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Testing of Iodine Filters for Nuclear Installations

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABSTRACT

Work has been done to test the removal efficiency of iodine filters for nuclear installations.
The test method in use includes laboratory tests of the adsorber material (under MCA-conditions for temperature, relative humidity, pressure, loading, stay-time and bed depth) and in-place tests at the site of the installation.

For removal of methyl iodide under high relative humidity, KI-impregnated charcoal is widely used. Most of the data for charcoal removal efficiency available today result from experiments with tracer amounts of $CH_3{}^{131}I$, mixed with $CH_3{}^{127}I$. Considering MCA-conditions, the validity of those data should be confirmed for specific activities on charcoal 10^3 - 10^6 times higher. Experimental data are given for realistic loadings with $CH_3{}^{131}I$ up to 1.0 Ci/g charcoal under 100 % relative humidity.

For standard laboratory adsorber tests a method is discussed for humidification and control of gas streams up to 100 % relat. humidity at elevated temperatures; the apparatus used will be described. Experimental data are given for the removal efficiency and the adsorption of water in charcoal samples from long-time tests under 100 % relat. humidity. The overall test period was up to 98 h, simulating the phase of elevated pressure and, therefore, high iodine release from the reactor containment. The reproducibility of long-time tests under extremely high relative humidity is shown and discussed.

A description of the hardware for in-place tests is included, results are given.

INTRODUCTION

Reactor stations in Germany with its high population density are in need of reactor safeguards to protect the population from accidental fission product release. Therefore, most reactor off-gas systems are equipped with iodine filters. The removal efficiency of those filters has to be tested and it has to be shown that the performance under simulated MCA-conditions will meet the values established in the hazards report of the respective installation. The adsorber material of the iodine filter is tested in the laboratory under standard conditions and simulated MCA-conditions. Additional in-place tests are carried out on the reactor site. These tests are generally produced under normal operating conditions and no effort is made to get high humidity or elevated temperature.

LABORATORY TESTS

Test apparatus

For testing of adsorber material we developed a test apparatus. Tests can be performed in the temperature range between 10 and 80°C with relative humidities of the sweep gas between 5 and 100 %. The depth of the test bed may be up to 50 cm, stay-time and the period of loading can be adjusted to any realistic value considering iodine filters. It is well known [1, 2] that the charcoal removal efficiency for organic iodine compounds is influenced very strongly by the high relative humidity of the sweep gas. For iodine filter systems of water cooled reactors, the operational range of interest very often is near or at 100 % R.H. Small changes in this region may have a significant effect on the adsorber performance. For competitive laboratory test runs with different types of charcoal a very accurate control of the humidity of the sweep gas is needed. Hence, a major effort was taken for accurate control of the relative humidity in our test apparatus.

Fig. 1 is a flow diagram of the test apparatus. The sweep gas enters the equipment through a particle filter (1) and the gas

flow is measured (2). Then the sweep gas is mixed with steam (3) and transported through a heated tube (4) to a special cooler (5). This cooler is double-walled and has a very narrow coil. Through wall and coil there is a constant flow of coolant from a thermostate. In this "dew point cooler" the dew point of the sweep gas is fixed in a range of ± 0.1°C. The condensate from excess steam is sampled in (6). The sweep gas is taken from the lower third of the dew point cooler and sucked through a double-walled heated tube (7). The temperature of the outer tube wall is only 1 - 2°C above the dew point to avoid heat flow to the dew point cooler. Then the sweep gas is sucked through a long "thermostate tube" (8) inside a water filled thermostate (11). The test agent, mostly $CH_3{}^{131}I$ + $CH_3{}^{127}I$ in air, is added to the sweep gas at the central part of the thermostate tube. Inside the thermostate tube the gas is controlled within ± 0.03°C. The gas is then sucked through a deep glass wool filter (9) and beds of the adsorber material under test (test beds (10)) inside the thermostate (11). The offgas from the test beds is heated so that the relative humidity will drop below 30 % and is sucked through heated charcoal beds (safety beds (12)). The remaining activity of the gas stream is removed by the first section of the safety beds. Then, most of the humidity in the offgas is removed by a cooler with exactly controlled temperature (13), filtered by an additional charcoal trap for safety (16) and discharged to the exhaust through a membrane pump (18). The relative humidity in the apparatus can be measured directly by psychrometric methods and can be calculated from the volume of the condensate (14). The dew point method is used as well. Different temperatures in the dew point cooler (5) and the thermostate (11) allow the humidity of the gas to be varied from 5 to 100 % R.H. The whole apparatus works nearly fully automatically; we performed runs up to 98 h at 99 - 100 % R.H. without condensation in the test beds.

As slight condensation in the adsorber material would obscure the results of high humidity runs, the gain in weight of the subsequent charcoal beds by water adsorption is measured after each run. The time dependence of the water adsorption and the total adsorption in equilibrium with the humidity of the sweep gas is measured in a run without any activity.

Fig. 2 indicates the results of a water adsorption experiment with a total bed depth of 50 cm. The charcoal was divided into three subsequent beds and the time dependence of the water adsorption of each bed was measured. Air of 30°C and 100 % R.H. was used. Most of the adsorption took place in the first 7 hours, equilibrium was reached in a period shorter than 13 hours in the first charcoal bed. Considering the pressure drop in the charcoal, the H_2O-partial pressure will be somewhat lower in each subsequent bed and so will be the total amount of adsorbed water. The exact data of this run are given in Table I.

Standard Test Procedure

To compare the efficiency of different types of charcoal we perform a standard test procedure. Test conditions are: 20 h pre-humidification of the charcoal with wet air of 30°C, 100 % R.H. and a loading time of additional 20 h with a mixture of $CH_3{}^{131}I$ + $CH_3{}^{127}I$ in the wet air. After loading, the wet air stream is maintained for additional 2 h. In Table II the removal efficiency under standard conditions is given for some charcoals. For one non-impregnated and one impregnated charcoal also the results of re-runs are given to demonstrate the reproducibility of tests under extremely high relative humidities for both types of charcoal.

