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APPENDICES

APPENDIX A

Chemical Analysis of rock samples.

The major and minor constituents of rock samples were analyzed by using the "rapid Analysis of silicate, carbonate, and phosphate rocks" (Shapairo L.,1975): two solutions method, A and B, as follows:

Preparation of solution A

Solution A is used in the determinations of SiO_2 and Al_2O_3. Portions of the samples and standards are decomposed by fusion with NaOH at a comparatively low temperature for about 10 min in nickel crucibles. After cooling, the melts are leached with water, and the solutions are acidified with hydrochloric acid.

Reagents

NaOH solution, 30-percent: Dissolve 450g of NaOH pellets in 1,500 ml of water in a stainless steel beaker. Cool and store in a plastic bottle.

HCl, 1+1: Prepare 1l.

Standard

Standard were prepared from the mixture of $CaCO_3$, $MgCO_3$, SiO_2 and Al_2O_3 as:

$CaCO_3$	$MgCO_3$	SiO_2	Al_2O_3
90%	6%	2%	2%
85%	9%	3%	3%

And the working standard are 0.5%, 1%, 1.5%, 2%, 3%, 4%, and 6% for SiO_2 ; 0.5%, 0.75%, 1% and 1.5% for Al_2O_3.

Procedure

1. Transfer 5-ml portions of 30-percent NaOH solution, measured with a plastic graduate, to a series of 75-ml nickel crucibles. one crucible will be needed each sample, two for standards, and one for a blank. The crucibles should be cleaned with dilute HCl before use.

2. Evaporate the solutions to dryness over a hot plate. Slight spattering can be ignored.

3. Accurately weigh 200 mg of each powder sample and two 50-mg portions of prepared standard . As each portion is weighed, transfer to a crucible containing the fused NaOH.

4. Cover and heat the crucibles to dull redness for about 10 min. Remove each crucible from the heat and swirl the melt around the sides. Allow the melts to cool.

5. Transfer the crucibles plus contents to a series of 1l plastic beakers.

6. Add about 300ml of water to each beaker. Allow to stand for at least 1hour.

7. Stir the contents of each beaker using a plastic rod.

8. Add 20 ml of 1+1 HCl to each beaker, and stir. If the sample solution is not clear, add another 5 ml of 1+1 HCl and wash crucible by distilled water before taking out from the solution.

9.Transfer the clear sample solutions to the volumetric flask 500-ml, make volume and keep them in polyethylene bottles.

Preparation of solution B

Solution B is used in the determination of total iron, MgO, CaO, P_2O_5. The samples are digested on a steam bath overnight with a mixture of HF, H_2SO_4 and HNO_3. This procedure decomposes almost all the minerals that are normally present.

Organic matter is destroyed by the addition of a few drops of a mixture of perchloric and nitric acids after heating to sulfuric acid fumes.

Reagents

Solution B acid mixture: Working under a fume hood, transfer the contents of a 454-g bottle of HF (48 percent) to a 1-L polyethylene bottle. Chill the HF in a bath of cold water. Keep the polyethylene bottle in the cold-water bath, add 165 ml of concentrated H_2SO_4, mix, and allow to cool. Add 40 ml of concentrated HNO_3 and mix.

$HClO_4$-HNO_3Mixture : Mix 100ml of $HClO_4$ (72 percent) and 100ml of concentrated HNO_3 Store in a 200-ml glass-stop-pered Pyrex bottle.

Hydrazine-sulfate solution, 0.2 percent: Dissolve 0.2 g NH_2 NH_2 SO_4 in 100 ml of water, freshly prepare for each run.

Procedure

1. Transfer 0.500g of each sample to a Teflon beaker.

2. Add approximately 2drops of distilled water for wetting to each beaker containing powder sample.

3. Add 1:1 nitric acid (HNO_3) drop wise to each sample until there is no gas evolved.

4. Add 15 ml of mixture of HF, H_2SO_4 and HNO_3 to each sample.

5. Cover the beakers, put them on the hot plate at 80°C for 4 hours and continue heating at 100°C for 30 minutes and increase the temperature to 150° and 180°C respectively until there is no more while fume of SO_3 evolved.

6. Open the Teflon covers, place beakers on a hot plate with temperature 100°, 150° during ½ hour, then addition of temperature to 180°-200° continued until the samples will be dried.

