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GLASS STOPCOCKS

By Martin Shepherd

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PREFACE

A glass stopcock is one of the most indispensable tools ever devised by the ingenuity of man to assist in the struggles of the laboratory, particularly in the traffic control of gases. Stopcocks can be satisfactorily employed to meet even the exacting demands of high-vacuum work, provided they are properly made, nicely lubricated, and correctly used. This Circular describes the proper construction, grinding, lubrication, use, and care of stopcocks from the viewpoint of the user. In addition, attention is called to the intriguing possibilities of stopcocks especially designed to perform both usual and unusual operations. Examples of unconventional design are illustrated and described. The Circular should serve three groups: those who have not used stopcocks, but will do so when experimental work makes this necessary; those who have used and discarded them as unsatisfactory; and those who use them regularly.

LYMAN J. BRIGGS, *Director.*

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ABSTRACT

This general discussion of stopcocks is written from the viewpoint of the user rather than the maker. Various types of cocks, their construction, grinding, lubrication, and care are discussed.

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I. INTRODUCTION

The author once made the statement that a well-constructed, nicely ground, and properly lubricated stopcock was capable of maintaining a vacuum *indefinitely*. A colleague ironically agreed that *this* was just the trouble. The word indefinitely was accordingly changed to the expression "over long periods", since our experience had been definite enough.

It seems, however, that both of these views are accepted creeds, and it is most probable that the explanation for the disagreement is that different experimenters have used different materials, and, like everything else in the world, there are good and bad stopcocks, good and bad lubricants, and good and bad techniques of use.

Our own experience has been quite different from that of the old school who stoutly maintain that a stopcock is a located leak. It is perfectly true that even good stopcocks will leak occasionally. But it is equally true that the expectation of this event is not such as to cause great concern. Moreover, if a stopcock *is* leaking, the leak is visible and easily located, which is usually not true of other types of leaks.

We have known a system of stopcocks, numbering from 7 to 18 cocks, to maintain a vacuum (0.1 mm Hg) over periods as long as 2 years, on occasions when apparatus had been set aside. We have known a single stopcock to maintain a vacuum (0.0002 mm Hg) within a 22-liter flask used as a stratosphere sampler for a period of 1 year. Complicated apparatus with over 100 stopcocks has been known to maintain a vacuum (0.001 mm Hg) for weeks and months at a time.

Since good stopcocks perform so satisfactorily in this respect, there is no reason for avoiding them even in exacting work, save in exceptional cases when reaction with lubricants or some especial feature of

the work would interfere. On the other hand, there is plenty of reason to employ them, for they are a useful and indispensable tool, devised by the ingenuity of man to assist in the struggles of the laboratory, particularly in the traffic control of gases. As a matter of fact, their real possibilities have probably scarcely been explored, for by deviating from conventional design, stopcocks can be produced capable of performing all sorts of useful tricks.

The object of this circular will have been accomplished if the imagination of the user is stimulated with respect to special design; if the beginner learns what to look for in a good cock and how to use it, once it is secured; and if a few of the opposite creed can be converted to the use of these efficient traffic managers.

II. TYPES OF STOPCOCKS

In speaking of the stopcock, the inner rotating member (male grinding) is variously termed the plug, the key, the tap, the stopper; the outer member in which this rotates (female grinding) is called the barrel or the shell; the tubes sealed onto the outer member are called leads or outlets. The choice of terms seems to be a matter of personal taste and is therefore not to be interferred with.

The conventional types of stopcocks are well known and their usefulness pretty well explored. These include the so-called "two-way" and "three-way" cocks. There are also the tee and 120° types, the latter being an old friend in our laboratory, rediscovered and patented elsewhere many years after its first appearance. Variations of these types have outlets through the key, making them useful for atmospheric vents, etc.

Concerning the work to be assigned to these standard types, there are two comments. The first is that if a number of cocks are to be assembled to perform multiple routing of gases, the various combinations possible should all be considered—not merely the first arrangement which may occur. The object is to accomplish the desired flow with the fewest cocks.

