CHAPTER -3

Methodology
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Methodology includes field study and sampling, petrography, separation of mineral fractions for U-Pb isotope studies and trace element studies, processing of rock samples for Rb-Sr and Sm-Nd isotope studies, preparation and calibration of columns for element separation based on ion-exchange chromatography, preparation and calibration of isotope tracers and their standardization, and determination of elemental and isotopic abundances from the samples using analytical instruments such as Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES) and Thermal Ionization Mass Spectrometer (TIMS).

3.1 Field Study, Sampling and Petrography:

Field study was carried out primarily to understand the geological setting and distribution of various rock types in the study areas (Hutti, Kolar and Ramagiri areas) marked in Figs. 2.2, 2.4 and 2.5 and to collect representative samples for geochemical and isotope studies. An extensive field study of geology of the Kolar, Ramagiri and Hutti areas was carried out during December 2000 and January 2001. During June 2001 and December 2001 and in January 2003 sampling of granitoids around the Hutti Schist Belt was undertaken. A joint field study of the Hutti area was carried out with Mr. Alexander Schmidt and Dr. Klaus Mezger (University of Muenster, Germany) as a part of a DST-DAAD project during February – March 2002. One more field work and sample collection from the Hutti Schist Belt was carried out during January 2005. Survey of India topographic sheets 57K/8, 57L/1 and 57L/5 for Kolar area, (covering 12°50’ N to 13°05’ N latitudes and 76°10’ E to 76°25’ E longitudes) 57F/7 and 57F/11 for Ramagiri area (covering 14°10’ N to 14°25’ latitudes and 77°25’ E to 77°45’ E longitudes) and 56D/11, 56D/12, 56D/15, 56D/16 and 57A/9 for the Hutti area (covering 15°50’ N to 16°20’ latitudes and 76°35’ E to 76°55’ E longitudes) were used for the field work. Details of sample locations are given in Appendix I.

3.1.1 Hutti Granitoids

Field study of the Hutti Schist Belt and the surrounding granitoids was carried out in several phases for a better understanding of the field relationships and disposition of different rock types. Several traverses were taken along and across the
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schist belt. The main objective of sample collection was to do detailed isotope study of the granitoids and the schist belt rocks. Samples of granitoids were collected from the northern, western and eastern granitoids surrounding the schist belt. Metabasalts and felsic volcanics were collected from various locations in the schist belt marked in Fig. 2.2. The locations of the samples from the schist belt were same as that of the locations reported in the geochemical study of these rocks by Girithearan and Rajamani (1998). The samples of the granitoid rocks were collected from the same locations where they were sampled for geochemical and petrogenetic studies reported in Basir (2000) in order to integrate the isotope study with the previous geochemical studies.

North of Golapalli village, about a km on the eastern side of the Shorapur road exposures of granitoids are found (Plate 3.1a). They are granodioritic in composition (Basir, 2000) with large phenocrysts of pink feldspars (~2-3 cm) in the groundmass made of quartz, feldspars, biotite and hornblende. The rocks are epidotized at some places. Flow foliations are seen in these rocks.

The granodiorite in thin section is inequigranular, medium-grained, has abundant quartz, orthoclase, microcline and minor plagioclase feldspars. Hornblende is the common mafic mineral present along with minor biotite. Plagioclase is intergrown with K-feldspar forming anti-perthite (Plate 3.1b). Sericitization and epidotization can also be observed. Titanite is the most abundant accessory mineral followed by opaques, zircon and apatite.

South of Yelagatti village are pink granitic rocks that have large phenocrysts of K-feldspar (~2-3 cm) embedded in a quartz-feldspar matrix (Plate 3.1c). These rocks are referred to as Yelagatti granitoids by Srikantia (1995) although, in appearance, they are similar to those occurring north of Golapalli village. The Golapalli and Yelagatti granitoids are referred to as northern granitoids in the present study and samples from these locations were collected. The contact between the northern granitoids and the schist belt is soil covered and hence their relationship is not clear.

The thin section of the Yelagatti granitoid shows abundant quartz, megacrysts of K-feldspar that are sericitized, plagioclase, biotite and hornblende. Microcline is relatively smaller in size exhibiting tartan twinning. Abundant small-sized titanites are found which are invariably surrounded by opaques, probably ilmenite and/or magnetite. Zircon crystals that are pale pink in colour are also present.
Northeast of Palkanmardi village within the schist belt there is a unique unit which has been reported as "Polymictic Conglomerate" by earlier workers. This "conglomerate" is characterized by rounded to angular, unsorted clasts up to 60 cm in size, which are closely placed or scattered in a coarse- to fine-grained matrix of metamorphic amphibole and detrital feldspar and quartz (Vasudev et al., 2000). The clasts are of granite, aplite, pegmatite, granodiorite, vein quartz, greywacke, fine-grained metabasalt and metagabbro. A field photograph showing clasts of granodioritic rocks in a matrix of mafic material is shown in Plate 3.1d. On the northwestern side of the schist belt, in the bed of the Hutti river the contact between the metabasalts of the schist belt and the granitoids could be seen where the granitoids appear intrusive into the metabasalts of the schist belt.

On the western side of the schist belt, about half a km southeast of Kardikal and about 3 km west of Lingsugur, extensive outcrops of granodiorite are found. These rocks are made up of quartz, plagioclase, minor K-feldspar, amphibole and abundant titanite. The contact between the western granitoids and the schist belt rocks is soil covered.