7.Remove the beakers from the hot plate, allow them to cool for a few minutes, and then add about 225ml of water, 5ml of concentrated HNO_3, and 1ml of the hydrazine sulfate solution. Heating to boiling.

8. Cool the solutions to room temperature, then transfer them to 250-ml volumetric flasks.

9.Make to volume, mix, and transfer to the polyethylene bottles.

Determination of silica (SiO_2) by colorimetry

For determination of silica (SiO_2), a molybdenum blue method (Bunting,1944) is used.

Reagents

1. Ammonium molybdate solution: Dissolve 6.0g of $(NH_4)6MO7O_{24}.4HO2$ in 1l of water.

2. Tartaric acid solution: Dissolve 16g of $H_2C_2H_4O_6$ in 1l of water.

3. Reducing solution: place 0.28g of sodium sulphite, 3,6g of sodium bisulphite, and 0.06g of lamino-2 napthol-4-sulphonic acid into a 1l bottle, add 1l of water, and stir to dissolve.

4. Acidified water: add 5 ml of 1+1 $H_2SO_4$to 1l water.

Procedure

1. By using autopipette transfer 8 ml of the solution A reagent blank solution, 8 ml of each standard solution, 8 ml of each sample solution to 150 ml beakers.

2. Using dispenser add 25 ml of acidified water to each beaker.

3. Add 25 of the molybdate reagent to each beaker. Allow to stand for 10 min.

4. Add 25 ml of the Tartaric acid solution to each beaker.

5. Add 25 ml of the reducing solution to each beaker. Allow to stand for at least 45 min.

6. Determine the percent of SiO_2 for each solution at 640 nm using the reagent blank as the reference by an UV-Visible spectrophotometer, Genesys 10.

Determination of alumina (Al_2O_3) by colorimetry

Alumina (Al_2O_3) is determined by measuring the absorption of light at 475 nm by solution in which aluminum has been converted to a calcium aluminum alizarin red-S complex (Parker and Goddard, 1950).

Reagents for Al_2O_3

1. Complexing solution: to 880 ml of water add 0.3g potassium ferricyanide $K_3Fe(CN)_6$; 40 ml of hydroxylamine hydrochloride (NH_2OHHCl) solution, 10 percent; and 80 ml of a $CaCl_2$ solution (Dissolve 14g $CaCl_2$ in 30 ml concentrated HCl and dilute to 1l).Prepare this solution the same day it is to be used.

2. Thioglycolic acid solution : dilute 3 ml of pure acid to 1l of water.

3. Buffer solution: dissolve 80g of sodium acetate NaC2H3O_2.3H2O, in 975 ml H_2O and add 24 ml glacial acetic acid.

4. Alizarin red-S stock solution, 0.05-percent: dissolve 0.5 g the pure dye in 1l of water and filter.

5. Alizarin red-S, 0.01-percent: dilute 200 ml of the stock solution to 1l with water.

Procedure

1. By using autopipette, transfer 15 ml each of the sample(solution A), reagent blank, the standard solution, and the sample solution to a series of 150 ml beakers.

2. Add the following reagents to each beaker, preferably with a dispenser.

Add 25 ml of the complexing solution.

Add 25 ml of the thioglycolic acid solution. Allow to stand for 5 min.

Add 25 ml of the buffer solution. Allow to stand 10 min.

Add 25 ml of the 0.01 percent alizarin red-s solution. Allow to stand for 45-75 min.

Determine the percent of Al_2O_3 at 475 nm for each solution using the blank solution as a reference with an UV-Visible spectrophotometer, Genesys 10.

Determination of total iron (Fe_2O_3) by colorimetry

The method for determining total iron (Fe_2O_3) is based on the orange color developed with orthophenanthroline after the iron is reduced with hydroxylamine hydrochloride and the solution is buffered with sodium citrate (Bandemer an Schaible,1944).

Reagents for Fe_2O_3

Hydroxylamine hydrochloride, 10 percent: Dissolve 100g NH_2OHHCl in 1l of water.

Orthophenanthroline solution, 0.1 percent.Dissolve 1g C12H8N_2.H2O in 1l of water.

Sodium citrate solution, 10 percent.Dissolve 100g Na3C6H5O_7.2H2O in 1l of water.