The second is that we have not found the "three-way," tee, and 120° types always suitable for vacuum work, because as ordinarily made, there is too little ground surface between the bores of the key—and if this condition is remedied, the cock may become rather larger than one would wish. The first failure of the lubricant is opposite the bore of the key, and this may permit leakage *across the bore* from one outlet to the other, even though there is no leakage from the outside to the inside of the system. Such leakage does not ordinarily occur at atmospheric pressures in cocks of this type; but the more exacting demands of high vacuum work have best been met, in our experience, by eliminating these types in favor of the simple oblique bore type with two connecting tubes. As many of the latter type as are necessary for the required distribution of gases are sealed together in a manifold suitably designed for the specific purpose at hand. Although this results in a few more cocks here and there, those present are to be trusted. A good idea of such an assembly is presented in figure 1, which is a section of our apparatus used for the purification of gases by rectification. A drawing of the stopcock used in this apparatus is given in figure 2.

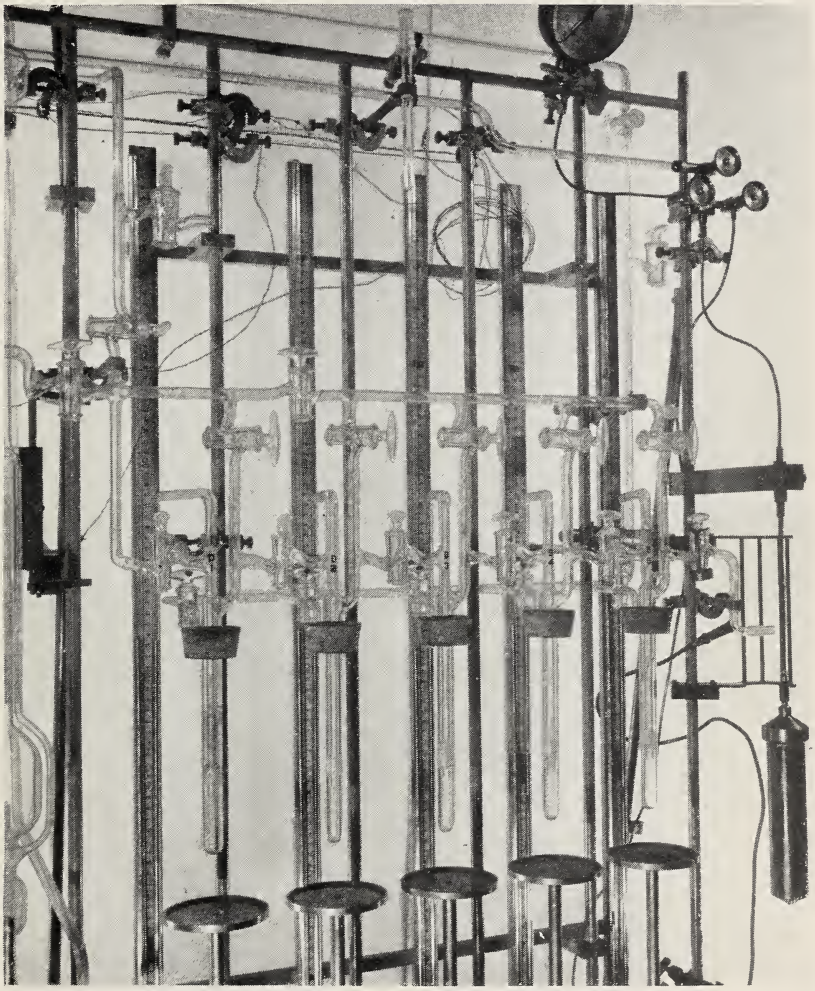

FIGURE 1.—*Section of an apparatus showing assembly of simple oblique-bore stopcocks for vacuum work*

FIGURE 5.—*Stopcock which functions both as a tee cock of a gas distributor and a cock at the top of a bubbling-type absorption pipette.*

In addition to the conventional types mentioned, there are now cataloged several modifications of the type which can be best described by saying that it is merely the top of one arm of the standard glass-stoppered U-tube used for drying and absorptions. This type has two advantages for vacuum work. It permits the use of large bores without excessive size or weight, and it is held tightly seated when evacuated. It will undoubtedly find increasing use in vacuum work, and it is surprising to find such an item absent from older (and many new) catalogs. After all, in the form of our old friend the U-tube, it has been commonly used for years.

Special stopcocks for particular manipulations have been described, but again it is a matter of surprise to find how little the imagination of the experimenter has been stirred by the delightful possibilities here involved. A few examples will suffice to illustrate the latent possibilities, and perhaps serve to inspire a caluclating regard of the reader's own laboratory equipment.

