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TRAPPING OF KRYPTON IN URANIUM CARBIDE SURFACES

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by

O. GAUTSCH and C. MUSTACCHI

1966



Joint Nuclear Research Center Ispra Establishment - Italy

Physical Chemistry

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European Atomic Energy Community — EURATOM Joint Nuclear Research Center — Ispra Establishment (Italy) Physical Chemistry Brussels, March 1966 — 22 Pages — 4 Figures — FB 40

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SUMMARY

The trapping of Kr in UC surfaces during neutron irradiation was investigated and about 10^{13} atoms of Kr were found embedded per cm² x atm. of Kr after 5 x 10^{18} thermal nvt.

Trapping of Kr was also observed in CaF_2 and Au which were thermally evaporated and recondensed in presence of Kr. The Kr concentration in the recondensed layers was 2.10^{-4} and 3.10^{-5} atoms of Kr per atom of solid and unit Kr pressure, for CaF_2 and Au respectively.

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Introduction (°)

Scope of these experiments was an estimate of the surface trapping of inert gases in UC powders during irradiation. Such a trapping was reported to be of possible importance in attenuating the build-up of fission gas pressure in fuel elements (1). Two types of experiments were made to obtain information on the amount of trapping and- if possible - on the trapping mechanism:

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- a) Fused, loosely packed UC powders of different grain sizes, with an apparent density of about 50 % T.D., were irradiated in quartz vials in presence of known quantities of Kr + Kr⁸⁵. Five samples of the same type (2 with UC-Kr, 3 with UC-Xe) were exposed only to gamma radiation. This was made to account for a possible gammainduced attachment of gas to the UC during decay. Two samples of UC were irradiated without Kr + Kr⁸⁵ being present, to obtain the amount of fission Kr⁸⁵.
- b) CaF₂ and Au were evaporated and recondensed on glass surfaces in presence of known quantities of Kr + Kr⁸⁵, to assess the Kr concentrations in the deposits. Gettering effects could be a possible mechanism for inert gas trapping on the surface of gas-filled fuel element pores, where solid matter is vaporized and recondensed due to knock-out (2) and sputtering.

(°) Manuscript received on January 12, 1966

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1. Irradiations

1.1. Portions of about 1 g of UC powders (13.3 g/om³, 6.6 g/om^{3±}3 % when loosely packed) were irradiated below 300°C. In one vial ZrO instead of UC was exposed to neutrons, to account for a possible trapping in absence of fission. Data about the irradiation capsules are shown in Table 1.1.1. After irradiation the powders were dissolved, Kr separated from other gases liberated during the dissolution, and condensed into a quartz vial which was eventually sealed and submitted to gamma counting of Kr⁸⁵. Details on the preparation of the irradiation and counting vials are described in Appendix A.

From the activity rate and the weight of the UC powder the specific activity (ops/g) corresponding to the total Kr^{85} released from each sample was obtained and normalized to the flux and irradiation time of KCPR 8 (of. Table 1.1.1.). Thus the specific activity of KCPR 6 through KCPR 18 was proportional to Kr^{85} from in-pile trapping, fission, and gamma-induced attachment during the cooling time.

The specific activity of KCPR 8 and 9 due to fission Kr⁸⁵ only, was subtracted from the above values to yield the specific activity of trapped Kr⁸⁵. This result was divided by the Kr pressure, which existed in the irradiation vials at ambient temperature, to obtain the specific activity per unit Kr pressure. From this last the specific activity of KCPR 6 f and 15 f (again normalized to unit Kr pressure) proportional to the "gamma" attached Kr⁸⁵ during the cooling time was eventually subtracted to give the specific activity per unit Kr pressure of the in-pile trapped Kr⁸⁵ as ops/g atm. Results are shown in Table 1.1.2. The gamma activity rates of the Kr⁸⁵ photopeaks were transformed to atoms by means of a Kr⁸⁵ reference source of the same geometry as the counting samples, containing 846 CPS = 3.5.10¹⁴ ± 10 % atoms of Kr⁸⁵:

(1) $\frac{\text{Kr atoms trapped}}{\text{g atm}} = \frac{\text{ops}}{\text{g atm}} \cdot \frac{3.5 \cdot 10^{14}}{846} \cdot \frac{100}{\text{\% Kr}^{85}}$

The UC-Xe samples were processed in a similar way, without separating the Xe from other off-gases. The quantity of Xe was determined by mass-spectrography^X.

