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**In 1982, the name of the International Superphosphate Manufacturers' Associations (ISMA) was changed to International Fertilizer Industry Association (IFA).*

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LTE/70/21

A METHOD OF DETERMINING THE CAKING TENDENCY OF A FERTILISER AND THE EFFECTIVENESS OF ANTICAKING AGENTS

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INTRODUCTION

It is well known that mineral fertilisers tend, to a greater or lesser extent, to cake during storage, according to their moisture content. This caking of fertiliser granules into large agglomerates, tending to form solid and compact lumps, is due principally to a recrystallisation of salts which forms bridges between the granules, binding them together.

To avoid this phenomenon, the finished product is covered by suitable substances called "anti-caking agents".

METHOD AND APPARATUS

Into a small impermeable PVC bag (dimensions 360 x 130 mm, with PVC 0.3 mm thick) there are introduced, one after the other, the following :

- a thick card disk, 75 mm diameter.
- about one kg of the granulated product to be tested.
- another thick card disk, identical to the first.

After having forced out the excess air, the top end of the bag is sealed to avoid the fertiliser coming into contact with the outside environment.

Then the prepared bags are hung on metal rails, which are located inside an impervious cylinder (figures 1 and 2).

At Priolo two pieces of apparatus are available for these trials : one of about 8 m^3 for pressure tests at the ambient temperature, the other of about 3 m^3, equipped with electrical resistance heating and a thermostat, and linked to a pressuri-

sation system (compressed air system), in order to allow trials at temperatures above the ambient temperature to be carried out.

The product in the bags in the cylinder is submitted, by means of pressurisation, to an established pressure, which is of the order of 0.7 kg/cm^2. Generally, for trials at different temperatures, two heat levels, 25° C and 45° C, are chosen.

The capacity of the cylinder with the thermostat is about 320 bags ; the ambient temperature cylinder has a capacity of approximately 1,000 bags. The duration of the treatment varies from 10 to 40 days. After the caking test, the product in each bag is in the form of a solid cylinder with the following dimensions : Ø = 75 mm ; h = 150 mm (figure 3).

These cylinders act as test-samples for evaluation of the breaking load and tendency to disaggregation.

The fertiliser samples are submitted to the tests in absolutely impermeable bags in order that the caking determined in the test-samples should be due entirely to the nature of the anti-caking agent, to the product and to the moisture content of the latter, and not to external conditions.

After having carried out the caking tests, the breaking load is determined by submitting the whole test-sample (and not only a part of it) to increasing pressures, up to the breaking up or disintegration of the product).

The breaking resistance test is carried out using a dynamometer (figures 4, 5 and 6) composed of a screw press actuated by hand or electricity, and of a manometer recorder.

15 sampled test-samples (bags) are made up for the test. After each predetermined interval (usually after 10, 20, 30 and 40 days) 3 test-samples are taken, of which 2 are for the determination, in duplicate, of the breaking load and the third for determination, again in duplicate, of the disaggregation index. Three of these 15 test-samples are kept as a reserve.

VALUES DETERMINING THE TENDENCY TO CAKING

The effectiveness of the anti-caking treatment is estimated by evaluating the resistance to breaking by compression of the test-samples submitted to the caking trials, and measured according to the pressure necessary (in kg/cm^2) to bring about the breaking.

The evaluation of the degree of caking of a fertiliser would, however, be incomplete if it were limited to the measurement of breaking load only. We have, therefore, considered it advisable to use a second measure for expressing the degree of cohesion of the granules or, conversely, the tendency of the product to disaggregation.

By "disaggregation capacity" is understood the tendency of agglomerated products to disaggregate into granules when they are submitted to given determined forces. This capacity gives an indication in principle, but one indicative of the practical outcome, of the recoverability of a caked product.

The measure of the disaggregation capacity consists of a determination of the time, in seconds, which is necessary for about 450 gms of product to disaggregate or to break down again into separate granules.

