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DEPARTMENT OF
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BUREAU OF MINERAL RESOURCES, GEOLOGY AND GEOPHYSICS

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METHODS OF SAMPLE PREPARATION AND ANALYSIS USED IN THE BROAD SOUND ESTUARY STUDY PROJECT

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SUMMARY

Methods of sample preparation used in the treatment of about 350 samples of unconsolidated sediments collected from the Broad Sound area are described. Ground fractions were prepared for P_2O_5 determinations by colorimetric analysis, trace element determinations by direct reading optical spectrometer and atomic absorption techniques, and mineralogical identification by X-ray diffraction. The weight loss and CO_2 evolution methods of carbonate analysis used in the section's laboratory are described in some detail, as are also the operating procedures for the automatically recording settling tube and pipette methods of grainsize analysis.

Sample preparation methods for the study of the organic component of the sediments, heavy minerals, and preparation for thin sections of the calcareous and non-calcareous fractions are briefly described.

INTRODUCTION

The study of recent sediments of the Broad Sound area necessitated the sedimentological and geochemical analysis of a large number of samples. In order to deal with these samples within the limited time available it was necessary to devise a flow chart which would produce the required analyses with the minimum duplication of sample treatment.

This report has been produced both to document the procedures used in the Broad Sound investigation and also to assist other workers who may be involved in the formulation of analytical techniques in a study of this type in the future.

SAMPLE COLLECTION AND TREATMENT

Approximately 350 samples of Holocene sediments were collected in the Broad Sound area on a 2-km sample grid. Subaqueous samples were collected by dredging; sub-aerial samples were collected by clearing surface litter and then digging out a 30-cm cube sample which was then quartered until the required size was obtained. The samples, together with a number of samples collected from other selected localities, were then subjected to the series of steps shown in the flow chart (Fig. 1). Each of the steps involved will now be considered.

Sample drying and grinding

The bulk sample was transferred to a 20-cm evaporating dish and allowed to oven-dry at 60°C for up to 3 days. It was considered desirable to dry at this comparatively low temperature in order to minimize the possibility of mineral transformations (such as aragonite to calcite). Also, excessive heat was found to 'bake' samples, producing grain aggregates which then gave incorrect grainsize analyses. Where samples were washed before drying in order to remove sodium chloride, the sample was first dispersed in 500 ml of water. The suspension was then poured onto a 0.125 mm Buchner vacuum filter funnel lined with No. 41 filter paper and washed with distilled water. The sample was subsequently transferred from the filter paper to an evaporating dish and dried at 60°C.

The dried sample was split using a metal sample splitter, and samples for geochemical analysis were manually ground using a porcelain mortar and pestle until all the sample passed through a nylon sieve of the appropriate mesh size.

The splitter, brushes, sieves, glazed paper, etc. were cleaned thoroughly after each sample to prevent contamination.

Analyses of ground material

Samples were submitted for analysis as follows:-

Phosphate

Colorimetric Analysis

10 g of sample was ground down to 50 mesh and then submitted to Australian Mineral Development Laboratories (AMDEL) for P_2O_5 determinations.

XRD mineralogy

5 g of sample was ground down to 200 mesh and submitted to the BMR Laboratories.

Trace element analysis (DROS)

5 g of sample was ground down to 200 mesh and submitted to the BMR Laboratories for determination of the following trace elements by Direct Reading Optical Spectrometer (DROS)