Standard iron solution:

Add 0.4910 g $FeSO_4(NH_4)2SO_4$.6H2O to a 500 ml volumetric flask. Add 16 ml of 1+1 H_2SO_4,10 ml of concentrated HNO_3, and about 300 ml of water. Stir until completely dissolved, then add water to the mark, and mix. The Fe_2O_3 concentration in this solution is 0.2 mg /ml, which is equivalent to that in a solution prepared from a sample containing 10 percent Fe_2O_3.

Procedure

1. With a autopipet, transfer two 5 ml portions of the standard iron solution and 5 ml of each sample solution B to a series of 100-ml volumetric flasks. Use an additional flask for the preparation of a reagent blank solution.

2. With a dispenser, 5 ml of the hydroxylamine hydrochloride solution to each flask and allow to stand for 10 min.

3. Add 20 ml of the Orthophenanthroline solution to each flask.

4. Add 10 ml of the sodium citrate to each flask and dilute to 100 ml with water.

5. After 1 hr. determine the percent of Fe_2O_3 at 555 nm for each solution by using the blank solution as the 100 percent reference, by an automatic UV-Visible spectrophotometer, Genesys 10 .

Determination of total CaO+MgO by titration

Determination of total CaO+MgO is done by titration with EDTA and Eriochrome black T as an indicator (Biederman and Schwarzenback, 1948; Pribil, 1953).

Reagents

Standard solution for CaO+MgO: Transfer 0.500g of Calcium Carbonate (Low in alkalies), AR.grade, to a 150-ml beaker, add about 25 ml of water and 5ml of HNO_3, cover with a watch glass, and bring to boil on a hot plate. Cool to room temperature, add 8ml of 1+1 H_2SO_4, dilute with water to 250 ml in a volumetric flask, and mix.

EDTA solution, 0.3 percent: Dissolve 12 g of disodium ethylenediamine tetraacetate in 4l of water.

Erichrome black T solution, 0.05 percent: Dissolve 50 mg of the dye in 100 ml of water. Do not store for more than 3 days.

Hydroxylamine hydrochloride solution, 10 percent: Dissolve 100 g NH_2OHHCl in 1l of water.

Buffer solution: Dissolve 66 g of NH_4Cl in 500 ml of water, add 500 ml of concentrated NH_4OH and mix.

Complexing solution: Dissolve 64 g of KCN in 600 ml of water, add 400 ml of triethanolamine, and mix.

Methyl red solution, 0.02 percent: Dissolve 200 g of the pure dye in 1l of water.

Magnesium sulfate solution: Dissolve 6 g of $MgSO_4$.7H2O in 1l of water.

Procedure

1.With autopipette, transfer two aliquots 25 ml of the standard solution for CaO+MgO, an aliquot of the magnesium sulfate solution, and aliquots of each B solution to a series of 400-ml beakers.

2. Add 5 ml of the hydroxylamine hydrochloride solution to each of the beakers and allow them to stand for 5 min.

3. Add about 200 ml of water to each beaker.

4. Add 5 ml of complexing solution to each beaker by using dispenser.

5. Add 25 ml of the buffer solution to each beaker.

6. Add 2 ml of the magnesium sulfate solution to each beaker, including the beaker already containing magnesium sulfate.

7. To one of the solutions containing the standard CaO+MgO, add 1 ml of the Erichrome black T solution and 1 ml of the methyl red solution.

8. Decant about 10 ml of the solution into small beaker and set aside for the moment.

9. Adjust the level of EDTA in the buret to zero, then add the EDTA rapidly to the solution in the 400-ml beaker in slight excess of that needed to get complete color change from red to blue.

10. Pour the solution which had been set aside back into the 400-ml beaker. The color of the solution should change back to red.

11. Titrate carefully until the red color of the solution disappears and the solution is dull grey-green in color. This is the end point.

12. Repeat steps 6-11 with each of the sample solution, the magnesium sulfate solution, and finally with the remaining standard CaO+MgO solution.

Calculations

1. Divide the volume for the titrate of the magnesium sulfate solution by 13.5 to abtain the volume of the EDTA equivalent to 2 ml of the magnesium solution.

2. Subtract the volume of EDTA equivalent to 2 ml of the magnesium solution from each titration volume to obtain the volume of EDTA required for the 25 ml of each solution.

