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THE MEASUREMENT OF SOME STORAGE CHARACTERISTICS OF GRANULAR FERTILIZERS

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1. SUMMARY

A laboratory technique to measure the caking characteristics of granular fertilizers is described. The method involves packing fertilizer inside a thin rubber sheath, closed at the top by a hard disc and at the bottom by a hard base, through which there is a connection to atmosphere. The specimens are stored at constant temperature under a fixed load, applied by means of compressed air, to simulate the conditions in a pile of sacks or in a bulk heap. After the storage period the specimens are subjected to a compression test to measure the caking intensity. Good correlation has been shown with experiments made on the large scale in the factory.

2. INTRODUCTION

Caking has always been a major problem in the fertilizer industry. It was expected that the introduction of granulation would alleviate the problem, but the improvements achieved have tended to be partly offset in recent years by the introduction of more highly concentrated fertilizers, containing a large proportion of water-soluble constituents.

Large-scale experiments in the factory to study caking are expensive and difficult to carry out because of the large number of variables involved. For this reason several workers have developed techniques to carry out experiments in the laboratory. Two typical examples are those used at the United States Department of Agriculture, described by Adams and Ross (1), and at the Chemical Research Laboratory, Teddington, England described by Davies, Ditcham and Greaves (2).

The procedure used in the laboratory normally involves a period of storage under load to form the 'cake', followed by a compression test to measure the intensity of caking. In the techniques already referred to the load is applied to the top of the fertilizer, contained in a metal cylinder. This method has the disadvantage that an equal pressure is not applied throughout the sample of fertilizer. The height of the cylinder is made less than the diameter to reduce this effect, but this in turn causes inaccuracies in the compression test because, when the ratio of height to diameter is

with the aid of the mild steel tube (Figure 1E). The top disc, like the base, was originally of mild steel, but this also is now of either stainless steel or polyvinyl chloride. Another modification is the groove in the top disc to locate the 'O' ring.

The completed specimen, ready for the storage test, is shown in Figures 1G and 2b. The rubber sleeves were originally of natural rubber latex but Neoprene latex is now used to withstand chemical attack by the fertilizer, and also because of its superior properties for long periods of storage at 35°C. Natural rubber was found completely unsatisfactory for a limited number of tests carried out at 45°C.

5. THE STORAGE TEST

The completed specimens are connected by rubber pressure tubing to a copper manifold which accommodates 10 specimens and is shown in Figure 3. In the first experiments 10 specimens were used for each test, but a statistical analysis of the results over a period of 3 years showed that 5 specimens would be sufficient. In one hundred experiments recently carried out, the compression strengths of the five specimens were within ± 13% of the mean value.

The manifold is placed in a closed cylindrical container to enable air pressure to be applied to the specimens. In the original apparatus the cylinders were constructed of Perspex, which, while satisfactory at 25°C and a pressure of 3 p.s.i., developed leaks in cemented joints when used for tests at 35°C and a pressure of 6 p.s.i. The cylinders are now constructed from 2 foot lengths of 6 inches diameter Q.V.F. glass pipeline, each end being closed by a disc of Perspex ½ inch in thickness. The ends are held in position by tie rods which extend the length of the cylinder. Two connections are provided at the front of the cylinder, one being used for the application of the compressed air and the other for connection of the manifold to the atmosphere. The front of the cylinder is easily removed for either insertion or withdrawal of a manifold by removing six wing nuts. The present practice is to accommodate two tests each of five specimens on one manifold. 112 cylinders are in use, which enables 224 tests to be carried out at a given time.

In the original apparatus the pressure was indicated and controlled by mercury manometers fitted with platinum contacts. This arrangement has now been replaced by a supply of air from a compressor at 40 p.s.i. which passes through reducing valves. Experiments are now carried out at 6 p.s.i., whereas the earlier work was all limited to 3 p.s.i.

Another modification is the fitting of bubblers containing saturated salt solutions in the air supply lines to the cylinders. The object is to control the humidity of the air such that any leakage through the rubber sleeves to the fertilizer does not cause change in moisture content of the samples. The humidity of the air is controlled to a value depending on the chemical composition of the fertilizer under test.

The constant temperature room in which the tests are carried out is at 35°C whereas the first experiments were at 25°C. Specimens are stored for periods ranging from one week to six months, this range covering the usual storage periods of bagged fertilizers in commercial practice. In a given test, normally, 20 or 25 specimens are used, batches, each of five specimens, being removed at intervals over a six-months period in order to study the effect of time on the storage properties.