The same apparatus as that employed for granulometric determination is normally used : an apparatus with automatic mechanical sieves in which the sieves, suitably blocked, are subjected to a movement which reproduces manual action.

This apparatus has an automatic time-switch, allowing absolutely reproducible and comparable runs.

About 450 gms of the product - i.e. approximately half a test-sample, divided into four slices - are placed on a sieve with 5 mm mesh.

5 steel balls with a 20 mm diameter are added and the apparatus is put into motion. The apparatus is stopped when the disaggregation of the product is finished, or, in any case, after a maximum of 60 seconds.

MEASURE OF THE TENDENCY TO CAKING

The tendency to caking "I" is determined by the following expression :

$$I = i_c + i_s$$

where I = caking index

i_c = breaking load index

i_s = disaggregation index

It may be assumed that $i_c = 50$ when the breaking load reaches 3 kg/cm^2 (values over 3 kg/cm^2 are not of interest). Thus we have :

$$i_c = a. 16.66 \quad (1)$$

where a = breaking load in kg/cm^2.

Concerning the disaggregation index, one may also assume a value of 50 when the tube is unchanged after 60 seconds (which is produced by breaking loads about 3 or greater than 3) and, on the other hand, equal to zero when the test-sample shows no tendency to caking, and equal to 25 when the test-sample is completely disaggregated after 60 seconds.

In these conditions, the mathematical expression satisfying the three above conditions is as follows :

$$i_s = 25 - (60 \text{ sec} - t \text{ sec}) . 0.4166 + \frac{\% \text{ not disaggregated}}{4} \quad (2)$$

and (1) and (2) give

$$I = a . 16.66 + 25 - (60 \text{ sec} - t \text{ sec}) . 0.4166 + \frac{\% \text{ not disagg'd}}{4}$$

The caking index is then always between 0 and 100.

Experience enables us to compile the following practical scale of values (a scale which obviously is not rigorous but which, nevertheless, is sufficiently precise) :

I	between	0	and	10	= excellent
I	"	10	"	30	= good
I	"	30	"	50	= moderate
I	"	50	"	70	= bad
I	"	70	"	100	= very bad

PROTECTION INDEX

By protection index (I_p) is understood the degree of protection (as a percentage) against caking of a fertiliser treated with anti-caking agent relative to a control sample of the same, untreated, fertiliser.

The index results in the following expression :

$$I_p = 100 - \frac{I \text{ treated product}}{I \text{ untreated product}} . 100$$

CONCLUSION

The method of evaluating the caking index, as described in this paper, provides results with a very satisfactory degree of reproducibility.

In view of the simplicity of the apparatus used, the capacity of the cylinders under pressure can be chosen in such a way as to allow a considerable number of tests to be

carried out simultaneously and economically, with the following advantages :

1) Simultaneous carrying out of trials using different anti-caking agents and/or different fertilisers, and also a series of test-samples with a range of percentages for each type of anti-caking agent.

2) The possibility of obtaining, in the reduced time interval of 10 days, the first indicative results and in the space of 40 days the final results (after the first interval of 40 days, the index stays approximately constant).

3) The possibility of evaluating, using several cylinders at different temperatures or using one cylinder only with successive tests at different temperatures, the behaviour of a given product in relation to temperature.

