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DESORPTION OF WATER BY CHARCOAL

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by

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June, 1963

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DESORPTION OF WATER FROM CHARCOAL

by

H. Tjerkstra

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ABSTRACT

Regeneration facilities for the charcoal in the main fission product delay units have to be provided because of the possibility that the water content might rise above an acceptable level and thereby shorten the delay time of the fission gases Xenon and Krypton. Therefore, the influence of different parameters on the desorption of water from charcoal has been investigated. As a result the regeneration time for the Dragon delay units can be calculated.

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DESORPTION OF WATER FROM CHARCOAL

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1. INTRODUCTION

The adsorption of water on charcoal has the effect of diminishing its adsorption capacity for other adsorbates. Because of the possibility that the water content in the main fission product delay beds might rise above an acceptable level and thereby shorten the delay times of the fission gases Xenon and Krypton, regeneration facilities for the charcoal have to be provided. The regeneration time and efficiency will largely depend on the temperature and, if a continuous gas purge is used, on the gas flow rate and the initial moisture content. At present almost complete H_2O desorption is necessary, and this would require a temperature above 180°C which might be technically inconvenient. In order to determine suitable regeneration conditions, the mechanism of the desorption of water from charcoal has been investigated.

Described below are the characteristics of the main system which had to be closely simulated during the experiments. The main fission product delay units consist of a series of inter-connected charcoal-filled U-tubes of progressively increasing diameter. The gas entering is divided into eight parallel channels, each consisting of five vertical U-tubes 2 in diameter, connected in series. To prevent overheating of the charcoal near the entry, the effective diameter of the first vertical leg of the first U-tube in each of the eight paths was reduced by the insertion of a solid central core $1\frac{3}{8}$ in diameter, leaving the charcoal packed in an annulus 3/16 in wide. On leaving the 2 in bore tubes, the eight gas streams are re-combined into two parallel streams each of which passes next through an 8 in bore U-tube and finally through a 15 in bore U-tube. The two parallel streams leaving the 15 in U-tubes are then combined into a single stream which leaves the delay unit via the outlet branch. The quantity of charcoal per delay bed is 1566 kg, of which 60% is in the 15 in bore U-tubes. For regeneration, helium will be used as purge gas at a maximum flow rate per bed of 5 g/sec at 20 atm. The maximum water content of the charcoal before regeneration is expected to be 6% and the maximum tolerable water content after regeneration 0.5%.

2. EXPERIMENTAL METHOD

Simulating the relevant characteristics of the main delay units the experiments were carried out under the following conditions:-

(a) 16.8 g, 8-14 mesh coconut charcoal type 208 C - supplied by Sutcliffe, Speakman & Co. Ltd., - was used as a sample.

(b) Argon with a water content < 4 vpm was used as the drying gas. Similar behaviour with respect to the desorption mechanism is expected from helium, which will be used as the purge gas in the main delay beds.

(c) The experiments were carried out at atmospheric pressure as it is considered that the influence of the pressure of the carrier gas is negligible.

(d) The influence of temperature was investigated at 60, 90 and 120°C at a flow rate of 2.5×10^{-1} (cc/g . sec).

(e) The influence of the flow rate was also investigated at 60 and 120°C in order to extrapolate the results obtained to the conditions of the main delay beds with a flow rate of 1.3×10^{-3} (cc/g . sec).

It is possible to make allowances for the slightly different heat transfer in the main delay units and in the experiments.

2.1 Experimental Procedure

The charcoal sample was placed in a container (Fig. 1.1) and was regenerated first for at least 15 hours at 200°C and a pressure of 25×10^{-3} mm Hg. A longer regeneration showed no improvement. The charcoal thus prepared was regarded as being free from water. The sample was then saturated with dry argon (water content < 4 vpm) at the temperature at which the different experiments were to be carried out and then weighed. The water content of charcoal is always related to this weight. The saturation with water up to 4-7% by weight was carried out by passing over the charcoal air, previously equilibrated with water at room temperature. For investigation of the desorption mechanism, the bulb containing the charcoal was placed in a thermostatically controlled oil bath and dry argon was passed through it at a constant rate. The argon first flowed through a small glass coil immersed in the same oil bath in order that it should be at the same temperature as the charcoal. The degree of regeneration was checked by the weight loss of the charcoal. When the water content of the argon at the outlet of the sample fell below 500 vpm an electrolytic water analyser was connected which enabled the water desorption to be calculated from the flow rate and the difference of the water content at the inlet and outlet. Since an electrolytic water analyser requires a minimum flow rate of 100 cc/min this instrument can only be used for experiments with a flow rate above this value. At lower flow rates, therefore, the degree of regeneration could be checked only by the weight loss of the charcoal.

Fig. 1.2 shows the experimental set-up. The dry gas (high purity argon total impurity content < 50 vpm) is passed first through a 5Å molecular sieve column. An "Elliott" moisture meter was used to indicate the water content of the gas emerging from the column, which in none of our experiments exceeded 3 vpm. The flow rate was controlled by a flow meter in front of the charcoal sample. For weight measurements the bulb could easily be disconnected from the other components. The first weighing of the sample was normally carried out after one hour of regeneration and the time interval between further weighings was increased in relation to the decrease of the water concentration of the charcoal. In the cases where an electrolytic water analyser was used, this instrument was connected at the outlet from the charcoal sample. When the moisture meter indicated a constant water vapour concentration for more than 3 hours, or no further weight loss could be detected after at least 8 hours, the experiment was concluded. A semi-automatic analytical balance with a sensitivity of .5 mg (0.001 g per scale division) was used for the weighings.

2.2 Experimental Results

Fig. 2 shows the relation between desorption, regeneration time, and temperature, at a constant flow rate of 2.5×10^{-1} (cc/g . sec). The water content of the charcoal was not the same at the start of the different experiments and, therefore, the regeneration curves have been superimposed according to their original water content.

Starting at a water concentration of about 7% the desorption can be considered as linear with respect to time down to a water content of roughly 0.8% at 60°C, 0.5% at 90°C, and 0.3% at 120°C. Therefore, at a given flow rate and temperature, the desorption rate can be considered as constant in this range. Below these concentrations the desorption mechanism seems to change completely and the desorption rate decreases rather suddenly by a factor of 10^{-4}. The humidity of the charcoal decreases so slowly in this nearly horizontal part of the desorption curve that a change in the water content of the charcoal can hardly be detected.

The following Table shows the desorption rate in the linear range and in the flat part of the desorption curve and the final water concentration achieved at the corresponding temperature at a flow rate of 2.5×10^{-1} (cc/g . sec).

TABLE 1

Temperature (°C)	Desorption Rate in the Linear Range	Desorption Rate in the Flat Part	Final Water Content
	g/g . sec	g/g . sec	% by weight
60	1.4×10^{-5}	2.8×10^{-9}	.32
90	2.46×10^{-5}	1.4×10^{-9}	.15
120	3.4×10^{-5}	3.6×10^{-10}	.03

The influence of the flow rate on desorption at 60 and 120°C is shown in Fig. 3 and Fig. 4.1. The regeneration curves are knee-shaped and the two parts depend in different ways on the flow rate and temperature.

(a) In the steep part of the regeneration curve the desorption velocity is apparently affected by changes in flow rate and, at 60°C, almost proportional to it.

(b) In the flat part of the regeneration curve the influence of the flow rate is negligible. In this region the water concentration is determined by the temperature only.

TABLE 2

Flow Rate		Desorption at 60°C g/g . sec	Desorption Rate at 120°C g/g . sec
cm/mm	cc/g . sec		
260	2.5×10^{-1}	1.4×10^{-5}	3.4×10^{-5}
78	7.7×10^{-2}	0.4×10^{-5}	-
50	4.9×10^{-2}	0.3×10^{-5}	1.28×10^{-5}
25	2.4×10^{-2}	0.16×10^{-5}	9.5×10^{-6}

2.3 Experimental Accuracy

The charcoal was regenerated at 200°C and at a pressure of 25×10^{-3} mm Hg for at least 15 hours. No additional release of water was found during the next 24 hours and we assume, therefore, that the charcoal is free from water after the above treatment. According to other authors [1] there is no additional water release when the temperature is raised from 150°C to 300°C, and this agrees with the above assumption. Heating the charcoal up to 1000°C causes an additional weight loss, which probably results from desorption of other gases rather than from residual water. The effect of small amounts of residual water in the charcoal can be neglected.