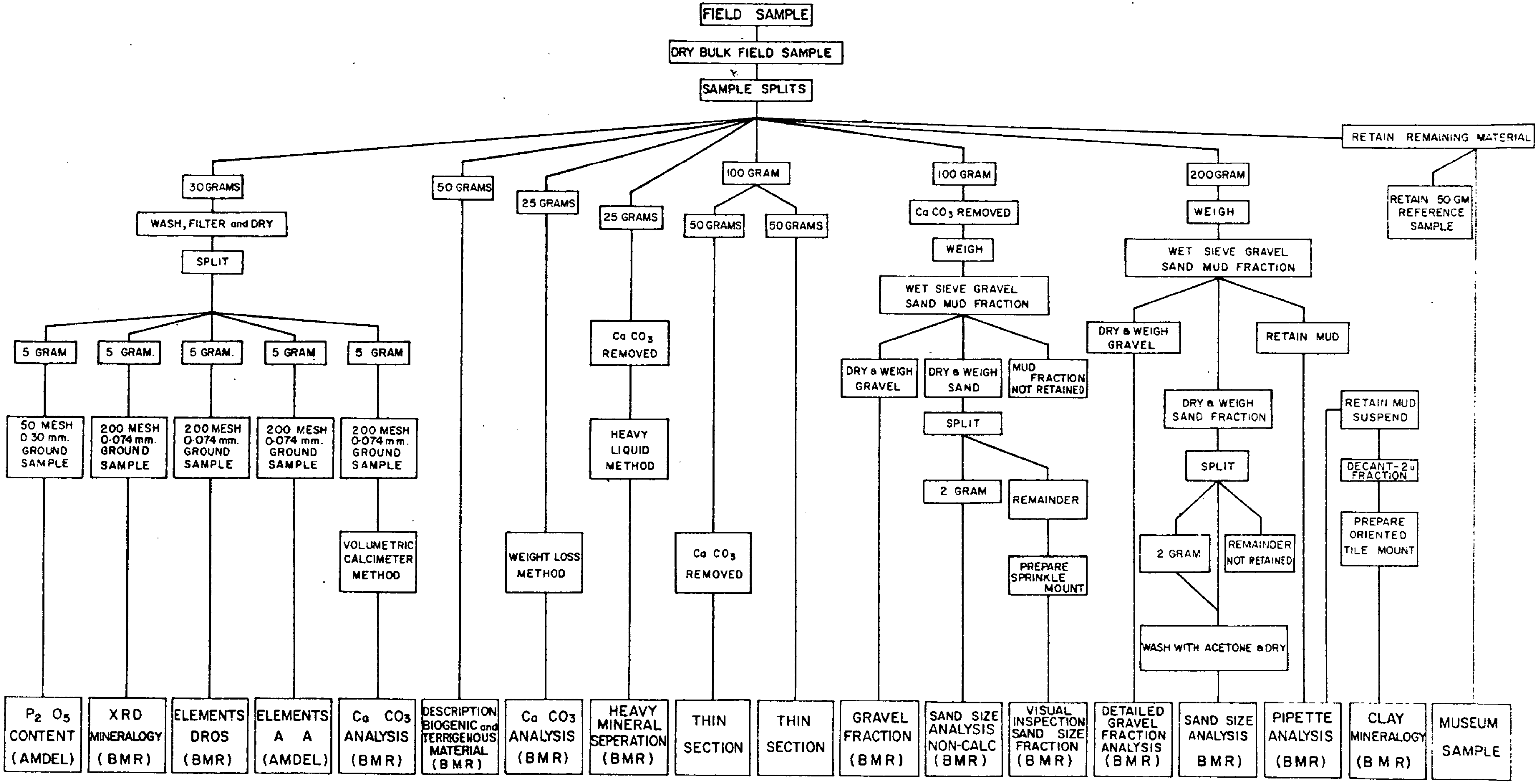
Ba, Ca, Co, Cr, Cu, Fe, La, Mg, Mn, Ni,
Sc, Sr, Ti, V, Y, Zn.

Trace element analysis (AA)

5 g of sample was ground down to 200 mesh, and submitted to AMDEL for Atomic Absorption Analysis (AA). The following elements were determined: Pb, Zn, Cd, Mo.

FIG. I

SAMPLE TREATMENT FLOWSHEET FOR THE BROAD SOUND ESTUARY STUDY PROJECT



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$CaCO_3$ Analysis

(a) Weight Loss Method. 25 g of finely ground sample required; method detailed in Appendix II.