List of Figures and Drawings

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FIG.1

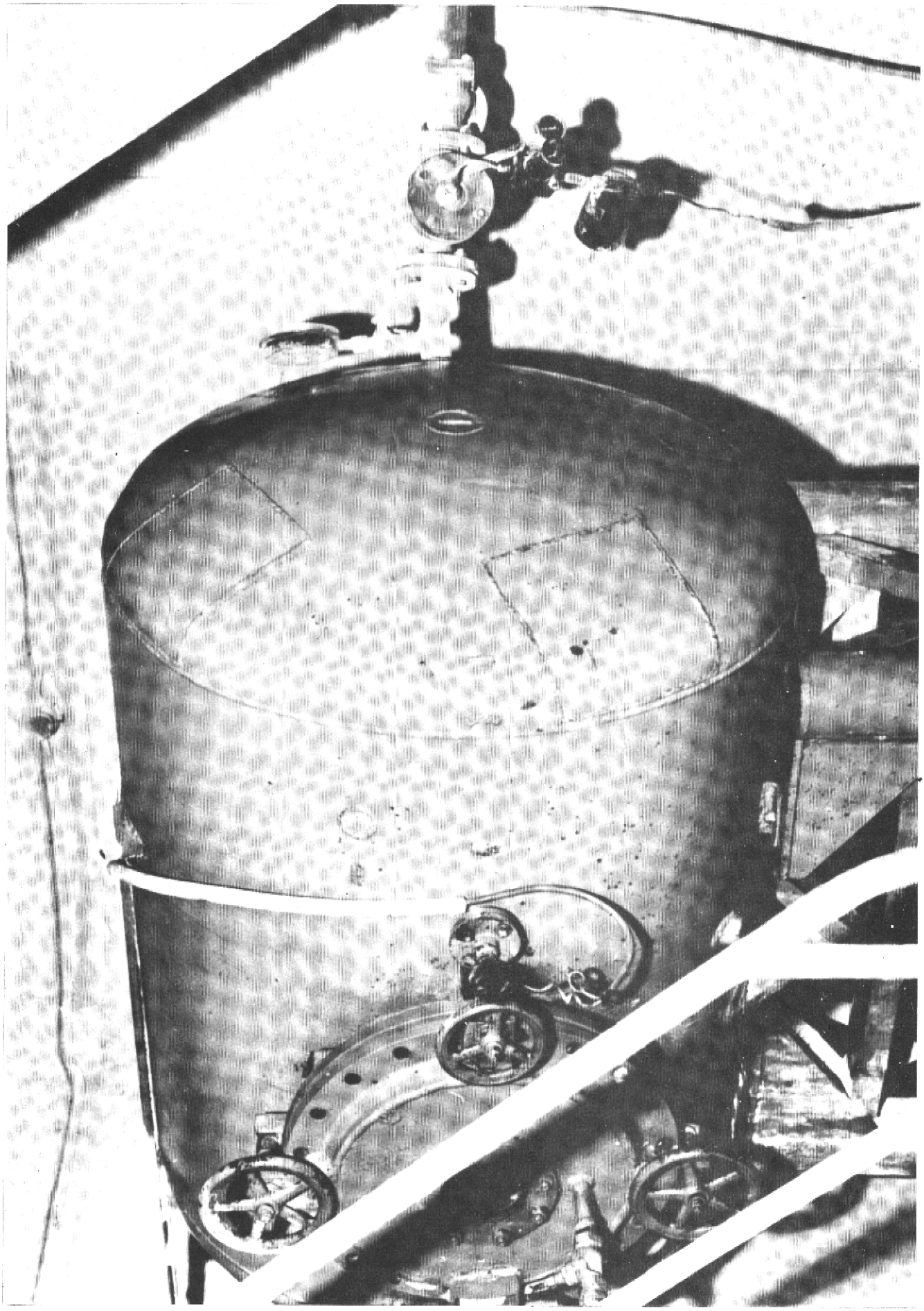


FIG. 2

FIG. 3