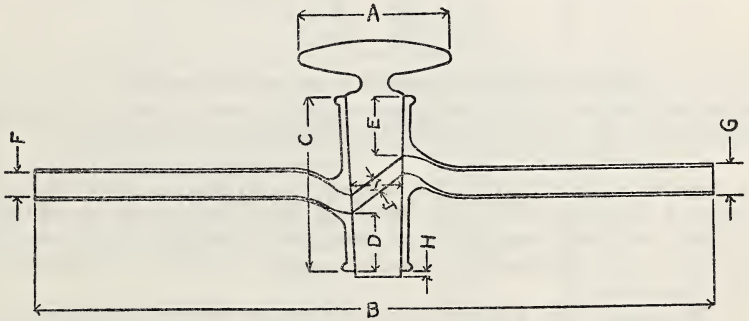

FIGURE 2.—*Drawing of oblique-bore stopcock used for vacuum work.*

Dimensions in millimeters: *A*, approximately 42 to 44; *B*, approximately 200; *C*, approximately 47 to 49; *D*, 15 to 17; *E*, 15 to 17; *F*, 7, *G*, 10; *H*, 1 to 2; *I*, 15; *J*, 3.5.

Key and barrel must be finely ground to match perfectly. Only finest grade of high vacuum grinding is acceptable. Glass should be thoroughly annealed and aged before grinding. Ground surfaces must be free from scratches, chips, or striations. Bores of key and barrel must coincide and be free from chipping. Key to be hollow blown and free from pinholes where insert tube is sealed. Cock to be supplied without lubricant, oil, wax, or other dressing, and free from grinding compound. Key to be wrapped in tissue. Entire cock to be made of the same kind of glass. Put no trade-marks, maker's name, other markings, raised rings or other mold or tool marks upon barrel. Put no pencil marks on grindings. Outlet tubes to be straight when 20 millimeters away from barrel, and on the same horizontal axis. Only finest workmanship throughout is acceptable.

Long ago the author was considerably annoyed by the difficulty in adjusting the manometer of a gas analysis apparatus exactly to the balance point, and by the tragedy which attended too abrupt withdrawal of gas from the absorption pipettes and consequent fouling of the distributor with reagent. After a period of suffering in silence and otherwise, the simple control cock of figure 3 was designed.[1] This device has saved hours of work and worry.

Some years ago, the author and his colleague, J. R. Branham, had occasion to satisfy their curiosity concerning the relative merits of ascarite and a solution of potassium hydroxide employed for the absorption of carbon dioxide in volumetric gas analysis.[2] Out standard apparatus was used, and the ascarite was put into a tube whose

[1] Martin Shepherd, *A simple control stopcock for gas analysis apparatus*, BS J. Research **4**, 23 (1930) RP130.
[2] Martin Shepherd and J. R. Branham, *Critical study of the determination of ethane by combustion over platinum in the presence of excess oxygen*, BS J. Research **11**, 783 (1933) RP625.

two ends were connected to two adjacent vertical leads of tee cocks of the distributor or manifold of the apparatus. This arrangement was used for a long time, but, in the end, the loss of a valuable connection and space, and the turning of two cock handles to do one job, inspired the design of the stopcock pictured in figure 4. It replaces two tee cocks, carries its own absorption tube, and the turn of one handle causes the gas to flow straight through, or to loop-the-loop via the absorption tube.

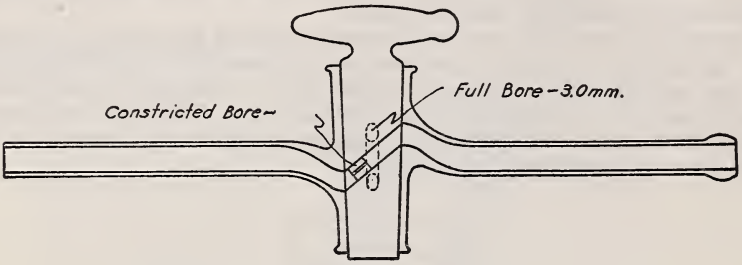

FIGURE 3.—*Control stopcock for gas analysis apparatus.*

Stopcock plug 48 millimeters long, 14 millimeters mean diameter. Must be ground to mirror finish and hold high vacuum. Bore of plug and barrel must accurately coincide and not be chipped. When plug is turned 90° from present position, the full bore (shown dotted) coincides with barrel openings.

