## NEW ZEALAND OCEANOGRAPHIC INSTITUTE



# TEXTURAL, CHEMICAL AND MINERALOGICAL ANALYSES OF MARINE SEDIMENTS

by Willem J.M. van der Linden

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## TEXTURAL, CHEMICAL AND MINERALOGICAL ANALYSES OF MARINE SEDIMENTS

bу

Willem J. M. van der Linden

#### INTRODUCTION

This manual is a compilation of the analytical methods in sedimentary petrography commonly used by the New Zealand Oceanographic Institute. The methods described are not very different from techniques used elsewhere. However, for practical purposes and because of specific requirements special instruments have been built and modifications made to well established analytical techniques.

The selection of a quantitative analytical method should be based on the sampling method. It is no use carrying out a detailed grain-size analysis when the equipment does not produce a representative sample. Samples of the bottom of the sea can be obtained by many devices. The best piece of sampling equipment is a corer which brings an undisturbed sample to the surface. Using this is not always practicable because of factors such as the nature of the bottom, texture of the sediment and weather conditions. The next best is a grab. A conical shaped canvas bag draped over the grab prevents material being washed out while being hauled to the surface. Dredged bottom samples are usually of limited value but a more or less representative sample is obtained provided there are no rapid changes in sediment properties over the course of the dredge haul. A canvas bag mounted inside a dredge will greatly improve the quality of the sample obtained.

In most cases dessication and decay of organisms in the sample have a marked effect on the grain size especially of the finer particles. Clay minerals may form aggregates which are not always destroyed by subsequent wetting and organic decay may reduce grain size of soluble minerals. As on the spot analyses are mostly impossible the manner in which samples are stored is very important. Air-tight packing and the use of preservatives and dispersants will greatly increase the value of a sample.

It is doubtful whether results of grain size analyses by different techniques are comparable. Certainly there is no simple conversion method and it is therefore advisable to use a standardized technique. It will, however, be clear that the method applied will depend both on sampling equipment and on the detail and accuracy required.

## GRAIN SIZE ANALYSIS (sieve-pipette method)

## 1. REQUISITES

- A. Instruments and Apparatus
  - a. 6 x beakers, 600 ml + 6 x watch glasses, diam. 5"
  - b. 1 x precision balance (Mettler K5)
  - c. washing and filtering equipment -
    - 1 x vacuum pump (Speedivac)
    - 2 x Buchner funnels for filter paper, diam. 12.5 cm
    - 2 x filtering flasks, 1000 ml
    - filter paper (Schleicher und Schull 55 no. 595 diam. 12.5 cm) rubber tubing
    - 2 x tube clamps
  - d. coarse-fine separation unit -
    - 1 x retort stand
    - 1 x clamp and bosshead
    - 1 x glass funnel, diam. 6"
    - 1 x sieve, diam. 4", mesh 240 (64)
    - 1 x 4" length of rubber tubing to fit over funnel stem
    - 1 x tube clamp
    - 1 x brush
    - 1 x pipette, 50 ml
  - e. thermostatically controlled hotplates or water baths
  - f. dry-sieving unit -
    - 1 sieve shaker (Rotap)

1 set of st	ieves, diam.	8"	
$\max_{no_\bullet}$	μ	mesh no.	/u
1	16,000	30	500
$\bar{2}$	8,000	44	353
4	4,000	60	250
8	2,000	85	178
12	<b>1,</b> 400	120	125
16	1,000	170	89
22	700	240	64

g. pipetting unit -

 $1 \times \text{pipette}$  stand (enabling horizontal and vertical pipette movement)

1 x vacuum suction pipette, 20 ml

1 x vacuum pump (Speedivac) rubber tubing

7 x spoutless measuring cylinders, 1000 ml

7 x rubber bungs

1 x thermometer (allowing readings between  $10^{\circ}$  and  $30^{\circ}$ C with an accuracy of 0.5°C)

1 x stopwatch

1 x three-way stopcock

- h. 31 x nickel evaporation dishes, 50 ml
- i. 1 x desiccator
- j. 1 x drying oven (temp. up to 120°C)
- k. 1 x analytical balance (Mettler B5) or1 x multipurpose balance (Mettler H3)
- 1. squeeze bottles, spatulas, forceps, marking ink

#### B. Chemicals

- a. 10% peroxide ( $\rm H_2O_2$ )

  To make up one litre of 10%  $\rm H_2O_2$  solution take 250 ml of the standard 130 Vol. 0.  $\rm H_2O_2$  solution ( $^{\pm}$  39%) and add 750 ml of demineralized water.
- b. 1 N hydrochloric acid (HCl)

  To make up one litre of 1N HCl take 100 ml of the standard 35.4% HCl solution and add 900 ml of demineralized water.
- c. 6N hydrochloric acid (HCl)

  To make up one litre of 6N HCl take 600 ml of the standard 35.4% HCl solution and add 400 ml of demineralized water.
- d. \*20% sodium dithionite (sodium hydrosulphite)  $\rm Na_2S_2O_4$  To make up a 20%  $\rm Na_2S_2O_4$  solution add 10 grams of  $\rm Na_2S_2O_4$  to 50 ml of cold water.
  - $N_{\bullet}B_{\bullet}$  The solution should be prepared immediately before use.
- \* Solutions (d) and (e) only to be used if sample contains a high amount of iron.

- e. \*1% (10 grams of NaCl/litre)
- f. peptizer (0.1 mole Na-oxalate + 0.02 mole Na<sub>2</sub>CO<sub>3</sub> per litre).

  To make one litre of peptizer dissolve 13.4 grams of dry Na-oxalate and 2.12 grams of dry Na<sub>2</sub>CO<sub>3</sub> in 1 litre demineralized water.

#### 2. PROCEDURE

For practical purposes it is advisable to work with six samples at a time.

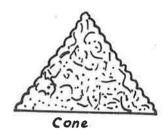
#### A. Initial Amount

Homogenise the sample by carefully mixing with a spatula. Depending on overall grain size distribution take amounts of 50, 25 or 10 grams to ensure an adequate concentration of the suspension in the pipette cylinders (5-20 grams/litre).

- 50 grams if the material is predominantly coarse sand.
- 25 grams if the material is predominantly sand or sandy mud.
- 10 grams if the material is predominantly muddy.

For coarser grained material (average sizes 2000 µ) another analytical method should be used. For instance up to and including the pebble class samples can be separated into size classes by using sieves with apertures of 4,000, 8,000 and 16,000 µ. For even larger size classes it is necessary to measure the intermediate diameter (b-axis) of a certain number (usually 50 to 100) of pebbles, cobbles or boulders to provide estimates of size comparable to sieve separated size fractions. Errors caused by such comparisons are normally introduced because of differences in petrologic composition. (Sieve separation provides weight percentages in the various size classes, measurement of b-axes provides approximate volume percentages.)

It is often necessary to sample from bulk. To ensure that the ultimate sample to be analysed is truly representative of the bulk and that repeat tests can be made if necessary divide the sample either with a sample splitter or by the quartering method. The latter is illustrated step by step in Fig. 1. The original bulk quantity is mixed carefully and shaped into a cone on a flat surface. Flatten the cone to a circular pile of uniform thickness. Divide it into four quarters with two right angle cuts separating the material slightly with a lateral movement. Remove two opposite quarters. Mix the remaining quarters completely, form a cone, and repeat the procedure until the sample is the desired size.





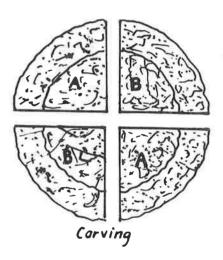




Fig. 1 Sampling from bulk. The quartering method. (after Milner, 1962)

#### B. Peroxide Treatment

To remove organic material the samples are treated with  ${\rm H_2O_2}$ . Put the initial amount (50, 25 or 10 grams) in a numbered 600 ml beaker and add 50 cc of 10%  ${\rm H_2O_2}$  solution. Cover the beaker with a watch glass. Add small amounts of 10%  ${\rm H_2O_2}$  as reaction ceases and place on a water-bath. If there is almost no further reaction remove the watch glass to allow some evaporation.

