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ENGINEERING PRACTICE SCHOOL
CARBIDE AND CARBON CHEMICALS CORPORATION

AECD-4232

MEMORANDUM
MPS-X51

June 22, 1949

TO: R. F. Baddour
FROM: R. N. Saleeby, leader, J. R. Cheshire, and W. P. Jensen
SUBJECT: Analyses of Decay Curves of Irradiated Plastics

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A general program is being conducted at the Oak Ridge National Laboratory to investigate the effects on plastic materials of exposure to radiation in the pile. In the present investigation, several plastics were irradiated, and on removal from the pile, their rates of decay were determined by measurement of gamma activity in an ionisation chamber. (For details of procedure, see Appendix).

RESULTS

Decay curves for the various plastics, together with their graphical analyses, are shown in Figs. 1 to 8. The radioactive isotopes probably present in the irradiated plastic, and the stable parent isotopes from which they were formed, are listed in Table I. Also presented are the approximate weight percentages of the parent element in the plastic, where data necessary for this calculation were available.

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Table I

ANALYSES OF DECAY CURVES OF IRRADIATED PLASTICS

Plastic	Observed Half-Lives of Radio-active isotopes	Probable Radio-active isotope	Stable parent isotope	Weight percent Parent Element in Plastic
Saran	37 min 5.1 hr 8 days	37 min Cl^{38} ? ?	Cl^{37} ? ?	36.2
Ethocel	40 min. 14.4 hr 67 hr	37 min Cl^{38} 14.8 hr Na^{24} 63 hr Sc^{48}	Cl^{37} Na^{23} Ti^{48} (?)	0.072 0.01
Teflon	27 min 14.7 hr	37 min Cl^{38} 14.8 hr Na^{24}	Cl^{37} Na^{23}	0.041 0.003
Bakelite (paper filler)	43 min 5.3 hr 16 hr	37 min Cl^{38} ? 14.8 hr Na^{24}	Cl^{37} ? Na^{23} (?)	0.086
Fluorothene	37 min	37 min Cl^{38}	Cl^{37}	27.2
Bakelite (Asbestos filler)	2.35 hr 9 hr 20.2 hr	2.59 hr Mn^{56} ? ?	Mn^{55} ? ?	0.0083
Casein	4.3 hr 15.2 hr	? 14.8 hr Na^{24}	? Na^{23}	0.15
Polystyrene	32 min 15.2 hr	37 min Cl^{38} 14.8 hr Na^{24}	Cl^{37} Na^{23}	0.203 0.001

A question mark (?) indicates uncertainty in the chemical identification.

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DISCUSSION OF RESULTS

Method of Graphical Analysis

The observed half-lives of the isotopes causing activity in the irradiated plastics were obtained by a graphical method. This method depends upon the fact that radioactive disintegrations are first-order reactions. Thus a plot of activity of a single radioisotope versus time yields a straight line on semi-logarithmic paper. Since the decay curve for an irradiated plastic represents the disintegration of several isotopes having different half-lives, a straight line is not obtained except at a time when the activity of a single component greatly exceeds that of the others. If the curve is extended over sufficient time, the activity of the isotope of longest half-life appears as a straight line. When this straight portion of the curve is extrapolated to zero time, the slope of this line defines the half-life, which is the time required for the activity to be reduced by one-half. If the straight line is then graphically subtracted from the decay curve for the plastic, the resulting subtraction curve will also have a straight portion, corresponding to the decay of the second longest lived isotope. By repeating the procedure of extrapolating the straight portion of the curve to zero time and subtracting curves, the several straight lines which comprise the decay curve for the plastic may be obtained.

This method of analysis does not yield the chemical identity of a radioactive isotope, but only its half-life. It does not distinguish between two different isotopes having the same or nearly the same half-life. The accuracy of the analysis depends on choice of the best curve representing the experimental data, and any error made in the drawing of a given line is magnified in all subsequent constructions. The accuracy is often further impaired because the determination of a straight line for the isotope of longest half-life must be made from data obtained when the activity is low and accuracy of the counting instrument is relatively poor.

Chemical Identification of Radioactive Isotopes

Several factors were considered in the chemical identification of the isotopes producing activity in the irradiated plastics. Determination of each of the radioactive isotopes was based upon the following requirements:

1) The isotope must decay by gamma ray emission.

2) The known half-life of the isotope must be nearly the same as that determined by graphical analysis of the decay curve.

3) The radioisotope must be formed from an element likely to occur in the plastic, either as a chemical constituent or as an impurity. A study of the chemical process by which the plastic was made and the spectrographic analysis of the ash of the plastic (see Appendix)

indicated some of the stable isotopes present.

4) The radioisotope must be formed by a neutron capture reaction, such as n-γ, n-α, or n-p. The n-γ reaction involves capture of a thermal neutron, for which the cross section is usually high, whereas the other two reactions occur principally with fast neutrons, for which the capture cross section is usually low. Therefore the n-γ reaction is the one most likely to occur in the pile.

In addition to these requirements, two other factors influenced the choices. The stable isotope from which the radioisotope is formed should have a high capture cross section for thermal neutrons, or should be relatively abundant in the natural element.

Calculation of the Amounts of Parent Elements in Plastics

The data obtained in this work by analysis of the decay curves were used for quantitative determination of certain of the elements present in the plastics. The basis of the calculation is that the degree of activity, A_0, of a radioactive isotope at the moment of removal of the sample from the pile depends upon the number of atoms of the isotope present, thus upon the number of atoms of its stable parent isotope in the plastic before irradiation. The relationship is expressed by the following equation (1),

$$A_0 = nv\sigma N(1 - e^{-\lambda t}),$$

where nv is the neutron flux, neutrons/(sq cm)(sec) during irradiation,

σ is the cross section for neutron capture for the parent isotope,

N is the number of atoms of parent isotope subjected to irradiation,

λ is the decay constant for the radioisotope, and

t is the time of exposure to irradiation.

A_0 is obtained by extrapolation to zero time of the decay curve for the isotope as previously discussed. For greater accuracy the values of A_0 used in the calculations were determined by extrapolation on large-scale plots, rather than with those presented in this report. Since experimental data on neutron cross sections are not highly accurate, and since the graphical analysis is a subjective method, the accuracy of the quantitative determination may not be high. However, the method is extremely valuable for the quantitative determination with fair accuracy of elements present in such small amounts that they would not be detected in chemical methods of analysis.

Analysis of Plastics

Of the three radioisotopes observed in irradiated Saran (Fig. 1), only one, 37 min Cl^{38}, could be identified with certainty. Saran (vinylidene chloride-acrenitrile copolymer) contains 47.3% chlorine. The amount calculated from the experimental data was 36.2%. The 5.1 hr isotope could be 4.4 hr Br^{79}, but no evidence could be found indicating the presence of bromine in Saran.

