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DR-2971

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EXPERIMENTAL STUDIES CONCERNING THE DRYING OF VOLOXIDIZER OFF-GASES

Prepared by the
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July 1981

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Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
NTIS price codes—Printed Copy: A07 Microfiche A01

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Consolidated Fuel Reprocessing Program

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Under Subcontract S-7164

for

OAK RIDGE NATIONAL LABORATORY

Oak Ridge, Tennessee 37830

operated by

UNION CARBIDE CORPORATION

for the

DEPARTMENT OF ENERGY

Contract No. W-7405-eng-26

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CONTENTS

	<u>Page</u>
HIGHLIGHTS	v
1. INTRODUCTION	1
2. EXPERIMENTAL STUDIES ON VARIOUS DESSICANTS	6
2.1 Experimental Apparatus and Procedures	6
2.2 Discussion of Results	10
3. DEVELOPMENT OF A COLUMN-MOUNTED MOISTURE DETECTOR	14
3.1 Description of the Column-Mounted Moisture Detector	15
3.2 Results	24
3.2.1 Resistance measurements	25
3.2.2 Capacitance measurements	31
4. ADSORPTION ISOTHERMS FOR LINDE TYPE 3A MOLECULAR SIEVES	38
4.1 Equipment	38
4.2 Procedure	42
4.3 Results	45
4.4 Study Performed on Linde Type 3A Molecular Sieves (Binder R)...	51
4.5 Special Runs Performed on Linde Type 3A Molecular Sieves (Binder R)	53
5. IODINE ANALYSIS USING A COMMERCIAL OXIDANT MONITOR	56
5.1 Apparatus Description	56
5.2 Experimental Procedure	58
5.3 Results	61
6. EXPERIMENTS USING CARTRIDGE-TYPE BEDS	66
6.1 Apparatus and Procedure	66
6.2 Results of the Cartridge-type Bed Studies	71
6.3 Results of Cycling Test Beds	76
6.4 Results of Heated Top Bed Runs	80
REFERENCES	84
APPENDIX A	85

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HIGHLIGHTS

This report presents the results of an experimental program conducted to aid in the design of a tritium retention system to remove tritiated water from voloxidizer off-gases. The retention system is expected to be a fixed-bed adsorption unit using a commercially available desiccant, such as molecular sieves, to dry the off-gases. The presence of iodine in the off-gas stream somewhat complicates the drying process since some iodine will be retained in the drying bed along with the tritiated water.

The present work represents a follow-up to a study in which a small-scale (2-in.-diam by 30-in.-long) packed column of Linde Molecular Sieves (LMS) type 3A was repeatedly loaded and regenerated using a non-radioactive simulated voloxidizer off-gas containing water and iodine vapor. Both water and iodine loadings were measured and the regeneration characteristics of the bed were observed.

The following studies were carried out:

- (1) Testing of other desiccants - a series of tests were conducted on LMS type 3A, 5A, AW-300, Davison 3A, Norton Zeolon-500, Drierite and alumina. These tests generally showed LMS type 3A to be superior because of its high water loading and low iodine retention.
- (2) Development of a column-mounted moisture detector - a device which uses changes in the electrical properties of the bed packing to determine the amount of water loading was successfully tested.
- (3) Adsorption isotherms - the equilibrium water loading on LMS type 3A was measured as a function of gas-phase water concentration and temperature. An equation to predict equilibrium water loading was developed for use in mathematical modeling.

(4) Iodine analysis using a commercial oxidant monitor - response and recovery times were measured for a commercially available instrument used to measure iodine concentrations in air.

The results were not promising at the high iodine concentrations employed.

(5) Tests on cartridge-type beds - a series of tests were conducted using three small drying beds connected in series. They were used to obtain basic data for evaluating the concept of using disposable cartridge-type beds for tritium retention. These tests also provided information concerning the location of the iodine retained in a larger drying bed.

One further finding of this study was the importance of the clay binder (used in pelletized molecular sieves) in obtaining satisfactory or acceptably low iodine retention. Tests on two samples of LMS type 3A having different binders showed considerable differences in the amount of iodine adsorbed during the air drying operations.

1. INTRODUCTION

Voloxidation is being developed as a process for the removal of tritium from spent nuclear reactor fuel prior to aqueous reprocessing. The current voloxidizer concept consists of a rotary kiln in which sheared fuel elements are heated to approximately 600°C in an oxidizing atmosphere. Under these conditions, tritium will be released from the fuel and exit the voloxidizer as tritiated water vapor in an off-gas stream. It is planned to remove the tritiated water from the off-gas immediately after it exits the voloxidizer by first cooling and filtering the off-gas and then passing it through a gas-drying system.

The gas-drying system would consist of several packed beds of desiccant material which are operated in parallel. In such a system, one bed would be on-stream while the other bed or beds would be in a regeneration mode. A typical regeneration procedure consists of heating the bed to approximately 300°C and simultaneously backflowing a dry gas through the bed. The water removed during regeneration is condensed and collected for ultimate disposal.

The entire operation is cyclic. When the on-stream bed is nearly completely loaded with water, it is necessary to direct the gas flow to a newly regenerated bed and begin regeneration operations on the fully-loaded bed. Such procedures are common in many industrial gas-drying operations.

One consideration not found in ordinary air drying operations, but of major concern for this particular case, is the presence of iodine vapor in the off-gas stream. Ideally, it would be desirable for the iodine to pass through the bed and not be adsorbed. Previous experiments showed that this was not the case and that some iodine was adsorbed along with the water.^{1,2} The adsorbed iodine was recovered during regeneration and was

collected in the regeneration gas condenser.

In order to measure the effect of iodine on the drying capacity of desiccants (primarily molecular sieves), an experimental program was initiated at Tennessee Technological University under the sponsorship of the Oak Ridge National Laboratory. An apparatus was constructed which would allow small-scale (approximately 2-in.-diam by 30-in.-long) packed beds to be loaded using a simulated voloxidizer off-gas containing water and iodine vapor. Using this apparatus, the following properties of a test bed could be determined:

- (1) The amount of iodine retained in the bed during a drying cycle.
- (2) The water loading during a drying cycle.
- (3) The shape of the water "break-through curve".
- (4) The iodine and water removal rates from the bed during regeneration.

It appeared that the potassium-substituted form of zeolite which has approximately a 3 Å pore size (known commercially as Linde Molecular Sieve (LMS) type 3A) would be the most promising for the intended purpose. This particular pore size would permit the passage of water molecules into the interior of the zeolite structure but would screen out the larger iodine molecules, thus allowing most of the iodine to pass through the bed and not be adsorbed.

A long-term study of LMS type 3A was undertaken. This study dealt primarily with 1/16-in. pellets containing a small quantity of a kaolin binder to hold the smaller zeolite crystals together. The study was concerned with effects which might not be apparent except after long-term exposure, such as chemical deterioration. Therefore, the study consisted of repeated cycling of a single bed of material through loading and regeneration.

The results of the first part of this study have been presented.³

In general, a decrease in water loading capacity with time was observed, but the effect was not believed attributable to the presence of iodine. A similar decrease has been found in drying operations that did not involve iodine. Iodine retained on the bed was found to be from 0.2 to 0.4 g of I_2 per kg of bed. Water loading at breakthrough varied from about 16% of dry bed weight initially to about 11% after 1100 h of operation. The original report provides a more complete description of the problem, the experimental apparatus, and the experimental procedures.³

Additional experimental runs have been conducted on LMS type 3A and on other candidate desiccants. One purpose of these additional runs was to determine if the type 3A molecular sieves are superior to other commercially available drying agents, and to determine the best operating conditions and procedures for use.

This report presents data obtained on the following desiccants:

- (1) Linde type 3A molecular sieves (1/16 inch pellets)
- (2) Linde type AW-300 molecular sieves (1/16 inch pellets)
- (3) Linde type 5A molecular sieves (1/16 inch pellets)
- (4) Linde type 3A molecular sieves (1/8 inch pellets)
- (5) Davison 3A molecular sieves
- (6) Norton Zeolon - 500 molecular sieves
- (7) Drierite
- (8) Aluminum oxide

In addition to the fixed-bed drying studies mentioned above, several other investigations were made to address certain problem areas recognized as possibly requiring some development effort in order to successfully design and operate an iodine-containing off-gas drying system and to obtain such basic data as would be required to construct a mathematical model of a drying

system. The following additional studies are reported:

Development of a column-mounted moisture detector. This study consisted of designing and testing a device to measure the change in electrical properties of the bed packing material which results from the water adsorbed within the packing. It is believed that such a device would be useful in detecting water breakthrough in a prototypical drying column. Available hygrometers appear to be unreliable for determining outlet gas dew points because of the corrosive effects and interferences caused by iodine in the gas stream.

Study of an in-line iodine monitor. A commercially available oxidant monitor (Mast Model 724-6) was obtained and modified to detect iodine in air at concentration levels of interest in this study (~ 0.0015 g I_2 /g air). Response times to increasing and decreasing iodine concentrations and useful life of the instrument were determined.

Adsorption isotherms. An apparatus was assembled to measure the equilibrium loading of water on LMS type 3A at various temperatures. Runs were conducted both with and without iodine in the feed gas. Since a knowledge of the equilibrium loading at various temperatures is necessary to successfully model a non-isothermal drying bed, a mathematical relation was developed from the data that generates equilibrium water loading at any given temperature and water partial pressure.

Test runs on cartridge-type beds. A series of experiments was conducted using several relatively short drying beds connected in series. In the initial set of experiments, test runs were continued until breakthrough occurred in the last bed. By measuring the amount of retained iodine in each of the small beds, it was possible to predict the expected distribution of retained iodine throughout a long drying bed. Similar measurements were made to

determine the water loading profile throughout the entire series of beds.

In another set of experiments, the drying system was operated in such a way as to simulate the operation of a disposable cartridge-type drying system. In the three-bed systems used, water breakthrough in the second bed was used to mark a cycle time. As each cycle ended, the first bed was removed, the second bed was moved up to the first position, the third bed was moved to the second position, and a new third bed was added to the system. This mode of operation was employed through several cycles. Iodine and water retained on each of the beds was measured. This concept might be used if it was desired not to regenerate the drying material, but rather to remove the loaded drying material for separate processing or storage.

2. EXPERIMENTAL STUDIES ON VARIOUS DESICCANTS

One purpose of this program was to conduct scoping studies on drying properties of commercially available desiccant materials. From earlier tests, the LMS type 3A appeared to be satisfactory for the intended use of drying voloxidizer off-gases. However, it was thought prudent to obtain experimental data on other materials should unforeseen problems preclude the use of LMS type 3A.

Samples of several desiccants were obtained and tested through successive drying and regeneration cycles at essentially identical conditions using the same experimental apparatus as was used in previous studies on LMS type 3A. Typically, the first few runs were made with an air stream containing only water vapor. After the adsorptive capacity of the material was established for water, iodine was added to the air stream in subsequent runs to determine the effect of iodine on water retention and the amount of iodine that was adsorbed by the bed.

2.1 Experimental Apparatus and Procedures

The apparatus used in this study was designed to simulate a small-scale voloxidizer off-gas drying system. It consisted of an inlet gas supply system, a 2-in.-diam by 36-in.-long fixed-bed adsorption column, and a column regeneration system. A schematic diagram of the apparatus is shown in Figure 2.1. Figure 2.2 is a photograph of the drying system.

As shown in Figure 2.1, inlet air at a pressure of ~ 100 psig enters the system through a cartridge filter and flows through a predrying column to remove most of the initial moisture. The pressure is reduced and the dried air is directed to a header where the flow is split using three control valves connected to rotameters. One flow path leads through a humidifying system

Fig. 2.1 Schematic diagram of experimental equipment.

Fig. 2.2. Overall view of experimental equipment.

consisting of water-filled bottles through which the air is bubbled. A second path leads through an iodine vapor generating system consisting of tubes packed with iodine crystals through which the air flows. The third air stream is untreated. The three streams are recombined to provide the simulated voloxidizer off-gas at the desired water and iodine vapor concentrations.

Weight losses of water in the humidifying section and of iodine in the iodine generating section are used along with measured air flow rates to calculate average vapor concentrations for a run. In addition, a dew point hygrometer and an iodine analysis technique are used to periodically measure these concentrations during a run.

The simulated voloxidizer off-gas then passes through the desiccant-packed test column in a downward direction. The depth of packing in the column is approximately 30 in. The column is fabricated from 2-in.-diam Pyrex glass tubing and fitted with titanium flanges. The column is mounted in a larger insulated metal cylinder which contains a Cal-rod heating coil that serves to heat the column during regeneration. During normal loading operations, the column is not heated.

The dried gas leaving the column is routed to a fume hood. Provisions are made to continuously sample the exit gas for dew point determination. Iodine concentration is determined periodically. Outlet gas dew points are measured using an aluminum oxide film-type hygrometer (Veekay Model 36). Iodine concentrations in the exit gas are determined by contacting a known volume of the gas with potassium iodide solution and analyzing this solution using either a spectrophotometric technique (for I_2 concentrations $< 0.00008 N$) or sodium thiosulfate titration (for I_2 concentrations $> 0.00008 N$).

A different flow pattern is used during regeneration. The inlet air

is first dried and the pressure is reduced as before. At the header, the flow is directed through a separate rotameter and passed through the column in the opposite direction (upward). The flow rate is ~ 10% of that used during loading runs. The heater surrounding the column is activated and the run continues until thermocouples within the column indicate a temperature of 270°C. This typically takes about 4 h.

