CHAPTER IV

METHODS OF INVESTIGATION

On account of their diverse nature, extreme variation in grain size and lithology, the rocks of the Blaini Formation are one of the difficult populations to be sampled and investigated. Diversification of techniques of investigations can only help in such a study in which an attempt has been made to concentrate more on petrological and geochemical investigations. Nevertheless, the field investigations form the primary basis for any type of such works. The techniques used during the present investigations of the Blaini Formation of Simla Hills have been discussed in this chapter.

Field Methods:

The study area is one of the most accessible areas in the Himalaya and as such one of the best explored. It has been found that the earlier geological maps prepared by the pioneers of Indian Geology like Pilgrim and West (1929) and Auden (1934) are very much helpful as base maps. These maps show well-marked outcrops of the Blaini Formation quite accurately. The Survey of India’s topographic sheets No. 53 E/4, 53 F/1 and 53 F/5 were utilised for the field work. Due to the extensive nature of the
outcrops of the Blaini Formation, the Girl river section offers the best opportunity to carry out the mapping of various members. This has been done on 4" = 1 mile (1 cm = 167 m) scale. In rest of areas, mapping on this scale has not been done due to the limited thickness and extent of this formation. Lithostratigraphic columns of the Blaini Formation were prepared in all the sections of the study area.

Sampling of the different litho-units of the Blaini Formation has been a difficult task particularly because no composite and representative sample can, in totality, statistically be designated as a true sample of the Blaini diamictites. Hence it became obligatory for the author to undertake random sampling as well as the lithostratigraphically controlled sampling of these units. One of the representative sections, i.e., Girl river section, was selected for close unit-wise sampling for the sedimentological and geochemical investigations as the succession here is complete and less disturbed. In all, a large number of samples — about 500 in number — were collected. Selected samples from this collection were utilised for the laboratory investigations.

**Thin Section Study:**

A large number of thin sections of all the
lithologies of the Blaini Formation were prepared and studied petrographically. Slight difficulty was encountered in studying the diamicrites in thin sections. However, thin sections were prepared of all the components of the diamicrites collectively as well as separately. Modal analyses of different lithologies have been carried out by the method of point counting using the mechanical stage and counting about 500 grains in each case. In carrying out modal analysis of the diamicrites, fine grained clastics up to the sand size were counted and larger ones were neglected.

**Grain Size Analysis and Textural Parameters:**

Much has been written on the techniques of the grain size analysis used for sedimentary rocks and various methods like sieving, thin-section study and pipette analysis, etc., have been suggested. This is due to the large variation in the nature and the physical properties of these rocks. These techniques have been detailed in various text books on sedimentary rocks (Krumbein and Pettijohn, 1938; Muller, 1964; Carver (Ed.), 1971; Folk 1974; etc.). For the grain size analysis of the rocks of the Blaini Formation, different techniques were adopted for diamicrites and sandstones. These are discussed below separately.
**Diamictites**

The importance of the grain size analysis of the sedimentary rocks lies in the determination of grain size of the clastic particles as they were deposited. However, a number of post-depositional factors render its exact determination a difficult task. In case of diamictites, the large unwieldy size of the components add more to the problem and virtually at times it becomes impossible to do the mechanical analysis satisfactorily by any one method. The grain size analysis of the diamictite samples was performed by the method of sieving. Only the friable samples were taken. The samples having abundant chemical cement were rejected. All the samples analysed are from near the village Hlon in Giri river section. Bulk samples (about 2-3 kg. each), from base to top, of the unit were taken on grid pattern at 2-3 metres interval. The samples were disaggregated first with the fingers and then with the porcelain mortar by gentle pounding. Grains coarser than -2 $\varnothing$ were separated by hand. The left over material was reduced in bulk by the method of coning and quartering and finally sieved at 1/2 $\varnothing$ interval. Mud fraction was not further analysed. From the data, thus accruing, the cumulative weight percent frequency curves were drawn.
The formulae of graphical parameters for the grain size distribution proposed by Folk and Ward (1957) require to incorporate the 5th and 95th percentile values. However, Dyer (1970) modified the formulae of standard deviation, skewness and kurtosis for sandy-gravels. He substituted the 95th and 5th percentile value by 75th and 25th percentile values in the original formulae of Folk and Ward (1957). The formula for the mean size has been adopted as such by Dyer (1970). For the Blaini diamictites, the grain size parameters have been calculated using the formulae of Dyer (1970), which are given in table 4.