6. THE COMPRESSION TEST

After the required storage period the specimens are removed from the manifold and the compressive strengths measured individually. In the test apparatus the pressure applied to the base of the specimen is gradually increased and at a certain value the specimen shears as shown in Figure 5. In the original apparatus the pressure was applied mechanically by springs of known spring factor. This apparatus is suitable for compressive strengths up to 30 p.s.i. but not sufficiently robust for higher values. The introduction of more concentrated fertilizers, together with the increases in temperature and pressure during the storage period, has resulted in strengths considerably higher than 30 p.s.i. being encountered. Another disadvantage of the mechanical apparatus is that, for specimens of unknown compressive strength, it is necessary to change the spring at least once and perhaps twice during the test, and each time this necessitates the release and then re-application of the pressure; results obtained in this way have a low level of accuracy. The compression test apparatus now in use and shown in Figures 6 and 7 was designed and constructed in our own workshops. The stress is applied to the base of the specimens by means of compressed air instead of by the mechanical compression of a spring as in the original apparatus. The maximum pressure which can be applied is 100 p.s.i. During a test the air pressure is increased slowly by opening the fine control valve fitted to the apparatus. The pressure is recorded on three dial gauges, each covering a section of the range, to enable accurate readings to be made. For the measurement of "blank" specimens and also for pressures up to 10 p.s.i., where the breaking point is not well-defined, a separate support is used for the specimens. Compression of the specimen closes an electrical circuit and an indicator is illuminated at the breaking point.

Three examples of the failure of specimens are shown in Figure 5. Figure 5a shows the type of failure to be expected with caked material and Figure 5b that with free-flowing granular fertilizer. Figure 5c shows that specimens of small ratio of height to diameter should not be used, since the plane of failure does not completely cut the circumference of the specimen.

7. PRE-TREATMENT OF SAMPLES

Often, the tendency of a fertilizer to cake is greatest during the first few days after its production. If such a fertilizer is stored during that time, e.g. in a bulk heap or as a bottled sample, and then disturbed, its subsequent caking is less. It is therefore the practice to commence the laboratory storage tests on the day of manufacture, whenever this is practicable.

The size range of the fertilizer used in the tests is 1/16 - 3/16 inches. No effect on the caking tendency has been shown by variation in granule size within this range.

8. RESULTS

Typical results of storage tests for an NPK compound fertilizer are shown in Figures 8 and 9. The moisture contents of the fertilizer are indicated.

A fertilizer is considered to possess free-flowing characteristics if the breaking strength after 6 months storage at 35°C and 6 p.s.i. does not exceed 10 p.s.i.

9. ACKNOWLEDGEMENTS

The late Dr. A.L. Whynes was responsible for the development of the apparatus and the earlier experimental work.

Thanks are expressed to the Directors of Fisons Fertilizers Ltd., for permission to publish this paper.

10. REFERENCES

(1) Adams, J.R., and Ross W.H., Ind. Eng. Chem. 33, 121 (1941).

(2) Davies, C.R., Ditcham, J.R., and Greaves, W. Fertiliser Society (London) Proceedings No.5 (1949)

(3) Dee, T.P. and Whynes, A.L., J. Sci. Food Agr. 8, 577 (1957)