The petrography of the thin section of the above sample shows abundant quartz with K-feldspar, plagioclase, hornblende and anhedral biotite. The rock is equigranular and medium to coarse-grained. Quartz and orthoclase intergrowth showing graphic texture is also observed. Titanites are ubiquitous while trace amounts of zircon and apatite are also found. Sericitization and epidotization are common (Plate 3.2a).

On the eastern side of the N-S arm of the schist belt the contact between the schist belt and the granitoids could be observed east of the village Gajalagatta where the granitoids are intrusive into the schist belt rocks (Plate 3.2b). A sample of the granodiorite near Gajalagatta was collected (Plate 3.2c). About a km west of Watgal and south of the Lingsugur-Raichur road there is a hillock which is entirely made up of granodiorites, a sample of which was collected. The rocks from these two locations are composed of quartz, plagioclase, K-feldspar, biotite, hornblende and considerable titanite.

The thin section study of the Gajalagatta granodiorite showed a mineralogical composition of quartz, plagioclase, alkali feldspars, hornblende and minor biotite. Plagioclase feldspars are coarse-grained, present as megacrysts and exhibit characteristic polysynthetic twinning. Quartz and alkali feldspars occur as interstitial
grains between plagioclase feldspars. Titanite, opaques, zircon, apatite and epidote are present as accessory minerals (Plate 3.2d).

The granodiorite from Watgal is inequigranular and medium to coarse-grained on the thin section. It contains quartz, microcline, orthoclase, plagioclase, flaky biotites and hornblende. Titanite is a ubiquitous accessory mineral phase and is found as relatively larger euhedral grains. Zircon, epidote, apatite and opaques are also present in accessory amounts.

A sample of granite outcropping near Kasamodddi village present in the eastern granitoids is found to be composed predominantly of quartz and pink coloured K-feldspar. The granite is medium-grained and has mafic minerals in minor amounts.

Based on thin section study, quartz, microcline showing tartan twinning, orthoclase and perthite were found to be the major mineral constituents of the pink coloured Kasamodddi granite. Plagioclase and biotite occur in minor amounts and this granite is medium-grained and equigranular. Few grains of accessory minerals such as titanite, zircon and opaques could be traced in thin section. Absence of mafics except for the few flakes of biotite in this section is conspicuous.

3.1.2 Hutti Metavolcanics

On the E-W arm of the Hutti Schist Belt, south of Chinchargi village, metabasalts showing pillow structure at places are encountered. Based on the grain size three varieties of metabasalts could be found, viz., a coarse-, a medium- and a fine-grained variety which were sampled (Plates 3.3a, 3.3c and 3.4a). Another medium- to coarse-grained amphibolite outcropping to the east of Chinchargi was also sampled.

The petrographic study of the thin sections of the above metabasalts showed that they are amphibolites, predominantly made up of magnesiohornblende, whose composition was confirmed by the XRD analysis of their sample powders (Appendix II). The long axes of amphiboles are oriented sub-parallel to each other which defines the schistosity. Plagioclase feldspars that are labradorite in composition are present as the next major mineral in these samples. Epidotization of the plagioclase feldspars is not uncommon. Minor quartz and chlorite are also present. Plates (3.3b, 3.3d and 3.4b) show the photomicrographs of the thin sections of these three varieties of metabasalts.
The medium- to coarse-grained inequigranular metabasalt sampled east of Chinchargi also shows magnesiohornblende, plagioclase feldspar (labradorite), and quartz in that order of abundance. Bending of cleavage due to shearing could be observed. Amphiboles also exhibit simple twinning (Plate 3.4c).

Amphibolitic metabasalts are found east of the Uti-Palkanmardi road. These rocks are fine- to medium-grained and show well-developed schistosity. In the thin section the sample is medium-grained and is composed of amphiboles that are magnesiohornblende in composition and plagioclase feldspar. Minor quartz, chlorite, titanite and opaques are also present. The sub-parallel orientation of amphiboles along their c-axes defines the schistosity. The mafic and felsic minerals show imperfect banding (Plate 3.4d).

On the Hutti river bed, near Madrainakota, two varieties of metabasalts are found. The coarse-grained variety is massive while the medium- to fine-grained variety is schistose (Plate 3.5a). The contact between these two varieties appears to be tectonic (Plate 3.5b). Their thin section study showed presence of hornblende, plagioclase feldspar and quartz in that order of abundance. The medium- to fine-grained variety shows parallel arrangement of hornblende along its long axis which defines the schistosity. Both samples show epidotization at places (Plate 3.5 c and d).

The rocks of the N-S arm of the schist belt are well foliated and have undergone higher degree of shearing compared to the rocks of the E-W arm of the schist belt. Quartz, quartzo-feldspathic and carbonate veins intrude the rocks of the schist belt. However, the intensity of veins is much higher along the N-S arm relative to the E-W arm. Outcrops of chlorite-carbonate schist are found at several places, near Maski, Pamankallur and Gajalagatta to name a few (Plates 3.6 a and b). These rocks show well-developed schistosity striking roughly N-S. A sample of each was collected for isotope geochemical studies. An outcrop of talc-tremolite schist was also sampled west of Pamankallur.

Thin sections were made from the N-S arm metabasalts and their petrography was studied. The chlorite-carbonate schist collected east of Maski shows abundant chlorite, calcite, quartz and plagioclase. The rock is fine-grained and the shearing is pervasive and anastomosing.

