A SIMPLIFIED LABORATORY FLOW-RATOR

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ABSTRACT

In the investigations taken up on the Catalytic Cracking of Kerosene Oil, the authors felt the need of a laboratory flow-rator which could maintain a reasonably steady and accurate rate of flow of oil during the test run of cracking. Several types of flow-rators described in literature were examined but found unsatisfactory for the purpose. A description of a simplified and fairly accurate all-glass flow-rator of the variable area type, designed by the authors, is given in this paper. The method of calibration of the flow-rator under varying temperature conditions is given. The accuracy of the flow-rator has been verified by actual test-run results and by making a comparison of the material balance. The results indicate an accuracy within 5%.

INTRODUCTION

A large variety of laboratory flow-meters designed to operate on different principles are described in literature. Although meters working on thermo-electric and electronic drop counting principles seem to be interesting, the Mariotte bottle type is found to be suitable for laboratory purposes. Zentner[1] employed an apparatus in which the liquid is siphoned from a Mariotte bottle and delivered through a capillary. An improved form of the above flow-meter has been described by Fabris and Peacock[2] in which, the liquid is used to measure the pressure differential across the capillary.

Many difficulties, however, were encountered by us in testing the Mariotte bottle type, constructed in our laboratories. It was found that considerable time elapsed before the liquid attained definite differential height in the two limbs for a correct reading of a steady flow-rate to be taken. Further, it was difficult to set up the complicated all-glass construction in three dimensions on a wooden frame, and several parts were broken and had to be frequently repaired, necessitating its recalibration every time.

The final design of the flow-meter constructed by us was much simplified as a result of a number of unsuccessful attempts to modify the older types. This is one of variable area type in which the liquid can be made to flow at varying rates by changing the amount of opening of a stop-cock. One of its main features is its all-glass nature without any rubber connection, where

contact with kerosene oil is undesirable. An all-glass constant head device is another important feature of the meter, and the incorporation of an angular scale to measure a specific opening of the stop-cock aperture is decidedly an advantage over the older types. It has, at the same time, been necessary to use a grease for the stop-cocks which should be insoluble in kerosene oil. A mannitol-dextrin-glycerine grease[3] has been found highly suitable.

DESCRIPTION OF THE FLOW-METER AND ITS OPERATION

The design and set up of the meter is shown in Fig. 1. The liquid is siphoned from the reservoir (R) at the top into a wide glass tube (A) and after filling it to a certain level it overflows through the tube (C). At the bottom of (A) is fused a capillary tube (B) the other end of which is fixed to one limb of a three-way stop-cock (D). The liquid, after passing through the restricted aperture of the stop-cock, is carried through its another limb (E) into the reaction tube by means of an extension piece (H). The extension piece (H) can be connected to the flow-meter tube (E) by means of fused standard ground glass-joint (G). An aluminium pointer (P) is mounted by means of a small clamp on the stop-cock plug which moves on a circular brass scale (S).

The details of the stop-cock (D) are shown in Fig. 2. When the plug of the stop-cock is in position (XX') its aperture is just closed completely and hence no flow of liquid takes place. As the plug is rotated the flow rate of liquid through the stop-cock gradually increases and reaches a maximum at the position (YY'). Further rotating the plug in the same direction again gradually reduces the flow rate.

A thermometer (T) is fixed in the tube (A) to indicate the temperature of the liquid. It is necessary to provide a plug of cotton-wool at the end of the siphon tube in the reservoir (R) to avoid dust particles coming in. The flow meter is fixed on a vertical wooden board which, in its turn, stands on a horizontal base plate. The meter can be adjusted to an exactly vertical position by means of levelling screws (S_1 and S_2).