The gas leaving the column flows into a vertically-mounted Pyrex glass condenser which is cooled to ~ 5°C with refrigerated coolant. The water condensed is collected in a receiver and the exit gas is bubbled through a potassium iodide solution to absorb any residual iodine. Iodine also appears in the condenser and in the collected water. An iodine balance to determine the amount of iodine retained on the bed is made by combining all solutions containing iodine and analyzing the solution as described previously.

Provisions are made for regenerating the predrying bed and for flowing cool air through the column heating chamber after regeneration is completed.

2.2 Discussion of Results

One interesting problem developed somewhat unexpectedly during the course of the test program. A new batch of the LMS type 3A material was ordered and a sample was tested in the apparatus used to measure adsorption isotherms. The results indicated a much higher iodine retention than had been found in earlier runs on LMS type 3A material. Consultations with the manufacturer revealed that the clay binder used to form the pelletized material had been changed from previous batches. The new binder material (called "EPG") has a different pore size distribution than the older type binder (called "R"). The sieves made with the EPG binder supposedly have better mechanical and chemical properties than those manufactured with the R binder.

It appears that the particular type of binder employed has a major effect on iodine retention. Consequently, in reporting data obtained on LMS type 3A, the binder type (either R or EPG) is specified. The earlier work,³ which investigated the effect of long-term cycling on bed operation, was done using LMS type 3A with binder R.

Table 2.1 presents a summary of the experimental results for the various drying agents tested. A more complete description of the test conditions and experimental findings for each material tested are presented in Appendix A.

The first entry in Table 2.1 is essentially a compilation of the data obtained in the previous study.³ The 16% water loading represents the loading found in the first few runs of the test program. After about 50 cycles of loading and regeneration, the loading was observed to decrease to about 11%. The iodine loading of 0.3 g per kg of bed was the lowest of any of the desiccants studied.

The Linde AW-300 (4 Å pore size) and Linde type 5A (5 Å pore size) show higher iodine retention. This is particularly true of the LMS type 5A and apparently indicates the iodine molecules have little difficulty entering the interior of pores sized 5 Å or greater. An extremely high iodine loading was found for Norton Zeolon-500, a material which also has pores in the 4 to 5 Å range.

Tests were made using LMS type 3A, binder EPG, using both 1/8-in. and 1/16-in. pellets. The 1/8-in. pellets were found to adsorb slightly less water (16% versus 19%) and significantly less iodine (2.5 g/kg versus 5.8 g/kg) than the 1/16-in. pellets. This indicates that pellet size could be an important consideration in the design of a voloxidizer off-gas drying system. It was also noted that the material with EPC binder did

Table 2.1. Summary of results of H₂O and I₂ loading on various desiccants

<u>Desiccant Type</u>	<u>Description</u>	<u>Water Loading</u> (% of dry bed weight)	<u>Iodine Loading</u> (g/kg of dry bed weight)	<u>Comments</u>
Linde Molecular Sieve, type 3A	1/16-in. pellets, binder R	16	0.3	Water loading decreases to 11% after extended use.
Linde Molecular Sieve, type AW-300	Acid Resistant, Nom. Diam. 4 ⁰ Å 1/16-in. pellets	8	1.5	
Linde Molecular Sieve, type 5A	1/16-in. pellets	15	21.0	I ₂ loading very high.
Linde Molecular Sieve, type 3A	1/8-in. pellets binder EPG	16	2.5	
Davison 3A ⁰	Spherical, 4-8 mesh beads	16	2.7	
Norton Zeolon-500	1/16-in. pellets, 4-5 Å	13	24.5	I ₂ loading very high.
Drierite	CaSO ₄ , 8 mesh	1.4	0.3	H ₂ O loading very low.
Alumina	Al ₂ O ₃ , 6-10 mesh	10.5	6.8	
Linde Molecular Sieve, type 3A	1/16-in. pellets, binder EPG	19	5.8	I ₂ loading high but H ₂ O loading does not decrease as rapidly as with binder R.
Linde Molecular Sieve, type 3A	1/16-in. pellets, binder R	17	1.1	Same material as first entry above but ordered 1 yr. later. Gave higher I ₂ loading.

Summary of Test Conditions:

Bed: 2-in. diam x 30-in.-long, weight 1.2 kg.

Gas Flow: Face velocity 1.5 fps, Air was ~ 50% saturated with H₂O and I₂ at 25°C.

Temperature: Ambient.

(Loading values are given at water breakthrough.)

not show as rapid a decrease in water loading after several cycles as was observed with the same material having the R binder.

The Davison 3A material gave essentially the same results as the LMS type 3A, EPG binder, 1/8-in. pellets. Of the two non-zeolites tested, Drierite showed extremely poor water loading and alumina showed only fair water loading and somewhat higher iodine loading than was found for the type 3A molecular sieves.

The last entry in Table 2.1 applies to 1/16-in. pellets of LMS type 3A, binder R, and should compare with the first entry in the table. However, this material was ordered from the manufacturer approximately one year after the material described in the first entry. The water loading of the two materials is comparable but the iodine loading is about four times higher for the material ordered at the later date. While no explanation of this can be offered with certainty, it appears that the difference might be attributable to variations in the R binder material used in the two batches.

An overall view of the results indicates that LMS type 3A with binder R seems to be superior because of its high water and low iodine loadings. However, this material may not continue to be commercially available because the Linde Division is converting to the use of EPG binder. LMS type 3A with EPG binder (1/8-in. pellets) and Davison type 3A would appear to be approximately equivalent.

3. DEVELOPMENT OF A COLUMN-MOUNTED MOISTURE DETECTOR

The development of a dependable means for detecting water breakthrough in a fixed-bed drying column was an important part of the overall study. Most of the earlier work made use of commercially available hygrometers which were used to measure the exit air dew point.³ These hygrometers were either of a type based on the change in capacitance of a small alumina element (Veekay Model 36) or which used an optical system to detect the formation of mist on an automatically cooled mirror (General Eastern Series 1311). The latter type was more direct and required no calibration curve.

Both methods were generally unsatisfactory for use with iodine-containing air on a continuous basis. The capacitance probe was found to be subject to corrosion and to generally deteriorate and lose calibration. The direct method using the cooled mirror was found to be unusable because of the higher dew point for iodine and the deposit of solid iodine on the mirror which provided false dew point readings.

A method for moisture detection based on properties of the packed bed rather than the air stream was thought to be usable. It was known that the electrical properties of the powdered form of molecular sieve materials are greatly influenced by water loading. Consequently, a column-mounted electrode was built and tested to determine the feasibility of using the electrical properties of pelletized column packing materials to determine water loading. Among the factors to be determined were:

1. Whether the change in electrical properties of the pelletized material was sufficiently great to be used to measure water loading.
2. Whether the presence of iodine would obscure any changes taking place due to water loading.

If successful, such a column-mounted electrode could be placed near the exit end of a prototypical drying column and the electrical properties could be monitored to detect when that section of the column was becoming

heavily loaded with water. It was believed that such a device should have the following characteristics:

1. Corrosion resistance to the moist, iodine-containing air stream.
2. Ability to withstand the high ($\sim 300^{\circ}\text{C}$) regeneration temperatures.
3. Sufficient ruggedness to allow its placement in the bottom of a packed-bed of solid pellets.

It was further recognized that it may be advantageous to place several such units at various heights within a packed-bed and allow the mass-transfer zone to be monitored as it proceeds through the bed. These units could also be used to provide an indication of the completeness of the regeneration cycle.

Another method considered for use in detecting water breakthrough depends on thermocouples placed near the exit of the bed that sense the rather large heat effects within the mass-transfer zone associated with the phase change of the water. One weakness of this method is that it is time dependent. It is influenced by the loading history of the bed. For instance, if the gas flow is interrupted and later restarted, the results based on temperature readings may not be dependable. Thus, the use of bed temperature measurements to detect water breakthrough is felt to be a convenient back-up method, but probably should not be considered as a primary means for breakthrough detection.

3.1 Description of the Column-Mounted Moisture Detector

It was desired to fabricate a column-mounted electrode system to be used in conjunction with the 2-in.-diam Pyrex glass columns used in the adsorption studies. Since titanium had been shown to be resistant to corrosion in the iodine-water system, it was selected as the material of

construction for the electrodes.

The apparatus was fabricated from two 2-in.-long sections of different sized titanium pipe. The larger pipe was 1.773 in. ID and 1.911 in. OD. The smaller was 1.250 in. OD and 1.051 in. ID. These sections were mounted concentrically leaving an annular gap of 0.261 in. Leads to the two cylinders were made using 0.061-in.-diam titanium wire welded to each cylinder. The leads were passed through hollow Mullite ceramic insulators and brought out of the apparatus through the bottom end plate using 1/4-in. stainless steel compression fittings with Teflon inserts. Saureisen cement was used to effect a seal between the lead wires and the ceramic insulators. Figures 3.1 and 3.2 are photographs of the test cell.

The test cell was inserted into the bottom of a 15-in.-long by 2-in.-diam Pyrex glass tube, as shown in Figure 3.3. The tube was then packed to a depth of approximately 12 in. with 1/16-in. pellets of LMS type 3A molecular sieves.

So as to insure correct alignment of the two electrodes during the column packing operation, a spacing tool was developed. It consisted of a 4-in.-long hollow aluminum cylinder with inside and outside diameters that exactly matched the gap between the two concentric titanium electrodes. Six 3/8-in.-wide slots cut around the circumference of the tool extended upward approximately 3 in. A wire was attached to the top of the tool. During the column packing operation, the tool was inserted between the electrodes and packing material was added until the electrodes were covered by approximately 1/2 in. of packing material. During this operation, some of the pellets would fall through the slots and become located between the electrodes. Next, the packing tool was slowly withdrawn from the top using the attached wire. The combination of slow withdrawal of the tool and

Fig. 3.1 Moisture detector test cell.

Fig. 3.2 Bottom view of moisture detector cell.

Fig. 3.3 Moisture detector cell mounted in test column.

gentle tapping of the bed resulted in a uniform filling of the gap between the electrodes. The column was then filled to the desired level with the packing material as shown in Figure 3.4.

In some of the tests it was desired to vary the depth at which the electrodes were located in the bed. Since the cell was rigidly attached to the bottom base plate by the ceramic insulators, this was accomplished by filling the bottom of the bed with glass beads up to the desired level before adding the packing material to the bed. Using this procedure, experiments were made with the bottom edge of the cell varying from 0 to 2 in. above the bottom of the molecular sieve packing.

After completion of the **packing operation**, a top closure was installed on the Pyrex glass tube and the bed was connected to the inlet gas source as shown in Figure 3.5. The inlet gas source was the same as that used for studies described previously in this report.

Two electrical parameters were examined: electrical resistance and capacitance. Resistance measurements between the electrodes were made using a Keithley Model 160B Digital Multimeter with a supplementary analog output. Digital display of resistance was limited to 2000 megohms, but an analog override provision allowed readings as high as 4200 megohms to be measured with a blanked digital display. As analog values fall below 2000 megohms, the digital display is reestablished. The analog output voltage was coupled to a Houston Ommiscribe B5116-5 recorder to allow continuous tracking during each run. Analog values were calibrated to the recorder response voltage, such that the chart could be read directly in megohms.

Capacitance measurements were made using a Hewlett Packard 4261A digital LCR bridge. Meter parameters were set as follows: 1000 Hz

Fig. 3.4 Moisture detector cell installed in packed test column.

Fig. 3.5 Overall view of moisture detector test apparatus.

excitation, DC Bias = 0, Test signal 1.0, Internal trigger, Circuit mode auto. The frequency at which capacitances were measured could be varied, such that either 1 KHz or 120 Hz excitation could be selected. The device was connected directly to the titanium lead-outs of the electrode assembly during each run. Lead capacitance was found to be 0.2 pF. All measurements were made at frequency of 1 KHz except on one run where capacitance measurements at both 1 KHz and 120 Hz were made.

Since the capacitance measurements described above could not be continuously recorded, a circuit was designed to convert the change in observed capacitance into a recordable signal. An oscillator circuit was used that featured a change in output frequency with the changing capacitance. This output frequency was converted into a DC voltage and recorded on a 0 to 10 v. strip chart recorder. This system was used on some of the latter runs although most of the data presented are based on manually recording the digital capacitance outputs.

A typical test procedure consisted of initiating the feed gas flow to the packed bed; periodically (every 5 min.) making the desired electrical measurements, both before and after water breakthrough occurred; and then either regenerating or repacking the bed in preparation for the next test.

Water breakthrough in the column was determined by using a Veekay Model 36 hygrometer. The hygrometer probe was located in the exit gas line of the bed. The same experimental procedures were used in these tests as were used in the earlier studies on larger columns. Breakthrough typically occurred about one hour after the test commenced.

Two gas flow rates were used throughout these tests - either 108.5 scfh, (giving a face velocity of 1.4 fps), or 78.5 scfh, (giving a face velocity of 0.95 fps). The humidity and iodine concentration of the inlet gas were

not measured directly during this series of tests. Instead, the rotameters in the inlet gas supply system were set at the same values as were used in the earlier tests on larger columns. For the runs involving only air-water mixtures, the humidity of the inlet gas using these rotameter settings has been found to be about $0.012\text{g H}_2\text{O/g air}$. For those runs using air-water and iodine, the humidity is essentially the same as above and the iodine concentration averages about $0.0015\text{g I}_2/\text{g air}$.