(b) Volumetric Method using Collins' Calcimeter (Fig. 2). 1-2 g of finely ground sample required. This method was used for supratidal (including supratidal mangrove) samples where there was known to be significant quantities of gypsum. The method is detailed in Appendix II.

Analyses of unground material

Biogenic and Terrigenous material

Visual observations (by binocular microscope) were made on a 50-g sample, split from the bulk field sample.

Heavy minerals

A 25-g split sample was wet-sieved to obtain the sand fraction only; carbonate was removed by treatment with dilute HCl. The non-calcareous sand fraction was washed with acetone and allowed to dry at room temperatures. The sample was then accurately weighed, heavy minerals separated with bromoform by the standard technique, and grain mounts prepared.

Thin sections

(a) Calcareous

A 50 g representative sample was split from the bulk field sample and submitted to BMR transit room for thin sectioning by contract. All samples required impregnating before sectioning.

(b) Non-calcareous

A 50 g representative sample was split from the bulk field sample; the $CaCO_3$ was removed with dilute HCl. The remaining non-calcareous sample was submitted to the BMR transit room for sectioning. All samples required impregnation.

Grainsize Analysis

Non-calcareous gravel and mud fractions

$CaCO_3$ was removed from a weighed 100 g sample split from the bulk sample. The gravel fraction (over 2.00 mm), obtained by wet sieving the sample, was oven dried, weighed and retained. The mud fraction was wet sieved and not retained.

Sand fraction - non-calcareous

(2.0 - 0.0625 mm.)

The sand fraction was obtained by wet-sieving the original sample to remove the gravel and mud fractions; the residue was oven dried and weighed. A 2 g split was taken, washed with acetone and allowed to dry at room temperature; part of each remaining sand fraction was mounted onto a glass slide for visual inspection. The 2 g split representing the non-calcareous sand fraction was used for detailed sand grainsize analysis using the Rapid Sediment Analyser (settling tube), following the instructions given in Appendix 1.

Calcareous gravel fraction

An approximately 200 g split from the bulk sample was wet-sieved into gravel, sand, and mud fractions which were retained for detailed size analysis.

The gravel fraction was dry-sieved using a mechanized Endecott sieve-shaker; the following sieve sizes were used:

Sieve Mesh Size	Millimetres
8	2.00 - 2.38
7	2.38 - 2.83
6	2.83 - 3.36
5	3.36 - 4.00
	4.00 - 4.76
	4.76 - 9.53
No mesh; wire squares used	9.53 -16.00
" " " " "	16.00 -64.00

Each size fraction was individually weighed and recorded.

Calcareous sand fraction

Identical method to that used for the non-calcareous sand fraction using the settling tube.

Mud fraction - pipette method

Analyses were undertaken on samples containing greater than 5 percent mud. Approximately 15 g of material is the recommended amount for a pipette analysis. Larger samples result in interference between grains and encourage flocculation, whereas samples of less than 5 g increase weighing errors.

Using distilled water, an appropriate amount of sample was wet-sieved; the retained mud fraction was then transferred to a 100-ml measuring cylinder; the water level was brought up to the 1000 ml mark using distilled water diluted with a 1 percent dispersant (sodium hexametaphosphate).

The sample was mixed with a stirrer, and the settling time commenced when stirring was completed. Before each reading the 20-ml pipette was held at the required depth. The fluid sample was withdrawn and then transferred from the pipette into a 50-ml beaker. The pipette was washed through with distilled water (using a wash bottle). The 50-ml beaker plus sample was then oven dried at 110°C for 24 hours, weighed accurately, and the results recorded.

A schedule indicating withdrawal depths and times used as a reference when sampling is given in Table 1.

Table 1. Schedule of withdrawal depths and times used for pipette analysis at various temperatures.

