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AN AUTOMATIC SAMPLE COLLECTING VACUUM PUMP[1]

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The mercury displacement pump, which has been generally abandoned for the simple production of high vacua in favor of the more rapid mercury vapor pump, is still of value when the gas pumped is to be collected for further investigation. Numerous automatically operated pumps of the Töpler type have been described.[4] Without discussing the ingenious features of these systems, several of which are quite useful, it may be said that the majority of them have one or more of the following faults: (1) vortices are formed in the bulbs during operation; (2) the mercury entering the pump head when low pressure prevails is not sufficiently controlled to prevent fracture occurring here or in the delivery tube; (3) in order to avoid the first two faults, the operation of the pump is made excessively slow; (4) the mercury delivered from the pump has to be returned to the system by hand; (5) the mercury in the pump is not well enough protected from contamination and oxidation to permit the continued operation of the pump for long periods of time.

A pump designed by the senior author a number of years ago eliminates these and other faults and at least three pumps have been built and put into successful operation. The third pump to be constructed has been in continuous use for the past three years, and its behavior has proved entirely satisfactory. The design and operation of this unit will be described.

[1] Publication approved by the Director of the Bureau of Standards, Department of Commerce, Washington, D. C.

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[4] Beutell and Oberhopper, *Chem.-Ztg.*, **43**, 705 (1919); *C. A.*, **14**, 662 (1920); Bianu, *Bull. sec. sci. acad. Roumaine*, **5**, 58 (1916); *C. A.*, **14**, 1769 (1920); Johnson, This Journal, **34**, 909 (1912); Klein, *J. phys.*, **9**, 44 (1910); *Compt. rend.*, **148**, 1181 (1909); Maass, This Journal, **37**, 2654 (1915); Morley, *Sill. J.*, **47**, 439 (1894); Parnfil, *J. chim. phys.*, **11**, 801 (1914); Porter, *Ind. Eng. Chem.*, **16**, 731 (1924); Stedman, *Trans. Roy. Soc. Canada*, **15**, 93 (1921); Steele, *Chem. News*, **102**, 53 (1910); *Phil. Mag.*, **19**, 863 (1910); Maass, This Journal, **41**, 53 (1919).

The pump control is by mercury displacement in a separate cycle made to operate on the suction of a water aspirator or any of the mechanical