**Sandstone**

The Blaini sandstones could not be disaggregated due to the consolidated nature of the rock. As such, the thin section technique was adopted for the grain size analysis. According to Olaister and Nelson (1974), the thin section technique has advantage over the sieving method because this technique could be applied to ancient consolidated rocks, could be used over small samples and the diagenetic changes could also be recognised and accounted for. The quartz grains are the most suitable to measure the size. The longest dimensions of 200 quartz grains in each thin section of the Blaini sandstones were
**TABLE 4: Formulae for the graphic size measures used in the present work**

<table>
<thead>
<tr>
<th>Measure</th>
<th>or san s ones</th>
<th>or am c es</th>
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<tbody>
<tr>
<td>Graphic Mean</td>
<td>[ M_z = \frac{Q_{16} + Q_{50} + Q_{84}}{3} ]</td>
<td>[ M_z = \frac{Q_{16} + Q_{50} + Q_{84}}{3} ]</td>
</tr>
<tr>
<td>Inclusive Graphic Standard Deviation</td>
<td>[ \sigma_I = \frac{Q_{84} - Q_{16}}{4} + \frac{Q_{95} - Q_{5}}{6.6} ]</td>
<td>[ \sigma_D = \frac{Q_{84} - Q_{16}}{4} + \frac{Q_{75} - Q_{25}}{2.57} ]</td>
</tr>
<tr>
<td>Inclusive Graphic Skewness</td>
<td>[ Sk_I = \frac{Q_{16} + Q_{84} - 2Q_{50} + Q_{95} - Q_{25}}{2(Q_{84} - Q_{16}) + 2(Q_{95} - Q_{25})} ]</td>
<td>[ Sk_D = \frac{Q_{16} + Q_{84} - 2Q_{50} + Q_{75} - Q_{25}}{2(Q_{84} - Q_{16}) + 2(Q_{75} - Q_{25})} ]</td>
</tr>
<tr>
<td>Graphic Kurtosis</td>
<td>[ K_G = \frac{Q_{95} - Q_{5}}{2.44(Q_{75} - Q_{25})} ]</td>
<td>[ K_D = \frac{Q_{84} - Q_{16}}{1.5(Q_{75} - Q_{25})} ]</td>
</tr>
<tr>
<td>Simple Sorting Measure</td>
<td>[ S_{o_s} = \frac{1}{2}(Q_{95} - Q_{5}) ]</td>
<td>-</td>
</tr>
<tr>
<td>Simple Skewness Measure</td>
<td>[ \alpha_s = (Q_{95} + Q_{5}) - 2(Q_{50}) ]</td>
<td>-</td>
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</tbody>
</table>
recorded at 1/2 \( \phi \) interval by the method of point counting, using the mechanical stage, under the microscope. The grain size up to 5 \( \phi \) was measured by this method and the material finer than this class was grouped into the next class, following Glaister and Nelson (1974). The data were plotted on simple graph paper for the number frequency distribution and on probability graph paper for the cumulative frequency distribution. The graphical parameters, viz., mean size, standard deviation, skewness and kurtosis have been calculated, by finding the percentile values from the cumulative curves, using the formulae of Folk and Ward (1957). Two more parameters, viz., simple sorting measure and simple skewness measure have been calculated using the formulae of Friedman (1967). All these formulae are given in table 4.

Statistical parameters for the distribution of grain size in the Blaini sandstones have also been calculated by the method of moments. The IBM 1620 Computer of the Department of Mathematics, Panjab University, Chandigarh, has been utilised for this purpose. The first four moments and the statistical parameters have been calculated by the formulae given by Griffiths (1967).

In recent years, the environmental discrimination
has been done on the basis of grain size distribution by the method of sieving on loosely consolidated recent sediments by a number of authors. The size distribution determined by the thin section method cannot be directly compared with the sieve-size data. A large number of factors have been proposed to convert the thin section data into either loose grain or sieve size data (Krumbein, 1935; Sahu, 1964b, 1966b). Sahu (1964b, 1966a, b, c) has developed a procedure for the grain size analysis of thin sections on weight frequency basis. This method requires the determination of the nominal section radius of about 500 grains in each thin section and from this data weight frequency distribution of grains can be obtained by applying conversions. The theory and procedure of the thin section size analysis has been thoroughly discussed by Sahu (1974a, b, c, 1976, 1977). Though Sahu's method may be more desirable, for the present work, this method was not used since longer dimensions of only 200 grains have been measured. Recently, Adams (1977) has proposed certain conversions by which sieve size statistics can be obtained from the thin section data. The low correlation coefficients for conversions of skewness and kurtosis indicate that these two parameters cannot be reliably estimated from thin section data. Sahu's (1964a) discriminant functions have
been utilised for the environmental interpretation of the Blaini sandstones and the results have been obtained from both thin section and converted sieve size statistics.