Readings	Phi (ø)	Depth	TIMES 19°C	TIMES 21°C	TIMES 23°C
1	4	20 cm	20^{s}	20^{s}	20^{s}
2	4.5	20 cm	$1m58^{s}$	$1m52^{s}$	$1m46^{s}$
		RESTIR			
3	5	10 cm	$1m57^{s}$	$1m52^{s}$	$1m46^{s}$
4	5.5	10 cm	$3m54^{s}$	$3m44^{s}$	$3m33^{s}$
5	6	10 cm	$7m54^{s}$	$7m32^{s}$	$7m11^{s}$
6	7	10 cm	$31m12^{s}$	$29m44^{s}$	$28m21^{s}$
7	8	10 cm	2hrs5m	1hr59m	1hr53m
8	9	10 cm	8hrs22m	7hrs58m	7hrs36m

The water temperature often changed significantly between the 7th and 8th readings and the time for the 8th reading was adjusted accordingly.

After the 8th reading, the top 10 cm of the clay suspension was syphoned off, this clay fraction size being 2 microns or less.

Clay Mineralogy - XRD

An oriented clay mount was made directly from the -2 micron suspended material (previously gained from pipette analysis). The mount was prepared by slowly applying the clay suspension to a circular 18 mm x 1.5 mm ceramic tile which has been mounted on a vacuum tube. Sufficient fluid was applied to deposit a uniform thickness of clay which thoroughly concealed the granular texture of the tile; the clay mount was then allowed to dry beneath an inverted beaker at room temperature for about 2 days before X-ray analysis.

APPENDIX 1

INSTRUCTIONS FOR DETERMINATION OF GRAINSIZE USING THE SETTLING TUBE AND HEWLETT PACKARD 7004A X-Y RECORDER

by

W. Mayo

Sample Preparation

1. Remove size fractions greater than -1 phi (2 mm) and less than 4 phi (0.0625 mm).
2. Wet sponge with distilled water.
3. Use less than 2 g.
4. Sprinkle sample evenly over the wire gauze and make sure sample is wet.
5. Ensure average temperature of top and bottom of tube is between 20°C and 22°C.

Operating Instructions

1. Switch on both wall mounted switches.
2. Depress "power" switch on recorder and wait a few seconds.
3. Insert paper and depress "chart" switch.
4. Depress "servo" switch.
5. Press "pen" switch and adjust zero with the two "zero" knobs (DO NOT TOUCH VERNIER KNOBS - LEAVE IN THE FULLY CLOCKWISE POSITION).
6. If necessary test ink level using "start" and "reset" buttons - if needed fill through hole in drum.
7. Press "pen" switch to lift pen from paper.
8. Set sweep rate on 5, 10, or 20 depending on grainsize of sample and the range on 2.
9. Rotate wire gauze on to tube mouth with a smooth even movement (too fast disturbs the water; too slow starts recorder prematurely).

10. After sufficient time has been allowed for the smallest (4 phi) particles to have fallen (about 3 minutes) press "servo" switch.
11. Lift wire gauze from tube.
12. Press "servo" switch again.
13. Repeat for next sample after any finer particles have fallen through tube.
14. Sediment should be cleared from the sample collecting pan (with moveable rod at the base of the tube) regularly. THE RIGHT-HAND SERVO KNOB WILL HAVE TO BE TURNED QUICKLY CLOCKWISE WHEN THE PAN IS CLEARED TO PREVENT DAMAGE TO THE RECORDER.
15. Use only distilled water in tube.
16. Ensure tube is always topped up by using the water valve.

Calculation Procedure

Moments Method (Statistical)

1. The distances of pen travel for the 21 quarter-phi intervals (between -1 phi and 4 phi) for three different sweep rates are given in the following table.
2. Measure the amount of deflection (from the zero line) for each of these distances from the recorded graph.
3. Record these readings directly on data sheets.
4. Computer will give mean, variance, standard deviation, skewness, kurtosis.