The quantitative determination of Xe by Dr. S. Facohetti, Analytical Chemistry Service, CCR Ispra, is gratefully acknowledged.

<u>Table1.1.1.</u>

Sample	mean grain size of UC, cm.10 ⁴	Kr in vial, om ³ NTP	% Kr ⁸⁵	Kr pressure in vial at room temp., atm.	Irradiation time, hours	Thermal neutron flux, n/cm ² sec.
KCPR 6	175 (100-250)	1.36	4.9	1.6	275	6.4 · 10 ¹²
KCPR 13	108 (90-125)	5 .2	2.9	4.1	269	$5.5 \cdot 10^{12}$
KCPR 14	108 (90-125)	5 .2	2.9	4.1	269	5.7 · 10 ¹²
KCPR 17	11 (<44)	5.2	2.9	4.7	269	$5.9 \cdot 10^{12}$
KCPR 18	11 (<44)	5.2	2.9	5.1	280	$6.7 \cdot 10^{12}$
KCPR 19	5 (ZrC)	5.2	2.9	5.1 (ZrC)	280	6.7 • 10 ¹²
KCPR 8	175 (100–250)	-	-	-	275	5.9 • 10 ¹²
KCPR 9	175 (100–250)	_	-	-	275	5.9 · 10 ¹²
KCPR 6 f	175 (100–250)	1.36	4•9	1.6	-	(gamma)
KCPR 15f	108 (90-125)	5.2	2.9	4•7	-	(gamma)
XCPR 1	108 (90-125)	-	-	3.2 (Xe)	-	(gamma)
XCPR 2	108 (90-125)	-	-	3.2 (Xe)	-	(gamma)
XCPR 3	108 (90-125)	-	· -	3.2 (Xə)	-	(gamma)

UC: 4.8 ± 0.2 % C, 1500 ppm 0₂, 1000 ppm N₂, 20 ppm H₂ (the KCPR 17 and 18 powder had 5700 ppm 0₂, and X-ray analysis showed weak oxide patterns) ZrC: 88.3 % Zr, 0.25 % 02, 11.5 % total C, 0.4 % free C.

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Sample	Kr ⁸⁵ acti observed,	vity,ops/g normalized to KCPR 8	<u>Kr atoms</u> g atm.	Specific surface cm ² /g	<u>Kr atoms</u> om ² atm.	Recoil fragments/om ²	Kr atoms trapped per recoil fragment and atm.
KCPR 6	3460	31 90	1.5.10 ¹⁶	320	4.7.10 ¹³	2.8.10 ¹⁴	0.17
KCPR 13	11 36	1 350	3.0.10 ¹⁵	540	5.6.10 ¹²	2.8.10 ¹⁴	0.02
KCPR 14	2895	3070	9.0.10 ¹⁵	540	1.7.10 ¹³	2.8.10 ¹⁴	0.06
KCPR 17	4350	4390	1.2.10 ¹⁶	1460	8 .2 •10 ¹²	2.0.10 ¹⁴	0.04
KCPR 18	4355	3750	8.4.10 ¹⁵	1460	5.8.10 ¹²	2.0.10 ¹⁴	0.03
KCPR 19	110	94	3.0.10 ¹⁴	-		_	_
KCPR 8	219	219	-	-	-	-	-
KCPR 9	204	199	-	-	-	_	_
KCPR 6 f	206	206	1.1.10 ¹⁵	320	3.2.10 ¹²	_	_
KCPR 15f	336	336	1.0.10 ¹⁵	540	1.9.10 ¹²	-	-
XCPR 1	-	-	3. 8 . 10 ¹³	540	0.7.10 ¹¹	_	_
XCPR 2	-	-	2.4·10 ¹⁴	540	4.4.10 ¹¹	-	_
XCPR 3	-	-	6.9.10 ¹³	540	1.3.10 ¹¹	_	
Reference	Source 846	CPS -	-		-	-	-

Note: For KCPR 17 and 18 no separate sample was exposed to gammas. The average value of 6 f and 15 f was used to correct for the attached Kr.