The 14.4 hr and 40 min isotopes observed in Ethocel (Fig 2) were identified as 14.8 hr Na^{24} and 37 min Cl^{38}. These isotopes are formed from Na^{23} and Cl^{37}, respectively, by n-γ reactions. Both sodium hydroxide and ethyl chloride are used in the manufacture of Ethocel. The 67 hr isotope could be 63 hr Sc^{48}, formed from Ti^{48} by n-p reaction. (4) Spectrographic analysis indicated the presence of titanium in Ethocel but 63 hr Sc^{48} has not been included in any compilation of isotopes appearing in literature since 1944, and its occurrence is somewhat doubtful.

The elements producing activity in Teflon were identified as sodium and chlorine. The end group in the Teflon polymer is $-CCl_3$, which would account for the presence of the 37 min Cl^{38} activity. The occurrence of sodium in the Teflon was indicated by spectrographic analysis.

In the polymerization of Bakelites, both sodium hydroxide and hydrochloric acid are frequently used as catalysts. Analysis of the decay curve for paper-filled Bakelite (Fig. 4) indicated activity derived from both sodium and chlorine. Spectrographic data did not indicate the presence of sodium in the plastic, so that this identification is somewhat in doubt.

Two decay curves for Fluorothene (Fig. 5) were obtained by use of two samples, because the first curve showed an increase in activity after 11 hours and a check determination was desired. The second curve showed no activity increase, but the two curves did not agree except in the initial steep portion, so that complete analysis of the data was impossible. Only 37 min Cl^{38} could be definitely determined. The drawing of the straight line representing this activity without complete analysis of the decay curve was justified in the case of the Fluorothene because any activities which should have been subtracted from the total decay curve would be so small that neither the slope nor the intercept of the initial steep portion would be noticeably affected. The appearance of chlorine activity was to be expected since Fluorothene contains 30.5% chlorine. The amount of chlorine in Fluorothene as calculated from the experimental data was 27.1%.

Of the three radioactive isotopes detected in asbestos-filled Bakelite (Fig. 6) only the 2.35 hr isotope could be identified. This was 2.59 hr Mn^{56}. The presence of manganese was confirmed by spectrographic data.

Two radioactive isotopes were detected in Casein (Fig. 7). The 15.2 hr activity was identified as 14.8 hr Na^{24}. The spectrographic data showed presence of sodium in Casein. Some of the 4.3 hr activity might have been

caused by 2.5 hr Ca^{49}, which is formed from Ca^{48}. The spectrographic data showed very strong Ca in the plastic. Calculation of the amount of calcium in Casein, based on the assumption that all the 4.3 hr activity was due to 2.5 hr Ca^{49}, gave a figure one and one-half times as great as the actual weight of the Casein sample irradiated. This indicates that the 4.3 hr activity was not in the main due to 2.5 hr Ca^{49}. The isotope causing this activity could not be identified. It might have been manganese, but this was not detected in the spectrographic analysis.

Analysis of the decay curve for polystyrene (Fig. 8) indicated sodium and chlorine activity. Sodium was detected in the spectrographic analysis. Hydrochloric acid is probably used in the manufacturing process, and would account for the chlorine.

The possibility of occurrence in a plastic of a radioactive isotope from two different sources, or of two different isotopes having the same half life, may throw doubt on some of the analyses. For example, in the case of Teflon, the activity having 14.7 hr half life was identified as 14.8 hr Na^{24}, formed from Na^{23} by n-γ reaction. The n-α reaction of Al^{27} also yields Na^{24} (6), and the spectrographic analysis showed very strong aluminum, so that some of the activity reported as due to sodium was in face due to aluminum. However this amount must have been small, because of the very low capture cross-section for Al^{27}, which is about 1/1000 of that for Na^{23} (6). Another example is afforded in the case of asbestos-filled Bakelite. The 2.35 hr activity was reported as 2.59 hr Mn^{56}, formed from Mn^{55}. The spectrographic analysis showed moderate Ni. By a n-γ reaction, 2.6 hr Ni^{65} is produced from stable Ni^{64} (6). Hence some of the 2.35 hr activity was due to nickel. The capture cross-section for Mn is about four times that of the Ni, but the natural abundance of the Mn isotope which produces a daughter isotope having 2.6 hr activity is about ninety times that of Ni (6). Therefore most of the activity must have been due to Mn rather than to Ni.

LOCATION OF ORIGINAL DATA

The original data of this investigation appear in Notebook no. 1073, pp 16-24; Notebook no. 1300, pp 63-69; log sheets of pile operations, June 7 to June 20, 1949, located in files of the Operations Division, CRNL.

R. H. Saleeby, Jr.

J. R. Cheshire

W. P. Jensen

APPENDIX

A. Detailed Procedure

After irradiation of the plastic in hole no. 22 of the ORNL pile, the gamma activity was counted in a 100% geometry ionization chamber. This instrument was calibrated at different activity levels, using cobalt sources of known activity as standards. A calibration curve was available to correct for chamber efficiency, the efficiency with which gamma rays produce ions in the chamber, at energy levels different from that of the Co^{60} standard source.

Samples for spectrographic analysis were ignited in porcelain crucibles for 24 hr at 1100 C in a muffle furnace. The ash from these samples was analyzed spectrographically by the Chemistry Division. Results of the analyses are given below.

RESULTS OF SPECTROGRAPHIC ANALYSES

Material	Polystyrene	Fluorothene	Saran	Casein	Bakelite (paper filled)	Teflon	Ethocel	Bakelite (asbestos filled)		Polystyrene	Fluorothene	Saran	Casein	Bakelite (paper filled)	Teflon	Ethocel	Bakelite (asbestos filled)
Ag	VFT	VFT	VFT	—	—	—	—	—	Mn	S	S	S	—	—	M	—	W
Al	T	T	T	VFT	M	VS	FT	VW	Mo	—	—	—	—	—	—	—	—
As	—	—	—	—	—	—	—	—	Na	VW	VW	VW	T	—	T	—	FT
Au	—	—	—	—	—	—	—	—	Ni	S	S	S	—	—	M	—	M
B	—	—	—	—	—	T	—	—	Pb	—	—	—	—	—	FT	—	—
Ba	—	—	—	—	—	—	—	—	Rb	—	—	—	—	—	—	—	—
Be	T	T	T	—	—	—	—	—	Sb	—	—	—	—	—	—	—	—
Bi	—	—	—	—	—	—	—	—	Si	W	W	W	—	FT	S	W	S
Ca	VW	VW	VW	VS	VS	S	FT	W	Sn	—	—	—	—	—	—	—	—
Cd	—	—	—	—	—	—	—	—	Sr	FT	VFT	VFT	—	—	—	—	
Co	T	T	T	—	—	—	—	FT	Ta	—	—	—	—	—	—	—	—
Cr	M	M	M	—	—	M	W	M	Ti	T	T	T	—	—	T	VS	VFT
Fe	VS	VS	VS	—	S	W	T	VS	V	—	—	—	—	—	—	—	—
K	—	—	—	—	—				W	—	—	—	—	—	—	—	—
Li	—	—	—	—	—	—	—	—	Zn	—	—	—	—	—	—	—	—
Mg	VW	VW	VW	M	W	VW	VFT	S									