A slightly more complicated variety is pictured in figure 5. This replaces a tee cock of the gas analysis distributor and, in addition, the special stopcock supplied with standard absorption pipettes of the bubbling type. The traffic controls possible, for the various positions of the handle, are:

(1) Bypassing the pipette entirely, the gas flowing straight across the distributor.

(2) Connecting the burette to both arms of the pipette and at the same time closing off the portion of the distributor beyond the pipette.

(3) Connecting the burette to the bubbling arm of the pipette, and at the same time closing off the short arm of the pipette and the portion of the distributor beyond the pipette. The last two operations may be performed in the sequence necessary for an absorption, that is, (2)→(3)→(2), which is an essential feature of the design.

Perhaps the most pretentious hybrid devised by the author is pictured in figure 6. This stopcock presented not only an intriguing problem in design, but an unusually interesting problem in construction—a problem which was most skillfully met by the Bureau's chief glassblower, E. O. Sperling. A brief account of its development will serve well to illustrate the latent possibilities in stopcock design.

When the National Geographic Society, in cooperation with the United States Army Air Corps, made their historic stratosphere flights, one of the important scientific problems undertaken was the determination of the composition of air at the great heights attained. This work was entrusted to the National Bureau of Standards. Sampling the air was an important part of the whole problem, because a sample of this consequence should necessarily be certified as being a true sample. Accordingly, a special sampler was designed. This was a flask, to the bottom of which was sealed a small cup. The capillary inlet to the flask was projected through the cup by means of

a ring seal. If the flask were first evacuated to receive the sample, and if, following this evacuation, a column of mercury were placed in the capillary inlet, the presence of this mercury *before* sampling would assure the fact that the sampling cock had not leaked and the flask was *still* evacuated. Furthermore, if the mercury could be withdrawn, the sample next taken, and the mercury then replaced, its presence in the sampling inlet would prove that the sampling cock had not leaked *after* the sample (at reduced pressure) was taken. Finally, if the cock did leak at any time, the mercury would be displaced into the cup, from whence it could not be returned to the sampling inlet by any conceivable manipulation.

The first device designed to accomplish these necessary steps is shown in figure 7. Rather than burden the reader with the explanation of its complete operation, it may be stated that 50 separate operations of 5 stopcocks were required to do the job. This seemed a good deal to ask of an army officer some 21.5 kilometers away from the earth and with other matters to occupy his mind. Major Stevens actually went through this procedure during the flight of *Explorer I;* but when *Explorer II* departed, the sampler taken used the stopcock shown in figure 6. This cock not only performed all the required procedures, but did so by means of four simple 90° turns of the indicating handle.

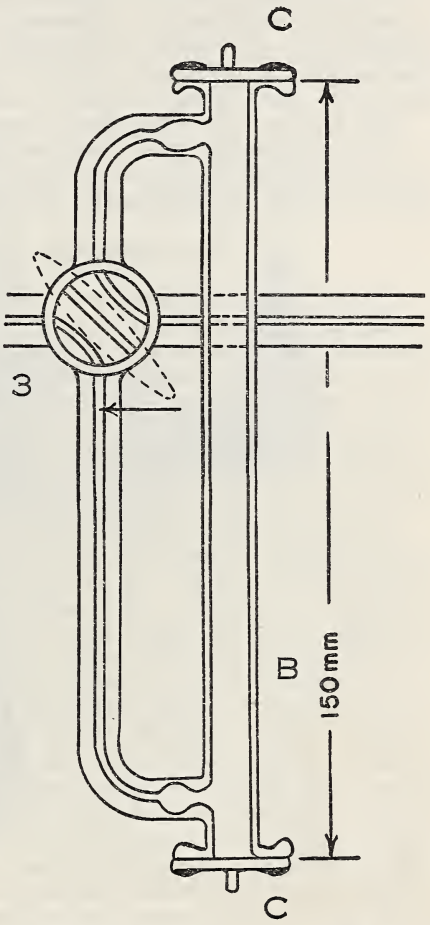

FIGURE 4.—*Special stopcock with absorption tube.*

The operations are simple, and may be described step by step. The cock was made ready for use by nearly filling the hollow handle, *B*, with mercury, leaving a small bubble of air above the mercury in this handle. It was then lubricated and seated. The flask and the hollow key, *D*, were evacuated. Mercury from *B* was then allowed to flow into the capillary, *A*, until a column about 5 cm long had formed. The apparatus was then ready for use.