3. The percent CaO in pure $CaCO_3$ is 56.0, there fore to calculate a factor:

$$\frac{56.0}{\text{Average of titration volumes for 25 ml of standards}} = \text{factor}$$

4. Calculate the percent total CaO+MgO, as CaO in each sample:
(Factor) × (titration volume for 25 ml of the sample solution)
= percent CaO+MgO calculated as CaO.

5. The result obtained for CaO+MgO as CaO is used in the following manner:
Percent MgO (by atomic absorption)×1.39 = CaO equivalent of MgO,
then,
CaO+MgO (from step 4)- CaO equilavent of MgO=percent CaO.

Determination of MgO by atomic absorption (AAS) Spectrophotometry.

Prepared reagents

1. $CaCO_3$ standard solution : same as standard used for determination % total CaO+MgO

2. Working standard solution:
   - 1% Mg solution
   - La_2O_3 solution

Procedure

1. Transfer 4 ml of standard solution and solution B of each sample, using an auto-pipet, to a series of 200-ml beakers.

2. add 16 ml of lanthanum solution to each beaker, preferable with a dispenser.

3. Add 40 ml of water to each beaker with a dispenser. These solutions are ready to be analyzed.

4. Run the standards to prepare a calibration curve and continue running the samples by AAS from Perkin Elmu, model AA300.

Determination of Phosphate (P_2O_5) by colorimetry

The phosphate content of a sample is determined by measuring the light absorbed at 420 nm by a solution containing the yellow molybdovanadophosphoric acid complex (Kitson and Mellon, 1944). The wavelength of 420 nm was chosen to provide adequate sensitivity and to minize the effect of iron. At this wavelength, each 4 percent total iron as Fe_2O_3 in the sample will still cause the value of P_2O_5 to be high by 0.01 percent unless a correction is made.

Reagents

Molybdovanadate solution: Dissolve 5.0g of sodium metavanadate, $NaVO_3$, in 400 ml of 1+1 HNO_3. Dissolve 75g of sodium molydate ($Na_2MoO_4.2H_2O$) in 400 ml of water. Mix the two solutions and dilute to 2 l.

Standard P_2O_5 solution: The USGS rock standards are used:

	SCo-1	STM-1
10 ml	0.076 % P_2O_5	0.064 %P_2O_5
5 ml	0.038 % P_2O_5	0.032 %P_2O_5
2 ml	0.015 % P_2O_5	0.012 %P_2O_5

Procedure

1. Transfer 25 ml of water to serve as a reagent blank, two 25 ml portions of the standard solution, and 25 ml of each B solution to a series of 100 ml beakers.

2. Add 25 ml of the molybdovanadate solution to each beaker preferably with a dispenser.

3. Allow the solution to stand for 5 min, then determine the percent of the P_2O_5 at 420 nm, using UV-visible spectrophotometer, model Gesys-10.

Appendix B

Limestone staining procedure.

Potassium ferricyanide and Alizarin Red S in 1.5% HCl

Reagents:

1. Etching solution : 1.5% HCl

2. Staining solution : mixture of soultion I : solution II = 3 : 2
   - Solution I : dissolve 0.2g. Alizarin Red S in 100 ml 1.5% HCl.
   - Solution II : dissolve 2g. potassium ferricyanide in 100 ml 1.5% HCl.

Procedure

1.Immerse sample in etching solution (dilute HCl at room temperature) for approximately 15 seconds.

NB: the success of the staining depends on the quality of the etch; cold solutions give poor results; weak etching gives a patchy stain; and over-etching produces a very dense stain.

2. Immerse sample in the staining solution for 30-35 seconds.

NB: the solutions must be freshly prepared for each staining session.

3. Wash stained sample gently in distilled water and dry in a stream of warm air.

Appendix C.

