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Retention of ^{131}I Volatilized from Aqueous Solutions by Sorbents

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Abstract

By far the largest proportion of the ^{131}I that was volatilized from alkaline aqueous solutions was in a form whose retention by various sorbents was lower than that of I_2, but higher than that of CH_3I. This form is called "HOI" although no direct identification exists. With all the fresh impregnated activated carbons investigated, the retention of "HOI" was higher than 99 % at a residence time of o.1 s. The influence of purging was insignificant.

Abscheidung von aus wässrigen Lösungen verflüchtigtem ^{131}I an Sorbentien

Kurzfassung

Bei weitem der größte Anteil des ^{131}I, das aus alkalischen wässrigen Lösungen verflüchtigt wurde, war in einer Form, die schlechter als I_2 und besser als CH_3I an verschiedenen Sorbentien abgeschieden wurde. Diese Form wird "HOI" genannt, obwohl keine direkte Identifikation vorliegt. Bei allen untersuchten frischen imprägnierten Aktivkohlen war bei einer Verweilzeit von 0.1 s die Abscheidung von "HOI" größer als 99 %. Der Einfluß des Spülens war nicht signifikant.

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1. Introduction

In the exhaust air of nuclear power plants gaseous radioiodine occurs largely in the form of elemental iodine (I_2) or methyl iodide (CH_3I). However, also other gaseous iodine species are to be found some of which may be retained to a small extent only by the activated carbons contained in iodine filters. The percentage of the penetrating iodine species in the exhaust air of nuclear power plants is very low in general, however, large proportions (order of magnitude: 50 %) cannot be entirely excluded /1/. The nature of the penetrating iodine species is not known.

As for I_2 und CH_3I, many data are to be found in the literature on the retention of these species by activated carbons /2-4/. However, only a few data have been published on the retention of other iodine species. And these may be quite contradictory. This applies in particular to hypoiodous acid (HOI).

It appears that iodine which evaporates from aqueous solutions may be largely in the form of HOI under certain conditions /5-14/. This species may be formed by hydrolysis of I_2 in aqueous solutions:

$$I_2 + H_2O \rightleftharpoons HOI + H^+ + I^-$$

It must be emphasized that there is only indirect evidence for the existence of gaseous HOI, e.g. the particular sorption behavior of iodine volatilized from aqueous solutions. Direct measurements of gaseous HOI (e.g. by mass spectrometry) were not completely conclusive or negative /11,15,16/. Therefore, some authors use the formula HOI for convenience only. This is also our attitude. (To indicate this we use the designation "HOI" in the remaining part of this report.)

This paper deals mainly with the retention of "HOI" by activated carbons. In addition, sorbents that are used in iodine species samplers are dealt with to a certain extent. Some early results obtained in our laboratory have already been published /12/.

2. Previous Investigations

In this chapter a review is given of investigations described in the literature on the retention of "HOI" or, strictly speaking, of iodine volatilized from aqueous solutions. Both activated carbons and sorbents used in iodine species samplers are dealt with.

Activated carbons are usually impregnated (KI, TEDA or other compounds) to efficiently retain as many iodine species as possible even under adverse conditions. To the contrary, sorbents used in iodine species samplers are prepared in such a way that a few or even a single iodine species is retained to a high extent whereas other iodine species are retained to a negligible extent only. (Thus, by using these specific sorbents in an appropriate order, the proportions of the various iodine species contained in a mixture may be easily determined.)

Only very few data are to be found in the literature on the retention of "HOI" by activated carbons. In one paper /8/ results of investigations with three activated carbons are presented. The results may be summarized as follows (room temperature, relative humidity of 50-100 %, face velocity of 22 cm/s):

(a) The penetration of a TEDA-impregnated carbon was slightly lower than that of a KI-impregnated carbon and much lower than that of a carbon without impregnant.

(b) The penetration increased with increasing relative humidity, purging time and aging time.

(c) The penetration decreased with increasing "HOI" concentration.

With the fresh TEDA-impregnated carbon, at a relative humidity of nearly 100 % and at a residence time of 0.1 s, the penetration was about 7 % at a purging time of 2 h and about 13 % at a purging time of 60 h.

We have published some results on the retention of "HOI" by a KI-impregnated carbon under similar conditions (40 °C, ≈ 100 % R.H., 25 cm/s) /12/. At a residence time of 0.1 s the penetration was around 2 % at a purging time of 2 h and around 9 % at a purging time of nearly 190 h. (At higher residence times a smaller difference in penetration was observed.)

As for the sorbents used in iodine species samplers, literature data of two I_2 sorbents (DSM11, CDI) and two "HOI" sorbents (IPH, ISORB C3) are presented in the following paragraphs. (These sorbents were also used in the investigations described in this paper. See Table 2.)

The data published on the penetration of DSM11 and CDI by "HOI" are essentially as follows:
With DSM11 the penetration was approximately 90 % at a residence time of 0.1 s and a purging time of 2 h (30 °C, 20 % R.H., 25 cm/s) /12/. CDI exhibited a penetration of 96-97 % at a residence time of 0.1 s (purging time, temperature and relative humidity not given; face velocity: 25 cm/s) /7/.

Some more data are available on the penetration of IPH by "HOI". In the paper cited above /7/ a penetration of about 4 % at a residence time of 0.1 s is reported (information on the test conditions as above). In another paper /8/ a penetration of about 40 % at a residence time of 0.1 s with no purging after loading is given (room temperature, 50 % R.H., 22 cm/s). In tests under various conditions, generally a penetration of less than 10 % was found at a residence time of 0.1 s and a purging time of 2 h (approximately 20-40 °C, 10-90 % R.H., 15-30 cm/s) /9/. A higher penetration was observed with a "bad" batch of IPH in these tests. In other investigations a penetration of about 10 % was observed at the same residence time and purging time (30 °C, 20 % R.H., 25 cm/s) /12/.

As for ISORB C3, a penetration of around 1-3 % was found for "HOI" at a residence time of 0.1 s and a purging time of 2 h (22 °C, 40-100 % R.H., 25 cm/s) /10/.

3. Experimental

Data of the commercial activated carbons investigated are contained in Table 1. The activated carbons 207B (KI), 207B (TEDA), Kiteg II and Radshield 25 have been on the market for several years, whereas 207BPA (KI) and Q have been developed recently. For comparison, aged 207B (KI) and (fresh) 207B without impregnant were also examined. Data of the other sorbents investigated are given in Table 2. As already mentioned, two I_2 sorbents (DSM11, CDI) and two "HOI" sorbents (IPH, ISORB C3) were used.

"HOI" was prepared by passing gaseous I_2, contained in a helium stream, with glass frit diffusers through two successive 0.1 N NaOH solutions. (In some comparison tests also solutions with a lower NaOH concentration were used.) The iodine evolved from the two 0.1 N NaOH solutions contained negligible proportions of I_2 und CH_3I (see Chap. 4). For comparison, also tests with I_2 and CH_3I were performed. These species were prepared by standard procedures /4/.

The values of the test parameters are presented in Table 3. The activated carbons were tested at the adverse relative humidity of 98-100 %. The purging time was either 2 or 168 h (1 week) in these tests. In the tests of the other sorbents the relative humidity was 20 % and the purging time 2 h. (The comparison tests with I_2 and CH_3I were carried out under the same conditions with a purging time of 2 h.)

The test beds were sectioned to establish the retention or penetration as a function of the bed depth or residence time. The backup beds, consisting of 207B (KI), were operated at the favorable retention temperature of 80 °C. Both in the test beds and backup beds the total residence time was 0.4 s each.

The test apparatus corresponded in principle to our standard test apparatus /4/. However, in the investigations with "HOI" two parallel trains were installed each containing test beds and backup beds as described above.

The iodine species were tagged with ^{131}I. Activities of about 10^{-3} Ci ^{131}I per test were used. In the "HOI" tests, 10^{-2} to 10^{-1} % of this activity was evolved from the two o.1 N NaOH solutions. As the detection limit for ^{131}I retained on individual beds was approximately 10^{-11} Ci, the minimum detectable penetration was 10^{-3} to 10^{-2} % in the "HOI" tests.

The reproducibility of the test results is illustrated with the results of two different (not parallel) tests under the same conditions. Fig. 1 shows the penetration as a function of the bed depth in these tests. The values of two additional tests were in the same range (not shown).

When interpreting semilogarithmic graphs of penetration versus bed depth (Fig. 1), one should bear in mind the following principles:

(a) the type of an individual species with respect to its sorption behavior is characterized by the slope of the corresponding straight line (possibly after resolving the curve);

(b) the percentage of an individual species is given by the point of intersection of the corresponding straight line with the ordinate axis.

(These principles apply if no desorption occurs. With desorption it is more difficult to interpret these graphs.)

In the tests mentioned above, according to Fig. 1 by far the most ^{131}I was in a form that was strongly retained (steep part of the penetration curves). This species is called "HOI". A small percentage (10^{-2}-10^{-1} %) was in a form that was retained to a small extent only (flat part of the penetration curves). Small percentages of penetrating iodine species may also be found in tests with other iodine compounds (e.g. I_2) /17,18/.

Table 1: Data of the activated carbons investigated

Designation	Base material	Particle size (mesh)[a]	Impregnant	Supplier
207B (KI)	coal	8-12	KI	Sutcliffe Speakman, U.K.
207B (TEDA)	coal	8-12	TEDA	Sutcliffe Speakman, U.K.
Kiteg II	coconut shell	8-16	KI, tert. amine	Nuclear Consulting Services, U.S.A.
Radshield 25	coconut shell	8-16	tert. amine	Charcoal Engineering, U.S.A
207BPA (KI)	coal	8-12	KI	Sutcliffe Speakman, U.K.
Q	coconut shell	8-16	quinuclidine	Nuclear Consulting Services, U.S.A.
207B (KI)[b]	coal	8-12	KI	Sutcliffe Speakman, U.K.
207B	coal	8-12	none	Sutcliffe Speakman, U.K.