Clast Fabric:

The clast orientation studies in the Blaini diamictites have been performed at a few selected stations in the Giri river section. Apparent long axes of about 100 elongated or disc shaped clasts, having an apparent axial ratio 2 : 1, were measured at each station and the data have been plotted on equal area net. As the beds are inclined, the tilt correction was applied according to the method given by Potter and Pettijohn (1963). For this purpose, the data on the equal area net were rotated to the horizontal about the strike of the bed, by an angle equal to the dip of the beds. Care was taken to select only those stations which show the minimum effect of tectonic disturbance.

Scanning Electron Microscopy:

The surface texture studies of the quartz grains from the Blaini diamictites have been done by the Scanning Electron Microscope. The quartz grains were picked from the
sieved material of the diamictites. Generally, the grains are of medium sand size. Care was taken to choose only those quartz grains which were complete and without any hammer mark. Only monocrystalline grains were taken into account. The cementing material on the grains was removed by cleaning them with very dilute hydrochloric acid. After this, the grains were washed with distilled water a number of times. The cleaned quartz grains were coated with carbon. There was no tilt of the samples, i.e., the samples were kept in horizontal position and the photographs were taken by the Scanning Electron Microscope (Model JSM-50A).

The sample preparation of the quartz grains was done by the author while the carbon coating of the grains was performed and the photographs were taken at the Department of Geology, Memorial University of Newfoundland, St. John's, Newfoundland, Canada.

Staining of Carbonate Rocks:

Much importance has been assigned to distinguish different carbonate minerals by the staining techniques and a number of schemes have been proposed (Friedman, 1959; Warne, 1962; Evans, 1963; Dickson, 1965). During the present work, Dickson's (1965) and Warne's (1962) methods
were followed, respectively, for thin sections and rock chips staining of the Blaini carbonates. Both the techniques are briefly discussed below:

Dickson's (1965) technique of staining the thin sections of the carbonate rocks involves the following three steps:

**Step 1:** Etching of the thin section with 1.5% HCl for 10 to 15 seconds.

**Step 2:** Staining the etched thin section with a mixture of 0.2% Alizarin Red S and 2% Potassium Ferricyanide solution mixed in the ratio of 3:2 for about 1 minute (the solutions were prepared in 1.5% HCl).

**Step 3:** Staining with the solution of 0.2% Alizarin Red S for 10 to 15 seconds.

By the above technique the different carbonate minerals take the following stains:

- Calcite: Pink to red
- Ferron calcite: Purple
- Dolomite: Colourless
- Ferron dolomite: Light to deep turquoise
Warne's (1962) scheme is very illustrative and can be applied to both thin sections and rock chips. By this method, about 13 carbonate minerals can be distinguished. Polished rock chips of the Blaini carbonates were stained by this method involving the following steps:

Step 1: Etching of the rock chips with 3% HCl. Since little effervescence was seen, slight heating was done for about 1 minute.

Step 2: Etched samples were stained with 0.1% Alizarin Red S solution prepared in 2% HCl.

On staining by this solution, the minerals exhibit the following colours:

Calcite, witherite, high magnesium calcite and aragonite - deep red;

Ankerite, Fe dolomite, strontianite and cerussite - purple;

Anhydrite, siderite, dolomite, rhodochrosite, magnesite, smithsonite and gypsum - no colour.

Step 3: Etched samples are again stained with a mixture containing equal volumes of the
above solution of Alizarin Red 3 and 30% sodium hydroxide solution and boiled for five minutes. Each of the unstained mineral in the previous step will be stained to purple except siderite which stains dark brown to black.

Further differentiation of the carbonate minerals is possible by using different solutions as enumerated by Warne (1962).

X-ray Study:

On account of the limited facilities available to the author, it was possible to get the X-ray diffraction analysis of only five samples of the Blaini carbonates performed at the Department of Geology, Delhi University, Delhi. The powdered material (-200 mesh) was analysed by Phillips X-ray diffraction apparatus model PW 1130/00 using CuKα radiation and Ni-filter. The samples were run between 27° and 36° (2θ values) with 1°/minute rotation. The characteristic d-values (Å), calculated from the diffractogram, were utilised for the precise mineral identification.
The author undertook some geochemical investigations of the Blaini rocks of the study area. The matrix of the Blaini diamicritites were analysed for their major and trace element constituents, whereas in the case of shale and limestones, only the trace element determinations were carried out. The samples were pulverised in carborundum steel mortar, sieved and finely powdered in agate mortar. Care was taken to prevent the incorporation of clasts or sand size grains while preparing the samples of the diamicritite matrix for analysis. The procedures followed for the major and trace element determinations are discussed below:

