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GOVERNMENT OF INDIA
ATOMIC ENERGY COMMISSION

STUDIES ON MOLECULAR SIEVES FOR THE REMOVAL OF
MOISTURE FROM AIR STREAM

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ABSTRACT

Studies on molecular sieves were taken up with a view to provide sufficient experimental data for the design of an adsorbent bed for the removal of moisture to very low level from air stream containing traces of radioactive xenon and krypton, so that delay of xenon and krypton on activated charcoal columns can take place with a higher dynamic adsorption coefficient.

Experimental studies included the evaluation of molecular sieves for their moisture removal efficiencies at different face velocities and different bed thicknesses. Adsorption capacity at removal efficiency of 98.5% was determined for molecular sieves type 4A of local and foreign origin. For local molecular sieves type 4A adsorption capacity at 95% removal efficiency was found out at lower moisture contents of air streams. Regeneration characteristics of a saturated bed were also studied on local molecular sieves type 4A.

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1. INTRODUCTION

Synthetic molecular sieves are crystalline zeolitic aluminosilicates. They differ from conventional adsorbents primarily in their ability to adsorb smaller molecules while excluding larger ones so that separation can be made on the basis of molecular size. These selective adsorptive properties are due to a crystal structure incorporating inter-connecting channels and cavities of definite and uniform size. Molecules having appropriate dimensions can enter and be adsorbed in the internal cavities. The molecules in the cavities are then held thereby Vander Wall's and electrostatic forces.

For the removal of moisture molecular sieves type 3A, 4A, 5A and 13X can be used (which have pores or cavities of diameter 3\AA , 4\AA , 5\AA and 13\AA respectively) as the diameter of water molecule is 2.76\AA .

Although the molecular sieves are somewhat more expensive than other desiccants they offer the following advantages:

- (a) they provide higher adsorption capacity at lower relative humidities.
- (b) they can be used to adsorb water vapour selectively.
- (c) higher moisture removal efficiencies can be achieved.
- (d) they can be used even after many regeneration cycles with a little degradation in their performance.

The aim of the studies on molecular sieves was to evaluate the basic parameters such as removal efficiency and adsorption capacity for moisture under dynamic conditions. The results obtained can be used for the design of a large scale adsorption system for removal of moisture to a very low level. Further, these studies enable the comparison of molecular sieves of foreign and local origin for purposes of substitution wherever possible. The parameters evaluated include:

- (i) Pressure drop against the face velocity of air at a constant bed thickness
- (ii) Pressure drop against the bed thickness at a constant face velocity of air
- (iii) Moisture removal efficiency at different bed thicknesses and at different face velocities of air
- (iv) Comparative studies on molecular sieves type 4A of local and foreign origin for adsorption capacity at a removal efficiency of more than 95% and for the variation in the removal efficiency with the period of operation for constant moisture content of the air stream.
- (v) Adsorption capacity of local molecular sieves at 95% removal efficiency at lower moisture content of the air stream
- (vi) Regeneration characteristics like temperature, face velocity, moisture content and volume of the air to be purged through the saturated bed to bring about the required regeneration.

2. TYPES OF MOLECULAR SIEVES STUDIED.

The types of molecular sieves that were taken up for evaluation are given in table I.

3. PARAMETERS STUDIED

3.1. Pressure Drop Characteristics

Experimental evaluations were carried out to study the pressure drop variation with respect to bed thickness and face velocity of air stream.

3.2. Moisture Removal Efficiency

The moisture removal efficiency of a molecular sieves bed varies with duration of adsorption as well as with the moisture content of the inlet air. So it becomes important to know the removal efficiency at different intervals and at different moisture contents in the inlet air.

3.3. Adsorption Capacity

Adsorption capacity is a significant parameter for the design of an adsorption bed. Adsorption capacity of a bed depends upon the lowest removal efficiency at which the bed can be operated. If the removal efficiency is very high, adsorption capacity for moisture will be low. Hence evaluation of adsorption capacity at specific removal efficiency is of utmost importance.