At this stage it is advisable to leave overnight on the water-bath with the watchglass cover.

#### C. Acid Treatment

To remove carbonate cement and iron oxide films the samples are treated with HCl.

After the peroxide treatment cool the sample, clean the watchglass over the beaker with water (squeeze bottle) and add 50 cc of 6N HCl gradually, to start with, to avoid too violent a reaction on unknown sample material. Shake the sample every so often. If there is no further reaction add more 6N HCl. Repeat until all reaction has ceased. Put the sample on a water-bath for 30 minutes.

- N.B. 1. This treatment attacks iron and affects heavy minerals such as apatite, olivine and monazite. Extended HCl treatment should be avoided. If a sample has a high iron content it is advisable to remove excess iron by treatment with sodiumdithionite (E).
  - 2. Where information is required on the grain size distribution of sediment, including clastic or non-clastic carbonates, HCl cannot be used for disintegrating purposes. Instead treat the sample with a NaOH solution or a detergent.

#### D. Removal of Acid, Fig. 2

Place a Buchner funnel on a filtering flask and connect the flask with rubber tubing to a vacuum pump. Place a filter paper in the funnel and wet this thoroughly after applying vacuum. Once the filter sets without leaks in the funnel carefully place the sample on the filter.

If no dithionite treatment (E) is needed (the usual case) proceed from here to the washing procedure (F). After filtering the filter and funnel should be cleaned and the sample put back in the 600 ml beaker.

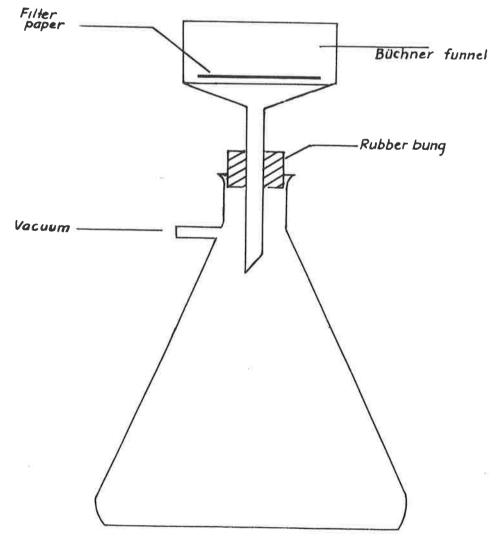


Fig. 2 Washing and filtering unit.

## E. Sodium Dithionite Treatment

This method ensures iron is removed without attacking other minerals.

The sample is placed in the beaker with only a small quantity of water on top. Loosen the sample with a spatula and add 50 cc 20% Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>. Place beaker for 20-30 minutes on the waterbath and shake regularly. The sample turns grey. Let it cool and filter as in (D) with a Buchner funnel and filtering flask.

## F. Washing (Set up as in D)

- 1. If the sample was treated with Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> wash with a 1% NaCl solution and after suction has been applied repeat a few times especially for very fine sediments. To prevent sulphur precipitation, which would be a nuisance if the filter bursts, empty the filter flask before continuing.
- 2. Wash the sample through the Buchner funnel with demineralized water 5 times and return to the 600 ml beaker. Clean funnel and filter thoroughly into the beaker.

## G. Coarse-fine Separation. Fig. 3

Attach a short length of rubber tubing fitted with a tube clamp to the stem of a 6" funnel. Clamp the funnel on a retort stand. 64  $\mu$  sieve (diam. 4") in the funnel and a 1000 ml pipette cylinder underneath. Put the sample on the sieve and separate the material finer than 64  $\mu$  using a brush and demineralized water. It is advisable to use sufficient water to cover the sieve mesh. Empty the funnel into the cylinder and repeat the procedure until all particles smaller than 64  $\mu$  have passed through the sieve. The fraction coarser than 64  $\mu$  is put on a nickel evaporation dish and left to dry on a water bath and later in a drying oven for 10 minutes at 120°C. Clean the funnel into cylinder and add 50 cc of peptizer to the fine suspension. Fill the cylinder up to the 1000 ml mark with demineralized water. Close the pipette cylinder with a rubber bung, shake the suspension and leave standing overnight on the pipette stand to ensure complete dispersion. Add one cylinder of demineralized water and 50 cc of peptizer, in which a thermometer is placed, to a series of six cylinders containing suspension.

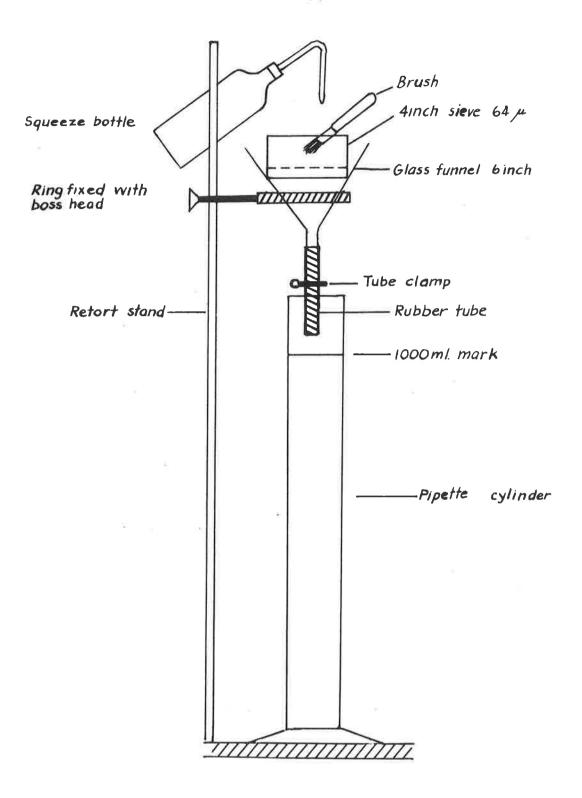


Fig. 3 Coarse-fine separation unit.

#### H. Sieving

After drying the fraction coarser than 64  $\mu$  is brought on the sieve series and shaken for 15 minutes. The sieve fractions are put into nickel dishes and subsequently weighed on an analytical or multi-purpose balance to the nearest 0.0001 gram.

All material that passes the 64  $\mu$  sieve is also weighed and, after being moistened with alcohol is added to the pipette cylinder. Enter all results on the computation list (pp.9, 10).

## GRAIN SIZE ANALYSIS (Computation List)

Geology Lab. N. Z. O. I. Wellington

Sample No.			
Analysed by:	.1. / 3		
Bulk sample: wet/mois		December 11 O /HOL/	
Initial amount	gm	Treated with ${\rm H_{2}O_{2}/HCl/}$	1Va2S2C4
Beaker No:		Dinatting town	°C
Pipette Cylinder No:		Pipetting temp.:	<u> </u>
1st sample	gm	2nd sample	gm
Dish No.:	gm -	Dish No.:	gm
Fraction < 64 $\mu$	gm x 50 =	gm	
Fraction < 32 µ	gm	32 <b>-</b> 64 µ	gm
Dish No.:	<u>gm</u> -	,	
	$gm \times 50 =$	gm	
Fraction < 16 \mu	gm	ىر 16 <b>-</b> 32 بىر	gm
Dish No.	gm -	,	
	gm x 50	gm	
Fraction < 8 µ	gm	8 <b>-1</b> 6 µ	gm
Dish No.:	gm	,	
	gm x 50 =	gm	
Fraction < 4 µ	gm	4 <b>-</b> 8 $\mu$	gm
Dish No.:	gm -	,	
	gm x 50 =	gm	
Fraction < 2 µ	gm	ير 4–2	gm
Dish No.:	gm -	,	
	gm x 50 =	gm	
Correc	tion:		gm
Peptizer:		gm < 2 /1	<b>9</b>
Dish No.:		gm <b>-</b>	
Correction:	·	gm x 50 =	gm
* The correction sho	ould always be		·

the finest fraction estimated. (Normally 8  $\mu$ ).

