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Trapping of Fission Product Iodine with Silver Impregnated Molecular Sieves

J.G. Wilhelm





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TRAPPING OF FISSION PRODUCT IODINE WITH SILVER IMPREGNATED MOLECULAR SIEVES

J. G. Wilhelm

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TABLE OF CONTENTS

INTRODUCTION 2 EXPERIMENTS WITH UNIMPREGNATED MOLECULAR EXPERIMENTS WITH Ag-IMPREGNATED MOLECULAR SIEVE IN WET AIR Removal Efficiency as a Function of the Relative Humidity of the Sweep Gas 4 Removal Efficiency for Higher Concen-METHYL IODINE REMOVAL EFFICIENCY OF Ag-IMPREGNATED MOLECULAR SIEVE LINDE 13 X AT ELEVATED TEMPERATURES Experiments with Dry Air as the Sweep Gas ... 5 Experiments with Dry Helium as the Sweep Conclusions: Runs with Dry Gases at High AGING OF Ag-IMPREGNATED MOLECULAR SIEVE LINDE 13 X IN SUPERHEATED STEAM AT HIGH EXPERIMENTS WITH DIFFERENT SILVER LOADINGS OF THE MOLECULAR SIEVE 8

Page

LIST OF TABLES

Page

<u>No.</u>	Title	Page
I	CH ₃ ¹³¹ I Removal Efficiency of Unimpregnated Molecular Sieve in Wet Air	10
II	CH ₃ ¹³¹ I Removal Efficiency of Ag-Impregnated Molecular Sieve Linde 13 X in Air of Different Relative Humidities	11
III	CH ₃ ¹³¹ I Removal Efficiency of Ag-Impregnated Molecular Sieve for Different Methyl Iodide Concentrations and Loadings	. 12
IV	CH ₃ ¹³¹ I Removal Efficiency of Ag-Impregnated Molecular Sieve in Dry Air at 200, 300 and 650°C	• 13
V	CH ₃ ¹³¹ I Removal Efficiency of Ag-Impregnated Molecular Sieve in Dry Helium at 200 and 300 [°] C	• 14
VI	CH ₃ ¹³¹ I Removal Efficiency of Ag-Impregnated Molecular Sieve before and after Aging in Superheated Steam	. 15

LIST OF FIGURES

<u>No.</u>	Title	Page
1	Penetration of CH ₃ ¹³¹ I through Linde Molecular Sieve Type 13 X	16
2	Penetration of CH ₃ ¹³¹ I through Ag-Impregnated Molecular Sieve Linde 13 X as a Function of the Relative Humidity	17
3	Penetration of CH ₃ ¹³¹ I through Ag-Impregnated Molecular Sieve Linde 13 X as a Function of the Bed Depth	18
4	CH ₃ ¹³¹ I Removal Efficiency of Molecular Sieve Linde 13 X as a Function of the Ag-Impregnation, 70 % R.H.	19
5	CH ₃ ¹³¹ I Removal Efficiency of Molecular Sieve Linde 13 X as a Function of the Ag-Impregnation, 100 % R.H.	20

- III -

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ABSCHEIDUNG VON SPALTJOD MIT SILBERIMPRÄGNIERTEN MOLEKULARSIEBEN

Zusammenfassung

Durch Brennbarkeit und relativ niedrige Desorptionstemperaturen wird der Einsatz von imprägnierten Aktivkohlen zur Abscheidung von Spaltjod aus den Abgasen kerntechnischer Installationen eingeschränkt.

Treten höhere Abgastemperaturen auf oder muß mit einer starken Erwärmung des Adsorbermaterials durch die Zerfallswärme von radioaktiven Spaltprodukten gerechnet werden, sollten temperaturfeste, nicht brennbare Adsorbermaterialien für Spaltjodfilter verwendet werden.