**Major Element Analysis:**

The major element oxides - SiO\(_2\), Al\(_2\)O\(_3\), total iron as Fe\(_2\)O\(_3\)(Fe\(^{+}\)), TiO\(_2\), K\(_2\)O, Na\(_2\)O, CaO and MgO were determined by the method of wet chemical analysis following the procedure of Shapiro and Brannock (1962). The 'Solution A' was prepared for the determination of SiO\(_2\) and Al\(_2\)O\(_3\) using 0.1 gm of rock powder and 'Solution B' was prepared using 0.5 gm of rock powder for the determination of TiO\(_2\), MnO, Fe\(^{+}\), K\(_2\)O, Na\(_2\)O, CaO and MgO. The optical densities of the solutions were measured by Hilger
and Watts UVISPEK Spectrophotometer for the determination of SiO$_2$, Al$_2$O$_3$, MnO, TiO$_2$, Fe$^t$.

The Na$_2$O and K$_2$O were determined on BBL's Flame Photometer. CaO and MgO were estimated volumetrically by E.D.T.A. method (versene method). Samples of the Blaini carbonates were also analysed, using this method, for their CaO and MgO content.

**Standards Used**

For the determination of SiO$_2$ and Al$_2$O$_3$, two international standards, 0-2 and BCH-1, supplied by the U.S.G.S., were used. For the determination of other oxides, the following analar grade synthetic chemicals were used as standards:

<table>
<thead>
<tr>
<th>Oxide determined:</th>
<th>Standards Used:</th>
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<tbody>
<tr>
<td>CaO</td>
<td>Calcium carbonate</td>
</tr>
<tr>
<td>MgO</td>
<td>Magnesium sulphite hydrated</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>Sodium chloride</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>Potassium chloride</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>Potassium titanyloxalate</td>
</tr>
<tr>
<td>Fe$^t$</td>
<td>Ferrous ammonium sulphate</td>
</tr>
<tr>
<td>MnO</td>
<td>Manganese sulphite</td>
</tr>
</tbody>
</table>
Trace Element Analysis:

The trace element determinations were carried out on the Jarrell-Ash 1.5 Meter Compact Direct Reading Spectrometer (Model 66-000) at the Oil and Natural Gas Commission Laboratory, Dehradun. The instrument is based on a 1.5 meter Paschen-Runge mounting, covers a spectral range of 2000Å to 9000Å I Order at an average dispersion of 5.45Å/mm and 2000Å to 4000Å II Order at an average dispersion of 2.75Å/mm.

The trace elements determined for the present work are: Mo, Ge, V, Co, Cu, Ni, Ba, B. The powdered sample (-200 mesh) and spec-pure graphite powder are mixed in a 1:4 ratio and the resultant powder is thoroughly mixed for about half an hour in an agate mortar to make it homogeneous.

Two spec-pure graphite electrodes were used. The anode is shaped to give a small cavity in which about 20 mg of the above mixture is filled in. The cathode is shaped point sharp. The two electrodes are placed in the arc stand having 2 mm analytical gap inbetween. The D.C. arc is produced between the two electrodes causing excitation of the sample. On excitation, it emits the characteristic radiations. Pre-exposure and exposure timers are provided.
with the instruments. Pre-exposure time was kept as 2 seconds which is required to stabilise the arc. During this pre-exposure time the mercury reference lamp do not rotate but on completion of pre-exposure time, it rotates allowing the radiant energy to pass through the slit for the exposure time which is 10 seconds. On completion of the exposure time, the arc is automatically extinguished and the mercury lamp comes to its original position.

The radiant energy falls on the diffraction grating which disperses it into its specific wave lengths giving characteristic spectrum. The principal lines of each element pass through the exit slits behind which the photomultiplier tubes are installed. The photomultiplier tubes convert light energy into electrical energy and charge the condensers. The charged condensers are discharged one by one giving the proportionate counts in the electronic read-out system. All the counts are automatically typed by the attached IBM typewriter. Three runs were made of each sample. The mean of the three readings is used for ppm determination.

Standards Used

The graphite base 34 Standard Powders provided by Jarrell-Ash Company are used in these determinations.
The respective standard powders for high (1000 ppm), medium (100 ppm and 10 ppm) and low (1 ppm) concentrations were run before each set of samples. Curves of the standard counts versus ppm concentrations are drawn on log-log graph paper for each element. The ppm concentration of each element in the analysed samples is determined from these curves. The USGS Standards G-2 and BCAS-1 were also analysed for these trace element contents in order to check the precision of the present data.