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IN-PILE GAMMA DOSIMETRY MEASUREMENTS WITH OXALIC ACID

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Introduction

The purpose of the work here reported was to measure γ dose rates in the NASA-Plum Brook Reactor. Measurements in an aqueous environment were required in an in-core location during reactor operation at full power of 60 Mw. and at 40 Mw. This meant that γ dose rates up to 4 w.g.$^{-1}$ would be encountered. The present study is the first in which chemical dosimeters have been used to directly measure γ dose rates in the PBR during operation at power.

Concentrated oxalic acid (H_2Ox) was chosen as the only chemical γ dosimeter both compatible with the lead-type capsule system to be used and capable of measuring the high γ dose rates present. Draganić et al[1-5] have pioneered and recommended the use of oxalic acid for γ dosimetry measurements in reactors and have reported[1] that measurements of total doses up to 1.6×10^8 rads with it are feasible. Thus at 4 w.g.$^{-1}$ an H_2Ox dosimeter sample can be irradiated for up to 6.7 minutes.

The stoichiometric decomposition of oxalic acid according to the simple equation,

$$H_2C_2O_4 \xrightarrow{\gamma} H_2 + 2CO_2 \tag{1}$$

does not actually take place under γ irradiation, although Marković and

Draganić[4] have recently reported that their data support the conclusion that the decomposition is a first order rate process. Instead the decomposition proceeds by a series of free radical reactions yielding less than two molecules of CO_2 per molecule of H_2Ox decomposed and variable amounts of H_2 depending on the irradiation conditions.[1] The presence of other carbonaceous (presumably polymeric) products in the irradiated dosimeter solutions is immediately evident from the deep yellow to brownish color of the solutions. CO is not produced in the decomposition and was not observed in the present work.

The decomposition yield from the γ irradiation of H_2Ox is reported[2] to be independent of the γ dose rate for dose rates up to 10^{10} rads sec.$^{-1}$. In addition the yield is independent of the sample temperature at least up to 80° C. and unaffected by impurities in the oxalic acid.[3] No significant change in the pH of concentrated H_2Ox occurs under irradiation, even to 90% decomposition, so this variable does not affect the radiation yield.

The irradiation facility used in this study allowed the sampling and gas chromatographic analysis of the off-gas from the dosimeter samples during irradiation. Hence the CO_2 production rate, as well as that of H_2 and O_2, could be measured as a function of time during irradiations of more than a few minutes duration. An important objective of the present work was to determine whether such CO_2 measurements could be used to determine constant (and varying) γ dose rates.

Experimental

The irradiations in this study were performed in the lead-type capsule irradiation facility constructed for PBR Experiment 64-06. The capsule is made of Zircaloy-2 and is situated in core piece LB-11 of the PBR. Fig. 1, a top view of the reactor core, shows this location. The capsule is mounted vertically in a "vertical adjustable facility tube" (VAFT), which allows its elevation to be adjusted as desired over a 9" range. Fig. 2 shows a side elevation view of the PBR core and indicates the vertical position of the capsule relative to the fueled region of the reactor with the VAFT full in and full out.

A simplified plumbing schematic of the 64-06 facility is shown in Fig. 3. All the plumbing apart from the capsule and the three 50 ft. long lines leading to it is located in a control panel on the lily pad above the reactor pressure vessel and is suitably shielded. Apart from aqueous samples inserted in it, the facility is always maintained helium-filled.

An air-free sample is loaded into the charging tank and from there is charged down into the capsule by a suitable helium pressure head. At the conclusion of the irradiation an increased pressure head is applied to force the sample back up into the receiving tank. From there the sample may be discharged to the hot drain or collected for later analysis. Except during sample transfer operations and special irradiations, a constant pressure of 120 psia. is maintained in the system by the back pressure regulator.

More detailed views of the capsule are shown in Fig. 4. As indicated, downward flowing primary coolant water is utilized to cool the outside of the capsule. The two chromel-alumel thermocouples measure the wall and sample temperatures. On charging an aqueous sample into the helium-filled capsule (situated at a fixed VAFT position) the temperature recorded by the sample thermocouple undergoes a sharp drop as the liquid cools the thermocouple junction. This decrease was used in the dosimeter irradiations to mark the beginning of each irradiation. Similarly, on discharging a sample from the capsule the temperature read by the sample thermocouple undergoes a rapid increase. This was used to mark the end of each irradiation. These times were easily determined to an accuracy of a few seconds.

A 40 ml. sample fills the capsule to about the extent shown in Fig. 4. During irradiation the sample can be sparged (as is indicated) with helium gas which is introduced through the bottom line at any desired rate. This rate is read on one of the inflow rotometers. When the gas chromatograph is used to analyze the off-gas from the sample being irradiated, the effluent gas stream is routed up the top capsule line, and the side line is closed off. Inside the 0.085" i.d. top line about 2" above the top of the capsule the effluent gas stream encounters the end of a 0.008" i.d. capillary tube (indicated in the bottom drawing of Fig. 4). A constant flow of gas from the effluent stream passes into and up the capillary tube and finally through the gas chromatograph sampling valve, as shown in Fig. 3. The constant flow rate through the capillary tube is determined by the 105 psi. pressure drop from the bottom end to the sampling valve. The remainder of the effluent gas flow passes on up the top capsule line, out of the system through the back pressure regulator, and through a flow transducer which accurately measures its mass flowrate.