Mit silberimprägnierten Molekularsieben konnten hohe Abscheidegrade für Methyljodid und elementares Jod bei realtiven Luftfeuchten bis zu 100 % erreicht werden. Die Abscheideleistung dieses Materials wurde in trockener Luft, in Dampf-Luftgemischen und Helium untersucht; in trockener Luft bei Temperaturen bis zu 650°C. Weiter wurden Alterungsversuche in Héißdampf durchgeführt.

Für ¹³¹J in Form von Methyljodid werden experimentell gefundene Abscheidegrade in Abhängigkeit von Art, Feuchte und Temperatur des Trägergases, der Silberimprägnierung und Bett-Tiefe der Molekularsieb-Filter (bzw. Verweilzeit) und der Beladung mit Methyljodid angegeben. Der Einsatz von silberimprägnierten Molekularsieben für die Reinigung der Sicherheitsbehälter-Atmosphäre wird diskutiert.

Abstract

The use of impregnated charcoal for the removal of fission product iodine is limited by relatively low desorption temperatures. In oxidizing gases, also ignition of the charcoal could occur. For trapping of fission product iodine in gases at high temperature and in the case of dangerous heat generation from fission product decay a nonflammable heat resistant adsorber material should be available for filtering devices.

High removal efficiencies for methyl iodide and elementary iodine in air with relative humidities up to 100 % were measured by filter beds made out of silver impregnated molecular sieve. Long time experiments were performed to test the removal efficiency of this material in dry air, steam-air mixtures, steam and helium; for air at temperatures up to 650° C. Experimental data are given for the CH_3^{131} I removal efficiency of silver impregnated molecular sieve Linde 13 X as a function of the type, humidity and temperature of the sweep gas, the amount of silver used for impregnation, bed depth of the molecular sieve (and stay time, respectively), and the loading level of methyl iodide. The use of the molecular sieve in gaseous reactor coolants and for the cleanup of containment atmospheres is discussed.

INTRODUCTION

The different types of Linde molecular sieves are well known for their selective adsorption ability. For elementary iodine and methyl iodide molecular sieve type Linde 13 X in particular shows good adsorption ability, but no useful removal efficiencies can be obtained when co-adsorption of water occurs. Because molecular sieves are excellent drying agents, this is valid for practically all purposes of off-gas cleaning.

By Ag-impregnation of the molecular sieve the removal efficiency can be much increased by providing a partner for chemical reaction in the molecular sieve. It was first shown by W. J. MAECK et al. ¹⁾ that elementary iodine and methyl iodide can be removed from gas streams with high efficiencies by the use of Ag-impregnated molecular sieves.

The work reported here is performed to get more information for the practical use of fission product iodine removal from off-gas streams. The method and the apparatus used are reported in detail elsewhere²⁾. To measure the removal efficiency for methyl iodide and elementary iodine, they were mixed with ¹³¹I in form of tracer amounts of $CH_3^{131}I$ and $^{131}I_2$, respectively. The radioactively labeled material was introduced into a sweep gas stream and sucked through successive test beds of the molecular sieve. The methyl iodide or elementary iodine which penetrated the test beds was trapped downstream in 7 successive charcoal beds (safety beds) made of KI-impregnated charcoal. Complete removal in the safety beds was assured by a stay time of about 1 sec and a decreased relative humidity of the sweep gas by heating of the safety beds.

After the run, the ^{131}I activity in the test beds and in the safety beds was measured by γ -spectroscopy. The removal efficiency was calculated from the ^{131}I activity in the test beds compared with the total activity in the test and safety beds.

- 2 -

- 3 -

The molecular sieve Linde 13 X:

$$Na_{86} \left[(Alo_2)_{86} (Sio_2)_{106} \right] \cdot x H_2^0$$

was impregnated with silver by ion exchange of the Na⁺ with Ag⁺. Mostly 1 n AgNO₃ solutions were used for the ion exchange.

Some previous runs not reported here were performed with elementary iodine. Because the removal efficiency for elementary iodine was always much higher than for methyl iodide and the removal of elementary iodine is obviously no problem compared with the removal of methyl iodide, the first experimental work reported here was concentrated on the removal of methyl iodide from gas streams.

