# 5. EXPERIMENTAL SET UP AND PROCEDURE

Fig. 5.1 through Fig. 5.4 show the schematics of the complete experimental set up employed to verify the mathematical model proposed in Chapter 3. The set up comprises of hydraulic and electrical circuits, which are described in detail below. The experimental procedure is also described.

## 5.1 Hydraulic Circuit

The hydraulic circuit (Fig.5.1) consists of the following major components:

- HDPE Calibration test section and collector test section.
- 35 Liter HDPE Tank.
- Centrifugal pump.
- Rotameter (28-280 lph of electrolytic solution)
- Copper Reference (anode) and test (cathode) electrodes
- Necessary PVC flexible pipe and fitting, and valves.

### 5.1.1 Calibration Test Section

This set up was made to calibrate the flow in the risers versus the polarising current established between the test and

reference electrodes, as discussed in Chapter 4. The dimensions of the riser and manifold are kept the same as that of the collector test section. This is shown in Fig. 5.2. It consists of 2200 mm long 20 OD HDPE riser pipe welded by extrusion technique to HDPE 50 OD pipe at both the ends. The 20 OD pipe was provided with 50 mm radius at both the ends to rest on the outer surface of 50 OD pipe. Also, the ID of 20 OD pipe was placed accurately on the opening of 50 OD pipe of the same diameter. This was achieved by using a specially tapered tool. This was to prevent any projection of the riser into the manifold, which would have caused flow disturbance at the tee junction.

The test electrode (1.4 mm copper wire) is provided at 120 mm from the entrance to avoid any entrance effects due to the tee junction. The reference electrode is placed at the exit 200 mm away from the tee junction. Similarly, at the inlet 500 mm of free running pipe of the same diameter is provided to avoid disturbance by any fittings/ joints upstream.

### 5.1.2 Collector Test Section

The collector test section is shown in Fig. 5.3. It comprises of 10 numbers of 2200 mm long 20 OD HDPE pipe, both ends of each were welded by extrusion to 50 OD HDPE manifold in a similar fashion as was done with the Calibration Test Section. The risers are spaced at 100 mm centre to centre. At the inlets and outlets of the manifold, HDPE/MS flanges are provided. The projection caused due

to this was carefully filed to avoid any flow disturbance.

Test electrodes (1.4 mm copper wire) are provided at 120 mm from the tee junction in all the risers as was done in the calibration test section.

### 5.1.3 HDPE Tank

The 35 litres capacity tank is provided to contain the electrolyte (0.5 N sodium hydroxide, 0.01 N potassium ferricyanide and 0.01 N potassium ferrocyanide).

### 5.1.4 Rotameter

The glass tube rotameter with SS float and other construction materials inert to the electrolyte is used with a flow range of 28-280 lph of electrolyte solution.

### 5.1.5 Reference and Test Electrodes

The Reference electrode (anode) is made of 50 mm nominal diameter, 60 mm long copper tube. The internal surface area is thus large enough such that the polarising current is independent of distance from the test electrode provided in each riser. It is mounted at the exit of test section.

The test electrodes (cathodes) are simple 1.4 mm copper wires inserted through the wall of the risers. The test electrode being a controlling electrode, they were prepared very carefully. Diameters of each were made equal and it was ensured that the same length was immersed in the riser. To insert the electrode, a circular hole of diameter slightly less than that of electrode was made. The electrode was then inserted to provide a tight fit and was leak proof.

### 5.1.6 Centrifugal Pump

A mono-block centrifugal pump is used to circulate the electro lyte. The casing is made of gun-metal with graphite seals.

### 5.1.7 PVC Flexible Pipings, Valves and fittings

The major components are connected with PVC flexible pipes. The valves and fittings are all made of gun-metal. Straight run HDPE pipes of 50 OD of 10 diameters are provided at the inlet and outlet of Collector Test Section to avoid any flow disturbance.

## 5.2 Electrical Circuit

The electrical circuit is simple and is shown in Fig. 5.4. A stabilised DC Power supply is used to apply voltage across the reference (anode) and test electrodes (cathodes). The power supply is 0-5 V, 0-2 A range and is able to operate as constant voltage or

current source. To check the applied voltage and measure the polarising current developed, a very accurate 4 1/2 digital mutimeter is used. The milliampere range of 2 to 20 mA was especially checked, and found to be very accurate within +/- 0.2 % +/- 0.01% f.s. The accuracy in the microampere range is similar.

A four pole-eight way switch is used to enable measurement of voltage and current using the same multimeter. In one position the DC power supply and the multimeter is in parallel for voltage measurement and in another they are in series to measure current. A single pole-eleven way selector switch is provided to connect the test electrodes in the risers to measure the polarising current.