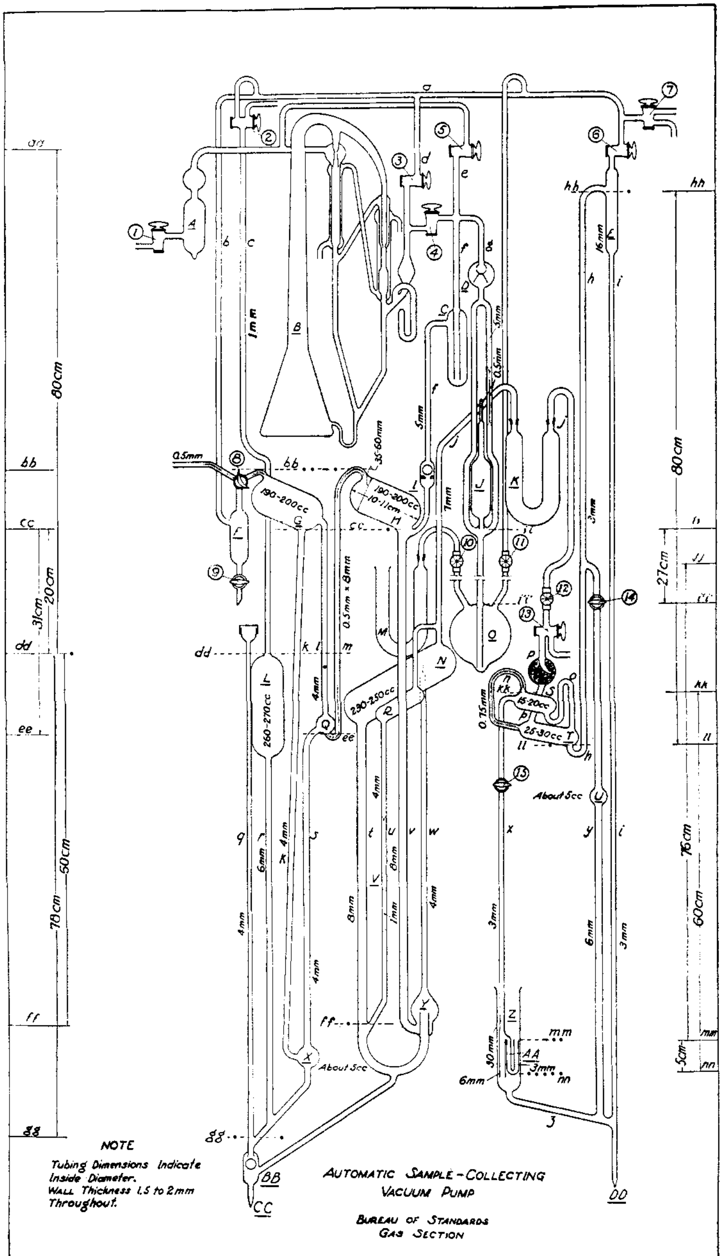

Fig. 1.—Automatic sample collecting vacuum pump.

vacuum pumps commonly on the market. Its operation may be described with reference to the line drawing (Fig. 1) while details of construction and assembly are apparent from a study of the photograph (Fig. 2).

The line drawing has been simplified by the following lettering system. (1) Figures designate stopcocks. (2) Capital letters designate bulbs or other major parts of the unit. (3) Lower case letters designate tubing connecting these parts. (4) Double lower case letters indicate dimensional levels.