The chlorite-carbonate schist occurring west of Pamankallur is fine-grained and composed chiefly of chlorite which is predominantly the Mg-rich clinochlore, as confirmed by XRD analysis (Appendix II). Calcite is the next major mineral which is
secondary and introduced by CO₂-rich hydrothermal fluids. Quartz, plagioclase and epidote were also observed. The minerals are arranged along the schistosity (Plate 3.6c). Another sample collected west of Pamankallur, called as talc-tremolite schist by Giritharan and Rajamani (1998) is extremely fine-grained and shows highly altered chlorite grains, occurring along with quartz, and calcite.

The chlorite-carbonate schist collected south-west of Gajalagatta is rich in chlorite and calcite followed by quartz and minor plagioclase. The calcite grains are larger in size compared to the fine-grained chlorite (Plate 3.6d). Presence of relict amphiboles surrounded by fine-grained chlorite is noticed in several samples. It is inferred that the present mineral assemblage developed from an amphibolite precursor due to intense shearing in the presence of large volumes of fluids. Presence of quartz-calcite veins along the schistosity indicates that these were formed prior to later phases of deformation and the fluid was rich in CO₂.

Two samples of felsic volcanics were collected from the Hutti Schist Belt. One of them found southwest of Uti in the E-W arm of the Hutti Schist Belt is fine-grained (Plate 3.7a). The thin section showed presence of quartz phenocrysts in a matrix made up of quartz and plagioclase feldspar. Minor amount of alkali feldspars such as microcline is also present. Based on the mineralogy it is suggested that this rock may be dacitic or andesitic in composition (Plate 3.7b).

The felsic volcanic rock sampled east of Gajalagatta in the N-S arm of the Hutti Schist Belt is rhyolitic in composition having quartz phenocrysts (Plate 3.7c). The thin section showed the sample to be fine-grained with the groundmass made up of predominantly quartz with embayed quartz phenocrysts (Plate 3.7d). Pyroclastic rocks showing pitted structure are also found to occur in and around Wandali (Plate 3.8a).

Based on the above field and petrographic studies it is proposed that a clear distinction exists between the metabasalts occurring in the E-W and N-S arms of the Hutti Schist Belt in terms of mineral assemblage, extent of shearing and intensity of quartz-calcite veining. The metabasalts of the E-W arm distinctively contain amphiboles as predominant ferromagnesian mineral along with calcic plagioclase, labradorite and have negligible quartz-calcite veins. The metabasalts of the N-S arm are invariably enriched in chlorite, formed by the alteration of amphiboles preserved as relict grains, and characterized by intense quartz-calcite veining.
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3.1.3 Kolar Granitoids

Several traverses were taken along and across the schist belt and its environs to understand the disposition of the schist belt and the surrounding granitoids. Samples were collected from the western Dod and Dosa gneisses at locations south of KGF-Bangarapet road near villages Dasarhoshalli and Devghanhalli. Good exposures of granodiorites (Dosa gneisses) and quartz diorites (Dod gneisses) are found in these areas. The granodiorites and the quartz diorites are made up of abundant quartz, feldspar, biotite, hornblende and specs of accessory titanite. Mafic enclaves and quartz and quartzo-feldspathic veins are also common.

The petrographic study of the thin sections of the above samples shows that these granitoids are medium-grained, inequigranular and composed predominantly of quartz, oligoclase and microcline. Biotite and hornblende constitute the mafic minerals with epidote often associated with biotite. Subhedral to euhedral titanite, apatite, opaques and euhedral grains of colourless to pale pink zircons are found as the common accessory minerals in thin sections. The plagioclase feldspars are altered as evidenced from the presence of fine, micaceous sericite on the surface and association of epidote along their margins. Titanites show a pale brown to dark brown pleochroism. Some of the titanite grains show zoning.

Huge blocks of granitoid rocks crop out near Patna village, northwest of KGF and west of the schist belt. These show poor gneissosity with the composition ranging from granite to granodiorite and contain mafic and microgranular enclaves, ranging in size from 0.1 to 10 m (Plate 3.8b). The rocks contain abundant quartz, K-feldspar, plagioclase, biotite, hornblende, epidote and titanite. A sample was collected from this outcrop.

Thin section study of the Patna Granite showed that it is comprised of abundant quartz and feldspars. The feldspars are predominantly orthoclase showing simple twinning with minor microcline exhibiting tartan twinning. Plagioclase feldspars present are highly altered and exhibit epidotization at places. Biotite constitutes the mafic mineral and they also show twinning at places. Titanites are present in good numbers which are anhedral to euhedral (Plate 3.8c). Opaques, viz., magnetite and ilmenite are found associated with titanite.

From the eastern Kambha Gneisses three samples were collected. One sample was collected from an exposure of granodiorite gneiss east of Guttapalli village, south of the KGF-Kyasamballi road. The rock is made of quartz, plagioclase, biotite, minor
hornblende and plenty of titanites. A sample of granodiorite gneiss was collected from 1 km northeast of Satu village. South of the KGF-Kuppan road, immediately south of the village Tippanapalli a good exposure of granitic to granodioritic gneisses is observed. The rock has abundant quartz, feldspar, plagioclase, biotite, hornblende, epidote and remarkably big titanites up to 1 cm in size. All the sample locations from the Kolar area are marked in Fig. 2.4 and are from the same locations and outcrops which were sampled earlier for geochemical and geochronological studies of Balakrishnan and Rajamani (1987) and Krogstad et al. (1991 and 1995), in order to integrate the data obtained in the present work with already existing data.