At the completion of a run, the bed was either repacked with new drying material or regenerated and reused. Repacking was done only when the type of packing was changed and most runs used regenerated packing. Regeneration was accomplished by heating the test bed overnight in a convection oven set at a temperature of 275°C .

Two types of LMS type 3A molecular sieves were studied: binder R and binder EPG. These two types of packing had been found to have different properties, particularly iodine retention, during the loading tests described in Section 2.

3.2 Results

The test results are presented as plots of the measured electrical parameter versus time. The time scale has been shifted so that the time of water breakthrough for any particular run is denoted as zero and other time measurements are denoted either as negative (before breakthrough) or positive (after breakthrough) quantities. This procedure allows a comparison of several runs on the same graph even though the water breakthrough times may be different.

Also, since the resistance measurements were found to vary over a large range during the tests, resistances are plotted on a logarithmic scale. Capacitances, which were found to vary less drastically, are plotted

using a linear scale.

3.2.1 Resistance Measurements

Plots of the resistance measurements are presented in Figures 3.6, 3.7, 3.8, and 3.9. Figures 3.6, 3.7, and 3.8 are the results of runs using R binder material and Figure 3.9 shows the results for runs using EPG binder.

It may be seen in Figure 3.6 that a rapid decrease in resistance was observed using the hygrometer. For the runs plotted, the cell was mounted with the bottom edge 1.4 in. above the bottom of the packing. No apparent difference is seen between the high and low flow rate runs.

Figure 3.7 is a plot of 3 runs; two being made without iodine in the gas and one with iodine present. Gas flow rates and cell location are the same for each run. This figure indicates that a more rapid change in resistance was observed in the run using iodine-containing moist air. The initial response in these runs occurred from 5 to 10 min. before the hygrometer indicated moisture breakthrough.

Figure 3.8 presents three runs made without iodine. Gas flow rate was varied, being high (1.4 fps) in two runs and low (0.95 fps) in one run. Response by the cell occurred at times from 1 min. before to 6 min. after the hygrometer indicated breakthrough. Results are inconclusive as to the effect of gas flow rate. The first response of the low flow rate run occurred between the first responses of the two high flow rate runs. It is noted that the low flow rate run indicated a more rapid change in resistance after the resistance had decreased to about 2000 MΩ.

A comparison of the first responses of the runs in Figure 3.8 to those in Figures 3.6 and 3.7 seems to indicate a shift relative to hygrometer

Fig. 3.6. Cell resistance versus time for different gas flow rates.

Fig. 3.7. Cell resistance versus time for different iodine concentrations.

Fig. 3.8. Cell resistance versus time for different gas flow rates with no iodine present.

Fig. 3.9. Cell resistance versus time for different gas flow rates with cell located at the bottom of the bed.

breakthrough. No satisfactory explanation for this has been found. It is noted that the humidity probably varied somewhat from run to run and that better control of such variables as gas flow rate, temperature and humidity is probably necessary to obtain more reproducible data.

Figure 3.9 presents results using LMS type 3A with binder EPG using moist air without iodine. Results are presented for a high flow rate (1.4 fps) and a low flow rate (0.95 fps) run. A major difference between these runs and the other runs discussed is that the cell had been lowered such that its lower edge was coincident with the bottom of the packing. As can be seen, this change in position of the cell resulted in resistance responses coming well after breakthrough was indicated by the hygrometer. The differences were 24 min. and 38 min. for the high flow rate and low flow rate runs respectively. Both runs indicate rapid resistance changes, similar to those seen using the material with binder R.

An additional run was made using binder R material in which dry air containing iodine only (no water) was passed through the bed. Over a period of several hours, the resistance of the bed remained above the maximum measurable value of $4200M\Omega$, indicating that iodine alone has no measurable effect on bed resistance.

Based on these measurements, the following conclusions are made:

1. For the cell configuration used, the resistance of a new or regenerated bed of 1/16-in. pellets of LMS type 3A is greater than $4200M\Omega$.
2. A change in water content of the packing within the cell results in a dramatic decrease in resistance, with values as low as $30M\Omega$ being obtained.

3. Iodine in the moist gas stream causes the decrease in resistance to be more pronounced than for the case of no iodine.
4. Proper vertical location of the cell in the packed bed will allow the cell response to closely match water breakthrough as indicated by a hygrometer in the exit gas stream. Location of the cell at the bottom of the bed resulted in the cell response.
5. The bed can be regenerated at a temperature of 275°C with the cell in place without any apparent change in the cell operating characteristics.

3.2.2 Capacitance Measurements

Results of the capacitance measurements are presented in Figures 3.10, 3.11, 3.12 and 3.13. Figure 3.10 shows the results of four runs conducted at high and low gas flow rates with and without iodine in the gas stream. The test material was LMS type 3A with R binder and the cell placement was 1.4 in. above the bottom of the bed. The capacitance of the cell with clean packing material was found to be 25 pF. In this particular configuration, a change in capacitance could be detected 10 to 15 min. before breakthrough was observed using the hygrometer in the exit line.

These data indicate a measurable effect of gas flow rate for the two runs made without iodine. As might be expected, the observed capacitance changes more rapidly for the high flow rate run. The pair of runs made in which the gas stream also contained iodine gave essentially the same results for both high and low flow rates. The measured capacitances for the runs containing iodine were found to lie between the high and low flow rate runs made in the absence of iodine.

Fig. 3.10. Cell capacitance versus time for various gas flow rates and iodine concentrations.

Fig. 3.11. Cell capacitance versus time for different cell locations relative to the bottom of the bed.

Fig. 3.12. Cell capacitance versus time for different measurement frequencies.

Fig. 3.13. Capacitance measurement circuit output voltage versus time for cell location at the bottom of the test bed.

Regeneration of the bed was accomplished between each of these runs and the capacitance of the installed detector always returned to approximately 25 pF.

Figure 3.11 presents the results of six runs using LMS type 3A with EPG binder. This figure illustrates the effect of cell placement on the response to breakthrough. Cell placement positions were 2, 1.5, 1, and 0 in. above the bottom of the bed. The curves show the expected trends in that the runs with the cell placed higher in the bed indicate capacitance changes a few minutes before breakthrough, while the run made with the cell located at the bottom edge of the bed does not show a capacitance change until ~ 20 minutes after breakthrough. These runs were all conducted in the absence of iodine. The effect of flow rate, which was seen for the material with R binder in Figure 3.10, does not appear in this set of data.

The capacitance of the clean packing, obtained from both the new and regenerated samples of material with EPG binder, was found to be about 45 pF. This represents a significant change from the value of 25 pF found for the material with R binder.

Figure 3.12 presents the capacitance results obtained on a low flow rate, iodine containing run using material with R binder. Capacitance measurements were made at two different frequencies - 1 KHz and 120Hz. The graph shows essentially no difference in the measured capacitance for a clean dry bed. However, as the capacitance begins to increase with the onset of breakthrough, capacitances measured at the lower frequency are observed to change more rapidly than those measured at 1 KHz. This figure indicates the importance of frequency in the measurement of capacitance for this particular system. All measurements of capacitance, with the exception of this particular run, were made at 1 KHz.

The capacitance measurements presented in Figures 3.10, 3.11, and 3.12 were obtained manually during the course of the experimental runs. For the runs presented in Figure 3.13, new circuitry was used to obtain and continuously record readings from the column-mounted moisture detector. This circuitry converts capacitance to a direct-current voltage which is recorded on a strip chart recorder. Figure 3.13 is a plot of voltage output versus time for a pair of runs using material with EPG binder. The detector was positioned at the bottom edge of the bed. Both runs were of the high flow rate type, one with and one without iodine in the feed gas. No detector response was evident until about 20 min. after breakthrough because of the particular placement of the detector. Once the response began, the run with the feed gas containing iodine showed a more rapid decline in voltage than the run made with the feed gas containing water only.

Based on these results the following conclusions are made relative to column-mounted moisture detectors.

1. For the cell configuration used, the capacitance of a clean bed of 1/16-in. LMS type 3A pellets is different depending on the binder used. For the R binder, the capacitance was 25 pF, and for the EPG binder, the capacitance was 45 pF.
2. A change of water content of the packing within the cell results in a significant change in capacitance, with values over 200 pF being obtained.
3. The effect of iodine in the gas is of secondary importance compared to the effect of water on capacitance measurements.
4. Breakthrough can be successfully monitored by a capacitance cell mounted near the bottom of a drying column.

4. ADSORPTION ISOTHERMS FOR LINDE TYPE 3A MOLECULAR SIEVES

The objective of this part of the study was to determine the equilibrium water loading capacity of 1/16-in. pellets of LMS type 3A molecular sieves (binder EPG) as a function of partial pressure of water vapor and the temperature of adsorption. The experimental technique consisted of passing an air stream of known moisture content across a small packed bed of molecular sieves until the bed became completely saturated. Water loading was determined by the weight increase of the bed. Runs were made at several temperatures ranging from 26 to 90°C. Figure 4.1 is an overall view of the test apparatus and Figure 4.2 shows the apparatus used for regeneration of the beds.

4.1 Equipment

4.1.1 Adsorption column

The adsorption column was made from a Pyrex glass tube (1.0-in.-I.D. x 14.0-in.-long). The height of the molecular sieve packing inside the column was 10.0 in. and was constant throughout the study. The molecular sieve bed was supported by a 0.5-in.-deep glass-wool packing. A similar glass-wool packing was also inserted above the molecular sieve bed.

4.1.2 Isothermal cabinet

An isothermal cabinet was used to maintain a constant temperature atmosphere surrounding the adsorption column. The isothermal cabinet consisted of an oven manufactured by Blue M Electric Company and had a controlling band of about $\pm 1.5^{\circ}\text{C}$.

4.1.3 Predrier

Air supplied to a humidifier was first dried in a predrier. The predrier was a 3.5-in.I.D. by 36-in.-long stainless steel column packed with LMS type 3A molecular sieves. The column was provided with a bottom air inlet and a top air outlet. The molecular sieves were supported by a stainless-

Fig. 4.1 Adsorption isotherm test apparatus.

Fig. 4.2 Regeneration apparatus for isotherm study.

steel wire screen and 1/2 in. of glass-wool packing.

4.1.4 Humidifiers

The humidifiers were Pyrex glass gas washing bottles (capacity 125ml) manufactured by the Fisher Scientific Company. Two bottles were used in parallel to divide the total flow between each humidifier, and each bottle was provided with a perforated silica disc to generate fine air bubbles in distilled water.

4.1.5 Heater for the inlet air to the adsorption column

The heater for the inlet air to the adsorption column consisted of a Pyrex glass column (1-in.-I.D. x 14.0-in.-long), wrapped with 1/2-in.-wide electric heating tape and insulating tape. The electric current to the heating tape was controlled by a variable transformer unit manufactured by the Superior Electric Company.

4.1.6 Instrumentation

The gas pressure at the inlet of the adsorption column was measured by a U-tube manometer manufactured by Meriam Instrument Company, Model 10AA25 WM. Temperatures at various points inside the isothermal cabinet and of the gas stream were recorded using copper-constantan thermocouples. Water vapor concentrations in the inlet and outlet air streams were measured by a General Eastern Series 1311 Sensor Dew Point Hygrometer. This hygrometer was an auto-optical condensation-type instrument.

4.1.7 Accessories

Flow rates of the air through the humidifier and bypass were controlled and measured by rotameters manufactured by the Fisher Scientific Company. Some of the earlier runs were conducted with iodine vapor added to the air stream. For these runs, part of the bypass air was passed through a small packed bed of iodine crystals and then combined with the humidified air

stream. An electric weighing balance manufactured by Mettler Instrument Corporation, Model PL 3001, was used for weighing purposes.

4.2 Procedure

In preparation for each isothermal loading run, the adsorption column was packed with freshly dried molecular sieves. Unused molecular sieves were dried by low pressure dry ambient air at 300°C . Figure 4.3 illustrates the flow diagram for the regeneration cycle. Regeneration was carried out for 3 to 4 h at 300°C . The dry molecular sieves were then cooled to room temperature under vacuum. A glass column containing the dry molecular sieves was then placed in the isothermal cabinet.

Compressed utility air was used as a carrier gas during the loading cycle. The compressed air was first throttled from 70.0 psig to 5.0 psig by using a diaphragm-type pressure regulator. The air was then passed through the predrier.

At the beginning of an experiment, the humidifier was bypassed and an equivalent flow of dry air was maintained through the bypass rotameter to the adsorption column and the temperature inside the isothermal cabinet was brought to the desired value; after approximately 30 min., the flows of dry air through the humidifier and the by pass were manipulated to obtain the desired value of the dew point of the gas to the adsorption column. Figure 4.4 presents a flow diagram of the loading cycle. The dew point of the air entering the adsorption column was measured occasionally and controlled to within $\pm 0.5^{\circ}\text{C}$. Temperatures at various points inside the oven and at the inlet of the adsorption column were continuously recorded and controlled to within $\pm 1.0^{\circ}\text{C}$. The run was continued for approximately 2 to 3 h after a steady-state concentration of water was achieved in the exit air from the adsorption column. The gas pressure at the inlet of the column was recorded

Fig. 4.3. Flow diagram for the regeneration cycle.

Fig. 4.4. Flow diagram for the loading cycle.

during the run and was used to calculate the partial pressure of water in the air. By knowing the initial weight of the bone-dry molecular sieves and the final weight of the loaded molecular sieves, the equilibrium water loading of the molecular sieves was obtained. At a specific isotherm temperature, the dew point of the air entering the adsorption column was varied from saturation (at $\sim 21^{\circ}\text{C}$) to -18°C . During this study, isotherm temperatures were varied from 26°C to 90°C .