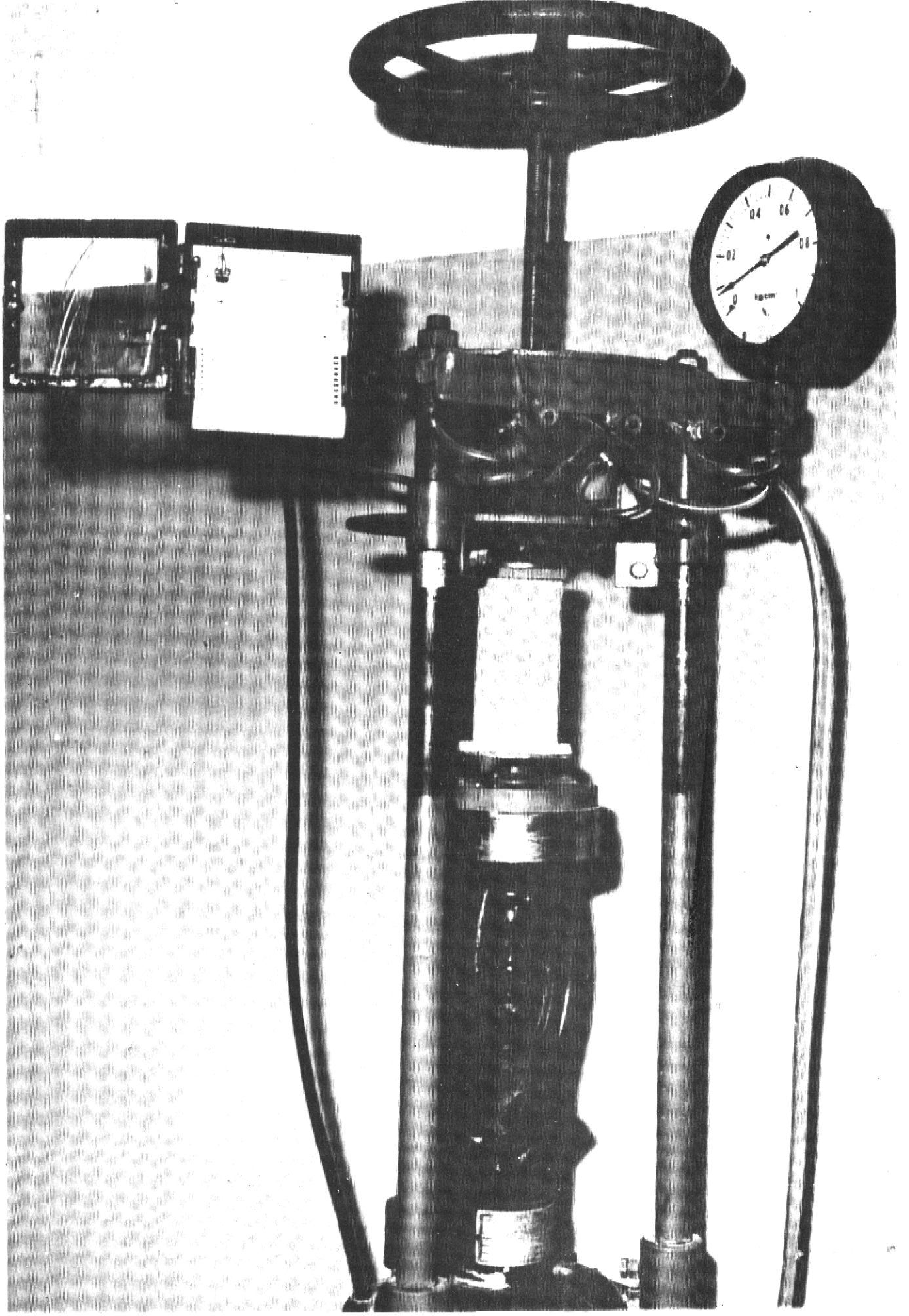


FIG. 4

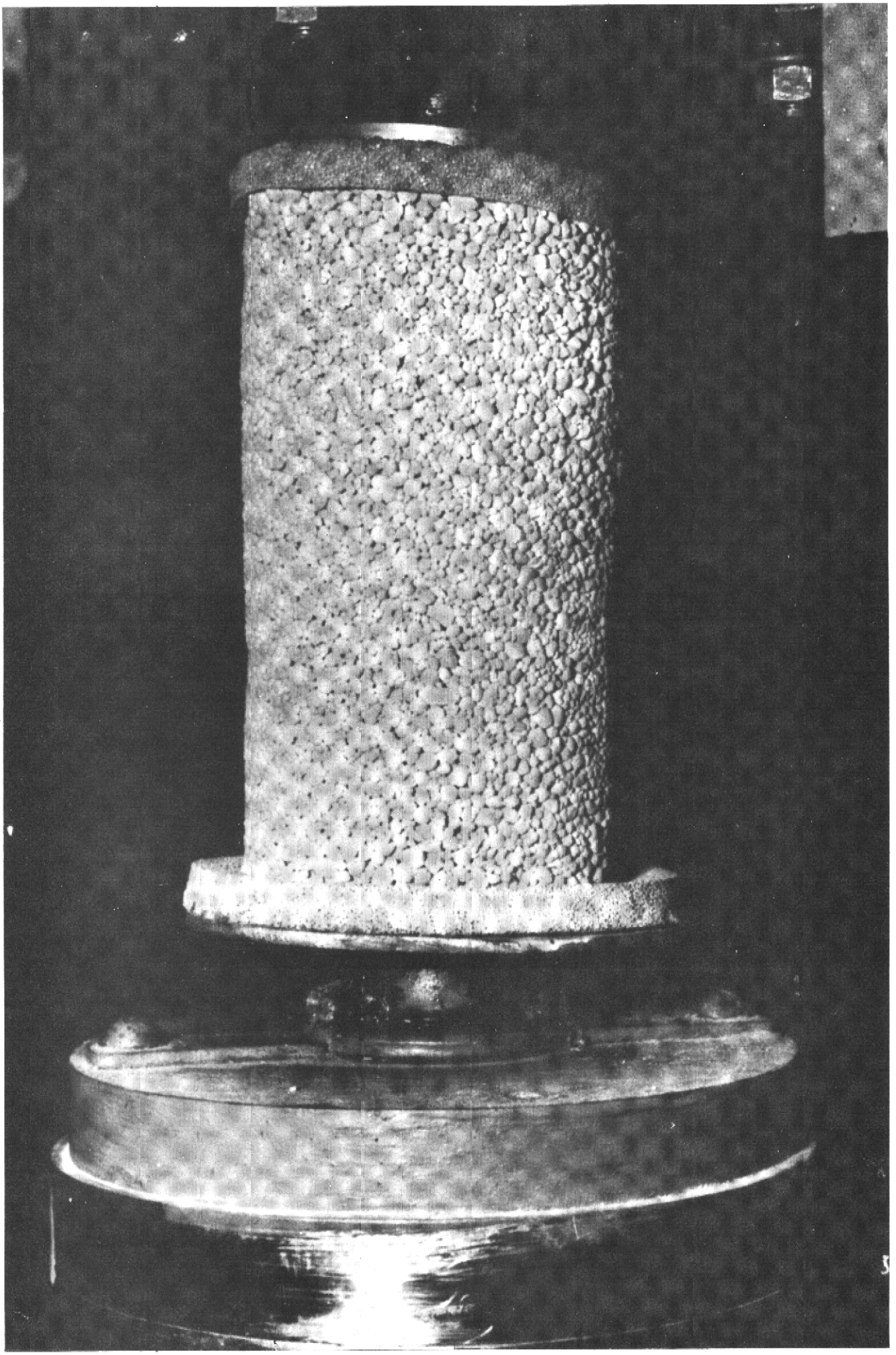


FIG.5