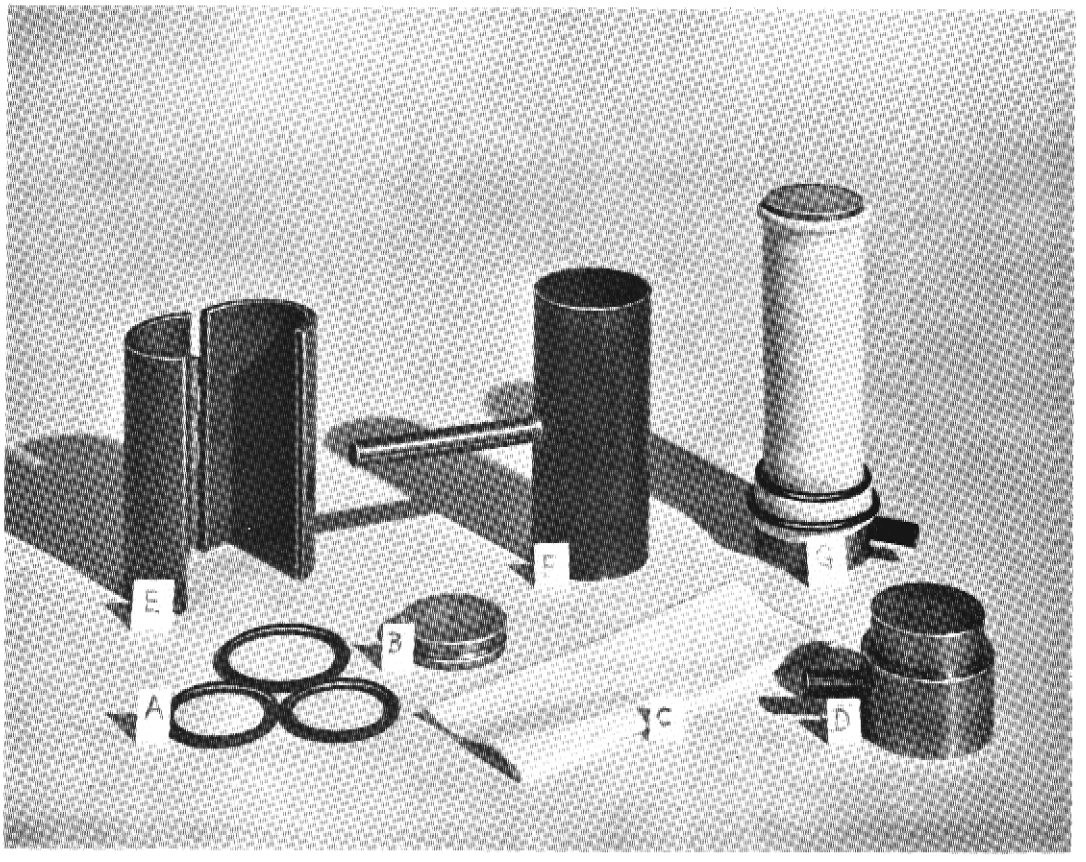

Figure 1.
Components for the Preparation of a Specimen.

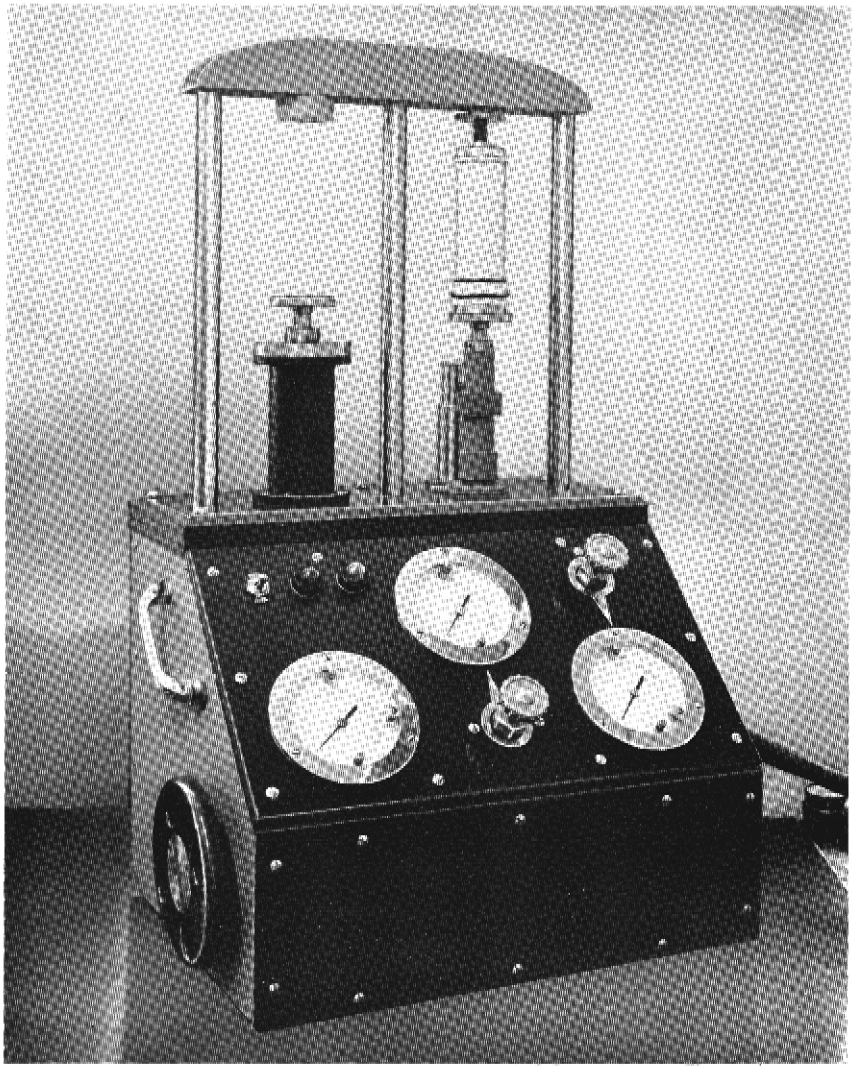

Figure 6.
The Compression Apparatus with a Specimen inserted.