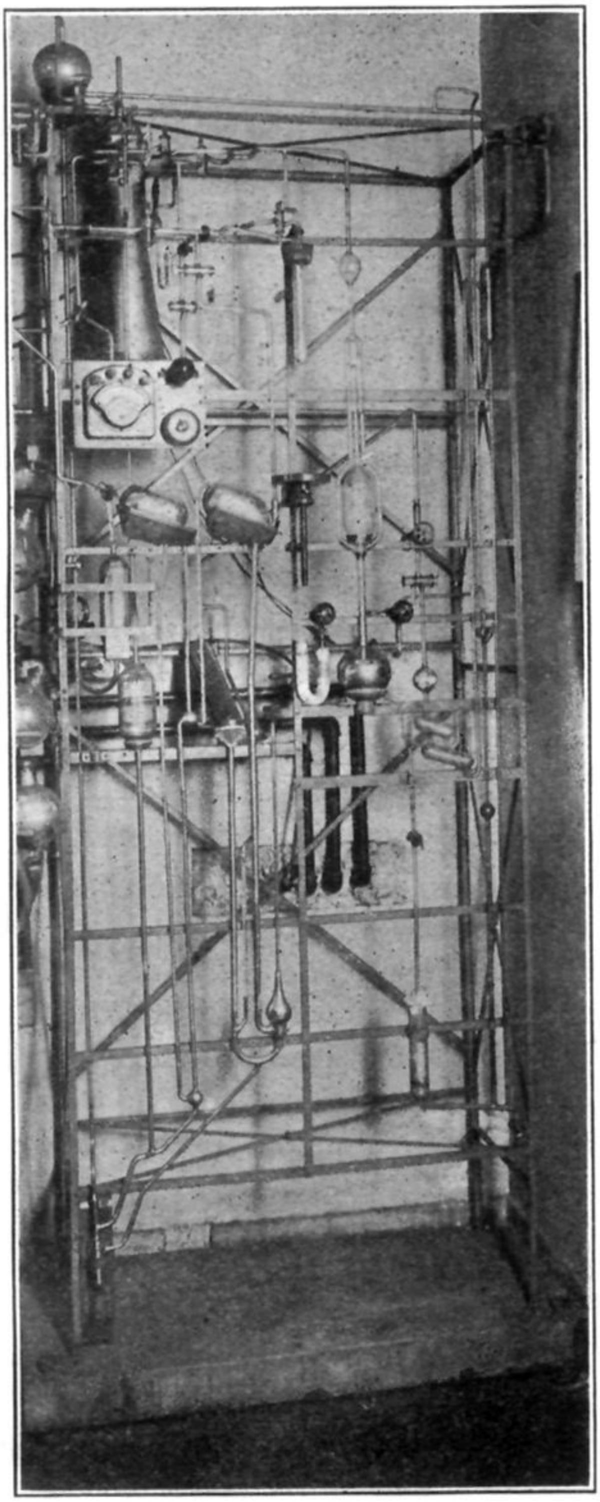

Fig. 2.—Automatic sample collecting vacuum pump.

The space to be evacuated is connected to the pump through cock 1 and drying tube *A* containing phosphorus pentoxide sublimed onto glass wool. The gas may enter the displacement pump directly through 5 or through a mercury vapor pump of the Stimson[5] type located at *B*. The Stimson pump is capable of producing a high vacuum in an apparatus of reasonable size within a few minutes, with the comparatively high backing pressure of 2 to 4 cm. The displacement pump itself has, then, a very small volume to evacuate, that is, merely the connections *f* and *g* and bulb *C* and trap *D*.

In case gases condensable at liquid air temperatures are to be pumped, condensation bulb *C* is used. After condensation, stopcocks 4 and 5 are closed and the condensate is vaporized into *H* and *G* or by-passed to a storage system. This step often saves much of the time ordinarily spent in pumping.

A McLeod gage, represented with its connections by *J*, *O*, *D*, *M*, *g*,

[5] Stimson, *J. Wash. Acad. Sci.*, **7**, 477 (1917).

10 and 11, permits the operator to know the pressure at the inlet *f* of the displacement pump proper.

The controlling or actuating system, which connects to the pump reservoir *N* through tube *j*, comprises the system represented at the right side of the drawing by the bulbs *S* and *T*, reservoir *Z*, interconnecting tubes *h*, *i*, *x*, *y* and *z*, and cocks 12, 13, 14 and 15.

The operation of this control unit may be described by tracing one complete cycle. Its purpose is to apply alternate vacuum and atmospheric pressure to the pump reservoir *N*.

The reservoir *Z* is filled with mercury until the volume above level *nn* is nearly equal to the volume of bulb *S* and tube *x*, and barometric columns in tubes *y* and *i*, including bulb *U*. The mercury must fill *Z* somewhat above level *mm;* the correct amount may be determined by trial. A vacuum from an aspirator or other vacuum pump is maintained through one inlet of stopcock 7. This reduced pressure is communicated through tube *h*, bulb *T*, tube *n* and bulb *S* to tube *x*, drawing the mercury from *Z* into *x*, through stopcock 15, by which its flow may be regulated, into bulb *S*. *Simultaneously, this reduced pressure is communicated through the cycle h, T, p, mercury trap and dust filter P, and tube j to the pump reservoir N.* The mercury seal in U-tube *AA* is not broken until the mercury in *Z* reaches level *nn*. When this occurs, air rushes through *AA* and *x* into *S* and the sudden increase of pressure in *S* causes the mercury to begin to siphon through *o* into bulb *T*. The suction through 7 is immediately interrupted by a barometric column in *h*. *The main reservoir, N, of the pump is now open to atmospheric pressure through AA, x, S, n, T, P, and tube j.* Tube *y* and the lower part of *h* now serve as a siphon to return the mercury from *T* to *Z*, and the time required for this operation is controlled by stopcock 14. When *T* is empty, air enters *h*. The slug of mercury in *h* above *jj* is swept into trap *E*, from which it returns to *Z* through *i*. Air also enters the upper end of *y*. Mercury flows but slowly through the partially closed cock 14. Air flows many times as fast as mercury through the same opening, hence, when air reaches 14 the mercury in *y* and *U* drops suddenly, covering the open end of *AA* and preventing the mercury in *Z* from being carried up in small portions by an "air lift" as it would be if all the mercury returned slowly to *Z*. When *AA* has been sealed over, the starting condition is restored and the cycle repeats itself. The air admitted to *N* is dried in K to prevent fouling of the mercury.