Graphical Method (Folk and Ward)

1. Determine 5%, 16%, 25%, 50%, 75%, 84%, and 95% of the total deflection (= deflection at 4 position).
2. Read off phi value corresponding to value of these from recorded graph.
3. Parameters for mean, standard deviation, skewness, and kurtosis may be worked out directly on calculator or programmed on Canon Canola calculator.

DISTANCES OF PEN TRAVEL

Particle Diameter (in phi)	Time of fall (secs)	Distance of travel in cms for given sweep rates 5 sec/inch (1.83 sec/cm)	10 sec/inch (3.65 sec/cm)	20 sec/inch (7.30 sec/cm)
-1.0	7.4	4.0	2.0	1.0
-0.75	8.2	4.5	2.2	1.1
-0.5	9.1	5.0	2.5	1.2
-0.25	10.3	5.6	2.8	1.4
-0.0	11.9	6.5	3.3	1.6
0.25	13.4	7.3	3.7	1.8
0.50	15.3	8.4	4.2	2.1
0.75	17.9	9.8	4.9	2.5
1.00	21.1	11.5	5.8	2.9
1.25	25.5	14.0	7.0	3.5
1.50	30.0	16.4	8.2	4.1
1.75	35.0	19.2	9.6	4.8
2.00	41.8	22.8	11.4	5.7
2.25	50.0	27.4	13.7	6.9
2.50	60.0	32.8	16.4	8.2
2.75	71.5	39.0	19.5	9.8
3.00	85.0	46.5	23.3	11.7
3.25	100.0	54.6	27.3	13.7
3.50	117.0	64.0	32.0	16.0
3.75	140.0	76.5	38.3	19.4
4.00	175.0	95.7	47.9	24.0

Notes: As the supplied graph paper is 38 cm wide, with a sweep rate of 20 all required information is recorded in one sweep. With a setting of 10 two sweeps are required and with a setting of 5 three are needed.

The sweep rate selected depends on the grainsize of the sample.

APPENDIX II

METHODS FOR THE DETERMINATION OF $CaCO_3$.

(a) Weight loss method

A 25 g sample is placed in a 500-ml beaker containing distilled water, dispersed by stirring and allowed to settle. Organic material (plant fragments) is then removed by decanting. The sample is then washed with distilled water in a 125-mm Buchner funnel. The sample is transferred by washing with wash bottle containing 10 percent H_2O_2 from the filter paper into a 12.5 cm evaporating dish; the remaining organic material is removed by adding 10 percent H_2O_2 and digesting in a hot water bath until most effervescence has ceased. With some samples this may take two days. The sample is washed through filter paper and oven dried at 110°C. A suite of numbered weighing bottles and numbered filter papers is oven dried at 110°C overnight. The numbered weighing bottles with and without the numbered filter papers are accurately weighed. The dried sample is transferred into a weighing bottle, dried at 110°C for approximately one hour, and then weighed accurately.

The sample is transferred from the weighing bottle into a 500-ml beaker containing dilute HCl, stirred oocasionally and allowed to stand until effervescence ceases. A check is made with litmus paper which ideally should record pH1: the sample is now washed onto numbered filter paper positioned in 75 mm long stemmed funnel and continually washed with distilled water until litmus paper is unaffected. The sample and filter paper are placed into the appropriate (numbered) weighing bottle and oven dried at 110°C overnight. Finally the bottle, filter paper, and sample residue are weighed and recorded with other weights previously obtained.

(b) Volumetric method using the Collins' Calcimeter.

This method was used for the supratidal (including mangrove) samples which were known to contain significant quantities of gypsum.

The approximate amount of sample to be used was determined from the amount of carbonate present, which has been previously found by the weight loss method: the greater the carbonate content, the less the amount of sample required. Thus for a sample containing 5 percent $CaCO_3$ approximately 2 g of material is required whereas for a sample containing 10 percent $CaCO_3$ approximately 1 g of material is required.