Test Procedure under Simulated MCA-Conditions

For purposes of reactor licensing, the main point of interest is in test runs under conditions expected in the MCA. Table III shows the results of tests for the MCA-iodine filter of the Obrigheim reactor[3]. This filter was built for a removal efficiency better than 99.9 % for fission product iodine. Relative humidity may be up to 100 % at a temperature up to 31.2°C; at higher temperatures the relative humidity will drop because there is a limited supply of steam by a leak between the first and the second containment. The stay-time of the offgas in the iodine filter with a bed depth of 50 cm will be 1 sec and the total operating period after an accident would most likely not exceed 48 h.

Considering the leak rate between the first and the second containment and a total amount of 10 % of the iodine converted

to methyl iodide, the loading of the charcoal in the iodine filter will not exceed 1.5 µg CH_3I/g of charcoal. With these data in mind we selected the test conditions listed in Table III. In Fig. 3 the penetration of the charcoal beds is shown versus the bed depth for those tests (curve b). There is a slight deviation in the plot from a straight line in direction of higher removal efficiencies with increasing bed depth. We think this is due to the pressure drop in the charcoal connected with a decreasing H_2O partial pressure in the successive layers of charcoal and affecting the amount of adsorbed water. From Fig. 3 it can be concluded that in this region of very low CH_3I-loading, over a range of nearly four orders of magnitude, there is no pronounced effect from the total amount of $CH_3{}^{131}I$ and $CH_3{}^{127}I$, reaching the successive layers of the impregnated charcoal. The total uptake of water in the first charcoal bed versus time for one of the two runs is given in Fig. 4. The diagram shows that condensation was clearly avoided. The total water inventory of the charcoal is the same as in a previous water adsorption test of a much shorter duration, indicated in Table I.

To be safe from overloading of the MCA-iodine filters we perform our tests with at least twice the maximum mass of CH_3I per g of charcoal expected in an accident. However, there is some concern about the validity of experimental results with tracer activity of $CH_3{}^{131}I$ for MCA-conditions. The specific activity of organic iodine may be higher than in the test runs by a factor of 10^6 and more.

Tests with Methyl Iodide of High Specific Activity

Today it is generally accepted that the trapping of radioactive $CH_3{}^{131}I$ by impregnated charcoal is mostly due to an isotopic exchange of ^{131}I in the form of $CH_3{}^{131}I$ in the gas phase with ^{127}I on the charcoal, mostly as $K^{127}I$ or $^{127}I_2$. For this isotopic exchange the relation of active organic iodine in the gas stream to inactive iodine on the charcoal will be much more unfavourable in a MCA situation than under the widely used laboratory test conditions. The upper loading limit for activity in the form of organic iodine compounds should be established.

We performed some ~~tests~~ with mixtures of $CH_3{}^{131}I$ + $CH_3{}^{127}I$ of higher specific activity. To avoid trapping of CH_3I by adsorption on the charcoal as far as possible, we used air with 98 - 100 % R.H. for the sweep gas. Therefore, the results of the experiments should reflect the influence of the changed relationship for an isotopic exchange.

To avoid extreme handling and shielding problems we used small charcoal beds with a diameter of 6 mm. The runs were performed with pellets of KI impregnated charcoal of 0.8 mm diameter and a length of 2 - 3 mm. There will be more channeling than in tests with larger test bed diameters, but in the experiments the results were consistent enough to be sure that this effect would not obscure effects of activity overloading.

In Table IV the results of some runs are indicated. For comparison, and to test the reproducibility, run 1, 2 and 3 were performed on an intermediate activity level. The activity (in the form of $CH_3{}^{131}I$) reaching the first test bed was between 3 and 8 mCi per g of charcoal, mixed with a total of 20 µg $CH_3{}^{127}I$. In run 4 the activity was increased to 1 ± 0.2 Ci/g of charcoal. Considering the somewhat lower reproducibility of experiments with very small charcoal beds, the removal efficiency is nearly the same in all four runs and no overloading effect can be seen. In Table IV also one run (No. 5) is included with a relative humidity slightly lower than in runs 1 - 4. The total activity (in the form of $CH_3{}^{131}I$) reaching the first charcoal bed was 0.3 Ci/g of charcoal. The removal efficiency is better than in runs 1 - 4, which demonstrates the sensitivity of the results to small changes in the relative humidity. Fig. 5 shows the penetration of the charcoal beds versus bed depth for run 4. Each point represents one of the successive charcoal beds. To the first and the last point are assigned the values for the activity per g of charcoal reaching that particular charcoal bed. Within the accuracy of the experiment a straight line can be drawn through the points and no sign of a pronounced bend in the region of high activity loading is observed.

IN-PLACE TESTS

Test Agent, Operating Conditions.

In-place tests should establish the leak rate of the iodine filter system and the condition of the adsorber material. For the test agent, therefore, a mixture of $CH_3{}^{131}I + CH_3{}^{127}I$ is used in case the iodine filter is filled with impregnated charcoal. For filter systems of an older design (and lower removal efficiency, of course) a mixture of $^{131}I_2$ and $^{127}I_2$ will be used. For technical and financial reasons, the in-place tests are performed under normal operating conditions of the iodine filter system.

Pre-Run in the Laboratory, Activity of Test Agent.

In the test procedure a pre-run in the laboratory is included with the original charcoal under conditions expected for the in-place test. This will indicate the maximum removal efficiency of the iodine filter, excluding mechanical leaks and aged charcoal. In Fig. 3 curve (a), the results of a pre-run for an in-place test of the Obrigheim iodine filter is shown. In this case, the removal efficiency is high enough to prevent activity from being detected downstream of the iodine filter without a mechanical leak or badly aged charcoal. The total activity used in the in-place tests is calculated with respect to the removal efficiency of the iodine filter system, established in the hazards report of the respective installation. We think that the activity should be high enough to indicate a leak which produces 1 % of the maximum allowable penetration.

Arrangement and Samplers for In-Place Tests.

The arrangement used for the Obrigheim in-place test is shown in Fig. 6. To avoid the use of extremely high activity, a relatively high gas flow through the downstream sampler is maintained (m^3/h). The gas flow through the upstream sampler normally is lower by a factor of $\sim$ 100. The volume of the impregnated charcoal in the sampler is sufficiently large to have at least a total stay-time of 1 sec in tests performed with methyl iodide. The upstream and downstream samplers are fitted

with charcoal beds of the same diameter and bed depth to avoid geometrical problems in quantitative γ-spectroscopy. To compensate for the higher gas velocity in the downstream sampler a larger number of charcoal beds is used. Owing to the different superficial gas velocities the distribution of the activity in the upstream and the downstream samplers is not the same. For measurement the samplers therefore have to be disassembled, the charcoal of each bed has to be mixed very carefully. Each charcoal bed is measured separately to check the quantitative removal of the test agent in the sampler.