			% Cum.%	
( > 16,000 µ) (8,000 - 16,000 µ) (4,000 - 8,000 µ) (2,057 - 4,000 µ) 1,400 - 2,057 µ Dish No.:	or > 2057 µ gm gm -	gm gm		
1,000 - 1,400 µ Dish No.:	gm -	gm		
700 - 1,000 µ Dish No.:	gm -	gm		
500 - 700 µ Dish No.:	gm -	gm		
353 - 500 µ Dish No.:	gm <u>qm</u> -	gm		
250 - 353 µ Dish No.:	gm gm -	gm		
178 - 250 µ Dish No.:	gm gm -	gm		
125 <u>-</u> 178 µ Dish No.:	gm -	gm		
89 - 125 µ	gm -	gm		
64 - 89 µ Dish No.:	gm qm -	gm		
$32 - 64 \mu$ $16 - 32 \mu$ $8 - 16 \mu$ $(4 - 8 \mu)$ or $< 8 \mu$ $(2 - 4 \mu)$ (or $< 4 \mu$ )	l 1 <b>)</b>	gm gm gm gm gm qm +		

TOTAL

gm

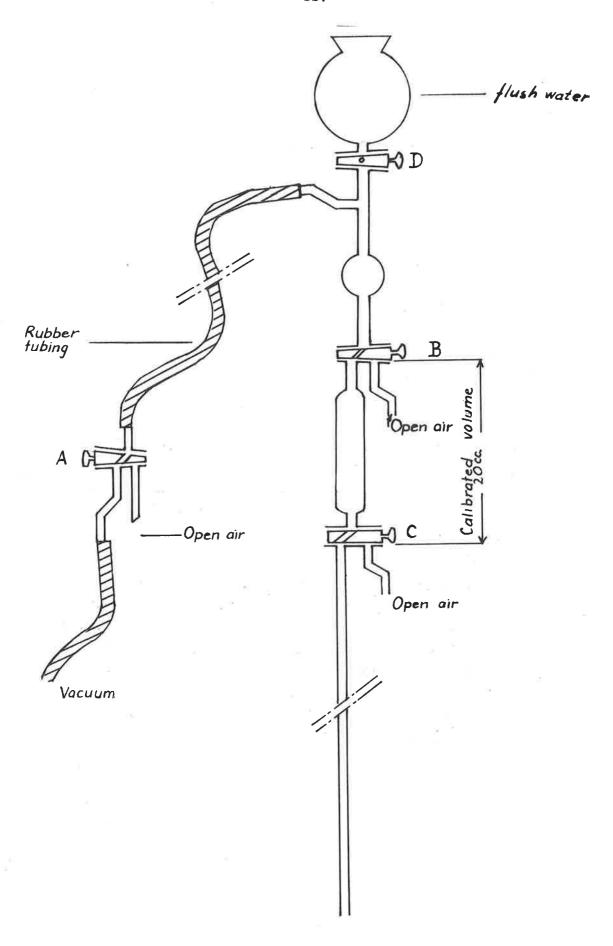


Fig. 4 Pipette (construction by Chemistry Division, DSIR) With taps C and D closed and taps A and B in the position indicated lower the pipette in the cylinder. Open C as indicated and let the suspension rise in the pipette until well over B. Close C and, by turning A, connect the pipette to the open air. Remove excess sample by opening B to open air. Clean the upper part of the pipette by quickly opening and closing D. Now turn B back to its original position. Collect the sample at C by turning the tap and flush the pipette again (into evaporation dish) by quickly opening and closing D.

#### I. Pipetting. Figs 4, 5.

(The procedure as described applies to the pipette and pipette stand used by  $N_{\bullet}$   $Z_{\bullet}$   $O_{\bullet}$   $I_{\bullet}$ )

Place a rubber bung on the pipette cylinder and shake the sample to loosen all material from the bottom and to obtain a homogenous suspension. Immediately after putting the cylinder down take a sample (20 cc) at a depth of 20 cm.

Empty the pipette sample into a nickel evaporation dish and place the dish on a water bath. For a control repeat the procedure of taking a sample at a depth of 20 cm immediately after renewed shaking of the cylinder. This provides two samples of the total fraction smaller than 64  $\mu$ . After all moisture has evaporated place the samples for 10-15 minutes in a drying oven at 120°C. The weight of one should differ from the other by no more than 2%

Shake the cylinder and place it on the pipette stand. After 4 minutes take a sample at the depth indicated in Table 1. (The temperature is read off the thermometer in the water-peptizer cylinder). This provides the total fraction smaller than 32  $\mu$  per 20 cc. Without further shaking samples are taken, in the same way, after 16 minutes and 30 minutes at the appropriate depths to obtain the fractions smaller than 16  $\mu$  and smaller than 8  $\mu$  respectively.

For a normal analysis it is not necessary to subdivide the < 8  $\mu$  traction as the silt-clay ratios in a given area are fairly constant for the tail end of a grain size distribution. Should this information be required, however, proceed in the same way taking further samples according to the time-temperature-depth table. The various fractions after evaporation on the water bath and subsequent drying in an oven for 10-15 minutes at 120°C are weighed on the analytical balance to the nearest 0.0001 gram.

TABLE 1
Pipetting depth in cm.

					M		
Γ	Cemp C	Fraction < 32 \mu	Fraction <16 µ	Fraction < 8 $\mu$	Fraction < 4 \mu	Fraction < 2 \mu	
		4 min.	16 min.	30 min.	60 min.	<b>1</b> 20 min.	
	10	16.9		7.9			
	10.5	17.1	i	8.0			
	11	17.4		8.1			
	<b>11.</b> 5 ,	<b>17.</b> 6		8.2			
·	12	<b>1</b> 7.8		8.4			
	12.5	18.1		8.5			
	13	18.3		8.6			
	13.5	18.6		8.7			
	14	18.8		8.8			
	<b>14.</b> 5	19.1	8	8.9			-
	15	19.3		9.0	4.4	2.2	
	<b>1</b> 5.5	19.6		9.2	4.5	2. 2	
	16	19.8		9.3	4.5	2.3	
	<b>16.</b> 5	20.1		9.4	4.6	2.3	
	17	20.3		9.5	4.6	2.3	
	17.5	20.6		9.6	4.7	2.4	
	<b>1</b> 8	20.8		9.8	4.8	2.4	
	<b>1</b> 8.5	21.1		9.9	4.8	2.4	
	19	21.4		10.0	4.9	2.4	
	19.5	2 <b>1.</b> 6		10.1	4.9	2.5	
	20	21.9		10.3	5.0	2.5	
	20.5	22.2		10.4			
	21	22.5		<b>10.</b> 5			
	21.5	22.7		<b>10.</b> 7			
	22	23.0		<b>10.</b> 8			
	22.5	23.3		10.9			
	23	23.6		11.0	ä		
	23.5	23.8		<b>11.</b> 2			
	24	24.1		11.3			
	24.5	24.4	:	11.4			

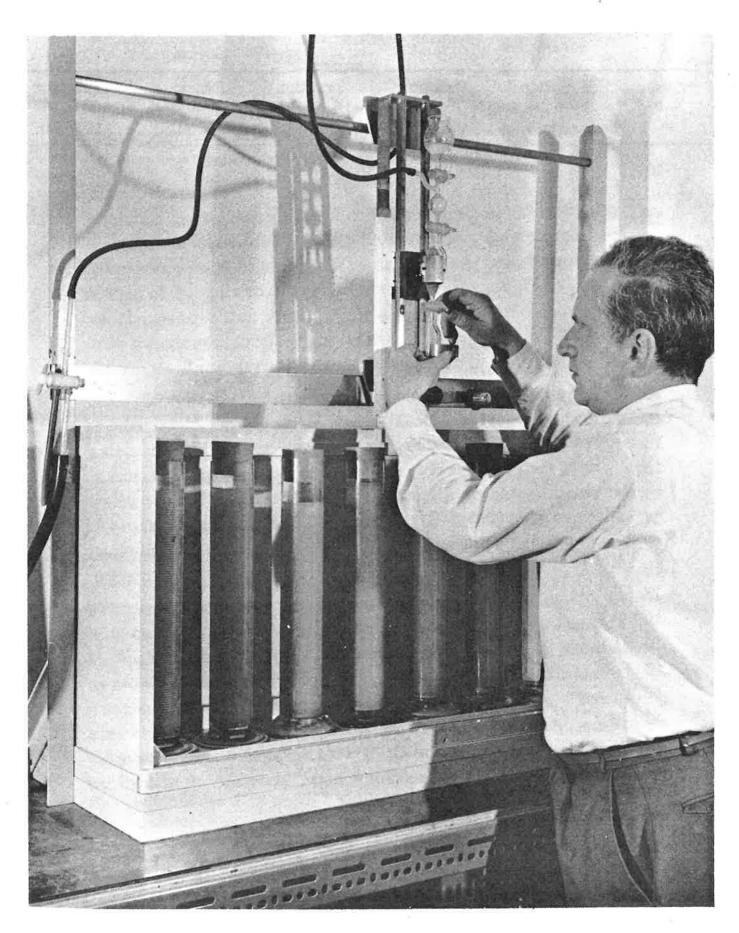


Fig. 5 Pipette stand with 6 cylinders (construction by Physics and Engineering Lab., DSIR)