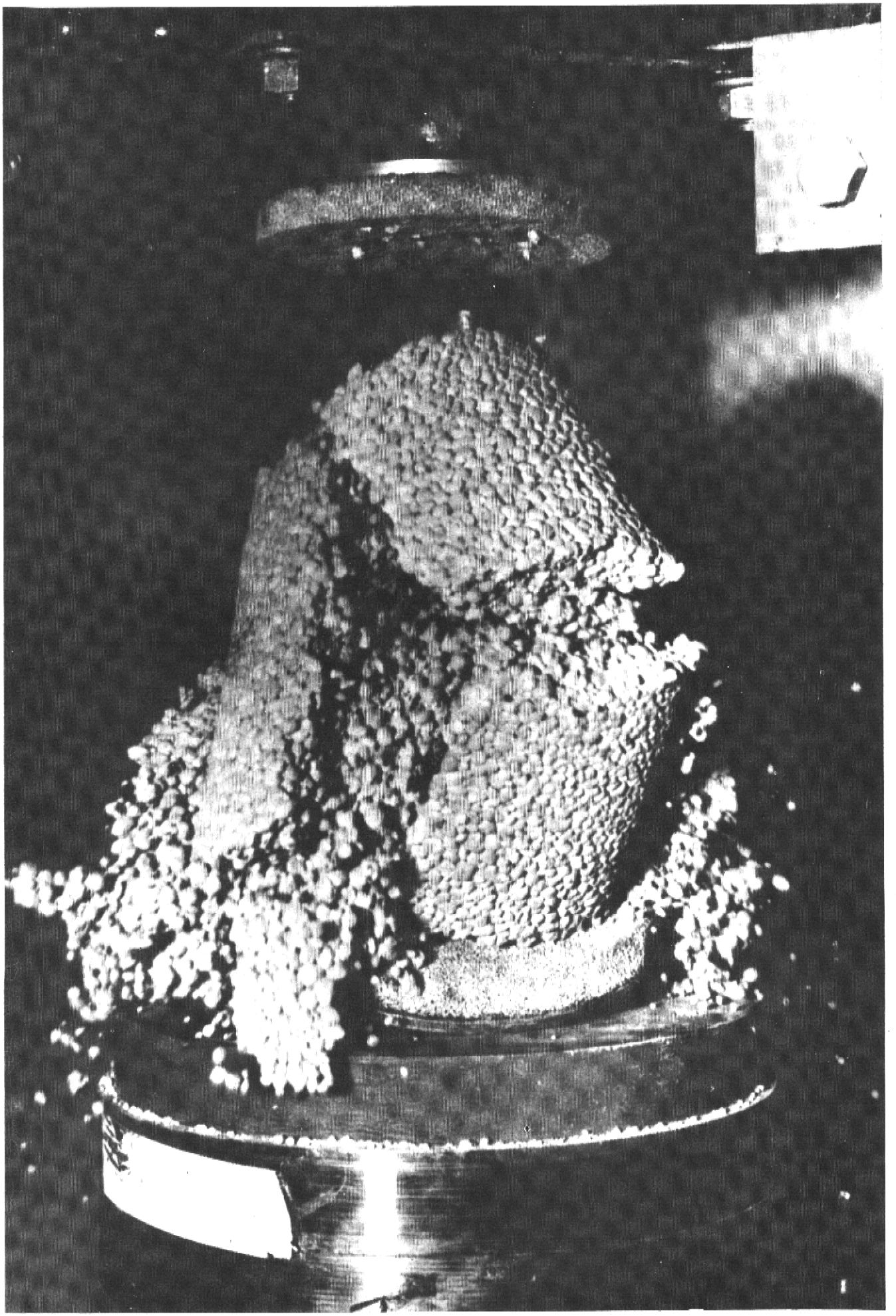
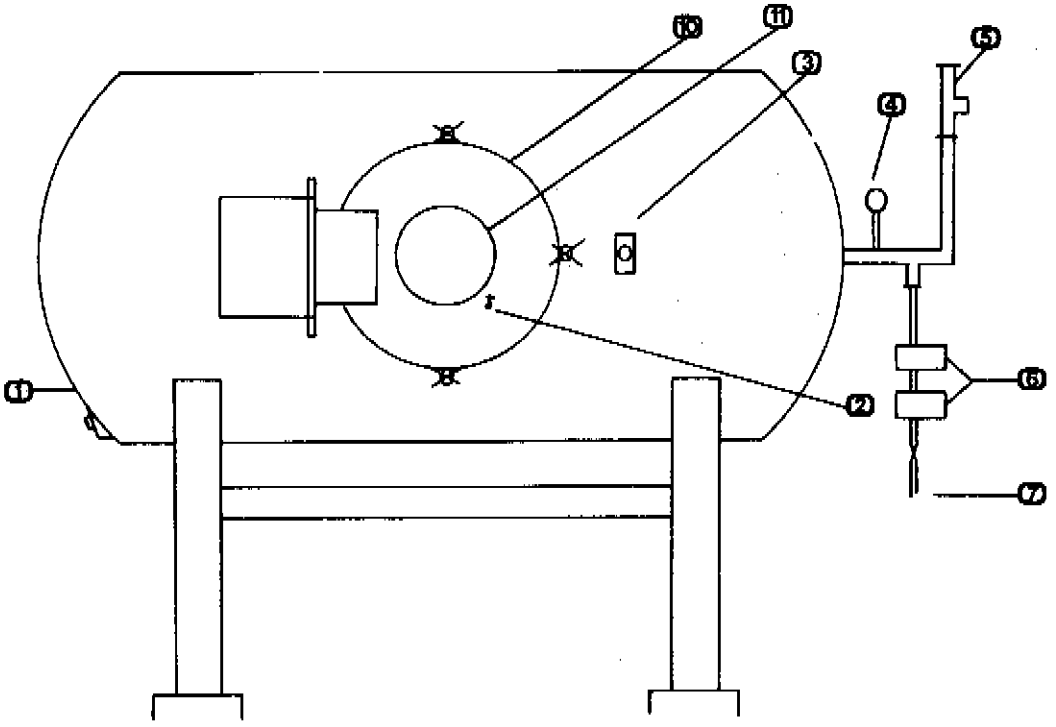