Due to oil traces from the thermostat, and small fluctuations in the experimental conditions, the limit of experimental accuracy cannot be expected to be better than ± 2 mg. Using a charcoal sample of 16.8 g this error corresponds to an accuracy of roughly 10^{-4} g/g. The final water concentration, measured at 120°C, is nearly of the same order, therefore, an experimental error of 100% is possible in the final stages of the experiment. On the other hand the water content of the charcoal achieved at this stage is already so low that this accuracy is sufficient from a practical point of view. A higher accuracy at very low water concentration could be obtained only at the cost of using a completely different and more complicated experimental set-up.

Some experiments have been performed to trace the temperature change inside the charcoal sample during the experiment. The two graphs of Fig. 4.2 show the delay time in heating the charcoal sample up to 120°C starting at a water concentration of 4% (lower curve) or 0.5% (upper curve). The time lag in heating up the charcoal to 120°C is nearly twice as long at the higher initial water concentration as at the lower. Due to the high desorption rate, the time required to achieve a given temperature under the conditions of the experiments is increased. Assuming an isothermal desorption, the desorption curve would probably be somewhat steeper at higher water concentrations than found in the experiments.

3. DISCUSSION AND INTERPRETATION

3.1 Adsorption Equilibrium

The relevant adsorption and desorption characteristics can be seen from the adsorption isotherm. Fig. 5 shows two typical adsorption isotherms of water vapour on charcoal determined by A. S. Coolidge [2]. With the multi-molecular adsorption theory of Brunauer, Emmett and Teller and taking account of capillary condensation [3] it is possible to represent this type of adsorption isotherm, as follows:-

$$V = \frac{V_m\, C\, P/P_o}{1 - P/P_o} \; \frac{1 + (1/2\, ng - n)\, (P/P_o)^{n-1} - (ng - n + 1)^n + 1/2\, ng\, (P/P_o)^{n+1}}{1 + (C - 1)\, P/P_o + (1/2\, Cg - C)\, (P/P_o)^n - 1/2\, Cg\, (P/P_o)^{n+1}} \qquad (1)$$

where:

V = volume adsorbed

V_m = volume adsorbed when the entire adsorbent surface is covered with a complete unimolecular layer

n = maximum number of layers that can be adsorbed on the wall of the capillary

P/P_o = relative pressure, where P_o denotes the vapour pressure of water at the given temperature.

C = constant, given in good approximation by the following equation

$$C = e^{E_1 - E_L/RT} \qquad (2)$$

E_1 = heat of adsorption in the first layer

E_L = heat of liquefaction

R = Gas constant

T = Temperature ^{o}K

g = constant, which can be determined by the expression

$$g = e^{Q/RT}$$

Q = additional heat of liquefaction, which takes into account that the last adsorbed layer in a capillary is attracted on both sides

$$Q = 2 \, . \, \sigma \, . \, S$$

σ = surface tension

S = surface area covered by 1 mol of liquified gas spread into a unimolecular layer.

The characteristic "S" shape of the isotherm is due to:-

(1) the low value of C in the low pressure part of the curve

(2) the capillary condensation in the middle pressure range

(3) the limitation of the thickness of the adsorbed layers by the pore size in the saturated part of the curve.

With the following constants, equation (1) fits satisfactorily the shape of the isotherm on "pure" sugar charcoal illustrated in Fig. 5.

$V_m = 115 \text{ cm}^3$ S.T.P. $n = 3$

$C = 0.08$ $g = 28$

In the case of low pressures $P/P_o \ll 1$, equation (1) contracts to

$$V = \frac{V_m \, C \, P/P_o}{(1 - P/P_o)\,(1 + (C - 1)\,P/P_o)} \qquad (3)$$

The water isotherm is clearly convex toward the pressure axis at low pressures, which means that according to equations (2) and (3) $E_1 < E_L$. This has been proved experimentally by A. S. Coolidge.

From the above it can be concluded that charcoal is a hydrophobic substance and the removal of absorbed water from charcoal should be comparatively easy, but the results of the water desorption experiments seem to contradict the hydrophobic nature of the charcoal. This might be explained as a result of the presence of impurities in the charcoal. According to the information of the manufacturer the ash content of the coconut charcoal 208 C, used in the experiment is 2-3% and has the following composition:-

	% on ash basis
Silica (SiO_2)	17
Iron oxide (Fe_2O_3)	8
Alumina (Al_2O_3)	7
Lime (CaO)	3
Magnesium Oxide (MgO)	4
Phosphates (P_2O_5)	2

	% on ash basis
Carbonates (CO_2)	10
Sulphates (SO_3)	2
Alkalies by difference	47

The experiments of A. S. Coolidge prove that small amounts of hydrophilic impurities probably siliceous have an influence on the adsorption of water on charcoal at low concentrations. Fig. 6 shows two adsorption isotherms of water on the same type of coconut charcoal at low relative pressures. Curve A-A represents the isotherm of untreated charcoal with an ash content of 1.7%, curve B-B the isotherm of the charcoal with an ash content of 0.4%, treated with hydrofluoric acid for several days extracting most of the impurities. The ultimate capacity of the charcoal was not affected by the treatment but the amount adsorbed at relative pressures below 0.3 became much less. The hydrophobic character of charcoal is, therefore, more pronounced on curve B-B.

The phenomenon of hysteresis associated with the adsorption of water on charcoal found in other experiments [4] might be explained as an effect of impurities. The amount adsorbed on the impurities is sometimes so large that blocking of the narrow pores could occur.

The difference curve (Fig. 6 dotted line) between the isotherm on untreated charcoal and extracted charcoal should give the adsorption isotherm of water vapour on the impurities in charcoal. (Because of the limited accuracy of the data at very low pressures the isotherm constants were calculated from data at intermediate pressures up to a relative pressure of 0.3.) This adsorption isotherm can be represented by equation (3), where

$$V_m = 66 \text{ mg/g charcoal and } C = 5.3$$

If one relates, tentatively, V_m to 1 g of impurities, then $V_m = 3300$ mg per g of impurities which is an unusually high value. However, it does not seem possible to explain the extra adsorption by using values of V_m and C of different order of magnitude, such as is found in literature for silicates and metal oxides, (i.e. $V_m = 80$ mg/g and $C \gtrapprox 100$). The adsorbed amounts calculated by using the latter values turn out to be too small at least by a factor of 10. It could be that the high number of adsorption sites, (proportional to V_m) found in the present case is due to lattice defects at the interface between charcoal and finely dispersed impurities. This assumption would also explain why the value of the adsorption energy does not correspond with any single impurity.

3.2 Rate Behaviour

Assuming that the bend in the desorption curve occurs when the residual water is mainly adsorbed on the impurities, we may conclude from our experimental results that the adsorption isotherm of water on the charcoal used in our experiments differs from the one found by Coolidge. The influence of the impurities seems to be negligible at a water content above 0.8% in our case, whereas Coolidge found an influence up to 5%. Nevertheless, at a somewhat higher water content the agreement between our results and the isotherm given by Coolidge is reasonable (see numerical results) and, since no more accurate adsorption isotherm is available, further calculations have been based on the isotherm given by Coolidge.

3.2.1 Charcoal Model

The following model has been assumed for the desorption mechanisms.

A charcoal grain of linear dimensions L is penetrated by parallel pores of equal length L and equal diameter d. The number of pores N per grain is such, that the total internal surface equals the charcoal surface S found by experiment. The external surface of the grain can be neglected in comparison with the internal surface.