Container System for Test Agent

Fig. 7 shows a drawing of the container system for handling and transport of the radioactive test agent and a charcoal sampler. The inner container filled with the test agent is built in form of a U-tube closed with bellows seal valves and quick-connects with double end shut-off. The U-tube is cast in a lead cylinder for shielding. This inner container is used also as a recipient for the distillation of the methyl iodide. This avoids handling of unshielded radioactive agents. The inner container is transported to the iodine filter to be tested inside a gas-tight transport container which is fitted with additional shielding and a charcoal trap. Before the transport container is opened, it is flushed with air so that gaseous activity will be trapped in the charcoal. The charcoal trap is not shielded; therefore a leak of the inner container can be detected by simple γ-measurement on the outside of the charcoal trap without opening of the transport container.

Data on the In-Place Test of the Obrigheim MCA-Iodine Filter.

Finally, some data on the in-place test of the Obrigheim MCA-iodine filter are given.[4] The activity of the $CH_3{}^{131}I$ was 100 ± 20 mCi, mixed with 5 ± 1 mg $CH_3{}^{127}I$.
The gas flow through one upstream sampler was 24 N l/h, through one downstream sampler it was 2500 N l/h. Two charcoal samplers of each type were used. The charcoal volume of one downstream sampler was 765 cm^3. After an injection time of 15 min the air

flow through the iodine filter was maintained for 2h. The temperature was 21.5 ± 0.5°C, R.H. 55 ± 2 %. The removal efficiency η of the iodine filter in the in-place test was

$$\eta \geq 99.9998\ \%$$

(under simulated MCA conditions the removal efficiency of the charcoal was 99.975 ± 0.015 %).

The simple design of the iodine filter (two containers in parallel with a charcoal fill much higher than the perforated sheet metal welded to both ends of the container and a total charcoal volume of 1 m^3) made a result as that received from the in-place test likely, because most sealing problems are avoided.

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[2] COLLINS, D.A., TAYLOR, L.R., TAYLOR, R., "The development of impregnated charcoals for trapping methyl iodide at high humidity", UKAEA Rep. TRG 1300 (W) (1967)

[3] WILHELM, J.G., "Nachweis des Abscheidegrades der MCA-Jodfilter Obrigheim, Laborversuche", Externer Bericht der GfK Nr. 20/67-4, (1967)

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Table I: Water Adsorption of Impregnated Charcoal

Charcoal: Norit 0.5 % KI, pellets, diam.: 4 mm, length 4 - 8 mm, 3 successive charcoal beds, total bed depth 50 cm.
Wet air: 30°C, 100 % R.H., superficial air velocity: 42 cm/sec.

Duration of air flow (h)	Water adsorbed in wt. % of dry charcoal		
	bed 1	bed 2	bed 3
1	23.64	11.58	7.37
3	44.61	32.02	19.54
7	46.33	45.22	43.78
15	46.49	45.57	45.16
31	46.49	45.69	45.28
37	46.57	45.81	45.70

Table II: $CH_3{}^{131}I$-Removal Efficiency of Activated Charcoal under Standard Conditions

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

Loading: about 5 µg CH_3I/g charcoal, total ^{131}I activity: 0.1 - 0.2 mCi.

Sweep gas: air, temperature: 30°C, R.H.: 100 %, atmospheric pressure, superficial velocity: 15 m/min.

Duration of air flow: pre-humidification 20 h, CH_3I injection: 20 h, air flow continued for additional 2 h, total: 42 h.

Charcoal	$CH_3{}^{131}I$-Removal Efficiency (%)			
Bed depth (cm)	2.5	5.0	7.5	10.0
Stay-time (sec)	0.1	0.2	0.3	0.4
Norit, in pellets, 0.8 mm diam., unimpregnated, length 2-3 mm (1)	0.92	1.69	2.55	3.47
same as above (2)	0.73	1.42	2.06	2.80
average values of (1)a.(2)	0.83	1.56	2.31	3.14
Norit RCX 1 % KI. 0.8 mm diam. (same as above, but impregnated)	81.24	97.48	99.63	99.94
Norit 0.5 % KI, in pellets 2 mm diam., length 4-8 mm	41.35	64.97	83.01	91.51
Norit RX 3210 1 % BaI, in pellets, 1 mm diam., length 3-6 mm a)	72.79	94.38	98.79	99.77
SS 207 B 0.5 % KI (UK) (1)	78.52	96.85	99.56	99.94
same as above (2)	74.45	95.73	99.34	99.89
average values of (1)a.(2)	76.49	96.29	99.45	99.92

a) CH_3I injection: 2 h, air flow continued for 20 h, loading ($CH_3{}^{127}I$): 50 ± 10 µg/g of charcoal.

Table III: $CH_3{}^{131}I$-Removal Efficiency of Impregnated Charcoal under Simulated MCA-Conditions

Charcoal: Norit 0.5 % KI in pellets, 2 mm diam. 3 successive beds, total bed depth: 50 cm, diam.: 2.5 cm.

Loading: 3.5 ± 0.5 µg CH_3I/g charcoal, total ^{131}I-activity: 0.15 ± 0.05 mCi.

Sweep gas: air, temperature 31.2°C, relative humidity 100 %, atmospheric pressure, superficial velocity: 30 m/min.

Duration of air flow: pre-humidification 48 h, CH_3I injection: 48 h, air flow continued for additional 2 h, total: 98 h.

Bed depth (cm)*	Stay-time (sec)	$CH_3{}^{131}I$ Removal Efficiency (%)		
		Run 1	Run 2	average
6.25 ± 0.4	0.125	54.56	52.18	53.37
12.50 ± 0.4	0.250	85.21	81.34	83.28
18.75 ± 0.4	0.375	93.53	91.94	92.74
25.00 ± 0.4	0.500	97.85	97.36	97.61
31.25 ± 0.4	0.625	99.32	99.23	99.28
37.50 ± 0.4	0.750	99.74	99.68	99.71
43.75 ± 0.4	0.875	99.93	99.92	99.925
50.00 ± 0.2	1.000	99.98	99.97	99.975

* The charcoal from the single beds was poured out in fractions. Therefore, the tolerance for one fraction is higher than for the total bed depth.