## 5.3 Experimental Procedure

### 5.3.1 Preparation of Electrolyte Solution and Quality check:

The following electrolyte system was chosen:

0.5 N of sodium hydroxide.
0.01 N of potassium ferrocyanide.
0.01 N of potassium ferricyanide.

The required quantities of each of the above chemicals were weighed by an electrical balance accurate upto 0.1 mg, and dissolved in distilled water. The polarising current is very sensitive to the concentration of ferricyanide ions. Thus, after every preparation

the concentration was determined by iodometry. Subsequently, as long as the electrolyte was used this was continued to monitor the ferricynaide concentration. The electrolyte was discarded if more than 1 % change was detected.

### 5.3.2 Preparation of the test set up

Both for the calibration and collector test runs, the followings were observed to ensure repeatibility and minimise errors:

- After charging the electrolyte into the 35 l HDPE tank, the pump was switched on at the maximum flow rate to flush out any air. This was done for 30 minutes.

- Flow rate was then adjusted to the desired value by observing the rotameter. Half-an-hour was allowed for the flow to stabilise.

- For any fresh change of the electrolyte the system was flushed with distilled water and drained.

### 5.3.3 Rotameter Calibration

The rotameter was calibrated for the complete range. The time required for collecting known volume of 0.5 N sodium hydroxide was noted. The temperature during the experiments was also noted. The results are given in Table 5.1. It is also shown in Fig. 5.5.

### 5.3.4 Calibration of Flow vs Polarising Current

Employing the Calibration Test Section, polarisation curves were obtained at different flow rates. At each flow rate the current was measured at different DC potential applied across the reference and test electrode. The voltage was increased till limiting current was reached. The results are given in Table 4.2. Fig. 5.6 shows the polarisation curves at different flow rates.

It is worthwhile to mention that the resulting current fluctuated quite rapidly due to sensitive instrument used which could detect local disturbance around the test electrode. Thus, in Table 5.2 a range of current is presented for each flow rate and DC potential. It was ensured, however, that for each flow rate and applied voltage the measured current falls within the range all the time. Thus, for each point 15 minutes was allowed for.

Fig.5.7 shows the relationship between the limiting current and the flow rate of chosen electrolyte. It can be observed that it is highly linear.

Table 5.1 Calibration of Rotameter with Indifferent Electrolyte 0.5 NaOH, Temperature 29°-32° C.

| S.No. | Rotameter, mm | Vol. collected,l | Time,s (i) | (ii) | (iii) | Mean | Flow rate, l/s |
|---|---|---|---|---|---|---|---|
| 1. | 200 | 2 | 25.90 | 25.19 | 26.04 | 25.74 | 0.0777 |
| 2. | 190 | 2 | 26.80 | 26.86 | 26.76 | 26.81 | 0.0746 |
| 3. | 180 | 2 | 28.50 | 28.27 | 28.56 | 28.44 | 0.0703 |
| 4. | 170 | 2 | 29.80 | 29.73 | 29.77 | 29.77 | 0.0672 |
| 5. | 160 | 2 | 31.79 | 31.33 | 31.83 | 31.65 | 0.0632 |
| 6. | 150 | 2 | 33.50 | 32.79 | 33.85 | 33.41 | 0.0599 |
| 7. | 140 | 2 | 35.80 | 35.76 | 35.09 | 35.55 | 0.0563 |
| 8. | 130 | 2 | 38.75 | 38.82 | 38.02 | 38.58 | 0.0518 |
| 9. | 120 | 2 | 40.90 | 40.58 | 41.08 | 40.86 | 0.0489 |
| 10. | 110 | 2 | 43.60 | 43.28 | 43.54 | 43.50 | 0.0460 |
| 11. | 100 | 2 | 46.85 | 46.54 | 46.95 | 46.78 | 0.0428 |
| 12. | 90 | 2 | 51.55 | 51.07 | 51.61 | 51.41 | 0.0389 |
| 13. | 80 | 2 | 59.28 | 59.59 | 59.17 | 59.35 | 0.0337 |
| 14. | 70 | 1 | 33.67 | 34.01 | 34.00 | 33.89 | 0.0295 |
| 15. | 60 | 1 | 37.44 | 37.64 | 37.62 | 37.57 | 0.0266 |
| 16. | 50 | 1 | 43.69 | 44.20 | 43.90 | 43.93 | 0.0228 |
| 17. | 40 | 1 | 48.73 | 49.07 | 48.27 | 48.69 | 0.0205 |
| 18. | 30 | 0.5 | 31.35 | 31.50 | 31.26 | 31.37 | 0.0159 |
| 19. | 20 | 0.5 | 37.40 | 36.99 | 37.30 | 37.22 | 0.0134 |