The sample is weighed and placed in flask B (Fig. 2) together with tube A which contains dilute HCl to the level indicated. Taps 1 and 2 are opened. Flask B is stoppered and immersed in the water in the tank and fastened by the clip positioned on the mounting board. The tank water is agitated to produce a uniform temperature by closing tap 2 and blowing air through tube C. Tape 2 is opened and the rubber bulb D squeezed until water in burette E stands at zero, at which moment tap 1 is closed. Flask B is then inverted, thus bringing the acid into contact with the sample and evolving CO_2.

The level in the U-tube is re-adjusted by squeezing bulb D and shutting tap 2, and the volume of gas is measured on the burette. Note the two readings were recorded, consequently it is not essential that the burette reading be set at zero before CO_2 evolution.

The percentage $CaCO_3$ in the sample is obtained by dividing the volume of CO_2, evolved (ml) by the weight of the sample (g), and multiplying the quotient by 0.5091.

A standard set of samples ranging from 5 percent to 40 percent $CaCO_3$ was used to test this method for accuracy and precision. Repeat analyses for each standard were always within 3 percent of the average of the repeat values.

The average of each set of repeats had to be multiplied by a factor of 1.14 to equate the average to the known value of the standard. This factor has been included in the above formula.

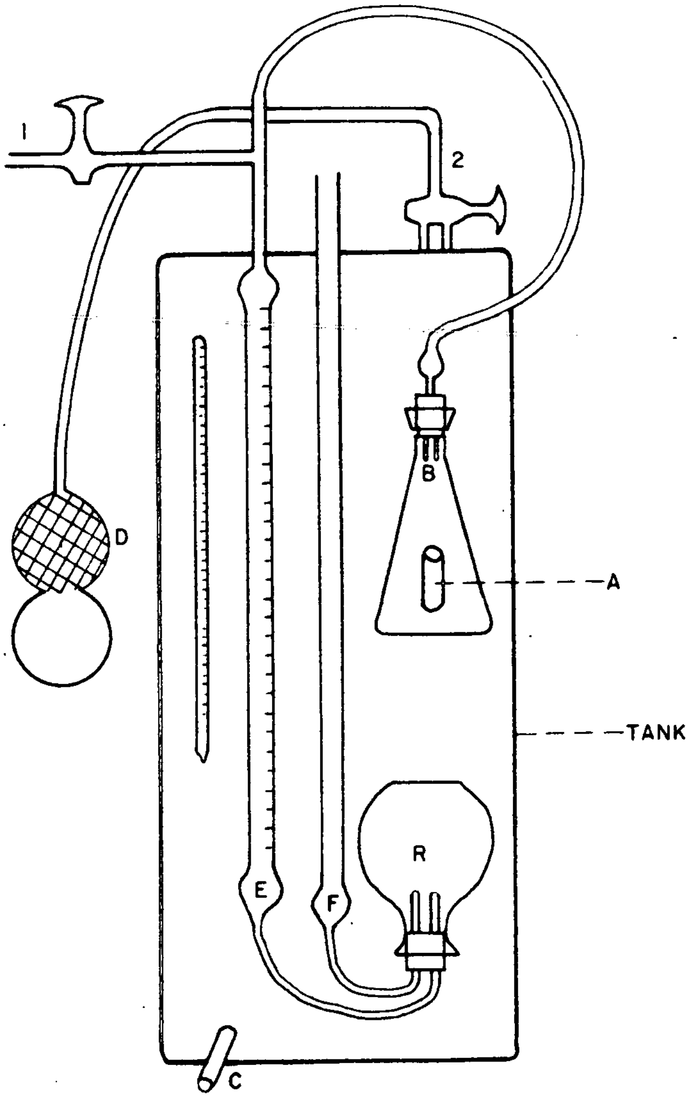

FIG. 2 COLLINS CALCIMETER

To accompany Record 1973/73

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