When a gas chromatographic analysis is desired of the off-gas stream passing through the capillary tube, the sampling valve is turned 180°. This introduces a 0.5 cc. sample of the off-gas into 'he carrier gas stream leading to the absorption column of the chromatograph. A column filled with Porapak T and operated at 40° C. was used in this work to separate the H_2,

O_2, and CO_2 in the samples analyzed. The gas chromatograph employs a standard thermal conductivity detector to measure the separated gases as each comes off the end of the column, and the detector response is continuously recorded on a strip chart. Since the H_2 and O_2 in each sample come off the column rapidly while the CO_2 appears only after a considerable delay, several successive off-gas analyses can be initiated at constant time intervals and thus overlapped without obscuring any of the peaks recorded. A typical series of analyses overlapped in this manner is shown in Fig. 5. Here the initiation of each analysis is indicated by a low, broad pressure peak with a sharp leading edge. The sharp H_2 and O_2 peaks appear, in that order, within 1.5 minutes. Not until about 11 minutes after the start of the analysis does the CO_2 peak occur. This delay allowed a total of seven analyses to be started at 1.5 minute intervals before the first CO_2 peak appeared. Of course, then no more analyses could be initiated until all the CO_2 peaks had been recorded.

The intensity of each recorded gas chromatograph peak was measured by the peak amplitude. This was then compared with analyses of standard gas mixtures to obtain the absolute concentration of the gas in the sample analyzed. Several precautions were observed and minor corrections made in analyzing the data to assure the validity and accuracy of this approach. Since the helium flow rate through the sample under irradiation was always known, the percent composition of H_2, O_2, and CO_2 measured in the gas chromatograph analyses could be directly converted to absolute production rates of each of these gases in the dosimeter sample.

The dosimeter samples irradiated were 0.60 M H_2Ox solutions prepared from reagent grade $H_2Ox \cdot 2H_2O$ using deionized water. The concentration of H_2Ox remaining in each irradiated sample, as well as that in the starting solution, was determined by titration of a 10 ml. aliquot with CO_2-free, standardized NaOH (0.25 N). Prior to titration all possible dissolved CO_2 was removed from the sample by boiling for 30 minutes. Titration was done to a phenolphthalein endpoint and also followed with a pH meter in order to observe any anomalous behavior in the titration-pH curve. Apart from the reduced amount of H_2Ox present, the titration-pH curves obtained from the irradiated dosimeter samples were found to be indistinguishable from that of the starting solution. However, a slow downward drifting of the equilibrium pH was observed at pH's above ~6 during the titration of irradiated samples. This was attributed to the slow neutralization of weakly acidic groups in the high molecular weight products present in the irradiated samples, the same products responsible for the characteristic deep yellow to

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brownish color of the irradiated solutions. By performing the titrations in less than 5 minutes this effect was minimized and could be neglected.

Many of the irradiated samples also showed a distinct haziness and a few had a small amount of a fine yellowish precipitate. The samples also showed a small but definite amount of zirconium γ activity. These precipitation effects are most likely due to the formation of zirconium oxalates from the action of the H_2Ox on the zirconium in the capsule. Thus they may cause a slight loss of H_2Ox in the irradiated samples. However, the magnitude of and error due to these effects was estimated to be negligible.

Experimental details concerning the irradiation facility and the dosimeter irradiations performed in this study are summarized in Table I.

Results and Discussion

Draganić's[2] original empirical equation relating total γ dose to amount of H_2Ox decomposed in a dosimeter sample may be rewritten,

$$\log D = \log \bar{R\rho}'t = a \log C + b \tag{2}$$

where

D = total absorbed γ dose in w. min. ml.$^{-1}$,

R = γ dose rate in w.g.$^{-1}$, assumed constant during irradiation,

$\bar{\rho}'$ = average density at 25° C., in g. ml.$^{-1}$, of dosimeter sample during irradiation,

t = irradiation time in min.,

C = molarity of H_2Ox decomposed in sample during irradiation.

Values of the constants a and b for 0.60 M H_2Ox dosimeter samples have been recalculated in the above units from the values given by Draganić[2] and are,

$$\text{for } 0.004 < C < 0.158, \ a = 0.999, \ b = 1.530 \tag{2a}$$

$$\text{for } 0.158 < C < 0.531, \ a = 1.400, \ b = 1.850 \tag{2b}$$

The constants given in equation (2b) cover a decomposition range from ~26 to 89% of the initial 0.60 M H_2Ox concentration. The irradiations in this study produced decompositions of from 55 to 93%. Therefore equation (2)

with the constants given in equation (2b) was used to calculate the constant γ dose rate, R, for each test from the measured values of C and t and the correct value of $\bar{\rho}'$ obtained from tables.[6] C was of course determined from the titration of the irradiated sample. The results are presented in Fig. 6, in which the calculated γ heating rate R is plotted vs. the average height of each sample above the core horizontal midplane. A straight line has been drawn to fit the results obtained. Available PBR reference values for the γ heating in LB-11 are also shown in the figure, with their 20% error bars. These reference values were all calculated from data obtained in the PBR mock-up reactor.