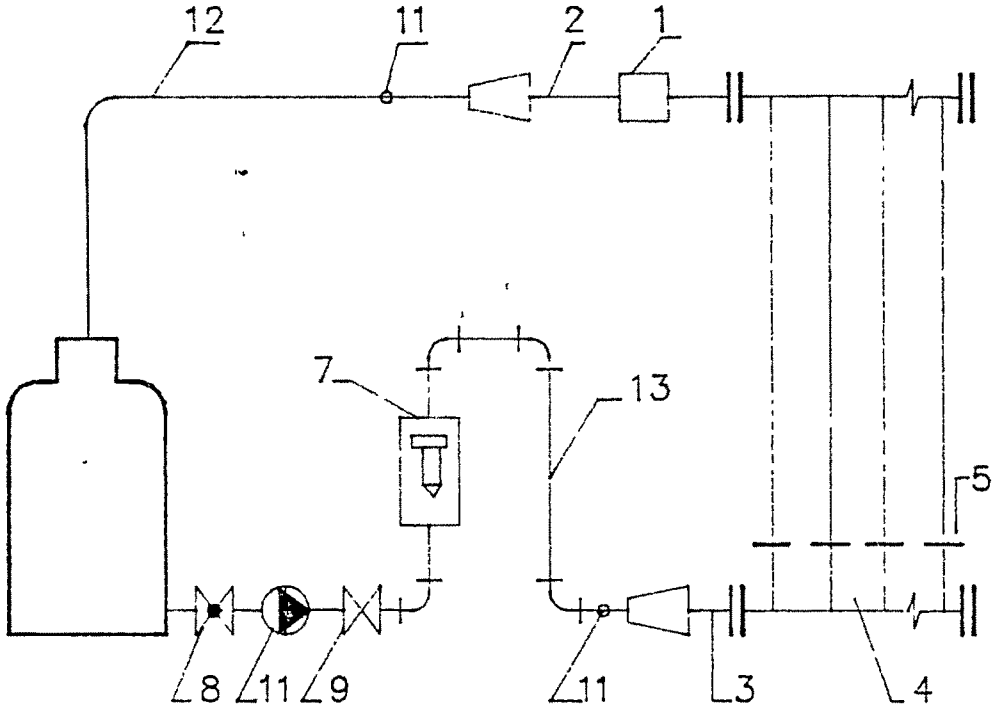
Table 5.2 Calibration of Flow Rate vs Polarising Current, Temperature 34° C.

| S.No. | Rotameter reading, mm | Voltage, V | Polarising Current, mA | | |
|---|---|---|---|---|---|
| | | | Min | Max | Mean |
| 1. | 200 | 0.0376 | 2.654 | 2.672 | 2.663 |
| | | 0.05083 | 4.538 | 4.613 | 4.576 |
| | | 0.6060 | 5.437 | 5.557 | 5.497 |
| | | 0.7045 | 6.459 | 6.548 | 6.504 |
| | | 0.8066 | 6.935 | 7.347 | 7.141 |
| | | 0.9093 | 6.875 | 7.452 | 7.164 |
| | | 1.0073 | 6.847 | 7.616 | 7.141 |
| | | 1.1017 | 6.728 | 7.554 | 7.141 |
| 2. | 175 | 0.2058 | 1.355 | 1.548 | 1.452 |
| | | 0.4068 | 3.360 | 3.572 | 3.466 |
| | | 0.5007 | 4.468 | 4.508 | 4.488 |
| | | 0.6076 | 5.466 | 6.072 | 5.769 |
| | | 0.7031 | 5.999 | 6.342 | 6.171 |
| | | 0.8041 | 6.043 | 6.581 | 6.312 |
| | | 1.0028 | 6.009 | 6.611 | 6.310 |
| 3. | 150 | 0.1080 | 0.573 | 0.630 | 0.602 |
| | | 0.1983 | 1.498 | 1.561 | 1.530 |
| | | 0.3096 | 2.588 | 2.623 | 2.606 |
| | | 0.4034 | 3.638 | 3.683 | 3.660 |
| | | 0.5067 | 4.539 | 4.607 | 4.573 |
| | | 0.6045 | 5.433 | 5.537 | 5.485 |
| | | 0.7031 | 5.463 | 5.680 | 5.572 |
| | | 1.0051 | 5.541 | 5.710 | 5.625 |
| 4. | 125 | 0.1991 | 0.940 | 0.970 | 0.951 |
| | | 0.2981 | 1.940 | 2.092 | 2.016 |
| | | 0.3996 | 2.981 | 3.063 | 3.022 |
| | | 0.5068 | 3.854 | 3.930 | 3.892 |
| | | 0.6034 | 4.357 | 4.763 | 4.560 |
| | | 0.7065 | 4.378 | 4.832 | 4.605 |
| | | 0.8001 | 4.453 | 4.842 | 4.648 |
| | | 1.0046 | 4.541 | 4.779 | 4.660 |
| 5. | 100 | 0.2062 | 0.905 | 0.941 | 0.923 |
| | | 0.3051 | 1.834 | 2.029 | 1.932 |
| | | 0.4029 | 2.124 | 2.858 | 2.491 |
| | | 0.5010 | 3.419 | 3.655 | 3.537 |
| | | 0.6076 | 3.631 | 4.113 | 3.872 |
| | | 0.7017 | 3.541 | 4.093 | 3.817 |
| | | 0.9996 | 3.504 | 4.098 | 3.801 |