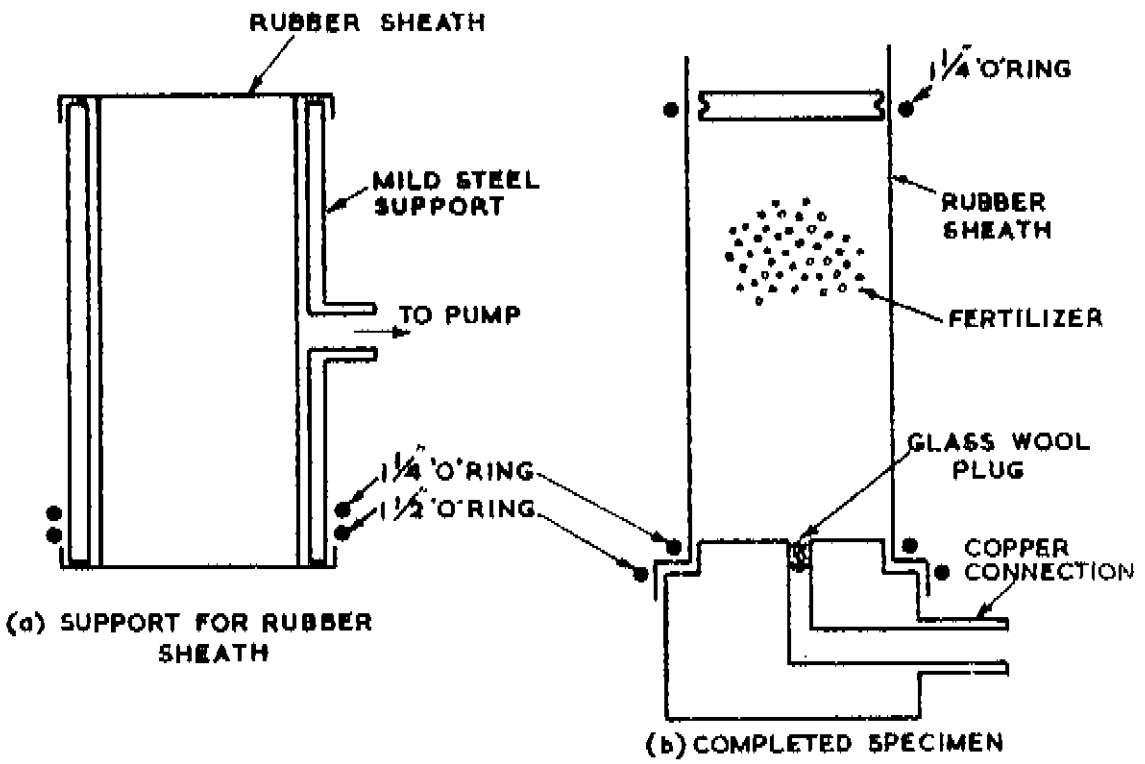

FIGURE 2.

PREPARATION OF A SPECIMEN.