Dividing the results from equation (1) by the measured specific surface (B.E.T.) of the unirradiated powders, the number of Kr atoms trapped or attached per om² and atm was estimated, assuming the specific surface did not change during irradiation. To obtain the number of Kr atoms trapped per fission fragment escaped and atm (i.e. recoil fragment emerging from the powder grains) the number of the fragments emitted per unit surface of the powder was calculated as shown in Appendix B. All experimental errors pertaining to the measurements are also listed. For the smallest powder ($11 \cdot 10^{-4}$ om grain size) with experimentally determined particle diameter, the grains were assumed spherical (of Appendix B).

1.2. It can be seen from Table 1.1.2. that neutron irradiated ZrC(3) trapped about 10 times less Kr atoms/g atm. than UC, in spite of its larger specific surface. The in-pile trapping observed in UC must thus be caused by fission fragments and/or beta and gamma radiation from fission products. The average value for in-pile trapping amounts to $1.7 \cdot 10^{13}$ Kr atoms/cm² atm., (within a factor of about 3). Trapping per unit surface does not appear to be correlated to the powder grain size. This implies that the depth (or distribution of depth) of burying is not a function of grain or pore size.

An average value of $2.5 \cdot 10^{12} \pm 55 \%$ Kr atoms/cm² atm was observed for the gamma exposed samples 6 f and 15 f. Their difference in grain size was a factor of about 2, leading again to the belief that attachment of gas per unit surface does not depend on grain size. A lower attachment rate was found for UC-Xe. The average value is $2.1 \cdot 10^{11}$ Xe atoms/cm² atm., within a factor of about 2. The only difference between

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the UC-Kr and UC-Xe samples consisted in the absence of the Kr^{85} beta radiation in the later.

The average number of Kr atoms trapped per recoil fragment and atm was 0.06 (within a factor of 3) independent of grain size, which means that about 17 fission fragments escaping are required to trap one Kr atom/atm.

In analyzing the above results, the thermal release and recoil of Kr⁸⁵ (or its precursors) from the UC powders during the low temperature irradiation were neglected. Normalization to unit Kr pressure was performed, assuming linearity with pressure (4) whatever the trapping mechanism is.

2. Thermal evaporations

2.1. To obtain an order of magnitude for the trapping of Kr and its concentration in recondensed solids, CaF_2 and Au were used as getterers in absence of any effects related to fission. The only radiation present was that of Kr⁸⁵. Details on the experimental procedure are given in Appendix C and the results for Au-Kr are shown in Table 2.1.1.

Table 2.1.1.

Sample N ^O	1/1	1/2	2/1	2/2	2/3
Atoms of Au condensed per cm ²	2.5.10 ¹⁷	1.9·10 ¹⁷	3.2·10 ¹⁷	3.2.10 ¹⁷	3.9.10 ¹⁷
Atoms of Kr found per cm and atm	6•10 ¹² ±85%	2•10 ¹³ ±41%	1•10 ¹³ ±87%	0	0
Atoms Kr/atom Au	2•10 ⁻⁵ ±85%	10•10 ⁻⁵ ±41%	3•10 ⁻⁵ ±87%	0	0

The average values are $7 \cdot 10^{12}$ Kr atoms/om² atm and $3 \cdot 10^{-5}$ Kr atoms/atom of solid and atm, within a factor of about 3. These results must be regarded with caution, not only because of the large experimental errors involved, but also because of the extrapolation of the trapped quantities and Kr concentrations from the experimental Kr pressures of about $1.3 \cdot 10^{-3}$ atm to 1 atm. Possibly the structure of the deposits is pressure dependent. It should be noted that the large amounts of Kr trapped per om² and atm are not to be directly compared to the amounts trapped during irradiation. The former Kr quantities have a meaning only insofar as to yield values of Kr concentrations. The total amounts of gettered Kr will of course depend linearly on the thickness of the condensed layers.