### J. Calculation of Results

The weights of the pipette fractions are entered on the computation list (pp. 9) and the total amounts per litre calculated (multiplication by 50). Subtracting the total amounts finer than a given size gives the distribution over the various size fractions. From the fraction smaller than 8  $\mu$  (4  $\mu$  or 2  $\mu$ ) the total amount dry weight of peptizer is subtracted. This amount, indicated on the computation list as correction, is 0.776 grams. Every time new peptizer solution is prepared a test sample should be taken from the solution in the waterpeptizer cylinder (G) to verify that the correction factor is indeed 0.776 grams. After evaporation and drying the sample weight should be 0.015-0.016 grams.

The calculated results of the size classes smaller than 64  $\mu$  are added to the column presenting the results of the sieve analysis and the total of all fractions estimated. With a slide rule the percentages and cumulated percentages are now calculated to the nearest 0.0001 gram.

TABLE 2

Conversion of mm to Ø diameters

Tolerance less than 1% unless otherwise indicated.

	_				
mm	Ø		mm	Ø	
16,000	<b>-</b> 4	ń	0 <b>, 1</b> 78	2 <b>,</b> 5	1
8,000	<b>-</b> 3	interval 1 Ø	0,125	3,0	interval 0.5 Ø
4,000	- 2	11110211112	0,089	3 <b>,</b> 5	
2,000	- 1	4	0,064	4,0 ( <del>*</del>	1% {
1,400	- 0,5 ( <del>-</del>	3%)	0,032	5,0	
1,000	0,0		0,0 <b>1</b> 6	6,0	interval
0,700	0,5 <b>(</b> ±	3% interval 0,5	ø 0,008	7,0	1%
0,500	1,0	1	0,004	8,0	1
•	•	4	0,002	9,0	, i
0,353	<b>1,</b> 5		•, ••-	,	
0,250	2,0	,			

### K. Evaluation of Data

To avoid time consuming calculations of logarithmic values the  $\emptyset$  (phi) scale is introduced:  $\emptyset = -\log_2 \xi$ 

in which  $\xi$  (zeta) is the size in mm.

Table 2 gives the conversion of mm to Ø units. Cumulative percentages are now plotted against phi diameter on arithmetic probability paper (Gormack Graph Paper, Christchurch, No.5G) and a smooth line drawn through the points. From the curve the following percentages are now read to the nearest 0.1 Ø: 5%, 16%, 25%, 50%, 75%, 84% and 95%. This permits calculations of the following statistical parameters (after Folk and Ward, 1957).

Calculation and Evaluation of results by computer.

A computer programme "Pebbles" was prepared for the calculation of size parameters. It is written in Algol, a language linking classical mathematical notations to the binary notation with which the Elliott 503 computer works.

The input to the computer, punched on eight channel paper tape, is in weights per size class. The computer prints out size ranges with their respective percentages and cumulative percentages and will then interpolate linearly between successive points. From this the distribution parameters are calculated and printed. They are followed by a graph of cumulative percentages against size in  $\emptyset$  units. A modal analysis of the size frequency distribution can be incorporated in the programme.

#### REFERENCES

- Milner, H.B., 1962 Sedimentary petrography, Vol. 1. Methods in sedimentary petrography. 4th ed. Allen and Unwin, London, 643 pp.
- Muller, G. M. 1964 Methoden der Sedimentuntersuchung. Schweizerbart'sche Verlagsbuchhandlung, Stuttgart, 303 pp.
- Geologisch Instituut der Ryks Universiteit Utrecht: Voorschrift voor Korrelgrootte-analyse. Unpubl.
- Folk, R. L.; Ward, W.C. 1957: Brazos River Bar: A study in the Significance of Grain Size Parameters. J. sedim. Petrol. 27(1) 3-26.

#### CARBONATE ANALYSIS

#### (Acidemetry)

#### 1. REQUISITES

- A. Instruments and Apparatus
  - a. 1 x agate mortar and pestle
  - b. 1 x sieve mesh 120 (125  $\mu$ ), diam. 4"
  - c. 1 x sieve mesh 30 (500  $\mu$ ), diam. 4"
  - d. 6 x beakers, 100 ml
  - e. 1 x analytical balance (Mettler B5)
  - f. thermostatically controlled hot plates
  - g. 1 x 50 cc burette, 0.1 cc divisions
  - h. 1 x pipette, 20 ml
  - i. 1 x Erlenmeyer, 250 ml
  - j. 1 x funnel, diam. 3"
  - k. filterpaper, diam. 5"

#### B. Chemicals

- a. 1N hydrochloric acid (HCl)

  To make up one litre of 1N HCl take 100 ml of the standard 35, 4% HCl and add 900 ml of demineralized water.
- b. 1N sodium hydroxide (NaOH)

  To obtain one litre of 1N NaOH dissolve 40 grams of dry
  NaOH pellets in demineralized water to make a litre
  solution.
- c. methyl-orange
- d. 100% CaCO<sub>3</sub>
- $\underline{\text{N.B.}}$  Keep both NaOH pellets and 100% CaCO $_3$  stored in a dessicator.

#### 2. PROCEDURE

It is advisable to work with six samples at a time. Crush the sample in a mortar to eliminate the effect of granular variation. By sieving through sieves mesh 30 and 120 the fraction 125-500  $\mu$  is obtained. From this take one gram (analytical balance) and put it in a 100 ml beaker. Add 20 cc (pipette) 1N HCl and 20 to 25 cc of demineralized water. Shake the beaker and put it on a hotplate (in fume cupboard) for 10 minutes at  $60^{\circ}$ - $70^{\circ}$ C.

After cooling filter the solution into a 250 ml Erlenmeyer and add a drop of methyl orange as indicator. The amount of HCl used is now estimated by titration with IN NaOH solution.

As the volume of NaOH required to neutralise the superfluous HCl is directly proportional to the carbonate percentage of the sample a simple nomogram can be used to estimate the carbonate concentrations. The nomogram (Fig. 6) is based on the analyses of pure (100%) calcium carbonate and of a blank. These analyses provide two endpoints on the graph which are joined by a straight line. The scale of the graph should be selected so that the volume of NaOH can be entered to the nearest 0.05 ml and the CaCO<sub>3</sub> percentages read to the nearest 1% (approx. 1 cm  $\equiv$  1 ml; 1 cm  $\equiv$  10%). An extra advantage of the nomogram is that the normality of the reagents is not too critical. Naturally a new nomogram must be constructed every time new reagents (HCl or NaOH) are used.

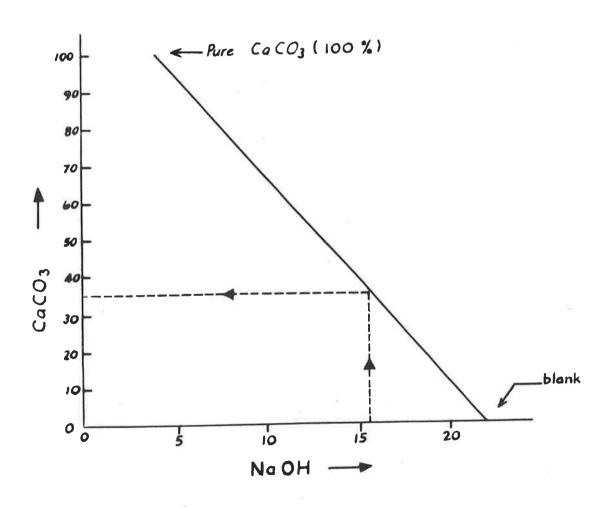


Fig. 6 Nomogram for the determination of carbonate percentages.