The petrography of the thin sections showed these gneisses to be rich in quartz, plagioclase, K-feldspar, biotite and minor hornblende. Epidote, titanite, apatite, opaques and zircon are present in accessory amounts (Plate 3.8d). The titanite grains in some samples are coarse measuring from about 2 mm to 5 mm. Zoning can be observed in some of the titanites. Allanite is present which shows characteristic pleochroic halo around it due to radioactivity. Epidotization and sericitization are also observed in some thin sections.

3.1.4 Ramagiri Granitoids

Field study and sample collection was carried out in the granitoids surrounding the schist belt on the same outcrops on which U-Pb geochronological studies were reported by Balakrishnan et al. (1999) and geochemical studies by Mohanta (1998) in order to integrate already existing data with the data generated in this study. Samples from the Ramagiri area were collected for geochemical study in titanites, as well as, for the Sm-Nd mineral isochron studies.

Exposures of granodiorites, quartz-monzodiorites, granites and quartz-diorites are seen in the western Gangam Complex, on either sides of the Nasanakota-Gangampalli road. The quartz-monzodiorite has abundant titanite apart from plagioclase, quartz and mafic minerals such as biotite and hornblende. The granodiorite shows porphyritic texture with abundant quartz, plagioclase and biotite and accessory titanite. Samples were collected from these localities.

Based on petrographic study of thin sections it was found that the Gangampalli quartz-monzodiorite and granodiorite are rich in plagioclase feldspars with the former having lower modal abundance of quartz. Quartz grains arranged along the foliation are fine-grained. K-feldspar is also present. Biotite is the major mafic mineral present.
Sericitization of plagioclase feldspar is common. Titanite, zircon, apatite and opaques are present as accessory minerals. Titanites are altered at some places and show minor zoning. Titanites are highly pleochroic from pale brown to dark brown. A photomicrograph of Gangam granodiorite is shown in Plate 3.9a.

In the central Ramagiri Complex, on the northern side of the Muttavakunta-Ramagiri road, good exposures of quartz-diorite composed of plagioclase, quartz, hornblende, biotite and titanite are found. Chloritisation is also observed in these rocks. A sample of the quartz-diorite was collected.

The petrographic study of the thin section this sample showed the rock to be primarily composed of plagioclase feldspars which are sericitized at several places. Minor epidotization can also be observed. Quartz grains are larger in size. Microcline showing cross-hatched twinning is also present. Biotite is present in large numbers. Anhedral to subhedral titanites are associated with opaque minerals (Plate 3.9b).

In the eastern gneissic terrane referred to as Chenna Gneisses, near Chennakottapalle, outcrops of granodiorite gneisses are found. These gneisses show migmatitic texture and are composed of plagioclase, quartz, biotite and hornblende. A diabasic dike cuts across at the eastern end of the outcrop. A sample of the granodiorite was collected from this location. A sample was also collected from the hill Δ 458, located east of Ramagiri village and west of Nagasamudram Gate. The hill is almost entirely made of granitic gneisses. The gneisses lack migmatitic texture and have relatively less biotite. The gneisses are made up of quartz, feldspar, hornblende and visible specs of titanite. Late pink granite intrusions in these gneisses are observed at several places. A field photograph of an outcrop of these rocks is shown in Plate 3.9c. The granitic gneisses are cut across by an approximately 400 m long diabasic dike. Sample locations are indicated in the geological map of Ramagiri area (Fig. 2.5).

The thin section of the Chenna Gneiss shows abundant quartz, K-feldspar, plagioclase and biotite. Titanites exhibiting pleochroism are present disseminated throughout the section (Plate 3.9d). Zircon grains pale pink in colour has been observed.
Plate 3.1

a. Pink granite outcropping near Golapalli village north of the Hutti Schist Belt. Megacrysts of K-feldspars are abundant.

b. Photomicrograph of megacrystic K-feldspar showing simple twin in Golapalli granodiorite. Anti-perthitic intergrowth can also be observed. Quartz and microcline also seen. Crossed Polars.

c. Porphyritic pink granite outcrop near Yelagatti, north of the Hutti Schist Belt.

d. Palkanmardi mixtite (polymictic conglomerate) showing clasts of granodiorite in a matrix of basic rock.
Plate 3.2

a. Photomicrograph showing altered feldspar, quartz, biotite and hornblende in Kardikal granodiorite. An euhedral titanite included in biotite is seen in the lower left part. Plane Polars.

b. Intrusive contact between the Eastern Granitoids and the metabasalts of the Hutti Schist Belt as seen near Gajalagatta.


d. Photomicrograph showing altered feldspar, hornblende and biotite in Gajalagatta granodiorite. A zircon grain is seen embedded in the flaky green biotite. Plane Polars.
Plate 3.3

a. Coarse-grained amphibolite occurring south of Chinchargi in the eastern part of the Hutti Schist Belt.

b. Photomicrograph of the coarse-grained metabasalt, sampled south of Chinchargi, with larger grains of hornblende along with albitic plagioclase feldspars. Plane Polars.

c. Medium-grained amphibolite occurring south of Chinchargi in the eastern part of the Hutti Schist Belt.

d. Photomicrograph of the medium-grained metabasalt showing abundant amphiboles, chiefly hornblende, along with albitic plagioclase feldspars. Sample collected south of Chinchargi. Plane Polars.
Plate 3.4

a. Fine-grained amphibolite occurring south of Chinchargi in the eastern part of the Hutti Schist Belt.

b. Photomicrograph of the fine-grained metabasalt, south of Chinchargi, predominantly made up of fine-grained hornblende and plagioclase feldspars. Plane Polars.