Coconut charcoal 8-14 mesh was used. The grains were assumed to be of cubical shape and to have a linear dimension of 1.6×10^{-1} cm. It was found that the average number, n, of grains per cm^3 is 135 and, under the above conditions one finds an intergranular voidage which agrees well with the voidage E found by other authors [5] $E = 0.45$.

The total pore volume of charcoal found in literature [6] is in the order of 0.5 cc/cc. A separation into macro and micropores shows that 47% of the pore volume represents macropores with an average pore diameter of 10^{-4} cm and the rest of the volume of micropores with a diameter of roughly 20 Å.

A rough order of magnitude calculation indicates that surface adsorption in the macropores is negligible compared with the adsorption in the micropores. The surface within the cylindrical pores is given by:

$$s = \frac{4V}{d}$$

where V is the volume of pore space and "d" is the pore diameter. Using the values for V given above, d = 20 Å for micropores and $d = 10^{-4}$ cm for macropores the surface of the micropores obtained is 5×10^6 cm^2/cm^3 and of the macropores 1×10^4 cm^2/cm^3.

In the following the calculations will be related only to the micropores.

The surface S is given by:

$$S = n \cdot N \cdot L \, \pi d \quad (cm^2 \cdot cm^{-3})$$

In the present case:

$S = 5 \times 10^6 \text{ cm}^2 . \text{cm}^{-3}$, $L = 1.6 \times 10^{-1}$ cm, $d = 2 \times 10^{17}$ cm,

$n = 135 \text{ cm}^{-3}$, $N = 3.68 \times 10^{11}$ pores/grain

3.2.2 Desorption at Low Water Content

It is assumed that in this region:-

(1) the water is adsorbed mainly at the impurities,

(2) the adsorbed layer of water is immobile.

The latter assumption may be justified as follows.

We have seen that the energy constant of the impurity isotherm is $C = 5.3$, which corresponds to a heat of adsorption per molecule of the first layer $E_1 = 12$ kcal/mol. For a molecule adsorbed on pure charcoal $E_1 = 9$ kcal. It follows that the motion of a water molecule away from an impurity on to the pure charcoal surface is associated with an activation energy per molecule of roughly 3 kcal. The fraction of free moving molecules on the pore surface at a given temperature is given by $f = e^{-\frac{\Delta E}{RT}}$. In the present case this fraction is negligible.

Therefore, the desorption can be considered as a diffusion process of water vapour through the pores space out of the grain under the influence of the partial pressure difference, i.e. of the concentration difference $C = C_i - C_o$ inside and outside the grain, C_i being the average concentration inside the pore and C_o the outside concentration in g mol . cm^{-3}. The outgassing rate W is given then by:-

$$W = N^* \times \frac{\pi}{4} d^2 \times D_g \frac{C_i - C_o}{L/2} \quad (\text{g mol . cm}^{-3} . \text{sec}^{-1}) \qquad (4)$$

where $N^* \times \frac{\pi}{4} d^2$ is the total area of cross section of all the pore openings in one cm^3.

$$N^* \frac{\pi}{4} d^2 = 2 \, n \, N \frac{\pi}{4} d^2$$

$$= 3.12 \; (\text{cm}^2 . \text{cm}^{-3})$$

L/2 is half the length of the pores

$$L/2 = 8 \times 10^{-2} \; (\text{cm})$$

The diffusion coefficient D_g is in the present case

$$D_g = \frac{1}{3} v \, d \; (\text{cm}^2 . \text{sec}^{-1})$$

because the gas flow is in the Knudsen region.

The mean molecular velocity v is given by:-

$$v = \sqrt{\frac{8 \quad RT}{\pi \ . \ M}}$$

The outside concentration C_o is given by:-

$$C_o = K_o \frac{W}{f + W} \qquad (5)$$

where

K_o = mol volume (g mol . cm^{-3})

W = description rate (g mol . cm^{-3} sec^{-1})

f = flowrate (g mol . cm^{-3} sec^{-1})

It turns out that the outside concentration can be neglected in the present case, since the partial pressure of water in the gas is in the order of 10^{-3} mm Hg.

The following Table shows the outgassing rate calculated according to equation (4) at the corresponding temperatures:

TABLE 3

Temp. T(^oK)	Diff. Coeff. D_g (cm^2/sec)	H_2O Content mg/g	Part Press mm Hg	Gas Concentration C_i (g mol/cm^3)	Outgassing Rate W (g/g . sec)
333	4.17×10^{-3}	3.25	1.44	6.95×10^{-8}	4.1×10^{-7}
363	4.34×10^{-3}	1.5	2.3	10.1×10^{-8}	6.15×10^{-7}
393	4.53×10^{-3}	0.3	1.3	5.6×10^{-8}	3.56×10^{-7}

A desorption rate of the order of 10^{-9} (g/g sec) (see Table 1) is arrived at experimentally.

The difference of 2-3 orders of magnitude between the experimental results and the estimate can be explained as follows:-

(1) As can be seen from the results given by different authors the reliability of the adsorption isotherm of water on coconut charcoal is limited to the one type of charcoal investigated. The uncertainty of the isotherm at low pressures can affect the outgassing estimate up to one order of magnitude.

(2) The model parameters have been chosen so as to satisfy certain experimental results, i.e. total surface, pore diameter and pore space. In order to simplify the model

straight pores were assumed, but this reduces the pore length to a minimum. Taking into account that the pores are in fact not straight and that the value found for the total surface gives the effective surface or microsurface, which would reduce the number of pores considerably, our estimate can easily be affected by two orders of magnitude.

3.2.3 Desorption at High Water Content

In this case it was assumed that:

(1) the main part is adsorbed on the charcoal and not on the impurities, and

(2) the adsorbed molecules are free to move along the pore surface as a two-dimensional gas and their motion is mainly influenced by mutual collisions.

The surface diffusion coefficient D_a is given by:-

$$D_a = \frac{1}{2} v l$$

where v is the molecular velocity and l is the free path.

The free path l will be given by, approximately,

$$l = \frac{v}{v \, m\rho}$$

where ρ is the diameter of the water molecules (2.7 x Å), and m is the number of molecules per cm^2.

The outgassing rate W can then be estimated according to the following equation:

$$W = N^* \left(\frac{\pi}{4} d^2 D_g \frac{C_i - C_o}{L/2} + \pi \, d \, \rho \, D_a \, A \frac{C_i - C_o}{L/2} \right) (\text{g mol} \cdot \text{cm}^{-3} \cdot \text{sec}^{-1}) \quad (6)$$

where ρ is again the diameter of the water molecules and A is the ratio between the amount of water in the vapour phase and in the adsorbed phase.

The above equation for W can also be written:-

$$W = N^* \frac{\pi}{4} d^2 \frac{D_g}{L/2} \left(1 + 4 \frac{\rho}{\alpha} \frac{D_a}{D_g} A\right) (C_i - C_o) \qquad (6a)$$

The numerical calculations were performed for a water content of 4%, which will be considered as a representative point on the steep part of the regeneration curve. The desorption rate will be determined for a regeneration temperature of 90°C.

The surface diffusion coefficient D_a is:

$$D_a = 4.5 \times 10^{-3} \text{ cm}^2 \cdot \text{sec}^{-1}$$

The partial pressure of water inside the charcoal and hence the concentration C_i was determined using the adsorption isotherm shown in Fig. 5, from which it follows that:

$$C_i = 4.65 \times 10^{-6} \text{ g mol} \cdot \text{cm}^{-3}$$

In the present case it is again found that $A \approx 10^4$.

Equation (5) gives the relation between outgassing rate, flow rate and outside concentration, which can also be written:-

$$W = \frac{C_o}{K_o - C_o} f \qquad (5a)$$

Equations (5a) and (6a) are represented in Fig. 7, from which the outgassing rate W is then found

$$W = 8.5 \times 10^{-7} \text{ (g mol} \cdot \text{cm}^{-3} \cdot \text{sec}^{-1})$$

$$W = 3.06 \times 10^{-5} \text{ (g/g} \cdot \text{sec)}$$

An outgassing rate in the order of 10^{-5} (g/g sec) was found experimentally.