Table IV: $CH_3{}^{131}I$-Removal Efficiency of Impregnated Charcoal on Loading with Methyliodide of High Specific Activity

Charcoal: Norit, Type RCX 1 % KI, in pellets, diam.: 0.8 mm, length: 3-6 mm, 10 successive test beds, bed diam.: 6 mm.

Loading with $CH_3{}^{131}I$ (^{131}I activity per g charcoal, calculated for the weight of the first test bed): Run 1: 3 ± 0.6 mCi/g; run 2: 3.5 ± 0.7 mCi/g; run 3: 8 ± 1.6 mCi/g; run 4: 1 ± 0.2 Ci/g; run 5: 0.3 ± 0.06 Ci/g.

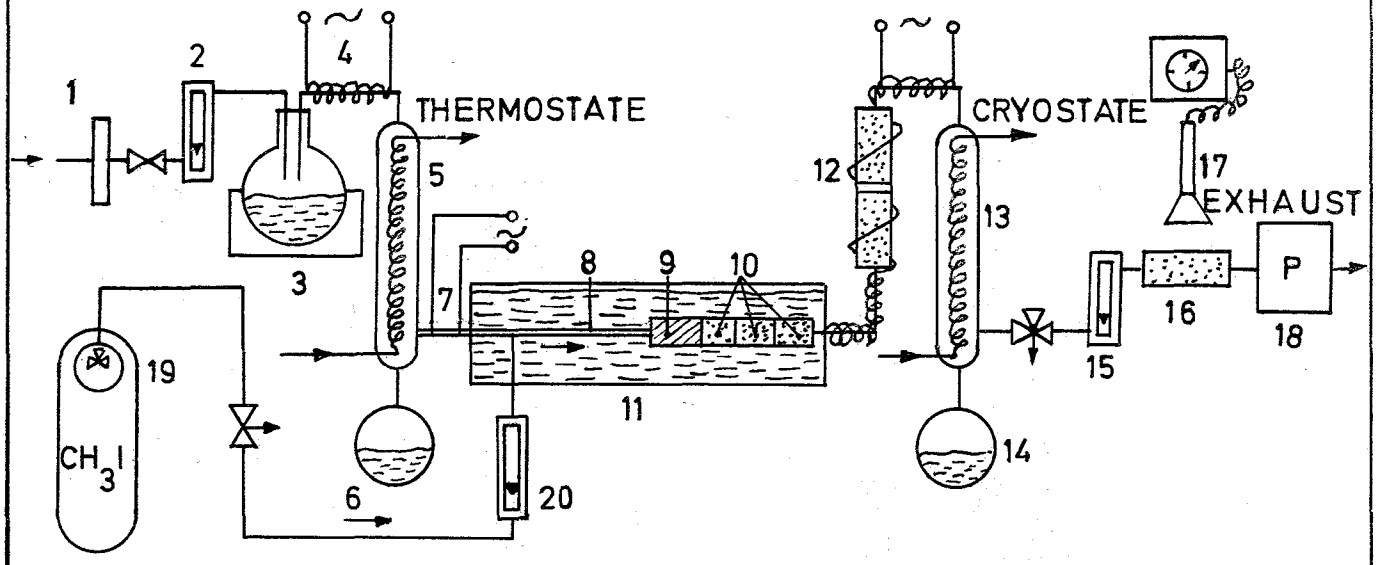
Loading with $CH_3{}^{127}I$: 20 ± 4 µg total for each run.

Sweep gas: air, 30°C, 98-100 % R.H., atmospheric pressure, superficial velocity: 15 m/min.

Duration of air flow: pre-humidification: 20 h, CH_3I-injection: 15 min, air flow continued for 22 h.

Bed depth (cm)	Stay-time (sec)	$CH_3{}^{131}I$ Removal Efficiency (%) Average			
		Run 1, 2	Run 3	Run 4	Run 5 a)
2.5	0.1	50.16	40.03	50.82	70.50
5.0	0.2	77.32	71.05	78.47	93.50
7.5	0.3	88.80	85.51	91.96	99.13
10.0	0.4	94.49	91.76	97.48	99.93
12.5	0.5	97.33	95.87	99.11	
15.0	0.6	98.75	97.94	99.69	
17.5	0.7	99.36	98.95	99.90	
20.0	0.8	99.67	99.44	99.95	
22.5	0.9	99.84	99.71	99.98	
25.0	1.0	99.91	99.85	99.99	

a) In run 5 the relative humidity of the sweep gas was slightly lower than in runs 1 - 4, only 4 test beds were used in this run.



1 PARTICLE FILTER	9 GLASS WOOL FILTER
2,15,20 FLOW METER	10 TEST BEDS
3 EVAPORATOR	11 BATH THERMOSTATE
4 HEATER	12,16 SAFETY BEDS
5 COOLER FOR DEW POINT SETTING	13 COOLER FOR DEHUMIDIZING
6,14 CONDENSATE	17 γ-SPECTROMETER
7 DOUBLE WALLED TUBE	18 PUMP
8 THERMOSTATE TUBE	19 PRESSURE CYLINDER WITH AGENT