The displacement pump proper comprises the reservoirs *N* and *R* discharging through tubes *t* and *u* and air trap *Y* into pump chamber *H*, which in turn delivers the gas through fall tube *m* and trap *Q* into the sample storage reservoir *G*, whence mercury from *L* may be made to displace the gas through stopcock 8 for delivery to any other apparatus sealed to 8. A system terminating in float valve *BB* and connected through tubes *q*, *r*, *s*, and trap *X* effects the automatic return to the reservoir *N* of mercury delivered on the compression stroke from the pump head *H* to the collecting bulb *G*.

Very pure mercury is introduced into the system through tube *q*, which may then be closed or left open to the air through a drying tube and dust filter. The amount of mercury must be just sufficient to fill the interconnecting tubes *k*, *l*, *m*, *r*, *s*, *t*, *u*, *v*, *w*, trap *Y*, the reservoir *G* with a slight excess in *L*, and the pump head *H* when *N* and *R* are just empty. The amount of mercury should be finally adjusted by trial, operating with needle valve 12 nearly closed to avoid breaking the pump head if too much mercury is in the system. When correct adjustment is obtained, the mercury from *N*, which empties quickly through the large tube *t* just fails to fill the pump head *H* and so avoids fracturing this part. The small bulb *R* contains just a little more mercury than is required to complete the displacement stroke, and discharges slowly through tube *u* containing a capillary stricture *V* of sufficiently small bore to prevent the mercury striking sharply against the pump head.

Before beginning to collect a sample, vacuum applied through cock 7, tube *a*, mercury trap *F* and stopcock 8 is made to draw mercury from reservoir *L*, connected to the atmosphere through cock 2, into reservoir *G*. When *G* is completely filled, 8 is closed, 2 turned to connect *L* to the vacuum and mercury withdrawn from *G* into *L*, leaving a barometric vacuum in *G* into which gas from *H* is discharged. This arrangement permits placing the pump reservoir *N* below *H*, the chamber into which it discharges, and the consequent operation of the pump by vacuum and atmospheric pressure rather than vacuum and positive pressure.

The gas delivered from *H* is effectively prevented from returning by trap *Q*, which also prevents gas bubbles being carried down tube *s*. The mercury which follows the gas through delivery tube *m* automatically returns to the pump through valve *BB*[6] when vacuum is again applied to *N*. The reservoir *N* is made of a tube approximately 35 mm. in diameter and 30 cm. in length, sloped at an angle of about 30° from horizontal to prevent the vortex action invariably observed in the usual reservoir. Bulbs *S* and *T* in the control system must be relatively narrow inclined tubes for the same reason. The delivery tube *m* must be constructed of tubing of 0.5–0.7mm. bore, in order to be completely effective. Larger bores do not deliver the discharged gas properly, leaving films which prevent the obtaining of high vacua. It is perhaps superfluous to note that the construction of *H* and *m* presents an exacting problem to the glass blower, since any surface approaching the horizontal is apt to trap a gas film and ruin the efficiency of the pump.

[6] The float valves *BB* and *I* are solid glass balls ground perfectly spherical and fitting against a 45° ground seat. Floats of this type do not fail as long as they remain clean.

It should be noted that the specified levels indicated by the double letters are quite significant to successful operation of the pumping cycle as well as the control cycle. The level *aa–dd* need not be 80 cm. unless the pump is made to operate on a barometric valve in tube *f*. The glass float *I* is to be preferred at this point. Construction should closely adhere to the other levels specified.