3.2.4 Numerical Results

The numerical estimates account reasonably for the difference in the desorption rate in the steep and flat part of the regeneration curves and in general the model shows satisfactorily the difference in the desorption mechanisms in the two sections of the regeneration curve and the parameters influencing the desorption.

Equation (6a) can be reduced to:-

$$W = K \cdot (C_i - C_o) \quad \text{(g mol} \cdot \text{cm}^{-3} \cdot \text{sec}^{-1})$$

where K can be assumed constant, because in our case the variation with partial pressure and temperature is comparatively small. The concentration inside the charcoal C_i is given by the adsorption isotherm. The outside concentration C_o is given by equation (5).

Fig. 7 shows clearly that the desorption rate in the steep part is mainly determined by equation (5a). From this result one can conclude that the pore resistance to diffusion is small and that, for the extrapolation of the experimental results to the conditions of the Dragon delay units, it may be assumed that the partial pressure of water inside the charcoal is roughly equal to that outside and that the outgassing rate W as function of the flow rate f can then be calculated according to equation (5a). On the other hand it is possible, using, equation (5), to estimate the concentrations C_i from the experimental desorption results. A reasonable agreement with Coolidge's isotherm is then found.

The following Table showsthe average desorption rate in the steep part of the regeneration curve calculated according to equation (5a) and found experimentally at a flowrate of 2.5×10^{-1} cc/g . sec.

TABLE 4

Temperature °C	Desorption Rate	
	Calculated g/g . sec	Found by Experiment g/g . sec
60	1.04×10^{-5}	1.4×10^{-5}
90	2.4×10^{-5}	2.46×10^{-5}
120	8.5×10^{-5}	3.4×10^{-5}

A similar agreement between the calculated degassing rate and the experimental value can be seen at different flow rates. The larger deviation of the value at 120°C is caused by a time lag in heating the charcoal sample up to 120°C (see experimental accuracy).

4. CONCLUSION

The influence of different parameters on the desorption of water from charcoal has been shown and it appears possible, using the adsorption isotherm of water on charcoal, to predict with sufficient accuracy the desorption rate for the regeneration of the charcoal in the main fission product delay units. The average desorption rate of charcoal containing between 7 and 0.5% of water at a flow rate of $f = 8 \times 10^{-7}$ g mol/g . sec in the delay units is summarised in Table 5.

TABLE 5

Temperature °C	Desorption Rate g/g . sec
60	3.8×10^{-8}
90	8.3×10^{-8}
120	2.25×10^{-7}

According to these experimental results, the maximum tolerable water content of 0.5% in charcoal can be achieved at a temperature of 60°C, but for the following reasons a higher temperature (120°C) of the heating medium is recommended for regeneration.

(1) The average temperature of the charcoal due to the temperature gradient through the charcoal filled tubes in the delay units - particularly in the 15 in tubes - will be somewhat lower than the temperature of the heating medium outside. Due to the high desorption rate a temperature difference of roughly 10-15°C may be expected.

(2) The transition range from the steep part of the regeneration curve to the flat part occurs definitely below a water content of 0.5% which reduces the time of regeneration considerably. The ultimate water content achievable at this temperature is well below the maximum tolerable water content required for the delay beds.

(3) At higher temperature the desorption rate is still rather high down to a water content in the charcoal below 0.5%. Therefore, the condensation of the water vapour from the helium stream can be performed at a more convenient temperature.

The regeneration curve for the charcoal in the main delay units is shown in Fig. 8. The present calculation of the desorption rate has taken into account that with a 60 kW heater 50 hours are required to achieve a temperature of 120°C for the heating medium and that the average temperature of the charcoal is 10°C below the temperature of the heating medium. According to Fig. 8 a period of 10 days is required to dry the charcoal from the maximum expected water content of 6% down to the maximum tolerable water content of 0.5%.

5. ACKNOWLEDGMENTS

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6. REFERENCES

[1] General Atomic, Division General Dynamics, G.A.-2861. Quarterly Progress Report for the period ending 31st December, 1961.

[2] A. S. Coolidge, J. A. Chim. Soc. 49 708 (1927).

[3] S. Brunauer, "The Adsorption of Gases and Vapours," Oxford University Press, London, 1944, p.169.

[4] Private Communication from Dr. F. A. P. Maggs, CDEE Porton Down.

[5] S. G. Donn and G. R. Donson IGR-TN/CA-842.

[6] See [3] p.374.

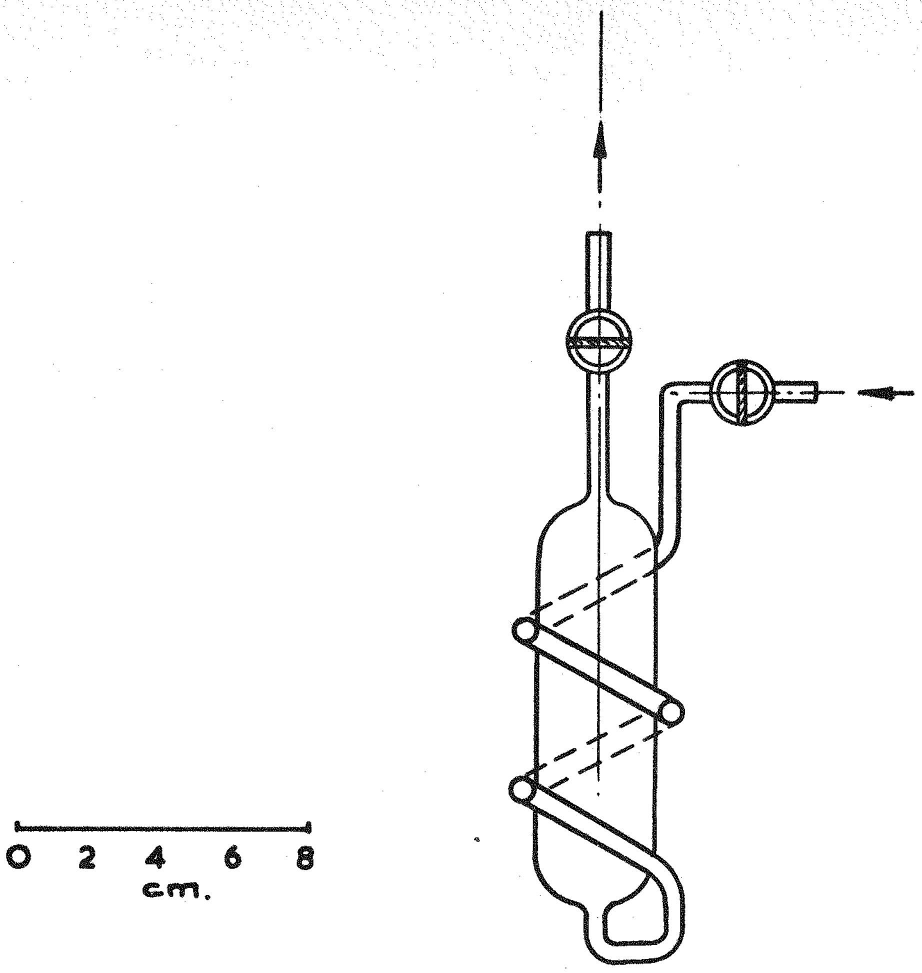

FIG. 1.1. GLASS BULB USED FOR CHARCOAL REGENERATION.