(1) To take the sample, the cock was first turned from the closed position 90° counterclockwise. This opened the bore, *F*, to the

capillary, *A*. The mercury dropped from *A* to the evacuated key, *D*. *Its presence there upon return of the sampler was evidence that it had been in place in the capillary before sampling, and that the flask was therefore evacuated before sampling.*

(2) The cock was then turned 90° more to connect the diagonal bore, *E*, with the capillary, *A*, and the sampling inlet, *G*. The sample flowed into the evacuated flask.

(3) The cock was then turned 90° further to connect the handle, *B*, with the capillary, *A*. Mercury from *B* was now forced by the air bubble into *A* to form a second seal. *If this seal was in place when the sampler was returned, it afforded direct evidence that the cock had not leaked and the sample had not been contaminated after it was taken.*

(4) The next 90° turn closed the cock.

These examples taken from our own experience can be amplified by scanning the literature dealing particularly with investigations of gases, but the possibilities are far from exhausted.

III. GENERAL CONSTRUCTION

If one has ever examined a well-constructed stopcock, he will recognize an inferior one at sight. Fortunately, good stopcocks are generally available now. This was not true a number of years ago. The ones in our storeroom were not to be trusted for vacuum work, and of those ordered especially, about 20 percent were reasonably satisfactory. At present, we expect and get 98 percent of excellent ones from a special order, and many of the ordinary stock are good. Yet there are still imperfect ones, and it is well to keep certain points in mind when ordering and inspecting.

In general appearance, the cock should be well proportioned. The handle should be large enough to permit easy turning, but not so large that it will interfere with proper assembly or knock elbows with its neighbor in the line. The barrel should be thick enough to insure strength, and smoothly marvered at the ends. The key should project slightly from the small end of the barrel, to permit the use of a key remover [3] in the event it becomes frozen in the barrel. The leads should be sealed onto the barrel with a suitable amount of building up for strength. They should be strictly in line, a point to watch carefully with cocks of the oblique-bore type. A suitable amount of ground surface should be interposed between the outer edges of the bores and the outer edges of the barrel, and also between the bores of the key and the bores of the barrel when the cock is in the closed position. A well-proportioned cock is shown in figure 2. The whole should be carefully annealed. The key and the barrel should be blown from the same kind of glass. We have found the combination of soft key and Pyrex barrel unsatisfactory, and the reverse arrangement has been reported so by others. The differential expansion of the glasses may cause leakage. We have observed this, and it has been noted elsewhere.

We have no definite information upon which to base a choice between a solid and a hollow key. It would seem reasonable to suppose that the latter would cause less trouble if exposed to sudden changes of temperature. Our own preference for it may be a carry-

[3] Two excellent devices for such operations are described by H. W. Bailey, Ind. Eng. Chem., Anal. Ed. 4, 324 (1932).

