

11. Dehydration of the silica under reduced pressure has no advantages over the common evaporation at ordinary atmospheric pressure.

12. Excessive time of dehydration, *viz.*, 4 hours, possesses no advantages.

13. Excessive amounts of sodium carbonate should be avoided, since the sodium chloride subsequently formed exerts a solvent action on the silica. The best proportions are 4-5 parts sodium carbonate to 1 of silicate. Less than 4 parts of sodium carbonate is frequently insufficient to completely decompose many silicates.

14. The nonvolatile residue has been found to be invariably free from sodium. Pure silica, on fusion with sodium carbonate, subsequently gives no nonvolatile residue.

MADISON, WISCONSIN.

NOTE.

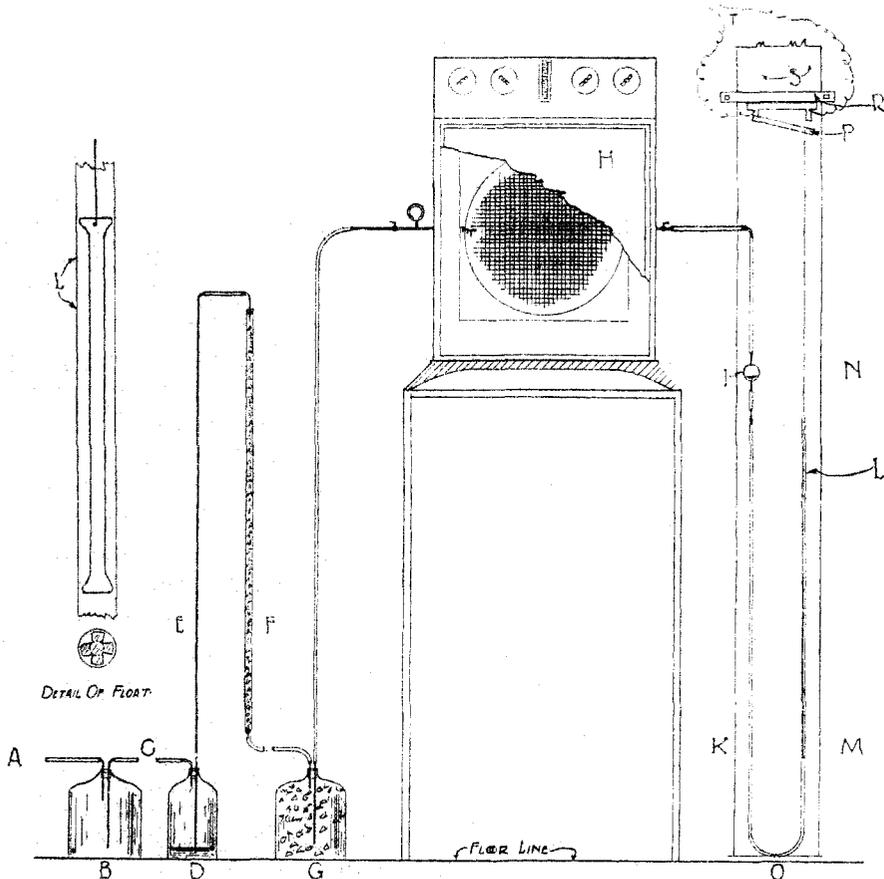
A Simple Device for Regulating the Pump Used in Exhausting a Vacuum Oven.—When operating a vacuum oven it is customary to keep the pump running continuously, since the best ovens on the market will not hold a vacuum more than a few hours, especially at elevated temperatures. With a good pump the initial exhaustion of the oven can be accomplished in a very few minutes. The subsequent leakage of air is, however, so slow that the continuous operation of the pump for days at a time seems a waste of power. The simple device described in this paper can be so adjusted as to start and stop the motor automatically for any desired range of vacuum, thus effecting a considerable saving of power. The materials of which it is constructed are to be found in any chemical laboratory, and very little mechanical skill is required to assemble them.