The procedure for determination limestone by loss on ignition is as follows:

1. Grind samples to a powder passing 105 mesh (0.105mm).

2. Weigh a 2 g sample in to a platinum crucible and dry in a laboratory oven for one hour at 105°C.

3. Record dry weight.

4. Place samples in a muffle furnace at 550°C for 25 minutes.

5. Remove, cool in a desiccators and weigh.

6. Calculate weight loss due to release of carbonaceous material and water.

7. Return samples to muffle furnace and increase temperature to 1100°C.

8. Leave samples for 25-30 minutes , remove, cool in a desiccator and weigh.

9. Calculate loss in weight between 550° and 1100°C = wt loss.

$$\%CaCO_3 \text{ content} = \frac{\text{Wt loss} \times 100}{2.000\text{g.}}$$

APPENDIX D

Phase analysis of the samples by X-ray Diffraction Powder Technique (XRD)

Procedure

1. The samples are grinded as power by using tungsten carbide disc mill, passing sieve No.325 mesh.

2. Pack each powdered sample in cavity of sample holder and flatten the surface by using glass slide.

3. Run each sample by the Bragg – Brentano X-ray Diffractometer; D8 Advance from Bruker from 2θ = 10 to 70 degree, step size = 0.04.

4. The diffractograms are identified using software package; search and match or " EVA " by comparing with database from International Centre for Diffraction Data (ICDD).

APPENDIX E

Brightness Test

The brigtness test is carried out by using colorimetter: Photo Volt model 577 from Seradyn, Inc, USA.

Method:

1, The samples are grinded by disc mill, and screened with sieve mesh No. 200

2, Dry the samples, fraction –200 mesh in an oven at 105 $\pm$ 3°C for 3 hours.

3, Set zero of the instrument by using an empty glass cup.

4, calibrate the instrument with the standard plates of brightness 64.1 and 71.0.

5, Measure the brightness of the samples by using the same cup as zero setting.

6, Read the brightness 3 times for each sample and calculate the average values.

APPENDIX F

End-uesd limestone standard

Table 5.1 Classification of limestone by purity (Cox et al.1977).

Category	Percentage ($CaCO_3$)	Percentage (CaO)
Very high purity	>98.5	>55.2
High purity	97.0-98.5	54.3-55.2
Medium purity	93.5-97.0	52.4-54.3
Low purity	85.0-93.5	47.6-52.4
Impure	<85.0	<47.6

Table 5.2 chemical properties of limestone for cement production (Lorenz,1991).

Chemical composition	Percentage(by weight)	Remark
Calcium carbonate ($CaCO_{3)}$	>42	For white cement:
Magnesium oxide (MgO)	<3	Fe_2O_3<0.01% and
Silica (SiO_2)	<15	Least MnO
Phosphorous pent oxide (P_2O_5)	<0.5	

Table 5.3 Chemical properties of limestone for sugar production (Harrison,1992).

Chemical composition	Percentage (by weight)
Calcium oxide (CaO)	>53.76
Magnesium oxide (MgO)	<4
Carbon dioxide (CO_2)	-
Silica dioxide(SiO_2)	<1
Alumina+Ferric oxide (Al_2O_3+Fe_2O_3)	<1.5

Table 5.6 Chemical properties of limestone for ceramic grade II (Lorenz,1991).

Chemical composition	Percentage (by weight)
Calcium oxide (CaO)	> 49.9
Magnesium oxide (MgO)	< 4
Silica (SiO_2)	<2
Alumina(Al_2O_3)+ Ferric oxide (Fe_2O_3)	<0.03
Sulphur trioxide (SO_3)	<0.1

Table 5.7 Chemical properties of limestone for agriculture (Lorenz,1991).

Chemical composition	Percentage (by weight)
Calcium oxide (CaO)	>47.6
Silica (SiO_2)	Not to high
Alumina (Al_2O_3)	< 1
Ferric oxide (Fe_2O_3)	< 1
Sodium and Potassium oxide(Na_2O+K_2O)	<0.05

Table 5.8 chemical properties of limestone for quicklime (Lorenz, 1991)

Chemical composition	Percentage(by weight)
Calcium oxide (CaO)	> 53.2
Magnesium oxide (MgO)	< 2
Alumina(Al_2O_3)+Ferric oxide (Fe_2O_3)	< 0.9