#### REFERENCE

Dulemba, J. L. 1963: Methodes d'Analyse des carbonates dans les Sediments. <u>Révue Géomorph. dyn. 4-6</u>: 62-71

#### HEAVY MINERAL ANALYSIS

### 1. REQUISITES

- A. Instruments and Apparatus
  - a. 1 x precision balance (Mettler K4)
  - b. 6 x beakers, 600 ml + 6 x watch glasses, diam. 5"
  - c. 1 x sieve mesh no. 30 (500 microns), diam. 4"
  - d. 1 x sieve mesh no. 240 (64 microns), diam. 4"
  - e. 1 x porcelain wash basin, diam. 8"
  - f. 1 x wooden pestle
  - g. thermostatically controlled hot plates or water bath
  - h. 6 x nickel evaporation dishes, 50 ml
  - i. 1 x drying oven (temp. up to 120°C)
  - j. 1 x dessicator
  - k. separation unit -

1 x funnel-battery or retort stand with 2 x clamps and bossheads

- 6 x separation funnels, 50 cc (Pyrex B 19/28)
- 6 x glass funnels, diam. 3"
- 12 x coloured glass bottles, 500 cc
- 6 x porcelain dishes, diam. 2"

filter paper, diam. 4"

- $1 \times \text{hydrometer}$  (2.6-3.0) or  $1 \times \text{pycnometer}$ , 50 cc
- 1. FRANTZ Isodynamic Magnetic Separator
- m. mounting set-up -
  - 1 x thermostatically controlled hot plate
  - 1 x thermometer  $(0^{\circ}-200^{\circ}C)$
  - 1 x copper thermometer holder object glasses + cover glasses

match sticks

- n. squeeze bottles, spatulas, stirring rods, marking ink, diamond pencil.
- o. 1 x 250 cc separation funnel

#### B. Chemicals

- a. 10% peroxide  $(H_2O_2)$ To make up one litre of 10%  $H_2O_2$  add 250 ml of the standard 130 Vol. 0.  $H_2O_2$  ( $^{\pm}$  39%) to 750 ml of demineralized water.
- b. concentrated hydrochloric acid (HC1)
- c. bromoform (sp. gr. 2.9)
- d. 99% alcohol (methylated spirits)
- e. canada balsam
- f. xylol (xylene)

#### 2. PROCEDURE

It is advisable to work with six samples at a time.

#### A. Preparation

Homogenise the sample by carefully mixing with a spatula. Put approximately 50 grams of sample (100 grams if predominantly silt and clay) in a 600 ml beaker and add 50 cc of  $10\%\,\mathrm{H_2O_2}$  solution to remove organic material. Cover the beaker with a watchglass and leave standing overnight in a fume cupboard.

Next add water and stir thoroughly. Remove all material finer than 64  $\mu$  and coarser than 500  $\mu$  by wet sieving through sieves mesh nos 30 and 240. To remove excessive light fraction wash the 64-500  $\mu$  fraction in a porcelain wash basin with tap water at the same time breaking down aggregates with a wooden pestle. Leave the suspension to settle for 30 seconds and decant. Repeat the procedure until the water stays clear.

Return the sample to the 600 ml beaker and add 50 cc of concentrated HCl. Put this on the waterbath in the fume cupboard for 20 minutes.

After adding cold water to the suspension to prevent the escape of fumes outside the cupboard wash the sample repeatedly in the porcelain wash basin as before.

N.B. The treatment with concentrated HCl will attack and dissolve the following heavy minerals: apatite, siderite, olivine, monazite and glauconite.

Next put the sample in nickel evaporation dish, dry it on the water bath or hotplate (temp. lower than 100°C) and later on stove at 120°C for 1 hour. Store the sample in a dessicator.

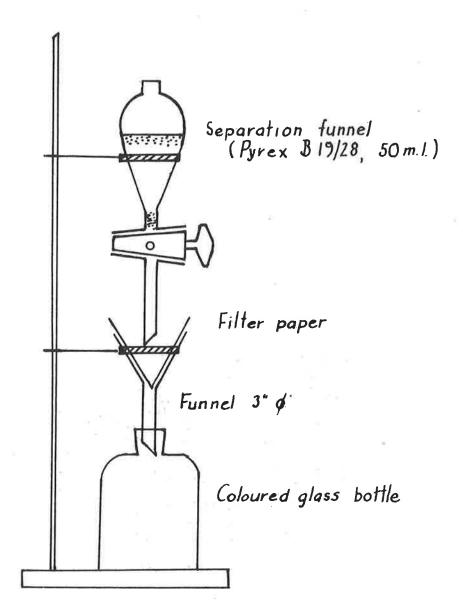


Fig. 7 Heavy mineral separation unit.

## B. Separation of the Heavy Fraction with Bromoform (Fig. 7)

Care should be taken with the handling and use of bromoform as its vapours are toxic if breathed in quantity. The use of bromoform outside a fume cupboard should be restricted to a minimum. Bromoform must be kept in the dark and separation of minerals with bromoform should be carried out in subdued light to avoid disintegration of the fluid and consequent decrease in density. As bromoform is also expensive every effort should be taken to avoid spilling.

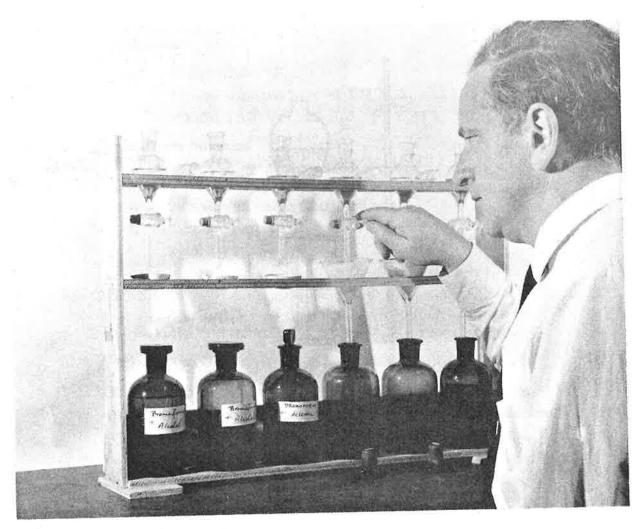


Fig. 8 Funnel battery for the separation of heavy minerals.

The use of a funnel battery enables a number of separations to be carried out at the same time (Fig. 8). First of all check the density of the bromoform (2.89) directly with a hydrometer (calibrated for densities between 2.6 and 3.0) or indirectly by using a pycnometer (50 cc). Three-quarters fill the separation funnel with bromoform. Add a spoonful of thoroughly mixed sample and stir carefully with a clean glass rod. Take care the fluid is not disturbed in the lower narrow part of the funnel. If the amount of heavy mineral that settles down is not sufficient add another spoonful of sample and stir again carefully.

By opening the tap slightly collect the heavies in a porcelain dish. Decant excessive bromoform through a filter back into the bromoform bottle. Empty the separation funnel by opening the tap also through a filter into the bromoform bottle and leave the tap open for 15 minutes.

To clean the separation funnel rinse it with wash fluid, a mixture of bromoform and 95% alcohol, and empty the contents through a filter into a wash bottle.

Clean the sample with 95% alcohol and decant super natant through a filter into the wash bottle. Repeat this last procedure three times.

Dry the heavy mineral sample in a stove at 120°C. Wash the light fraction in the same way as the heavy fraction, dry and store with sample no. in sample tube.

#### C. Magnetic Separation

This method separates the heavy mineral association in six groups according to their magnetic susceptibilities. This separation makes later microscopic identification much easier especially for inexperienced staff. The procedure described is the one for the FRANTZ isodynamic separator (Fig. 9).