cont'd

Table 5.2 cont'd

| S.No. | Rotameter reading,mm | Voltage, V | Polarising Current, mA | | |
|---|---|---|---|---|---|
| | | | Min | Max | Mean |
| 6. | 75 | 0.3008 | 1.597 | 1.638 | 1.618 |
| | | 0.4026 | 2.350 | 2.398 | 2.374 |
| | | 0.5013 | 2.653 | 2.942 | 2.800 |
| | | 0.6035 | 2.824 | 3.151 | 2.988 |
| | | 0.7021 | 2.666 | 3.049 | 2.858 |
| | | 1.0002 | 2.774 | 3.198 | 2.986 |
| 7. | 50 | 0.2017 | 0.387 | 0.439 | 0.413 |
| | | 0.3051 | 1.253 | 1.308 | 1.281 |
| | | 0.4021 | 1.931 | 2.031 | 1.981 |
| | | 0.5011 | 2.138 | 2.368 | 2.253 |
| | | 0.6036 | 2.005 | 2.328 | 2.167 |
| | | 0.7019 | 1.906 | 2.340 | 2.123 |
| | | 1.0002 | 2.052 | 2.328 | 2.190 |
| 8. | 25 | 0.2064 | 0.327 | 0.354 | 0.341 |
| | | 0.3086 | 0.996 | 1.025 | 1.011 |
| | | 0.4025 | 1.264 | 1.385 | 1.325 |
| | | 0.5055 | 1.163 | 1.423 | 1.293 |
| | | 0.6075 | 1.230 | 1.408 | 1.319 |
| | | 0.9026 | 1.256 | 1.424 | 1.340 |
| | | 1.0005 | 1.240 | 1.411 | 1.326 |




| Pt. | Qty. | Description |
|---|---|---|
| 1 | 1 | Coper Reference Electrode |
| 2 | 1 | Straight Run–Reducing Section |
| 3 | 1 | Straight Run–Reducing Section |
| 4 | 1 | HDPE Collecter– Test Section |
| 5 | 10 | Copper Test Electrode |
| 6 | 1 | 80 Ltr.HDPE Drum(wide mouth) |
| 7 | 1 | Rota Meter |
| 8 | 1 | Globe Valve(SS or GM) |
| 9 | 1 | Needle valve(SS) |
| 10 | 1 | SYP Iwaki Magnetic PP Pump |
| 11 | 12 | SS Hose Clips |
| 12 | | Flexible PVC Clear Pipe |
| 13 | | 20 OD HDPE Pipe |