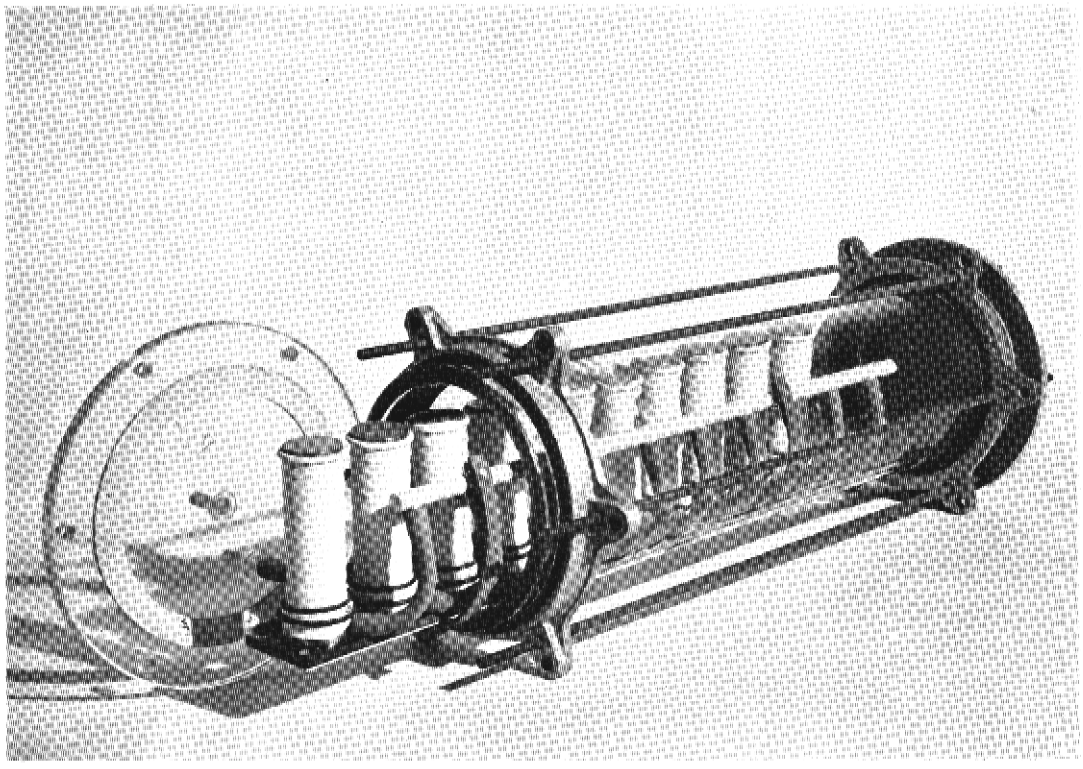

Figure 3.
Components of a Cylinder including a Manifold.

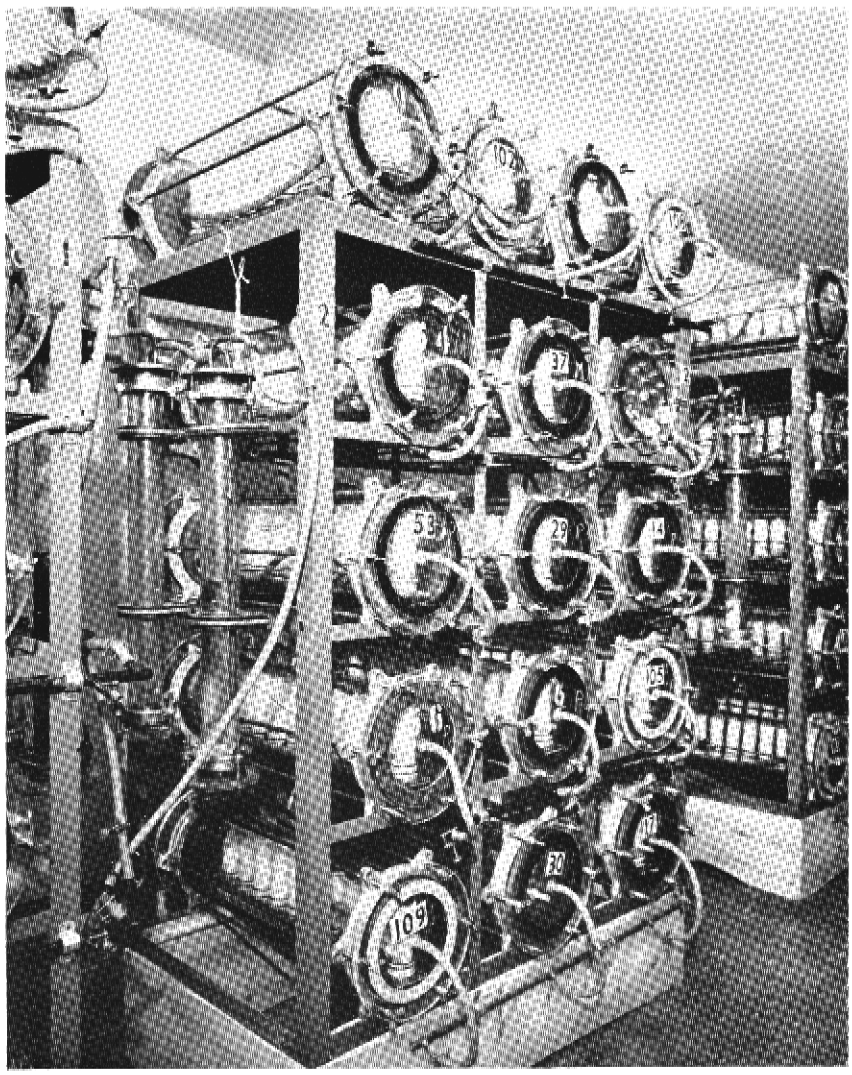

Figure 4.
Arrangement of Cylinders in a Frame.