CaF₂ is reported to recondense as a crystallized, porous layer (5) 2.2. being thus particularly suitable as a gettering medium. The experimental arrangement is shown in Fig. 2.2.1. About 10 g of CaF, powder were introduced into a quartz vial of 22.5 cm³, the system degassed to $6 \cdot 10^{-3}$ mm Hg and the Kr container (4.6 cm³ NTP of Kr, 3.0 % Kr⁸⁵, correction factor for decay 0.86) opened by HF induction heating. Then the gas was displaced into the quartz vial by rising the meroury level and sealed. The Kr pressure in the vial was 0.17 atm \pm 10 % at ambient temperature. The CaF, was kept at 1200°C for 1.2.10⁵ sec. The temperature of the "cold" wall, where CaF, recondensed, was about 200°C, so that thermal release of some Kr could have occurred during the experiment. After evaporation the "oold" wall was out from the vial and exposed to a vacuum of 10⁻³ mm Hg for an hour. X-ray analysis of the deposits showed only CaF, patterns. By absolute counting of the Kr⁸⁵ gamma radiation 2.6.10¹⁴ Kr atoms/om² atm \pm 66 % were found embedded. From chemical analysis

 $3 \cdot 10^{-4}$ g of Ca were obtained, corresponding to $1 \cdot 1 \cdot 10^{18}$ atoms of Ca + F/cm². The resulting Kr concentration was $2 \cdot 10^{-4}$ atoms Kr/atom of the solid and atm ± 66 %. Assuming a density of 3 g/cm³ for the recondensed layer, its thickness results to be $2 \cdot 10^{-5}$ cm.

3. Interpretation of data

3.1. It follows from Table 3.1.1./a that in-pile trapping of Kr in UC is caused by fission. The only events which may have a bearing on trapping must therefore be fission gammas or the emergence of fission fragments. The data obtained by irradiating ZrC disprove any major contribution of the fission-gamma field of the reactor. Therefore, fission fragments must be the cause of trapping. These fragments may:

- a) knock out matter (2), which on recondensing getters the gases (as in the CaF₂ experiment);
- b) leave open craters in the surface which, filled with gas, might become closed by globules (2) knocked out from the opposite wall;
- c) pass through the gas, cause ionization and dissipate part of their energy in elastic collisions with the gas atoms (1). Energetic gas atoms, capable of penetrating into the solid and sticking could be created thus;
- d) impinge on the pore surface with sufficient energy to cause significant sputtering, especially at high temperatures.

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Table 3.1.1. <u>a</u>

Sample	Type of reactor neutrons	radiatio gamma	on involu fission beta	reactor gamma	85 Kr gamma	Kr ⁸⁵ beta	Atoms Kr om ² atm	Atoms Kr Atom of solid atm
UC-Kr	+	+	+	+	+	+	1.7.1013	· · · · · · · · · · · ·
UC-Kr	-	+	-	-	+	+	2.6.10	
ZrC-Kr	+	-	- ,	+	+	+	< 10 ^{1,1}	
UC-Xe	-	+	-	-	+	-	2.1.10	· _
Au-Kr	-	_	-	-	+	+	-	3.10-5
CaF ₂ -Kr	-	-	-	-	+	+	_	2.10-4
					<u>b</u>	•		
mean pore size, cm	Surface the por	of 2 e,cm	Atoms o present	f Kr /pore atm [#]	Atoms of trapped	of Kr 1/pore at	Fraction	Recoil fragments required
2.10-4	1.2.10	-7	1.0.1	0 ⁸	2.1	o ⁶	2	3•107
11.10-4	3.6.10	-6	1.7.1	0 ¹⁰	6.1	7	0.4	1•10 ⁹
108.10-4	3.5•10	,-4	1.6.1	013	6.1	0 ⁹	0.04	1•10 ¹¹
175-10-4	9,2.10	-4	6.7•1	0 ¹³	1.6.1	0 ¹⁰	0.02	2.6.1011
2009-10-4	1.2.10	-1	1.0.1	0 ¹⁷	2•1	0 ¹²	0.002	3·10 ¹³

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*f (r³) **f (r²) Whatever the trapping mechanism is, it cannot be important in limiting the pressure build-up due to the thermal release of fission gases, because the overall amounts of gas trapped are small and decrease with increasing pore size as shown in Table 3.1.1./b. The values in Table 3.1.1/b were calculated assuming spherical pores and using the ideal gas law. The temperature of the gas was taken as 300° K, the atoms trapped as $1.7 \cdot 10^{13}/\text{cm}^2$ atm and 0.06/reooil fragment and atm (both have a factor of 3 as error). If the amount of gas present in a pore is a function of time, then of course the fraction trapped will be time-dependent.