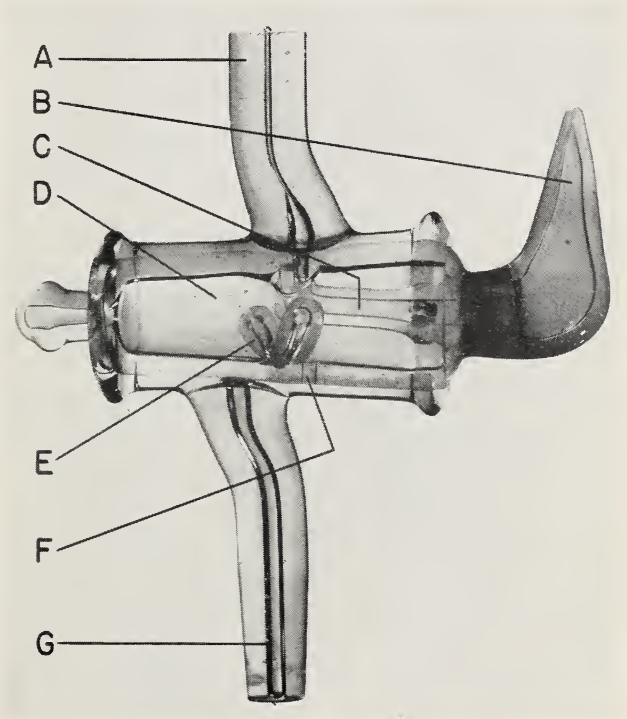

FIGURE 6.—*Special stopcock for stratosphere sampler.*

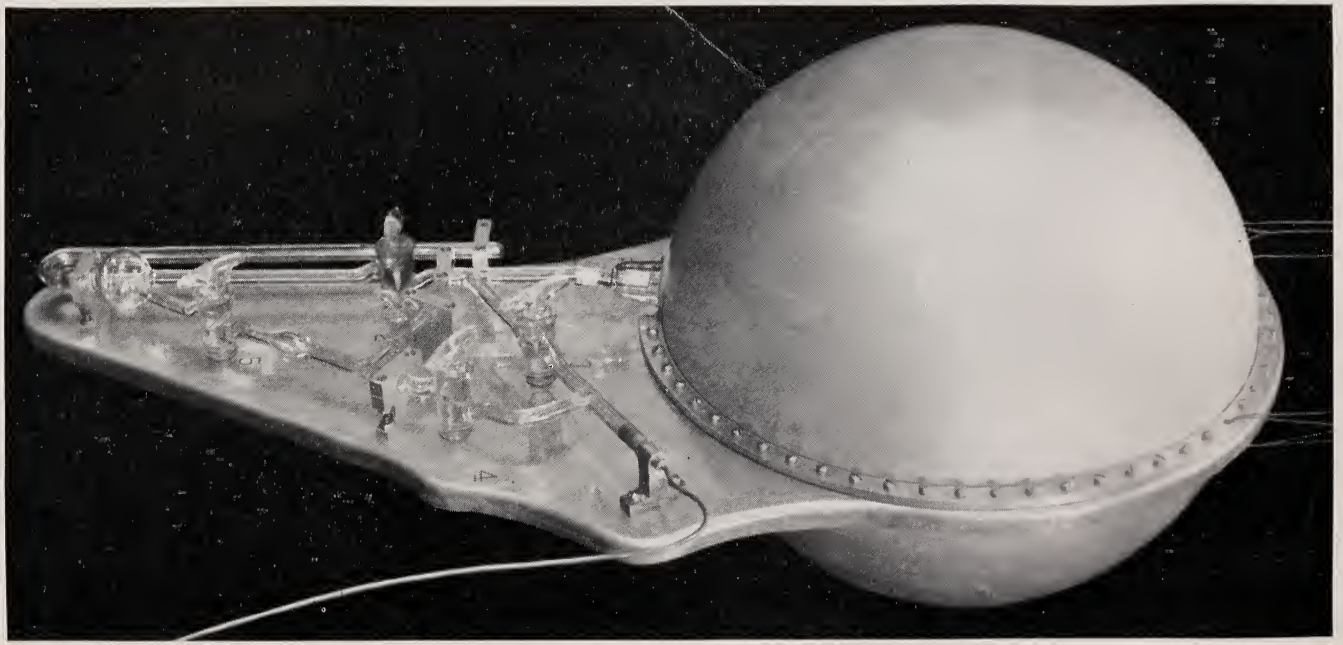

FIGURE 7.—*Former arrangement of the stratosphere sampler.*

over from the old days when hollow-blown cocks were usually good and those with solid keys usually were not—probably because a glass blower who could make a good hollow-blown cock would do the rest of the job well. Excellent cocks with solid plugs are now available, however, and if unusual bores are to be drilled in the key, they are often preferable. One danger concerning a hollow key which is hardly ever realized is the possibility of the insert tube cracking at the seal, and so presenting a source of leakage into and out of the hollow key itself that might be very insidious and difficult to locate. We have had only one such experience from hundreds of such cocks, however. It is probably easier to see the condition of the lubricant with the hollow key.

The last statement brings to mind the importance of having a clear view of the lubricant in the cock, for by the appearance of this one can judge surely whether the cock *may* be leaking or is *certainly* tight. For this reason, the maker's name or trade mark should never be put upon the barrel, but rather on one of the outlets near enough the barrel to insure its staying with the unit, or on the handle, or perhaps be omitted from the decorative scheme. For the same reason, the barrel should be free from striations or tool marks. Moulded raised rings around the barrel are not desirable, since they obscure the view of the lubricant, and usually at the most important point—near the bore.

The bores of key and barrel should register closely, and be free from chipping. If the maker has failed to achieve the proper register, and has then provided a connection by gouging a chunk of glass from the key, return the cock to him for appropriate disposal.

The length of the lead tubes may be relatively unimportant to the user. If the cocks are to be sealed into an apparatus, only as much tubing as is required should be ordered. Shorter cocks are easier to pack and store.

