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THE RADIATION PROCESSING RESEARCH AND DEVELOPMENT PROGRAM
AT BROOKHAVEN NATIONAL LABORATORY

by

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For the purpose of fostering peacetime uses of atomic energy, the U.S. Atomic Energy Commission has established a broad effort on the use of nuclear energy for the production of chemicals in the Radiation Division of the Nuclear Engineering Department at Brookhaven National Laboratory (BNL). The program includes radiation chemistry research, engineering development and demonstration, and economic evaluation. By way of introducing the present activities in the BNL program, a review of the various ways in which nuclear energy can be used in the chemical process industry is first presented.

The generic term "chemonuclear" has been assigned to any chemical process system using nuclear energy as the prime energy source. The type of effect which induces the chemical change, or produces the chemical is of importance in classifying the type of chemonuclear process, or reactor involved. Four types of effects are identified for this classification. These are radiation, thermal, electrical, and photolytic effects. The radiation effects include the interaction with matter of high energy particles, or photons carrying energy in the range of a million electron volts (MEV) or higher, and causing primarily ionization effects. Thermal effects include transfer of thermal energy to matter and usually is directed towards producing a thermodynamic equilibrium in the system. Electrical effects are concerned with interaction with matter of

particles carrying energy in the range of electron volts up to thousands of electron volts (EV to KEV). The photolytic effects utilize photon energy in the range of infrared through the visible to the ultraviolet radiation (IR, Visible, and UV). As is evident, this classification is somewhat arbitrary since it is primarily based on a division of the energy spectrum which is in reality continuous. It does, however, separate the more conventional methods of inducing chemical reactions from the less conventional, and demonstrates the various techniques in the utilization of nuclear energy. All these techniques must be compared when selecting an economically competitive process. A further division based on direct and indirect use of the nuclear fission process is necessary to complete the general classification. Figures 1 to 4 illustrate generalized flow sheets for each of the types of chemonuclear reactors mentioned in the above classification.

Figure 1 shows the general manner in which radiochemonuclear reactors are used either directly or indirectly in a chemical process. The direct use of reactors for producing radiation effects includes the direct interaction of fission fragment recoil energy with matter. The reactor is generally constructed of thin sections of unclad fuel material. The advantages of the direct utilization of fission fragment recoil energy are; (a) a major fraction up to 84% of the fission energy can be utilized in this manner; (b) it is possible to produce extremely high temperatures, in the order of thousands of degrees Kelvin in the thermal spike for extremely short periods of time, in the order of millimicroseconds; and (c) it is possible to effect extremely rapid quench-down rates, in the order of billions of degrees Kelvin per second⁽¹⁾. The characteristics of this type of interaction coupled with its economic advantage makes it worthy of its continued

investigation on a research and development basis. One of the problems with fission fragment chemonuclear reactors is the decontamination requirement for production of an acceptable product.

The neutron-gamma chemonuclear reactor utilizes clad nuclear fuel, and the chemical effects are derived from prompt and decay neutron, gamma, and beta energy in the reactor. The main advantages are that the reactor technology is similar to those developed for power purposes, and the contamination problem is much less severe. The main disadvantage is that a minor fraction, less than 16% of the fission energy is available for this process.

Fission fragment reactors are generally indicated for endothermic and non-chain processes, while neutron-gamma reactors are indicated for exothermic and chain reactions. The power requirement per unit production rate is as follows:

$$P_T = \frac{101.5}{GE_O M}$$

where:

P_T = power requirement in MW per ton per day of product,

G = G-value of the reaction in molecules/100 ev,

E_O = energy deposition efficiency, and

M = molecular weight of the product.

Generally endothermic and non-chain reactions have G-values ranging below 100, while exothermic and chain reactions are usually above this value and even range into the millions. Fission fragment reactors can probably be designed to operate on energy deposition efficiencies of 50% or better, while n- γ reactors have efficiencies in the range of 2 to 3%. Figure 5, thus shows that for these generalized conditions, fission fragment reactors are indicated in capacities of hundreds of kilowatts or more per ton/day for low G-value reactions,

and n- γ reactors for high G-value reactions. This generalization does not exclude the consideration of n- γ reactors for low G-value reactions, if the economics of the system prove attractive.