To start with, the wooden board is levelled with a spirit-level by adjusting the levelling screws (S_1 and S_2), and the stop-cock (D) is kept in closed position (XX'). The stop-cock (F) is opened to the desired extent so as to cause a continuous over-flow in thin stream through the tube (C), the limbs (B) and (E) being previously filled. When starting a test run the pointer (P) is brought quickly to the desired angle corresponding to a known flow rate (obtained from calibration curve, Fig. 4), the moment of thus setting the pointer being noted by means of a stop-watch. So long as the oil head in (A) is constant, *i.e.*, at the overflow level, and the room temperature steady

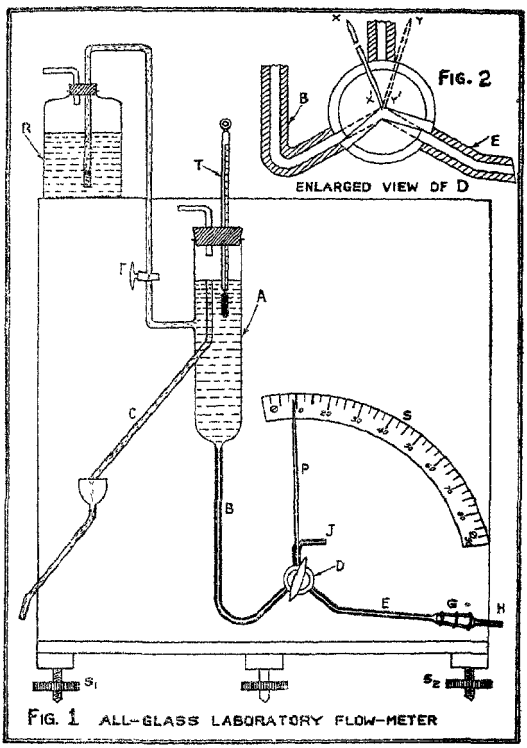

FIG. 1 ALL-GLASS LABORATORY FLOW-METER

for a particular angle indicated by the pointer, the flow rate remains practically constant. This is ensured further by keeping oil at a sufficiently high level in the reservoir (R) by occasionally adding fresh oil to it. The total amount of oil fed into the reaction chamber is calculated by the total period of run read from the stop-watch. After the desired run, the pointer is swiftly turned on to the position (XX') when the flow stops.

The communication through the stop-cock between (E) and third limb (J) helps in drawing out the liquid still remaining in (E) swiftly at the end of a run by the application of suction at (J). As a matter of fact, when the test run is still in progress for a predetermined period, a suction is created

in the line (J) by connecting it to a water jet aspirator, so that by turning the plug in the position to establish a communication between (E) and (J) the liquid is swiftly drawn out of the tube (E). This technique avoids further undesirable oil drops trickling down and coming in contact with the catalyst after a specified period of run.

The extension piece (H) fitted into the reaction tube is shown in Fig. 3.

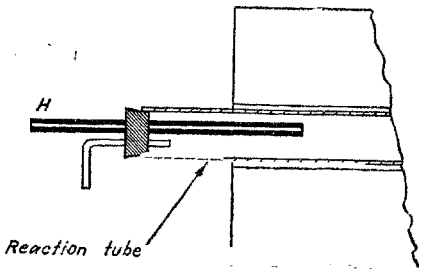

FIG. 3. Vapourising Furnace

CALIBRATION OF THE FLOW-RATOR

The flow-rator is calibrated by weighing the amounts of liquid collected in tared test-tubes, for known intervals of time, at different positions of the pointer. A graph is drawn showing the angle of the pointer and the corresponding flow-rate as shown in Fig. 4. It has already been pointed out that the stop-cock (F) (Fig. 1) should be so adjusted that a continuous overflow of the liquid is maintained irrespective of the rate at which the liquid is drawn out through (E). This is necessary obviously to keep the liquid head in (A), causing the flow, constant.

Due to varying temperature conditions, as it usually happens the temperature of the liquid in the reservoir (R) may not be constant during the entire period of a test-run. As such there is a chance of flow-rate being altered due to change in density and viscosity of the liquid. In such cases the actual rate of flow is taken to be that at an average temperature during the entire period. Hence it has been found necessary to calibrate the flow-rator at different temperatures combining with different openings of the stop-cock as far as possible.