3.2. To explain the trapping mechanism in terms of gettering of the CaF, type, let us assume a recondensed layer of knocked-out UC with a Kr concentration of $2 \cdot 10^{-4} \pm 66 \%$ Kr atoms/atom of solid and atm. Due to re-ejection such a layer reaches an equilibrium thickness of about 10^{-7} cm (2), corresponding to about 5.10¹⁵ atoms of solid/cm² (2) and hence to 10¹² Kr atoms/cm² atm. This value is too low to account for in-pile trapping. If 10³ atoms of the solid are knocked-out per recoil fragment (2) and associated with 0.06 Kr atoms trapped/recoil fragment and atm, the resulting Kr concentration should be 10⁻⁴ Kr atoms/atom of solid and atm, similar to that in CaFo. From these results one can deduce that in-pile trapping is not a mechanical burying of the CaF, type. The existence of a recondensed layer, observed on metallic collectors (2) is true for a gas-filled fuel element pore as well. In such a layer consisting of $5 \cdot 10^{15}$ atoms U+C/om², the Kr concentration should be:

$$\frac{1.7 \cdot 10^{13} \text{ Kr atoms trapped/cm}^2 \text{ atm}}{5 \cdot 10^{15} \text{ atoms of solid/cm}^2} = 3 \cdot 10^{-3} \frac{\text{Kr atom}}{\text{atom of solid and atm}}$$

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at saturation. This concentration is much higher than that of CaF_2 , where no re-ejection took place during trapping, and therefore improbable.

Another possibility of mechanical burying is offered by oraters, left in the surface of a gas-filled fuel element pore by escaping fission fragments. (Also in this case linearity of the trapping effect with the gas pressure will be assumed). Rogers et al. (2) showed that one globule of about $7 \cdot 10^{-7}$ cm in diameter is knocked out per every 16 recoil fragments. Suppose that the craters are invaded by gas atoms and then closed by a globule, arriving from the opposite wall. With $1.5 \cdot 10^{11}$ globules knocked-out per cm² at saturation (2), assume that the same number of gas-filled craters corresponding to a volume of $2.8 \cdot 10^{-8}$ cm³/cm² are closed hermetically at reactor shut-down. With 1 atm of Kr at 300° K the number of trapped Kr atoms, estimated from the perfect gas law, is roughly $6 \cdot 10^{11}$ atoms/cm² atm. This is about 10 times less than the lowest observed in-pile trapping.

3.3. The above types of mechanical burying being excluded as a possible trapping mechanism, the elastic collision theory (1) must be considered. According to this energetic gas atoms are generated by collisions with fission fragments and may penetrate into the solid, especially in small pores and at low gas pressures (4), where the energy losses of a gas atom due to multiple collisions between gas atoms are small. At higher gas pressures trapping would reach saturation and there would be no limiting of the fission gas pressure build-up.

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Conolusion

Gettering mechanisms of the previously described types do not account for the observed in-pile trapping. The experimental results are believed to be saturation values, because knock-out and thermal release of in-pile trapped gas during irradiation counterbalance trapping proportionally to the gas concentration in the surface. Whatever the trapping mechanism is, there will be saturation and no important attenuation of the fission gas pressure.

Appendix A

The powders and a Co flux monitor were introduced into quartz vials of known internal volume. All filling operations were carried out in Ar atmosphere with less than 250 ppm of H_2O by weight. The filled vials were attached to a Kr-filling assembly (of Fig. 1) which was first pumped down to about $2 \cdot 10^{-2}$ mm Hg. Then the Kr + Kr⁸⁵ container was punctured and the Kr slowly compressed into the cooled vial (liquid nitrogen) by the Hg, after which the vial was sealed.