It is apparent that pumping ceases when the pressure in *H* equals that in *G*. At this point the pump runs automatically without further transfer of gas from the pump head to the storage reservoir. The pump is then stopped at the termination of a compression stroke by turning cock 13 to the air, and the pumped gas transferred from *G* by mercury displacement from *L*, cock 2 being turned to the atmosphere for this purpose.

It is worthy of particular comment to note that the pumping cycle contains no stopcocks except the outlet cock No. 8. This is a very important feature, since stopcock lubricant will foul the mercury and make impossible the obtaining of high vacua. The mercury in contact with 8 is carried forward from the pump, and never backward into *G*. No lubricant can therefore be carried into the pumping system. The pump is so arranged that mercury must be added to the Töpler cycle through *q*, never through 8. In actual use, an original filling of pure mercury was perceptibly fouled at the end of two years' operation. If the outlet of *q* and inlet of *N* had been provided with suitable drying tubes, as suggested herein, it is believed the small amount of fouling that occurred over this long period might have been prevented. It is required, of course, that the mercury does not come into contact with a corrosive gas.

For draining and cleaning either the controlling or the pumping system, outlets are provided in the form of drawn-out glass tips at *CC* and *DD*.

The pump was mounted, as shown in the photograph, on a frame of angle iron, the bulbs containing heavy weights of mercury being set into plaster of Paris in suitable metal forms. This type of mounting is desirable when a permanent unit is to be set up. In the event of breaking any part, the whole is easily accessible to a hand torch.

The pump operates readily at a rate of one or two cycles per minute.

Summary

The automatic sample collecting vacuum pump here described has been in successful operation for several years. The pump is an automatic Töpler, which acts as the backing unit to a high speed mercury vapor pump. The automatic control is accomplished by an arrangement of mercury displacement within an auxiliary cycle, which can be operated by a water aspirator or any motor driven vacuum pump commonly on the market. No pressure above atmospheric is required for the operation of the pumping unit. The design incorporates features which prevent

fracture of the Töpler pump chamber and delivery tube. Stopcocks have been eliminated from the pumping cycle and a minimum quantity of dried and filtered air comes in contact with the mercury in the pump reservoir; the mercury is consequently not fouled through contact with lubricants or moist air. The mercury delivered by the Töpler pump is automatically returned to the cycle, while the gas pumped is automatically collected and may be transferred as desired to other units.

The control system is entirely independent of the vacuum pump and may find other applications in which alternating vacuum and pressure are desired.

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SOME PROPERTIES OF COLLOIDAL LEAD

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In the course of the preparation of colloidal lead suitable for clinical use a number of the properties of lead sols prepared by the Bredig method[1] have been studied. The more outstanding of these properties are presented below.

Apparatus and Method.—The apparatus was the same as that described by the author elsewhere.[2] The anode was $^1/_{16}$-inch commercial sheet lead from the National Lead Co. The cathode was a roll of "Pueblo" lead foil from the American Smelting and Refining Co. Baker's "C. P." reagents and Eimer and Amend's "C. P." and "Tested Purity" reagents were used and were not further purified. The approximate currents used at different times are given below, temporary fluctuations up to $\pm 10\%$ of the values given being caused by irregularities in the arc: 1.4 amps., 40 volts; 3.8 amps., 50 volts; 7.0 amps., 60 volts. The temperature range was 10–35°.

Properties.—The colloidal lead was dark gray and fluorescent when first prepared, turning blue-black after being centrifuged or being allowed to stand for a few hours. It was brown by transmitted light. The particles carried a positive charge, as shown by their migration in an electric field. When a current passed directly through a sol, the lead coagulated to tree-like growths on the electrodes.

When protected from air by paraffin seals about 5 mm. thick the sols would keep for from three to nine weeks before coagulating. If, however, they were left with a large surface in contact with the air, they rapidly

[1] Bredig, *Z. angew. Chem.*, **12,** 951 (1898).

[2] Woodard, *Ann. Sur.*, 607, Oct., 1927.