In the early part of this phase of the program, several runs were conducted in the presence of iodine. It was concluded from the results obtained that the effect of the iodine on the water loading capacity of the molecular sieves is not significant. Based on this observation, iodine was not added to the humid air stream in the later part of the study.

4.3 Results

A principal objective of the adsorption isotherm work was to generate a mathematical expression to predict the equilibrium water loading on molecular sieves as a function of the partial pressure of water in the gas phase and the temperature of adsorption. This mathematical expression could then be used in mathematical modeling of the adsorption process.

During this study, equilibrium water loadings were measured at various values of water partial pressure in the inlet gas stream and temperature of adsorption. During the study, the dew point of the air at the inlet of the adsorption column ranged from -18°C to 21°C , with temperature of adsorption ranging from 26°C to 90°C .

Various mathematical expressions were attempted to fit the experimental data by using a least-squares curve-fitting technique. The mathematical expression given by the equation (4-1) gave the best curve fitting of the experimental values.

$$W_m = \frac{(a_1)(P_{H_2O})}{1 + (a_2)(P_{H_2O}) + (a_3)(P_{H_2O})^2} \quad (4-1)$$

where

W_m = equilibrium water loading, $\frac{\text{g of } H_2O}{\text{Kg of dry molecular sieves}}$

P_{H_2O} = partial pressure of H_2O in the gas phase, cm.Hg.

a_1 , a_2 , and a_3 are arbitrary constants that are functions of temperature only. These constants can be represented by the following expressions:

$$a_1 = (3.795557E+03)(\exp(A01 + A02/T + A03/T^2 + A04/T^3)),$$

$$\frac{\text{g of } H_2O}{(\text{kg of dry molecular sieves})(\text{cm Hg})}$$

$$a_2 = (1.9342105E-01)(\exp(A05 + A06/T + A07/T^2 + A08/T^3)), (\text{cm Hg})^{-1}$$

$$a_3 = (3.74117E-02)(\exp(A09 + A10/T + A11/T^2 + A12/T^3)), (\text{cm Hg})^{-2}$$

where

$$A01 = -9.9170 \text{ E} + 02$$

$$A02 = 9.7269 \text{ E} + 05$$

$$A03 = -3.1866 \text{ E} + 08$$

$$A04 = 3.4889 \text{ E} + 10$$

$$A05 = -1.0528 \text{ E} + 03$$

$$A06 = 1.0368 \text{ E} + 06$$

$$A07 = -3.3936 \text{ E} + 08$$

$$A08 = 3.7105 \text{ E} + 10$$

$$A09 = -3.7480 \text{ E} + 03$$

$$A10 = 3.6128 \text{ E} + 06$$

$$A11 = -1.1600 \text{ E} + 09$$

$$A12 = 1.2421 \text{ E} + 11$$

and T = temperature of adsorption, $^{\circ}\text{K}$

Table 4.1. Comparison of experimental and predicted water loading for Linde molecular sieves, type 3A, 1/16-in. pellets with EPG binder.

Isotherm Temp. (°K)	Partial Pressure of H ₂ O (cm Hg)	Equilibrium Water Loading (g H ₂ O/kg of dry mol. sieve)		Percentage Error
		Experimental	Calculated	
299	0.033	165.1	159.0	-3.71
299	0.100	173.6	176.2	1.50
299	0.279	182.1	185.6	1.93
299	0.415	195.1	189.7	-2.77
299	0.962	205.6	204.2	-0.69
299	1.445	211.6	217.8	2.93
308	0.280	181.0	176.5	-2.46
308	0.281	176.7	176.6	-0.05
308	0.640	188.5	185.4	-1.65
308	0.641	187.1	185.4	-0.90
308	0.939	191.8	190.5	-0.68
308	1.359	198.0	197.1	-0.44
308	1.361	196.6	197.7	0.29
318	0.642	179.0	177.9	-0.63
318	0.643	180.9	177.9	-1.66
318	1.132	190.9	186.7	-2.20
318	1.138	189.2	186.8	-1.27
318	1.631	189.8	194.8	2.64
318	1.635	189.1	194.9	3.05
333	0.471	162.4	162.9	0.32
333	0.950	181.2	173.1	-4.45
333	0.962	169.9	173.3	2.03
333	0.963	173.3	173.4	0.04
333	0.986	174.5	173.8	-0.43
333	1.375	182.5	179.9	-1.44
333	1.384	182.6	180.0	-1.42
333	1.933	188.3	188.2	-0.04
333	1.936	184.8	188.3	1.88
348	0.096	115.4	111.8	-3.16
348	0.096	114.5	111.8	-2.33
348	0.206	133.8	135.4	1.20
348	0.207	131.5	135.4	2.99
348	0.334	137.6	145.8	5.98
348	0.334	139.4	145.8	4.58
348	0.522	151.9	153.0	0.72
348	0.524	151.5	153.0	1.02
348	0.928	162.8	159.8	-1.86
348	0.934	162.9	159.8	-1.88
348	1.335	166.5	163.3	-1.93
348	1.335	168.4	163.3	-3.03
363	0.097	75.7	74.0	-2.26
363	0.097	72.5	74.0	2.12
363	0.207	100.5	105.8	5.32
363	0.208	95.5	106.1	11.08
363	0.334	123.0	123.7	0.56

Table 4.1. Comparison of experimental and predicted water loading for Linde molecular sieves, type 3A, 1/16-in. pellets with EPG binder. (Cont.)

Isotherm Temp. (°K)	Partial Pressure of H ₂ O (cm Hg)	Equilibrium Water Loading (g H ₂ O/kg of dry mol. sieve)		Percentage Error
		Experimental	Calculated	
363	0.334	121.6	123.7	1.73
363	0.523	141.4	137.3	-2.92
363	0.523	135.8	137.3	1.09
363	0.931	150.2	150.1	-0.08
363	0.933	151.6	150.1	-0.97
363	1.346	158.2	155.8	-1.49
363	1.613	158.2	158.1	-0.06

Table 4.1 presents a comparison of experimental and calculated values of equilibrium water loading on LMS type 3A molecular sieves with EPG binder. Figures 4.5 and 4.6 graphically represent the experimental and calculated values of equilibrium water loading on molecular sieves.

Fig. 4.5. Adsorption isotherms for water on Linde molecular sieves, type 3A (binder EPG).

Fig. 4.6. Adsorption isotherms for water on Linde molecular sieves, type 3A (binder EPG).

4.4 Study Performed on Linde Type 3A Molecular Sieves (Binder R)

During the early stage of the isotherm study performed on LMS type 3A molecular sieves with binder EPG in the presence of iodine, iodine loading on the molecular sieves was observed to be approximately 3.5 g of iodine per kg of molecular sieves. This value of iodine loading is almost eight times the previous iodine loading observed on the main test column.³ As mentioned in Section 2.2, it was found from discussions with the manufacturer that the higher iodine loading on the new batch of the LMS type 3A molecular sieves was probably caused by the use of a different binder, trade name "EPG", in the preparation of 3A molecular sieves. LMS type 3A molecular sieves used during the previous loading runs on the main test column were manufactured by using the binder named "R".³ The use of binder R in the manufacture of LMS type 3A molecular sieves has been discontinued. In the light of the above developments, a request was made to obtain a new batch of molecular sieves manufactured using binder R; and several pounds of left-over samples were received from the Linde Division. Several loading runs were performed on this new batch.

Experimental results for 9 runs at a temperature of 26°C are presented in Table 4.2. From Tables 4.1 and 4.2, it is evident that the equilibrium water loadings for 3A molecular sieves with binder EPG and binder R are almost comparable with LMS 3A-R material showing about 19% loading and LMS 3A-EPG material showing 20 to 21% loading. However, iodine loading for the material with binder EPG is almost 4 times greater than that of the binder R.

Water loading at 100°C (run 10) was lower by 35.57% as compared to the water loading observed at 26°C. Iodine loading at 100°C was lower by 63.36% as compared to the iodine loading observed at 26°C. Since the decrease

Table 4.2. Equilibrium water loading on Linde type 3A molecular sieves (Binder R).

Run	Isotherm Temperature (°C)	Partial Pressure of Water (cm of Hg)	Inlet Iodine Concentration ($\frac{\text{g of iodine}}{\text{g of dry air}}$)	Water Loading ($\frac{\text{g of water}}{\text{kg of mol. sieves}}$)	Iodine Loading ($\frac{\text{g of iodine}}{\text{kg of mol. sieves}}$)
1	26.0	0.9410	0.0016	196.098	0.802
2	26.0	0.9460	0.0015	190.643	0.957
3	26.0	0.9510	0.0014	191.785	1.115
4	26.0	0.9553	0.0014	185.700	0.918
5	26.0	0.9640	0.0014	186.983	0.917
6	26.0	1.3920	0.0000	188.800	No iodine in the feed
7	26.0	1.4250	0.0000	193.900	No iodine in the feed
8	26.0	1.9620	0.0000	200.500	No iodine in the feed
9	26.0	1.9680	0.0000	199.300	No iodine in the feed
10	100.0	0.9700	0.0014	120.464	0.336

in iodine loading is larger than the decrease in water loading at the higher loading temperatures, it could be advantageous to load the bed at the temperature above the ambient condition to achieve a better separation between iodine and water.

4.5 Special Runs Performed on Linde Type 3A Molecular Sieves (Binder R)

The objective of the two special runs made in this study was to achieve a higher separation between iodine and water by modifying the loading and regeneration cycle.

During the first run, the molecular sieves were first loaded with water in the presence of iodine at 26°C up to equilibrium. The temperatures of the bed and the feed gas to the bed were then raised to 100°C . The run was continued for another 2.0 h with no iodine in the feed gas. Throughout this run, the water concentration in the feed gas to the bed was maintained at a constant value. During the second run, the loading procedure was the same as in the first run except for the later part of the loading run when dry air was passed over the bed at 100°C instead of moist air. Detailed information is presented in Table 4.3. The percentage decreases in iodine and water loading by continuing the run at 100°C are presented in Table 4.4. The percentage decrease in iodine loading was observed to be comparable to the percentage decrease in water loading during both runs. Hence, it does not appear possible to achieve a better separation between iodine and water by the modified loading and regeneration cycles tested in this study.

Table 4.3. Special runs performed on Linde type 3A molecular sieves (Binder R).

Run	Isotherm Temperature (°C)	Partial Pressure of Water (cm of Hg)	Inlet Iodine Concentration ($\frac{\text{g of iodine}}{\text{g of dry air}}$)	Water Loading ($\frac{\text{g of water}}{\text{kg of mol. sieves}}$)	Iodine Loading ($\frac{\text{g of iodine}}{\text{kg of mol. sieves}}$)
1					
1-1	26.0	0.9430	0.0015	190.1 ^a	
1-2	100.0	0.9430	0.0	128.3 ^b	0.515 ^c
2					
2-1	26.0	0.9670	0.0015	187.1 ^a	
2-2	100.0	0.0	0.0	108.1 ^b	0.526 ^c

Note: ^a The water loading was calculated by weighing the bed at the end of Phase 1.

^b The final water loading was calculated by weighing the bed at the end of Phase 2.

^c The final iodine loading was calculated by regenerating the bed at the end of Phase 2.

Table 4.4. The decrease in iodine and water loading due to the continuation of the runs at 100°C.

Run Number	Decrease in Water Loading ^a (%)	Decrease in Iodine Loading ^b (%)
1	32.49	46.19
2	42.22	45.04

Notes: ^aPercentage decrease in water loading was calculated by continuing the water loading with the end of phase I as a reference point.

^bPercentage decrease in iodine loading was calculated by considering the iodine loading in Run 2, Table 4.2 as a reference point.

5. IODINE ANALYSIS USING A COMMERCIAL OXIDANT MONITOR

The purpose of this part of the program was to evaluate a commercially available oxidant monitor for use as an iodine analyzer for gas streams. The device considered was a Mast Model 724-6 Oxidant Monitor, manufactured by the Air Monitoring Division, Mast Development Company, Davenport, Iowa.

It is recognized that in an operating reprocessing facility iodine may be analyzed using radioactive counting techniques. However, since much development work is scheduled to be performed in a non-radioactive pilot plant facility where detection systems based on radioactive properties cannot be used, it was desired to find a convenient and dependable iodine analysis method based on chemical properties.

5.1 Apparatus Description

The Mast Oxidant Monitor consists primarily of a microcoulomb sensor and basic electrical circuitry. A chemical solution, containing potassium iodide, is maintained in a reservoir and pumped through a closed system containing the microcoulomb sensor. At the sensor, the solution is brought into contact with an air sample containing the oxidant. The system was originally designed to detect ozone but has been found to work effectively with iodine and other halogens.

The following is a description of the theory of operation presented in the Mast Literature.⁴

The sensing of ozone in the air sample is accomplished by the oxidation-reduction of potassium iodide which is contained in the sensing solution. This reaction takes place on the cathode portion of the electrode support. In this region, any ozone in the air sample reacts with the sensing solution as follows: (other oxidants behave in a similar manner)

At the cathode, a thin layer of hydrogen gas is produced by the polarization current:

When the voltage is applied to the electrodes (about 0.24 Volts), the hydrogen layer builds to its maximum and the polarization current ceases to flow.

When free iodine is produced by the reaction with ozone, it immediately reacts with the H_2 as follows:

The removal of the hydrogen from the cathode allows a repolarization current of two electrons to flow in the external circuit, re-establishing equilibrium. Thus, for each ozone molecule reacting in the sensor, two electrons flow through the external circuit. Hence, the rate of electron flow, or current, is directly proportional to mass per unit time of ozone entering the sensor.