Fig.5.1 Experimental Test Setup (Electrochemical)–Hydraulic Circuit

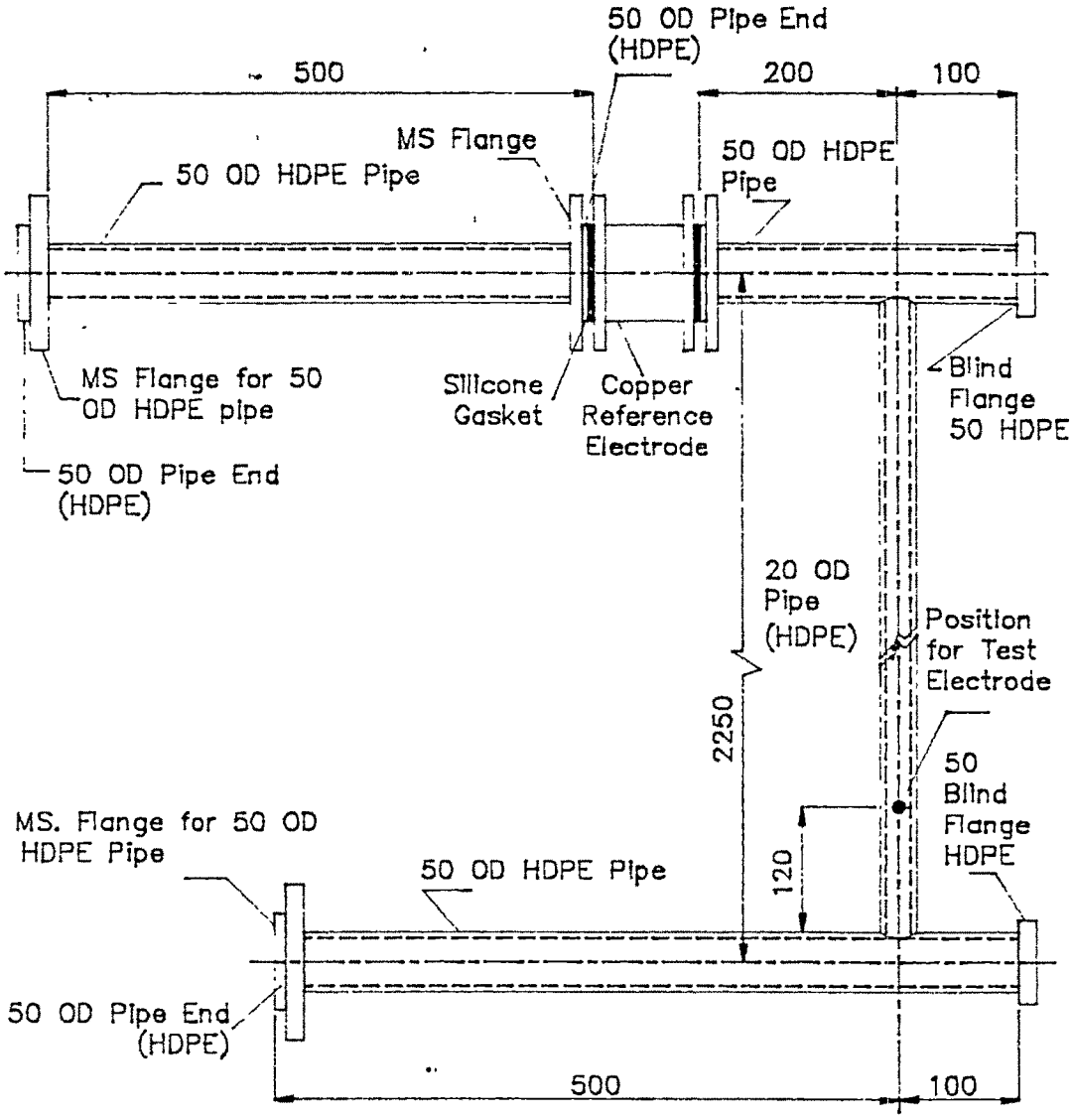



Fig. 5.2 Calibration Test Setup (Electrochemical)

