

**I.O.S.**

**A SHIPBOARD SYSTEM FOR EXTRACTING  
INTERSTITIAL WATER  
FROM DEEP OCEAN SEDIMENTS**

**P. S. RIDOUT**

**REPORT NO. 121**

**1981**

**NATURAL ENVIRONMENT  
INSTITUTE OF OCEANOGRAPHIC  
SCIENCES  
RESEARCH COUNCIL**

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ABSTRACT

A system is described which is used to extract large volumes of interstitial waters, on board ship, with procedures to minimise the effects of temperature, oxidation and contamination.

## INTRODUCTION

Knowledge of the composition of interstitial water is important to the understanding of the chemistry of deep ocean sediments. The analytical requirements include chlorinity, alkalinity, nutrients, dissolved gases, organics, major and trace elements. Shipboard sediment squeezing can provide interstitial water samples for most of these requirements, providing one is aware of errors which may arise from improper sampling, storage and handling of the material. Studies have shown that warming of marine sediments prior to pore water extraction can give rise to enrichments in potassium<sup>1,2</sup> and silica<sup>3</sup> and depletion in magnesium and calcium<sup>2</sup>. To overcome this 'temperature of squeezing' effect, sediment must be treated at 'in situ' temperatures. Additionally, some components of anoxic sediments are very sensitive to oxygen, especially phosphate<sup>4</sup> and iron<sup>5</sup>. It is essential, also, to minimise the storage time of the core prior to squeezing<sup>6</sup>.

Various systems have been developed for pore water extraction. They include leaching,<sup>7,8,9</sup> centrifugation,<sup>10,11</sup> liquid/gas displacement,<sup>12,13,14</sup> low and medium pressure gas/mechanical<sup>15-19</sup> and high pressure hydraulic/mechanical squeezers.<sup>20-25</sup> Leaching is little used as it is generally slow, difficult to control and would have limited use on board ship. The displacement systems, although giving good results with coarse grained sandy sediments, are inefficient when applied to fine grained or clay sediments. Low pressure systems have been used in many situations on a range of sediments. They produce good pore water samples but are generally slow in operation and do not produce large volumes of samples. Gas systems can use large volumes of inert gas which may not be generally available on board ship. High pressure, hydraulic systems are much quicker and can provide large volume samples for analysis, but may be heavy and unsuitable for trace metal studies due to their metal construction.

The aim of the system must be to provide a sample for analysis which is chemically representative of 'in situ' pore waters. Therefore, provision must be made for control of temperature, and for preventing contact with oxygen and contamination, whilst maintaining a system which is sufficiently transportable, reliable and uncomplicated for use on research vessels.

The system described utilises a mechanical, hydraulic system with pressures up to 4000 psi exerted on the sediment.

## THE SQUEEZING UNITS

A scale drawing of the unit is shown in Fig. 1. A full, detailed components list is provided in Table 1. The sediment is contained within the PVC unit which comprises a base (1), cylinder (2), securing ring (3), and a piston (4). Nitex nylon gauze (5) and a filter paper (6) located on the base, are held in position by the 'O' ring (7). The cylinder is fixed in place and the securing ring is tightened down using the wing nuts (8), on the stainless steel studs (9). The piston presses into the barrel, sealing on its two 'O' rings. (10) A syringe (60 ml) is inserted at the outlet port (11) of the base. The complete PVC assembly is positioned, on the hydraulic jack (12), inside the steel frame (13), so that the support plate (14) locates in a recess in the PVC base. The thrust pad (15) is placed on top of the piston. The pressure in the system is measured by a gauge (16) connected to the base of the jack with copper pipe (17,18).

The PVC construction allows no metal contact with either the sediment or the pore water. All mild steel parts are epoxy coated before use. Nitex gauze facilitates lateral flow to the outlet port which has a minimal dead volume. The filter papers used (Whatman 542) have good retention and wet strength properties. The cylinders should be no more than 3/4 filled with sediment for efficient squeezing.