First separate the ferro-magnetic minerals with a hand magnet. Minerals with lower susceptibilities are fed in the hopper and separation is carried out in successive steps with side inclination of the magnet and field strength set as indicated in Table 3. The inclination of the magnet in the direction of the chute (forward slope) is set at 30° for all steps. After each step the grains with higher susceptibilities are stored ready for mounting and the grains with lower susceptibilities are put through the separator for the next step.

## D. Mounting of Specimens

Set the hotplate for a temperature of 125°C some time prior to mounting. N.B. This temperature is critical. If the temperature is too low the balsam will not set, if too high the balsam becomes brittle and turns yellow-brown. Put an object glass on the plate and put some canada balsam on the slide with a glass rod. Leave for a few minutes to permit evaporation of volatiles. Mix the dry heavy mineral sample carefully and sprinkle an adequate amount of sample on the balsam. Spread the grains evenly over the slide and ensure that no air bubbles develop. With the help of two match sticks mount a cover glass in the following way:-

TABLE 3

Heavy minerals arranged in groups based on mass magnetic susceptibility

(after Hess, 1959)

Side Slo Magnetic at 0.4 A Imenite Farnet	Magnetic at 0.8 A hornblende hypersthene augite	Magnetic at 1.2 A diopside tremolite	Magnetic at 1.2 A titanite apatite	Non- magnetic zircon rutile
arnet livine	hypersthene	tremolite	6 6	
hromite	actinolite	enstatite spinel	andalusite monazite	anatase brookite
chloritoid	staurolite (light) epidote biotite chlorite tourmaline (dark)	staurolite (dark) muscovite zoisite clinozoi- site tourmaline (light)	xenotime	topaz fluorite kyanite sillimanite beryl
h	loritoid	(light) epidote biotite chlorite tourmaline	(light) (dark) epidote muscovite biotite zoisite chlorite clinozoi- site tourmaline tourmaline	(light) (dark) epidote muscovite biotite zoisite chlorite clinozoi- site tourmaline tourmaline



Fig. 9 FRANTZ Isodynamic Magnetic Separator.

Dip one match in canada balsam and stick the match to the cover glass. Now carefully bring the cover glass on the preparate supporting it on one side with another match. Gently let the cover glass drop down from one end (Fig. 10). Press the cover glass down firmly, put the slide aside and let it cool. After fifteen minutes the excessive balsam can be split off with a knife and the slide cleaned with xylene. Scratch the sample no. (and magnetic fraction) in the object glass with a diamond pencil.

## E. Bromoform Recovery from Wash Fluid

If, after some time, the fluid turns brown and its density decreases below 2.89 shake it with charcoal (e.g. medicinal tablets) and then filter the suspension.

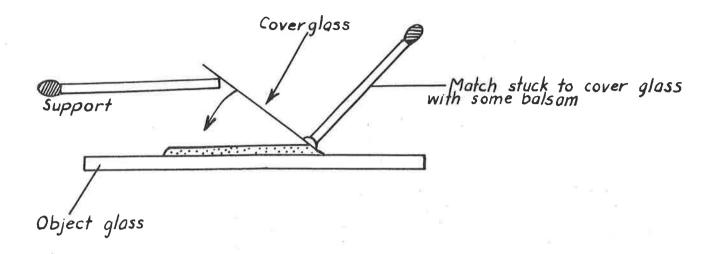


Fig. 10 Techniques for mounting mineral specimens for microscopic examination.

The bromoform can be recovered from the wash fluid as follows:add to half a bottle of wash fluid the same amount
of water and shake thoroughly. Leave the contents
for a few hours. The bromoform will separate to
the bottom, the alcohol-water mixture to the top.
Now bring a glass tube attached to a water tap deep
in the bottle and leave water percolating gently overnight. The water will remove all alcohol left in the
bromoform. Carefully decant excess water and do
the final separation in a 250 cc separation funnel.
Filter the contents back into a bromoform bottle.

- F. Identification of Heavy Minerals with the Polarizing Microscope

  Tables 4 and 5 are only meant as an aid for rapid identification.

  The use of the petrographic microscope and a working knowledge of crystallography is assumed. Table 4 provides a means of identification of the most common heavy minerals based on the following optical properties:
  - a. Isotropism or anisotropism
  - b. Extinction
  - c. Colour and pleochroism
  - d. Optic sign and optical character
  - e. Elongation
  - f. Birefringence

Table 5 is a check list with description of the optical characteristics of the various minerals. It is advisable to use this check list in combination with a handbook providing a more detailed description of the diagnostic properties such as Milner, H.B. (1962); Kerr, P.F. (1959); Tickell F.G. (1965); Winchell, A.M. (1951).

For every slide the ratio opaques and alterites versus transparent minerals should be estimated as well as the percentages of the various transparent minerals. In practice an identification of 100 minerals is needed to establish the ratio opaques, alterites/transparent minerals followed by a further identification of transparent minerals until the latter add up to 100. Identification should be carried out systematically by using a mechanical stage or point counter.

#### REFERENCES

- Hess, H. H., 1959: Notes on the Operation of FRANTZ Isodynamic Magnetic Separator. Advertising pamphlet for FRANTZ Isodynamic Separator, New York.
- Kerr, P.F. 1959: Optical Mineralogy. 3rd ed. McGraw-Hill, New York, 442 pp.
- Milner, H.B. 1962: Sedimentary petrography, Vol. 1. Methods in sedimentary petrography. 4th ed. Allen and Unwin, London, 643 pp.

  1962: Sedimentary petrography, Vol. 2. Principles and applications. 4th ed. Allen and Unwin, London, 715 pp.
- Müller, G. M. (1964): Methoden der Sedimentuntersuchung. E. Schweizerbart'sche Verlagsbuchhandlung Stuttgart, 303 pp.
- Geologisch Instituut der Universiteit Utrecht, 1962: Praktikum Bodem en Sediment Minerologie. Unpubl.
- Tickell, F.G. 1965: The techniques of sedimentary mineralogy. Elsevier, Amsterdam, 220 pp.
- Winchell, A.M. 1951: Elements of Optical Mineralogy. Pt 2, Description of minerals, 4th ed. Wiley, New York, 551 pp.

### TABLE 4

# Identification of heavy minerals based on their optic characteristics. (modified after Tickell, 1965)

\* Minerals partly or completely dissolved in HCl.

	Extinction	Colour	Pleochroism	Interference	Optical Sign	Elongation	Mineral	Birefringence	2 V			
Isotropic							Garnet Spinel Diamond Chlorite group					
					+	+ + + -	Zircon Cassiterite	0.055-0.062 0.097				
				1	-	1 1	Corundum Apatite * Beryl	0.008-0.009 0.003-0.005 0.004-0.008				
		Ω				+	Vesuvianite Brookite Enstatite	0.004-0.008 0.158 0.008-0.009	0°-30° 54°-80°			
		Straignt			+	+	Sillimanite Zoisite *	0.020-0.023	21°-30° 0°-60° 6°-19°			
Anisotropic	ght				2		<u>+</u>	Monazite * Topaz (Titanite)	0.049-0.051 0.008-0.010 0.092-0.141	44°-66° 23°-37°		
Aniso	Strai								•		+ +	Hypersthene Epidote Andalusite
					=	Dumortierite (Beryl)	0.011-0.020 0.004-0.008	20°-40°				
			roic		+	<u>+</u> +	Rutile  Xenotime	0.287 0.095-0.107 0.055-0.062				
		Coloured	pleochroic	1		+ + + +	Zircon Cassiterite Anatase	0.097	,			
		Ŭ	Non		-	-	Apatite * Vesuvianite (Beryl)	0.003-0.005 0.004-0.008 0.004-0.008	-			