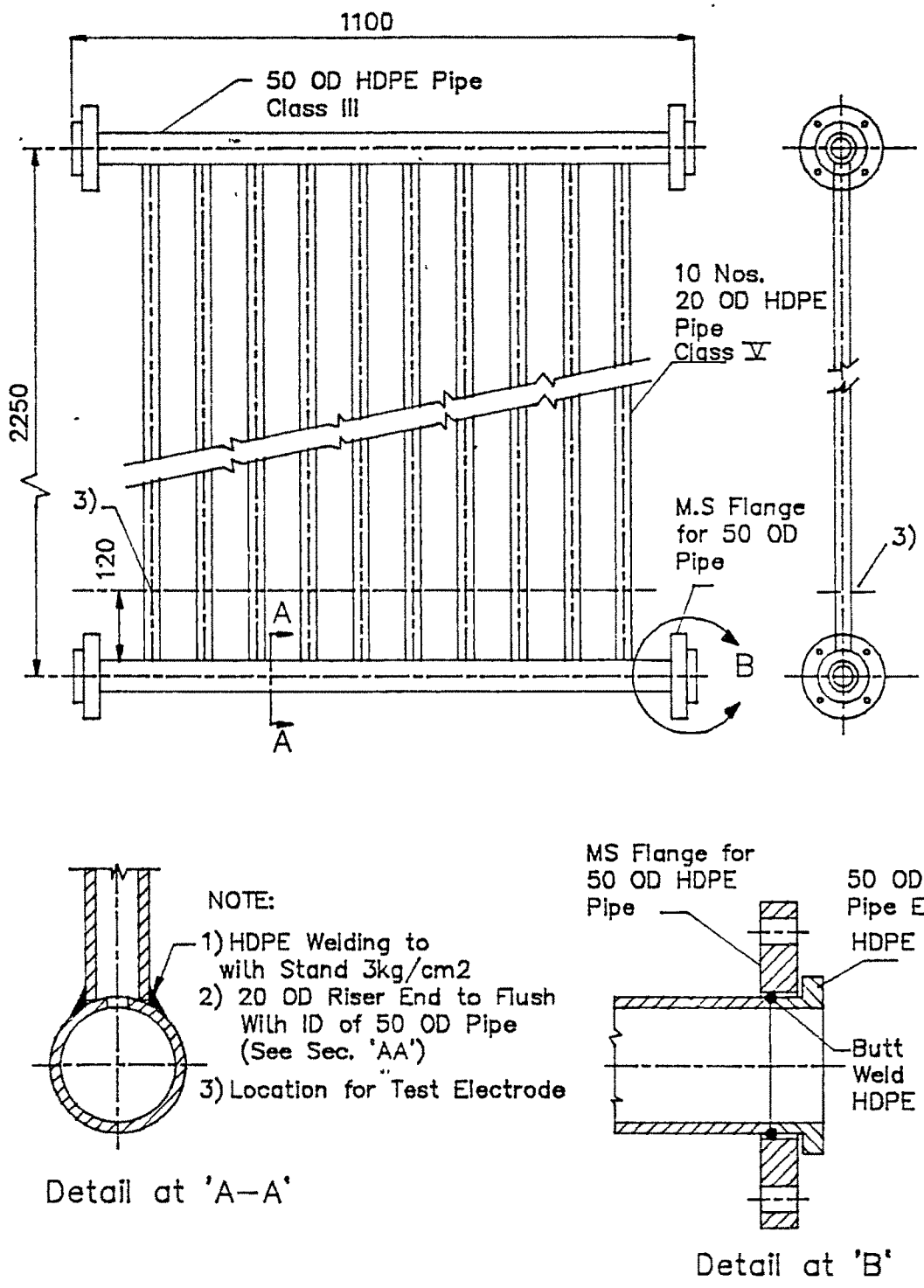



Fig. 5.3 Collector Test Setup

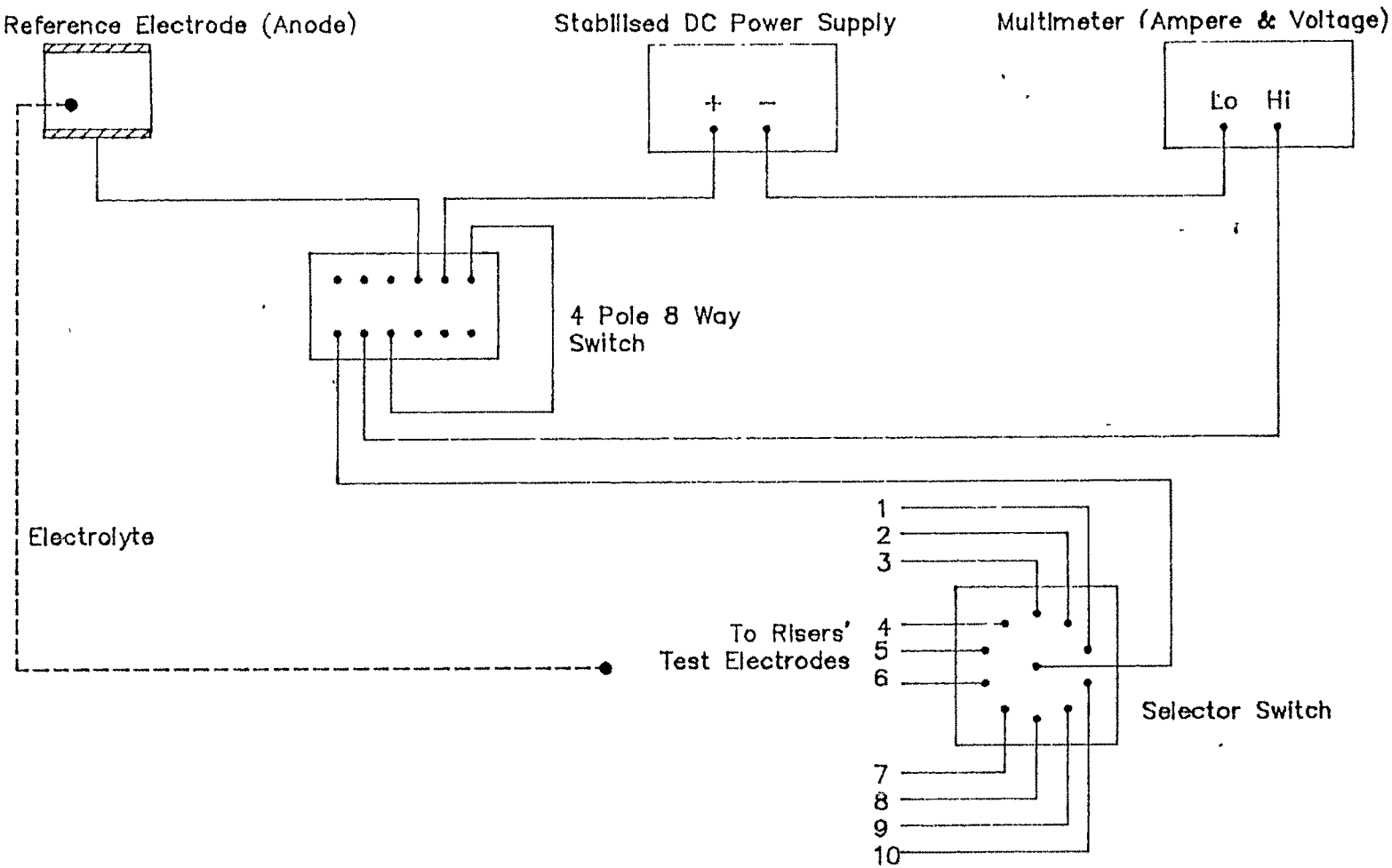