The steel frames are mounted, on a wooden support, in a domestic chest freezer (13.7 cu.ft ) modified to run between  $-5^{\circ}\text{C}$  and  $10^{\circ}\text{C}$ , with a circulation fan in the lid. The inner walls of the freezer are sprayed with vinyl to prevent corrosion. The hydraulic units can be operated from above without much disturbance of the cool air in the cabinet. (Plate 2)

This system was used on Discovery Cruises 108 and 110. The following general procedure was followed.



### SAMPLE COLLECTION AND STORAGE

Core samples were collected by insertion of a precleaned, butyric core liner (4" dia.) near the centre of a box core (1 foot square cross section). The dead space at the top of the subcore was flushed with nitrogen before capping. These sub-cores were 'dug out' as soon as possible and transferred to the cool cabinet, for temporary storage before sectioning.

### TRANSFER OF SUB-CORE TO SQUEEZERS

The sub-core was sealed into a large glove bag along with the squeezer units. The bag and its contents were thoroughly flushed with nitrogen, then the bag loosely inflated. A PVC piston, machined to fit the liner, was used to extrude the sub-core, which was sectioned into the PVC squeezer units using a plastic spatula. Each squeezer unit was sealed using its piston, then transferred to the frames, in the cooler, for squeezing. In order to reduce the storage time of the core, thereby reducing the risk of oxidation, the next set of sections to be squeezed can be prepared as above in a second set of PVC units and stored in the cool cabinet prior to squeezing.

### SQUEEZING

After allowing time for the units to come to temperature, each one was pressurised to a reading in the range 2000-4000 psi (the reading is dependent on sediment type). The pressure gauges provide a good indication of squeezing efficiency and serve to warn the operator of over-pressure and leakage. The first few ml. of water were discarded to flush the dead volume and the filter assembly, before a plastic syringe (60 ml.) was inserted into the outlet port. The pressure was maintained by further pressurisation at approximately 5-minute intervals. The bulk of the sample (generally 100-200 ml.) was collected within half an hour. The syringes were capped and stored in the cooler. Sub-samples were taken into glass vials for some analyses.

### CONCLUSION

This system is easily operated and maintained and is relatively inexpensive to build. Pore waters can be maintained at in situ temperatures and in an oxygen free environment. It was found that a large volume glove bag was essential for ease of operation and to prevent the bag splitting. However it may be of further benefit to inflate the bag in a perspex chamber in order to give it more structure. The diameter of the sub-core is also important. Initially, 2" diameter tubes were

used, but these compressed the sediment so that an untypical vertical section was obtained. Increasing the surface area to wall thickness ratio by use of the 4" diameter tube, minimised this compression effect. The pressure exerted on the sediments (up to 400 psi) is not thought to affect the pore water chemistry,<sup>23,26-28</sup> but is sufficiently high to provide samples in a relatively short period of time. The PVC units avoid sediment/metal contact but this does not eliminate all possible contamination and careful cleaning is necessary between squeezing operations.<sup>29</sup> The system provides relatively large volumes of pore waters for analysis within the limitations of a shipboard collection system.

#### Acknowledgement

The assistance of the staff of the Engineering Workshop at IOS Wormley is gratefully acknowledged.