All the values plotted in Fig. 6 are corrected to 60 Mw. reactor power (nominal full power) and to 24" rod bank height (the approximate height at mid-cycle). The reactor power correction was made by assuming the γ dose rate to be proportional to the power level. The corrections for rod bank height were made using correction factors calculated from γ heating values for a reactor location similar to LB-11. These values were available for rod bank heights of 16" (early cycle) and 28" (late cycle) and covered the whole vertical range of interest.

Fig. 6 shows that the γ dose rate values measured in this study, calculated as indicated above, are lower than but in fair agreement with the sparse PBR reference values available. The values obtained in this work show a nearly linear decrease with increasing height over the region studied. The slope and zero intercept of this linear relationship, however, are significantly different from those suggested by the PBR reference data.

Fig. 7 compares the line drawn to fit the γ dose rate values in Fig. 6 with temperature rise data obtained during earlier runs with water samples at 60 Mw. reactor power and 24" rod bank height. The temperature rise referred to is the difference between the readings of the wall and the sample thermocouples in the capsule. The temperature data which is plotted was taken at a high helium sparge rate to help ensure that turbulent heat-transfer conditions existed for all the measurements. Under these conditions the temperature rise should be approximately proportional to the heating rate, i.e., the γ dose rate. Fig. 7 shows this prediction fairly well borne out. The extrapolated zero intercept of the temperature rise curve is at ~18" above the core midplane, while the zero intercept of the γ heating line is at ~19".

In 1968, Marković and Draganić[4] proposed a different equation than equation (2) to relate the total γ dose to the extent of H_2Ox decomposition in H_2Ox dosimeter samples. This new relationship may be written as follows, using the same symbols as in equation (2),

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$$D = \overline{R\rho}'t = cC_0 \log \frac{C_0}{(C_0 - C)} \tag{3}$$

where

C_0 = initial H_2Ox molarity in dosimeter sample,

c = a constant = 66.7 w. min. mmol.$^{-1}$ (best value).

The new equation fits their data reasonably well and has the advantages (a) of describing the decomposition as a first order rate process and (b) of applying to any initial H_2Ox concentration, C_0, in the dosimeter sample. However, the validity of the new relationship is only claimed up to 60% decomposition of the H_2Ox, compared to 89% for the original equation, (2). Fig. 8 shows a comparison between relations (2) and (3) for values of $\overline{R\rho}'$ = 0.921 w. ml.$^{-1}$ and C_0 = 0.60 M. The percent decomposition of H_2Ox is plotted vs. the time of irradiation. For reference purposes the plots of both relations (2) and (3) are extended beyond the respective 89 and 60% decomposition limits given for them.

Since nearly all of the dosimeter samples irradiated in this study were decomposed to an extent greater than 60%, and many to ~90%, only relation (2), with the constants given in (2b), was used to calculate γ dose rates from the decomposition data. Satisfactory agreement was obtained between the dose rates calculated for samples irradiated to substantially different decomposition levels under similar test conditions. However, not enough such comparisons are available in these tests to definitely substantiate the validity of equation (2) and the constants given in (2b). Nor is such a verification provided by the general consistency shown in Figs. 6 and 7 between the γ dose rates from this work and both the available PBR reference data and the temperature rise measurements given.

The time derivative of relation (2) gives a prediction of the CO_2 generation rate as a function of time during the irradiation of 0.60 M H_2Ox. For a constant γ dose rate R, one can obtain the derivative expression

$$\log \dot{C}' = -\frac{a-1}{a} \log t + \frac{1}{a} \log \overline{R\rho}' - \log \frac{a}{k} - \frac{b}{a} \tag{4}$$

where

$\dot{C}'$ = mmols. CO_2 generated per min. per ml. in the sample (with the sample volume measured at 25° C.),

$\quad$ k = number of CO_2 molecules generated per molecule of H_2Ox
$\quad\quad$ decomposed.

The quantity k, which is just the ratio $\dot{C}'/\dot{C}$, may be a constant or may vary with C. Draganic[1] has reported it to have the constant value of 1.6 in air-free samples for H_2Ox concentrations > 0.025 M, but it is unclear whether this applies throughout irradiation or only initially.

The time derivative of relation (3) gives an alternate prediction of the CO_2 generation rate as a function of irradiation time, namely

$$\log \dot{C}' = -\frac{R\bar{\rho}'}{cC_0} t + \log R\bar{\rho}' - \log \frac{c}{2.303\,k} \tag{5}$$

in which all the symbols have been previously identified and again the γ dose rate R is taken as constant.

Four identical, lengthy irradiations of 0.60 M H_2Ox dosimeter samples were performed in which the CO_2 generation rate was determined at frequent intervals covering the whole irradiation time by the gas chromatographic technique described earlier. The γ dose rate during these irradiations, as calculated from the extent of H_2Ox decomposition using relation (2), was 0.91 ± 0.01 w.g.$^{-1}$. The recorded gas chromatographic response curve from one of these runs is reproduced in Fig. 5 and shows 12 separate CO_2 determinations. The CO_2 analyses from all the runs are plotted in Fig. 9, in which $\dot{C}'$, the CO_2 generation rate, is plotted as a function of the irradiation time.