c. Photomicrograph of the medium- to coarse-grained amphibolite rich in amphiboles, plagioclase feldspar and opaques. Sample collected east of Chinchargi. Bending of cleavage can be observed. Plane Polars.

d. Photomicrograph of the medium-grained metabasalt exposed east of Uti-Palkanmardi road, showing minerals oriented along the schistosity. Hornblende and plagioclase feldspars are the common minerals. Plane Polars.
Plate 3.5

a. Fine-grained schistose metabasalt found on the Madrainakota river bed north of Hutti village. The schistosity trends roughly E-W.

b. Contact between the medium- to fine-grained schistose (lower part of the photograph) and coarse-grained massive metabasalts (upper part of the photograph) occurring on the Madrainakota river bed north of Hutti village. The pen in the middle of the photograph lies parallel to the contact between these two varieties of metabasalts. The contact appears to be tectonic.

c. Photomicrograph of the coarse-grained massive metabasalt, found associated with the schistose variety in the Madrainakota river bed, made up of amphiboles, plagioclase feldspars and opaques. Crossed Polars.

d. Photomicrograph of the medium- to fine-grained amphibolite with amphiboles oriented along the schistosity collected from the schistose variety exposed in the Madrainakota river bed. Plane Polars.
Plate 3.6

a. Highly weathered chlorite-carbonate schist with the schistosity striking N-S occurring west of Pamankallur along the N-S arm of the Hutti Schist Belt.

b. Chlorite-carbonate schist exposed southwest of Gajalagatta village on the eastern side of the N-S arm of the Hutti Schist Belt exhibiting well-developed schistosity striking N-S. Quartz-carbonate veins are abundant in this outcrop.

c. Photomicrograph of the fine-grained chlorite-carbonate schist showing calcitic vein set in a matrix of chlorite. Sample collected west of Pamankallur. Crossed Polars.

d. Photomicrograph showing vein calcite along with fine-grained chlorite in a sample of metabasalt collected southwest of Gajalagatta. Crossed Polars.
Plate 3.7

a. Felsic volcanic sample exposed southeast of Uti, in the northeastern part of the E-W arm of the Hutti Schist Belt.

b. Photomicrograph of the fine-grained felsic volcanic made up of quartz and feldspar collected southeast of Uti. A large grain of microcline is also seen on the southwest quadrant of the photograph. Crossed Polars.

c. Fine-grained felsic volcanic rock with calcite veins exposed near Gajalagatta close to the contact between the Eastern Granitoids and the Hutti Schist Belt rocks.

d. Photomicrograph of the fine-grained felsic volcanic rock dominated by quartz and feldspar. Quartz grains are also present as embayed phenocrysts. Sample collected east of Gajalagatta. Crossed Polars.
Plate 3.8

a. A Pyroclastic rock exposed near Wandali village, east of the Hutti Schist Belt showing pitted structure on the surface.

b. Mafic enclave hosted by the Patna Granite occurring north of the Kolar Schist Belt.

c. Photomicrograph showing quartz, feldspar and a millimeter long crystal of titanite in Patna Granite. Crossed Polars.

d. Photomicrograph showing quartz, altered feldspar and a twinned euhedral titanite in the Tippanapalli granodiorite from Kambha Gneiss. Crossed Polars.
Plate 3.9

a. Photomicrograph showing quartz, feldspar, biotite and titanite in Gangam granodiorite. Crossed Polars.

b. Photomicrograph showing sericitized plagioclase and titanite in the granodiorite from Ramagiri Complex. Crossed Polars.

c. Granodiorite gneisses (Chenna Gneiss) outcropping east of the Ramagiri Schist Belt near Chennakotapalli.

d. Photomicrograph showing quartz, feldspar, biotite and anhedral titanite in the migmatized Chenna Gneiss. Plane Polars.
3.2 Mineral Separation:

U-Pb isotope geochemical study requires pure mineral separates of U-rich heavy minerals such as zircon and titanite from granitoid rocks. K-feldspar mineral separates are required to estimate Pb isotope composition of common lead. Further, titanite, hornblende and biotite mineral separates from granitoid rocks are required for the Sm-Nd isotope study on these minerals. Conventional mineral separation techniques were followed for the separation of minerals.

Mineral separation procedure starts with the crushing and powdering of sample. About 5 to 10 kg of the whole rock sample was cleaned and crushed using a hardened steel mortar and pestle to a size fraction of about -80 ASTM mesh (equivalent to a size between ~177 and ~149 μm). This size fraction is preferable as minerals in this size range seldom form aggregates and are suitable for density based separation using heavy liquids. This size fraction also has considerable finer rock powder which is effectively removed by continuous washing and decanting the suspension. The heavy minerals (having specific gravity >2.89) were then concentrated by panning followed by bromoform separation.

Bromoform is a heavy liquid with a specific gravity of 2.89 at 20°C. Heavy minerals are those that have a specific gravity greater than that of bromoform. About 500 ml of bromoform was taken in a separating funnel. About 100 g of the -80 ASTM mesh sample powder was transferred into the separating funnel and stirred with a laboratory stirrer continuously for 5 minutes to unclog loosely held mineral aggregates. After stirring, the separating funnel is left undisturbed for 30 minutes. The heavy minerals settle due to their higher specific gravity at the bottom of the separating funnel, which are collected through a filter paper. The separating funnel contents are then stirred further to collect more heavy minerals in the above mentioned fashion, until sufficient heavies are recovered from the 100 g fraction. This procedure was repeated for the heavy mineral separation in the remaining sample powder. The lighter fraction was retained for the separation of K-feldspar.