Fig. 5.4 Electrical Circuit for Electro Chemical Flow Measurment

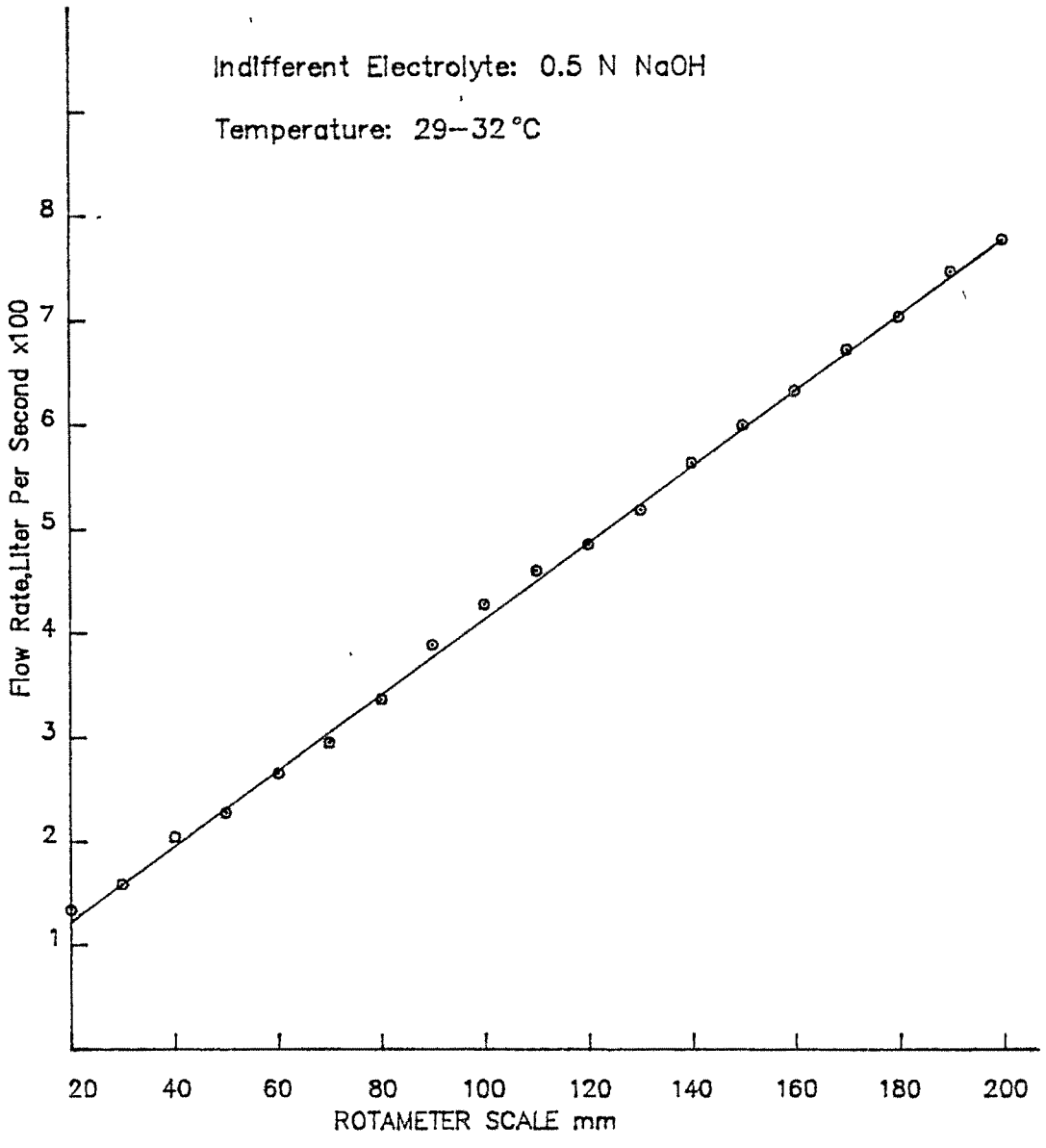



Fig.5.5 Rotameter Calibration.