Also indicated on Fig. 9 are the curves for $\dot{C}'$ predicted by the derivative relations (4) and (5), assuming a constant value of $k = 1.6$. These have both been displaced to the right on the abscissa by 2 minutes to account for the time required for a gas sample to travel from the dosimeter solution under irradiation up the capillary tube to the gas chromatograph sampling valve. The estimate of 2 minutes for this delay time was made from a knowledge of the gas generation rates, the helium sparge rate (~100 sccm.), the gas volume above the solution and below the end of the capillary tube (~12.5 cc.), the volume of the capillary tube and the expansion line at the top end of it (~2.8 cc.), the flow rate through the capillary tube (~36 sccm.), and the temperatures and pressures in the system.

The percent decomposition of the 0.60 M H_2Ox is indicated in Fig. 9 on the abscissa. This has been calculated from relation (2) and also includes the 2-minute gas sampling delay.

As seen in Fig. 9, the derivative expression (4), together with the constants given in (2a) and (2b), predicts the CO_2 generation rate $\dot{C}'$ will drop rapidly at about 26% H_2Ox decomposition from a previously constant value to a value ~70% of this, and then will continue to decrease at higher decomposition levels. Also as shown in the figure the derivative relation (5) predicts a continuous exponential decline of $\dot{C}'$ with increasing irradiation time. Again for comparison purposes this curve is carried beyond the 60% H_2Ox decomposition limit given for the validity of the parent relation (3).

From the rise of the experimental data at the beginning of the irradiations it is evident that a substantial reservoir mixing effect is included in the data, so that the gas samples analyzed were only approximately representative of gas mixtures actually in equilibrium with the solutions under irradiation. This effect is believed to occur principally in the gas volume immediately above the solutions in the capsule. Its magnitude in the runs plotted in Fig. 9 was reduced somewhat from that in previous runs by increasing the sample volume (to reduce the gas volume) and by increasing the helium sparge rate.

From Fig. 9 it will be noted that the maximum experimental values of $\dot{C}'$ approach within 10% the constant value predicted from (4) with the constants in (2a). Also, the curves drawn from (4) and (5) with $k = 1.6$ generally agree within ~20% with the experimental data after the initial rise of the measured $\dot{C}'$ values to their maximum. Taking account of the reservoir mixing effect mentioned above, the actual agreement in this region is even better than this for the portions of the two predicted curves lying below the experimental values. If the value of k is allowed to assume the theoretical maximum of two (see eq. (1)) near the maximum experimental values of $\dot{C}'$ and decrease slowly to about one for the values measured at the end of the irradiations, then the decrease in the experimental values of $\dot{C}'$ with t, including the estimated mixing effect, can be exactly fitted by relation (4) with the constants in (2b). Such a decrease in k with irradiation time is plausible in view of the build-up of high molecular weight products during irradiation. On the other hand, the data with the same mixing effect are also well fitted by equation (5) with a constant value of $k \approx 1.8$.

It will also be observed that a linear relationship between $\dot{C}'$ and t will empirically fit the decreasing $\dot{C}'$ values in Fig. 9 very well. Rather sparse gas chromatograph data obtained at other dose rates indicate that the slope of such linear fits is directly related but not proportional to the γ dose rate.

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Plotted in Figs. 10 and 11 are the measurements of $\dot{C}_{H_2}$ and $\dot{C}_{O_2}$ -- the generation rates of H_2 and O_2 -- made during the same four runs in which the CO_2 data in Fig. 9 were obtained. As in the case of the CO_2 measurements, $\dot{C}_{H_2}$ and $\dot{C}_{O_2}$ refer to the millimoles of H_2 or O_2 generated per minute per ml. in the sample (with the sample volume measured at 25° C.). The very low O_2 generation rate follows the pattern of the CO_2 generation rate quite closely, with the ratio of the O_2: CO_2 production rates $\approx 1.4 \times 10^{-3}$ at their maxima (t $\approx$7 min.) and $\approx 1.8 \times 10^{-3}$ at the end of the irradiations (t $\approx$32 min.). The H_2 generation rate, however, is inversely related to the CO_2 rate, the ratio of the H_2: CO_2 rates rising from $\sim$0.11 after 7 minutes irradiation time to $\sim$0.42 after 32 minutes.

A number of factors and effects of possible importance have been neglected in this study and should be mentioned. These include the local effects on the γ dose rate in LB-11 due to changes or anomalies in the fuel loading of the immediately adjacent fuel element LB-10 and due to the operation of the diagonally adjacent regulating and shim rods (the former being generally locked in a nearly full-out position). Also neglected was the contribution of other species than γ rays, in particular of recoil protons, to the radiation damage in the dosimeter samples. Similarly, the possible effect of sparging the dosimeter solutions with helium during irradiation has been neglected due to the relatively low sparge rates used. And, finally, the effect of the Zircaloy-2 capsule on the radiation environment in the dosimeter samples was disregarded. None of these effects is believed to have possibly been so large as to invalidate the results obtained in this study or the analysis made of them.

Conclusions

The major conclusions reached in this study are as follows:

--Concentrated oxalic acid can be reliably and readily used as a γ dosimeter in reactor environments with γ dose rates at least as high as 4 w.g.$^{-1}$.