VS = Very Strong
S = Strong
M = Moderate
W = Weak
VW = Very Weak
T = Trace
FT = Faint Trace
VFT = Very Faint Trace
— = Sought, Not Found

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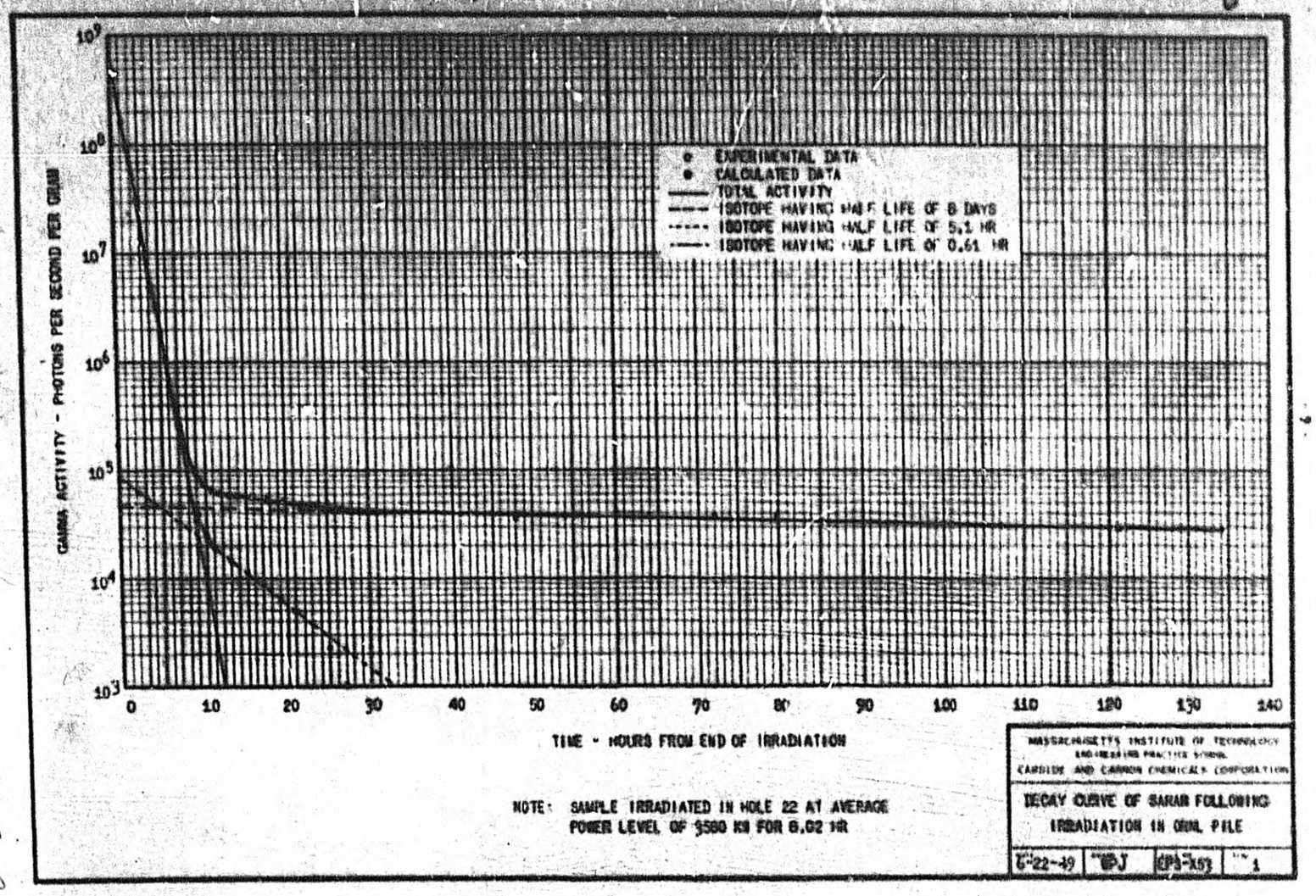