APPENDIX G

Some used Instruments

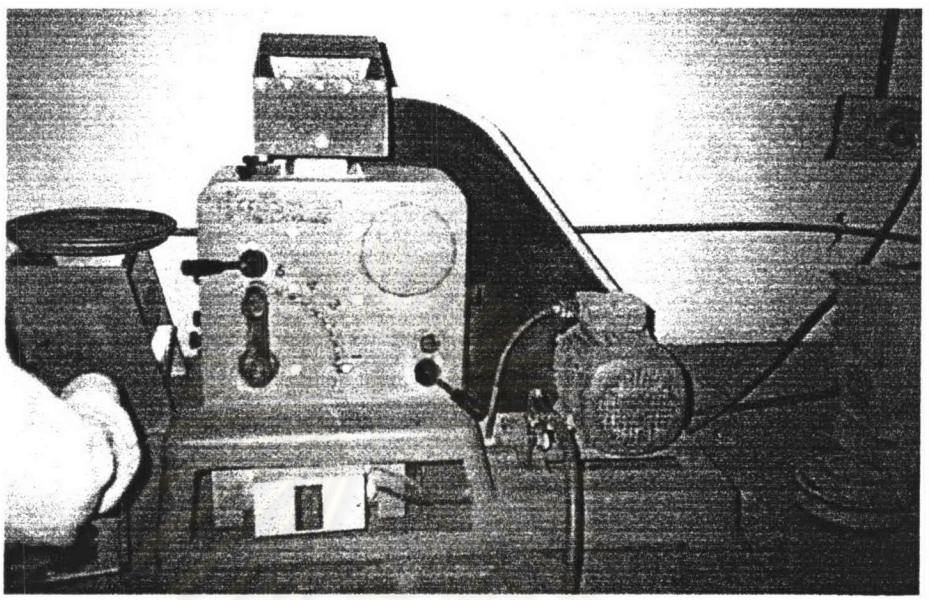

Fig. G1 Jaw Crusher, model I. FRITSCH

Fig. G2 Disc- mill. RETSCH

Fig. G3 Thermo - Spectronic, Genesys-10uv

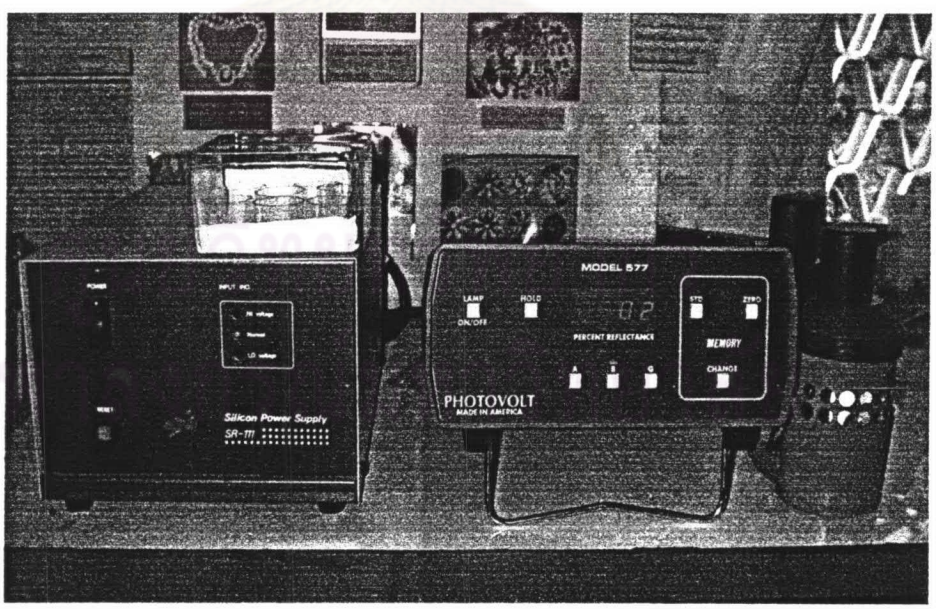

Fig. G4 Colorimetter: Photovolt, model 577

Fig. G5 Atomic Absorption Spectrometer-Perkin-Elmer AA-300

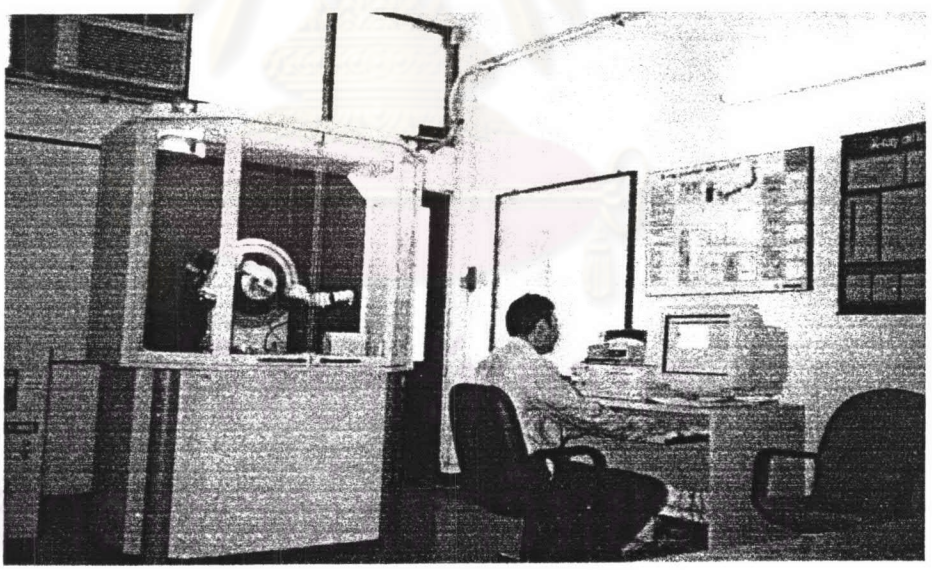

Fig. G6 X-ray Diffractometer-Bruker-D8-Advanced.

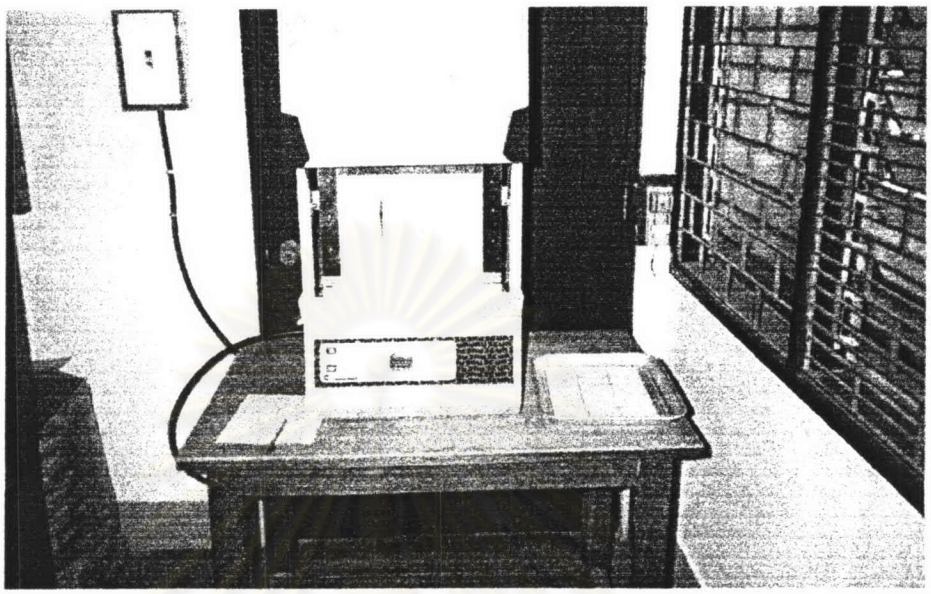

Fig. G7 Furnace Carbolite, RHF-140

Fig. G8 Titration.

Fig. G9 Polishing Machine.

Fig. G10 Cutting slabs for thin sections.

BIOGRAPHY

Mr. Keo Khamphavong was born on December 4, 1961 in Champasak province, southern Lao PDR. During 1977-1983, he studied the petroleum at Technical School in Baku city, Azerbaijan state, former Union of Soviet Socialist Republic (USSR), and received a Diploma Degree of Assistant Geophysicist in April, 1983. After graduation, he worked as a storekeeper for geological instruments in the Department of Geology and Mines (DGM), Vientiane, the Lao PDR until 1988.

From 1989 to 1994, he went to continue his study in the Ural State Academy of Mining and Geology, Ekaterinburg, Russia, and finally received the diploma Degree of Engineer-mining-geologist in June, 1994. Since August, 1994, he returned to work as a geologist in the Geological Division of Department of Geology and Mines, Vientiane, the Lao PDR.

From March, 2000, he has continued his study for Master Degree in the Department of Geology, Faculty of Science, Chulalongkorn University, Thailand. Under the financial support of the Department of Technical and Economic Cooperation (DTEC) of Thailand.