film over a surface capable of being wet by water, is "viscous" in proportion to the amount of solid present, and so may act as a lubricant between two moving surfaces.[1] For purely temporary inorganic lubrication such a mixture may operate satisfactorily, but because the water quickly evaporates, leaving a non-lubricating cake of solid, lasting lubrication is not obtained.

When, however, a hygroscopic inorganic liquid, such as a concentrated aqueous solution of a deliquescent salt or acid, is employed instead of water, drying out does not take place, the creamy state persists and lubrication is maintained for an indefinite length of time depending on the relation between the average atmospheric humidity and the relative hygroscopicity of

[1] See, for example, various U. S. Patents, such as Colby, 49,983, Sept. 19, 1865; Farkas, 1,253,362, January 15, 1918, and others.

the liquid. I have prepared many such mixtures, using solutions of calcium, zinc and ferric chlorides, antimony pentachloride,[2] sulfuric and orthophosphoric acids and similar substances as liquids, and kaolin, bentonite, talc, graphite, carbon, precipitated silica, etc. (all sieved to pass a 100-mesh screen), as the non-reacting insoluble solids. Bentonite and talc could not be used with acid liquids because a gas-forming reaction takes place resulting in a frothy mixture. Successful lubricants were obtained in considerable variety. Typical examples are listed in Table I.

TABLE I

LUBRICATING MIXTURES

10 g. of solid used in each experiment

Solid	Kaolin	Kaolin	Kaolin	Bentonite	Graphite	Silica
Soln., 50% concn. of	H_2SO_4	$CaCl_2$	$ZnCl_2$	$CaCl_2$	$CaCl_2$	$ZnCl_2$
Cc. taken	6.0	6–6.5	6.5	8.0	6.5	6.5

MATERIALS.—The kaolin was the N. F. V. product; the silica was an anhydrous, amorphous material (electro-silicon); the bentonite and graphite were fine, commercial powders; and the calcium and zinc chloride solutions were prepared from anhydrous C. P. granules.

Of all the mixtures prepared, the best for general use were those of kaolin and of graphite with calcium chloride solution. After three months, although exposed surfaces of these mixtures had fluctuated in liquidity considerably, lubricating films between ground glass surfaces still operated satisfactorily.

Lubricants of this general kind are distinctly limited in usefulness. Their pasty nature permits clogging of small passages; and too great pressure may thin the lubricating film excessively. Nevertheless, they are useful in organic work when protection against leakage of water-insoluble organic liquids and gases is desired; and some of them can be employed with certain inorganic gases (such as those which may attack the common organic lubricants), particularly when it is not important to maintain great purity of these gases. They offer the advantage of being easily and quickly prepared; and the range of possible mixtures is wide enough to permit a considerable degree of choice in both liquid and solid constituents and so enable any specific requirements to be more suitably met.

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[2] Mixture of antimony pentachloride solution and kaolin forms a colloidal material, the properties of which will be investigated.

The Constricted Mercury Arc.—The usefulness of the highly intense constricted mercury arc has been seriously limited by its rapid rate of deterioration.[1] The arc described here has been designed to balance the various factors so that it can be burned at a constant high intensity.

A quartz capillary 2 × 8 × 50 mm. was ground on the side away from the slit until a wall of about 0.5 mm. remained. The other side was ground until a face of about 3 mm. width was obtained, thus reducing the apparent size of the capillary. A very satisfactory polish was then obtained on both sides by cleaning with hydrofluoric acid and heating carefully with a sharp flame. A piece of glass tubing was ground open the length of the capillary and fitted to the rear flat side of the lamp in order to cool with running water. The lower electrode was sealed in with de Khotinsky cement. The lamp was wrapped with strips of cloth so that cooling of the cement and electrodes could be efficient and convenient. A stream of air was passed around the front face. By this arrangement the lamp could be run at 4–5.5 amperes and 20–25 volts per sq. cm. for upward of thirty hours with fairly constant intensity. At the expense of rapid devitrification (fifteen to twenty hours) the intensity can be kept constant by no air cooling on the front face, which prevents the formation of silica patches. A fairly constant intensity can be maintained by an intermittent air stream easily arranged by a long lag relay circuit. In case any deposit forms on the front side it can be removed quickly by stopping the air stream and moderately increasing the amperage.

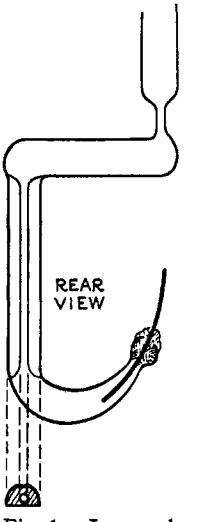

Fig. 1.—Lower electrode is sealed in with de Khotinsky cement. Constriction at upper electrode fits inserted iron wire or is packed with additional pieces to steady the upper meniscus.

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[1] Forbes and Harrison, THIS JOURNAL, **47**, 2449 (1925); Langer and Meggers, *Bur. Stds. J. Res.*, [5] **4**, 711 (1930).