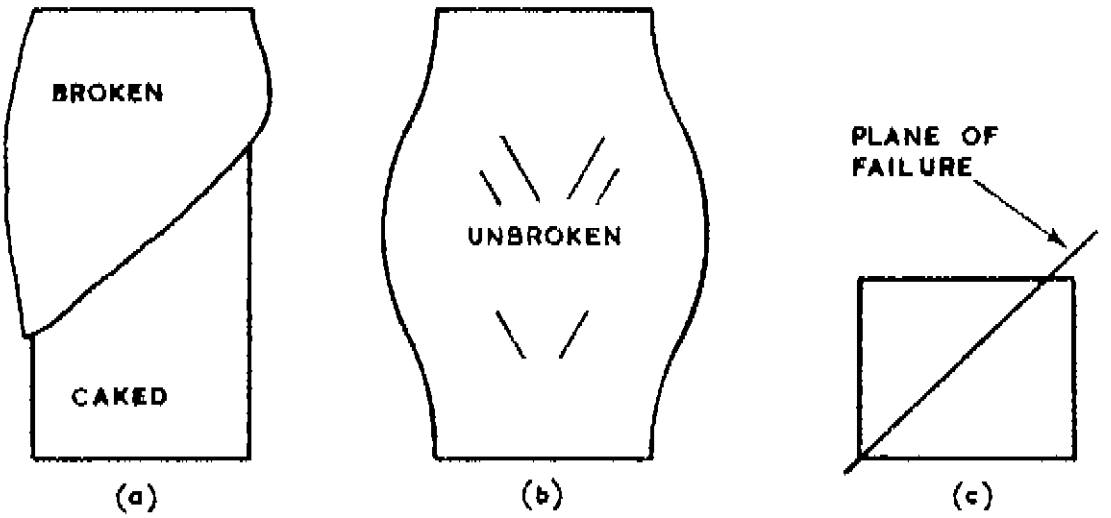

FIGURE 5.

TYPES OF FAILURE OF SPECIMENS IN THE COMPRESSION TEST.