NOTE: SAMPLE IRRADIATED IN HOLE 22 AT AVERAGE POWER LEVEL OF 3580 KW FOR 8.02 HR

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DECAY CURVE OF SARAR FOLLOWING IRRADIATION IN ORNL PILE

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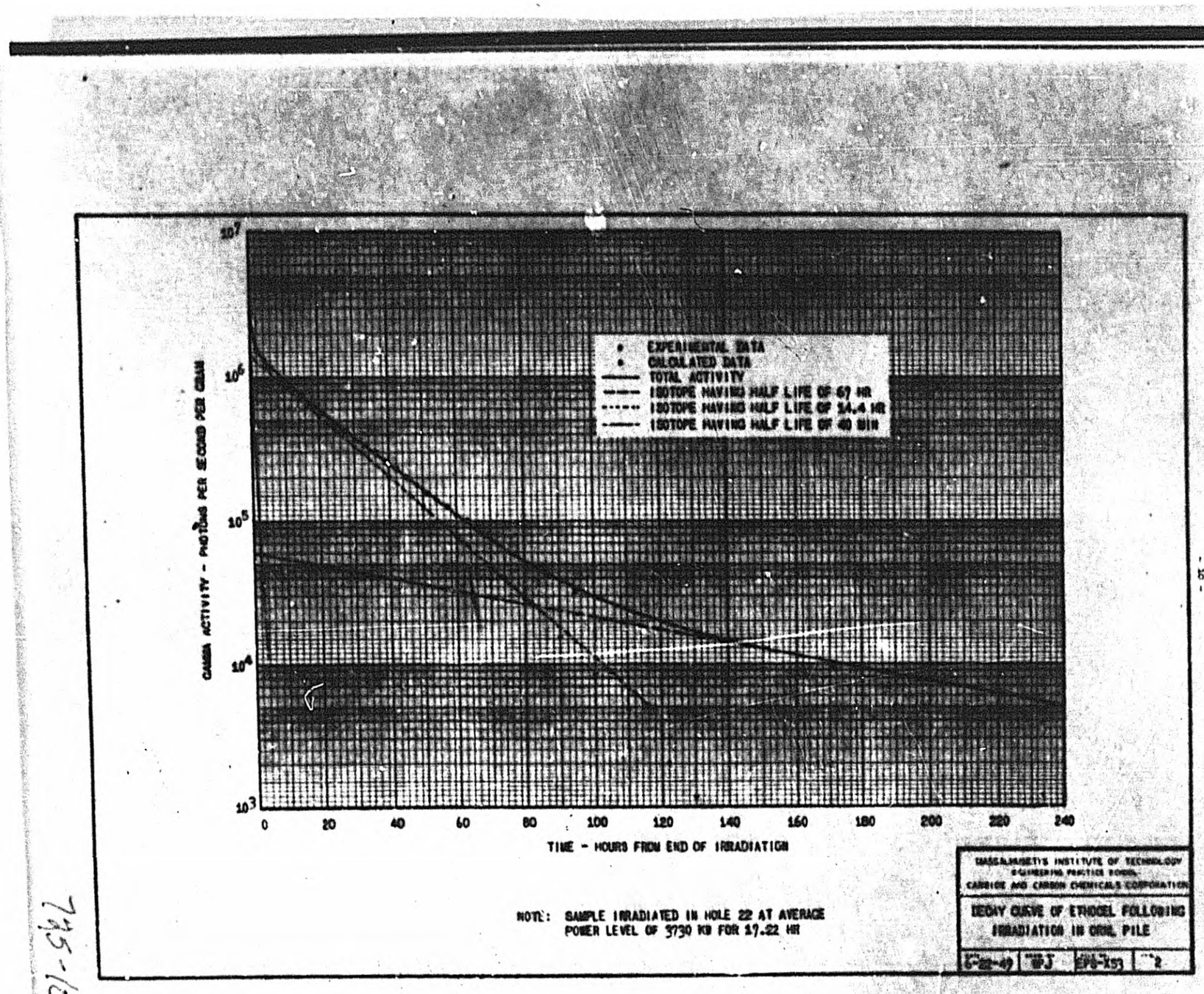
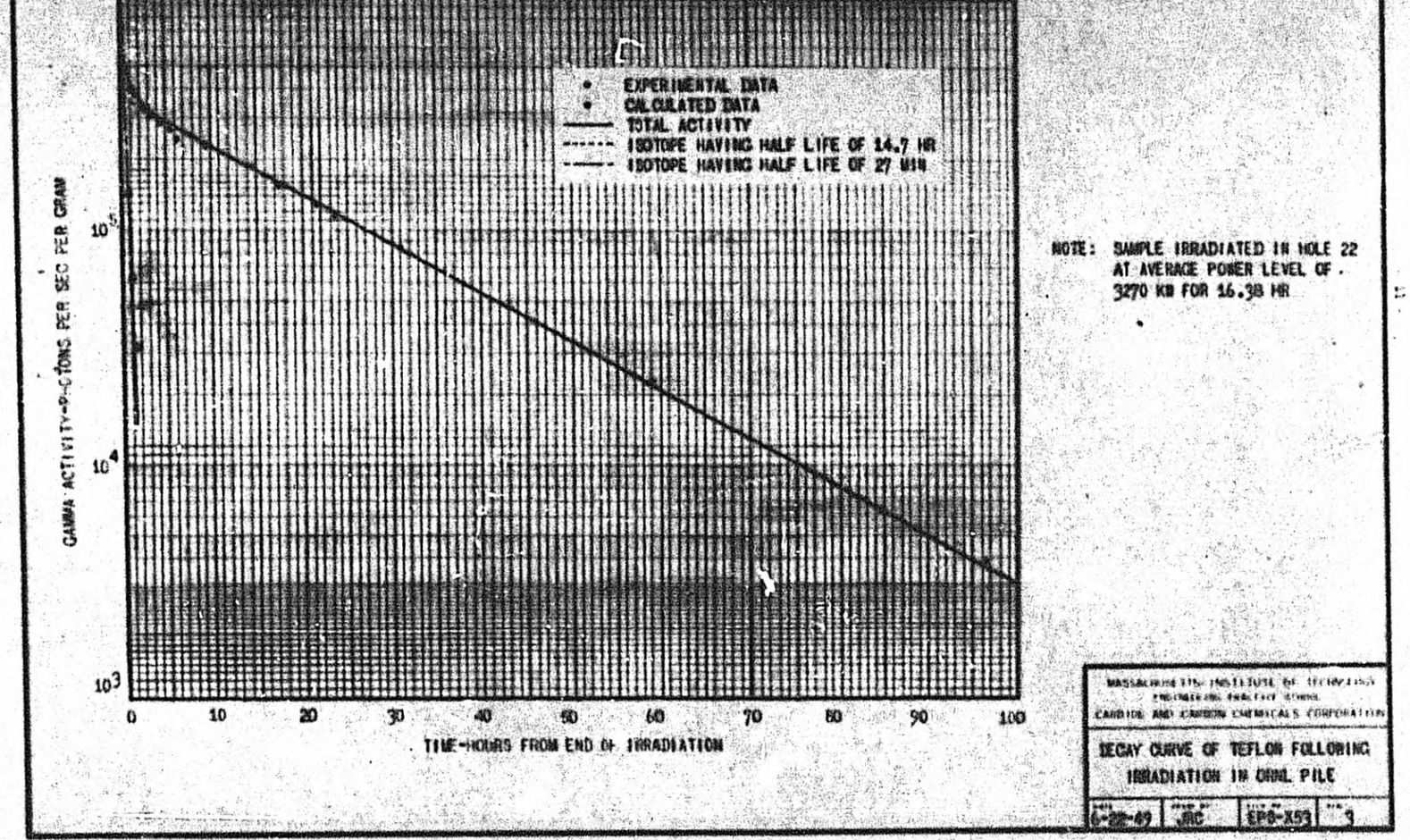
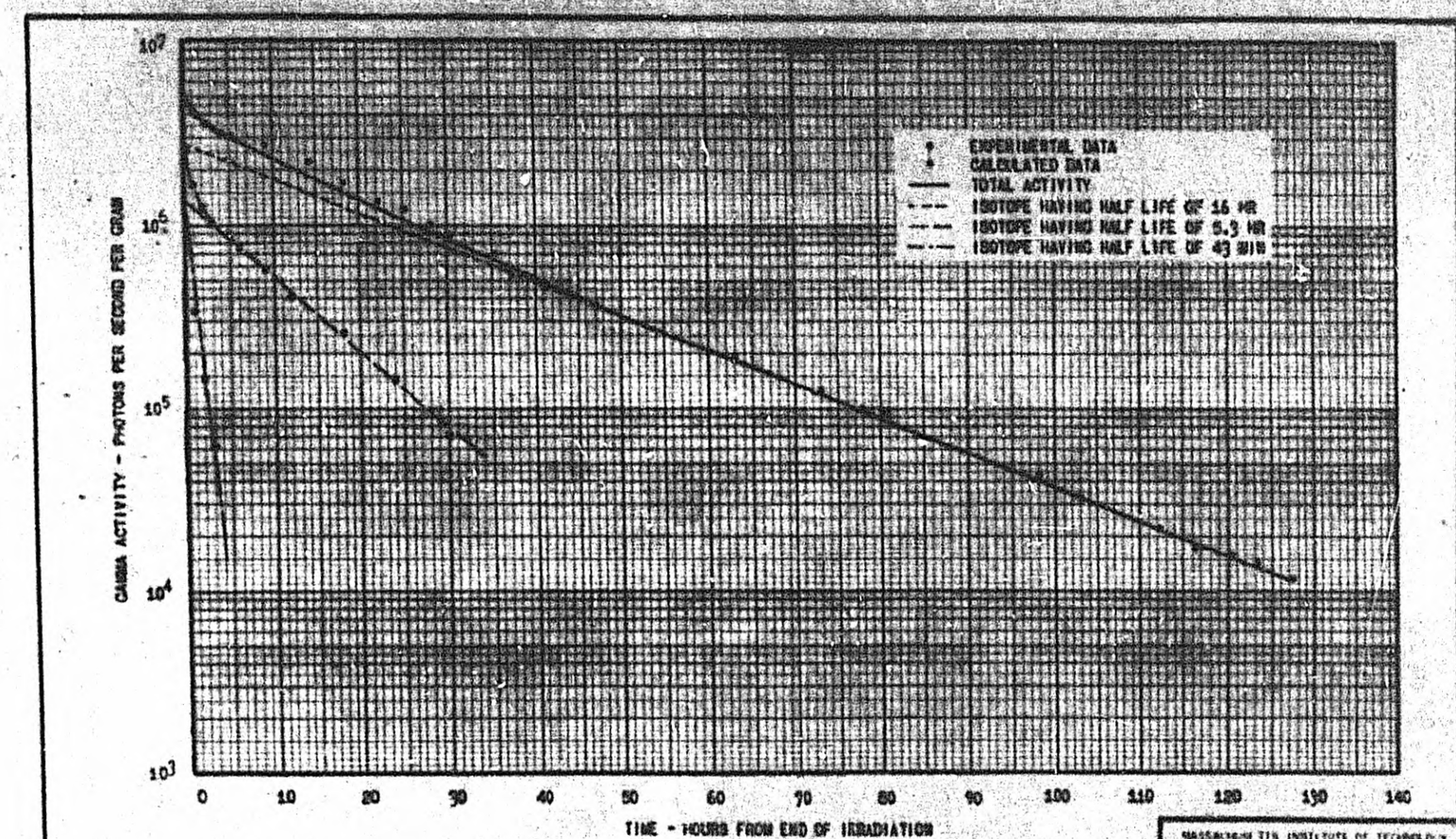