3.4. Regeneration

When an adsorption bed shows reduced removal efficiency it needs regeneration. Purging hot air through the bed desorbs the water molecules held in the cavities of molecular sieves and the saturated bed is regenerated. Studies were carried out to find the effect of temperature, moisture content, face velocity and volume of the regeneration air on regeneration of the bed.

4. EXPERIMENTAL ASSEMBLY AND RESULTS

4.1. Pressure Drop Studies

The experimental set up for study of pressure drop characteristics consists of a test bed and a suction device to provide air flow through the bed. The air flow is metered by a rotameter and the resistance across the bed is measured by a manometer connected across the bed. Figure 1 gives the graphical representation of the variation in pressure drop with bed thickness of molecular sieves of pellet diameter $1/16''$ and length $1/12$ to $1/2''$ and with face velocity of air.

4.2. Moisture Removal Efficiency Studies

The experimental set up for the molecular sieves bed for moisture removal efficiency and adsorption capacity for moisture is shown in figure 2. The experiment basically consisted of measuring the moisture content of air before and after passing through the bed. The moisture content was found in terms of dew point with the help of the Alnor Dew Pointer. The air was passed through the bed at a slightly positive pressure and the evaluation of moisture removal efficiency was carried out at different face velocities and at different bed thicknesses. Results of moisture removal efficiency studies for different types of molecular sieves are given in table 2. Figure 3 shows the relationship of moisture removal efficiency with period of adsorption of local and foreign molecular sieves type 4A. The adsorption capacity was found out at the end of operation by determining the weight of moisture adsorbed in the bed. Figure 4 gives the relationship of adsorption capacity at 95% moisture

removal efficiency with different lower moisture contents in the inlet air for molecular sieves type 4A (local). The different moisture contents in air were obtained by suitably mixing atmospheric air with dry air. The experimental set up is shown in figure 5.

4.3. Studies on Regeneration Characteristics

The experimental set up for these studies is shown in figure 6. The effect of temperature, moisture content, face velocity and the volume of air to be purged through the saturated bed was found out by purging air through the saturated bed at different conditions and finding the change in weight at the end of the regeneration operation. The experimental results are given in table 3.

For regeneration the direction of purging the air through the bed was opposite to that of stream that saturated the bed. This assured that the residual moisture at the head of the bed is not carried away with the air stream when the bed is again put in operation.

5. DISCUSSIONS AND CONCLUSIONS

The experimental results given in table 2 show that molecular sieves type 13X gives lower moisture removal efficiency than molecular sieves type 3A, 4A and 5A. The moisture removal efficiency for molecular sieve type 3A, 4A and 5A being of the same order, the selection for our specific use depends upon the highest adsorption capacity for moisture and the least adsorption of xenon. As the molecular diameter of xenon is 4.8 \AA , type 5A molecular sieves adsorbs xenon also and therefore, it can not be used. Out of 3A and 4A types of molecular sieves, type 4A has higher adsorption capacity for moisture than type 3A molecular sieves.

So the type 4A molecular sieves has been selected for the purpose of moisture removal when xenon is also present in the air.

The comparative studies on local and foreign type 4A of molecular sieves show that at a removal efficiency of 98.5% the adsorption capacity for moisture is 10.7% and 11.26% respectively. (gms. of moisture / 100 gms of molecular sieves).

From figure 4 it is found that adsorption capacity increases rapidly between 5000 to 9000 ppm of moisture in air and thereafter it increases slowly.

Experimental results given in table 3 show that air with 70% R.H. at 80°F can be used for complete regeneration under the following conditions.

| | |
|--|----------------|
| Temperature of air | = 240°C. |
| Face velocity of air | = 62.6 cm/sec. |
| Contact time of air | = 0.25 secs. |
| <u>Bed height of mol. sieves</u> Bed diameter | = 4.73 |
| <u>Total volume of air required to be purged</u> Vol. of molecular sieves to be regenerated | = 10,000 |