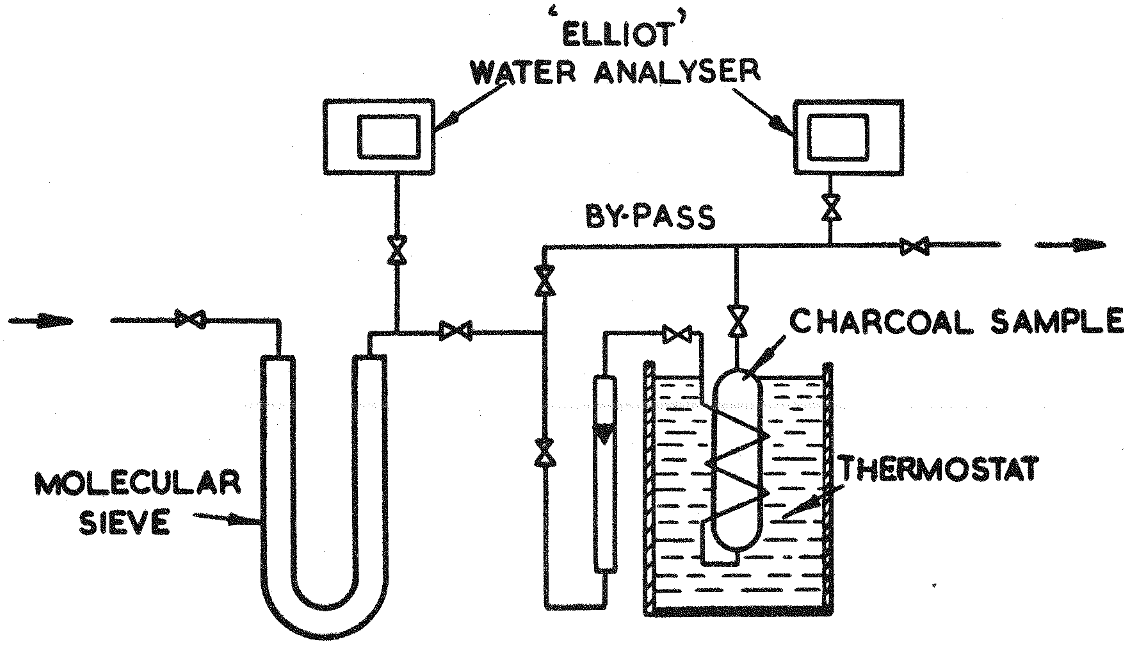

FIG. 1.2. EXPERIMENTAL SET-UP.