[a] 8-12 mesh: BS 410; 8-16 mesh: ASTM D2862

[b] Aged in the purge air of a boiling water reactor over about 18 months (operation not continuous)

Table 2: Data of the other sorbents investigated

Designation	Base material	Particle size	Impregnant	Supplier
DSM11[a]	silicid acid	1-2 mm (beads)	KI	Südchemie, F.R.G.
CDI[a]	diatomaceous earth	20-40[c] mesh	CdI_2	Science Applications, U.S.A.
IPH[b]	activated alumina	20-40[c] mesh	4-Iodophenol	Science Applications, U.S.A.
ISORB C3[b]	activated carbon	20 mesh[c]	-[d]	R.C. Contamination, Canada

[a] I_2 sorbent

[b] "HOI" sorbent

[c] ASTM D2862

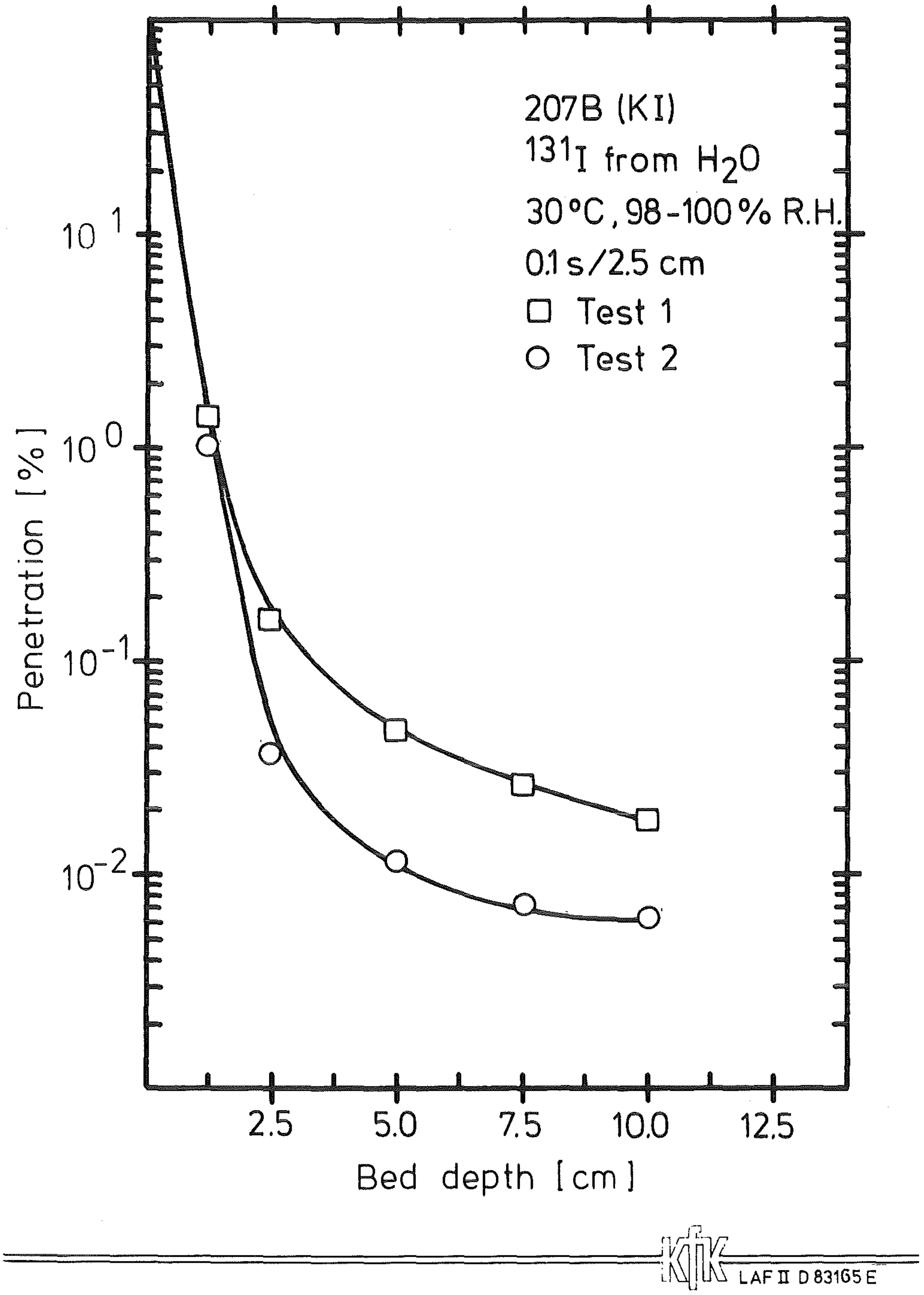
[d] Not known

Table 3: Test parameters

Parameter	Unit	Value
Temperature	^{o}C	30
Relative humidity	%	20 or 98-100
Face velocity	cm/s	25
Bed depth[a]	cm	10
Residence time	s	0.4
Preconditioning time	h	$\geq$ 16
Injection time	h	1
Purging time	h	2 or 168
Carrier concentration	g/m^3	10^{-7}-10^{-6}[b]

[a] 4 succesive test beds of depth 2.5 cm were used. The first test bed consisted of sections of depth 1.25 cm. (Bed diameter: 2.5 cm)

[b] 10^{-3} g/m^3 in the comparison tests with I_2 and CH_3I



Penetration as function of bed depth
in tests at the same conditions

Fig. 1[a]

[a] Purging time: 2 h (also in the other tables and figures unless otherwise stated); other parameters: see Table 3; different (not parallel) tests

4. Results

In this chapter the results are presented of the investigations on the retention of ^{131}I volatilized from aqueous solutions by the sorbents contained in Tables 1 and 2 under the conditions indicated in Table 3. ^{131}I volatilized from aqueous solutions is sometimes called "^{131}I from H_2O" here. As already indicated, the purging time was either 2 or 168 h. No reference to the purging time implies a duration of 2 h. Tests with different purging times but the same sorbent were conducted in parallel (using parallel test trains). First the activated carbons will be dealt with, then the other sorbents.

4.1. Activated Carbons

Table 4 and Figs. 2 and 3 give an overview of the penetration of the activated carbons by ^{131}I volatilized from aqueous solutions at two bed depths and purging times. The main results are as follows:

(a) The penetration found with different fresh impregnated carbons differed to a small extent only, at both purging times. At a purging time of 2 h, the penetration was of the order of 10^{-1} % at a bed depth of 2.5 cm and of 10^{-2} % at a bed depth of 10.0 cm. At a purging time of 168 h, the penetration was higher by factors of between 1 and 5.

(b) The penetration of the aged 207B (KI) was somewhat higher than that of the fresh 207B (KI). The difference was equivalent to factors of less than 5.

(c) The penetration of 207B (no impregnant) was much higher than that of 207B (KI). The difference was equivalent to factors of greater than 10.

Figs. 4 to 11 illustrate the penetration of the various carbons by ^{131}I from H_2O as a function of the bed depth at different purging times. In some cases the corresponding values for I_2 and CH_3I are also given (purging time: 2 h).

The penetration curves for ^{131}I from H_2O were generally steep up to a bed depth of about 2.5 cm and then flattened out. This applies to both purging times. At small bed depths (< 2.5 cm) the influence of the purging time was mostly less pronounced than at large bed depths.

Up to the maximum bed depth investigated (10 cm), in all the cases the penetration by ^{131}I from H_2O was higher than that by I_2 and lower than that by CH_3I. At somewhat higher bed depths a penetration higher than that of CH_3I can be anticipated for the fresh impregnated carbons. However, this penetration would still be lower than 10^{-1} % under the conditions investigated.

As explained in Chap. 3, the steep part of the penetration curves for ^{131}I from H_2O can be ascribed to "HOI" which was by far the predominant iodine species. The flat part of the penetration curves can be attributed to penetrating iodine species present in small proportions (10^{-2}-10^{-1} %).

The penetration of impregnated activated carbons by "HOI" as found in the present investigations was generally lower than that obtained in previous studies (compare Chap. 2). This discrepancy, corresponding to roughly one order of magnitude at a residence time of 0.1 s, is not fully understood. The purity of "HOI" may play an important part.

From the present more comprehensive investigations it is concluded that "HOI" should be no problem for iodine filtration even if this species occurred in substantial proportions in the air to be filtered. In particular, there appears to be no need for testing fresh or aged activated carbons with "HOI" routinely.