IV. GRINDING

No matter how well the cock may be made in other respects, if it is not well-ground it had much better be discarded at once, merely for the sake of economy. An example will suffice to illustrate this claim. Some time ago there was a job to be done, for which a simple apparatus was built. There were only seven stopcocks in the apparatus, but they were required to preserve low pressures within the unit. There were at the moment no suitable stopcocks in the private reserve (there have always been since). Accordingly, cocks from general stock were used, and they were not the equal of the same stock nowadays. At the end of 1 month, the job remained unfinished, no data of any value had been obtained, and the time had been spent in starting, finding a leak, starting again, finding another leak, and so on. The leaks were always in some of the seven cocks. By the end of the month a fresh supply of good cocks was at hand, the apparatus was reconstructed, and the job (a short one) was finished within the fortnight it should have originally taken. The good stopcocks cost $28.00 (they are cheaper now), the poor ones cost $13.30, the time lost approximated $250.

While the grinding must be *really good*, it is impossible to specify it in quantitative terms. In the old days we required bidders to submit samples, held the best sample as the criterion, and required

the maker to match it as nearly as we could judge. The whole business was rather abritrary, but we succeeded in securing siutable material after some struggle. Fortunately, the picture has changed considerably for the better. The advent of interchangeable ground-glass joints brought grinding technique to the foreground, and now well-ground cocks are available.

Such vague terms as "mirror finish," "satin finish," or "ground for high-vacuum work" have begun to have a real meaning to the maker, and that is all that seems necessary in the end. The last-quoted specification is perhaps the best when ordering.

We have tried various tests of the quality of the grinding. Two of these are: (1) measuring the rate of leakage of hydrogen through the dry grinding by means of the gas interferometer, and (2) measuring the rate of leakage of ether through the dry grinding. Such tests were not successful. From the purely practical viewpoint, we know that the cock when properly lubricated must show no streaks or gas pockets in the lubricant. If it will not do this, there is something wrong with the cock, or the lubricant, or the one who lubricated it; and the chances are the trouble is with the cock. If the cock passes this test, it may or may not be good. If it continues to maintain flawless lubrication upon use for periods of say 1 month, it is good. The reason for this hesitancy in passing judgment is simply that certain lubricants will fill in between relatively bad grindings and make the match of key and barrel appear good; but these lubricants soon wear out and disclose the flaws of grinding.

Stopcocks are generally lubricated when received from the maker. One then has to clean them to inspect the grinding. We prefer to receive them just as they are finished, with keys carefully wrapped in tissue. One can then determine at a glance whether or not the grinding is fine or coarse, free from scratches and striations, and washed clean of grinding compound. Robbed of the privilege of the obscuring lubricant, we have known makers to supply the cocks with a thin film of light oil. This makes the grinding look very good indeed, and we specify that such a dressing be omitted. In time, the user will learn to know a good grinding almost on sight.

V. LUBRICATION

There have been a considerable number of lubricants concocted for ordinary, specific, and high-vacuum uses. Some are designed to resist, as well as may be, solvents or reagents delivered through the cock. Others are intended for general use with gases at or near atmospheric pressure, such as those applied to the cocks of standard volumetric gas analysis apparatus. Some are designed to withstand slightly elevated or lowered temperatures. Those used in high-vacuum work are necessarily heavier than ordinary, for they must maintain a film that does not work out from between the grindings with the application of some pressure, and they are expected to do so for relatively long periods.

Such materials as rubber, parafin waxes, petroleum jellies and heavy oils, ceresin, chlorinated and esterified waxes, lanolin, graphite, sulfuric and phosphoric acids, highly distilled mineral oils and jellies from which the more easily oxidized fractions have been removed, etc., have been used singly or in combination. There have resulted

may salves, unguents, and some good lubricants—but no one that seems perfect.

For high-vacuum work, our own preference is a modification of the old Travers rubber-paraffin-petrolatum formula.[4] The proportions we use are (expressed in parts by weight) 7 parts of freshly milled pale crepe rubber, 1 part of 40° to 42° paraffin, and 6 parts of clear petrolatum. This mixture is stirred continuously in an oven thermostated at 155° C. Formerly this took about 190 hours to cook, but the time has been reduced to about 20 hours by bubbling air through the mixture—an idea that was suggested by Germann's observation[5] that a lubricant of this type lasted well in contact with oxygen *only* when it had been previously treated with this gas to remove the more easily oxidizable substances which tend to "cake out" as gummy solids.