Dry air as purge gas brings about the regeneration to completion under the following conditions:

| | |
|---|----------------|
| Temperature of air | = 150°C |
| Face velocity of air | = 32.8 cm/sec. |
| Contact time of air | = 0.28 sec. |
| <u>Bed height of mol. sieves</u> Bed diameter | = 2.05 |
| <u>Total vol. of air required to be purged</u> Volume of mol. sieves to be regenerated | = 2250 |

Table 1

TYPES OF MOLECULAR SIEVES STUDIED

| Sr. No. | Type | Source | Pellet length | Diameter | Packing Den. (gm/cc). |
|---------|------|---------|---------------|----------|-----------------------|
| 1 | 3A | Foreign | 1/12"-1/2" | 1/16" | 0.91 |
| 2 | 4A | Foreign | " | " | 0.92 |
| 3 | 4A | Local | " | " | 0.75 |
| 4 | 5A | Foreign | " | " | 0.91 |
| 5. | 13X | Foreign | " | " | 0.82 |

Table 2

MOISTURE REMOVAL EFFICIENCIES OF DIFFERENT TYPES OF MOL. SIEVES

Moisture content of Inlet air = 15,000 ppm(vol)

| Sr. No. | Face velocity of air in cm/sec. | Bed height in cms. | % Moisture removal efficiency at the start for molecular sieves. | | | |
|---------|---------------------------------|--------------------|--|-------|-------|-------|
| | | | 3A | 4A | 5A | 13 %. |
| 1 | | 5.0 | 99.15 | 99.35 | 99.42 | 97.84 |
| | 4.75 | 10.5 | 99.28 | 99.40 | 99.60 | 98.19 |
| | | 16.0 | 99.43 | 99.40 | 99.80 | 98.98 |
| 2 | | 5.0 | 99.36 | 99.40 | 99.74 | 98.53 |
| | 9.5 | 10.5 | 99.28 | 99.40 | 99.60 | 98.19 |
| | | 16.0 | 99.43 | 99.20 | 99.74 | 98.98 |
| 3 | | 5.0 | 99.66 | 99.46 | 99.66 | 98.53 |
| | 14.25 | 10.5 | 99.28 | 99.48 | 99.55 | 97.87 |
| | | 16.0 | 99.20 | 99.40 | 99.90 | 98.98 |

Table 8

REGENERATION OF MOLECULAR SIEVES TYPE 4A (LOCAL) WITH HOT ATMOSPHERIC
AIR AT VARIOUS TEMPERATURES FACE VELOCITIES AND OTHER PARAMETERS

| Relative humidity of air in air % at temp. | Temp. °C | Bed height of mol. sieves in cm. | Dia. of column in cm. | Bed height to dia. Ratio | Face velocity of air cm/sec. | Period of flow in mts. | Moisture adsorbed gms. | Moisture desorbed in gas . | %Removal of moisture | Vol. of mol. air passed in cc | Vol. of air passed in litres. | Vol. of mol. sieves. |
|--|----------|----------------------------------|-----------------------|--------------------------|------------------------------|------------------------|------------------------|----------------------------|----------------------|-------------------------------|-------------------------------|----------------------|
| 85% at 85°F. | 370 | 35 | 2.2 | 15.9 | 109.2 | 45 | 16.5 | 7.5 | 45.5 | 126 | 1225 | 9,070 |
| " | " | " | " | " | " | 60 | 17.5 | 8.5 | 48.6 | " | 1500 | 11,030 |
| " | " | " | " | " | " | 60 | 16.5 | 7.5 | 45.5 | " | 1500 | 11,030 |
| " | 325 | " | " | " | " | " | 16.0 | 9.0 | 56.3 | " | 1500 | 11,030 |
| " | " | " | " | " | 131.2 | " | 17.0 | 11.5 | 67.6 | " | 1800 | 13,235 |
| 70% at 80°C | 340 | 15.6 | 3.3 | 4.73 | 62.6 | 40 | 8 | 8 | 100 | 133 | 1280 | 9,624 |
| " | " | " | " | " | 48.8 | " | 8 | 8 | 75 | " | 1000 | 7,519 |
| 60% at 80°F (dry air) | 200 | 10 | 4.4 | 2.27 | 21.8 | 15 | 8 | 6 | 75 | 146 | 300 | 2,055 |
| " | " | " | " | " | " | 30 | 8 | 8 | 100 | " | 600 | 4,110 |
| " | 160 | 9 | " | 2.05 | 22.8 | 10 | 9 | 9 | 100 | 132 | 300 | 2,250 |
| " | 120 | " | " | " | " | 20 | 9 | 9 | 100 | " | 600 | 4,500 |