LABORATORY APPARATUS FOR THE DETERMINATION OF CHARCOAL REMOVAL EFFICIENCY

FIG. 1

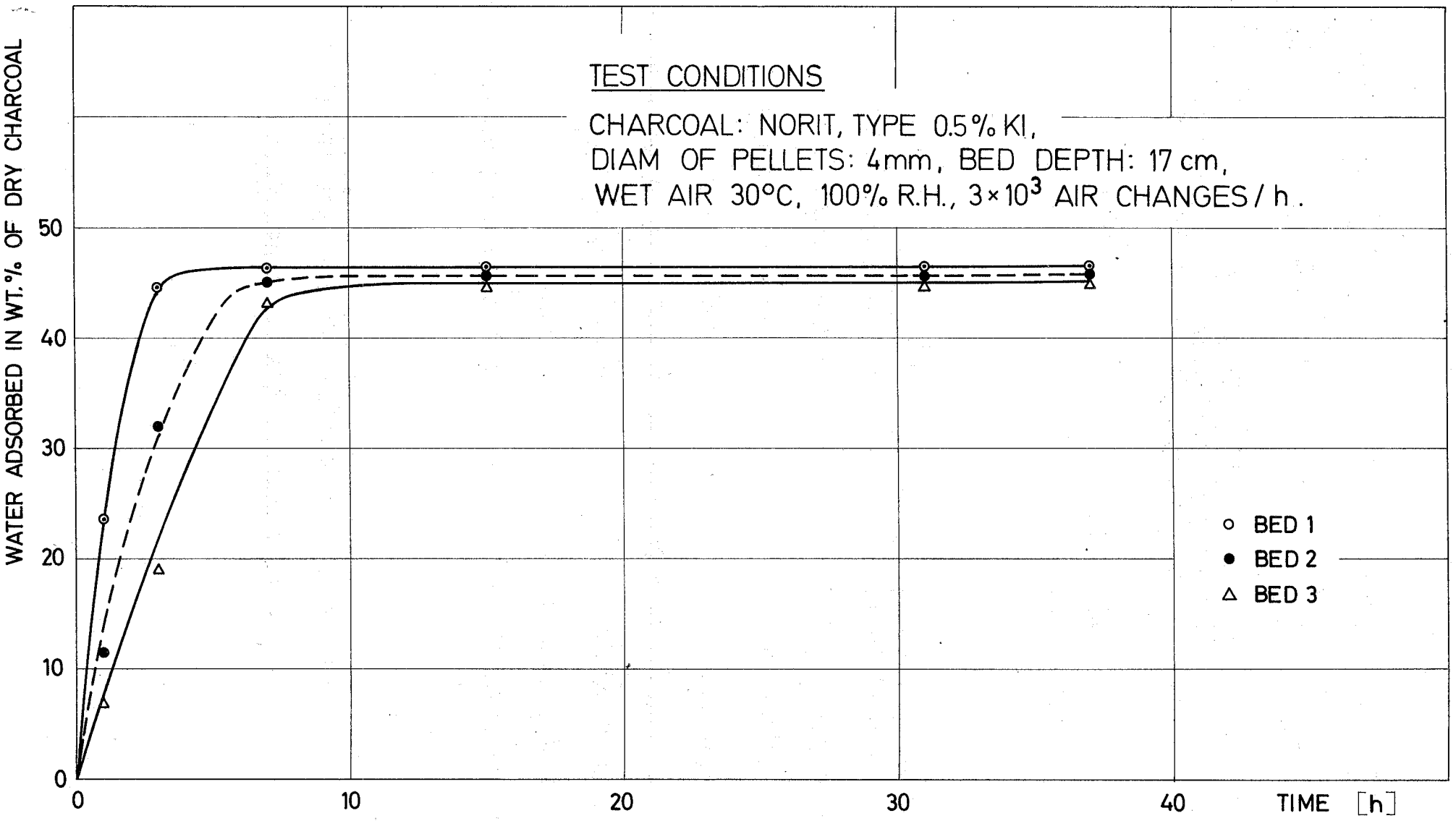


FIG. 2 WATER ADSORPTION OF IMPREGNATED CHARCOAL.