When iodine is the species to be detected, only the last two reactions above would be important.

After the solution passes through the detector, it flows through a small packed bed of Nylon beads, which removes any residual I_2 , and is finally passed back to the liquid reservoir. Both the liquid flow and the gas flow are controlled by calibrated pumps which are an integral part of the apparatus.

The Mast Oxidant Monitor has a maximum concentration range of up to 10 ppm. Since this is considerably less than the concentration range of iodine expected to be present in a typical voloxidizer off-gas stream, an effort was made to modify the monitor to obtain readings at higher concentration levels.

Four selectable concentration ranges are available on the Mast Oxidant Monitor; these are 0 - 1, 0 - 2, 0 - 5, and 0 - 10 ppm. The means of selecting these ranges is through a selector switch which acts to insert appropriately sized resistors into the final output circuit. A special gain control resistor was added at the "Aux" position of the sensitivity selector switch. It was found that an 8 k Ω resistor was sufficient to keep the highest iodine concentration studied on-scale for the instrument.

In addition to the indicating meter on the instrument, a strip chart recorder was used to obtain a permanent record of the concentration readings during the instrument tests.

Figure 5.1 is a photograph of the Mast Oxidant Monitor with a variable resistance source and recorder attached. Figure 5.2 shows an interior view of the instrument with the bellows-type air pump at the top, the packed section of nylon beads in the center, and the solution storage reservoir at the bottom.

5.2 Experimental Procedure

An iodine-air mixture of known composition was generated using the inlet gas make-up system that was used in testing the packed drying beds. The test gas was made by splitting a pre-dried stream of air such that part of it passed through a small bed packed with iodine crystals and part by-passed the bed. The two streams were then recombined. Iodine concentrations were set by the relative proportions of the gas passing through and by-passing the iodine bed. The Mast Oxidant Monitor was connected to sample a portion of the stream. The actual iodine concentrations were determined by absorption of the gas in KI solution and subsequent spectrophotometric analysis.

A typical test on the Mast Oxidant Monitor began with an initial period in which only by-pass air was allowed to flow through the system. To begin a test, the flow through the iodine bed was quickly initiated and the rotameters adjusted to give the desired iodine concentration. The time required to affect this change was typically less than 5 s.

The output of the Oxidant Monitor was continuously recorded on a strip chart recorder. For each iodine concentration level used, the output was found to reach a stable value. The time from the initiation of the

Fig. 5.1 Mast Oxidant Monitor.

Fig. 5.2 Interior view of the Mast Oxidant Monitor.

iodine flow until the instrument read 90% of the steady-state value was recorded. The tests were then continued for an additional 30 min. to check for any drift in the output reading. The test was concluded by stopping the iodine flow (but maintaining an equivalent by-pass air flow) and noting the time for the readings to decrease to 75, 50, 20, and 10% of the steady state readings.

5.3 Results

Table 5.1 and Figure 5.3 show the results of a series of tests made as described above. Figure 5.4 shows a superposition of these values, which graphically demonstrates the response rate, steady state value, and recovery rate as a function of I_2 concentration.

After the tests were concluded, a large reduction of the sensitivity of the unit was observed, amounting to a decrease in indicated iodine concentration values to approximately 20% of the values previously obtained. An analysis of this fault was accomplished by calibrating the internal liquid-side and gas-side pumps. The liquid side pump was found to have failed. The specific nature of the fault was traced to elastic check value membranes internal to the pump circuit; these showed embrittlement and consequent loss of elasticity. The liquid pump calibration during this diagnosis showed a reduction in output from that specified that coincided almost exactly with the consequent loss in system sensitivity. Verification of this effect, by overnight immersion of one of the elastic membranes in a strong KI solution, was positive. A complete darkening and embrittlement of the membrane was observed.

As a background to this failure, it is noted that the attempted measurements of I_2 levels exceeded the manufacturer's limit by a factor of over 100. Accumulated run time, consisting of preliminary work and the

Fig. 5.3. Mast Oxidant Monitor output versus iodine concentration in air.

Fig. 5.4. Response and recovery times for the Mast Oxidant Monitor (Model 724-6).

Table 5-1. Response and recovery times for a Mast Oxidant Monitor at high iodine concentrations

IODINE CONCENTRATION (g I ₂ /g x 10 ⁴)	STEADY-STATE VALUE (volts)	STABILIZATION TIME (min to 90% of steady state value)	Recovery Times (minutes to indicated % of steady state value)			
			75 %	50 %	20 %	10 %
0.40	0.0345	8.1	1.0	1.3	4.2	7.4
0.8	0.036	11.5	1.1	1.4	3.6	6.7
4.42	0.0655	3.8	2.3	4.3	7.6	11.7
9.06	0.072	2.5	3.4	6.0	9.0	13.0
12.80	0.077	1.8	2.7	7.0	11.9	17.1
18.00	0.087	1.4	4.1	6.5	11.2	16.0

studies themselves, exceeded 20 h. In addition, the unit was in storage for more than a year prior to the tests.

Based on an analysis of the system, it is believed that the following changes would be necessary before the Mast unit could be used successfully to monitor large iodine concentrations:

1. Replace the material used in the valve membranes with a composition more suited for use in the presence of halogens, such as a Vyton elastomer.
2. Conduct general maintenance on a more frequent basis than recommended by the manufacturer.
3. Evaluate a change in the makeup of the KI solution to allow a more efficient absorption of higher I_2 concentrations.
4. Dimensionally increase the size of the nylon bead column, doubling or tripling the quantity of nylon beads in the regeneration circuit.

6. EXPERIMENTS USING CARTRIDGE-TYPE BEDS

6.1 Apparatus and Procedure

The basic experimental set-up used in this phase of the study consisted of three separately packed 10-in.-deep by 1-in.-diam beds of drying material. The beds were made from Pyrex glass tubes that had ball joints at each end so that they were easily connected in series. Each bed was covered with a sleeve of foam rubber insulating material approximately 1/2-in. thick. Various delivery lines and sampling valves were made of Teflon.

The gas flow rate for the entire series of runs was maintained at 27.2 scfh. This corresponds to a face velocity of 1.4 ft/sec in the 1-in.-diam beds and is the same face velocity used in many of the runs in the 2-in. column. The same test air make-up section that was used on earlier tests on larger packed columns was used to deliver air at the desired moisture and iodine concentrations.

Testing was done using two types of 1/16-in. pellets of LMS type 3A, one type containing EPG binder and one type containing R binder. Flow was always downward through the three-bed series so that the top bed received the incoming gas. Figure 6-1 is a photograph of the test beds connected together without the insulation jacket. Figure 6-2 shows the same view with the insulation installed.

After completion of a particular run, the three-bed assembly was dismantled and each separate bed was capped and stored for final determination of retained water and iodine. The total loading (water and iodine) was determined by weight difference on each bed from weighings made before and after each run. Iodine loading on each bed was found by placing each bed in a tubular furnace operated at 275°C and passing a dry air stream through the heated bed. The air stream was then bubbled through a large container of

Fig. 6.1 Cartridge-type beds without insulation.

Fig. 6.2 Cartridge-type beds with insulation installed.

KI solution. After completion of the regeneration, the KI solution was spectrophotometrically analyzed for iodine and, from the concentration and volume of the adsorbing solution, the total amount of iodine on the bed was determined. Typically, the iodine loading was less than 1% of the water loading.

The initial sequence of runs was conducted until water breakthrough occurred at the exit to the bottom bed. The purpose of these runs was to determine water and iodine distribution throughout a saturated bed. The first 5 runs employed water only in the air stream and used LMS type 3A with EPG binder. The next 8 runs were essentially the same except iodine was also present in the air stream. The final 2 runs were made using LMS type 3A with R binder.

A second sequence of runs was made in a mode intended to simulate a cartridge-type disposable bed system. A typical run began with three new beds in series. The top bed received the incoming simulated off-gas. The system was run in this manner until water breakthrough occurred at the exit of the second (middle) bed. At this time the top bed was removed from the system, the middle bed moved to the top position, the bottom bed moved to the middle position, and a new bottom bed inserted in the system. The run was then continued until breakthrough again occurred at the middle bed.

After completion of one or more cycles of this operation, all of the individual used beds were weighed and regenerated to determine the water and iodine loadings. Runs of this type were conducted on 1/16-in. pellets of LMS type 3A with both EPG and R binder.

The final sequence of runs was conducted similarly to the second sequence except that the top bed was heated during the tests. This was done in an effort to reduce the retained iodine on the top bed without drastically

Table 6.1. Water loading on cartridge-type beds - LMS 3A, EPG binder

<u>Run No.</u>	<u>Date</u>	<u>Water Loading (g/kg solid)</u>			<u>Inlet Humidity^a (g H₂O/g Dry Air)</u>	<u>Inlet Air Temperature (°C)</u>
		<u>Top</u>	<u>Middle</u>	<u>Bottom</u>		
1	6/12/80	213.0	208.3	132.1	0.0179(near saturation)	24
2	6/16/80	210.8	194.9	85.7 ^b	0.0236(near saturation)	29
3	6/18/80	201.7	193.5	77.3	0.0199(near saturation)	26
4	6/19/80	182.5	184.3	107.3	0.0126	26
5	6/20/80	186.7	185.7	118.1	0.0120	26

Test Conditions:

Air Flow Rate: 27.2 scfh

Face velocity: 1.4 ft/sec

Flow direction: Top to bottom.

Air in at ambient temperature, beds insulated.

Water loading measured at breakthrough of the third (bottom) bed.

^a Inlet humidity calculated from water adsorbed and inlet air flow rate.

^b Breakthrough determined by temperature rise at bed outlet because of a hygrometer malfunction.

reducing the amount of water adsorbed.

6.2 Results of the Cartridge-type Bed Studies

The first five runs in this study were made without iodine in the air stream. The water loadings for these runs are presented in Table 6.1. The first three runs were made with air very nearly saturated at ambient conditions and the last two runs were made at a lower humidity.

For the runs conducted near saturation, the top bed showed a slightly higher water loading than the middle bed. For the two runs conducted at a lower humidity, loadings for the top and middle beds were almost identical. The effect of humidity can be seen in the fact that the top bed loaded to 20 or 21% for the high humidity runs but the same bed loaded only to about 18.5% for the runs conducted at humidities of 0.012 g H₂O/g air.

Runs 6 to 15 were conducted in the same mode as runs 1 to 5 except that iodine was present in the gas stream. The results for these ten runs are shown in Table 6.2. The runs conducted with material having binder EPG (runs 6 to 13) showed consistently higher loadings of both water and iodine than the runs with binder R (runs 14 and 15).

The following observations can be made of the data presented in Table 6.2.

1. Water loading was about 18% of dry packing weight for binder EPG and about 16% of dry packing weight for binder R.
2. Water loading in the top and middle beds was approximately equal. Loading in the bottom bed was typically about half that found in the top bed.
3. For the run taken 51 minutes past breakthrough (run 11) the water loading in the top and middle beds did not significantly increase over loading values at breakthrough in other runs. As expected,

Table 6.2. Total loading and iodine loading for three cartridge-type beds in series

Run No.	Total Loading, (g/kg solid)			Iodine Loading (g/kg solid)			Inlet Humidity (g H ₂ O/g air)	Binder	Comments
	Top	Middle	Bottom	Top	Middle	Bottom			
6	187.0	187.1	115.8	0.905	0.661	0.616	0.0119	EPG	Same beds used in Runs 1 to 5
7	183.9	181.8	106.0	1.653	0.867	0.677	0.0123	EPG	
8	172.5	179.3	91.4	1.152	0.807	0.591	0.0137	EPG	
9	178.0	184.8	69.2	1.090	1.015	0.785	0.0104	EPG	
10	176.3	183.0	87.3	1.185	0.757	0.915	0.0121	EPG	Bottom bed replaced with new packing
11	180.2	184.3	159.3	1.055	0.896	0.734		EPG	Run conducted to 51 minutes past break-through
12	177.5	195.3	93.8	0.931	0.845	0.795	0.0100	EPG	
13	185.7	183.9	111.5	1.192	1.011	0.985	0.0107	EPG	All three beds replaced with new packing of binder EPG
14	163.0	162.4	93.8	0.276	0.243	0.309	0.0109	R	All three beds replaced with new packing of binder R
15	157.4	159.1	95.1	0.230	0.200	0.273	0.0108	R	

Test Conditions:

Air flow rate: 27.2 scfh; Face velocity: 1.4 fps; Inlet I₂ conc.: 0.0012 g I₂/g air; Adiabatic operation.

the water loading in the bottom bed did show a significant increase over previous values.

4. The first 4 runs with binder EPG seemed to indicate a decrease in total loading in the bottom bed. For the next run (run 10), the bottom bed was changed with new binder EPG packing. An increase in the loading was noted although it was not as high as the original loading for the bottom bed.
5. Complete change-out of all three beds (run 13) resulted in loadings very similar to those in the original run (run 6).
6. Iodine loading was found to be approximately evenly distributed between the top, middle and bottom beds. These results are shown in Figure 6.3. Material with binder EPG was found to have loadings approximately 4 times as great as material with binder R. The EPG material consistently showed a decrease in loading from top to middle to bottom bed. The overall decrease was about 25%. The only exception was run 10 which was made using a newly replaced bottom bed.
7. The pattern mentioned above was not followed for the binder R material. This material showed a slight decrease in loading between the top and middle bed but then showed an increase in loading of the bottom bed to values greater than those found for the top bed.