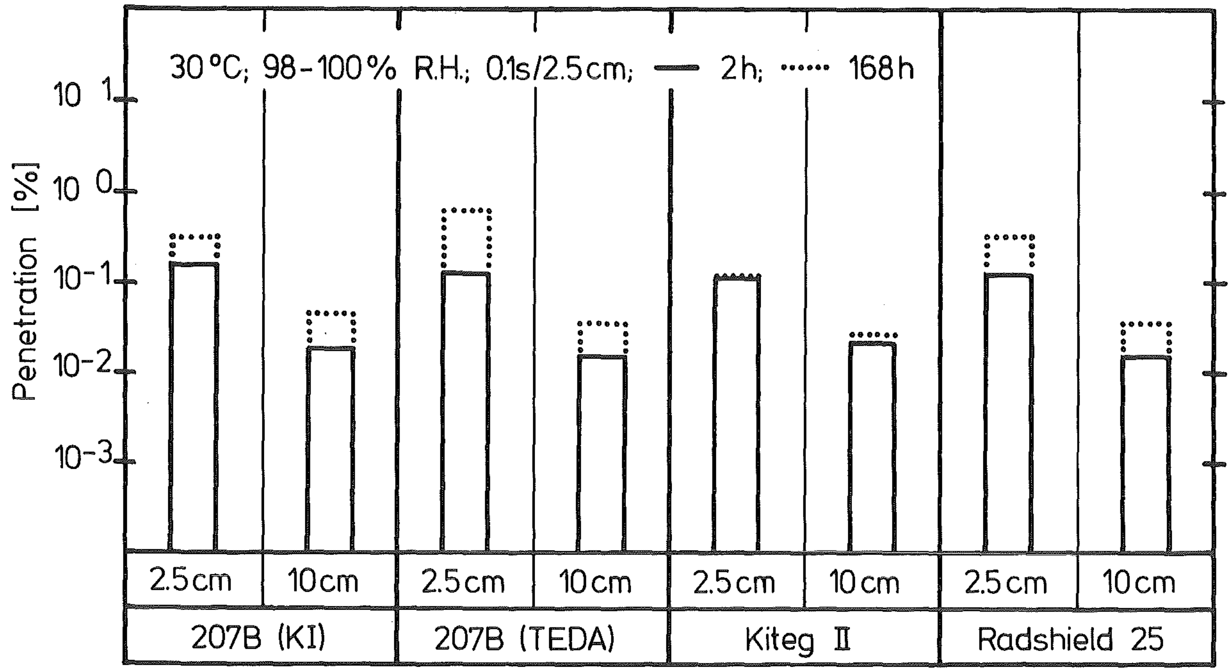
Table 4: Penetration of various activated carbons by ^{131}I volatilized from aqueous solutions at different bed depths and purging times
(compare Figs. 2 and 3)
Relative humidity: 98-100 %
Other parameters : see Table 3

Activated carbon	Bed depth (cm)	Residence time (s)	Penetration (%)[a] 2 h[b]	168 h[b]
207B (KI)	2.5 10.0	0.1 0.4	1.6(-1) 1.9(-2)	3.3(-1) 4.6(-2)
207B (TEDA)	2.5 10.0	0.1 0.4	1.3(-1) 1.6(-2)	6.8(-1) 3.7(-2)
Kiteg II	2.5 10.0	0.1 0.4	1.1(-1) 2.1(-2)	1.1(-1) 2.9(-2)
Radshield 25	2.5 10.0	0.1 0.4	1.2(-1) 1.6(-2)	3.4(-1) 3.9(-2)
207BPA (KI)	2.5 10.0	0.1 0.4	4.7(-1) 1.7(-2)	7.4(-1) 5.8(-2)
Q	2.5 10.0	0.1 0.4	1.9(-1) 7.2(-3)	2.4(-1) 1.6(-2)
207B (KI)[c]	2.5 10.0	0.1 0.4	7.2(-1) 7.6(-2)	1.4(0) 9.9(-2)
207B	2.5 10.0	0.1 0.4	3.6(0) 2.4(0)	6.4(0) 3.9(0)

[a] 1.6(-1)=1.6 · 10^{-1} etc.

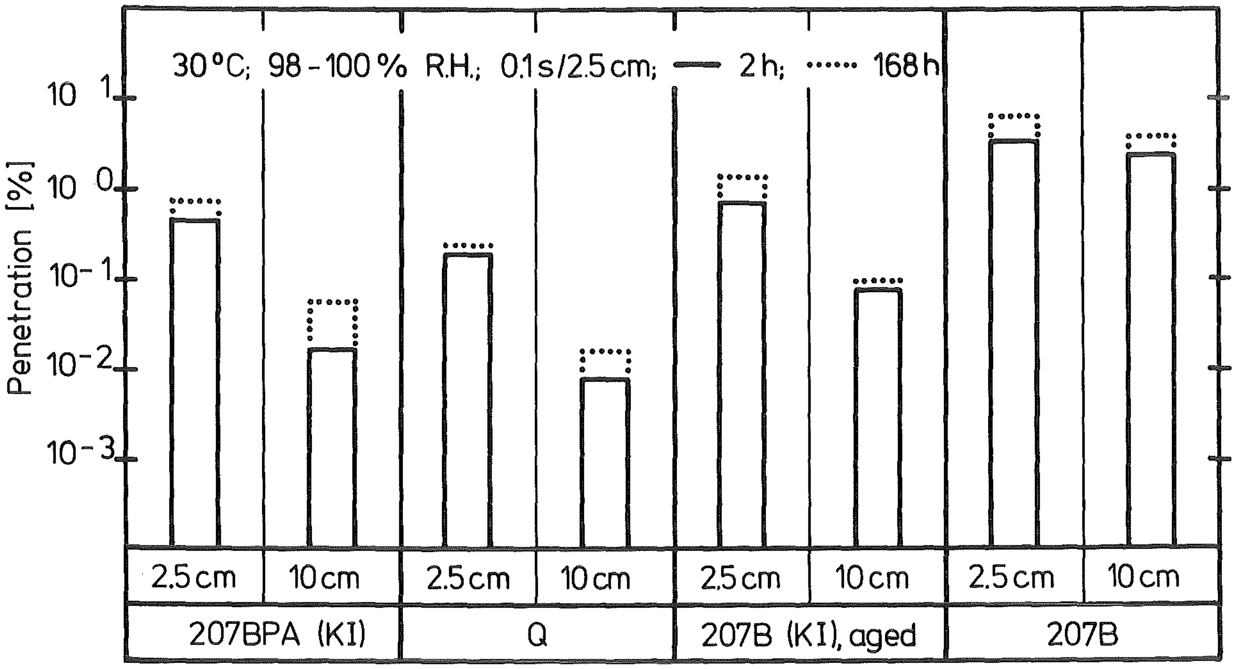
[b] Purging time

[c] Aged (see Table 1)



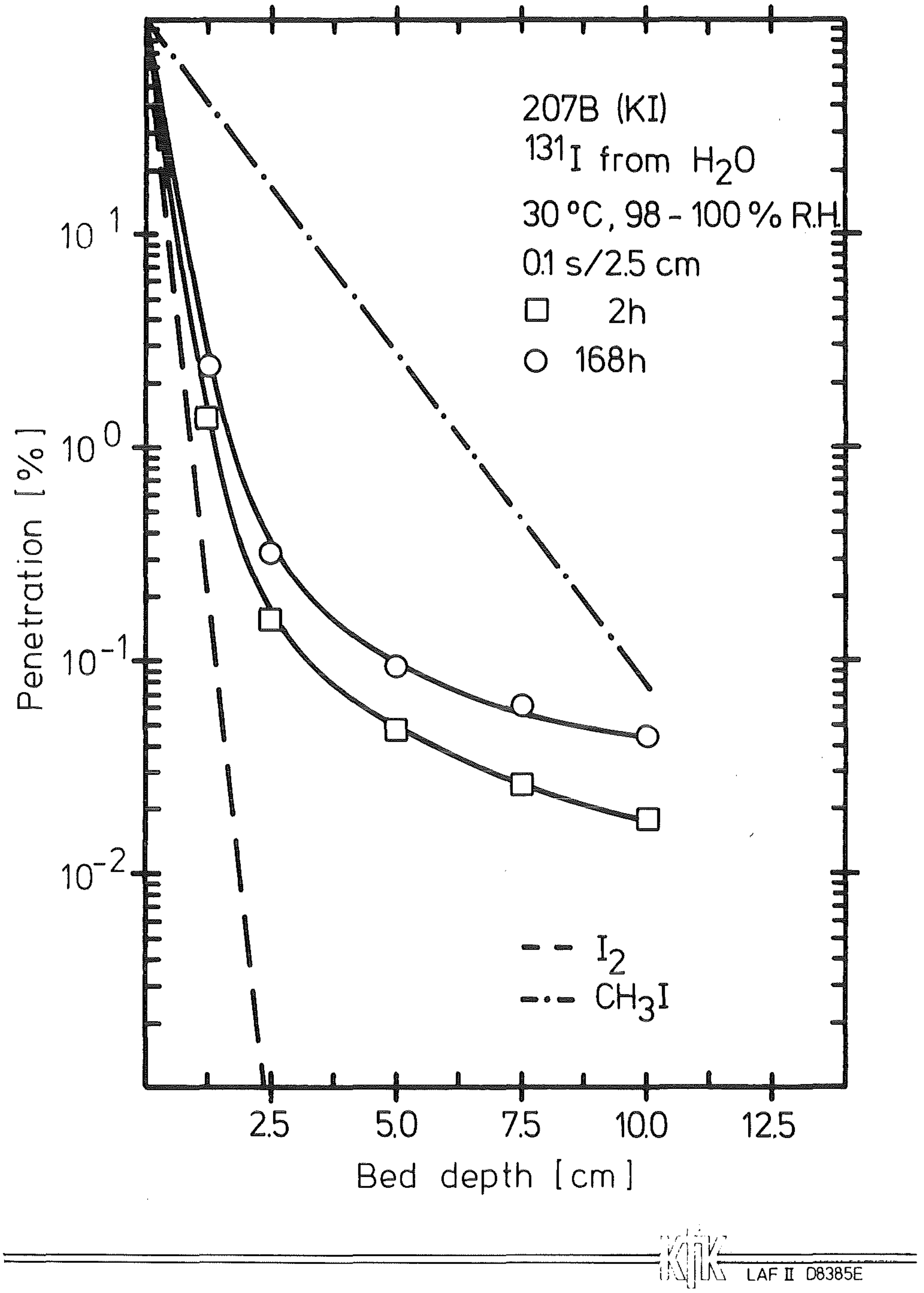
Penetration of various activated carbons by ^{131}I, volatilized from aqueous solutions, at different bed depths and purging times