## REFERENCES

1. MANGELSDORF, P.C., WILSON, T.R.S. & DANIELL, E., 1969. Potassium enrichments in interstitial waters of Recent marine sediments. Science, N.Y., 165, 171-173.
2. BISCHOFF, J.L., GREER, R.E. & LUISTRO, A.O., 1970, Composition of interstitial waters in marine sediments: temperature of squeezing effect. Science, N.Y., 167 1245-1246.
3. FANNING, K.A. & PILSON, M.E.Q., 1971, Interstitial silica and pH in marine sediments: some effects of sampling procedures. Science, N.Y., 173, 1228-1231.
4. BRAY, J.T., BRICKER, O.P. & TROUP, B.N., 1973 Phosphate in interstitial waters of anoxic sediments: oxidation effects during sampling procedure. Science, N.Y., 180, 1362-1364.
5. TROUP, B.N., BRICKER, O.P. & BRAY, J.T., 1974, Oxidation effect on the analysis of iron in the interstitial water of Recent anoxic sediments. Nature, Lond., 249, (5454), 237-239.
6. BISCHOFF, J.L. & KU, T.L., 1971, Pore fluids of Recent marine sediments: II. Anoxic sediments of 35° to 45° N, Gibraltar to mid-Atlantic Ridge. J. Sedim. Petrol. 41, 1008-1017.
7. EMERY, K.O. & RITTENBERG, S.C., 1952, Early diagenesis in California basin sediments in relation to origin of oil. Bull. Am. Ass. Petrol. Geol., 36, 735-806.
8. KULLENBERG, B., 1952, On the salinity of the water contained in marine sediments. Meddu. oceanogr. Inst. Göteborg, No. 21, Ser. B., 6, (6) 38pp.
9. SWARZENSKI, W.V., 1959, Determination of chloride in water from core samples. Bull. Am. Ass. Petrol. Geol. 43, 1995-1998.
10. POWERS, M.C., 1967, Adjustment of land derived clays to the marine environment. J. Sedim. Petrol., 27, 355-372.
11. RITTENBERG, S.C., EMERY, K.O., HULSEMAN, J., DEGENS, E.T., FAY, R.C., REUTER, J.M., GRADY, J.R., RICHARDSON, S.H. & BRAY, E.E., 1963, Biogeochemistry of sediments in experimental Mohol. J. Sedim. Petrol., 33, 141-172.
12. LUSCZYNSKI, N.J., 1961, Filter-press method of extracting water samples for chloride analysis. U.S. Geological Survey Water-supply paper 1544A, 1-8.

13. SCHOLL, D.W., 1963, Techniques for removing interstitial water from coarse-grained sediments for chemical analysis. *Sedimentology* 2, 156-163.
14. HARTMANN, M., 1965, A apparatus for the recovery of interstitial water from Recent sediments. *Deep-Sea Res.*, 12, 225-226.
15. SIEVER, R., 1962, A squeezer for extracting interstitial waters from Modern sediments. *J. Sedim. Petrol.*, 32, 329-331.
16. PRESLEY, B.J., BROOKS, R.R. & KAPPEL, H.M., 1967 A simple squeezer for the removal of interstitial water from ocean sediments. *J. Mar. Res.* 25, (3), 355-357.
17. REEBURGH, W.S., 1967, An improved interstitial water sampler. *Limnol. Oceanogr.*, 12, (1), 163-165.
18. ROBBINS, J.A., GUSTINIS, J., 1976, A squeezer for efficient extraction of pore water from small volumes of anoxic sediment. *Limnol. Oceanogr.* 21, (6), 905-909.
19. ROZANOV, A.G., MISCHENOKO, V.V. & YASHKICHEV, V.I., 1978, The 'Pneumo Press' - a device for extracting interstitial water. *Oceanology*, 18, (2), 229-231.
20. KRUIKOV, P.A., 1947, Metody vydeleniya pochvenuykh rastvorov (Methods for extracting soil solutions). *Sovremenny metody fizikokhimicheskogo issledovaniya pochv.* (Modern methods for Physico Chemical studies of soils), 2, 3-15 (Moscow).
21. KRUIKOV, P.A. & KOMAROVA, N.A., 1954, On the squeezing out of waters from clay at very high pressures. *Dokl. Akad. Nauk SSSR*, 99, 617-619.
22. SHISHKINA, O.V., 1956, A technique for obtaining ocean interstitial waters and investigation of their composition. *Trudy. Inst. Okeanol.*, 17, 148-176.
23. MANHEIM, F.T. 1966, A hydraulic squeezer for obtaining interstitial water from consolidated and unconsolidated sediments. U.S. Geological Survey, prof. paper 560-C, C256-261.
24. KALIL, E.K. & GOLDHABER, M., 1973, A sediment squeezer for the removal of pore waters without air contact. *J. Sedim. Petrol.*, 43, (2), 553-567.
25. SASSEVILLE, D.R., TAKACS, A.P. & NORTON, S.A., 1974, A large volume interstitial water sediment squeezer for lake sediment. *Limnol. and Oceanogr.*, 19, (6), 1001-1004.
26. BOLT, G.H., 1961, The pressure filtrate of colloidal suspensions - II Experimental data on homionic clays. *Kolloid Z.*, 175, 144-150.