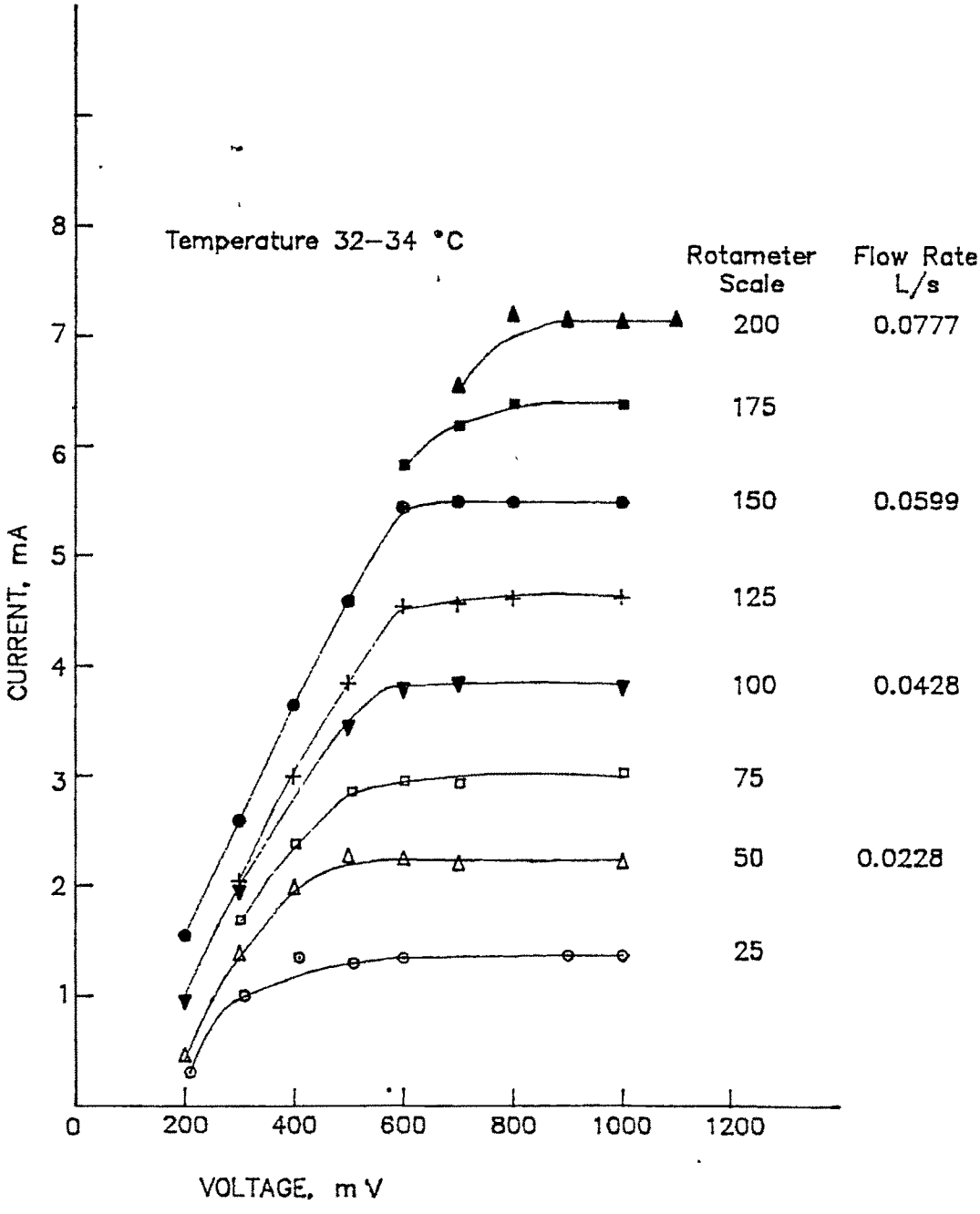



Fig. 5.6 Polarisation Curves at different flow Rates of 0.5 NaOH,01N $K_4$ $Fe(CN)_6$ and 0.01N $K_3Fe(CN)_6$