--For 0.60 M H_2Ox dosimeter samples decomposed between 50 and 90%, the γ dose rates calculated from the original Draganić relationship (2) are consistent with each other, with the available PBR reference data for LB-11, and with temperature rise data obtained on similar samples.

--Determinations of γ dose rates by the decomposition of concentrated H_2Ox have excellent reproducibility.

--Titration with NaOH in a CO_2-free aqueous environment is a convenient, accurate, and simple analytical method for determining the H_2Ox

remaining in irradiated dosimeter samples.

--Gas chromatograph measurements of the CO_2 generation rate during irradiations of 0.60 M H_2Ox dosimeter samples may be usable for determining γ dose rates providing (a) the CO_2 generation rate is known over the whole course of several similar irradiations at accurately known γ dose rates and (b) enough measurements of the CO_2 generation rate can be made to at least determine the (constant) rate of decrease of the generation rate with time.

--The technique just described may be capable of extension at low γ dose rates (<1 w.g.$^{-1}$) to the measurement of several different, constant γ dose rates with the same concentrated H_2Ox dosimeter sample.

References

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2. I. Draganić: Nucleonics 21, 33-35 (1963).

3. I. Draganić, B. Radak, and V. Marković: Intern. J. Appl. Radiation Isotopes 16, 145-152 (1965).

4. V. Marković and I. Draganić: Radiation Res. 35, 587-595 (1968).

5. V. Marković and I. Draganić: Radiation Res. 36, 588-600 (1968).

6. Handbook of Chemistry and Physics, 44th edit.; (Chemical Rubber Publishing Co., 1962): p. 2052.

Table I: Experimental Details Concerning Irradiation Facility and

H_2Ox Dosimeter Irradiations

capsule material: Zircaloy-2.

capsule location: core piece LB-11.

capsule vertical position: adjustable over 9'' range by a vertical
adjustable facility tube.

average sample height range above core horizontal midplane for 40 ml.
sample: 6.25-15.25''.

dosimeter samples: 28-45 ml. 0.60 M H_2Ox (air-free).

extent of H_2Ox decomposition in irradiated samples: 55-93%.

irradiation time: 4-35 min.

capsule filling time: ~1 min.

capsule emptying time: <1 min.

reactor power level: 40 and 60 Mw.

γ dose rates measured: 0.8-3.3 w.g.$^{-1}$.

sample temperature during irradiation: 55-88° C.

pressure during irradiation: held constant at 120 psia.; supplied by helium.

sample sparging rate during irradiation: 55-110 sccm (helium).
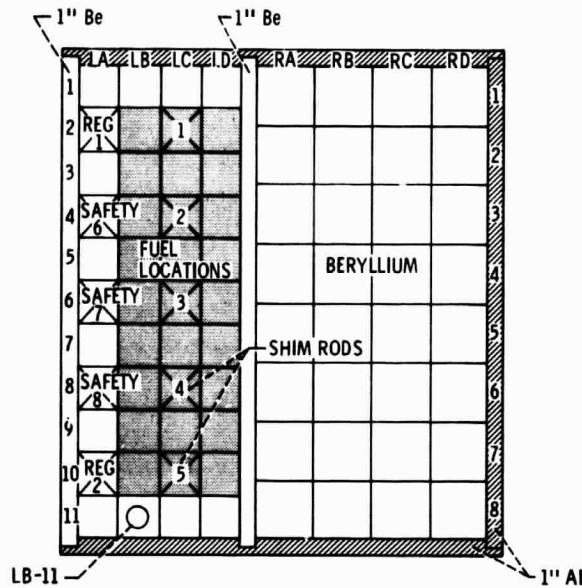
Figure 1. - Top plan view of Plum Brook reactor.
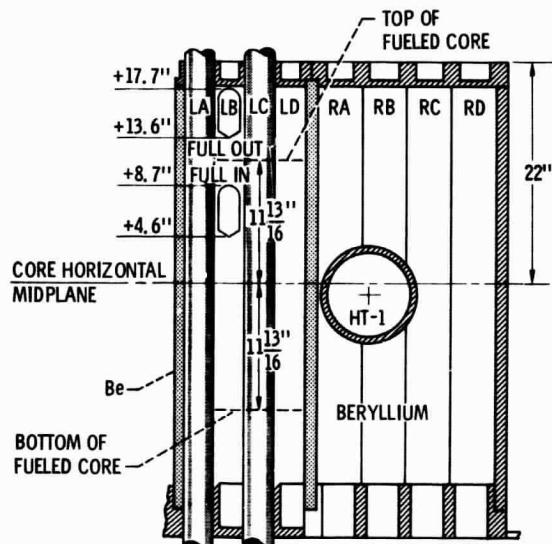
Figure 2. - Side view of Plum Brook reactor showing experiment 64-06 capsule elevations in LB-11.
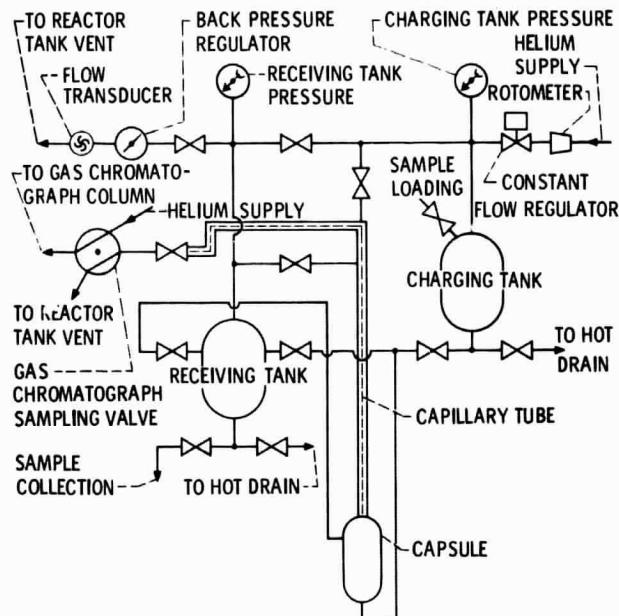
Figure 3. - Simplified plumbing schematic for experiment 64-06.
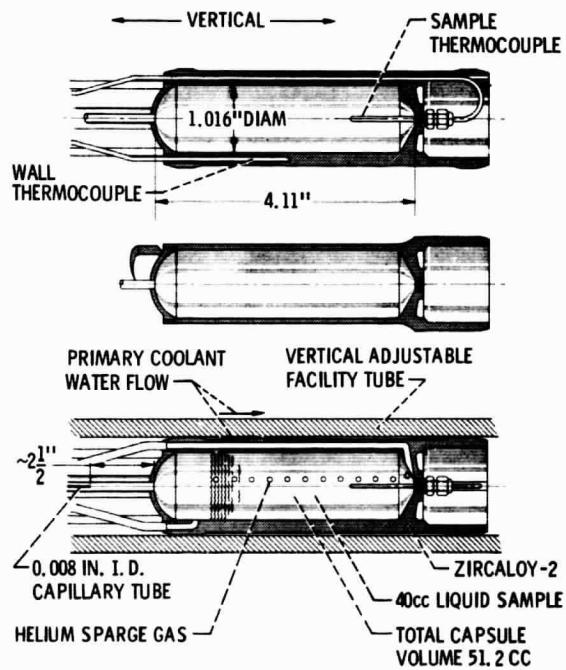
Figure 4. - Experiment 64-06 capsule. PBR core location LB-11.