Fig. 2



Penetration of various activated carbons by ^{131}I, volatilized from aqueous solutions, at different bed depths and purging times

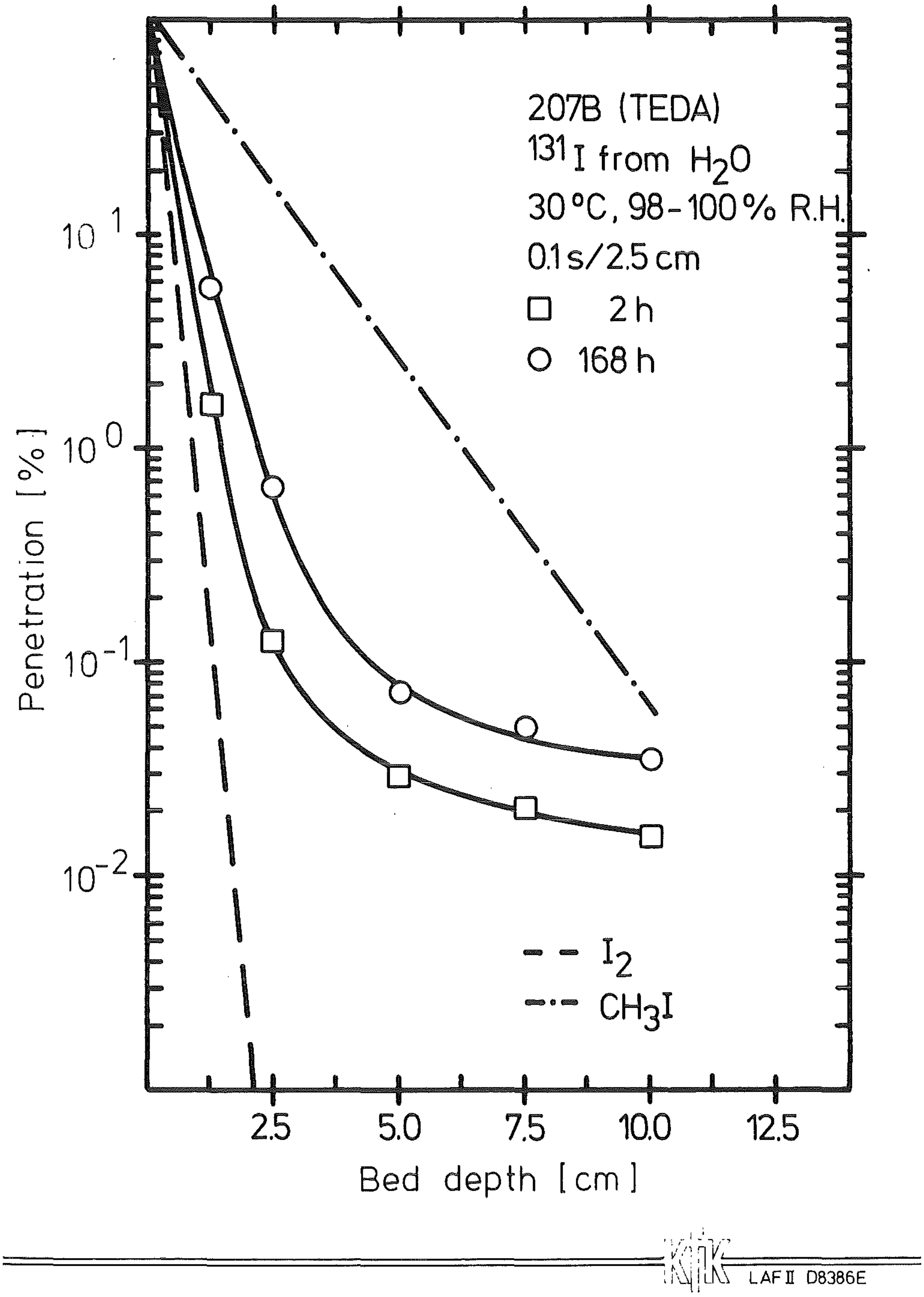
Fig. 3



Penetration as function of bed depth at different purging times

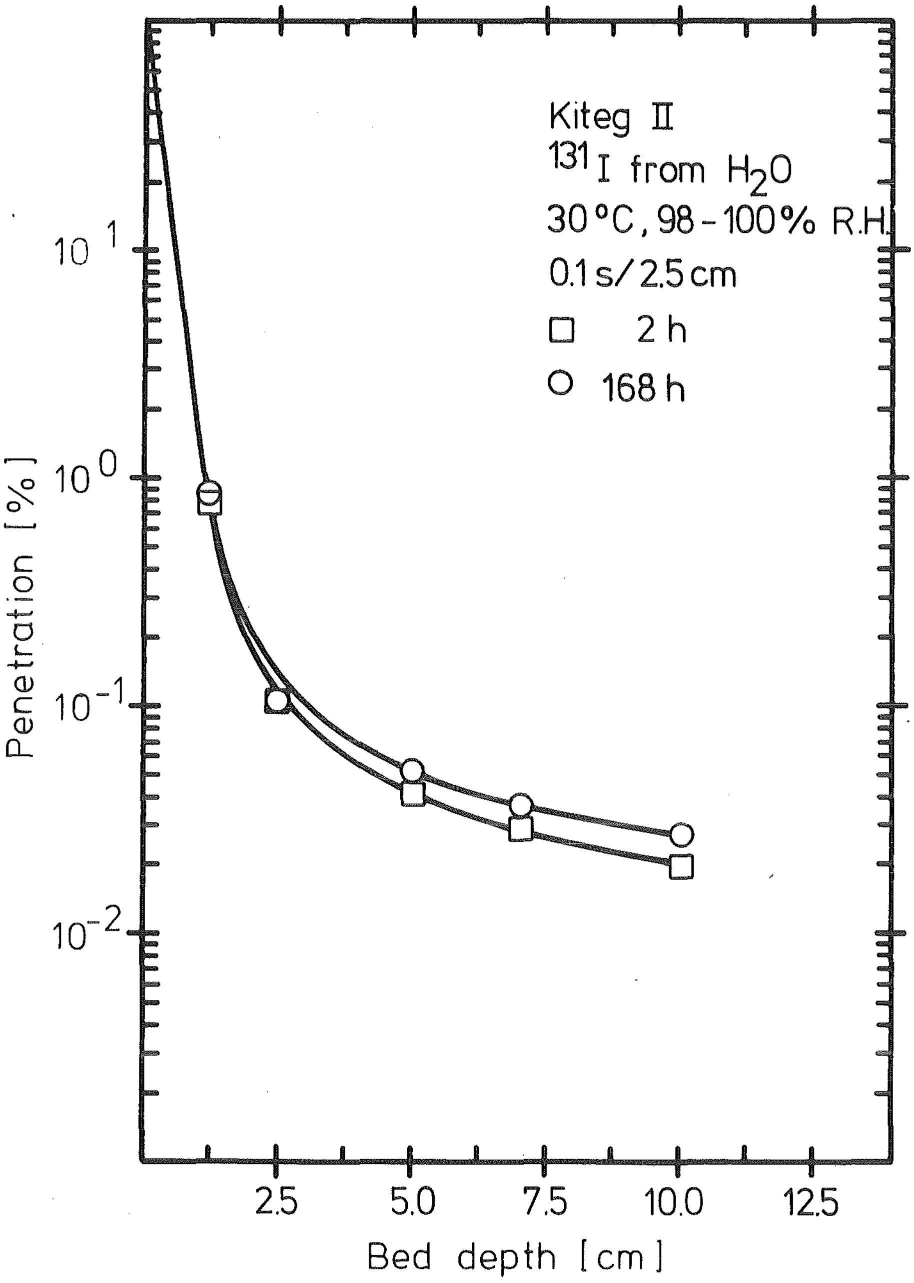
Fig. 4[a]

[a] 2 h: Test 1 (compare Fig. 1)