PRESSURE DROP STUDIES ON
MOLECULAR SIEVES OF PELLET SIZE $\frac{1}{16}$

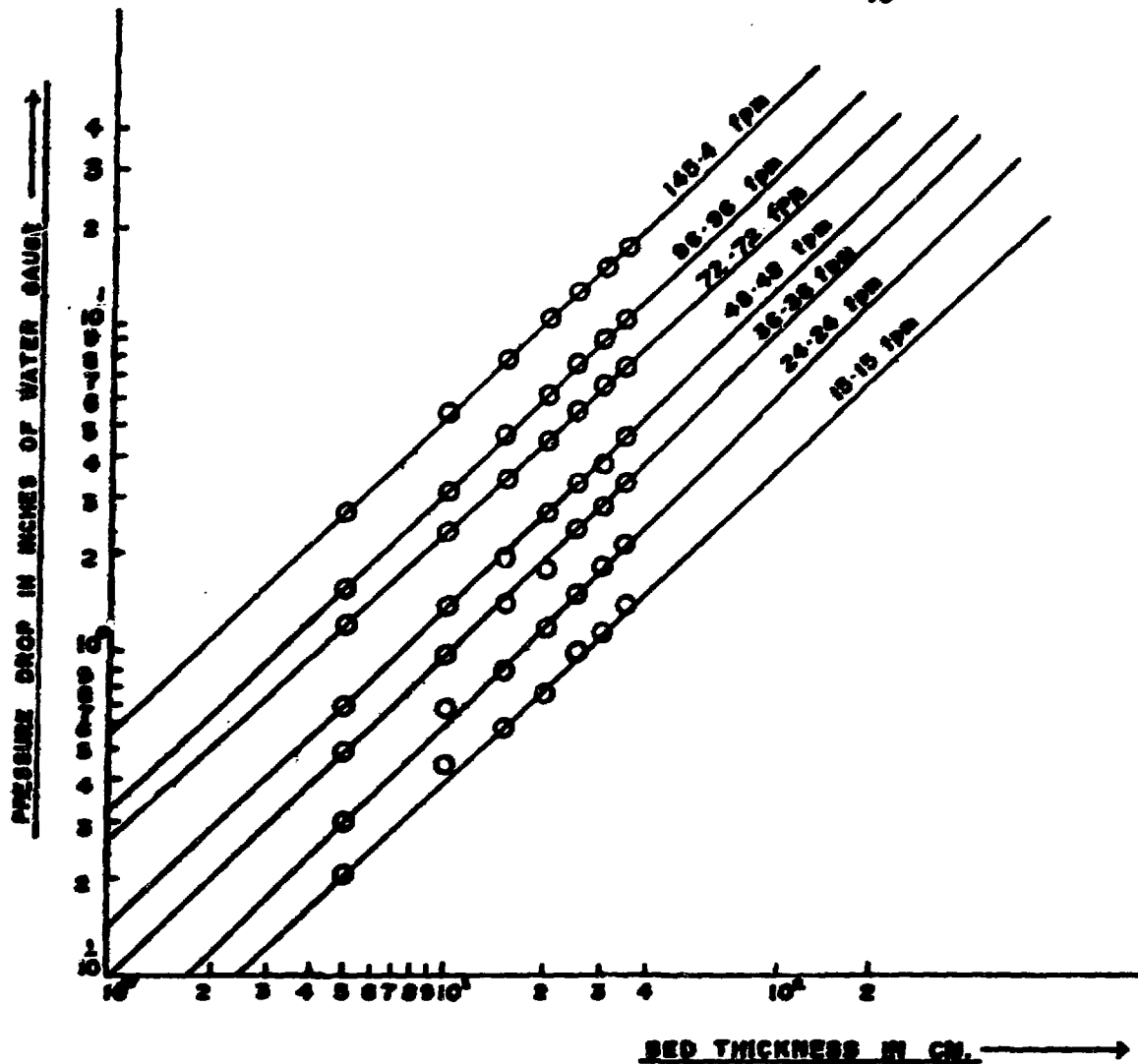
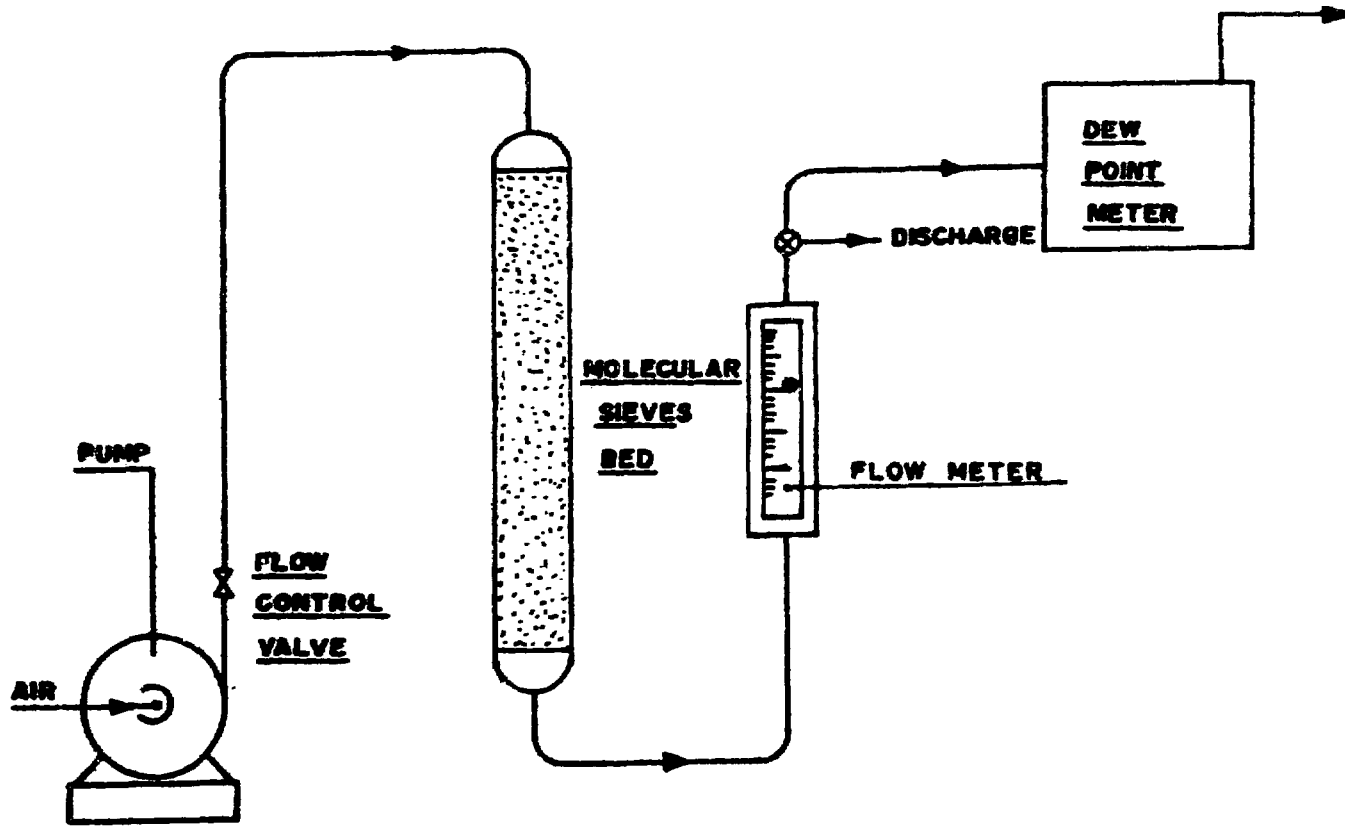