27. SHISHKINA, O.V., 1968, Metody issledoraniya morskikh i okeanicheskikh ilorykh vod (Methods of studying marine and oceanic pore fluids), In Porovye rastvory i metody ikh izucheniya (Interstitial waters and methods of studying them) (edited-Bogomolov, G.V.), 167-177.
28. SAYLES, F.L., 1970, Preliminary geochemistry. In Initial Reports of the Deep Sea Drilling Project, 4, 645-655, U.S. Government Printing Office, Washington, D.C.
29. ROBERTSON, D.E., 1968, Role of contamination in trace element analysis of sea water. Anal. Chem. 40, 1067-1072.

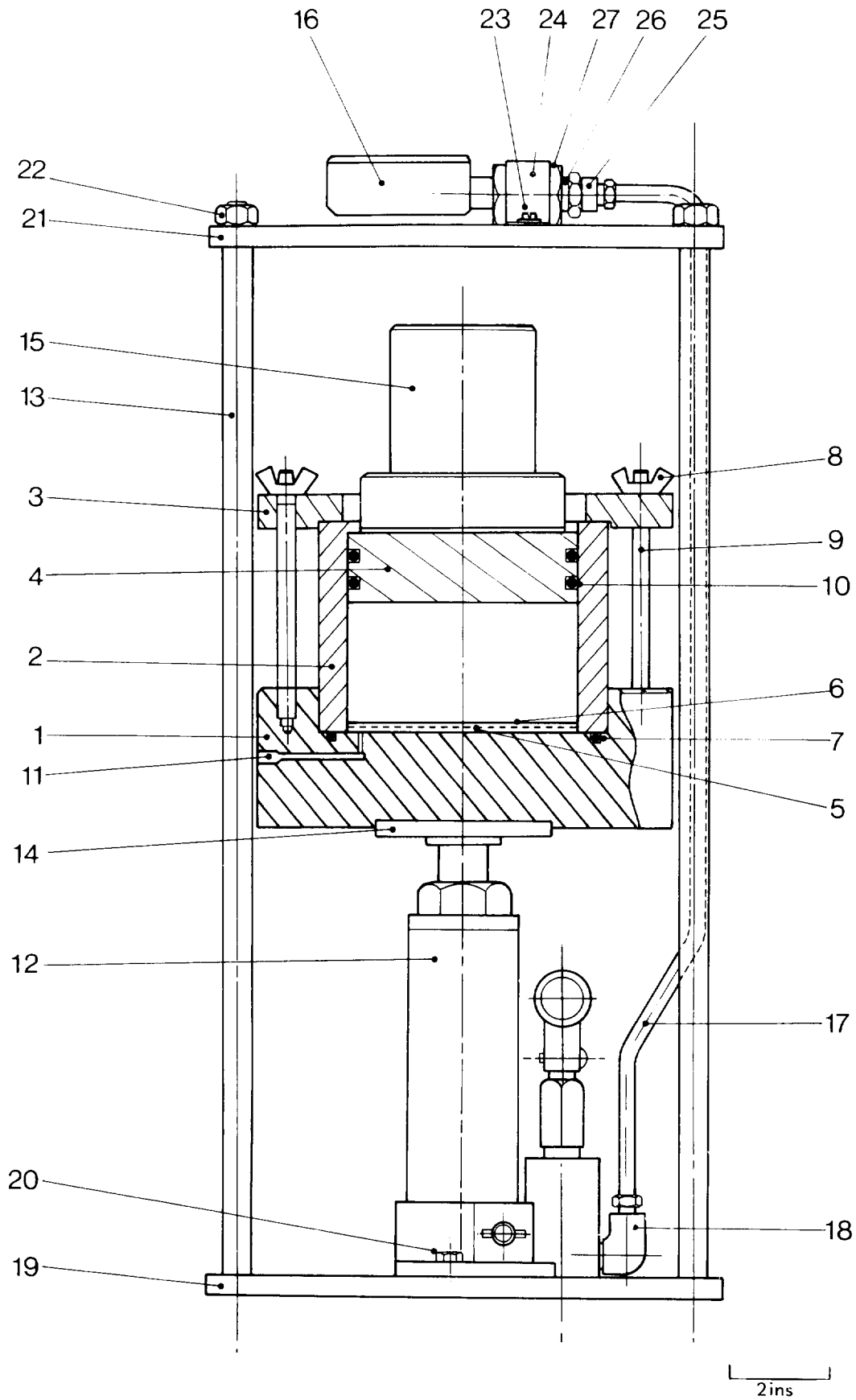


Fig.1 Scale Drawing of Sediment Squeezing Unit.

Table 1 - Components List (see Fig. 1)

| <u>No.</u> | <u>Description</u>         | <u>Size</u>                | <u>Material</u> |
|------------|----------------------------|----------------------------|-----------------|
| 1          | Cylinder Base              | O.D. 7"                    | Rigid PVC       |
| 2          | Cylinder                   | O.D. 4.92"<br>I.D. 3.940"  | Rigid PVC       |
| 3          | Retaining Collar           | O.D. 7"<br>I.D. 4.125"     | Rigid PVC       |
| 4          | Piston                     | O.D. 3.93"                 | Rigid PVC       |
| 5          | Gauze                      | 400µm mesh                 | Nylon (Nitex)   |
| 6          | Filter                     | Whatman 542                | Paper           |
| 7          | 'O' Ring                   | 50-245 Imperial            | Neoprene        |
| 8          | Wing nut                   | 1/4" BSW                   | St. steel       |
| 9          | Studding                   | 1/4" dia.                  | St. steel       |