For the indirect use of the radiochemonuclear system shown in Figure 1, spent fuel, separated fission products, or radioactive isotopes produced in a fission reactor can be utilized outside of a nuclear reactor in an irradiator device. The cobalt-60 isotope irradiator is the best known of the irradiators usually considered. Radiation effects can also be produced by high voltage machines which can be powered by a nuclear power reactor and is thus, also indicated as an indirect system.

Figure 2 indicates the use of thermochemonuclear reactors based on thermal effects. The direct system utilizes a nuclear reactor which is capable of generating temperatures sufficient for inducing thermochemical reactions, usually up to several thousand degrees Kelvin. Clad elements can be used to minimize contamination problems, however, high temperature material problems limit this concept for higher temperature processing. The indirect system includes a process steam or gas reactor for indirect transfer of thermal energy from the reactor through heat exchangers, or the fission fragment generation of endothermic chemicals, which can be combined outside of a reactor in a burner for release of thermal energy which would provide the thermal energy for use in a chemical process.

Figure 3 indicates the electrochemonuclear systems based on electrical effects. The direct system utilizes a fission fragment, or high temperature reactor for generation of chemicals which can be recombined in a fuel cell for power generation, or for chemical production. Direct thermionic and fission electric effects can be included here. In the indirect system nuclear power reactors through

conventional or unconventional power cycles are utilized to generate power for electrolytic and electric discharge cells.

Figure 4 indicates similar generalized flow sheets based on photo effects. The direct system generates light energy by direct nuclear⁽²⁾ interactions in matter, while the indirect system utilizes power reactors for producing IR, UV, and Visible light by means of conventional photocells.

The BNL radiation processing program is concerned with research and development of radiochemonuclear reactors, and the economic comparison of these with the other more developed systems mentioned above; i.e., the direct use of fission fragment energy for chemical processing, and the indirect use of nuclear energy in the forms of isotopes for the same purpose.

The fission fragment studies include; (a) an experimental program for the determination of the fission fragment energy deposition efficiency of various thin sectioned fuel elements, and the evaluation of G-values in static capsule tests for certain chemical reactions, such as the formation of NO_2 from N_2 and O_2 and CO from CO_2 ; (b) the development and fabrication of fission fragment sources and fuel elements; (c) the design and construction of a chemonuclear in-pile loop facility; and (d) the design and evaluation of various chemonuclear reactor and process concepts.

In the in-pile static capsule experiments, two-dimensional uranium-bearing materials are placed in quartz or aluminum containers filled with either nitrous oxide (N_2O) for dosimetry purposes, or with $\text{N}_2\text{-O}_2$ gas mixtures for NO_2 formation, or with CO_2 for CO formation. The uranium-bearing materials are either on ordered array of 3 micron glass fibers containing up to 40 w/o U-235 (93.5% enriched),

foils of 27 w/o U-235 alloy of aluminum, or foils of 20 w/o U-235, alloy of palladium in thicknesses as small as 2 microns. The experimental elements are held in quartz rings, as shown in Figure 6. The rings are then inserted in the capsule containers filled with the appropriate gas and are then placed in a neutron field inside the BNL reactor.

The nitrous oxide gas dosimeter is based on the decomposition of N_2O to NO_2 , N_2 , and O_2 . The decomposition of N_2O has been found to be independent of the type of radiation⁽³⁾. Based on ferrous sulfate dosimetry in a Co-60 gamma field, the $G(NO_2)$ value has been determined to be 3.8 and to be independent of absorbed dose. The $G(N_2)$ value has been found to be dose dependent according to the following equation:⁽⁴⁾