FIG. 1



EXPERIMENTAL SET-UP FOR THE
MOISTURE REMOVAL EFFICIENCY STUDIES
ON MOLECULAR SIEVES
(AT NORMAL WATER VAPOUR LEVEL IN AIR)

FIG. 2

COMPARISON STUDIES ON MOLECULAR SIEVES TYPE 4A FOR LOCAL AND FOREIGN MATERIAL

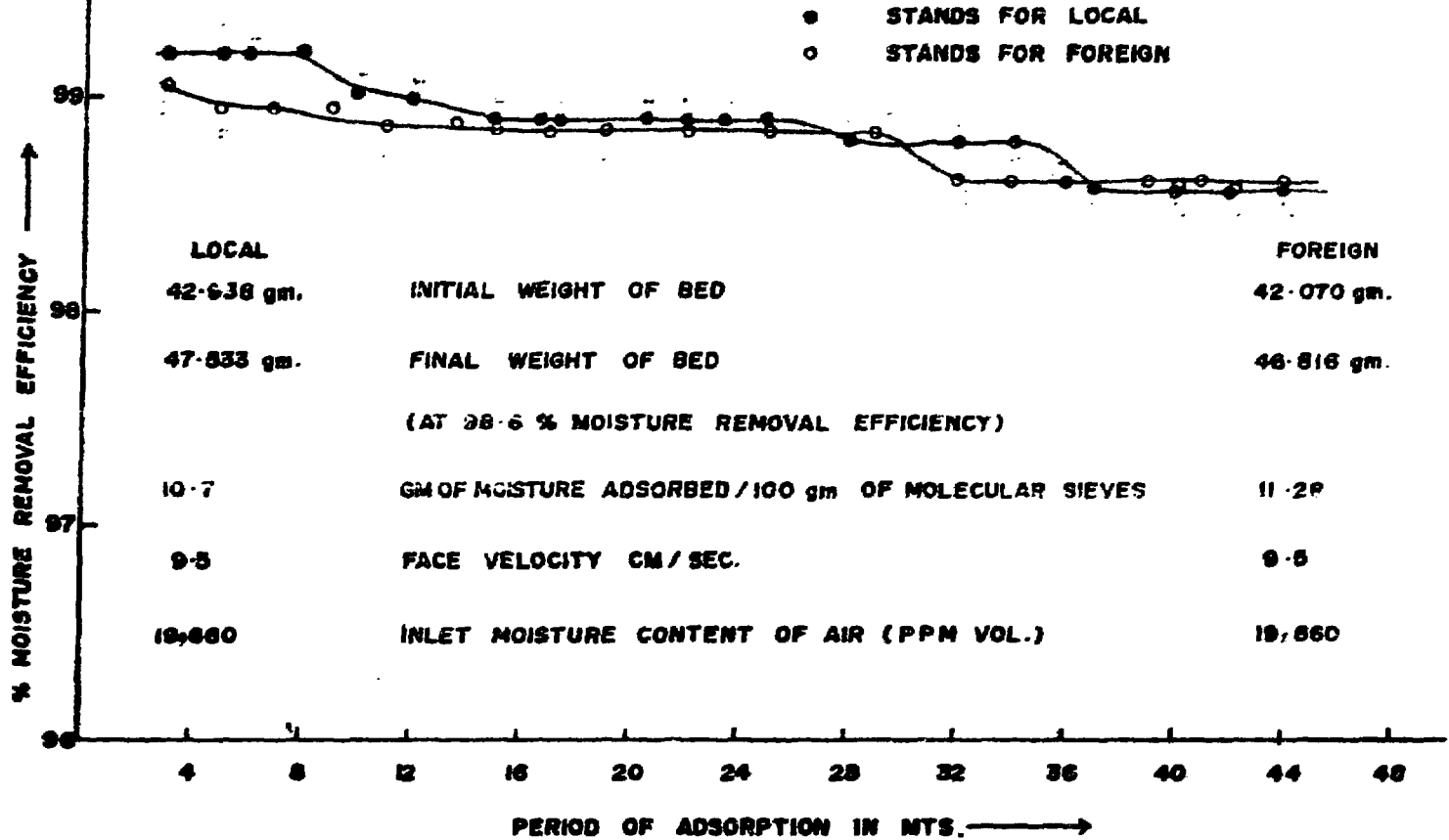


FIG. 3.