NOTE: SAMPLE IRRADIATED IN HOLE 22 AT AVERAGE POWER LEVEL OF 3730 KW FOR 17.22 HR

745-10


EXPERIMENTAL DATA
CALCULATED DATA
TOTAL ACTIVITY
ISOTOPE HAVING HALF LIFE OF 14.7 HR
ISOTOPE HAVING HALF LIFE OF 27 MIN
NOTE: SAMPLE IRRADIATED IN HOLE 22 AT AVERAGE POWER LEVEL OF 3270 KW FOR 16.38 HR
GAMMA ACTIVITY-DISINTEGRATIONS PER SEC PER GRAM
10^6
10^5
10^4
10^3
0
10
20
30
40
50
60
70
80
90
100
TIME-HOURS FROM END OF IRRADIATION
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
CAMBRIDGE AND CARBON CHEMICALS CORPORATION
DECAY CURVE OF TEFLON FOLLOWING IRRADIATION IN ORNL PILE
6-28-49
JRC
EPS-X53
3

7B7-11



NOTE: SAMPLE IRRADIATED IN HOLE 22 AT AVERAGE POWER LEVEL OF 3690 KW FOR 63.75 HR

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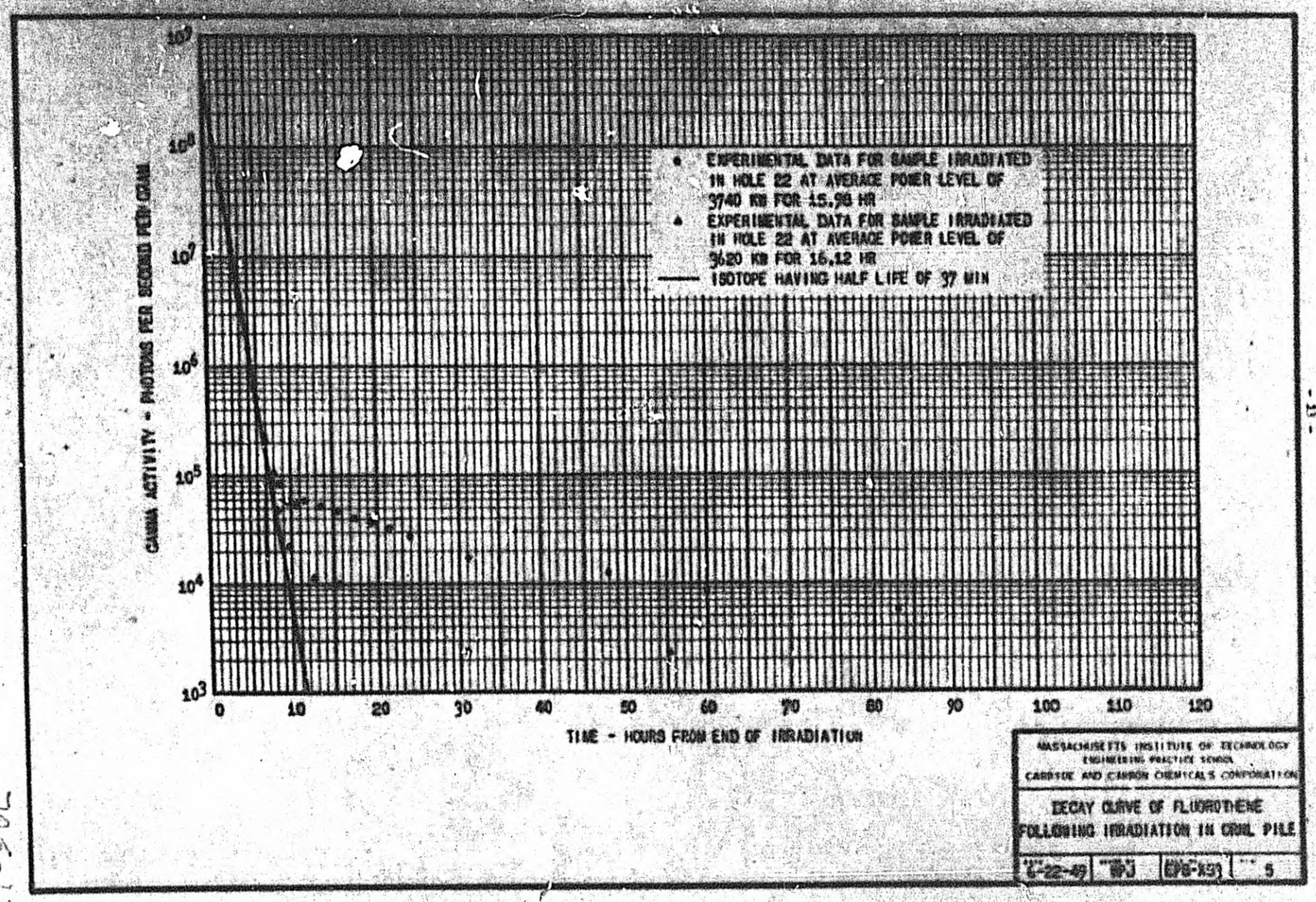
DECAY CURVE OF BAKELITE
(WITH PAPER FILLER)
FOLLOWING IRRADIATION IN ORNL PILE