The heavy mineral fraction separated using bromoform as described above in a granitoid sample is made up of biotite, hornblende, epidote, ilmenite, magnetite, apatite, titanite, zircon and allanite along with some lighter minerals such as quartz, K-feldspar and plagioclase feldspar that were found attached to some of the heavy minerals. Strongly magnetic minerals like magnetite were first removed by combing a hand magnet, wrapped in paper through the heavy mineral fraction several times. The
individual minerals were then separated based on their magnetic susceptibility, at different currents using Frantz® Isodynamic Magnetic Separator (Model LB-1). Details of the operation of the Frantz® Isodynamic Magnetic Separator and flow chart for mineral separation from granitic rocks are given in Mursky (1987). The chute positioned between the pole pieces of the electromagnet is kept at a particular forward slope and side slope. A magnetic field is produced by applying current to the electromagnet. The heavy mineral fraction is then passed through the chute, which is vibrated to get a constant flow of the sample fraction through the chute. The minerals that are more magnetic are separated from those that are less magnetic and are collected in separate buckets at the end of the chute. This step was followed by systematically increasing the magnet current and the individual minerals were thus separated. Titanites were separated at 1 A current and zircons were separated at 1.6 A current.

The non-magnetic fraction at 1.6 A includes zircon and apatite of the heavy mineral fraction, as well as, quartz and feldspars as they have similar magnetic susceptibilities and hence, are not well separated by the Frantz® magnetic separator. Another heavy liquid, di-iodomethane, having a specific gravity of 3.31 was used for the separation of apatite from titanite and zircon in a separating funnel. Further purification of individual minerals was done under a binocular microscope.

From the lighter fractions separated by bromoform, K-feldspars were separated initially using Frantz® magnetic separator and finally purified by handpicking under the microscope.

3.3 U-Pb isotope analysis on titanites and zircons:

U-Pb isotope analysis on titanites and zircons was carried out at the Zentrallabor für Geochronologie (ZLG), Institut für Mineralogie, Universität Münster, Germany. Separation of pure U and Pb from titanites and zircons by column chromatography is essential for this isotope study. Titanites and zircons using the magnetic separator were handpicked under a binocular microscope. The handpicked mineral grains were air-abraded using an abrasion mill to eliminate the rims that were affected by radiation damage to a higher extent relative to the core (Krogh, 1982). The abraded grains were cleaned several times with dilute acid and ultra pure water. About 2 to 5 mg of the grains were accurately weighed in a 3 ml Savillex® vial using a high precision analytical balance, and a known quantity of \(^{205}\)Pb tracer solution was added.
About 1 ml concentrated HF and few drops of concentrated HNO₃ were added to the vial, capped and put into Teflon®-inner lined metal bombs. The bomb was then kept inside a hot air oven at 200°C for several hours. After the sample was completely digested, the acid remaining inside the vial was dried out on a hot plate. About 1 ml of 3 N HCl was added to the dried beaker contents and left covered over the hot plate overnight. The column chromatography for the separation of U and Pb was then carried out using the procedure followed at the laboratory in Muenster (Tilton, 1973; Manhes et al., 1984; Mattinson, 1987; Mezger et al., 1991).

For the estimation of common Pb isotopic composition feldspar grains were leached by the addition of few drops of ultra pure water and HF and warming briefly on the hot plate. The acid-water mixture was then pipetted out and the feldspars were washed with ultra pure water three times. More acid (~10 ml) and less ultra pure water were added in the next step. When the feldspars become half their original size, the acid-water mixture was pipetted out and the remaining feldspars were washed several times and taken for Pb separation using anion exchange columns.

AG 1X 8, 100 - 200 (mesh) anion exchange resin was used in 0.2 ml Teflon® columns for the U and Pb separation. After equilibrating the columns with 1N HBr, the digested and dried samples were dissolved in 1 ml of 3N HCl and loaded into the columns. U and major elements were collected with 1N HBr followed by the collection of Pb in 1 ml of 6N HCl. The HBr cut thus collected was mixed with few ml of 7N HNO₃ and dried. The dried contents were dissolved in 0.5 ml of 6 N HCl and loaded into the same columns for the separation of U from major elements. After removing the major elements by washing with 6N HCl and 7N HNO₃, U was collected in about 1 ml of H₂O. The Pb and U thus collected were dried after adding 1 drop of 1M H₃PO₄. Any organic contaminants from the resin were removed by adding few drops of aqua regia and drying.

Pb was loaded on a single rhenium filament with silica gel and 6N HCl. U was loaded on a rhenium double filament assembly with 1N HNO₃. The Pb and U were analyzed on a Thermal Ionization Mass Spectrometer (TIMS, Model: VG Sector 54). NBS 982 standard for Pb and U-500 standard for U were run repeatedly to estimate fractionation correction factor and to check the accuracy and precision of the instrument.
3.4 Rb-Sr and Sm-Nd Isotope Analysis:

During the course of this Ph.D. work, a new isotope geochemical laboratory and Thermal Ionization Mass Spectrometer (TIMS, Model: Triton-Thermo Finnigan) were set up in the Department of Earth Sciences, Pondicherry University, Puducherry. The initialization of Rb-Sr and REE group separation and Sm-Nd separation and their isotope analysis required preparation of Rb-Sr and Sm-Nd isotope tracer solutions and their calibration and setting up of HCl and HDEHP columns.