ADSORPTION CAPACITY AT 95% MOISTURE REMOVAL EFFICIENCY OF MOLECULAR SIEVES

TYPE 4A (LOCAL) VS MOISTURE CONTENT OF INLET AIR FACE VELOCITY = 9.5 CM/SEC

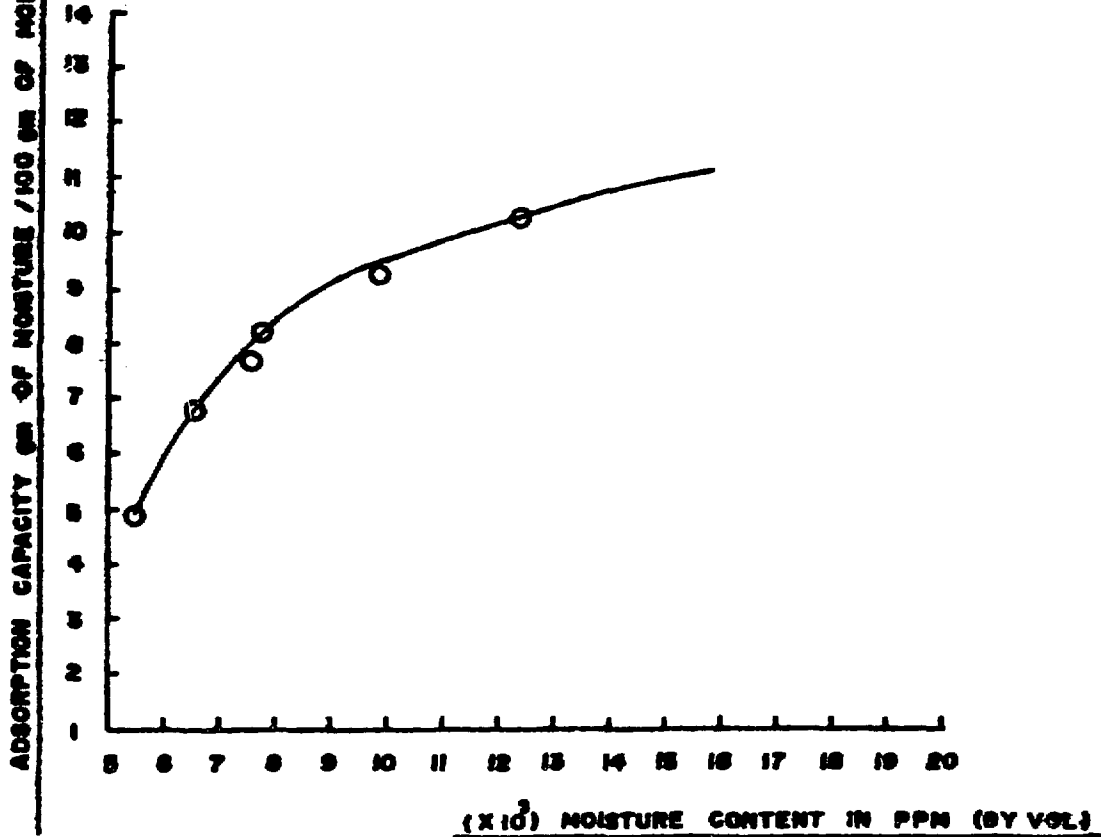