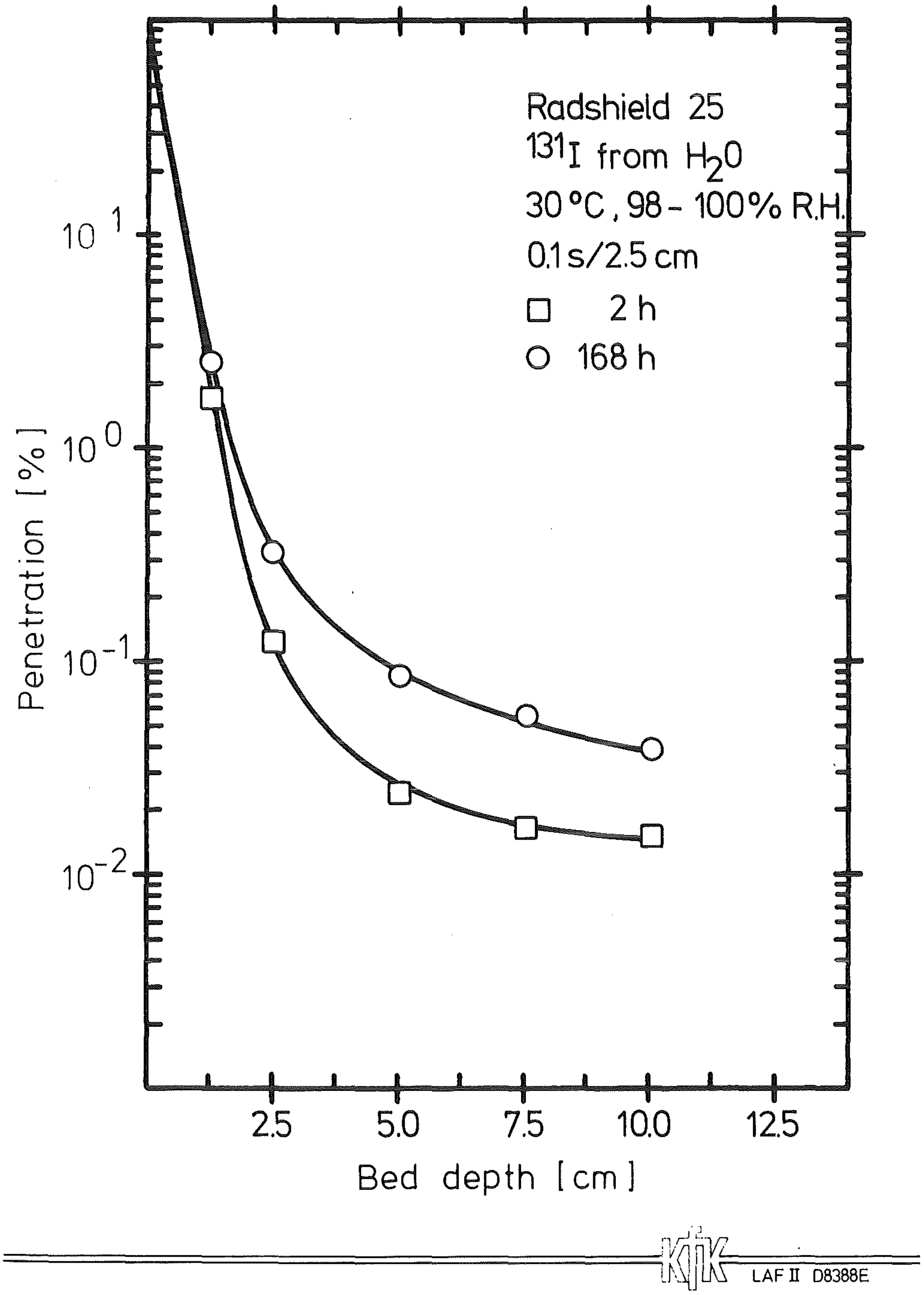
Penetration as function of bed depth
at different purging times

Fig. 5



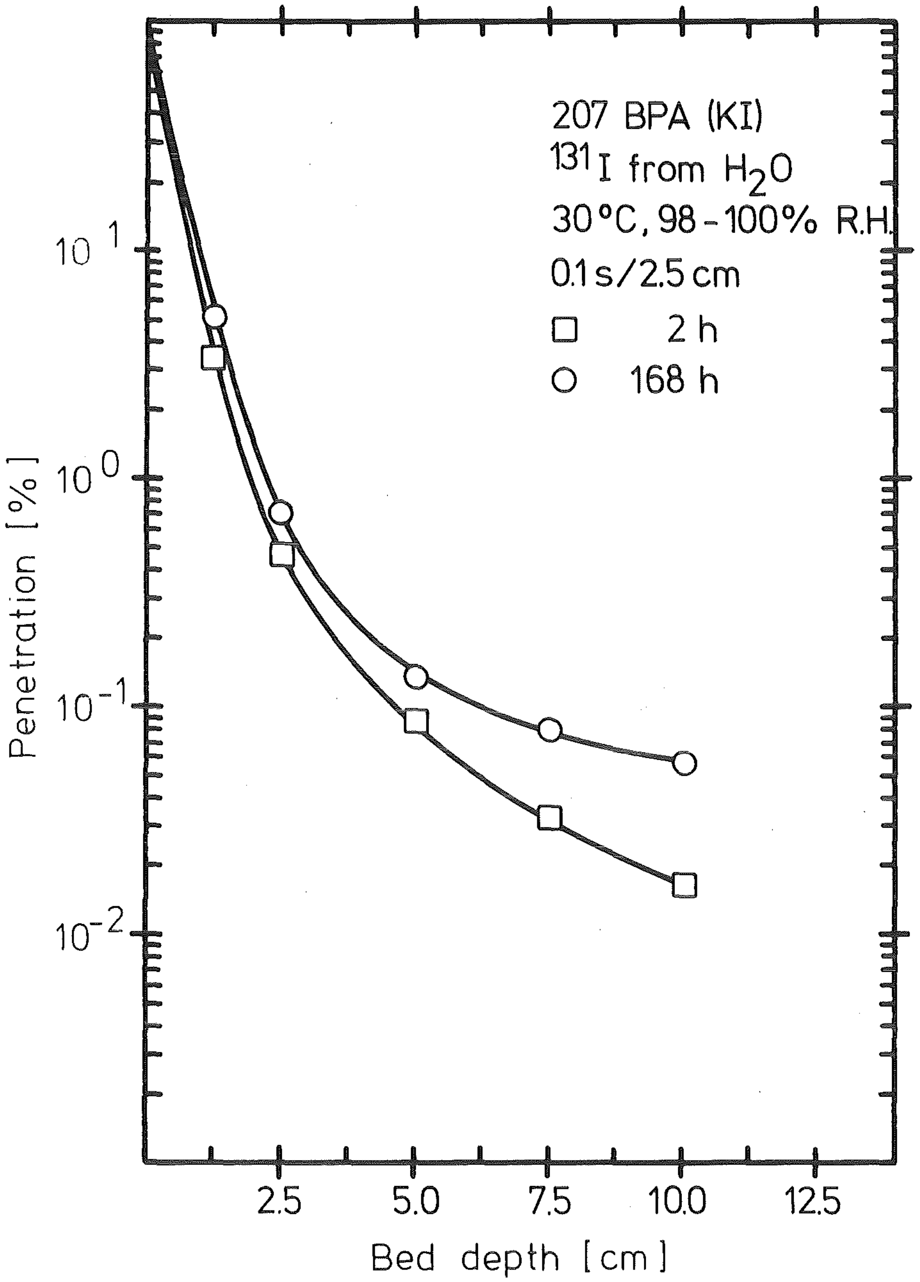
Penetration as function of bed depth at different purging times

Fig. 6



Penetration as function of bed depth at different purging times

Fig. 7



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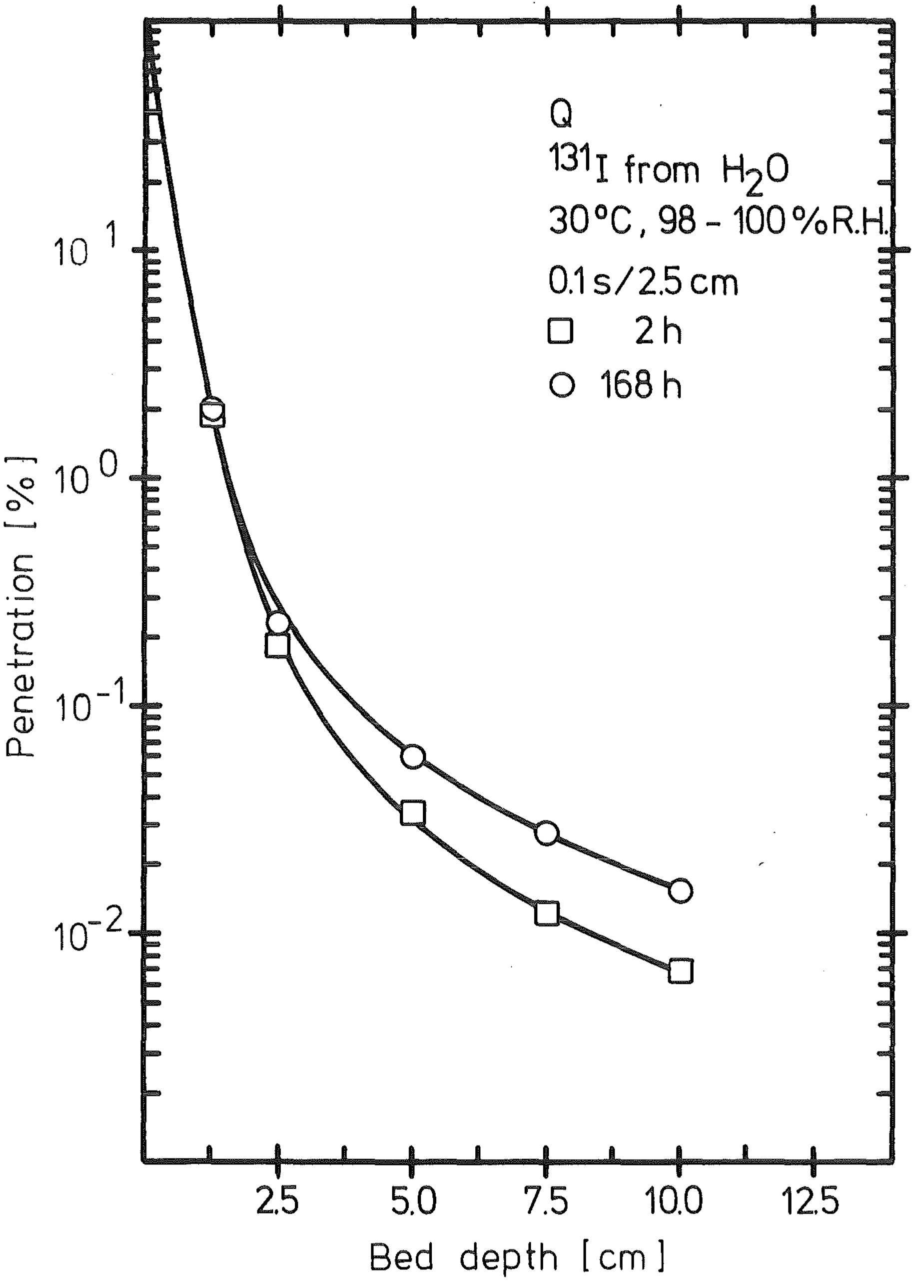
Penetration as function of bed depth at different purging times