RESPONSE

CO₂ PEAKS; X8

SAMPLE #26: 40 ML 0.6 M H_2Ox
REACTOR POWER 39.8 MW.
VAFT FULLY RETRACTED
CALCULATED γ HEATING: 0.91 W.G.$^{-1}$
He SPARGE RATE: 100 SCCM

CO₂ PEAKS, X8

H_2 AND O_2 PEAKS; X1

H_2 AND O_2 PEAKS; X1

TIME, 2 MIN

TIME ──▶
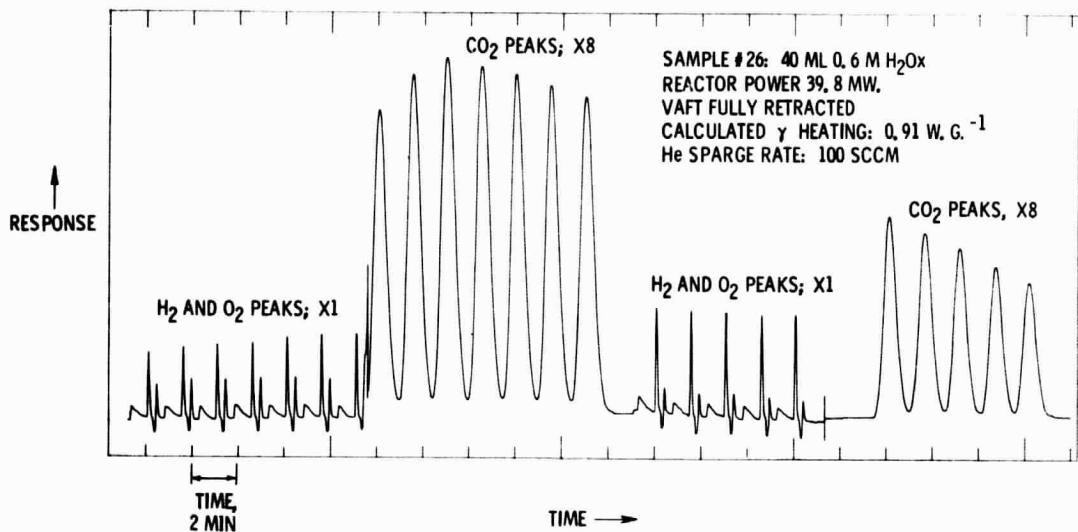
Figure 5. - Gas chromatograph recorder output.