The oven to which this device was attached is a Freas' Electrically Heated Vacuum Oven connected to a Crowell's Pressure and Vacuum Pump, the latter operated by a one-horse-power motor. When the oven is maintained at 100° temperature and 20 to 60 mm. pressure, the apparatus described below starts the motor 18 to 20 times per hour, allowing it to run about 30 seconds each time. Thus at an average pressure of 40 mm. the pump runs only one-sixth of the time. As stated above, the apparatus may be adjusted for any desired range of pressure, the motor starting at the maximum and stopping at the minimum pressure.

Referring to the diagram, *A* is the tube leading to the vacuum pump. The bottle *B* serves as a trap to catch any mercury that may be drawn out of *D*. The bottle *D* contains mercury, Tube *E* (6 mm. inside diameter) which is about 80 cm. long, projects 1 cm. into the mercury in *D*. When the pump stops, the atmospheric pressure raises the mercury in *E* corresponding to the vacuum, thus closing the chamber. When the pump is running, the air is required to bubble through only 1 cm. of mer-

cury. *F* and *G* are filled with calcium chloride to prevent the water given off by the samples in the oven from contaminating the mercury or the oil in the pump.

To the outlet of the vacuum oven *H* is attached the "U" tube *K L*. This tube, which contains mercury, is supported by the standard *O*. The arm *K* is about 90 cm. long and has an enlargement, *I* (3 cm. in diameter), situated 80 cm. above the lower end. The arm *L* is about 80 cm. long and 12 mm. inside diameter. It contains a glass float, *M*, which is



12 cm. long. This float is in the form of a plunger, with a small bulb at the lower end and small projections near the top. This keeps the float surrounded with mercury and prevents adhesion. The rod *N* connects the plunger *M* with switch *P*, which is an ordinary throw switch on a porcelain back, *R*. This switch is mounted on a block, *S*, which is so made that it can be adjusted by sliding it up and down on the standard *O*. The wires *T* lead to the motor.

When the desired vacuum is reached, block *S* is so adjusted that float *M* will just open switch *P*. As the air leaks into the oven, the column of mercury will raise the float *M* and close the switch, thus starting the pump.

The use of the bulb *I* reduces the pressure required to throw the switch to 25 mm. of mercury in place of 80 mm., the height which would be required were the tube without the bulb.

The above apparatus has been in constant use in our laboratory for two months and has given entire satisfaction.

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THE CONSTITUTION OF THE THREE SYMMETRICAL DINITROPYROCILLS.

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The nitration of pyrocoll, as first reported by Ciamician and Danesi,¹ led to the formation of a dinitropyrocoll of unknown constitution. The hydrolysis of this dinitro derivative yielded a mononitro- α -carbopyrrolic acid in which the position of the nitro group still remained an undetermined factor. In a study of the constitution of each of the three possible mononitro- α -carbopyrrolic acids,² it was found that the method of Ciamician and Danesi was the only practical way of preparing this one acid just mentioned. Eventually, when we were able to establish the position of the nitro group for each of the three acids, this acid became identified as β -nitro- α -carbopyrrolic acid. The constitution therefore of the intermediate product—the unknown dinitropyrocoll—was then open for consideration and in similar manner each of the three possible symmetrical dinitropyrocills may be studied.

The production of this one β -nitro- α -carbopyrrolic acid in the absence of isomers when the unknown dinitropyrocoll is hydrolyzed (a fact noted also by Ciamician), has led us to the conclusion that, during this nitration of pyrocoll, two nitro groups apportion themselves equally and symmetrically upon the two pyrrole nuclei in the pyrocoll molecule. That configuration, therefore, for this dinitropyrocoll, which alone will satisfy this condition and, by hydrolysis, permit of the total conversion into two molecules of β -nitro- α -carbopyrrolic acid, is designated as sym. β, β -dinitropyrocoll.

¹ *Gazz. chim. ital.*, 12, 39 (1882).

² THIS JOURNAL, 37, 2538 (1915).