Two main items of significance concerning these data are believed to be:

- a) Iodine is distributed rather evenly down the length of the series of beds.
- b) Materials with the EPG binder retain about 4 times as much iodine as material with the R binder.

Fig. 6.3. Iodine retention on three cartridge-type beds in series.

Table 6.3. Results for cyclical bed operation

Run No.	Total Loading (g/kg solid)				Iodine Loading (g/kg solid)				Binder Type	Comments
	Top 1	Middle 1	Bottom 1	Bottom 2	Top 1	Middle 1	Bottom 1	Bottom 2		
	Top 2	Middle 2								
16	191.3	188.4	89.1	1.2	0.977	1.027	1.084	1.586	EPG	Begin Runs with New EPG Beds
17	176.5	107.2	2.3		1.201	0.926	1.277		EPG	Hygrometer problems caused run to be stopped before breakthrough
18	177.7	177.5	102.4	1.2	1.069	0.872	0.795	1.009	EPG	
19	183.4	183.6	92.3	1.2	1.036	0.914	0.864	1.065	EPG	
20	176.7	179.4	64.1	.0	0.895	0.817	0.709	0.978	EPG	
21	174.4	177.9	75.7	1.2	0.851	1.043	0.739	0.910	EPG	
22	169.9	172.3	111.5	1.0	0.313	0.390	0.524	0.925	R	All Beds Replaced with New Beds Containing Binder R
23	157.6	158.8	121.6	1.0	0.284	0.354	0.418	0.925	R	
24	154.9	158.5	101.5	1.0	0.204	0.237	0.289	0.601	R	
25	87.1	66.9	36.0	62.6	0.243	0.210	0.125	0.186	EPG	Top Bed Maintained at 75 °C throughout Run
26	114.8	121.1	101.5	57.9	0.263	0.281	0.259	0.359	EPG	Top Bed Maintained at 50 °C throughout Run

The data on water retention found in this part of the study agrees well with that found in the part of the study reported on in Section 3. It will be recalled that this section presents data obtained using insulated 1-in.-diam columns while Section 2 presents data obtained using a 2-in.-diam uninsulated column. The results on iodine loading do not show the same good agreement. Iodine loadings in this part of the study were approximately 1 g/kg of solid for LMS type 3A with binder EPG and 0.3 g/kg for LMS type 3A with binder R. Similar tests on the 2-in.-diam uninsulated column resulted in iodine retentions of 5.8 and 1.1 g/kg respectively. One explanation for this difference in results might be that the insulated bed retains the rather large amount of heat generated by the adsorption of water for a longer time and thus results in higher bed temperatures which tend to retard iodine adsorption.

6.3 Results of Cycling Test Beds

In the cycling mode of operation, three beds in sequence were run until water breakthrough occurred at the exit from the middle bed. This period was designated Phase 1. For Phase 2, the beds were changed with the original middle bed becoming the top bed, the original bottom bed becoming the middle bed, and a new bottom bed being added to the line. The original top bed was removed, sealed, and stored for analysis. The run was then continued in Phase 2 until breakthrough again occurred at the exit of the middle bed.

A total of eleven runs were made in this mode. Nine runs (16 through 24) were adiabatic of which 6 used LMS type 3A with EPG binder and 3 used LMS type 3A with R binder. Two runs (25 and 26) were made with the top bed heated. Both of these runs used LMS type 3A with binder EPG. Temperatures of the top bed were maintained at 75°C and 50°C respectively.

Overall results are shown in Table 6.3, which presents total loading and iodine loading. Since iodine loading is of the order of one gram, total

loading is essentially equal to water loading except at very low total loadings. The test conditions were essentially the same as for the earlier runs with inlet humidities being approximately 0.012 g H₂O/g air and inlet iodine concentrations being 0.0015 g/g air.

Figure 6.4 shows loading accomplished during Phase 1 and Phase 2 for the adiabatic runs. Data on the top bed in Phase 1 show a loading of approximately 18% for LMS type 3A with binder EPG with the first run being higher (19%). These values are typical of first runs with new material. LMS type 3A with binder R shows a loading of about 16% with the first run (number 22) again slightly higher at 17%.

Data on the middle bed show that approximately half of the loading takes place during Phase 1 and half during Phase 2. The total loading for this bed agrees remarkably well with the total loading for the top bed. Again, it is observed that first runs give higher loading than later runs by about 1%.

The bottom bed shows essentially no loading during Phase 1 except for the approximately 1 gram which is no doubt iodine. During Phase 2, this bed shows very similar loading to the original middle bed with loading of approximately 50% of maximum total loading observed. The type R material seems to show a slightly higher total loading than type EPG.

Figure 6.5 shows the final iodine loadings for the set of adiabatic runs. Although the results are somewhat inconsistent, it is apparent that there is a significant difference in the iodine loadings of material with binder R and binder EPG. Iodine loadings for the EPG material are approximately 0.9 g/kg of solid and for the R material are approximately 0.3 g/kg of solid.

Another observable effect is the apparent decrease in I₂ loading between top, middle and bottom beds for the EPG binder and the increase in

Fig. 6.4. Total loading for three cartridge-type beds operated cyclically.

Fig. 6.5. Iodine loading for three cartridge-type beds operated cyclically.

loading seen for type R binder. All results showed a high iodine loading on the bottom bed during Phase 2. This bed was exposed to very little water vapor and was essentially adsorbing iodine without water vapor being present.

Two anomalous points on this figure should be mentioned. Run 16 shows a much higher iodine loading on the bottom bed in Phase 2. It has been pointed out that this was a first run on new material and original runs generally seem to show higher loading results than subsequent runs on the same material. Also the last point on Run 17 shows a higher than average iodine loading. This is probably because the run was stopped prematurely because of a hygrometer failure and the bottom bed did not load with water to the extent that the same bed did in the other tests. Past results have shown that iodine loading is influenced by the amount of water loading, being less if water is present. Consequently, the lower water loading of the bed provided the opportunity for more iodine loading to take place.

6.4 Results for Heated Top Bed Runs

For these two runs using EPG binder, the top bed was maintained at 75°C (run 25) and 50°C (run 26). This was accomplished by wrapping electrical heating tapes around the top bed and manually adjusting the voltage to the heating tapes so as to maintain the desired temperature as indicated by a thermocouple inserted about mid-way into the top bed. During a test, the temperature typically varied $\pm 5^\circ\text{C}$. The output of a second thermocouple, located at the exit of the middle bed, was also recorded.

It was intended to cycle the beds when breakthrough occurred at the middle bed, however, due to problems with the hygrometer, it is believed that breakthrough at this point was not accurately established. Abnormally high hygrometer readings were obtained and verified by high loadings on the bottom bed, a result which indicates breakthrough occurred through all 3 beds.

The reason for attempting these two runs was to see if iodine loading in the top bed (which would be disposed) could be greatly reduced without too large a loss in water loading. Even with the experimental difficulties encountered, it is believed that the results indicate that such an approach is not promising.

The results for total loading (which is essentially water loading) for the two high temperature runs are presented in Table 6.3 and in Figure 6.6. Figure 6.6 also contains data for run 20 which is judged to be a typical adiabatic run conducted at ambient conditions. Comparison of the total loading indicates a decrease from nearly 18% at 25°C to approximately 12% at 50°C and approximately 8% at 75°C. Similar results were obtained for the middle bed in Phase 1 which became the top bed in Phase 2.

Iodine loadings for the two high temperature runs are presented in Table 6.2. Iodine loadings of approximately 0.25 g/kg of solid were found for both of the runs. For the same material (LMS Type 3A, EPG binder), iodine loadings of 0.8 to 1.0 g/kg of solid were found for the runs conducted at 25°C. Thus, the effect of operating with the top bed maintained at a higher temperature was to reduce iodine loading to approximately 1/3 of values found without heating the bed; however, water loading was found to be reduced to about one-half of its value without heating.

From these data, it does not appear that a "clean separation", i.e., essentially all water being retained by the bed and essentially all iodine passing through the bed, can be obtained by simply controlling the bed temperature. It appears there will always be some retained iodine on the bed.

During the high temperature runs, the temperature of the gas exiting the middle bed was measured. These temperatures were found to be quite high, being about 100°C for the case where the top bed was maintained at 50°C, and

Fig. 6.6. Total loading for three cartridge-type beds at different top bed temperatures.

about 120°C for the top bed being maintained at 75°C. This effect is caused by the gas receiving both sensible heat from the hot bed plus the significant amount of heat received as a result of the water adsorption.

As mentioned earlier, breakthrough at the middle bed was not accurately observed during the two high temperature runs. It is apparent that breakthrough occurred in the middle bed from the relatively heavy loadings in the bottom beds as seen in Figure 6.6. It is suspected that the high gas temperatures contributed to the erratic hygrometer readings for these two runs.

Should the cartridge-type bed using external heating of the first bed approach be considered, it appears necessary that some provision for cooling the gas between beds will be necessary. Without this, subsequent beds will not operate very efficiently because of the high temperature of the gas.

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Appendix A

SUMMARY OF EXPERIMENTAL DATA

A.1 Linde AW-300 Molecular Sieves

A summary of the experimental conditions and results is presented in Table A.1.1. Results for each test run are given in Table A.1.2 and Table A.1.3.

During several regeneration runs, the bed was regenerated at a relatively low temperature until no more iodine or water was observed leaving the bed and then regeneration was completed at a high temperature (300°C). The fraction of I_2 and water that was recovered during regeneration at a low temperature were obtained. (See Table A.1.4). During each regeneration run, the entering regeneration air was at the ambient temperature.

Table A.1.4. Regeneration Characteristics of LMS AW-300.

Run Number	Low temperature of regeneration ($^{\circ}\text{C}$)	Fraction of I_2 recovered	Fraction of H_2O recovered
5	65	0.3453	0.0000
6	80	0.5221	0.1429
7	100	0.5507	0.2568
8	125	0.6767	0.3974

Table A.1.1. Summary of test results for Linde AW-300 molecular sieves

Material

Type: Linde AW-300
 Shape: 1/16-in. pellets
 Lot No.: 3940560031
 Part No.: 1044
 Base: Alumina-Silicate
 Heat of adsorption: 1450 Btu/lb H₂O
 Bulk density: 50 lb/cu. ft.

Bed

Weight of material: 1325.2 g
 Height of bed: 30.75 in.
 Diameter of bed: 2.0 in.

Operation

Number of cycles: 9
 Time for loading: 30 h
 Time for regeneration: 33 h
 Total exposure time: 276 h

Test results

Average water loading, g H₂O/kg of solid: 79.2
 Average inlet humidity, g H₂O/g air: 0.008
 Average inlet I₂ concentration, g I₂/g air: 0.0014
 Average iodine loading, g I₂/kg of solid: 1.49

Length of mass transfer zone

Run no.	Flow rate, scfm	MTZ, in.
5	108.5	5.84

Pressure drop across bed

Flow rate, scfm	Average pressure drop, in. Hg.
108.5	2.95

Table A.1.2.. Data collected on loading runs for Linde AW-300

Run	Duration of run (h)	Time of breakthrough (h)	Inlet air flow (scfh)	Inlet humidity (g H ₂ O/g air)	Inlet iodine concentration (g I ₂ /g air)	Minimum outlet dew point (°C)	Water loading at breakthrough (g/kg of solid)
1	3.3	3.3	108.5	0.0096	0.0014		89.0
2	3.5	3.5	108.5	0.0077	0.0014		74.7
3	2.8	2.8	108.5	0.0109	0.0019		83.0
4	3.3	3.3	108.5	0.0089	0.0014	-58	83.0
5	3.8	3.0	108.5	0.0094	0.0015	-53	80.3
6	3.2	3.2	108.5	0.0088	0.0015	-47	78.5
7	3.4	3.4	108.5	0.0075	0.0014	-52	70.9
8	4.0	4.0	108.5	0.0067	0.0012	-60	74.0
9	2.7	2.7	108.5	0.0000	0.0015		0.0

Table A.1.3. Data collected on regeneration runs for Linde AW-300

Run	Time of run (h)	Regeneration air flow rate (scfh)	Iodine collected (g/kg of solid)	Operating Temperature (°C)
1	3.3	9.1	0.91	25 to 270
2	3.3	9.1	1.23	25 to 270
3	3.3	9.1	1.34	25 to 270
4	3.3	9.1	1.58	25 to 270
5	3.9	9.1	1.32	25 to 340
6	3.3	9.1	1.38	25 to 310
7	3.3	9.1	2.17	25 to 270
8	3.3	9.1	1.99	25 to 310
9	6.0	9.1	4.78	25 to 340

A.2. Linde Molecular Sieve Type 5A

A summary of the experimental conditions and results is presented in Table A.2.1 . Results for each test run are given in Tables A.2.2 and A.2.3. Figures A.1.1 and A.1.2 indicate the amount of water retained on the bed as a function of time of operation and temperature of the bed during regeneration. Figure A.1.3 presents the amount of iodine retained on the bed as a function of time of operation and temperature of the bed during regeneration.

Table A.2.1 . Summary of test results for
Linde Molecular Sieve, type 5A

Material

Type: Linde Molecular Sieve Type 5A
 Shape: 1/16-in. pellets
 Lot No.: 59322667079
 Part No.: 1194
 Base: Alumina-Silicate
 Cation: Calcium form
 Heat of adsorption: 1800 Btu/lb H₂O
 Bulk density: 40.0 lb/ft³

Bed

Weight of material: 1148.5 g
 Height of bed: 30 in.
 Diameter of bed: 2.0 in.