FIG. 6



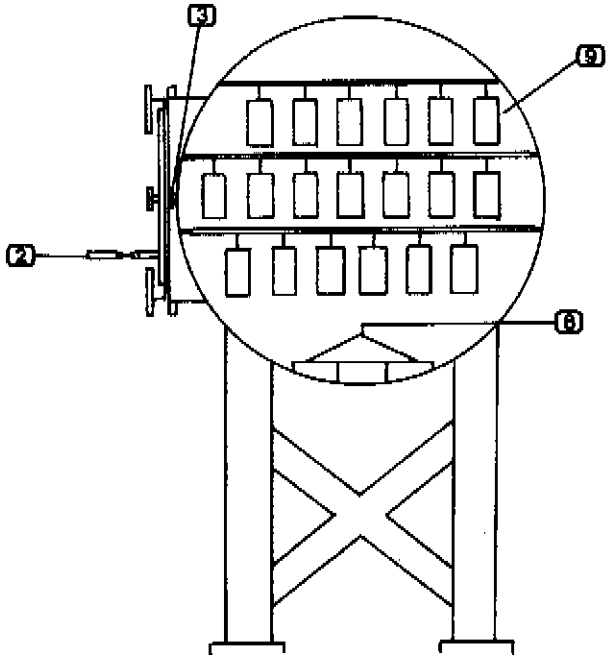


Schéma n°1

PTF/LTE/70/21
APPARATUS FOR CAKING TESTS

APPAREIL D'ESSAI
DE PRISE EN MASSE

1 - Internal light connection
2 - Vent
3 - Thermostat
4 - Pressure gauge
5 - Safety valve
6 - Pressure regulator
7 - Air
8 - Electrical wires
9 - Testing samples
10 - Manhole
11 - Ispection hole

1 - Eclairage intérieur
2 - Event
3 - Thermostat
4 - Jauge de pression
5 - Soupape de sécurité
6 - Régulateur de pression
7 - Air
8 - Fils électriques
9 - Echantillons d'essai
10 - Bouche d'accès
11 - Orifice d' inspection