	Extinction	Colour	Pleochroism	Interference	Optical Sign	Elongation	Mineral	Birefringence	2 V				
						+	Barite	0.012	36 <sup>0</sup> -38 <sup>0</sup>				
			ပ္ပ			+	Brookite	0.158	0°-30°				
			pleochroic			+	Monazite *	0.049-0.051	6° <b>-1</b> 9°				
			soc <sub>l</sub>	9	+	+	Sillimanite	0.020-0.023	2 <b>1</b> °-30°				
				2		+	Olivine *	0.037-0.041	53°-90°				
la l			Non			-	Topaz	0.008-0.010	44°-66°				
			4	8		+	(Olivine) *	0.037-0.041	53° <b>-</b> 90°				
					_	-	(Beryl)	0.004-0.008					
					+	+	Rutile	0.287					
						1		+	Anatase	0.061			
ł						+	Corundum	0.008-0.009					
		-73			-	-	Apatite *	0.003-0.005	i				
	Straight	Coloured				-	Tourmaline	0.015-0.040					
	tra	lot				+	Brookite	0.158	0°-30°				
	Ω	ü				+	Enstatite	0.008-0.009	54 <sup>0</sup> -80 <sup>0</sup>				
						+	Sillimanite	0.020-0.023	2 <b>1</b> °-30°				
ic			Pleochroic				+-	+	Staurolite	0.010-0.015	79°-90°		
Anisotropic						[당				+	Anthophyllite	0.016-0.025	70°-90°
sot									]     			+	Olivine *
Ani			[집	2			(Titanite)	0.092-0.141	23°-37°				
~				4		+	Hypersthene	0.010-0.016	45°-90°				
						+	Basaltic Hornblende	0.023-0.072	64 <sup>0</sup> -80 <sup>0</sup>				
						+	Glaucophane	0.013-0.018	0°-68°				
					-	+	(Olivine) *	0.037-0.041	53°-90°				
						<u>+</u>	Epidote	0.014-0.045	65°-90°				
						-	Dumortierite	0.011-0.020	20°-40°				
						-	Andalusite	0.007-0.011	83°-85°				
						+	Monazite *	0.049-0.051	6° <b>-1</b> 9°				
						+	Spodumene	0.013-0.027	54°-60°				
					+	+	Clinozoisite	0.005-0.011	65°-90°				
	(a)	Ω Ω					Augite	0.043	58°-62°				
	Oblique	Colourless		2			Titanite	0.092-0.141	23°-37°				
	Obl	lou		"		+	Kyanite	0.016	82 <sup>0</sup>				
	-	S				+	Muscovite	0.037-0.041	30°-40°				
	-				-	<u>+</u>	Epidote	0.014-0.045	65°-90°				
						<u>±</u>	Wollastonite	0.014	36°-42°				
	H						Axinite	0.010-0.012	70°-75°				

	Extinction	Colour	Pleochroism	Interference	Optical Sign	Elongation	Mineral	Birefringence	2 V						
						+	Monazite	0.049-0.051	6°-19°						
						-	Clinochlore	0.004-0.011	0°-50°						
			oic		+		Diopside	0.029-0.031	56°-61°						
			hr				Augite	0.043	58°-62°						
			pleochroic	2			Titanite	0.092-0.141	23°-37°						
						+	Tremolite-Actinolite	0.022-0.027	79°-82°						
			Non			+	Kyanite	0.016	82 <sup>0</sup>						
											v.	<u>+</u>	Epidote	0.014-0.045	65 <sup>0</sup> -90 <sup>0</sup>
						-	Clinochlore	0.004-0.011	0°-50°						
						+	Monazite *	0.049-0.051	6° <b>-</b> 19°						
ic			Coloured			+	Spodumene	0.013-0.027	54°-60°						
Anisotropic	ne	red				-	Riebeckite	0.004	80° <b>-</b> 90°						
sot	Oblique	lou			+	-	Clinochlore	0.004-0.011	0°-50°						
Ani	OR O	Co				-	Chloritoid	0.011-0.016	36 <sup>0</sup> -63 <sup>0</sup>						
7							Augite	0.043	58 <sup>0</sup> -62 <sup>0</sup>						
			oic				Titanite	0.092-0.141	23°-37°						
- 5			Pleochroic	2		+	Hornblende	0.019-0.026	52°-85°						
			leo			+	Basaltic Hornblende	0.023-0.072	64 <sup>0</sup> -80 <sup>0</sup>						
			E.			+	Glauconite *	0.020-0.032	16°-30°						
					_	+	Kyanite	0.016	82 <sup>0</sup>						
						+	Tremolite-Actinolite	0.022-0.027	79 <sup>0</sup> -82 <sup>0</sup>						
						+	Glaucophane	0.013-0.018	0 <sup>0</sup> <b>-</b> 68						
						±	Epidote	0.014-0.045	65°-90°						
	4 1					-	Riebeckite	0.004-0.006	80° <b>-</b> 90°						
						-	Aegirine-Augite	0.029-0.037	60°-81°						

TABLE 5

Description of Determinative Optical Characteristics of

	Remarks	Often well rounded dirty grains. Alteration products of minerals (e.g. Epidote). Sometimes on the edges the original mineral visible	Resembles aegirine but is distinguished by larger extinction angle. Most easily distinguished from hornblende by pyroxene cross sections and cleavage.	Basal sections:isotropic. Idiomorphic rounded form. Often dirty grains with clear, straight edgarims, sometimes striations. Dark coloured grains almost metallicopaque; sometimes anomalous bi-axial.
	Elonga- tion		Fast .	Slow +
istics of	Op <b>t.</b> sign and axial angle		2 + 2 - 60°-81°	<b>-</b>
ical Character y Minerals	Extinction		Oblique	Straight
ninative Opt mmon Heav	◁		Strong 0.029- 0.037	Strong 0.061
Description of Determinative Optical Characteristics of the Most Common Heavy Minerals	Relief		Very high n = <del>1</del> 1.75	Extremely high n = ±2,5
Descri	Pleochroism	Absent	Moderate, yellow- green - greenish	Absent, except in very thick grains pale blue or yellow, dark blue or orange
	Colour	Dirty, grey brown	Green - greenish black (opaque)	Yellow, brown, colourless, blue (trans- parent- opaque)
	Minera.	Alterite	Aegirine- Augite	Anatase

		32	•	
Remarks	Mostly transparent. Irregular form. Axial figure seldom observed. Different from topaz by optical sign and pleochroism and also by inclusions of graphite or carbonaceous material, furthermore often alterations which make the grains dirty.	Transparent, often small grains, Habit:rounded elongated prisms or eggshaped. Solution features because of HCl treatment. (Mostly not present).	Rounded prismatic or irregular fragments with coarse ragged teeth (in the last case a good axial figure is usually obtainable. Dispersion r > v	Inclusions more common than in hornblende. Often elongate large grains.
Elonga- tion	F 28.21	Fast -		Slow +
Opt. sign and axial angle	83°-85°	1 <del></del>	2 + 58°-62°	2 - 64°-80°
Extinction	Straight	Straight	Oblique + 45	Straight to oblique $0^{\circ}$ -12 $^{\circ}$
$\triangleleft$	Weak 0.007- 0.011	Weak 0.003-	Strong	Moderate to strong 0.023-0.072 own colour often dominant
Relief	High n = <b>11.</b> 64	High n = ±1.63	Very high n = ±1.72	High n = ±1.68
Pleochroism	Strong (often absent); colourless (greenish), pink or bloodred	Absent, weak in some coloured var.	Absent or weak (with violet or red)	Moderate, dark olive green or brown, dark brown pale yellow
Colour	Colourless, pale hues of red, green brown, violet	White, colourless sometimes greenish	Transparent bottle green (silver beet colour) yellow green brown, violet	Brown, brownish black, olive green
Mineral	Andalusite	Apalite	Augite	Basaltic Hornblende, Lamprobolite Basaltine Oxyhorn- blende

