CHAPTER III

MATERIALS AND METHODS

3.1. FIELD METHODS

Meltwater samples were collected in pre-washed polyethylene bottles (500 ml.) three times a day (730, 1200, 1700 hrs.) to broadly cover diurnal variations during the sampling period. The samples were collected 2 km. away from the snout during the period from 7th September to 19th September 1991. Electrical Conductivity (EC) of the water samples was measured in the field using a hand held Pentax EC meter. Sediment samples were collected at a few locations for their analysis in the laboratory.

3.2 LABORATORY METHODS

The water and sediment samples were later in the laboratory analysed following prevalent techniques in chemical analysis. A brief description of the methods followed is given below.
3.3 WATER ANALYSIS

3.3.1 pH and Electrical Conductivity (EC): pH and Electrical Conductivity of the samples were measured in the laboratory using Consort C-425 pH-conductivity meter. The instrument was set for standard pH 6.87 buffer at the water temperature and pH of the samples were taken. For EC measurements, the instruments was calibrated and set for 0.01M KCL standard. The conductivity was then measured in uS cm\(^{-1}\) for water samples.

3.3.2 Bicarbonate: Bicarbonate was determined in the laboratory by following the potentiometric titration method. In this method no indicator was used, instead, the pH of the samples as well as that of the standard solutions were maintained at 4.5 to signify the end points, by adding HCl (0.004 N). A graph was plotted for standards against the amounts of HCl consumed and from the graph the amount of bicarbonate in the samples were calculated.

3.3.3 Chloride: Chloride in the laboratory was determined by following the potentiometric titration method. Standards of various concentrations (1 to 5 ppm.) were prepared. 25 ml of each standard and water sample were titrated against 0.001 N silver nitrate solution. Potassium chromate was used as indicator. The end point of the reaction is the appearance of pink colour. Concentration of chloride in samples was determined from the graph plotted for standards against the amount of silver nitrate consumed.
3.3.4 **Sulphate**: Sulphate ion concentration was measured by turbidimetric method. The method is based on the principle that $\text{SO}_4^{2-}$ is precipitated in an acetic medium with Barium chloride, so as to form barium sulphate crystal of uniform size. Light absorbance of $\text{BaSO}_4$ suspension is measured by a photometer and the concentration is determined by comparison of the reading with standard curve. 100 ml. of standard solution and sample were measured in a volumetric flask and 20 ml. of buffer solution (prepared by dissolving 30 gm. of $\text{MgCl}_2.6\text{H}_2\text{O}$, 5 gm. of $\text{CH}_3\text{COOH}.3\text{H}_2\text{O}$, 1.0 gm. of $\text{KNO}_3$ and 0.111 gm. of sodium sulphate and 20 ml. of acetic acid (99%) in 500 ml. of distilled water and then the total volume made up to 1000 ml.) was added and mixed with the help of magnetic stirrer. While stirring spoonful of $\text{BaCl}_2$ crystal was added. This was stirred for sixty second with constant speed. After the stirring period, solution was poured into the absorbance cell of the photometer and turbidity was measured at $5 \pm 0.5$.

Sulphate ion concentration was estimated by comparing reading with calibration curve prepared by carrying sulphate standard through the entire procedure. Reliability of the calibration curve was checked by running a standard with every three or four samples.
3.3.5 **Dissolved Silica:** The dissolved silica concentration was determined by molibdosilicate method. Standard solutions of different concentration ranging from 0.5 to 10 ppm were prepared by dissolving Na$_2$SiO$_3$. 20 ml of each standard and water sample were taken. 10 ml of Ammonium molybdate solution and 15 ml of reducing reagent was added. Ammonium molybdate solution was prepared by dissolving 2 gm of Ammonium molybdate in 10 ml distilled water and 6 ml conc. HCl and the total volume made upto 100 ml by diluting it with distilled water.

Reducing agent was prepared by mixing 100 ml metal sulphite solution, 60 ml of 10% Oxalic acid and 120 ml of 25% sulphuric acid and the total volume made upto 300 ml using distilled water. Metal sulphite solution was prepared by dissolving 5 gm of Metol in 210 ml of distilled water and 3 gm of sodium sulphite was added and the total volume made upto 250 ml using distilled water.

The samples were stirred well and kept for three hours to complete the reaction. The optical density was measured for standard and water sample at 812 nm using GBC 911 UV/VIS Spectrophotometer.

3.3.7 **MAJOR CATIONS**

Calcium, Magnesium, Sodium and Potassium.

The analysis of cations was carried out on GBC 906
Atomic Absorption Spectrophotometer. Calcium and Magnesium were analysed in absorption mode and Sodium and Potassium in the emission mode. The instrument was calibrated using different chemical standards.