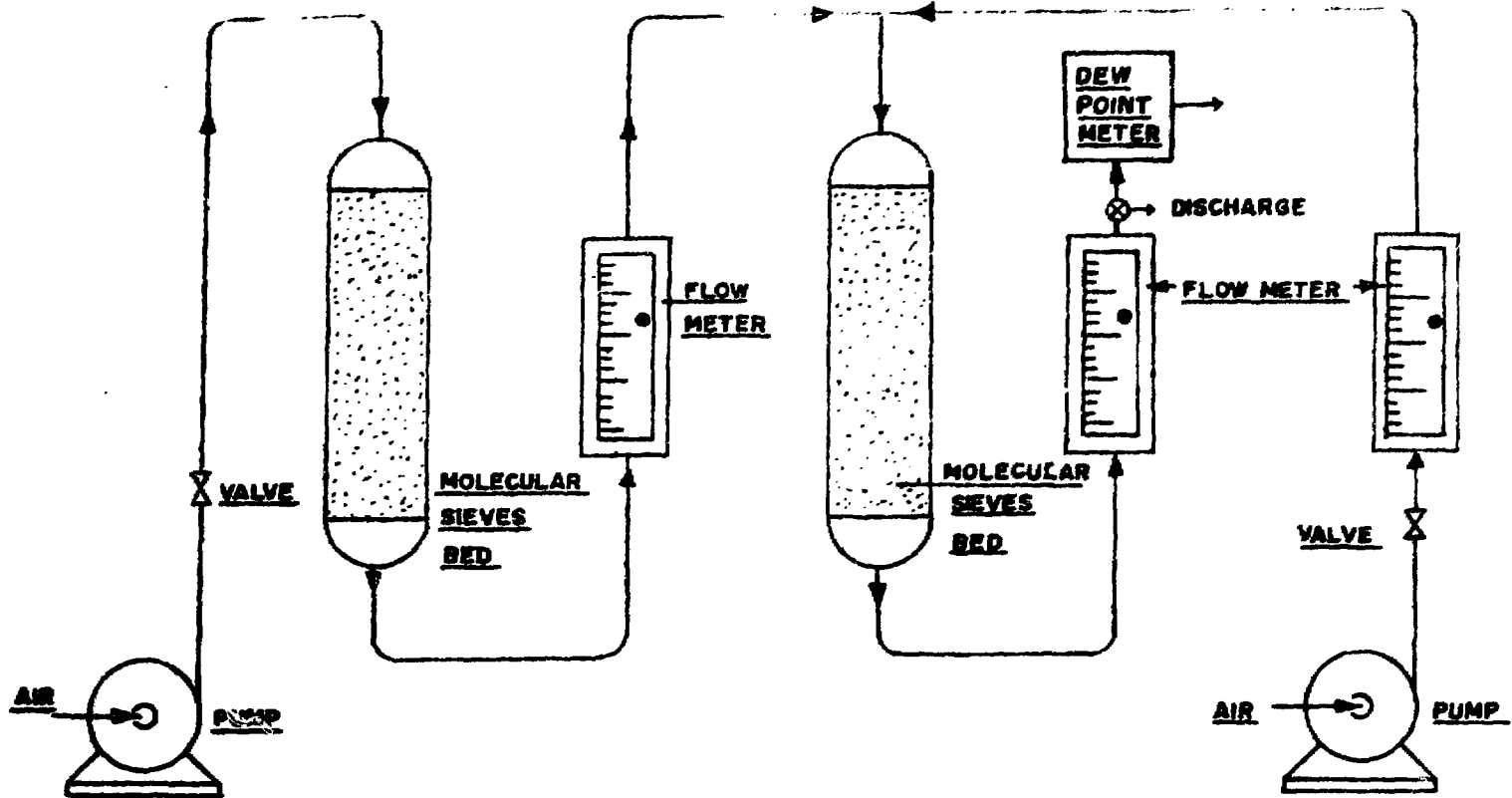


FIG. 4



THE EXPERIMENTAL SET-UP FOR THE
MOISTURE REMOVAL EFFICIENCY STUDIES
ON MOLECULAR SIEVES
(AT LOWER THAN NORMAL WATER VAPOUR LEVEL IN AIR.)

FIG. 5.