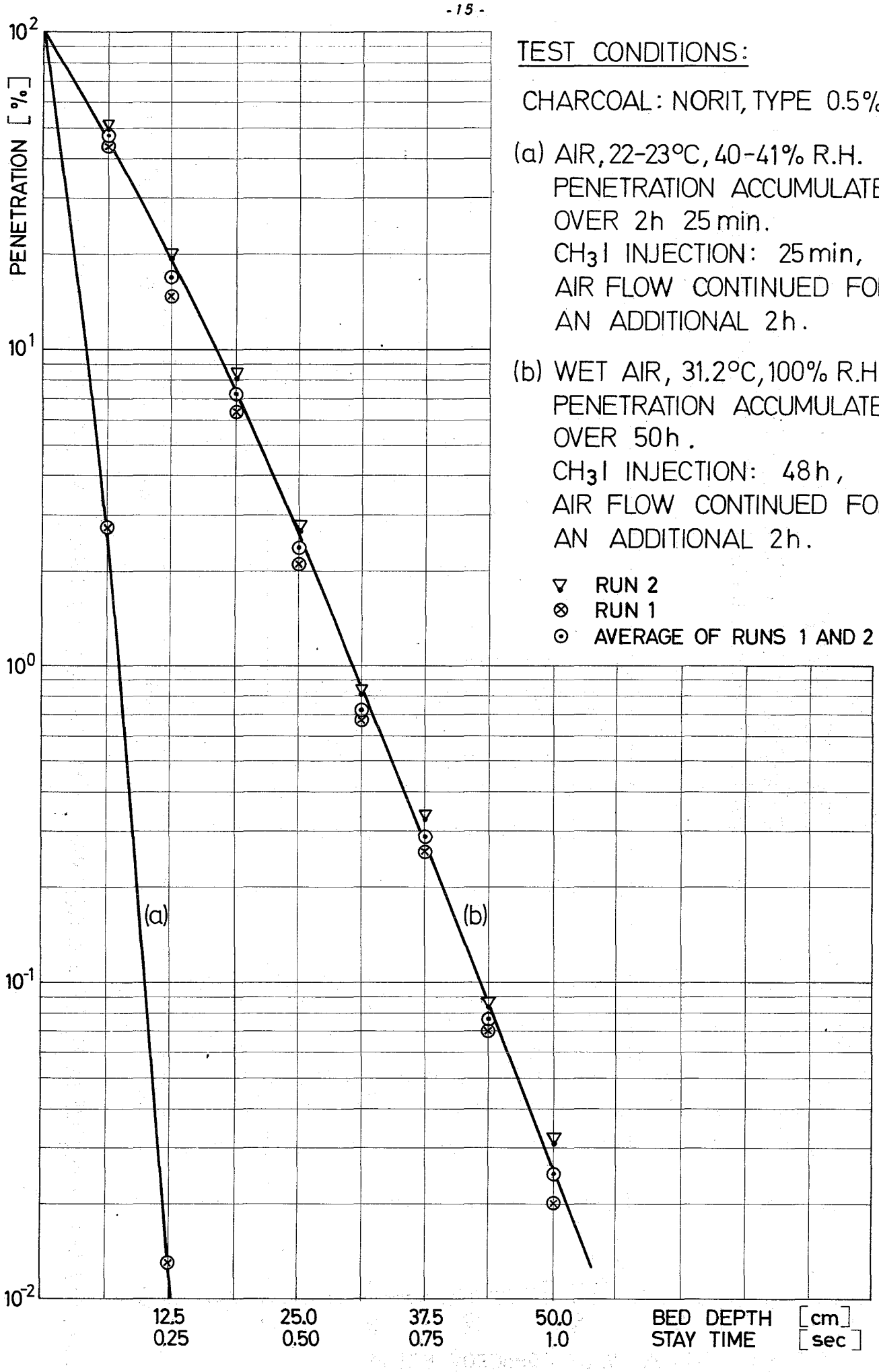


FIG. 3 PENETRATION OF $CH_3^{131}I$ THROUGH IMPREGNATED CHARCOAL OF THE OBRIGHEIM MCA-IODINE FILTER.

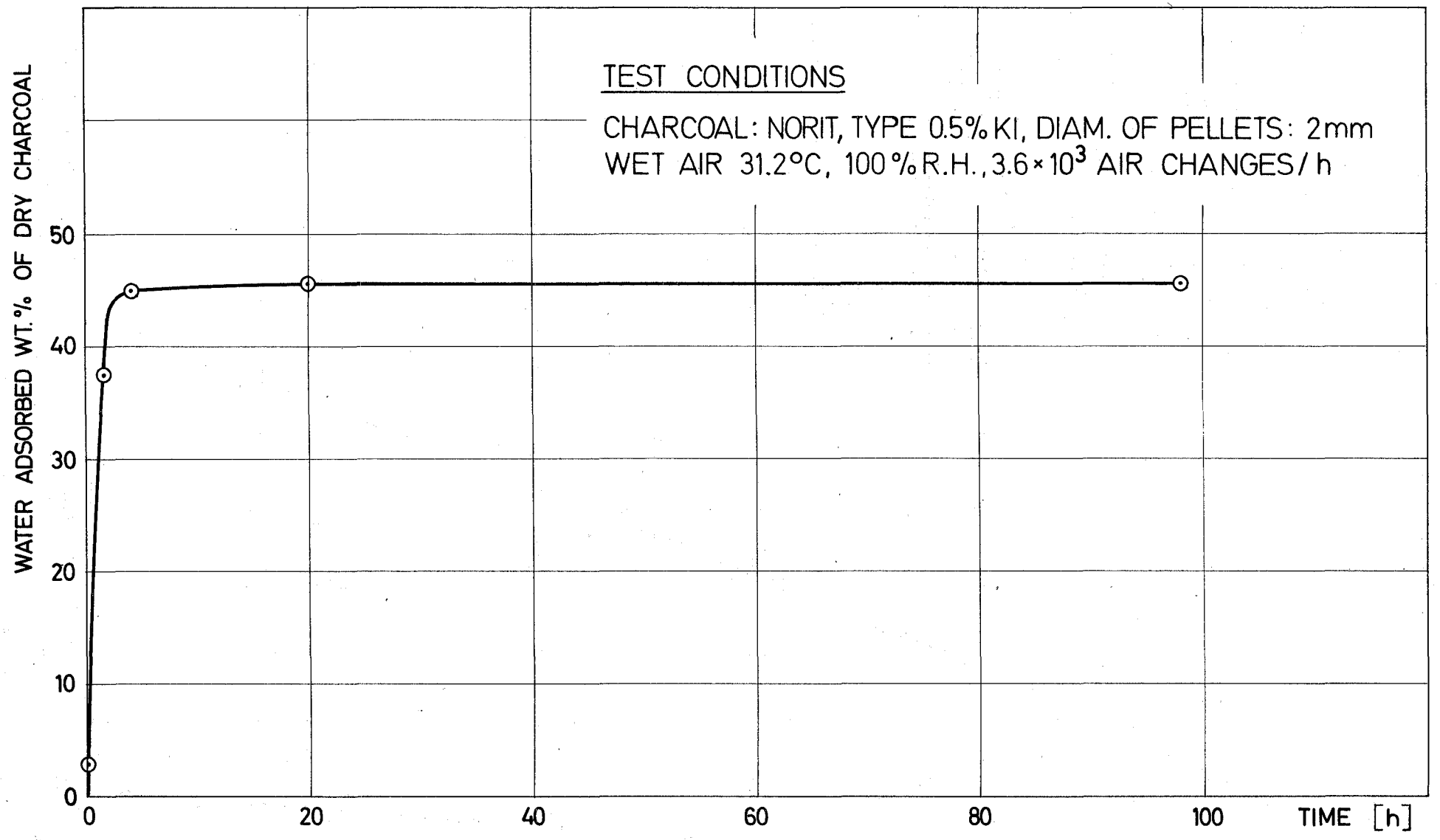


FIG.4 WATER ADSORPTION OF IMPREGNATED CHARCOAL IN REMOVAL EFFICIENCY TEST.