EXPERIMENTS WITH UNIMPREGNATED MOLECULAR SIEVE LINDE 13 X

For comparison some experiments were performed with unimpregnated molecular sieve Linde 13 X and dry and wet air as a sweep gas. There was no useful removal efficiency for methyl iodide when the molecular sieve was allowed to reach water adsorption equilibrium with the normal laboratory air (R.H. 30 - 60 %) and at higher relative humidities. Only dry molecular sieve (≤ 2 wt.% H₂O as delivered) showed reasonable adsorption of methyl iodide. In these experiments with dry molecular sieve, also relatively dry air was used and the time of gas flow was short enough to limit the total uptake of water by the molecular sieve to less than 4 wt.%.

In Figure 1 the results are given of two short runs with dry molecular sieve and one run at 85 % R.H. In this case (Table I), the molecular sieve is in equilibrium with the high humidity of the sweep gas. From the data given in Table I it can be concluded that only a very small amount of methyl iodide is still adsorbed in the test beds after 17 h of wet gas flow (gas flow through test beds: 440 1/h). This is important with respect to the results of methyl iodide removal experiments with Agimpregnated molecular sieve Linde 13 X. The additional wet sweep gas flow through the test beds in the following experiments is continued for ≥ 20 h, hence, practically all of the physically adsorbed methyl iodide should be desorbed from the test beds at the end of the experiments and only chemisorbed iodine is expected to remain in the Ag-impregnated molecular sieve.

EXPERIMENTS WITH AG-IMPREGNATED MOLECULAR SIEVE IN WET AIR

Removal Efficiency as a Function of the Relative Humidity of the Sweep Gas

Some runs were performed to test the removal efficiency of silver impregnated molecular sieve for methyl iodide in wet air. Low loadings with methyl iodide were used in this series of experiments because the point of interest was the influence of the relative humidity on the removal efficiency. The relative humidity of the sweep gas was varied from 25 -100 % in steps of 15 %. The time for pre-humidification of the molecular sieve was long enough to reach adsorption equilibrium with the water content of the sweep gas. The results of these runs are given in Table II and Figures 2 and 3. Most of the variations given in Figure 2 for the penetration of $CH_3^{131}I$ at 70 % R.H. are due to the use of different batches of Ag-impregnated molecular sieve Linde 13 X.

Tab. II shows relatively good removal efficiencies up to 100 % R.H. In this region of very low CH_3 I-loadings, no effect of the total amount or concentration of the methyl iodide on the removal efficiency (straight lines in Figure 3) is to be seen, the important parameter is the relative humidity.

The amount of Ag-impregnated molecular sieve in an air cleaning system is very much limited by the high price of silver. For air cleaning purposes the maximum relative humidity should be 70 % to save Ag-impregnated molecular sieve. By heating the off-gas with originally higher humidity this condition can be reached easily. Removal Efficiency for Higher Concentrations and Loadings

- 5 -

In a series of experiments performed with air of 30° C and 70 % R.H. as the sweep gas, the removal efficiency of Ag-impregnated molecular sieve was tested in the concentration range between 0.02 and 10 mg CH₃I/m³ air and with loadings up to 6.7 mg CH₃I per g Ag-impregnated molecular sieve (calculated for 10 cm bed depth). The results are given in Table III.

The removal efficiency decreases slowly with increasing loadings of the molecular sieve. This may be the result of the decreasing amount of silver available for reaction with iodine from methyl iodide. Within the accuracy of the experiments this effect is to be seen on the removal efficiencies given for loadings above 1,5 mg CH₃I/g Ag-impregnated molecular sieve (calculated for 10 cm bed depth). For practical purposes in the region of lower loadings, the removal efficiency can be assumed to be independent of loading. In the range investigated the experimental results do not show a pronounced effect of the concentration of the methyl iodide in the sweep gas.