Operation

Number of cycles: 9
 Time for loading: 79 h
 Time for regeneration: 81 h
 Total exposure time: 259 h

Test results

Average water loading, g H₂O/kg of solid: 154.9
 Average inlet humidity, g H₂O/g air: 0.0079
 Average inlet I₂ concentration, g I₂/g air: 0.0015
 Average iodine loading, g I₂/kg of solid: 21.0

Pressure drop across bed

Flow rate, scfm	Average pressure drop, in. Hg.
74.5	1.76
108.5	3.18

Table A.2.2 . Data collected on loading runs for
Linde Molecular Sieves, type 5A

Run	Duration of run (h)	Time of breakthrough (h)	Inlet air flow (scfh)	Inlet humidity (g H ₂ O/g air)	Inlet iodine concentration (g I ₂ /g air)	Minimum outlet dew point (°C)	Water loading at breakthrough (g/kg of solid)
1	9.4	9.4	74.5	0.0075	0.0000	-68	154.6
2	5.3	5.3	108.5	0.0087	0.0000	-63	149.9
3	16.5	13.1	74.5	0.0052	0.0000	-77	153.9
4	8.0	5.9	108.5	0.0078	0.0000	-66	151.6
5	5.8	5.8	108.5	0.0085	0.0014	-68	162.6
6	9.0	9.0	74.5	0.0077	0.0015	-67	156.1
7	13.1		108.5	0.0000	0.0013		
8	6.3	4.9	108.5	0.0096	0.0017		156.0
9	5.7	5.7	108.5	0.0083	0.0000	-68	154.3

Table A.2.3 . Data collected on regeneration runs for
Linde Molecular Sieves, type 5A

Run	Time of run (h)	Regeneration air flow rate (scfh)	Iodine collected (g/kg of solid)	Operating Temperature (°C)
1	8.0	9.1	0.00	25 to 350
2	5.9	9.1	0.00	25 to 350
3	6.8	9.1	0.00	25 to 350
4	6.0	9.1	0.00	25 to 350
5	6.2	9.1	21.00	25 to 350
6	8.6	9.1		25 to 350
7	18.1	9.1	55.10	25 to 350
8	15.8	9.1		25 to 350
9	6.0	9.1	0.00	25 to 350

Fig. A.2.1. Water removal during regeneration, LMS 5A, run 4.

Fig. A.2.2. Water removal during regeneration, LMS 5A, run 5

Fig. A.2.3. Iodine removal during regeneration, LMS 5A, run 6.

A.3. Linde Molecular Sieves type 3A, binder EPG, 1/8-in. pellets

A summary of the experimental conditions and results is presented in Table A.3.1. Results for each test run are given in Table A.3.2 and Table A.3.3.

During several regeneration runs, the amount of water recovered as a function of bed temperature was studied. Results obtained on run 1 are presented in Figure A.3.1. Similar results were obtained for other regeneration runs. The amount of I_2 recovered before the first drop of water was condensed was the subject of study in several additional regeneration runs. Results obtained during these runs are given below.

Run No.	Bed Temp., Top (°C)	Bed Temp., Bottom (°C)	Fraction of I_2 coming out before the first drop of water was collected in the condenser.
5	60.0	80.0	0.19
6	64.0	70.0	0.12
7	64.0	80.0	0.13
8	70.0	80.0	0.20

Table A.3.1 . Summary of test results for Linde Molecular Sieves, type 3A, 1/8-in. pellets

Material

Type: Linde Molecular Sieve, type 3A
 Shape: 1/8-in. pellets
 Binder: EPG
 Lot No.: 39403770566
 Part No.: 1030
 Base: Alumina-Silicate
 Cation: Potassium form
 Heat of adsorption: 1800 Btu/lb. H₂O
 Bulk density: 42.5 lb/ft³

Bed

Weight of material: 1059.1 g
 Height of bed: 30.0 in.
 Diameter of bed: 2.0 in.

Operation

Number of cycles: 8
 Time for loading: 65 h
 Time for regeneration: 60 h
 Total exposure time: 260 h

Test results

Average water loading, g H₂O/kg of solid: 158.3
 Average inlet humidity, g H₂O/g air: 0.0076
 Average inlet I₂ concentrations, g I₂/g air: 0.0016
 Average iodine loading, g I₂/kg of solid: 2.49

Length of mass transfer zone

Run no.	Flow rate, scfm	MTZ, in.
3	108.5	7.55

Pressure drop across bed

Flow rate, scfm	Average pressure drop, in. Hg.
74.5	1.10
108.5	2.12

Table A.3.2. Data collected on loading runs for
Linde Molecular sieves, type 3A, binder EPG, 1/8-in. pellets

Run	Duration of run (h)	Time of breakthrough (h)	Inlet air flow (scfh)	Inlet humidity (g H ₂ O/g air)	Inlet iodine concentration (g I ₂ /g air)	Minimum outlet dew point (°C)	Water loading at breakthrough (g/kg of solid)
1	5.7	5.7	108.5	0.0075	0.0000	-62	148.3
2	10.0	10.0	74.5	0.0075	0.0000	-74	179.8
3	12.1	8.8	74.5	0.0076	0.0000	-67	159.0
4	8.3	5.5	108.5	0.0077	0.0000	-64	147.9
5	5.9	5.9	108.5	0.0074	0.0013	-69	153.1
6	9.2	9.2	74.5	0.0075	0.0016	-63	164.8
7	5.5	5.5	108.5	0.0080	0.0016	-65	153.2
8	8.8	8.8	74.5	0.0077	0.0020	-57	160.0

Table A.3.3. Data collected on regeneration runs for
Linde Molecular Sieves, type 3A, binder EPG, 1/8-in. pellets

Run	Time of run (h)	Regeneration air flow rate (scfh)	Iodine collected (g/kg of solid)	Operating Temperature (°C)
1	6.5	9.1	0.00	25 to 360
2	7.3	9.1	0.00	25 to 360
3	6.6	9.1	0.00	25 to 360
4	6.0	9.1	0.00	25 to 360
5	8.8	9.1	1.69	25 to 360
6	8.3	9.1	3.02	25 to 360
7	8.2	9.1	2.54	25 to 360
8	8.3	9.1	2.69	25 to 360

Fig. A.3.1. Water removal during regeneration for Linde Molecular Sieves type 3A, Binder EPG, 1/8-in. pellets, run 1.

A. 4. Davison 3 Å Molecular Sieves

A summary of the experimental conditions and results is presented in Table A.4.1 . Results for each test run are given in Table A.4.2 and Table A.4.3.

During several regeneration runs, the amount of water coming out as a function of bed temperature was studied. Results obtained on run 1 are presented in Figure A.4.1 . Similar results were obtained for other runs. During regeneration runs 5 and 6, the fractional amount of I_2 that was removed by the time the first drop of water was collected in the condenser was measured. Results obtained during this study are given below.

Run No.	Top bed Temp. °C	Bottom bed Temp. °C	Fraction of I_2 removed before any water was collected in the condenser.
5	78.0	76.0	0.112
6	76.0	75.0	0.128

Table A.4.1 . Summary of test results for Davison 3A

Material

Type: Davison 3A molecular sieves
 Shape: Spherical
 Size: 4-8 mesh
 Grade: 562
 Drum no.: 3241
 Color: White

Bed

Weight of material: 1155.3 g
 Height of bed: 30.0 in.
 Diameter of bed: 2.0 in.

Operation

Number of cycles: 10
 Time for loading: 84 h
 Time for regeneration: 74 h
 Total exposure time: 356 h

Test results

Average water loading, g H₂O/kg of solid: 160.0
 Average inlet humidity, g H₂O/g air: 0.0081
 Average inlet I₂ concentration, g/I₂/g air: 0.0016
 Average iodine loading, g I₂/kg of solid: 2.74

Length of mass transfer zone

Run no.	Flow rate, scfm	MTZ, in.
3	108.5	9.68
7	74.5	7.95

Pressure drop across bed

Flow rate, scfm	Average pressure drop, in. Hg.
74.5	0.83
108.5	1.67

Table A.4.2 ... Data collected on loading runs for Davison 3A

Run	Duration of run (h)	Time of breakthrough (h)	Inlet air flow (scfh)	Inlet humidity (g H ₂ O/g air)	Inlet iodine concentration (g I ₂ /g air)	Minimum outlet (°C)	Water loading at breakthrough (g/kg of solid)
1	6.0	6.0	108.5	0.0083	0.0000	-55	159.2
2	9.3	9.3	74.5	0.0083	0.0000	-61	166.7
3	8.5	5.8	108.5	0.0083	0.0000	-58	152.2
4	9.4	9.4	74.5	0.0081	0.0000	-61	167.7
5	6.2	6.2	108.5	0.0081	0.0016	-69	158.6
6	9.9	9.9	74.5	0.0077	0.0020	-55	167.5
7	13.4	9.5	74.5	0.0079	0.0000	-64	164.7
8	5.8	5.8	108.5	0.0085	0.0000	-66	157.3
9	5.6	5.6	108.5	0.0080	0.0012	-62	142.0
10	10.3	10.3	74.5	0.0073	0.0015	-66	163.8

Table A.4.3 . Data collected on regenerations runs for Davison 3A

Run	Time of run (h)	Regeneration air flow rate (scfh)	Iodine collected (g/kg of solid)	Operating Temperature (°C)
1	6.3	9.1	0.00	25 to 380
2	7.7	9.1	0.00	25 to 380
3	6.6	9.1	0.00	25 to 380
4	7.4	9.1	0.00	25 to 380
5	8.8	9.1	2.65	25 to 380
6	7.4	9.1	2.58	25 to 380
7	7.0	9.1	0.00	25 to 380
8	7.8	9.1	0.00	25 to 380
9	7.5	9.1	2.38	25 to 380
10	7.7	9.1	3.35	25 to 380

Fig. A.4.1. Water removal during regeneration, Davison 3A molecular sieves, run 1.

A.5. Norton Zeolon-500

A summary of the experimental conditions and results is presented in Table A.5.1 . Results for each test run are given in Table A.5.2 and Table A.5.3 . Figure A.5.1 indicates the amount of water retained on the bed as a function of time of operation and temperature of the bed during regeneration.

Table A.5.1. Summary of test results for Norton Zeolon-500

Material

Type: Norton Zeolon-500

Shape: 1/16-in. pellets

Pore size: 4 to 5 Å

Bed

Weight of material: 930.1 g

Height of bed: 30.0 in.

Diameter of bed: 2.0 in.

Operation

Number of cycles: 6

Time for loading: 36 h

Time for regeneration: 37 h

Total exposure time: 187 h

Test resultsAverage water loading, g H₂O/kg of solid: 131.8Average inlet humidity, g H₂O/g air: 0.0082Average inlet I₂ concentration, g I₂/g air: 0.0018Average iodine loading, g I₂/kg of solid: 24.5Length of mass transfer zone

Run no.	Flow rate, scfm	MTZ, in.
3	108.5	6.41
4	74.5	5.17

Pressure drop across bed

Flow rate, scfm	Average pressure drop, in. Hg.
74.5	1.17
108.5	2.27

Table A.5.2 . Data collected on loading runs for Norton Zeolon-500

Run	Duration of run (h)	Time of breakthrough (h)	Inlet air flow (scfh)	Inlet humidity (g H ₂ O/g air)	Inlet iodine concentration (g I ₂ /g air)	Minimum outlet dew point (°C)	Water loading at breakthrough (g/kg of solid)
1	3.7	3.7	108.5	0.0087	0.0000	-64	118.8
2	6.7	6.7	74.5	0.0081	0.0000	-62	137.4
3	6.3	4.6	108.5	0.0082	0.0000	-68	137.5
4	8.7	7.0	74.5	0.0078	0.0000	-58	136.8
5	4.3	4.3	108.5	0.0082	0.0017	-64	128.7
6	6.5	6.5	74.5	0.0080	0.0020	-64	131.8

Table A.5.3. Data collected on regeneration runs for Norton Zeolon-500

Run	Time of run (h)	Regeneration air flow rate (scfh)	Iodine collected (g/kg of solid)	Operating Temperature (°C)
1	6.3	9.1	0.00	25 to 370
2	6.6	9.1	0.00	25 to 370
3	6.3	9.1	0.00	25 to 370
4	6.3	9.1	0.00	25 to 370
5	6.3	9.1	24.49	25 to 370
6	3.7	9.1		25 to 370

Fig. A.5.1. Water removal during regeneration, Norton Zeolon-500, run 1.

A.6. Drierite

A summary of the experimental conditions and results is presented in Table A.6.1. Results for each test run are given in Table A.6.2 and Table A.6.3. Figure A.6.1 indicates the amount of water retained on the bed as a function of time of operation and temperature of the bed during regeneration.

Table A.6.1. Summary of test results for Drierite

MaterialType: Drierite (Ca_2SO_4)

Shape: 8 mesh

Supplier: W. A. Hammond Drierite Company, Xenia, Ohio.

BedOperation

Weight of material: 1629.2 g

Number of cycles: 10 h

Height of bed: 30.0 in.

Time for loading: 13 h

Diameter of bed: 2.0 in.