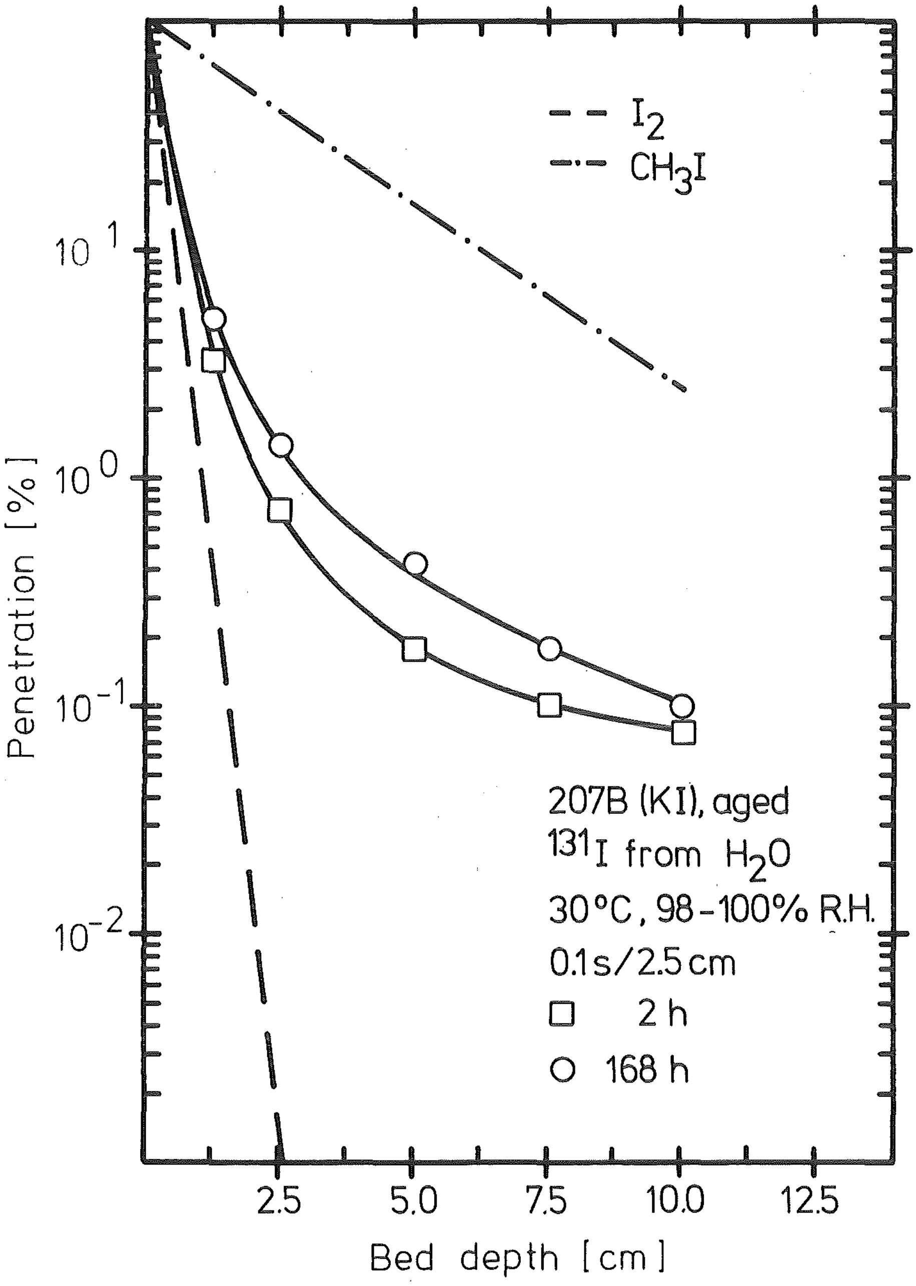
Fig. 8



LAF II D8390E

Penetration as function of bed depth at different purging times

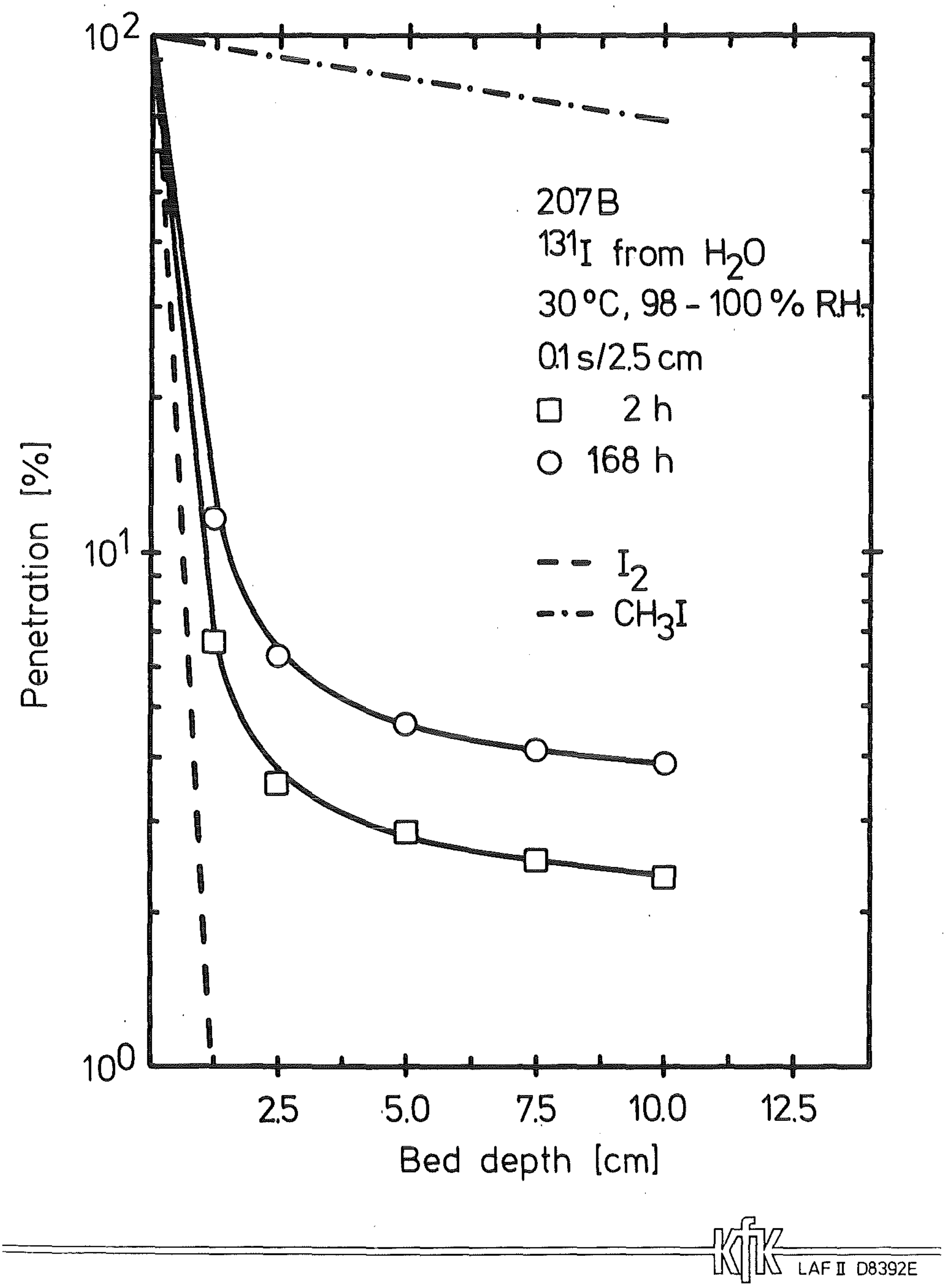
Fig. 9



LAF II D8391E

Penetration as function of bed depth
at different purging times

Fig. 10



Penetration as function of bed depth at different purging times

Fig. 11

4.2 Other Sorbents

Table 5 and Fig. 12 give an overview of the penetration of the other sorbents by ^{131}I volatilized from aqueous solutions at two bed depths. The main results are as follows:

(a) With the I_2 sorbents (DSM11, CDI) a very high penetration was found. At a bed depth of 2.5 cm the penetration was higher than 99 %.

(b) With the "HOI" sorbents (IPH, ISORC C3) a comparatively small penetration was observed. The penetration was between 1 and 10 % at both bed depths.

Figs. 13 to 16 show the penetration of the various sorbents by ^{131}I from H_2O as a function of the bed depth. In all the cases the corresponding values for I_2 and CH_3I are also presented. The penetration curves for ^{131}I from H_2O were concave for the I_2 sorbents and convex for the "HOI" sorbents (in the direction of increasing penetration). In all the cases the penetration by ^{131}I from H_2O was higher than that by I_2 and lower than that by CH_3I.

In particular from the penetration of the I_2 sorbents it is obvious that the ^{131}I volatilized from the standard aqueous solutions (p_H=13) contained negligible proportions of I_2. It is mentioned here that with decreasing p_H of the aqueous solutions increasing proportions of I_2 were found. From the penetration of the "HOI" sorbents it can be seen that also the proportions of CH_3I and similarly behaving iodine species were small (order of magnitude:1 %).