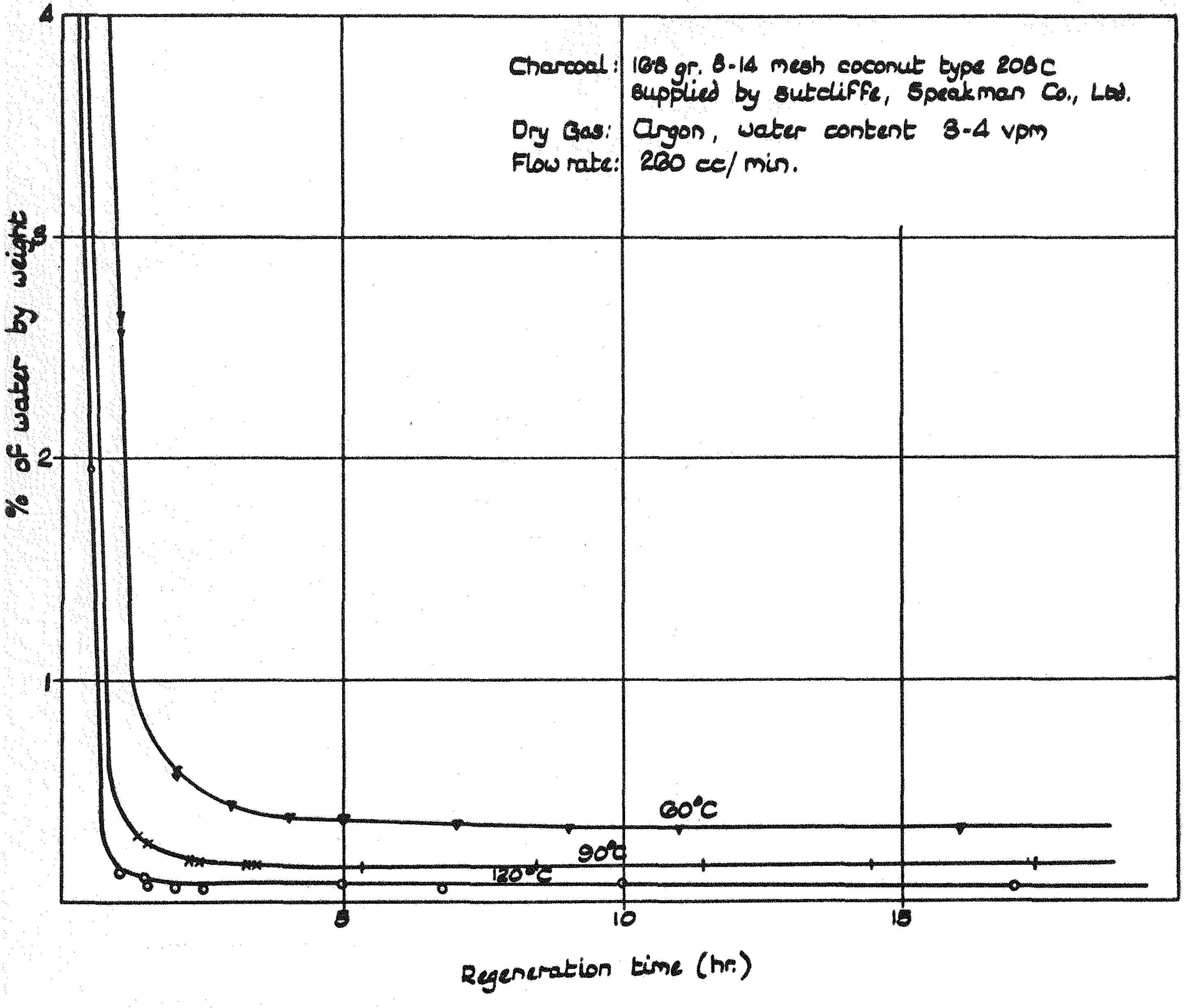

FIG.2. REGENERATION OF CHARCOAL AT DIFFERENT TEMPERATURES AND CONSTANT FLOWRATE

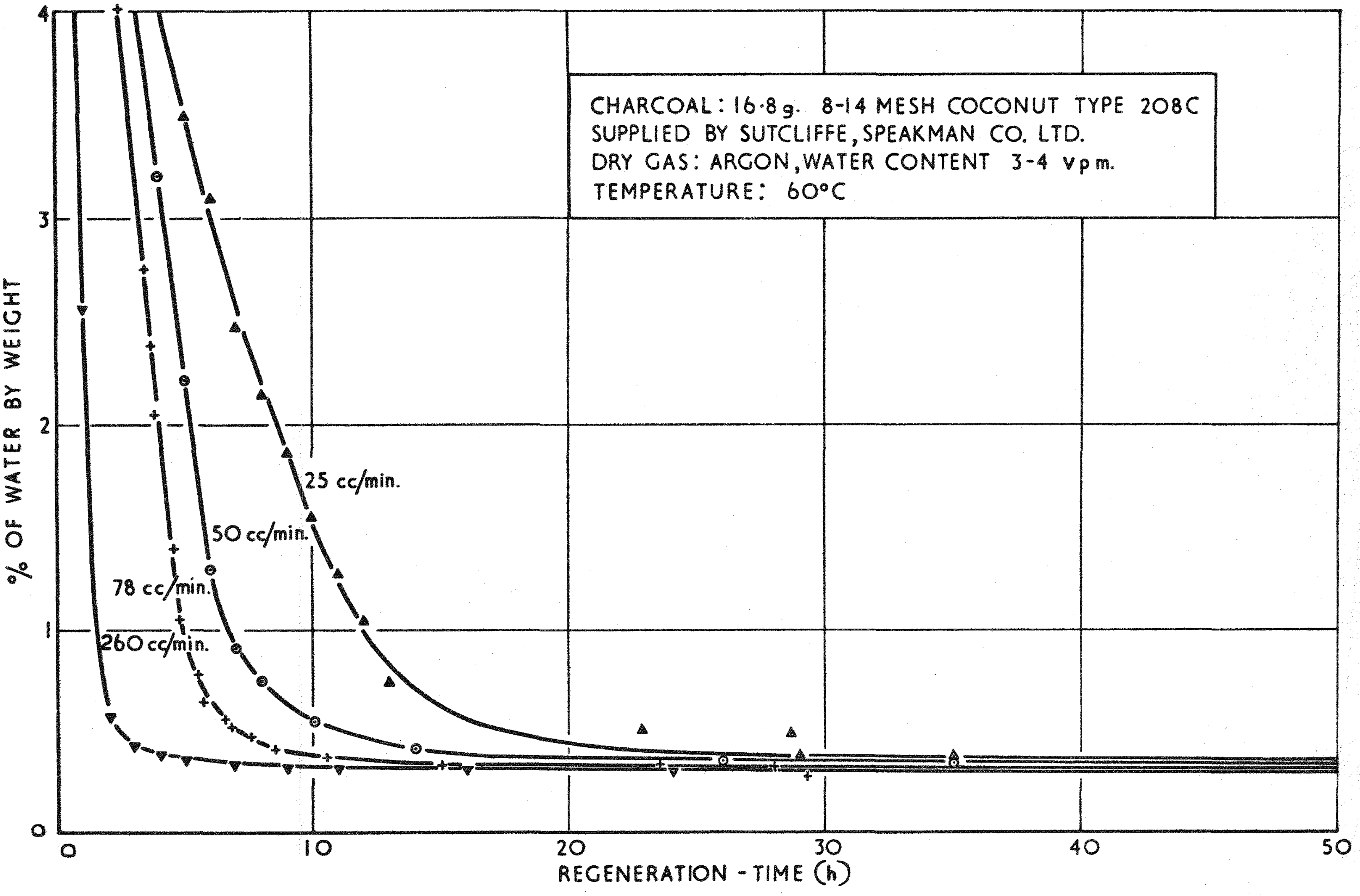

FIG. 3. REGENERATION OF CHARCOAL AT DIFFERENT FLOW RATES AND CONSTANT TEMPERATURE OF 60°C.

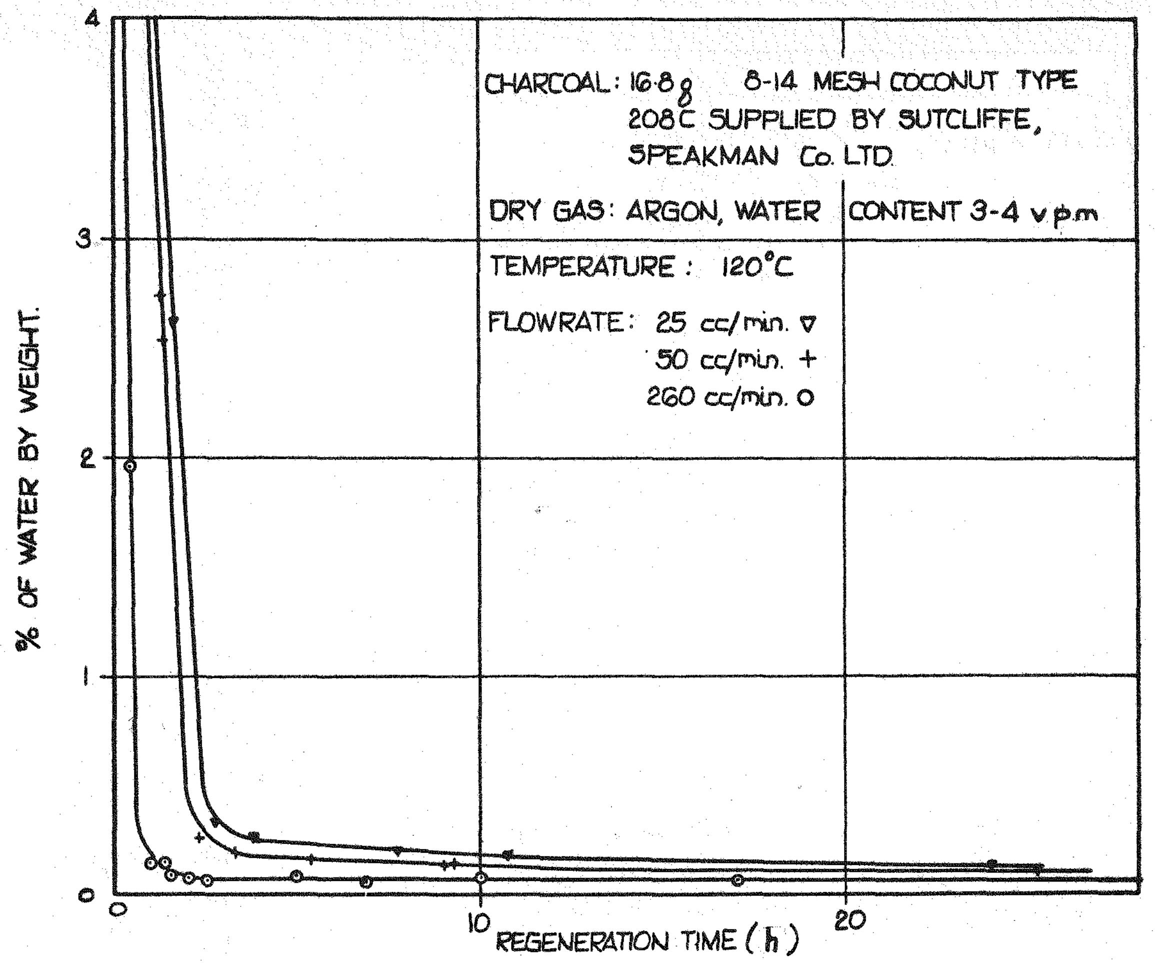

FIG.4.1. REGENERATION OF CHARCOAL AT DIFFERENT FLOW RATES AT 120°C.