Most of the ranges of loading and concentration expected for off-gas cleaning in nuclear reactor stations is covered by the given data. For off-gas cleaning in fuel reprocessing plants the maximum loading of the molecular sieve should be known. Hence, the experimental work will be continued toward higher loadings and with sweep gas containing NO and NO₂.

METHYL IODIDE REMOVAL EFFICIENCY OF Ag-IMPREGNATED MOLECULAR SIEVE LINDE 13 X AT ELEVATED TEMPERATURES

Experiments with Dry Air as the Sweep Gas

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Three runs were performed to test the removal of methyl iodide from air at 200, 300, and 650° C. The results are given in Table IV. The removal efficiency is > 99.99 % under these experimental conditions.

Experiments with Dry Helium as the Sweep Cas

Two runs were performed at 200 and 300° C (Table V). Technical grade helium was used with 99.995 % He, 10 vpm O_2 , 10 vpm H_2O , and 25 vpm N_2 . The data for the removal efficiencies from the He-experiments are very similar to those with air as the sweep gas. The different results for 200 and 300° C could be due to small mechanical leaks in the first test bed used for run 1. For removal efficiencies that high the data will be influenced strongly by small mechanical leaks.

Conclusions: Runs with Dry Gases at High Temperatures

The Ag-impregnated molecular sieve Linde 13 X gives excellent removal efficiencies for methyl iodide in dry air and helium at high temperatures.

From the distribution of a small amount of 131 I activity absorbed inside the glass tube, which is used as the wall material for the test beds, and from the activity removed by the stainless steel screens in front of the test beds the conclusion can be drawn that some part of the methyl iodide thermally decomposed to a more reactive chemical form like elementary iodine. The stay time of the sweep gas - methyl iodide mixture in the hot zone of the glass tube in front of the test bed was ≤ 0.8 sec.

AGING OF Ag-IMPREGNATED MOLECULAR SIEVE LINDE 13 X IN SUPERHEATED STEAM AT HIGH TEMPERATURE AND PRESSURE

The aging experiments in steam of high temperature and pressure should give an answer to the question of whether Agimpregnated molecular sieve could be used as a filter material to remove iodinecontinuously from superheated steam in primary

- 6 -

circuits. Also some conclusions should be derived from this experiments with respect to the service life under less rough steam conditions.

Ag-impregnated molecular sieve Linde 13 X was brought into a loop for generation of superheated steam, the boiler was warmed up and the steam flow through the molecular sieve started. During startup and after slowdown of the steam flow, condensation occurred in the molecular sieve bed. So, washout of the impregnation by water and steam could cause a loss of Ag from the molecular sieve. Runs were performed for aging the molecular sieve in superheated steam at 235° C, 30 atm and 510° C, 140 atm. After aging, the molecular sieve material was brought into the test apparatus and the remaining CH₃I removal efficiency was measured at 70 % relative humidity and 30° C (Table VI). The results of the runs with steam aged molecular sieve therefore can be directly compared with previous data.

With respect to the low removal efficiencies after aging (Table VI) it can be concluded that Ag-impregnated molecular sieve of this type cannot be used in steam of very high temperature and pressure for a convenient service time.

The molecular sieve aged at 510°C lost some of its silver during the aging test, the conductivity of the steam condensate showed a steep increase during the test. After the aging procedure the gray color of the unused molecular sieve had changed to brown, the material was very brittle. Microscopic inspection showed a rough spongy structure of the pellets compared to the denser material before aging. No measurable water adsorption of the aged and then dried molecular sieve was detected in humid atmosphere. It is believed that the crystalline structure of the molecular sieve is destroyed completely by the overheated steam.

The aging tests at 230°C and 30 atm did not change the Ag-impregnated molecular sieve so much. Hence, the experiments will be continued with superheated steam in the range between 150 and 300°C; also, experiments will be performed with overheated steam at much lower pressure.