3.4.1 Rb-Sr and Sm-Nd Isotope Tracer Solution Calibration

An isotope tracer (also known as spike) is an element with one of its isotopes enriched relative to its natural abundance. An isotope tracer solution is required for isotope dilution method of analysis for determination of elemental concentration with high order of accuracy and precision. Isotope dilution analysis involves addition of a known amount of isotope tracer solution to an accurately weighed sample solution. The abundance of the element to be determined and its isotopic composition in the tracer solution are precisely known. The isotopic composition of that element is either fixed (e.g. Rb) or measured using the TIMS (e.g. Sr) and the isotopic composition of the mixture is used for the determination of the concentrations of elements.

Rb isotope tracer with 98.10% enrichment of $^{87}\text{Rb}$ tracer and Sr isotope tracer with 68.7% enrichment of $^{84}\text{Sr}$ were obtained in powder form (RbCl and SrCO$_3$) and were made into solutions of known concentrations gravimetrically using high-precision analytical balance (Mettler Toledo AG245 and Sartorius ME 235S). 99.75% pure form of Rb metal was obtained from Alfa Aesar and used for the preparation of Rb calibrating solution of known concentration gravimetrically (concentrations of calibrating solutions given in Appendix III). For Sr, SRM 987 elemental standard (in the form of SrCO$_3$) was used as calibrating solution.

Sm isotope tracer with 99% enrichment of $^{152}\text{Sm}$ and Nd isotope tracer with 97.84% enrichment of $^{150}\text{Nd}$ were obtained as oxides and were made into solutions of known concentrations gravimetrically on a high-precision analytical balance. 99.9% pure form of Sm and Nd metals of Johnson Matthey Company provided by Prof. V. Rajamani, Jawaharlal Nehru University, New Delhi were used for the preparation of calibrating solutions of known concentration gravimetrically. The tracer solution concentrations were determined accurately by analyzing the tracer solutions against
the calibrating solutions repeatedly using the TIMS. The Rb and Sr tracer solution concentrations were re-checked against a solution of K-feldspar of known concentration provided by Prof. K. Mezger, University of Muenster, Germany. The Sm and Nd tracer solution concentrations were re-checked against Ames high purity Sm and Nd metals provided by Prof. K. Mezger, University of Muenster, Germany. The isotopic abundances of the Rb-Sr and Sm-Nd isotope tracer solutions were determined precisely using the TIMS. The isotopic compositions were also determined precisely using the TIMS. Double distilled (using sub-boiling quartz still) EMerck GR grade hydrochloric acid and Milli Q ultra pure water were used for the preparation of tracer solutions and calibrating solutions. The abundances of various isotopes in the tracer solutions are given in Appendix III.

3.4.2 Rb-Sr Column Calibration

Bio-Rad AG50-WX8 cation-exchange resin (200-400 mesh) was used in 3 ml primary HCl quartz columns for the separation of Rb, Sr and REE as a group. The columns were calibrated for the elements Rb, Sr, Ca, Fe, Mg, Al and REE. Known amount of the above pure elements was taken as a solution in 1 ml of 2N HCl and loaded onto the cation-exchange columns. The elution was carried out using calibrated double-distilled 2N HCl (EMerck GR) in one ml steps. The REE as a group were separated using calibrated 6N HCl. The cuts thus collected were analyzed using ICP-AES (Ultima 2, Jobin Yvon) and the data were plotted (Fig. 3.1).

Fig. 3.1 Plot showing concentrations (in ppm) of different elements that were separated using cation-exchange resin filled HCl column which was used for the calibration of these columns for the separation of Rb, Sr and bulk REE.
### 3.4.3 Sm-Nd Column Calibration

HDEHP (2-ethyl hexyl hydrogen phosphate) column was prepared following the procedure described by Richard *et al.* (1976) and Gioia and Pimentel (2000). About 1 g of Teflon® powder (provided by Prof. K. Mezger, University of Muenster, Germany) in 10 ml of acetone was coated with approximately 0.1 ml HDEHP by stirring the mixture for about 5 to 6 hours. 1 ml quartz columns were filled to a height of 0.5 cm with Bio-Rad AG1-WX8 anion-exchange resin followed by 3 to 4 cm HDEHP resin, which was pressed to optimum compaction. The column was then topped with about 0.5 cm with Bio-Rad AG1-WX8 anion-exchange resin.

Known amount of pure REE (La, Ce, Pr, Nd, Sm and Eu) element standards (obtained from SPEX CertiPrep, USA) were mixed and dried on a hot plate. This was dissolved in 200 µl of calibrated double distilled 0.18 N HCl and loaded onto the HDEHP column (procedure modified from Richard *et al.*, 1976 and Gioia and Pimentel, 2000). The elution was carried out using calibrated 0.18 N HCl, 0.3 N HCl and 0.4 N HCl in one ml steps. The cuts collected were analyzed using ICP- AES and the data were plotted. This procedure was followed separately for each of the HDEHP columns as the elution behaviour of these columns differ from each other due to amount of HDEHP coated Teflon® powder packed in each column and differences in the degree of HDEHP coating on the Teflon® powder. The plot for the REE elusion in one of the HDEHP columns is shown in Fig. 3.2. The separation in these conditions was good with complete removal of Sm from Nd.