6-22-49	JRC	EPS-X53	4

725-12



EXPERIMENTAL DATA FOR SAMPLE IRRADIATED IN HOLE 22 AT AVERAGE POWER LEVEL OF 3740 KW FOR 15.98 HR
EXPERIMENTAL DATA FOR SAMPLE IRRADIATED IN HOLE 22 AT AVERAGE POWER LEVEL OF 3620 KW FOR 16.12 HR
ISOTOPE HAVING HALF LIFE OF 37 MIN
GAMMA ACTIVITY - PHOTONS PER SECOND PER GRAM
TIME - HOURS FROM END OF IRRADIATION
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
CARBIDE AND CARBON CHEMICALS CORPORATION
DECAY CURVE OF FLUOROTHENE
FOLLOWING IRRADIATION IN ORNL PILE
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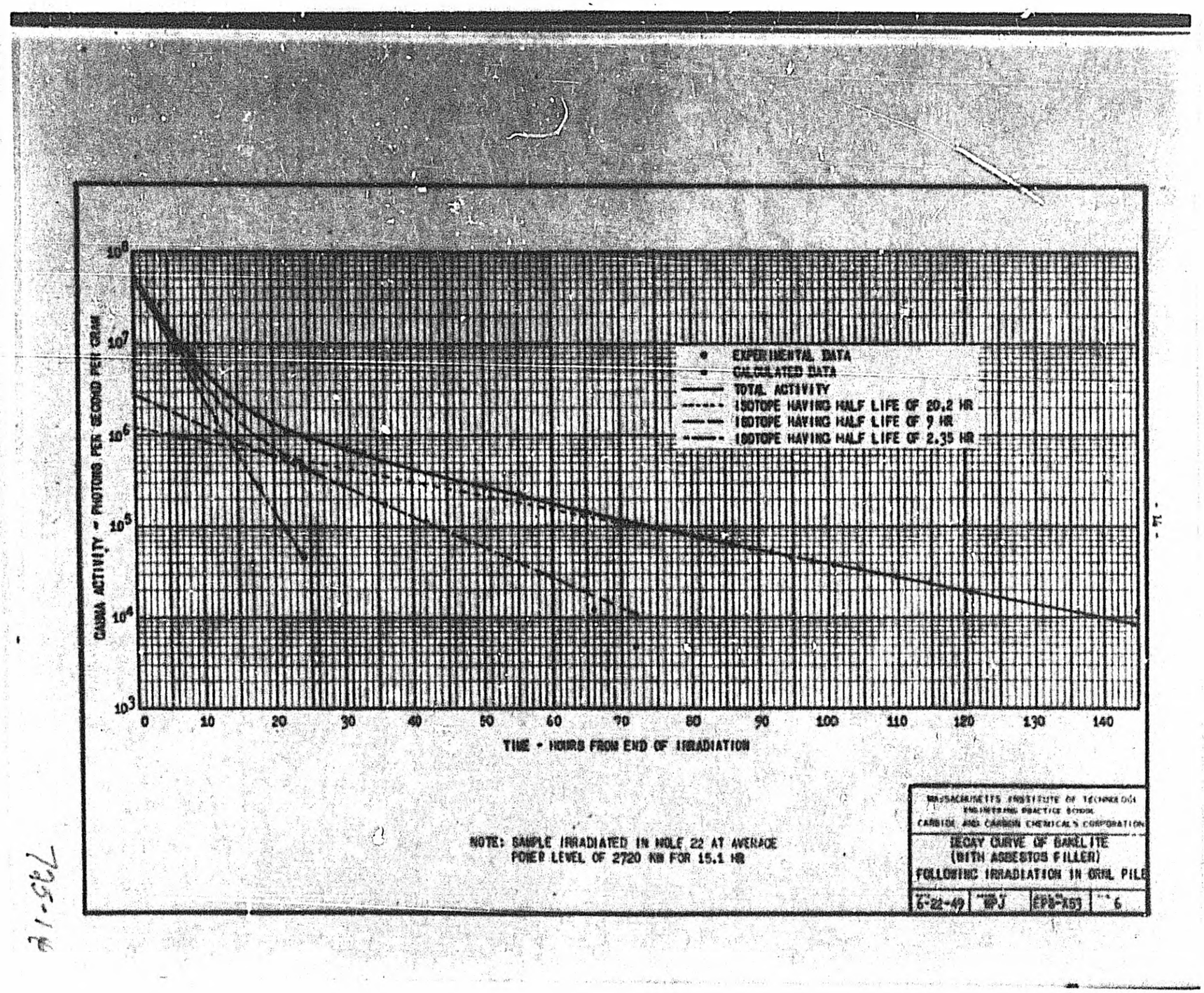
GAMMA ACTIVITY - PHOTONS PER SECOND PER CRM
10^8
10^7
10^6
10^5
10^4
10^3
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
TIME - HOURS FROM END OF IRRADIATION
EXPERIMENTAL DATA
CALCULATED DATA
TOTAL ACTIVITY
ISOTOPE HAVING HALF LIFE OF 20.2 HR
ISOTOPE HAVING HALF LIFE OF 9 HR
ISOTOPE HAVING HALF LIFE OF 2.35 HR
NOTE: SAMPLE IRRADIATED IN HOLE 22 AT AVERAGE POWER LEVEL OF 2720 KW FOR 15.1 HR
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
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CARBIDE AND CARBON CHEMICALS CORPORATION
DECAY CURVE OF BAKELITE
(WITH ASBESTOS FILLER)
FOLLOWING IRRADIATION IN ORNL PILE
6-22-49
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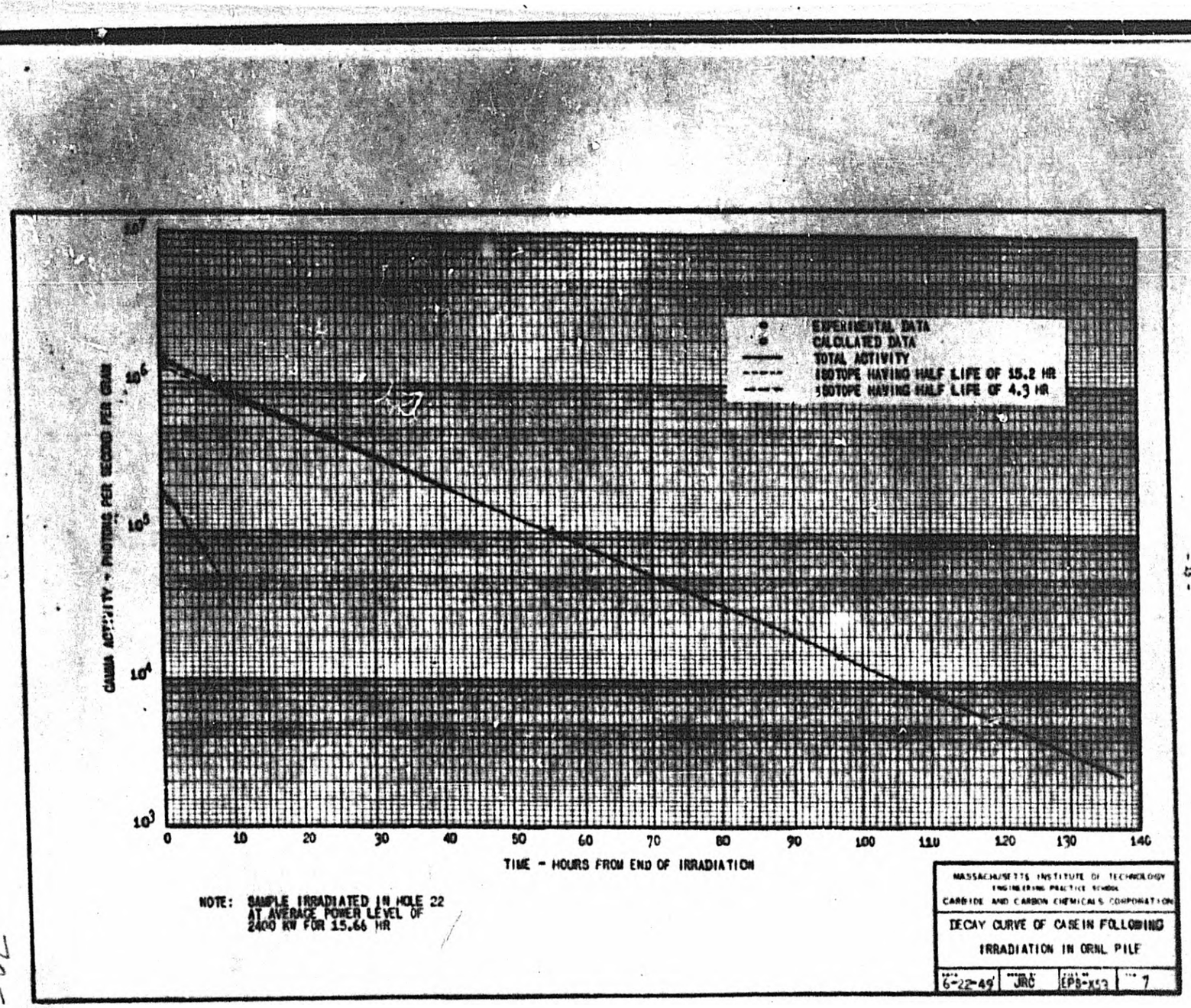
EPS-X53
6