- 7 -

EXPERIMENTS WITH DIFFERENT SILVER LOADINGS OF THE MOLECULAR SIEVE

The results of some experiments are given in Figure 4 and 5. There is a sharp decrease of removal efficiency with lower silver loadings and a certain amount of "non-reactive silver." Figure 4 clearly shows that for 70 % R.H. and a given amount of silver relatively high silver loadings and lower bed depth of the molecular sieve are more economical than lower silver loadings and higher bed depth. The same is valid for 100 % R.H., but for low bed depth the effect is somewhat obscured by the large amount of adsorbed water (Figure 5).

CONCLUSIONS

In dry air and helium Ag-impregnated molecular sieve Linde 13 X showed excellent removal efficiency for methyl iodide. At higher temperatures, 200, 300, and 650°C in these experiments, the removal efficiency increased further by the thermal decomposition of the methyl iodide.

The removal efficiency of Ag-impregnated molecular sieve Linde 13 X for methyl iodide is strongly influenced by the relative humidity of the sweep gas, but still at relative humidities near 100 % removal efficiencies between 90 and 99 % were measured for 10 cm bed depth and a stay time of 0.4 sec. For this bed depth, stay time and air at 30° C and 70 % R.H. as the sweep gas, variations in the loading with methyl iodide had only a slight influence in the investigated range from 0.01 to 6.7 mg methyl iodide per g Ag-impregnated molecular sieve (calculated for a bed depth of 10 cm), corresponding to concentrations between 0.023 and 10 mg of methyl iodide per m³ air.

From aging experiments in superheated steam of 510°C and 140 atm it can be concluded that Ag-impregnated molecular sieve

- 8 -

cannot be used as an iodine filter material in primary circuits under those rough conditions. The results at 235°C and 30 atm are much better; so the work will be continued in the range between 150 and 300°C and under lower pressure.

There is a strong influence from the amount of silver in the molecular sieve on the methyl iodide removal efficiency. The amount of silver used for impregnation of the molecular sieve by exchange should be high enough to allow a practically complete exchange of the Na⁺ with Ag⁺ in the molecular sieve Linde 13 X. For a given amount of silver and a relative humidity of 70 % and 100 % it was found that relative high silver loadings and lower bed depth of the molecular sieve are more economical than lower silver loadings and higher bed depth.

On the whole, Ag-impregnated molecular sieve Linde 13 X seems to be a good material to remove iodine from off-gas streams in dry and wet atmospheres, but its use for off-gas cleaning is restricted to cases in which the incombustibility of the material (high decay heat from fission product iodine loadings) is important enough to tolerate the high price of this material.

REFERENCES

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- (2) WILHELM, J.G., Testing of Iodine Filters for Nuclear Installations, Report of the Karlsruhe Nuclear Research Center, Germany, KFK 858 (November 1968)

- 9 -

Table I. CH₃¹³¹I REMOVAL EFFICIENCY OF UNIMPREGNATED MOLECULAR SIEVE IN WET AIR.

- Molecular sieve: Linde 13 X as delivered, pellets, diam. 1/16 inch, 4 successive test beds, bed depth: 25 mm, bed diam. 25 mm.
- Sweep gas: air, temperature: 30^oC, R.H.: 85 %, atmospheric pressure, superficial velocity: 15 m/min.

Duration of air flow: pre-humidification: 24 h, CH₃I injection: 1 h, air flow continued for an additional 17 h.

Loading: $3.0 \pm 0.6 \ \mu g \ CH_3^{127}I + 1.0 \pm 0.3 \ \mu Ci$ CH₃¹³¹I per g molecular sieve (calculated for 10 cm bed depth).

Bed depth (cm)	Stay time (sec)	CH ₃ ¹³¹ I removal efficiency (%)	CH ₃ ¹³¹ I penetration (%)
2.5	0.1	0.35 · 10 ⁻³	99.99965
5.0	0.2	$0.69 \cdot 10^{-3}$	99.99931
7.5	0.3	1.64 • 10 ⁻³	99.99836
10.0	0.4	2.72 · 10 ⁻³	99.99728

Table II. CH₃¹³¹I REMOVAL EFFICIENCY OF Ag-IMPREGNATED MOLECULAR SIEVE LINDE 13 X IN AIR OF DIFFERENT RELATIVE HUMIDITIES.