![Fig. 3.2 Plot showing concentrations (in ppm) of rare earth elements that were separated using HDEHP column which was used for the calibration of these columns for the separation of Nd and Sm from the rest of the REE.](image-url)
3.4.4 Rb, Sr, Sm and Nd Separation

Sample digestion for Rb-Sr and Sm-Nd isotopic analysis was carried out using the following procedure. About 200 mg of the −200 mesh (ASTM) powdered rock sample was taken and weighed precisely into a pre-weighed PTFE/Savillex® beaker. About 1 to 2 ml of HF + 1ml HNO₃ + few drops of HCl were added to the sample beaker and left on the hot plate at 110°C overnight with the lid on for digestion. The acid was then dried and about 1 ml of HNO₃ was added three times and dried to remove any traces of HF present. For the digestion of 10 to 20 mg of hornblende and titanite mineral separates proportionally lesser amount of acids were used.

Before the separation of individual elements for isotopic analysis, the beaker contents were dissolved in dilute HCl and split into two fractions. One of the fractions was used for the determination of the Isotopic Composition (IC) of Sr and Nd while the other was used for the determination of Rb, Sr, Sm and Nd concentrations by Isotope Dilution (ID).

The IC and the ID fractions were passed through Bio-Rad AG50-WX8 cation-exchange resin filled HCl columns. Rb and Sr were collected with 2N HCl and REE as a group was collected with 6N HCl. Rb and Sr fractions thus separated were purified further by passing through a set of secondary Bio-Rad (PP) columns that were also filled with Bio-Rad AG50-WX8 cation-exchange resin and calibrated for the above mentioned elements. The collected Rb and Sr solutions were dried.

The REE eluted as a group HCl column was dried and dissolved in 200 µl of 0.18 N HCl. The IC and ID REE fractions were passed through different HDEHP columns. Nd was collected with 0.3N HCl and Sm was collected with 0.4N HCl. A drop of ~0.5M H₃PO₄ was added to the collected Nd and Sm and dried. H₃PO₄ ensures visibility of Nd and Sm while picking up for loading onto the filament.

3.4.5 Mass Spectrometry

Loading of Rb and Sr for mass spectrometry was done using the following procedure. 1 µl of Tantalum oxide (TaO) activator was loaded on a pre-warmed rhenium (Re) single filament and dried at 0.5 A current. Rb was dissolved in 1 to 2 µl of 1N HCl and loaded on top of TaO on the filament and dried at 0.5 A current. 1 µl of Tantalum oxide (TaO) activator was again loaded on top of the sample making a sandwich of sample between layers of TaO. The filament was then heated to dull red colour for about 10 seconds and loaded on the turret of the TIMS for analysis. Sr was
dissolved in 1 µl of 1N HNO₃ and loaded between two layers of Tao on a rhenium filament and dried at 0.5 A current. The filament was then heated to dull red colour for about 20 seconds and loaded on the turret of the TIMS for analysis.

Nd and Sm were dissolved separately in 1 µl of 1N HNO₃ and were loaded on pre-warmed rhenium (Re) double filaments for mass spectrometry. A double filament assembly includes two filaments of which one is used as sample evaporation filament and the other is used as ionization filament. The sample was dried at 0.5 A current. The filament was then heated to dull red colour for about 20 seconds until all the H₃PO₄ evaporated and were loaded in the turret of the TIMS for analysis.

During the course of this study Sr isotope standard SRM 987 and Nd isotope standards La Jolla (provided by Dr. G. Lugmair, Scripps Institution of Oceanography, USA) and Ames were run repeatedly and the results are given in Appendix IV. Instrumental mass fractionation during the determination of Sr and Nd isotopic compositions in standards and samples was corrected by internally normalizing the measured ratios by the ratios $^{86}\text{Sr}/^{88}\text{Sr} = 0.1194$ for Sr and $^{146}\text{Nd}/^{144}\text{Nd} = 0.721878$ for Nd using exponential fractionation law. The mean value obtained on 40 analyses of SRM 987 for Sr is $0.710244 \pm 5$. The mean value obtained on 30 analyses of La Jolla for Nd is $0.511848 \pm 4$ and 25 analysis of Ames for Nd is $0.511969 \pm 4$.

3.5 Major and Trace Element Analysis of Titanites:

For the analysis of major elements on titanites thin and polished sections of the granitoid rocks from Kolar and Ramagiri area were made. These were coated with carbon and used for the analysis of major elements using an Electron Probe Micro Analyser (EPMA - model S x 50 Cameca) at the Department of Geology, Mysore University. The EPMA was calibrated with mineral standards such as biotite, apatite, anorthite, diopside, ilmenite and rutile. Suitable points on the titanite mineral grains were chosen and analyzed for the major elements.

For the analysis of trace elements, titanites were separated from the whole rock powders of granitoids from Ramagiri and Kolar areas by conventional heavy liquid and magnetic separation techniques, described earlier (section 3.2). The titanite mineral grains were further purified by handpicking under the binocular microscope. About 20 to 50 mg of titanite mineral grains from each sample were digested using 3 ml HF + 1 ml HNO₃+ 0.5 ml HCl acid (B-Solution). This digested solution was used for the analysis of trace elements such as Nb, Ta, Ba, Sr, Y, Zr, Th, U, Pb, Sc and
REE using an Inductively Coupled Plasma – Atomic Emission Spectrometer (ICP-AES, Model: Ultima 2, Jobin Yvon) (REE at Jawaharlal Nehru University, New Delhi and other trace elements at Pondicherry University). The REE analysis involved their separation and pre-concentration as a group by cation-exchange chromatography. Dowex – X 8 (100 – 200 mesh) cation-exchange resins was used in a two column procedure involving HNO₃ and HCl columns.