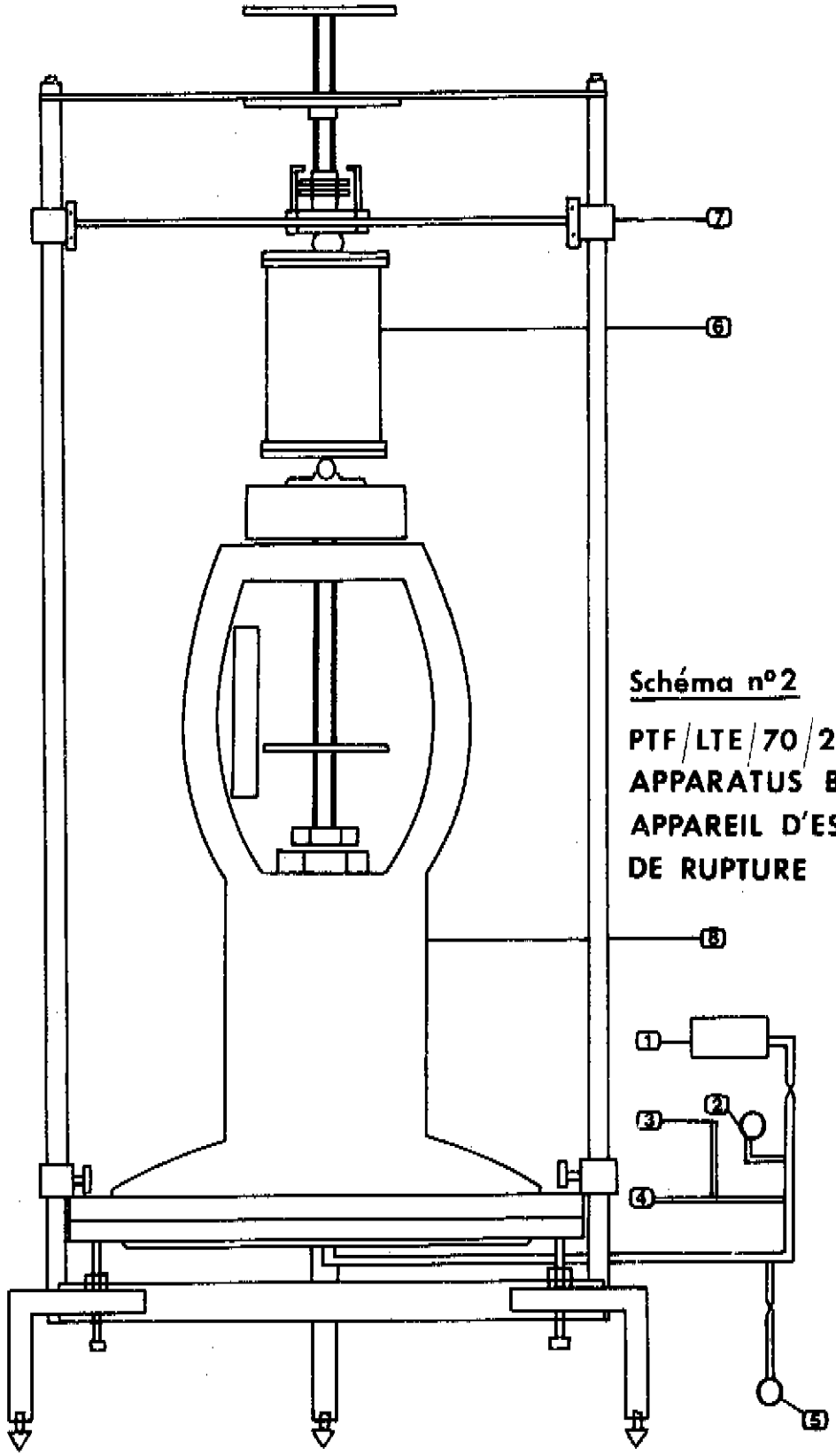


Schéma n°2

PTF/LTE/70/21
APPARATUS BREAKING LOAD
APPAREIL D'ESSAI DE LA CHARGE DE RUPTURE

1 = Pressure recorder
2 = Pressure gauge
3 = Vent
4 = Vent
5 = Pump
6 = Testing sample
7 = Slide
8 = Head of pneumatic valve

1 = Enregistreur de pression
2 = Jauge de pression
3 = Event
4 = Event
5 = Pompe
6 = Echantillon d'essai
7 = Glissière
8 = Tête de la valve pneumatique