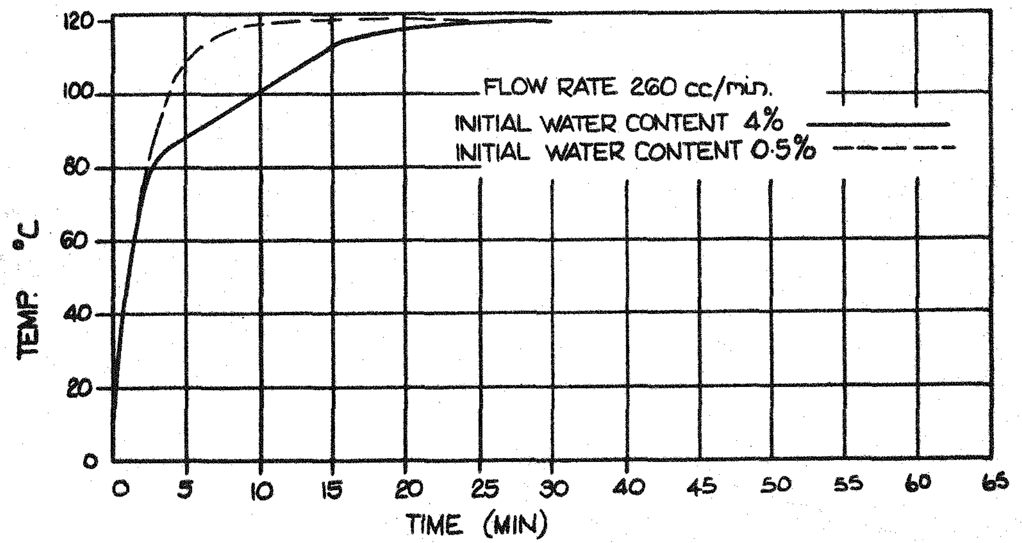

FIG.4.2. TIME LAG IN HEATING THE CHARCOAL SAMPLE UP TO 120°C

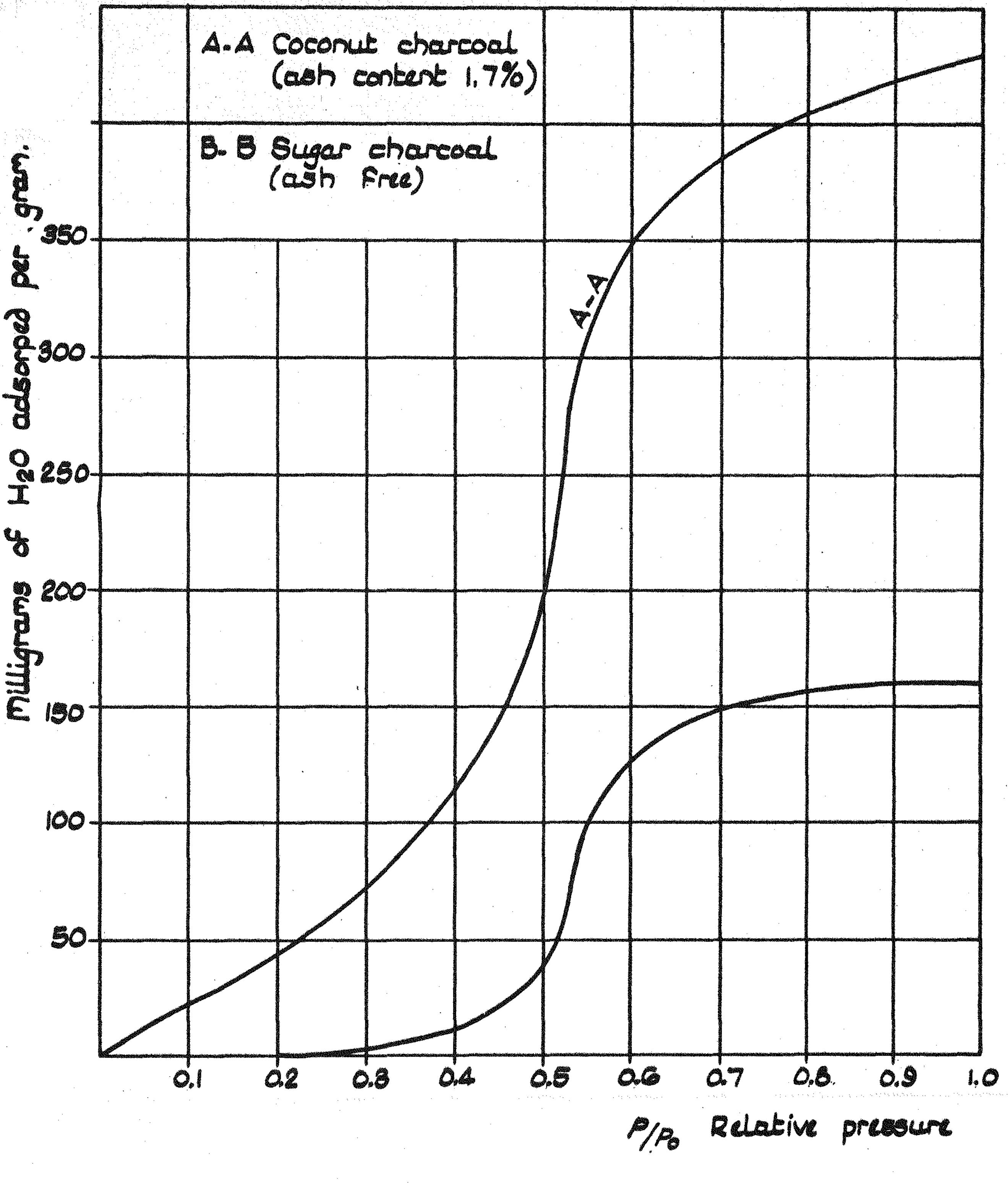

FIG. 5. ADSORPTION ISOTHERM OF WATER ON CHARCOAL AT 100°C

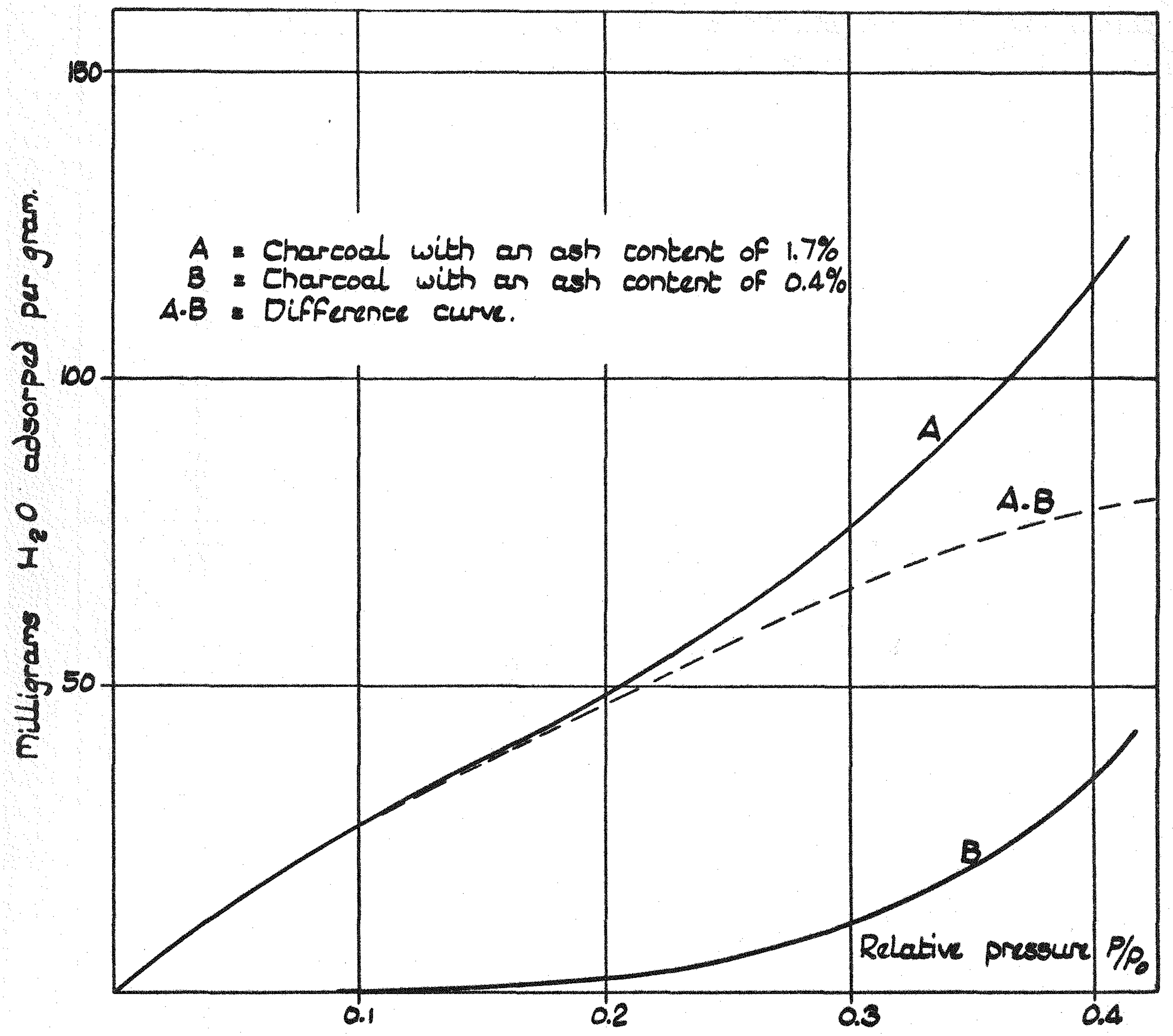

FIG. 6. ADSORPTION ISOTHERM OF WATER ON COCONUT CHARCOAL AT 100° C