After irradiation the UC powder was put into a dissolving assembly composed (Fig. 2) of a dissolver (A), a trapp for particles and condensible matter (B) (in which practically no delay of Kr was observed), a Zr-Ti furnace (C), and a charcoal trapp (D). While evacuating, the Zr-Ti and the charcoal were brought to 1150° C and 400° C resp. and degassed 2-3 hours. Then, at a vacuum of appr. $5 \cdot 10^{-2}$ mm Hg, the temperature of the Zr-Ti furnace (where the other gases set free by the UC dissolution, i.e. 90 ml CH_{4/g} UC, 8 ml H_{2/g}, and decomposition products of HNO₃ are absorbed) was lowered to 1000° C, that of the charcoal to ambient temperature. After this the vacuum line was disconnected, liquid nitrogen put around the glass wool trap (B) and 5 ml of 3 N HNO₃ added to the UC in the dissolver. The HNO₃ was heated to 80° C within 20 minutes and kept at this temperature for another 20 minutes, during which the gases were intermittently passed from the dissolver to the Zr-Ti furnace. Thereafter the dissolver was cooled to 50° C within 30 minutes (the gas always passing stepwise from the dissolver to the Zr-Ti furnace) turned off and the temperature of the Zr-Ti furnace lowered to 400° C within 30 minutes. Then the charcoal trap (D) (quartz vial cooled with liquid nitrogen) was opened and the Kr left to recondense for 45 minutes the vacuum gauge indicating 10^{-1} mm Hg at the end of the procedure when the vial was sealed.

Appendix B

The number of fission fragments escaped per unit surface from the UC powders is given by:

(1)
$$\mathbf{F} = 2 \mathbf{p} \sum_{\mathbf{f}} \mathbf{\phi} \mathbf{t} 4 \mathbf{T} \mathbf{r}^2 \mathbf{R} / 4 \mathbf{T} \mathbf{r}^2$$

= $2 \mathbf{p} \sum_{\mathbf{f}} \mathbf{\phi} \mathbf{t} \mathbf{R} = \frac{\mathbf{fragments escaped}}{\mathbf{cm}^2}$

for $r \gg R$. The escape probability p

for this condition is

$$p = \frac{1}{R} \int_{0}^{R} \frac{R-x}{2R} dx = 0.25$$

$$\sum_{f} = 0.136 \text{ om}^{-1}, \text{ macroscopic fission cross section}$$

$$R = 7 \cdot 10^{-4} \text{ om, mean range of fission fragments in UC}$$

$$\Phi = 5.9 \cdot 10^{12} \text{ n/om}^{2} \text{ sec, neutron flux}$$

$$t = 9.9 \cdot 10^{5} \text{ seo, irradiation time}$$

$$r = 54 \cdot 10^{-4} \text{ om, } 87.5 \cdot 10^{-4} \text{ om}$$

$$F = 2.8 \cdot 10^{14} \text{ recoil fragments/om}^{2}$$
For the powder of $11 \cdot 10^{-4}$ om grain size
$$(2) F = \frac{2p \sum_{f} \Phi t 4 \pi r^{3}}{4 \pi r^{2} 3} = \frac{2}{3} p \sum_{f} \Phi t r$$

With
$$r = 5.5 \cdot 10^{-4}$$
 cm = 0.8 R, $p = 0.687$
and $F = 2.0 \cdot 10^{14}$ recoil fragments/cm².

The error of the single measurements consists of:

- # 1 % on irradiation time
- ± 1% on counting
- ± 2 % Kr volume
- ± 5% volume of quartz vial

± 7 % Kr pressure

- ± 7 % neutron flux
- ± 10 % percentage of Kr⁸⁵
- ± 10 % Kr⁸⁵ reference source
- ± 20 % measured specific surface

atoms trapped g atm	•	±	52 %
atoms trapped om ² atm	I	±	72 %
recoil fragments om ²	1	±	8 %
atoms trapped	1	±	80 %

Appendix C

In the first experiment (samples 1/1 and 1/2) 0.24 g of Au were evaporated on 314 cm^2 . The experimental arrangement (shown in Fig. 3) was degassed to $4 \cdot 10^{-4}$ mm Hg and the Kr container punctured. A Kr quantity corresponding to 1.2 mm Hg ($4.76 \ \text{Kr}^{85}$) was admitted to the glass sphere, the temperature of which was kept below 70° C. After evaporation small splinters were broken from the sphere, their surface measured, and an absolute counting of the Kr⁸⁵ beta activity made, to obtain the total Kr atoms trapped. The quantity of Au was determined by activation analysis. The same type of experiment was repeated with 0.8 mm Hg Kr pressure and $4.86 \ \text{Kr}$ Kr 85 (samples 2/1, 2/2, 2/3).

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Fig. 3

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To disseminate knowledge is to disseminate prosperity — I mean general prosperity and not individual riches — and with prosperity disappears the greater part of the evil which is our heritage from darker times.

Alfred Nobel

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