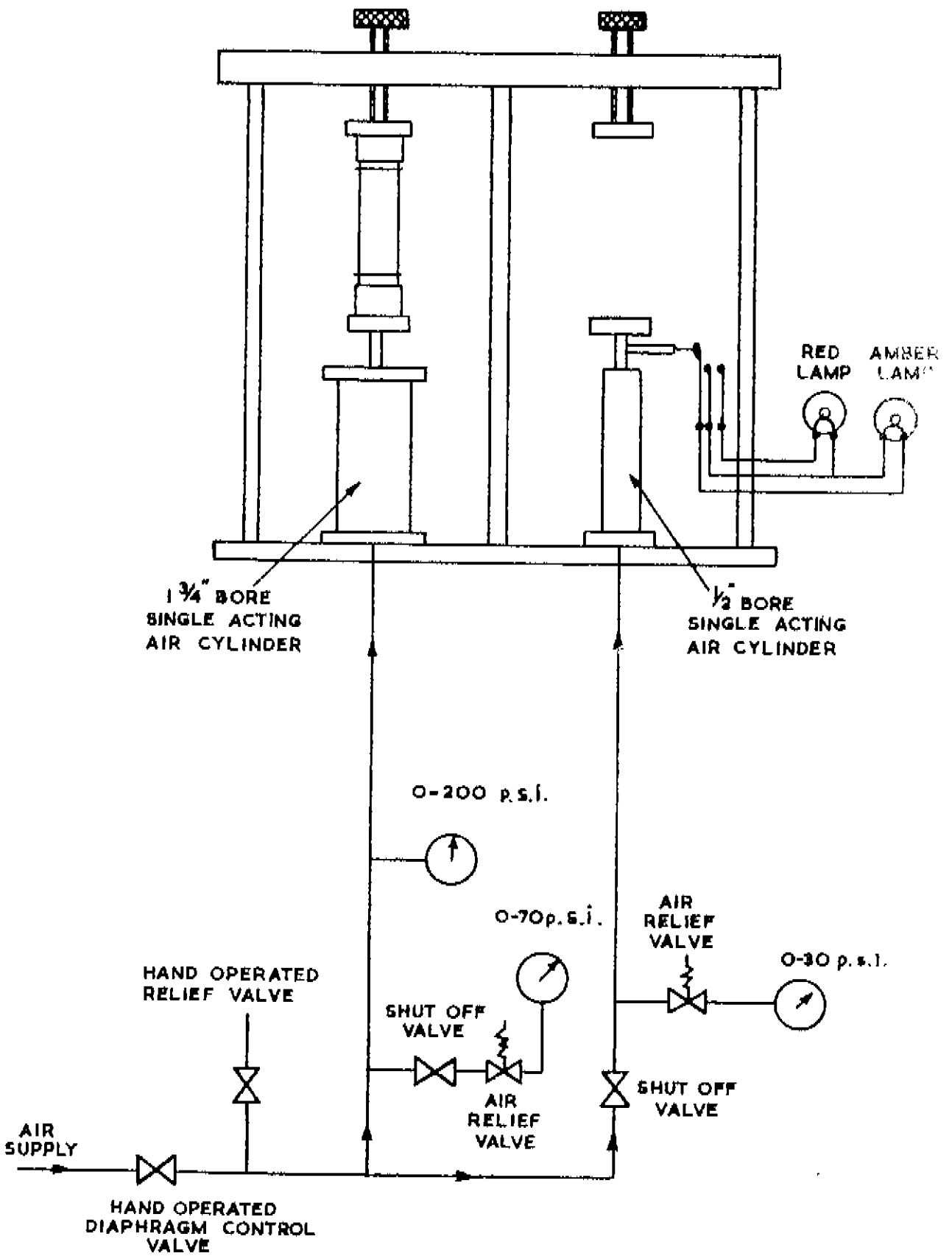

FIGURE 7.

LAYOUT OF THE COMPRESSION APPARATUS.

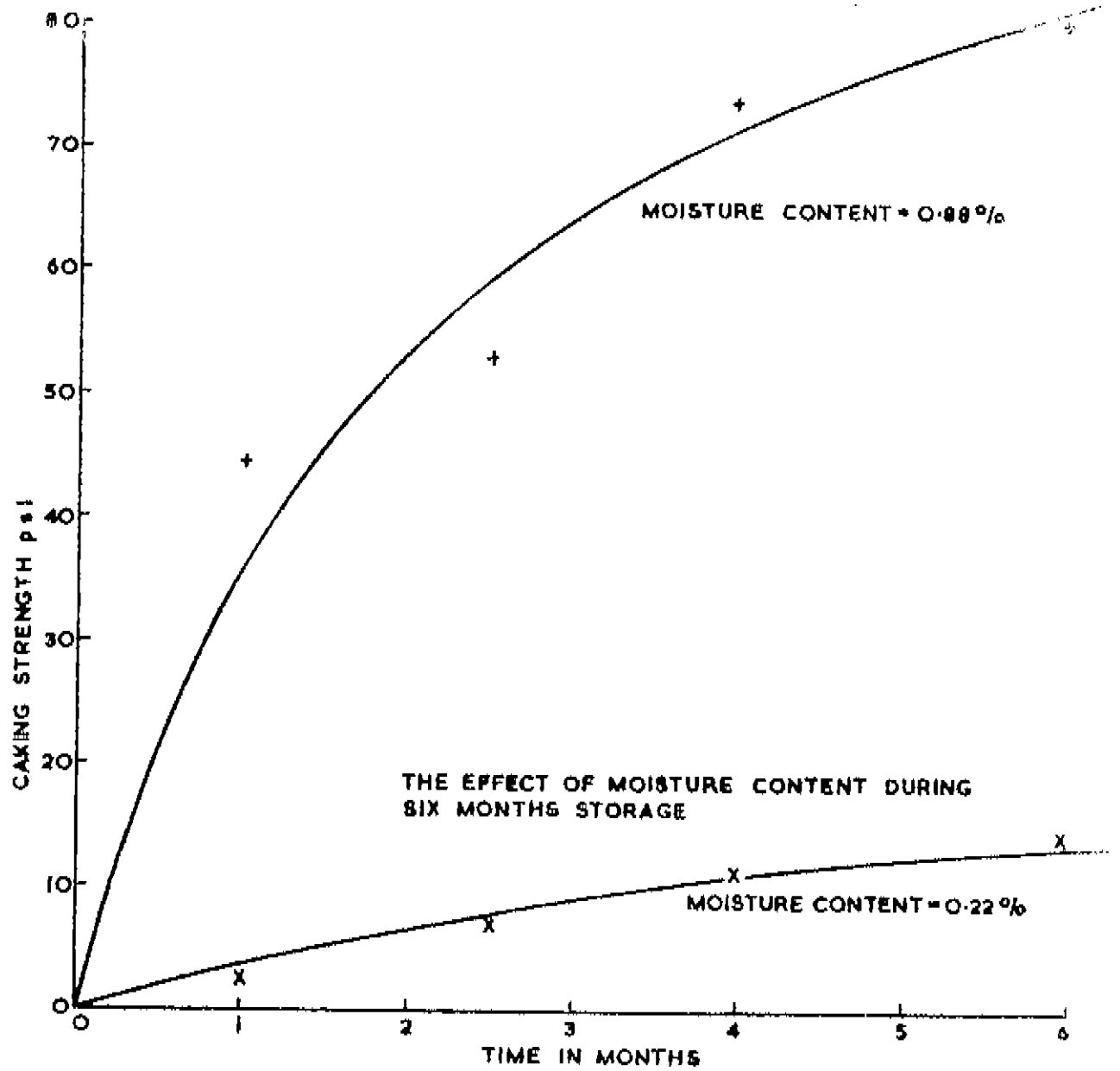

FIGURE 8.