			33.			
Remarks	Anomalous optical properties. Prismatic form, striation parallel, "dirty" through inclusions.	Mostly rounded, flaky grains (cleavage // 001 perfect). Good axial figure. Dispersion r > v	Differs from zoisite in its oblique extinction and larger optical angle. "Ultra blue" anomalous birefringence colours occasionally exhibited.	In contrast to kyanite, Diopside shows often good cleavage and fracture patterns.	Irregular, prismatic grains. "Dirty" colour often characteristic (Bronzite:intermediate in optical properties between Enstatite and Hypersthene)	
Elonga- tion	Slow +	Fast -	Slow + Fast -		Slow +	
Opt. sign and axial angle	2 + 0°-30°	2 + 36°-63°	2 + 65°-90°	2 + 56°-63°	2 + 54°-80°	
Extinction	Sometimes straight (often no extinction)	Straight to oblique 0°-20°	Oblique 2°-12°	Oblique 38°-45°	Straight	
◁	Very strong 0.158	Moderate- weak 0.011- 0.016	Weak 0.005-	Strong 0.029- 0.031	Weak 0.008-	
Relief	Extremely high $n = \frac{1}{2}2.6$	High n = <del>1</del> 1,72	High n = <del>1</del> 1,72	High n = ±1.67	High n = <b>±1.</b> 65	
Pleochroism	Sometimes, light yellow brown - dark (red) brown	Weak-strong; olive green, indigo-blue yellow green	Absent - weak green, pink, red	Absent	Moderate, increasing with higher Fe content yellow brown-green	
Colour	Brown and yellow	Olive greenblue, grey yellow	Colourless, yellow, pale green, brown	Colourless, pale green white, grey	Colourless, yellow, green	
Mineral	Brookite	Chloritoid	Clinozoisite	Diopside	Enstatite	

34.					( 103
Remarks	Irregular (equidimension- al?) angular grain. Clear to dirty. Characteristic bright colours (green, violet red rings on the edges). Strong dispersion r > v	Irregular form, high relief. No cleavages.	Pleochroic, often oblate grains characteristic. Inclusions common.	Prismatic cleavage parallel length or crossing at 124, sometimes solution marks. Inclusions uncommon.	Normally ragged, prismatic form. Often small inclusions (Schiller structure)
Elonga- tion	Some +		Slow +	Slow +	Slow +
Opt. sign and axial angle	2 - 65°-90°	×	2 <b>-</b> 0 <sup>0</sup> -68 <sup>0</sup>	2 <b>-</b> 52 <sup>0</sup> -85 <sup>0</sup>	2 45°-90°
Extinction	Straight- oblique 0°-5°		Oblique 4°-6°	Oblique 12-30° (some- times straight)	Straight
◁	Moderate- strong 0.014- 0.045		Moderate 0.013- 0.018	Moderate 0.019- 0.026	Moderate 0.010- 0.016
Relief	High n =±1,74	High - very high $n = \frac{1}{2}$ .	High n = <del>1</del> 1.63	High n = <b>±</b> 1,66	High $n = \pm 1.7$
Pleochroism	Weak or absent colourless, yellow green	Isotropic	Moderate-strong blue, violet, colourless yellow	Moderate - weak yellow, straw yellow pale green brown or dark	Weak-moderate (in ferriferous varieties) yellow green, pink or red yellow. (traffic-light)
Colour	Colourless, lemon, yellow, dark-green, hues of brown, grey	Colourless, pink, red, yellow- brown, green	Colourless- blue	Green-brown transparent	Yellow- brown, green, pink (trans-
Mineral	Epidote	Garnet	Glaucophane	Hornblende	Hypersthene

35 <b>.</b>					
Remarks	Mostly angular prismatic grains with sometimes cleavage parallel the length.	Often irregular formed, broken grains. Solution marks.	Often well formed prismatic grains or also broken grains. Sometimes characteristic oblique striation.	Mostly elongated prisms or fibres with distinct, sometimes irregular cleavage parallel the length. If equidimensional grain; low interference colours and cleavage less obvious.	Irregular form, determined by cleavage, haphazard boundaries. In a few cases perfect euhedra are seen. Inclusions common especially in dark varieties.
Elonga– tion	(Slow +)	Slow +	Slow +	Slow +	Slow +
Opt. sign and axial angle	2 - 820	2 + 53°-90°	+ +-	2 + 21 - 30°	2 + 79°-90°
Extinction	Oblique max. 32 <sup>0</sup> (0 <sup>0</sup> -32 <sup>0</sup> )	Straight	Straight	Straight	Straight
◁	Moderate 0.016	Strong 0.037- 0.041	Extreme 0.287	Moderate 0.020- 0.023	Moderate 0.010- 0.015
Relief	High n = <b>±1.</b> 72	High n = <b>-</b> 1,67	Very high n = ±2.8	High n = <del>1</del> 1,66	High n = <b>±1,</b> 74
Pleochroism	Absent, weak in dark coloured grains, colourless, violetblue, cobalt-blue	Absent	Weak, darkest colour // length of x, red brown, brown/yellow	Moderate or absent pale brown- yellow brown or green dark brown or blue	weak, colourless pale yellow, yellow reddish yellow-brown
Colour	Colourless	Colourless, light, yellow green	Red brown golden-yellow, red	Colourless, yellow pale hues of brown, green,	Straw <u>yellow</u> orange yellow,brown red-brown
Mineral	Kyanite	Olivine	Rutile	Sillimanite	Staurolite

36 <b>.</b>					
Remarks	Mostly, small irregular angular grains, sometimes dirty. Dispersion strong r > v Ultra blue - Yellow and red polarization colours.	Irregular glassy grain. To distinguish from Andulasite by absence of pleochroism, optical sign and transparency.	Mostly well rounded, no alterations, no fracturing or cleavage. Good axial figure only in grains without pleochroism.	Amphibole character, fibrous, ragged ends.Frequently alteration to chloritic matter. Inclustions of iron ore and carbonaceous matter.	
Elonga– tion		Fast -	된 88 1	Slow +	
Opt. sign and axial angle	2 + 23°-37°	2 + 44°-66°	: । च	2 - 79°-82°	
Extinction	Oblique, total extinction seldom observed	Straight	Straight	Oblique 10°-20°	
◁	Very strong 0.092- 0.141	Weak 0.008- 0.010 (0.003)	Strong 0.015- 0.040	Moderate- strong 0.022- 0.027	
Relief	Very high n = ±1.91	High n = <del>1</del> 1,62	High n = -1,65	High n = <del>1</del> 1.67	
Pleochroism	Absent, sometimes weak in strongly coloured varieties. Yellow or colourless, greenish pink or brownish yellow, pink or pale yellow.	Absent apart from some thick grains brownish-yellow, yellowish violet pink,	Very strong (brown var.) - weak (blue var.). green, yellow brown, blue or black, yellow violet or colourless	Absent - weak (in green varieties)	
Colour	Brown, vellow-brown, orange, yellow-green, olive green	Colourless yellow, blue,red, green	Yellow- brown, pink, yellow, orange, green blue, grey	Colourless- pale green	
Mineral	Titanite (Sphene)	Topaz	Tourmaline	Tremolite-Actinolite	

	9	37.			
Remarks	Prismatic, clear grains, characterised by colour and low birefringence.	Normally idiomorphic grains. A strong resemblance to coloured zircon, however less often inclusions.	Clear, mostly small grains; prismatic with pyramidal terminations. Idiomorphic mostly showing a certain degree of rounding. Sometimes zoning. Inclusions common.	Prismatic. Often green rod- like (amphibole) inclusions. Var. thulite (pink-red pleochr.) resembles andalu- site. Pol. col. deep blue ("ultra blue") characteristic.	
Elonga- tion	Fast •	Slow +	Slow +	Slow +	
Opt. sign and axial angle	1 - often anomalous 2 + 17 <sup>o</sup> -33 <sup>o</sup>	+	+	2 + 0°-66°	
Extinction	Straight	Straight	Straight	Straight, sometimes no extinction	
◁	Weak 0.004-	Strong 0.095- 0.107	Strong 0.55- 0.62	Weak- moderate 0.006- 0.018	
Relief	High n = ±1,71	Very high n = <del>1</del> 1,75	Very high n = 1.95	High n = <sup>‡</sup> 1, 70	
Pleochroism	Moderate or absent, variable, yellow green-colourless; yellow brown-brownish-grey; dark blue-colourless; redcolourless or grey	Moderate, yellow, green; pale yellow	In thick grains and strongly coloured varieties	Absent, sometimes moderate	
Colour	Brown, green-brown, red, green, pale-blue, yellow	Pale vellow - brown yellow, orange, pink	Colourless or pale hues of yellow, brown pink etc.	Colourless grey	
Mineral	Vesuvianite (Idocrase)	Xenotime	Zircon	Zoisite	