Time for regeneration: 35 h

Total exposure time: 105 h

Test resultsAverage water loading, g H_2O /kg of solid: 13.78Average inlet humidity, g H_2O /g air: 0.0084Average inlet I_2 concentration, g I_2 /g air: 0.0014Average iodine loading, g I_2 /kg of solid: 0.32Length of mass transfer zone

Run no.	Flow rate, scfm	MTZ, in.
3	108.5	28.8
4	74.5	18.7

Pressure drop across bed

Flow rate, scfm	Average pressure drop, in. Hg.
108.5	2.1
74.5	1.2

Table A.6.2 . Data collected on loading runs for Drierite

Run	Duration of run (h)	Time of breakthrough (h)	Inlet air flow (scfh)	Inlet humidity (g H ₂ O/g air)	Inlet iodine concentration (g I ₂ /g air)	Minimum outlet dew point (°C)	Water loading at breakthrough (g/kg of solid)
1	2.3	1.4	108.5	0.0070	0.0000	-67	22.0
2	1.8	0.8	108.5	0.0079	0.0000		14.8
3	1.0	0.7	108.5	0.0095	0.0000		13.9
4	1.1	1.1	74.5	0.0084	0.0000		13.9
5	0.9	0.9	74.5	0.0092	0.0000		12.5
6	1.2	1.2	74.5	0.0085	0.0000	-63	15.4
7	1.1	1.1	74.5	0.0073	0.0014	-67	12.8
8	0.7	0.7	108.5	0.0083	0.0014	-68	12.2
9	1.2	0.5	108.5	0.0095	0.0000	-29	10.0
10	1.6	.8	74.5	0.0084	0.0000	-39	10.4

Table A.6.3 . Data collected on regeneration runs for Drierite

Run	Time of run (h)	Regeneration air flow rate (scfh)	Iodine collected (g/kg of solid)	Operating Temperature (°C)
1				
2	4.0	9.1	0.00	25 to 320
3	2.9	9.1	0.00	25 to 320
4	3.1	9.1	0.00	25 to 320
5	3.1	9.1	0.00	25 to 320
6	3.6	9.1	0.00	25 to 320
7	3.8	9.1	0.36	25 to 320
8	3.3	9.1	0.28	25 to 320
9	3.3	9.1	0.00	25 to 320
10	4.0	9.1	0.00	25 to 320

Fig. A.6.1. Water removal during regeneration, Drierite, run 4.

A.7. Alumina

A summary of the experimental conditions and results is presented in Table A.7.1. Results for each test run are given in Tables A.7.2 and A.7.3.

During several regenerations runs, the amount of water coming out as a function of bed temperature was studied. Results obtained on run 2 are presented in Figure A.7.1. Similar results were obtained for other runs. During regeneration run 7, 20.4 g of iodine came out by the time the first drop of water was collected in the receiver. Corresponding bottom and top temperatures in the bed were 60°C and 82°C respectively.

Table A.7.1 . Summary of test results for Alumina

Material

Type: Alumina (Al_2O_3)
 Size: 6 to 10 mesh
 Source: Fisher Scientific Company

Bed

Weight of material: 1218.1
 Height of bed: 24.6 in.
 Diameter of bed: 2.0 in.

Operation

Number of cycles: 7
 Time for loading: 46 h
 Time for regeneration: 45 h
 Total exposure time: 208 h

Test results

Average water loading, g H_2O /kg of solid: 104.9
 Average inlet humidity, g H_2O /g air: 0.0078
 Average inlet I_2 concentration, g I_2 /g air: 0.0017
 Average iodine loading, g I_2 /kg of solid: 6.8

Length of mass transfer zone

Run no.	Flow rate, scfm	MTZ, in.
3	108.5	10.0

Pressure drop across bed

Flow rate, scfm	Average pressure drop, in. Hg.
74.5	1.05
108.5	1.85

Table A.7.2. Data collected on loading runs for Alumina

Run	Duration of run (h)	Time of breakthrough (h)	Inlet air flow (scfh)	Inlet humidity (g H ₂ O/g air)	Inlet iodine concentration (g I ₂ /g air)	Minimum outlet dew point (°C)	Water loading at breakthrough (g/kg of solid)
1	5.6	5.6	74.5	0.0085	0.0000	-56	97.5
2	4.2	4.2	108.5	0.0082	0.0000	-56	103.0
3	7.5	4.9	108.5	0.0076	0.0000	-60	112.8
4	10.5	8.1	74.5	0.0067	0.0000	-60	112.6
5	3.3	3.3	108.5	0.0092	0.0022	-59	90.4
6	7.0	7.0	74.5	0.0072	0.0017	-62	103.3
7	7.8	7.8	74.5	0.0071	0.0013	-67	114.4

Table A.7.3 . Data collected on regeneration runs
for Alumina

Run	Time of run (h)	Regeneration air flow rate (scfh)	Iodine collected (g/kg of solid)	Operating Temperature (°C)
1	5.5	9.1	0.00	25 to 320
2	6.0	9.1	0.00	25 to 320
3	6.4	9.1	0.00	25 to 320
4	6.6	9.1	0.00	25 to 370
5	7.2	9.1	6.74	25 to 370
6	5.3	9.1	6.10	25 to 370
7	7.6	9.1	7.63	25 to 370

Fig. A.7.1. Water removal during regeneration, Alumina, run 2

A.8. Linde Molecular Sieves type 3A, binder EPG, 1/16-in. pellets

A summary of the experimental conditions and results is presented in Table A.8.1. Results for each test run are given in Table A.8.2 and Table A.8.3.

Iodine concentration in the outlet stream during the loading runs was found to exceed the inlet concentration over a short period of time. Iodine loading for 1/16-in. pellets are almost double of that for 1/8-in. pellets of the same material. This could be because of the larger surface area available per unit volume in the case of the 1/16-in. pellets.

During several regeneration runs, the amount of water coming out as a function of bed temperature was studied. Results obtained on run 1 are presented in Figure A.8.1. Similar results were obtained for other regeneration runs.

During several regeneration runs, the amount of iodine and water coming out at a certain regeneration temperature was studied. Regeneration was first carried out for a sufficient period of time at a temperature of the study until no more iodine was observed coming out. Regeneration was then completed at 370°C. Results obtained are presented in the following table.

Regeneration temperature (°C)	Percent of I ₂ removed.	Percent of H ₂ O removed.	Time for which the bed was regenerated at the regeneration temperature, min.
21	2.798	0.0	60.0
60		19.57	350.0
80		21.85	325.0
95	48.99	28.66	180.0
120	46.43	53.38	215.0
145	52.33	75.16	295.0

Table A.8.1 . Summary of test results for Linde Molecular Sieves, type 3A, binder EPG, 1/16-in. pellets

Material

Type: Linde Molecular Sieves, type 3A
 Shape: 1/16-in. pellets
 Binder: EPG (new)
 Lot No.: 3944278
 Part No.: 1016
 Base: Alumina-Silicate
 Cation: Potassium Form
 Heat of adsorption: 1800 Btu/lb H₂O
 Bulk density: 42.5 lb/cu. ft.

Bed

Weight of material: 1090.8 g
 Height of bed: 30.0 in.
 Diameter of bed: 2.0 in.

Operation

Number of cycles: 12
 Time for loading: 96 h
 Time for regeneration: 110 h
 Total exposure time: 748 h

Test results

Average water loading, g H₂O/kg of solid: 188.5
 Average inlet humidity, g H₂O/g air: 0.0082
 Average inlet I₂ concentrations, g I₂/g air: 0.0016
 Average iodine loading, g I₂/kg of solid: 5.80

Length of mass transfer zone

Run No.	Flow rate, scfm	MTZ, in.
8	108.5	3.54

Pressure drop across bed

Flow rate, scfm	Average pressure drop, in. Hg.
74.5	1.17
108.5	3.37

Table A.8.2 . Data collected on regeneration runs for
Linde Molecular Sieves, type 3A, binder EPG

Run	Time of run (h)	Regeneration air flow rate (scfh)	Iodine collected (g/kg of solid)	Operating Temperature (°C)
1	6.4	9.1	0.00	25 to 370
2	7.0	9.1	0.00	25 to 370
3	7.4	9.1	5.42	25 to 370
4	13.0	9.1	6.58	25 to 370
5	10.8	9.1	6.18	25 to 370
6	13.3	9.1	7.23	25 to 370
7	7.8	9.1	0.00	25 to 370
8	5.8	9.1	0.00	25 to 370
9	12.3	9.1	4.51	25 to 370
10	6.5	9.1	0.00	25 to 370
11	8.0	9.1	5.13	25 to 370
12	12.1	9.1	5.57	25 to 370

Table A.8.3 . Data collected on loading runs for
Linde Molecular Sieves; type 3A, binder EPG

Run	Duration of run (h)	Time of breakthrough (h)	Inlet air flow (scfh)	Inlet humidity (g H ₂ O/g air)	Inlet iodine concentration (g I ₂ /g air)	Minimum outlet dew point (°C)	Water loading at breakthrough (g/kg of solid)
1	5.9	5.9	108.5	0.0089	0.0000	-63	177.2
2	8.8	8.8	74.5	0.0093	0.0030	-63	189.7
3	7.4	7.4	108.5	0.0077	0.0013	-67	191.3
4	7.2	7.2	108.5	0.0077	0.0015	-63	186.8
5	7.1	7.1	108.5	0.0079	0.0014		188.0
6	11.5	11.5	74.5	0.0071	0.0017	-63	188.9
7	8.3	7.2	108.5	0.0080	0.0000	-58	192.5
8	6.9	5.8	108.5	0.0095	0.0000	-55	186.8
9	6.8	6.5	108.5	0.0086	0.0018	-63	187.0
10	12.3	11.2	74.5	0.0076	0.0000	-58	196.3
11	7.4	7.4	108.5	0.0077	0.0014	-70	191.9
12	6.7	6.7	108.5	0.0082	0.0018	-63	185.4

Fig. A.8.1. Water removal during regeneration for Linde Molecular Sieves type 3A, binder EPG, 1/16-in. pellets, run 1.

A.9. Linde Molecular Sieves type 3A, binder R, 1/16-in. pellets

A summary of the experimental conditions and results is presented in Table A.9.1. Results for each test run are given in Table A.9.2 and Table A.9.3.

During several regeneration runs, the amount of water coming out as a function of bed temperature was studied. The results obtained during run 1 are presented in Figure A.9.1. Similar results were obtained for other runs.

During several regeneration runs, the amount of iodine and water coming out at a certain regeneration temperature was studied. Regeneration was first carried out for a sufficient period of time at the desired temperature of the study until no more iodine was observed coming out. Regeneration was then completed at 370°C. Results obtained are presented below.

Regeneration temperature (°C)	Percent of I ₂ removed.	Percent of H ₂ O removed.	Time for which the bed was regenerated at the regeneration temperature, min.
60	22.94	9.3	180.0
80	27.17	24.26	280.0
100	25.50	28.47	250.0

Table A.9.1 . Summary of test results for Linde Molecular Sieves,
type 3A, binder R

Material

Type: Linde Molecular Sieves, type 3A
 Shape: 1/16-in. pellets
 Binder: R
 Lot no.: 39457757105
 Base: Alumina-Silicate
 Cation: Potassium form
 Heat of adsorption: 1800 Btu/lb H_2O
 Bulk density: 42.5 lb/ft³

Bed

Weight of material: 1149.6 g
 Height of bed: 30.0 in.
 Diameter of bed: 2.0 in.

Operation

Number of cycles: 8

Test results

Average water loading, g H_2O /kg of solid: 153.9
 Average inlet humidity, g H_2O /g air: 0.0082
 Average inlet I_2 concentrations, g I_2 /g air: 0.0016
 Average iodine loading, g I_2 /kg of solid: 1.1165

Length of mass transfer zone

Run no.	Flow rate, scfm	MTZ, in.
3	108.5	4.16
4	74.5	3.70

Pressure drop across bed

Flow rate, scfm	Average pressure drop, in. Hg.
74.5	1.61
108.5	3.00

Table A.9.2 . Data collected on loading runs for
Linde Molecular Sieves, type 3A, binder R

Run	Duration of run (h)	Time of breakthrough (h)	Inlet air flow (scfh)	Inlet humidity (g H ₂ O/g air)	Inlet iodine concentration (g I ₂ /g air)	Minimum outlet dew point (°C)	Water loading at breakthrough (g/kg of solid)
1	6.6	6.6	108.5	0.0083	0.0000	-66	174.6
2	8.7	8.7	74.5	0.0083	0.0000	-70	159.7
3	7.1	6.2	108.5	0.0080	0.0000	-74	158.4
4	5.9	5.9	108.5	0.0079	0.0015	-69	149.7
5	10.0	8.3	74.5	0.0083	0.0000	-65	149.6
6	5.7	5.7	108.5	0.0081	0.0016	-67	148.5
7	5.3	5.3	108.5	0.0086	0.0017	-67	145.7
8	5.4	5.4	108.5	0.0084	0.0017	-66	145.3

Table A.9.3 . Data collected on regeneration runs for

Linde Molecular Sieves, type 3A, binder R

Run	Time of run (h)	Regeneration air flow rate (scfh)	Iodine collected (g/kg of solid)	Operating Temperature (°C)
1	6.2	9.1	0.00	25 to 370
2	6.1	9.1	0.00	25 to 370
3	6.1	9.1	0.00	25 to 370
4	8.1	9.1	0.91	25 to 370
5	6.0	9.1	0.00	25 to 370
6	8.2	9.1	1.16	25 to 370
7	7.2	9.1	1.42	25 to 370
8	6.4	9.1	1.17	25 to 370

Fig. A.9.1. Water removal during regeneration for Linde Molecular Sieves type 3A binder R, run 1.

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