CS-51126

ALL VALUES CORRECTED TO 60 MW AND 24 IN. ROD BANK HEIGHT

□ MEASURED VALUES
○ PBR REFERENCE VALUES CALCULATED
FROM MOCK-UP REACTOR DATA

γ
HEATING,
W.G.$^{-1}$

LINE FITTED TO
MEASURED VALUES

5 POINTS

DISTANCE ABOVE CORE MIDPLANE, IN.
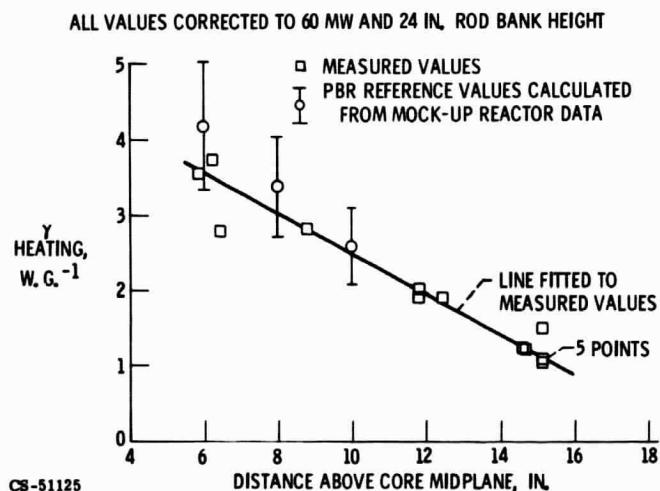
CS-51125

Figure 6. - Measured and reference γ heating vs distance above
core midplane.

ALL VALUES FOR 60 MW AND 24 IN. ROD BANK HEIGHT;
ΔT MEASUREMENTS AT HIGH He SPARGE RATE
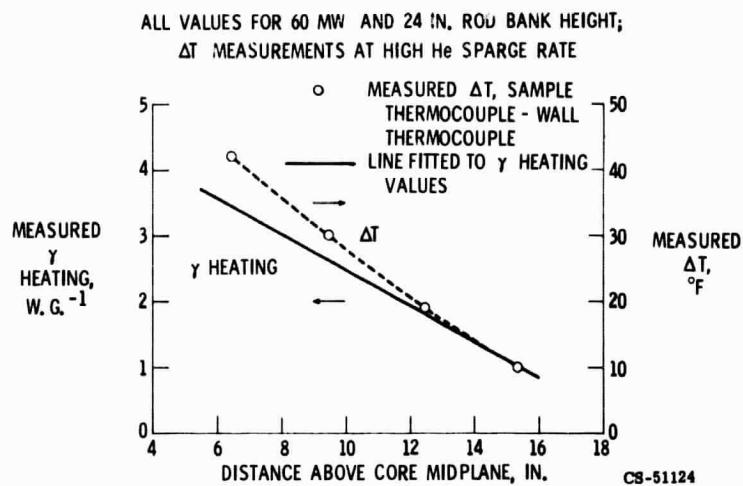
Figure 7. - γ heating and sample temperature rise measurements vs distance above core midplane.
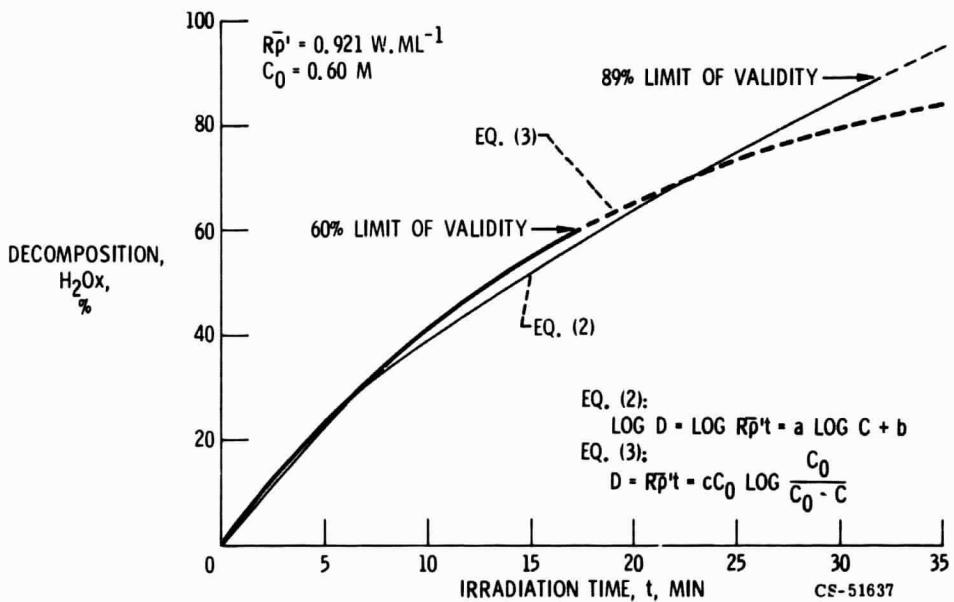