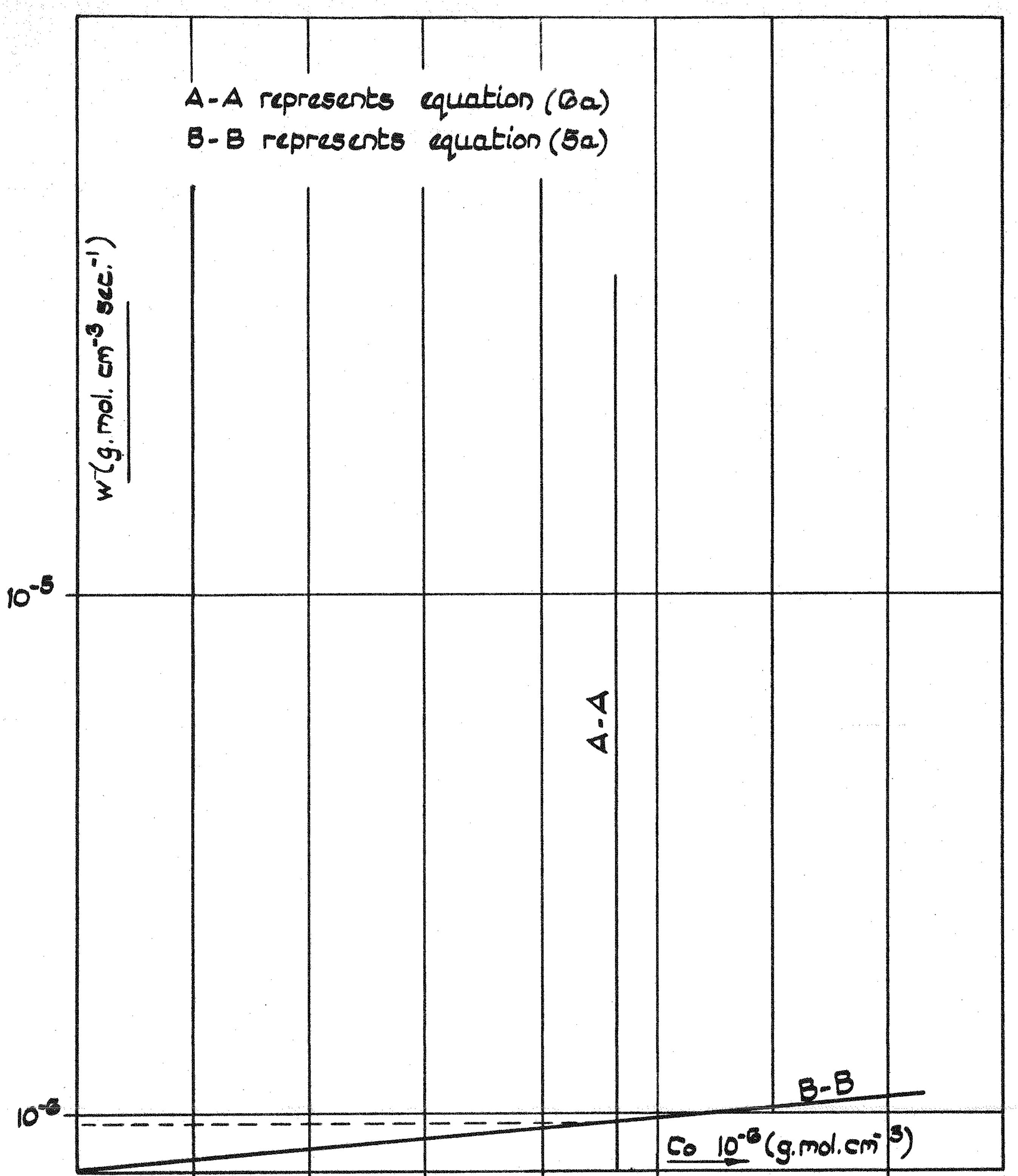

FIG. 7 DETERMINATION OF THE DESORPTION RATE W AT A FLOWRATE OF $2 \cdot 5 \times 10^{-1}$ CC/G.SEC. AND A TEMPERATURE OF 90°C

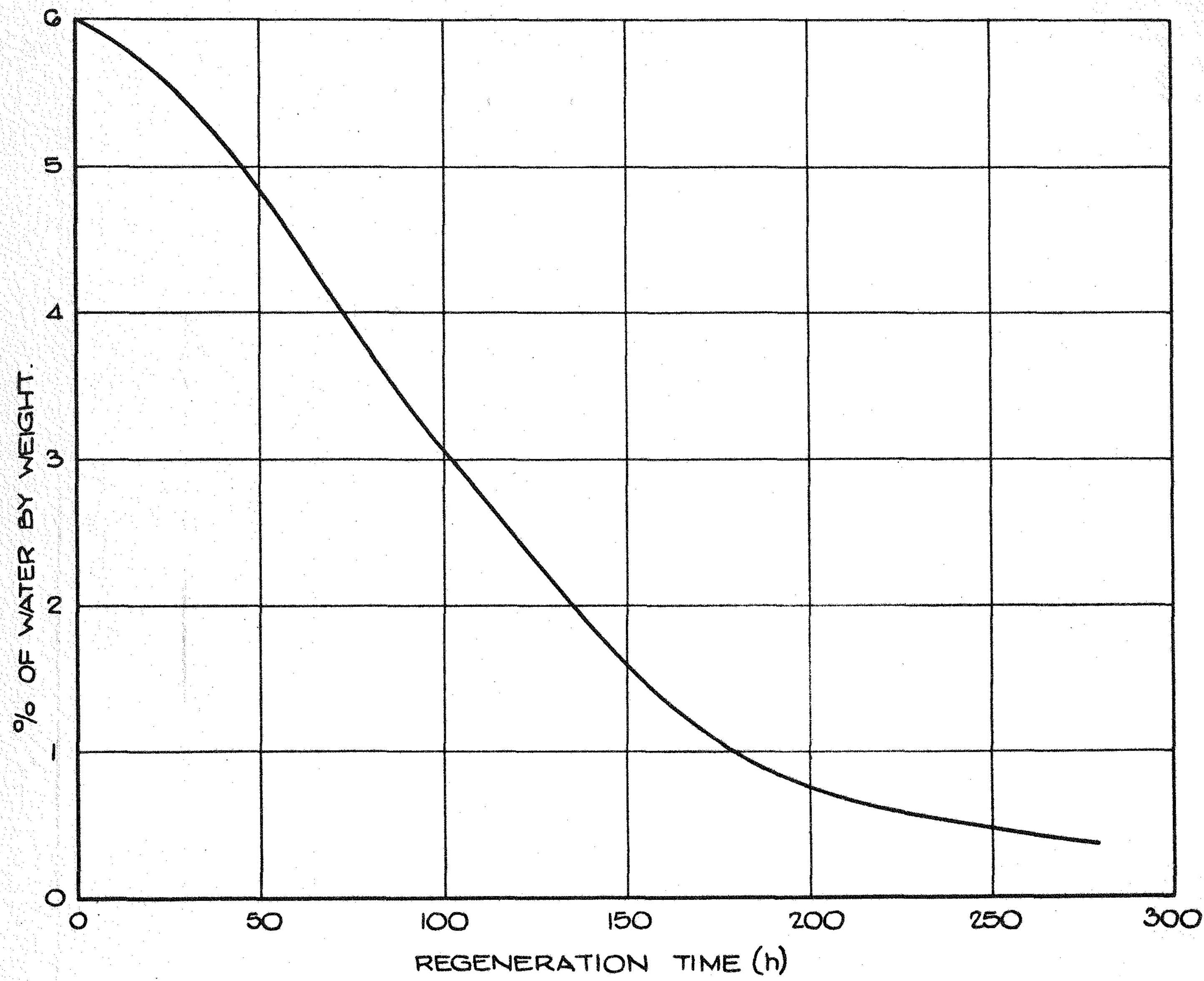

FIG. 8. REGENERATION OF CHARCOAL UNDER THE CONDITIONS OF THE MAIN DELAY UNIT.