| 10         | 'O' Rings                  | 71-1000 metric             | Neoprene        |
| 11         | Outlet port                | 3/32" C' bored to<br>5/32" | -               |
| 12         | Hydraulic jack             | 1 1/2 ton                  | -               |
| 13         | Tie rod                    | 3/8" dia.                  | Mild steel      |
| 14         | Support plate              | 3" dia.                    | Mild steel      |
| 15         | Thrust pad                 | 3.5" dia. at piston        | Rigid PVC       |
| 16         | Pressure gauge             | 0-5000 psi 60 mm dia.      | -               |
| 17         | Hydraulic pipe             | 6 mm dia.                  | 1/2 hard copper |
| 18         | Elbow                      | 90°                        | Plain brass     |
| 19         | Base plate                 | 8.75" x 8.75" x 0.375"     | Mild steel      |
| 20         | Hexagon Head Screw         | 1/4" BSF x 1/2"            | St. steel       |
| 21         | Top Plate                  | 8.75" x 8.75" x 0.375"     | Mild steel      |
| 22         | Hexagon nut                | 3/8" BSW                   | Mild steel      |
| 23         | Ch. Hd. M/C screw & washer | 2BA                        | Plain brass     |
| 24         | Connector clamp            | 25 wg.                     | Plain brass     |
| 25         | Male adaptor               | Straight                   | Plain brass     |
| 26         | Sealing Washer             | For 1/8" BSP thread        | Aluminium       |
| 27         | Pressure gauge connector   | 1" A/F Hexagon             | Plain brass     |