EXPERIMENTAL DATA
CALCULATED DATA
TOTAL ACTIVITY
ISOTOPE HAVING HALF LIFE OF 15.2 HR
ISOTOPE HAVING HALF LIFE OF 4.3 HR
GAMMA ACTIVITY - PHOTONS PER SECOND PER GRAM
10^7
10^6
10^5
10^4
10^3
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
TIME - HOURS FROM END OF IRRADIATION
NOTE: SAMPLE IRRADIATED IN HOLE 22
AT AVERAGE POWER LEVEL OF
2400 KW FOR 15.66 HR
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CARBIDE AND CARBON CHEMICALS CORPORATION
DECAY CURVE OF CASEIN FOLLOWING
IRRADIATION IN ORNL PILE
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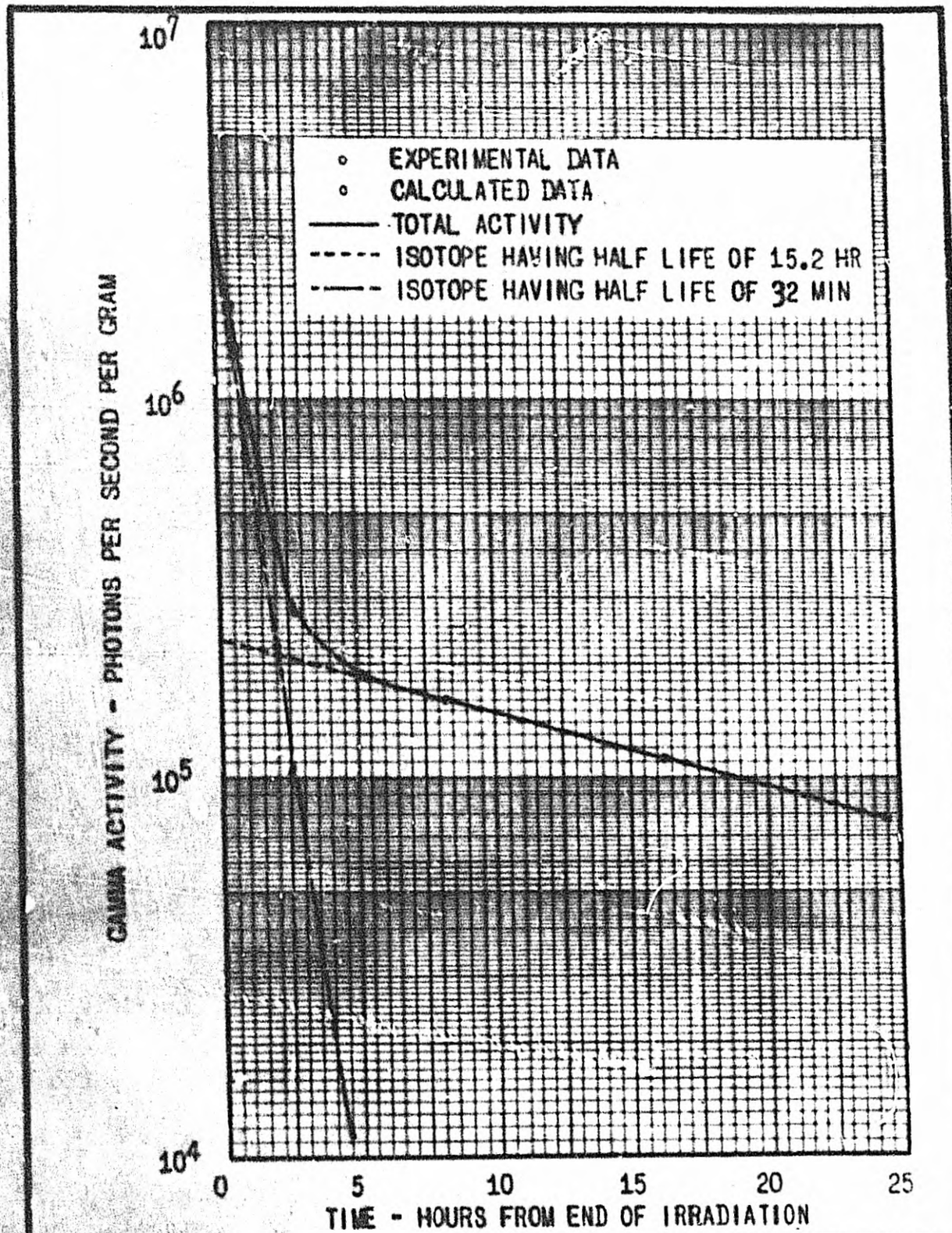

NOTE:
SAMPLE IRRADIATED IN HOLE 22
AT AVERAGE POWER LEVEL OF
.3675 KW FOR 63.75 HR

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DECAY CURVE FOR POLYSTYRENE
FOLLOWING IRRADIATION IN ORNL PILE

6-22-49	JRC	EPS-X53	8

B. Sample Calculations

Determination of quantity of parent isotope, Cl^{37}, in Saran sample, from activity of 37 min Cl^{38}.