Advantage has been taken of the slowly changing laboratory temperature from morning till evening, and readings of flow-rate at different positions of the pointer have been taken for short intervals to avoid any notable

fluctuation in temperature conditions. This has ensured a constant temperature of the liquid during short intervals of taking readings. The calibration curves for the sample of kerosene oil employed in this investigation at three different laboratory temperature conditions are shown in Fig. 4.

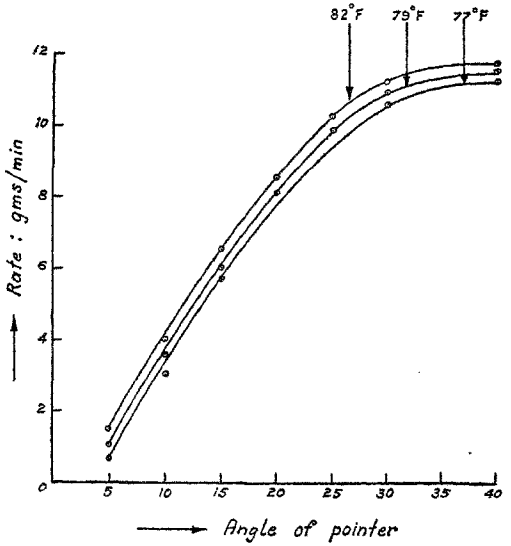

FIG. 4

TESTING THE ACCURACY OF THE FLOW-METER

Several calibration figures have been checked by actual test-runs and they have been found fairly accurate. For a desired flow rate condition to be maintained during a test-run, corresponding to a definite room temperature, the particular point from the calibration curve is chosen relating to a definite angular position of the pointer. Test-run is carried out by operating the flow-rator pointer as described above. The cracked products of the oil are all collected, the liquid by condensation in ice-cooled container and the gas in a gas-holder. The gas is analysed and its average molecular weight is calculated from its composition. The condensed liquid portion is weighed and the carbon deposited on the catalyst is burnt with oxygen and weighed in the form of carbon dioxide. The sum total of weights of liquid,

gas, and the deposited carbon is then compared with the flow-rate indicated by the flow-rator. A few typical results are given below, showing the accuracy of the flow-rator.

	Expt. I	Expt. II	Expt. III
Average temperature of oil ..	77 °F.	78 °F.	78° F.
Pointer angle ..	5°	10°	15°
Rate of flow of oil (from graph) (grams from minute) ..	0·75	3·5	5·8
Period of flow in minutes ..	30	4	3
Amount of oil passed (grams) ..	*22·50*	*14·00*	*17·40*
Condensed liquid products (grams) ..	12·42	10·12	16·44
Gas and *Nitrogen at N.T.P. (litres) ..	9·847	3·203	1·04
Composition of nitrogen and oxygen free gas (Per cent. by volume)			
CO_2 ..	Nil	Nil	Nil
C_nH_{2n} ..	37·88	52·81	46·06
CO ..	Nil	Nil	Nil
H_2 ..	14·16	14·62	19·36
CH_4 ..	44·78	21·45	24·15
C_2H_6 ..	3·18	11·12	10·43
No. of gram mols of gas ..	0·4395	0·1430	0·04643
†Weight of N_2 and O_2 free gas (gm.) ..	9·625	3·362	0·417
Weight of deposited carbon (gm.) ..	0·2861	0·0940	0·0172
Material balance (total of liquid and gas and deposited carbon) (grams)	*22·33*	*13·57*	*16·87*

* At the commencement of the run the system is purged out of air with nitrogen and also after the run to sweep out the gases into the gas-holder.

† The gas is found to contain a little amount of oxygen coming from the large amount of water in the gas-holder.

From the material balance it is found that the average difference between the quantity of oil passed as indicated by the flow-rator and the materials collected in the form of liquid, gas and deposited carbon is within 5% which, for all practical purposes, may be considered as fairly accurate.

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