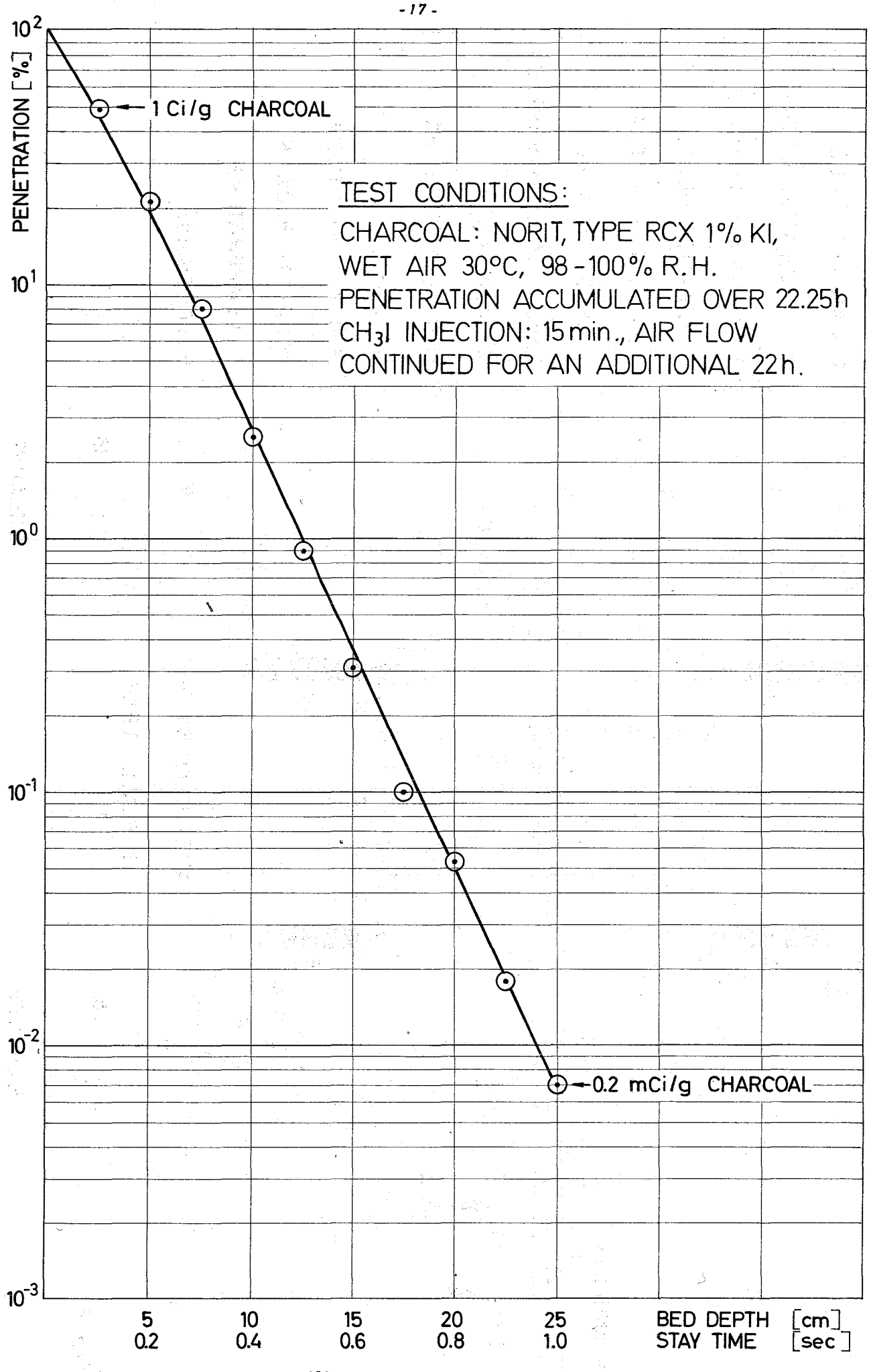
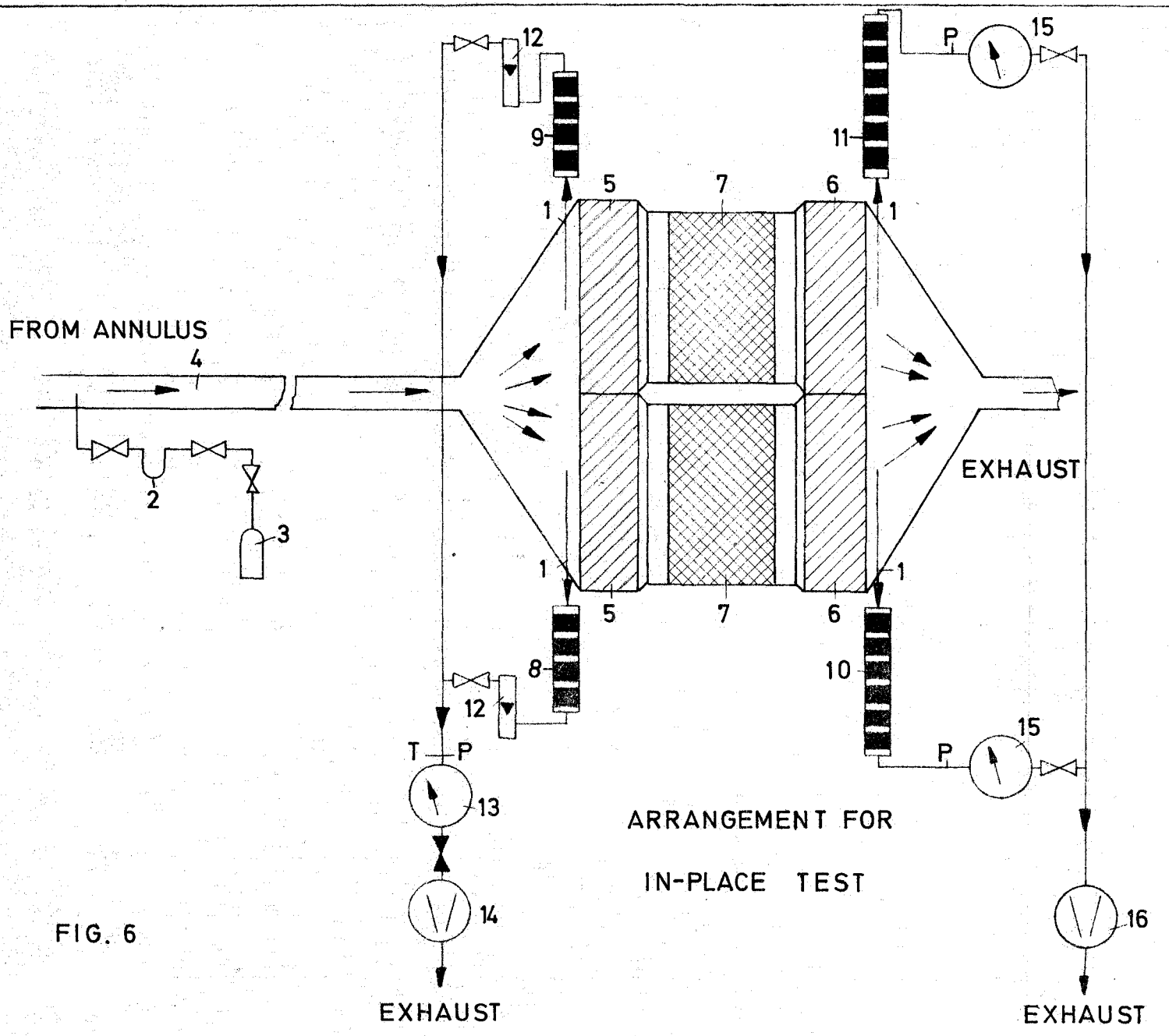


FIG. 5 PENETRATION OF $CH_3{}^{131}I$ FROM A METHYL IODIDE MIXTURE OF HIGH SPECIFIC ACTIVITY THROUGH IMPREGNATED CHARCOAL.