Data: (from Notebook No. 1300 p 65; Notebook No. 1073, pp 16-23; power data from pile operations log, June 7 to June 20, 1949).

Weight of Saran sample	0.0668 g
Time in pile	8.12 hr
Avg. power level	3580 kw

For any parent isotope the production of radioactive isotope by neutron bombardment is given by the following expression:

Net production rate = gross production rate - decay rate

$$\frac{dN'}{dt} = q - \lambda N' \qquad (1)$$

where N = number of atoms of radioisotope at the time t

λ = decay constant of the radioisotope, $\ln 2$/half life

t = time of exposure to irradiation

Solving eqn. (1),

$$\frac{dN'}{q - N'} = dt$$

$$-\frac{1}{\lambda} \ln(q - \lambda N') = t + \text{const}$$

$$q - \lambda N' = Ce^{-\lambda t}.$$

When t is zero, N' is zero, therefore $C = q$, and

$$\frac{q - \lambda N'}{q} = e^{-\lambda t}$$

$$-\lambda N' = qe^{-\lambda t} - q$$

A_0, the activity of the radioactive isotope in disintegrations per second at zero time (time of removal of the sample from the pile) equals λN.

$$A_0 = \lambda N = q(1 - e^{-\lambda t}) \qquad (2)$$

q, the gross production rate, is given by the relationship,

$$q = n v \sigma N \qquad (3)$$

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where nv = neutron flux, neutrons/(sq cm)(sec).

σ = cross section for slow neutrons, of the stable isotope, barns (σ x 10^{-24} in sq cm),

N = number of atoms of parent isotope exposed to irradiation 6.03×10^{23} x weight/At.wt

Thus,

$$A_0 = nv\sigma\,(10)^{-24}\,\frac{N}{\text{At. wt}}\,(6.03 \times 10^{23})(1 - e^{-\lambda t}) \qquad (4)$$

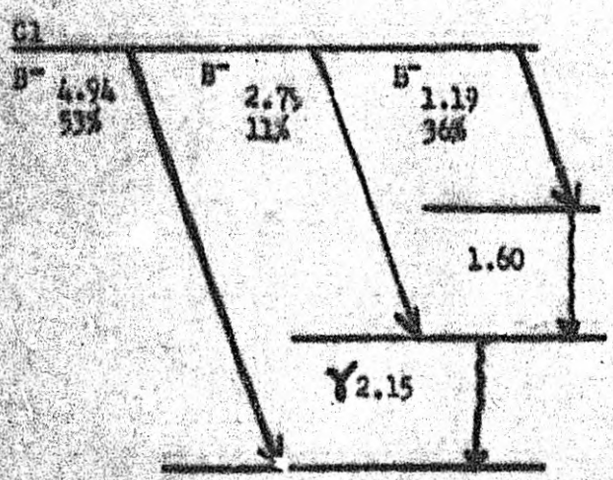
Rearranging,

$$N = \frac{N\,(\text{At. wt})}{(6.03 \times 10^{23})nv\;\;(10)^{-24}(1 - e^{-\;\;t})} \qquad (4a)$$

= weight of parent isotope.

A_0 is obtained from A_0' (observed activity at zero time) by employing corrections determined by the nature of the decay scheme of the radioisotope and by the efficiency of the counting instrument for the isotope.

Decay scheme for 37 min Cl^{38}: (6)



The decay scheme shows that per 100 disintegrations 36 gamma photons at 1.60 Mev and 47 at 2.15 Mev are emitted. Since there is a difference in energy of the gamma photons emitted by Cl^{38} from those emitted by the calibration standard, Co^{60}, correction of the readings of the ion counter must be made. The correction factor, obtained from a calibration curve prepared by Jones and Overman (3), is

$$\left[\frac{47}{(47+36)(1.3)} + \frac{36}{(83)(1.2)}\right] = 0.796$$

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Since there are 100 disintegrations per second for every 83 gammas,

$A_o = (0.796)(100/83)(A_o')$

$A_o' = 3.2 \times 10^7$ (Notebook No. 1073, p 23)

$A_o = 3.07 \times 10^7$ disintegrations per second.

Other values to be used in eqn. 4a are obtained from the reports indicated, as follows:

nv at the pneumatic tube ("rabbit" hole) is 0.57×10^{12} neutrons per sq cm per sec at a power level of 3600 kw, and varies in a linear manner with the power level (2)

$nv = 0.57 \times 10^{12}(3580/3600)$

$= 5.67 \times 10^{11}$ neutrons/(sq cm)(sec)

$\sigma = 0.56 \times 10^{-24}$ sq cm (or 0.56 barns) (6)

Use of this value for the cross section is based on the following considerations: 1) At the pneumatic tube, n is 0.72 neutrons/(cu cm)(watt) at a power level of 3600 kw (2). Therefore,

$v = 0.57 \times 10^{12}/(3.6 \times 10^6)(0.72)$

$= 2.2 \times 10^5$ cm/sec

$= 2200$ m/sec

This value of v corresponds to a neutron energy of 0.025 ev (6). 2) Two values, 0.4 and 0.56 ± 20% barns, are given (6) for the neutron absorption cross section of Cl^{37} at a neutron energy of 0.025 ev; 0.56 is the more recently determined value.

$\lambda = (0.693)(60)/37$

$= 1.12$ hr^{-1}

$e^{-t} = e^{-(1.12)(8.12)}$

$\cong 0$

Thus the weight of Cl^{37} in the Saran sample was

$$W = \frac{(3.07 \times 10^7)(37)}{(6.03 \times 10^{23})(5.67 \times 10^{11})(0.56 \times 10^{-24})(1)}$$

$= 5.94 \times 10^{-3}$ g

Since Cl^{37} occurs to the extent of 24.6% in chlorine (6),

Total chlorine $= 5.94 \times 10^{-3}/0.246$

$= 2.42 \times 10^{-2}$

Therefore the Saran contained (0.0242)(100)/0.0668 or 36.2% chlorine.

C. Literature Citations

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D. Acknowledgements

Appreciation is expressed for the advice and help given by O. Sisman throughout this investigation. Thanks are also extended to W. Kirkland and R. Towns for their aid in the taking of experimental data.

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