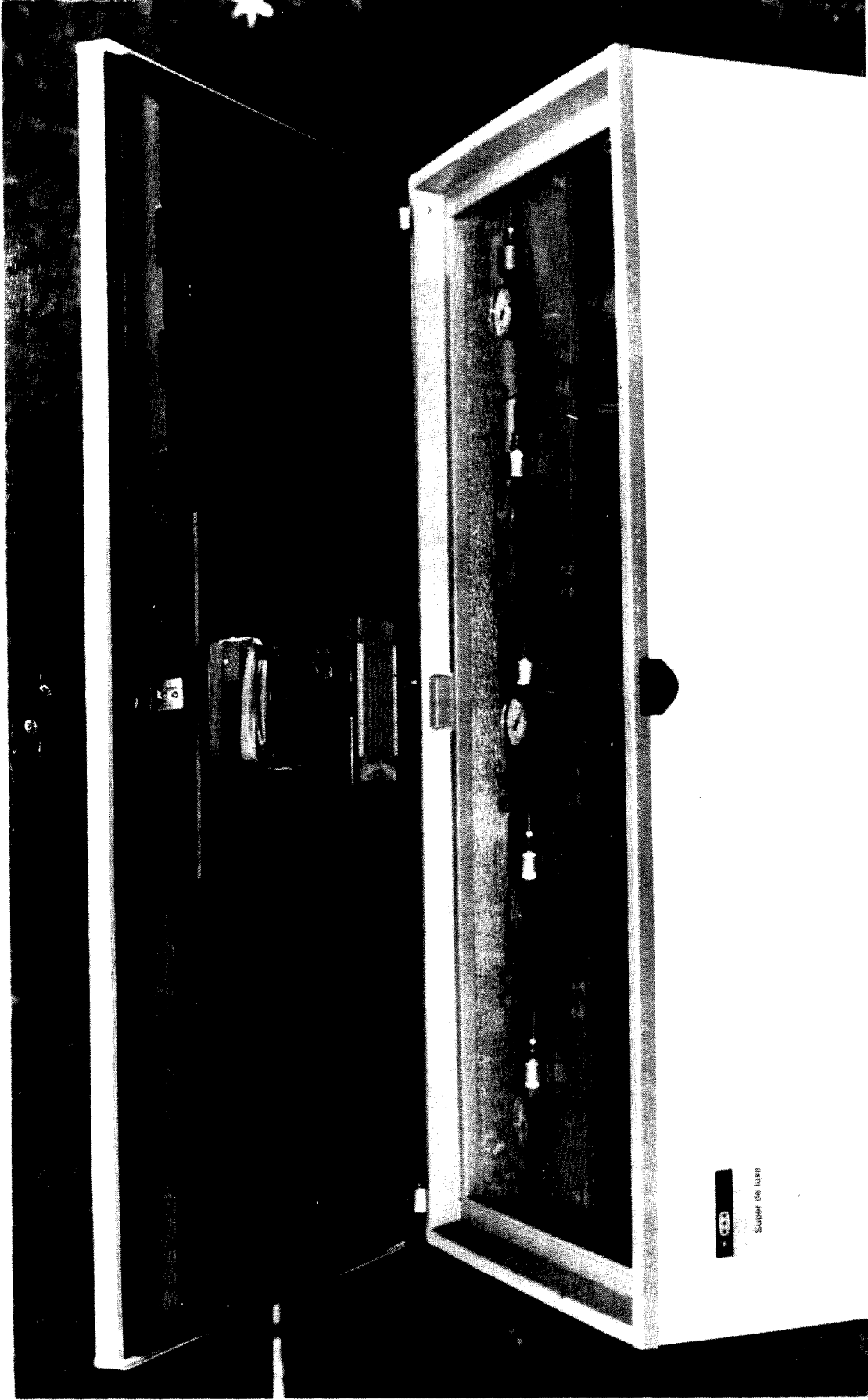


Plate 1

SQUEEZING UNITS FIXED IN COOLER CABINET