$$G(N_2O \rightarrow N_2) = 24.1 / A^{0.06}$$

The $G(-N_2O)$ value for decomposition of N_2O is thus, also dose dependent. The NO_2 , N_2 , and O_2 were analyzed by partial pressure measurements in a vacuum system, and the N_2 was further analyzed by gas chromatography. At a pressure of 3 to 4 atmospheres, the average range of the fission fragment in N_2O under these conditions is about 0.4 to 0.5 cm. The range in the glass fiber and the aluminum alloy is about 13.7 microns, while in the palladium alloy the range is about 6 to 7 microns. There are three modes of energy deposition in the in-pile capsule experiments; (1) fission fragment energy from uranium-bearing source; (2) the fast neutron and gamma pile background field; and (3) the energy due to neutron absorption reaction in $N^{14}(n,p)C^{14}$. The sum of the neutron-gamma reactor background radiation, and the n,p reaction on the nitrogen is estimated by making N_2O dosimetry measurements in-pile without the uranium source. The neutron-gamma reactor background radiation alone is estimated by making a measurement using the radiation decomposition of CO_2 inhibited

with small amounts of NO_2 ; the $G(-\text{CO}_2)$ value is about $4.0^{(5)}$. At lower pressures the n,γ and n,p reaction energy is insignificant, however, at higher pressures the contribution of this energy source becomes appreciable due to the increased mass of the reactant gas.

In figure 7, the fission fragment energy deposition efficiency is given as a function of the weight loading of U-235 in the exposed fiber⁽⁶⁾. The weight loading was achieved by varying the number of strands of fiber per unit area. The fixed array of fibers approximates a two-dimensional element because of the random close packed spacing of the strands in a planar dimension. The figure shows that a deposition efficiency of up to 50%, based on fission fragment energy, has been obtained with decreasing weight loading.

Figure 8 shows a few results with the 27 w/o U-Al metal alloy foils compared to a number of theoretical calculations using several different geometrical models. It is noted that the efficiency of a two-dimensional plate element is a function of the ratio of the plate thickness and the range of the average fission fragment in the material $(t/R)^{(7)}$. The experimental efficiency values obtained for the foils fall between the model assuming a constant G-value and linear and quadratic energy deposition along the path of the fission fragment. A maximum experimental value of about 40% was obtained with an element having a thickness of 6.3 microns.

In the formation of NO_2 and N_2O from N_2 and O_2 using the above fission sources a $G(\text{NO}_2)$ value of 1.2, and a $G(\text{N}_2\text{O})$ value of approximately 1.0 are the highest values obtained to date at about 3 atmospheres pressure. Attempts are being made to obtain G-formation values as a function of pressure, temperature, and foil thickness.

Because of the rather limited amount of material and restricted conditions imposed in capsule experiments, in order to obtain meaningful information, it was deemed advisable to construct an in-pile chemonuclear loop facility. The scope of the loop⁽⁸⁾ is to; (1) study fission fragment chemistry in a flowing gas system at pressures up to 1000 psig, temperatures up to 1000°F, and gas velocities up to 30 ft/sec; (2) measure the efficiency of fixed fission fragment source and fuel material as a source of fission fragment energy and to determine its stability under the conditions mentioned above; and (3) investigate the extent of the contamination problem and test the efficiency of certain types of equipment as decontamination devices. Figure 9 shows a flow sheet and Figure 10 a schematic of the design of the loop which will fit straight through two experimental holes in the Brookhaven Graphite Research Reactor. It will be possible to withdraw and replace fuel packages through a closure on one side of the in-pile section. The loop is designed for a maximum loading of 50 gm of enriched uranium which should generate about 5 KW of fission power. However, this maximum loading will be approached in a stepwise fashion starting with no loading, commencing with a study of the n, p and γ reactions. Gas circulation will be achieved by sealed gas bearing compressors capable of circulating 170 SCFM at a 15 psi pressure differential.. Continuous in-line gas analysis will be achieved by means of a process gas chromatograph, a mass spectrometer, and an infrared analyzer. It should also be possible to separate significant quantities of condensible product in a refrigerated by-pass line.

The development of chemonuclear source and fuel material has proceeded along the lines of attempting the fabrication of thin fuel elements having the required high temperature oxidation resistance

and mechanical stability. After some investigation an alloy consisting of a solid solution of uranium in palladium, containing up to 30 w/o uranium, has proved to be ductile enough to be rolled on a Sendzimir Mill to thicknesses of less than 3 microns. Much technique is needed to accomplish this, however, once achieved the Mill is capable of rolling long lengths of this material on a production basis. Tests in heated air stream up to 1000°F and 30 ft/sec have indicated that a very thin oxide film is formed on the surface of the foil and the foil loses some of its ductility, however, the foil generally appeared quite stable. In-pile burnup tests are in progress for determination of radiation damage, as it affects the strength of the material and mass transport from the material. Various configurations of packed sections with spacing between foils equivalent to the range of the fission fragment are being aerodynamically tested in high temperature high velocity air streams. A theoretical study is being initiated to determine the strength of material required to produce the most stable packed configuration under the flow conditions required. Both ordered and random arrays for a critical core loading are under consideration.