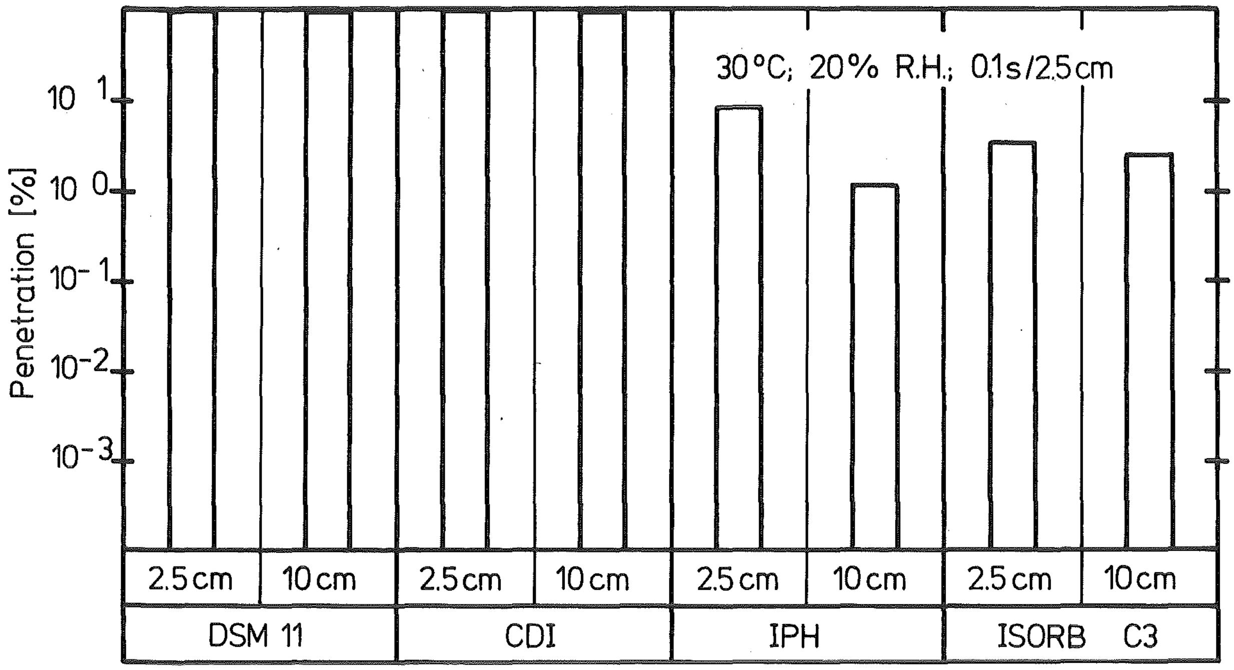
With the precision available in the tests with "HOI", the results obtained with the I_2 and "HOI" sorbents in the present investigations can be regarded as being largely in agreement with those of most of previous studies (compare Chap. 2).

Table 5: Penetration of various sorbents by ^{131}I volatilized from aqueous solutions at different bed depths (compare Fig. 12)

Relative humidity: 20 %
Purging time : 2 h
Other parameters : see Table 3

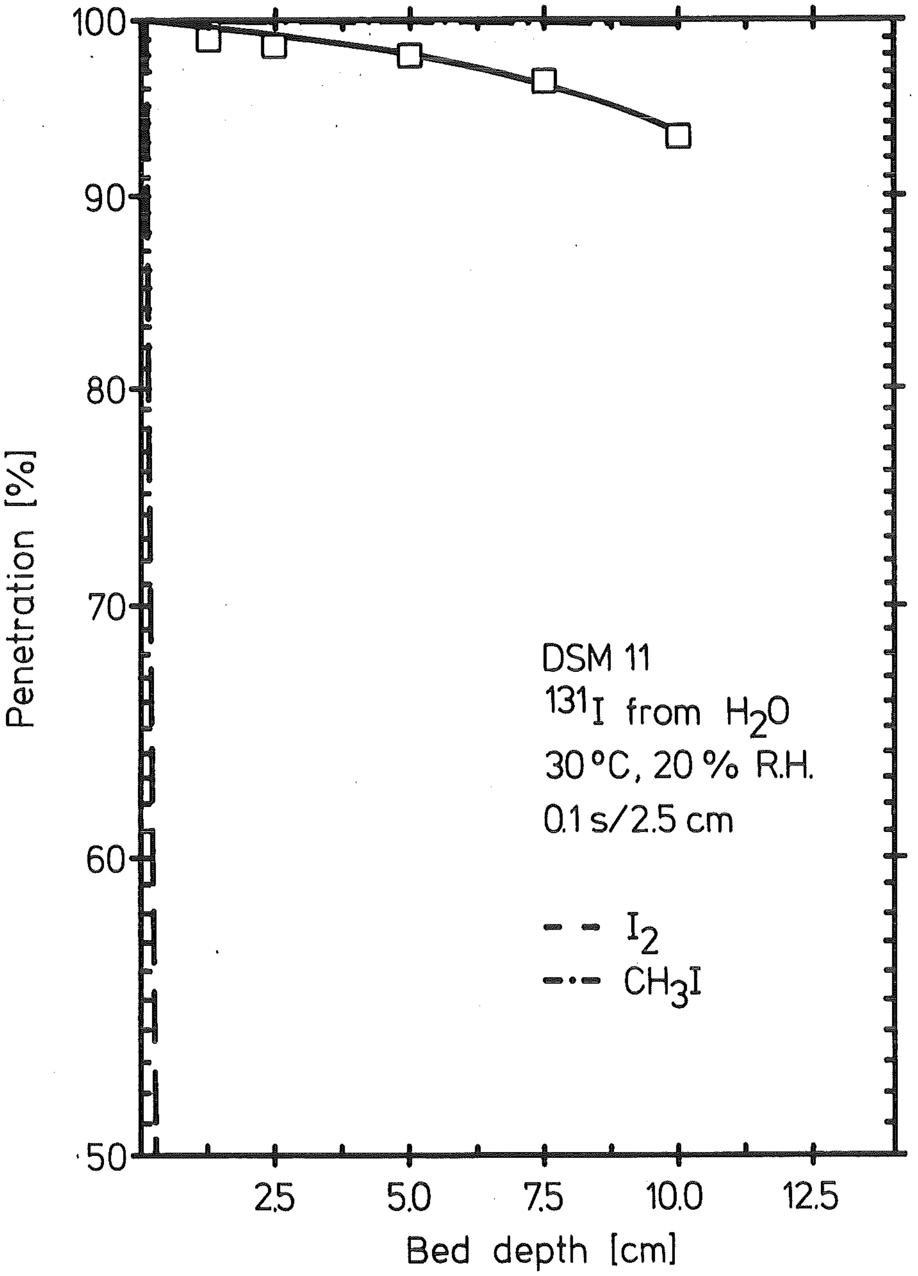
Sorbent	Bed depth (cm)	Residence time (s)	Penetration[a] (%)
DSM11	2.5 10.0	0.1 0.4	9.9(1) 9.3(1)
CDI	2.5 10.0	0.1 0.4	1.0(2) 9.5(1)
IPH	2.5 10.0	0.1 0.4	8.9(0) 1.1(0)
ISORB C3	2.5 10.0	0.1 0.4	3.5(0) 2.6(0)

[a] $9.9(1)=9.9 \cdot 10^{1}$ etc.



Penetration of various sorbents by ^{131}I, volatilized from aqueous solutions, at different bed depths