Molecular sieve: Linde 13 X, pellets, diam. 1/16 inch. Impregnation: 0.57 g Ag/g molecular sieve (as delivered),

4 successive test beds, bed depth: 25 mm, bed diam. 25 mm.

Sweep gas: air, temperature: 30°C, atmospheric pressure, superficial velocity: 15 m/min.

Duration of air flow: pre-humidification > 20 h,

 $CH_{3}I$ injection: 1 h, air flow continued for an additional 20 - 22 h.

Experimental conditions (CH313	³¹ i remo	OVAL EFF:	ICIENCY (%)
R.H. (%)	CH ₃ I loading	Bed depth (cm)	2.5	5.0	7.5	10.0
	(µg/g)	Stay time (sec)	0.1	0.2	0.3	0.4
25	30 <u>+</u> 6	96.14	99.88	99.994	(99.997)	
40	30 <u>+</u> 6	91.85	99.40	99.96	99.996	
55	30 <u>+</u> 6	85.50	97.85	99.68	99.963	
70	10 <u>+</u> 2	73.52	94.65	98.85	99.79	
85 85	3 <u>+</u> 0. 130 <u>+</u> 26	71.12 70.91	91.63 91.20	97.50 97.35	99.07 99.21	
100	3 <u>+</u> 0.	42.21	71.02	87.22	93.87	

^{*}µg CH₃I per g molecular sieve, calculated for 10 cm bed depth.

() = activity of test bed near background, therefore, data for removal efficiency too low.

Table III. CH₃¹³¹I REMOVAL EFFICIENCY OF Ag-IMPREGNATED MOLECULAR SIEVE FOR DIFFERENT METHYL IODIDE CONCENTRATIONS AND LOADINGS.

Molecular sieve and test beds: the same as in Table II. Sweep gas: air, temperature: 30°C, atmospheric pressure,

R.H.: 70 %, superficial velocity: 15 m/min. Duration of air flow: pre-humidification \geq 22 h,

 $CH_{3}I$ injection: 1 h (unless specified otherwise), wet air flow continued for an additional 20 - 22 h.

Experimenta	l conditions	CH ₃ ^{13⁻}	¹ i remo	DVAL EI	FFICIE	NCY (%)
CH _J I loading	CH ₃ I concentration	Bed depth (cm)	2.5	5.0	7.5	10.0
(mg/g)*	(mg/m ³)	Stay time (sec)	0.1	0.2	0.3	0,4
0.01(<u>+</u> 20 %	5) 0.023(<u>+</u> 20 %)		73.52	94.65	98.85	99.79
0.21 "	0.47 "		68.93	92.20	98.07	99.60
0.27 "	0.61 "		67.54	91.55	97.84	99.47
1.0** "	1.1 "		74.63	94.17	98.54	99.51
1.5 "	1.7 "	• •	73.72	94.30	98.85	99.76
3.6 "	.8,2 "		63.36	89.55	97.37	99.44
4 . 4 "	10.0 "		60.94	87.74	96.80	99.36
6.7** "	7.6 "		57.46	85.69	95.17	98.46

*mg CH₃I per molecular sieve, calculated for 10 cm bed depth. **CH₃I injection time: 2 h. Table IV. CH₃¹³¹I REMOVAL EFFICIENCY OF Ag-IMPREGNATED MOLECULAR SIEVE IN DRY AIR AT 200, 300 AND 650°C. Molecular sieve : the same as in Table II. Test beds: the same as in Table II, but only 2 successive beds. Sweep gas: air, atmospheric pressure, R.H. run 1: ≤ 0.1 %, run 2: ≤ 0.02 %, run 3: < 0.001 %. Superficial velocity: run 1: 21.4 m/sec, run 2: 26.1 m/sec, run 3: 19 m/sec. Duration of air flow: pre-conditioning with hot air 24 h, CH₃I injection 1 h, hot air flow continued for an additional 20 - 24 h.

