barrel and the surrounding liquor, but large enough to permit ready flow between them when desired. The pipet is fitted at the top with an ordinary glass stopcock, leaving a mouthpiece of glass about 2.5 cm. above the cock. Its total length is not longer than a stirring rod.

The sample to be analyzed is secured in a beaker, diluted suitably, and indicator added if necessary. The portion is adjusted when possible to require a set amount of standard solution. The stirring rod is introduced and the barrel filled. The pipet stopcock is closed and the standard solution run into the beaker as rapidly as possible from the buret to a slight excess, the buret stopcock being full open until the end-point is signaled. The pipet meanwhile functions as a stirring rod. Half of the withdrawn barrel portion is now drained into the beaker and titration repeated to excess, this time by additions of say twenty drops each. This cycle is repeated with say five drop portions. The rod is then rinsed in the solution by running liquid in and out several times. The final titration in one drop portions is rapid. The color variation or other indication of the end-point is fresh in the memory. Judgment is not strained. It was found even when duplicates were being run that it was quicker and easier to titrate by this method. A medicine dropper arrangement is similar but not satisfactory. It is not precise and not readily cleaned.

Fig. 1.—Diagram of pipet · stirring rod.

If the sample can be adjusted approximately to require a set volume of standard solution the titration (with a set procedure) will always occur in a fixed time.

CONTRIBUTION FROM THE
DEPARTMENT OF PHYSICAL CHEMISTRY
MCGILL UNIVERSITY, MONTREAL

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A New Glass Packing for Laboratory Fractionating Columns

BY C. D. WILSON, G. T. PARKER AND K. C. LAUGHLIN

A highly efficient packing suitable for general use in laboratory fractionating columns has recently been developed in this Laboratory. The packing is a glass modification of the single and double-turn wire helixes which have been used here with excellent results.

The apparatus required for making the glass coils is very simple: a brass (or other metal) rod of 3.2 mm. diameter and about 31 cm. long is equipped to hold a 2–3 mm. glass rod (by flattening a portion of the metal rod and drilling a hole or by slotting the end of the metal rod and bending the prongs to fit the glass rod). The end of a 2–3 mm. soft glass rod is inserted into this opening and heated in a moderate Bunsen flame to softening. The metal rod, held in the right hand at right angles to the glass rod, is then

rotated counter-clockwise, winding the softened glass in a spiral. After starting the spiral, the metal rod should rest on and be supported by the glass rod, and be about 3.2 to 6.4 mm. from the heated tip of the glass rod. By this process, the rod can be drawn to any desired diameter by varying the rate of turning. The space separating the turns is controlled by the angle at which the metal rod is held. With a little practice a uniform glass spiral can be wound having the size of No. 22 B. and S. wire, and about 10–12 turns per cm. This is easily removed from the rod when cool, and is broken up into single and double turns, which are sorted.

Fractionating columns packed with this material (usually 75% single, 25% double turns) have found a wide application in this Laboratory. This packing is far superior to the usual 5 × 5 mm. glass tubes. The flooding tendency is less (higher vapor velocity), enabling more rapid distillation, particularly under reduced pressures. The H. E. T. P. (height equivalent to a theoretical plate) of the new packing as determined by distilling a mixture of benzene and carbon tetrachloride[1] was 1.8 inches (4.57 cm.) in a total condensation, variable take-off column[2] with a packed section of 44 × 1.4 cm. and 2.0 inches (5.08 cm) in a similar column with 40 × 0.8 cm. packed section. The H. E. T. P. with 5 × 5 mm. glass tubes in a similar 40 × 1.4 cm. column was 4.0 inches (10.2 cm.). Thus by the use of the new packing the number of theoretical plates in a column can be more than doubled, while the distillation rate is also increased.

(1) Fenske, Quiggle and Tongberg, *Ind. Eng. Chem.*, **24**, 408 (1932).
(2) Whitmore and Lux, THIS JOURNAL, **54**, 3448 (1932).

STATE COLLEGE, PENNSYLVANIA

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The Homogeneous Reaction between Hydrogen and Fluorine

BY HENRY EYRING AND LOUIS S. KASSEL

It has been reported by Moissan and Dewar[1] that immediate explosions are obtained when fluorine is brought into contact with liquid hydrogen. Since it seemed to us improbable that molecular hydrogen and molecular fluorine could react without considerable activation, we attempted to repeat this experiment. We were prevented from doing so by experimental mishaps; we did find, however, that there is no appreciable homogeneous reaction between the gases at room temperature.

Fluorine was obtained from a generator of the usual type and conducted through copper tubing to the center of a 3-liter flask. Hydrogen and nitrogen from tanks could also be admitted near the center; an exit tube also was provided. The ordinary procedure was to flush the flask out with nitrogen, and then to admit first one, then the other, of our reactant gases.

(1) H. Moissan and J. Dewar, *Compt. rend.*, **124**, 1202 (1894); **136**, 641, 785 (1903).