Fig. 12



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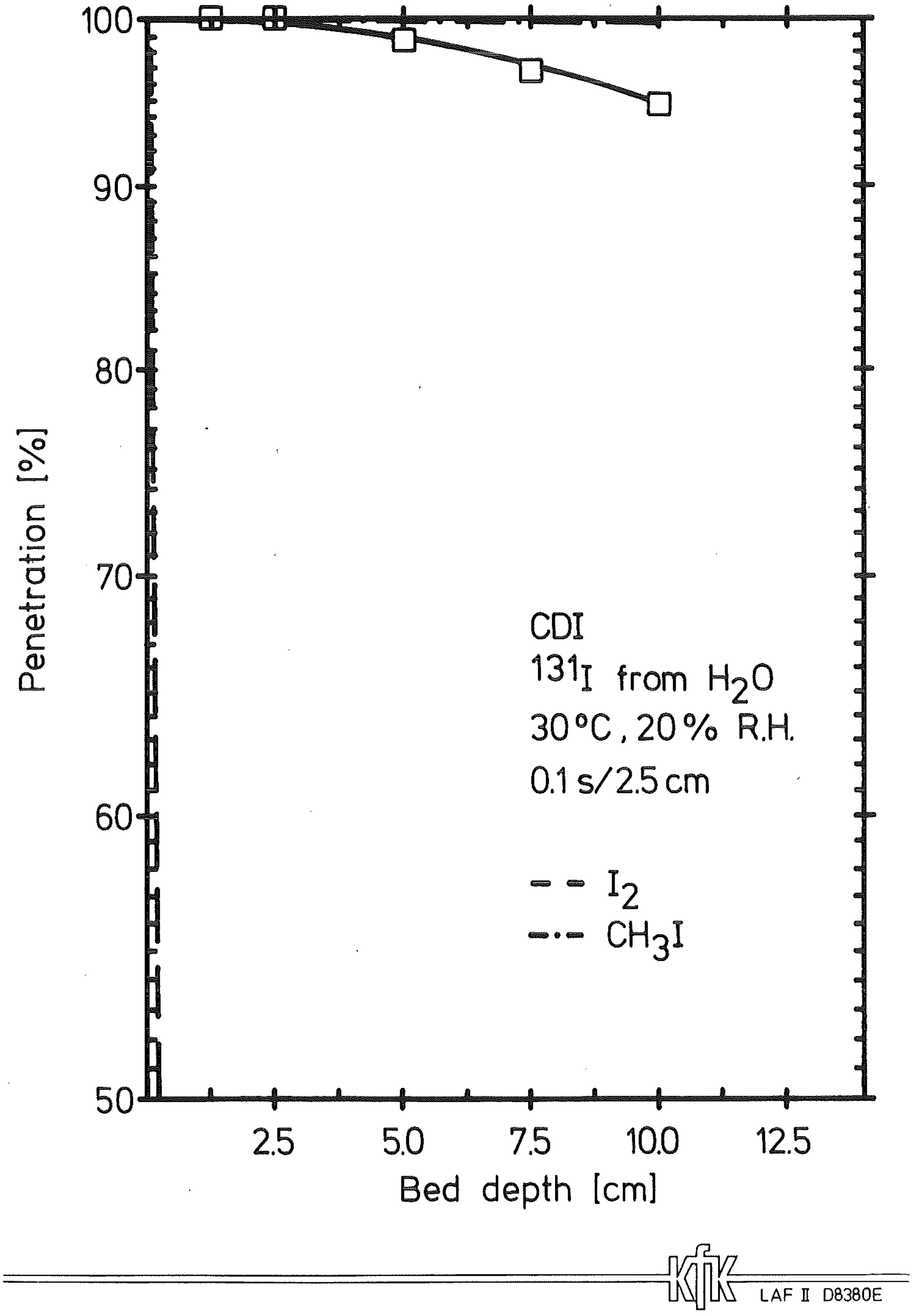
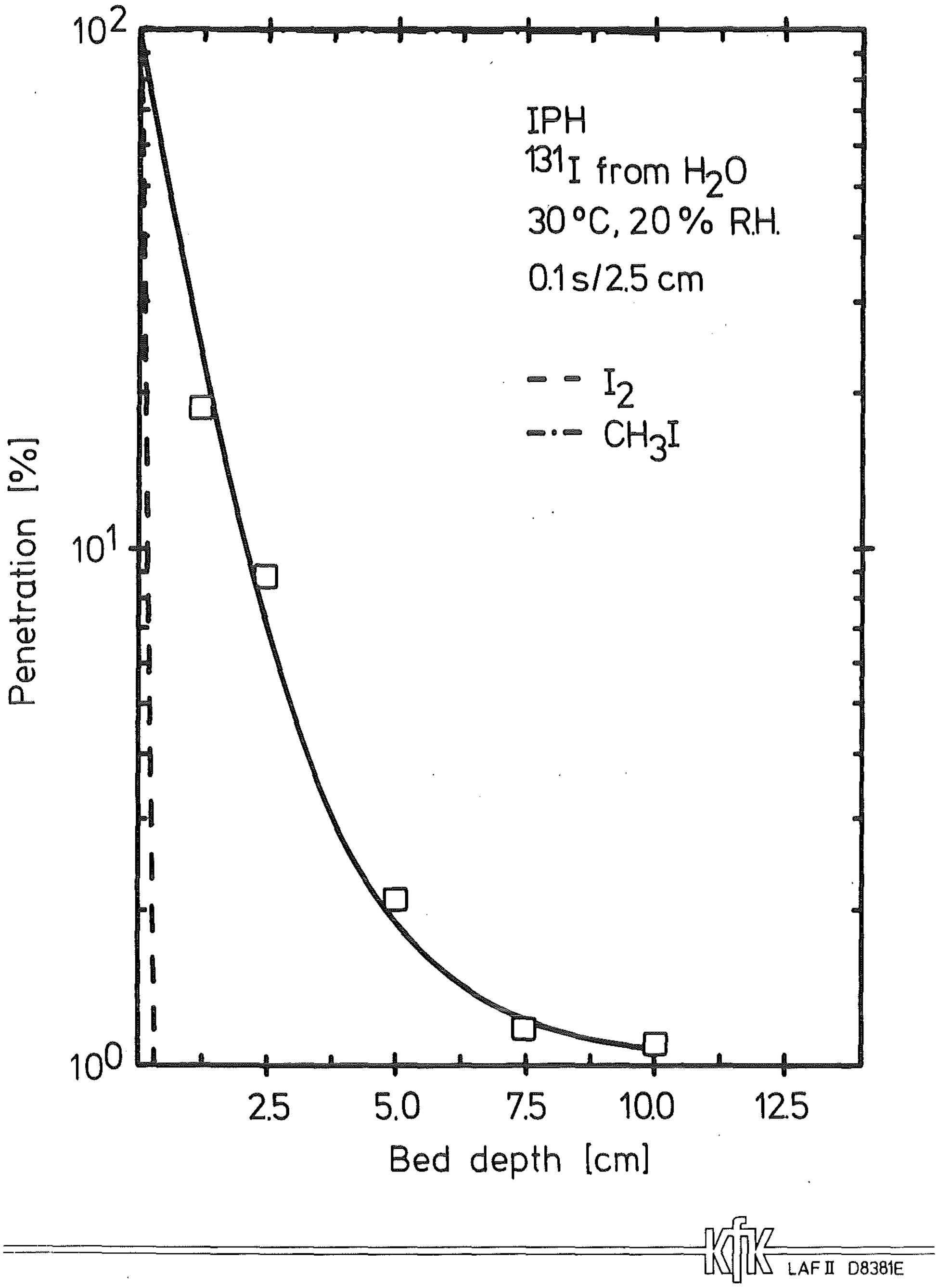
Penetration as function of bed depth

Fig. 13



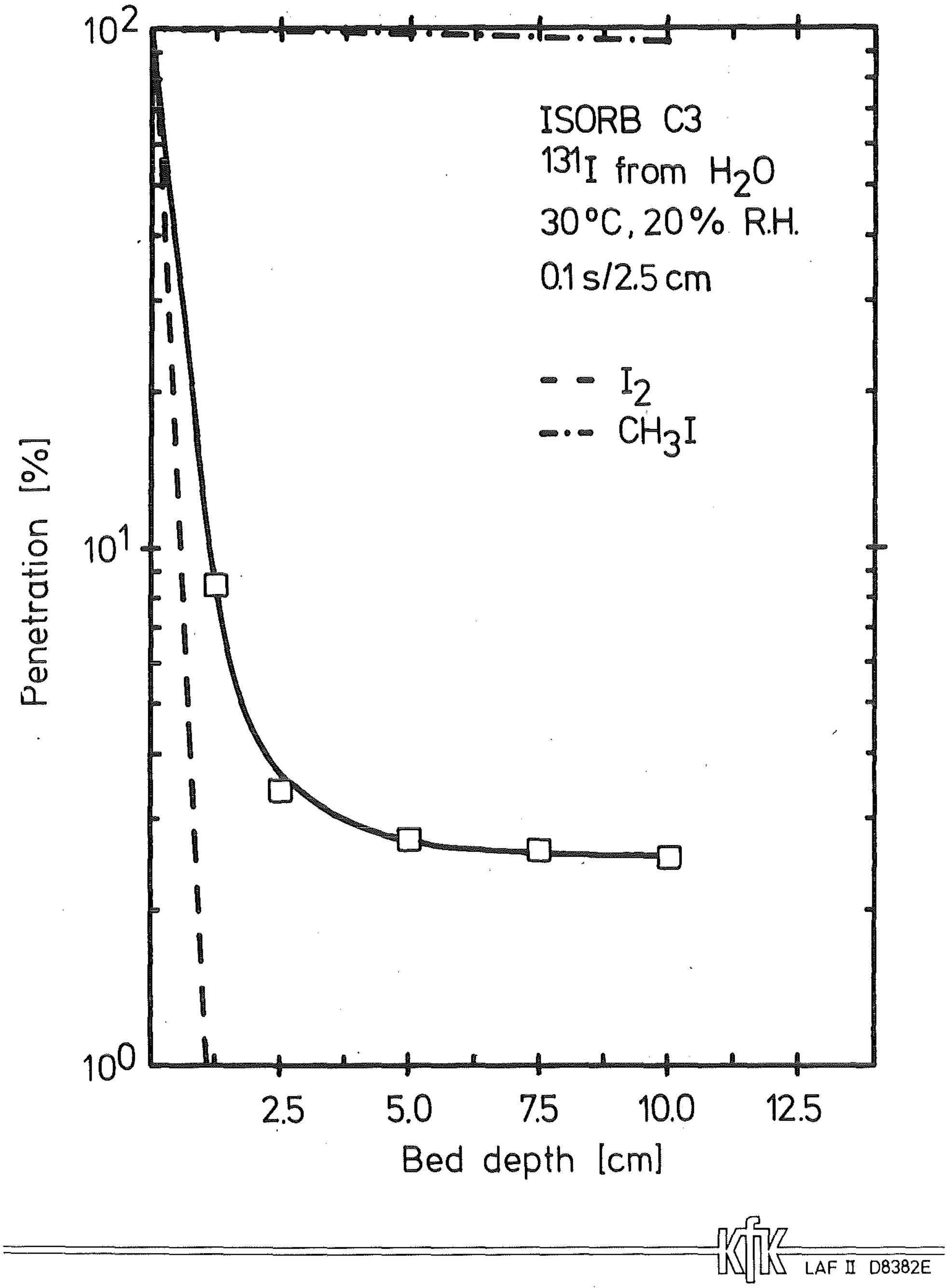
Penetration as function of bed depth

Fig. 14



Penetration as function of bed depth

Fig. 15



Penetration as function of bed depth

Fig. 16

5. Summary

Investigations were performed on the retention of ^{131}I that was volatilized from alkaline aqueous solutions by various activated carbons and sorbents used in iodine species samplers. By far the largest proportion of the ^{131}I was in a form whose retention was lower than that of elemental iodine (I_2), but higher than that of methyl iodide (CH_3I). This species is called "HOI" although no direct identification exists. A very small proportion was in a form whose retention was lower than that of both I_2 and CH_3I.

With all the fresh impregnated activated carbons investigated, the retention of "HOI" was higher than 99 % at a residence time of 0.1 s. The influence of purging was insignificant.

From these results it is concluded that "HOI" should be no problem for iodine filtration even if this species occurred in substantial proportions in the air to be filtered.

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