ARRANGEMENT FOR

IN-PLACE TEST

FIG. 6

1 SAMPLING TUBE

2 U-TUBE, TEST AGENT

3 PRESSURE CYLINDER

4 EXHAUST DUCT

5,6 PARTICLE FILTER

7 CHARCOAL FILL

8,9,10,11 CHARCOAL SAMPLER

12 FLOW METER

13 WET TEST METER

14 PUMP

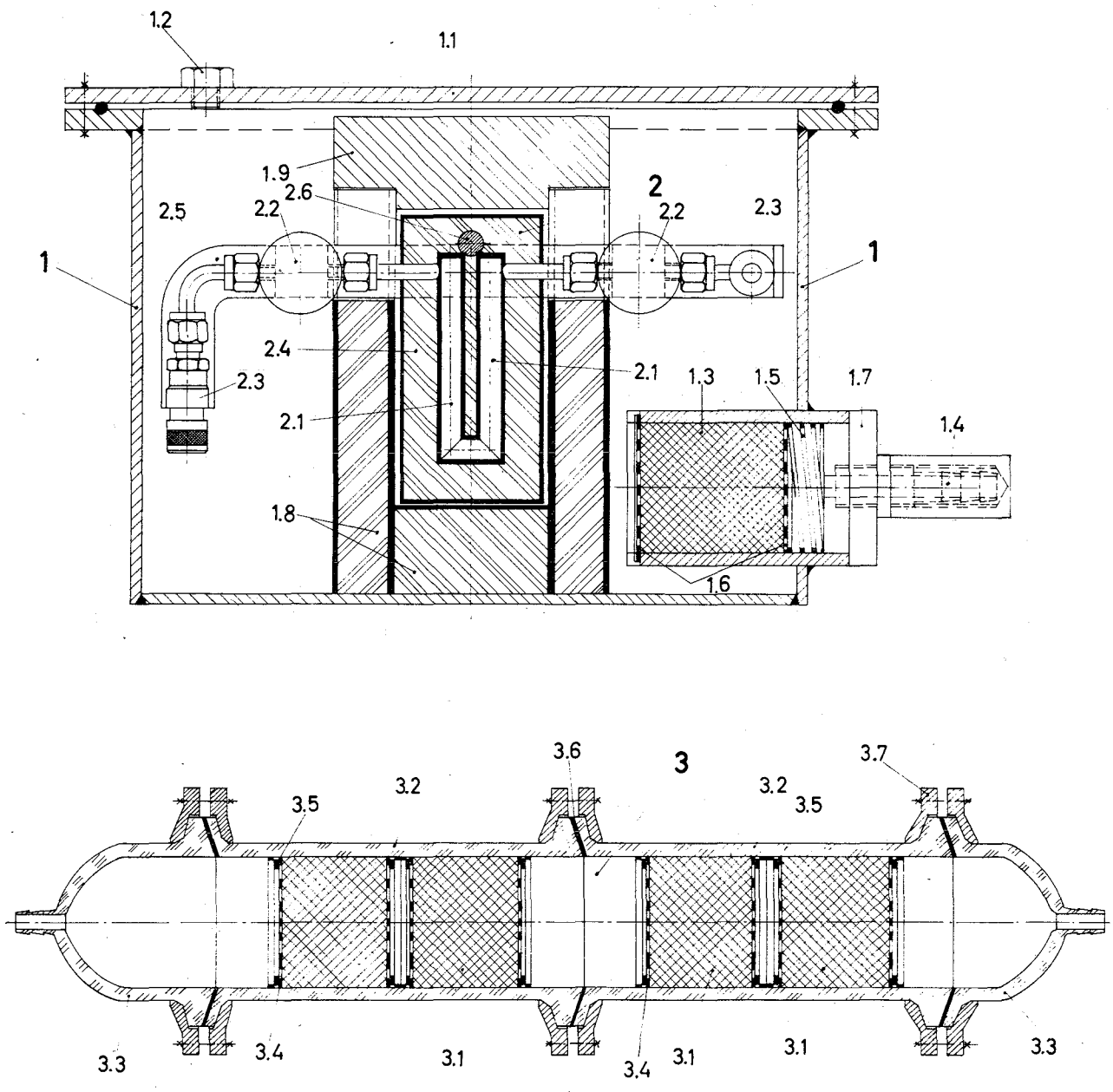
15 DRY TEST METER

16 RADIAL BLOWER

T TEMPERATURE

P PRESSURE

10.6.68 M. Groß



CONTAINER SYSTEM FOR RADIOACTIVE TEST AGENT,

CHARCOAL SAMPLER

FIG. 7

L E G E N D on Fig. 7

1	TRANSPORT CONTAINER
1.1	LID, GASTIGHT
1.2	AIR INLET (TO BE CLOSED GASTIGHT)
1.3	CHARCOAL FILTER
1.4	AIR OUTLET (TO BE CLOSED GASTIGHT)
1.5	SPRING
1.6	PERFORATED SHEET METAL
1.7	LID
1.8	SHIELDING
1.9	TOP OF SHIELDING, REMOVABLE
2	INNER CONTAINER
2.1	U-TUBE WITH TEST AGENT
2.2	BELLOWS SEAL VALVE
2.3	QUICK CONNECT WITH AUTOMATIC SHUT-OFF
2.4	SHIELDING, CAST AROUND U-TUBE
2.5	SUPPORT
2.6	HANDLE
3	CHARCOAL SAMPLER
3.1	CHARCOAL BED
3.2	GLASS-TUBE
3.3	END PIECE WITH TUBE CONNECTION
3.4	SCREEN
3.5	SPRING
3.6	TEFLON O-RING
3.7	FLANGE