Figure 8. - Comparison of H_2Ox dose equations.
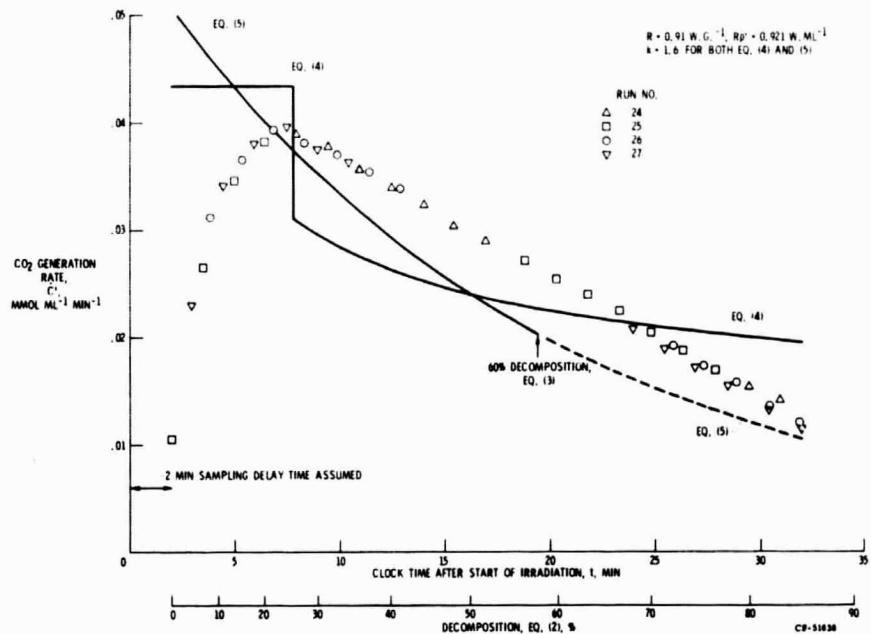
Figure 9. - CO_2 generation rate measurements.
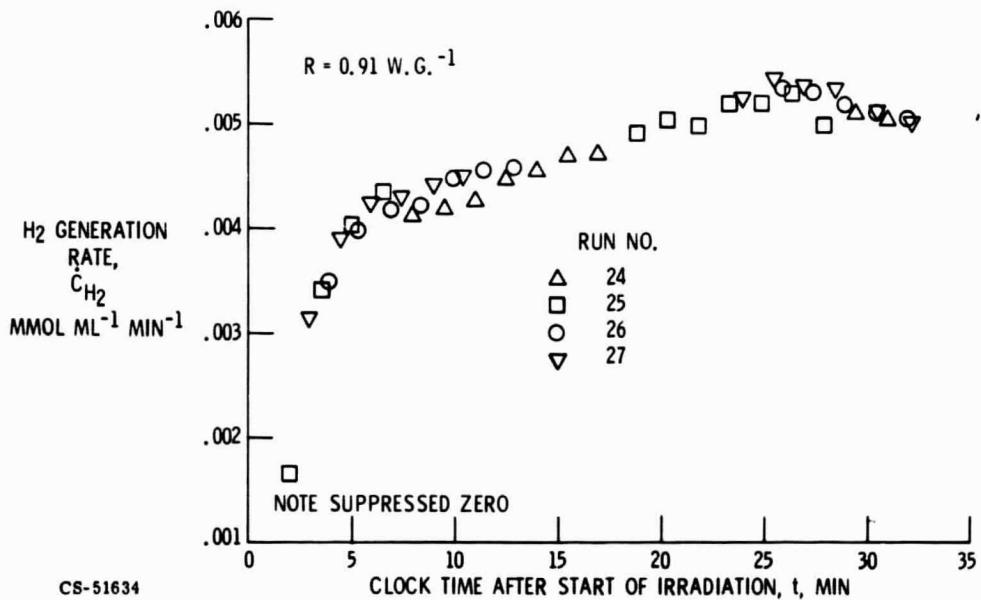
Figure 10. - H_2 generation rate measurements.
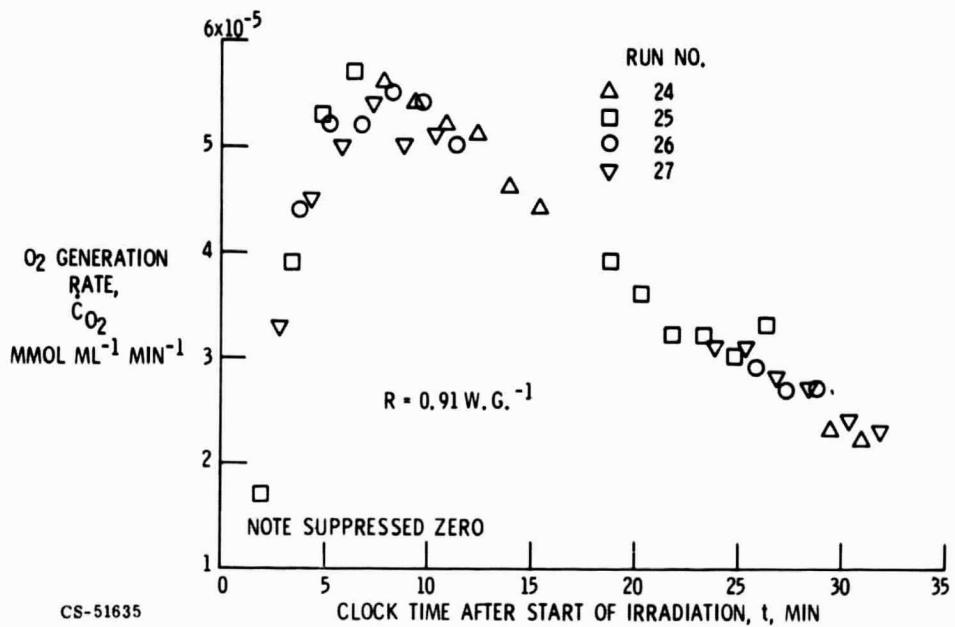
Figure 11. - O_2 generation rate measurements.