Run No.	CH ₃ I loading (µg/g) [*]	Tempe- rature (^O C)	Bed depth (cm)	Stay time (sec)	CH ₃ ¹³¹ I removal efficiency (%)
1	130	200	2.5 5.0	0.07 0.14	99.9843 99.9978
2	150	300	2.5 5.0	0.058 0.116	99.9871 99.9982
3	540	650	2.5 5.0	0.10 0.20	99.9760 99.9979

* μ g CH₃I per g molecular sieve, calculated for 5 cm bed depth.

Table V. CH₃¹³¹I REMOVAL EFFICIENCY OF Ag-IMPREGNATED MOLECULAR SIEVE IN DRY HELIUM AT 200 AND 300°C.

Molecular sieve and test beds: the same as in Table II, but only two successive test beds.

- Sweep gas: He, atmospheric pressure, superficial velocity for injection time and an additional 2 h of gas flow: 15 m/sec.
- Duration of He-flow: Pre-conditioning with hot He (80 1/h): 21 h, CH₃I injection: 1 h (He gas flow for injection time: 440 1/h), hot He flow continued for an additional 2 h with 440 1/h and 18 h with 80 1/h.

Run No.	CH _z I loading (µg/g)*	Tempe- rature (^O C)	Bed depth (cm)	Stay time (sec)	CH ₃ ¹³¹ I removal efficiency (%)
1	80	200	2.5 5.0	0.1 0.2	99.9937 (99.9980)
2	160	300	2.5 5.0	0.1 0.2	99.9993 (99.9995)

* μg CH₃I per g molecular sieve calculated for 5 cm bed depth.() = activity of the test bed near background, therefore, data for removal efficiency too low. Table VI. CH₃¹³¹I REMOVAL EFFICIENCY OF Ag-IMPREGNATED MOLECULAR SIEVE BEFORE AND AFTER AGING IN SUPERHEATED STEAM.

Conditions for $CH_3^{131}I$ removal test.

Molecular sieve: the same as in Table II, aged with super-

Test beds: the same as in Table II.

Sweep gas: air, atmospheric pressure, temperature: 30°C,

R.H.:70 %, superficial velocity 15 m/min.

Duration of air flow: pre-humidification \geq 22 h,

 CH_3I injection: 0.5 - 2 h, wet air flow continued for an additional 20 - 22 h.

Experimental conditions for			CH ₃ ¹³¹ I R	EMOVAL I	EFFICIEN	ICY (%)	a	
steam aging of molecular sieve								
Tempe- rature	Pres- sure	Steam flow	Time of steam flow	CH_I loading	2.5	Bed der 5.0	oth (cm) 7.5	10.0
(°C)	(atm)	(kg/h)	(h)	(mg/g)*	0.1	Stay ti 0.2	lme (sec 0.3) 0.4
	no aging			0.01	73.52	94.25	98.85	99.79
				1.5	73.72	94.30	98.85	99.76
235	30	≈ 300	16	2.0	48.08	75.28	88.46	94.67
235	30	≈ 300	32	2.2	26.61	45.53	61,59	73.85
510	140	≈ 300	16	2.2	0,28	0.54	0.72	0.91
510	140	≈ 3500	32	0.016	0.004	0.008	0.0013	-

* mg CH_3I per g molecular sieve, calculated for 10 cm bed depth.

- 15 -



Fig. 2 PENETRATION OF CH₃¹³¹I THROUGH Ag-IMPREGNATED MOLECULAR SIEVE LINDE 13 X AS A FUNCTION OF THE RELATIVE HUMIDITY.

BED DEPTH.

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AS A FUNCTION OF THE Ag-IMPREGNATION, 100 % R.H.