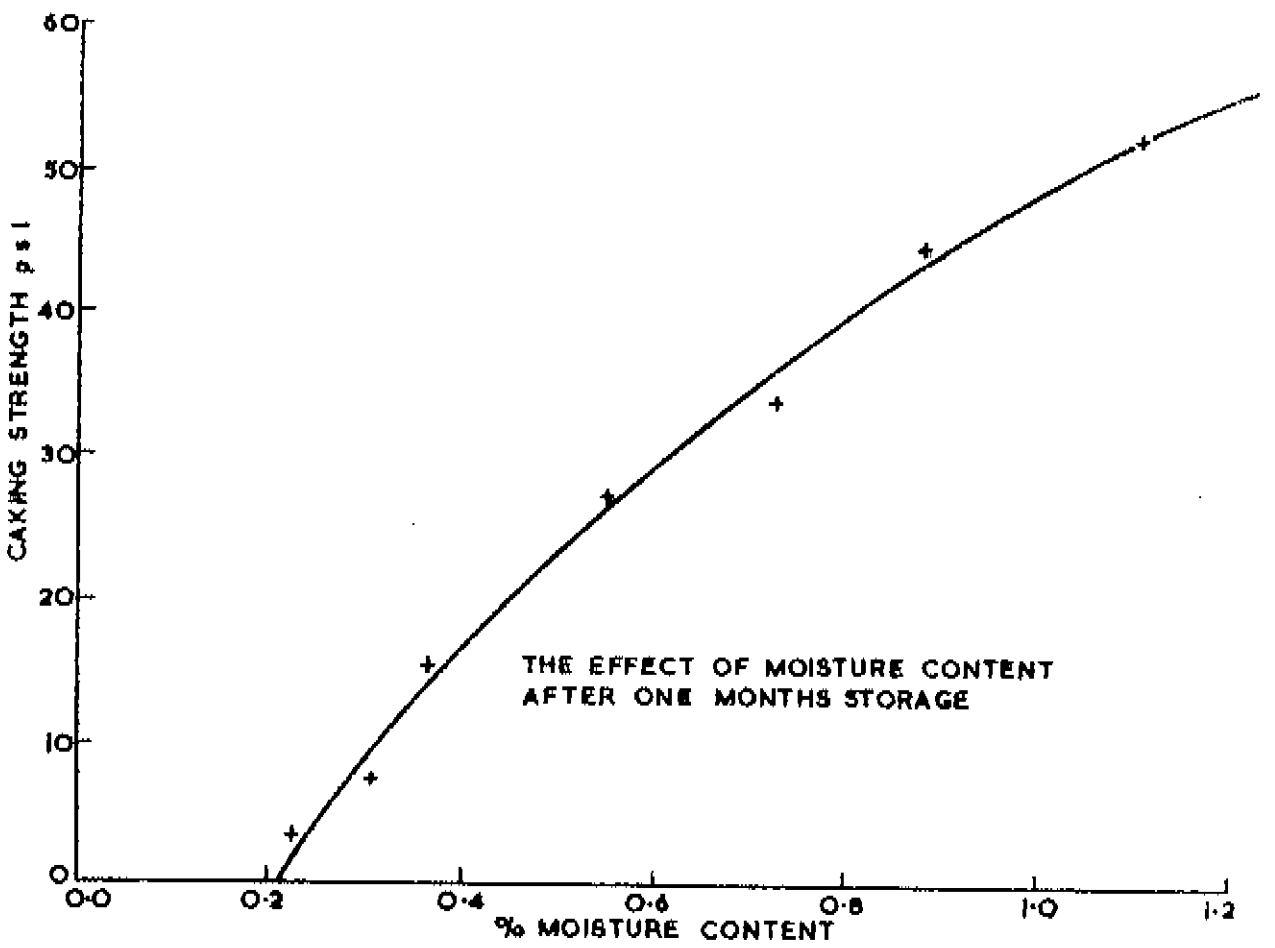

FIGURE 9.
RESULTS OF STORAGE TESTS FOR A N.P.K. COMPOUND FERTILIZER.