We have endeavored to standardize the preparation of this lubricant, but while the composition of the petroleum wax and jelly may be reasonably fixed, that of the rubber may vary considerably in milling; and again, while the temperature and air input may be fixed, the end point at which the rubber is just properly broken down is hard to judge, and no good way to determine it has been devised. As a consequence, the product is sometimes good and sometimes not.

We have noted one fact of practical value. A sample of this lubricant removed after processing say 18 hours may appear underdone; when placed between thumb and finger, which are then rapidly drawn apart and brought together in a succession of such movements, fine strands of the lubricant form and float away; and in this condition, it tends to form striations across the bore of a stopcock. A sample removed after 20 hours of heating may appear satisfactory; when placed between thumb and finger as above, it forms a long continuous strand which does not float away; in this condition it functions satisfactorily in the cock. But 1 year later, the 20-hour sample may be worthless, and the 18-hour sample satisfactory.

In general we have found it a serious mistake to include in the formula any substance which has a significant vapor pressure, with the expectation of getting rid of it later. Some of it is sure to be retained, and upon evacuation of the apparatus this will form bubbles in the lubricant of the stopcocks. We have known this to happen to such an extent that a mass of froth has completely blocked the bore of the cock at the inlets. (Incidentally, the high-frequency discharge ordinarily used to detect leaks will break down such a froth, but the technique is recommended only as an emergency measure.) In this connection, water is the most general source of trouble, and the lubricant should be protected from moisture. It is also important to remember that the fingers perspire, and if a too convenient digit is used as the applicator, the lubricant is apt to carry with it enough water to cause trouble later on.

In lubricating a stopcock, the following technique is satisfactory:

(1) The cock and particularly the ground surfaces are first carefully and completely cleaned. Useful agents are carbon tetrachloride, chloroform and benzol (all free from residue on evaporation), and a mild soap and water. If the first named does not work, the others, in the order named, should be tried. The ground surfaces must be dry

[4] Martin Shepherd and P. G. Ledig, Ind. Eng. Chem. **19**, 1059 (1927).
[5] J. Phys. Chem. **19**, 437 (1915).

and free from dust or cloth fibers. We find wooden medical applicators, cotton, and pipe cleaners (without bare wire ends) so convenient as to be almost indispensable.

(2) The lubricant is applied to the key by means of a smooth wooden applicator. Two streaks of lubricant are placed parallel to the longitudinal axis, 180° apart and away from the bore of the key.

(3) The key is held well above a soft Bunsen flame until the lubricant has melted to form two smooth semicircular ridges.

(4) When the key has cooled, it is inserted in the open position into the barrel, holding it centrally located as carefully as possible until it has started to seat. Gentle pressure is applied and at the same time the key is gently rotated back and forth through an angle of not over 5°, preferably less. The lubricant now forms V-shaped areas and gradually expels the air film completely.

(5) If the correct amount of lubricant has been applied, a uniformly even film results, and very little excess collects at the bores. If too much has been used, the amount accumulating at the bores will be relatively large and may easily cause trouble by forming a plug or film later on. (We have known such films, hard to see, to prevent proper evacuation and measurement of low pressures.) If too much or too little lubricant has been used, clean the cock thoroughly and try again. Practice will never make perfect at this game, but will considerably increase the number of initial successes. Patience is a distinct virtue, for once the job is done properly, the cock may be expected to stay in service for relatively long periods if the lubricant itself is good.

VI. GENERAL CARE

Some points which might have been included under this heading have been discussed in the foregoing sections. A few may now be added. These are as generally well known in many laboratories as they are little practiced in some of them.

Perhaps this may be putting it a bit strongly, but if a good stopcock is treated about as well as a good glass optical surface, the extra care involved will yield ample dividends. The ground surfaces should always be cleaned and protected from dust during periods when the apparatus is not in actual use. They should be protected from dirt at all times. The lubricant should never be allowed to stand opened to the air. Key and barrel should never be put together dry.

The key should be turned as slowly as the operation in hand will allow. With a heavy lubricant, the key *must* be turned slowly. This is preferably a two-handed task. One hand should be placed at the small end of the cock to afford slight support to the connecting line, the other used to press the key slightly in while turning it very slowly. A quick turn always tends to lift the key from its seat, and introduce striations in the lubricant. A considerable amount of work and often valuable material may be lost by impatience at these moments.

Washington, January 9, 1941.

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