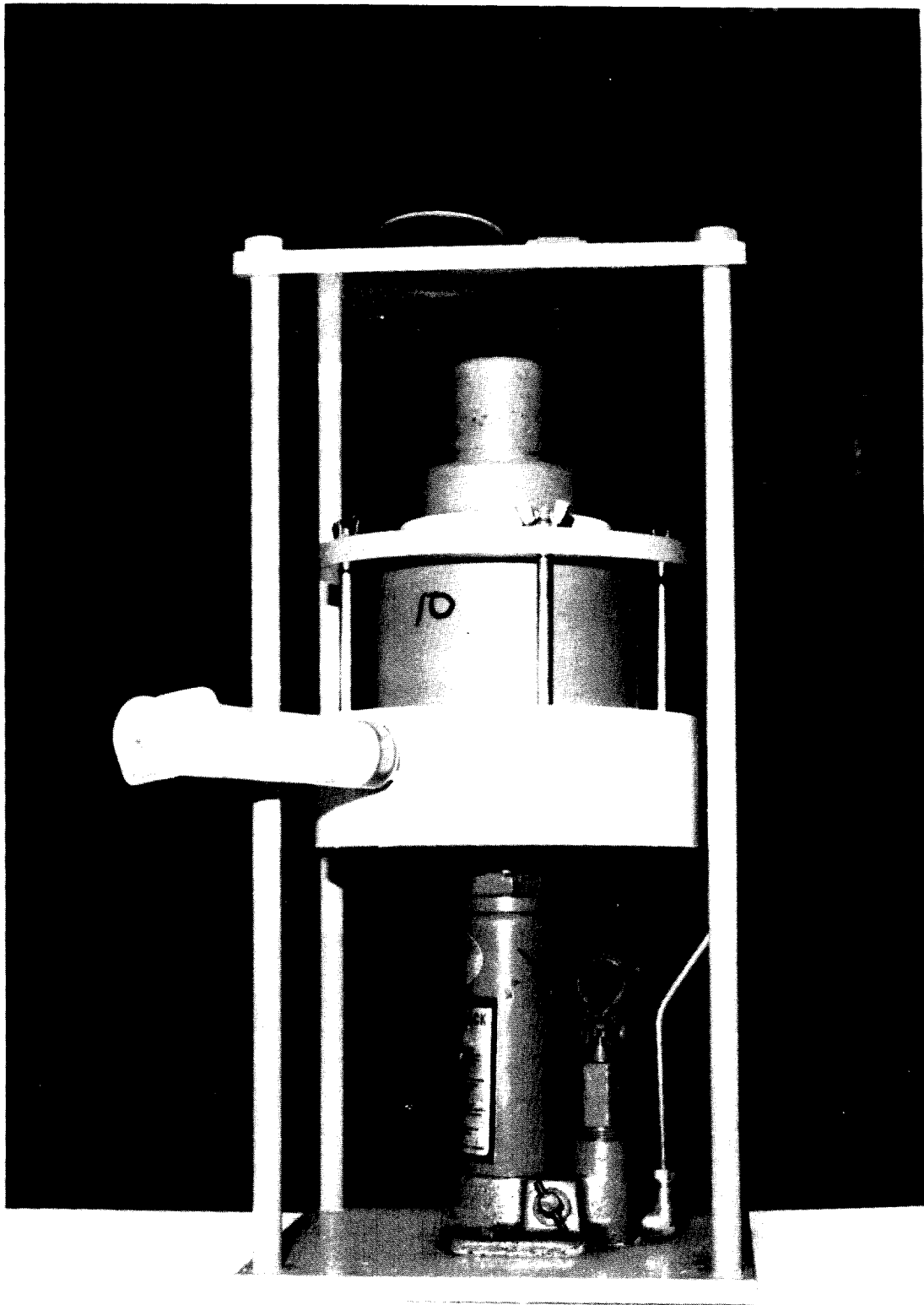


Plate 2

SEDIMENT SQUEEZING UNIT COMPLETE