Following complete design and economic analysis of a complete chemonuclear system for the production of fixed nitrogen⁽⁹⁾ additional studies have been made on chemonuclear systems for the production of CO from CO₂. A thin foil fueled externally moderated core design⁽¹⁰⁾ has indicated that a 19 ft. diameter core at 500 psig would produce CO equivalent to 1220 tons per day of fixed nitrogen. Unique dual-purpose two-region core designs have indicated some advantages in providing for independence of chemical and power production, however, under the conditions chosen, these systems suffer from relatively poor neutron coupling⁽¹¹⁾. A Chemonuclear Workshop

attended by a number of industry, university, and government agencies, has indicated that a basic research program concurrent with a generalized development program is indicated for advancing the technology and for further evaluating the usefulness of fission fragment chemonuclear reactors.

The studies on the indirect use of nuclear energy for the production of chemicals include; (a) an extensive investigation of the homo-polymerization of ethylene by Cobalt-60 gamma radiation, (b) a survey program on the copolymerization of ethylene with a number of other monomers, (c) a survey program of a number of promising exothermic systems induced by Cobalt-60 gamma radiation, and (d) the development of the fundamentals and demonstration of the technology of isotope radiation engineering applied to chemical processing.

The yield and rate of polymerization of ethylene in a Cobalt-60 gamma field was determined by a continuous pressure recording technique. The conditions under which the rate was measured varied in pressure from approximately 680 atm to less than 68 atm, in temperature from -78°C to 50°C , and in intensity from 17,000 to 920,000 rad/hr. Polymerization takes place in the liquid phase at -78°C at a very low rate. In the gas phase at 20° to 40°C a strong inhibition and retardation effect due to oxygen impurity was noted, and the induction period as oxygen concentration varied was determined. The experiments indicate that the effect of the presence of polymer on the polymerization rate is negligible when compared with the effect of either oxygen or ethylene pressure. The addition of argon acts as a diluent in decreasing the polymerization rate. For a series of runs using high purity gas containing < 5 ppm O_2 , the differential G values based on monomer alone were determined during the falling pressure period and

equations are derived to express the G-value and the polymerization rate as a function of ethylene pressure and radiation intensity. In the range of 150 to 600 atm pressure, 17,000 to 920,000 rad/hr intensity, and temperature in the range of 30 to 50°C, the G-value is proportional to the 0.82 power of the pressure, and to the -0.58 power of the intensity. At 335,000 rad/hr a maximum differential G-value of 37,200 was obtained at 651 atm, while at 17,000 rad/hr a maximum value of 136,000 was reached at 555 atm. These correspond to polymer formation rates of 165 and 29 gm/liter/hr, respectively. A rate expression was derived from the experimental data, as shown in Figure 11. The rate equation in terms of the formation rate Q in gm/liter/hr, pressure P in atmospheres, and the radiation intensity I in rad/hr is as follows: (12)

$$Q = 6.26 \times 10^{-4} P^{1.02} I^{0.42}$$

In experiments performed with gas containing oxygen impurity, additions of solid substrates of alumina and molecular sieves increase the G-value by a factor of 2 to 3 and the rate by a factor of 1.5 to 2 which indicates a surface effect.

The polymers produced throughout these experiments consisted of fine white powder with properties dependent on pressure and irradiation conditions. Generally, the polymers show intermediate density values of 0.93 to 0.94 having a high degree of crystallinity after annealing, and at lower total doses are non-crosslinked. Further rate data and polymer characterization awaits an experimental study at constant pressure conditions in static and flowing systems.

An extensive series of exploratory experiments have been made on the random copolymerization of ethylene gas with a number of liquid and gaseous monomers by Cobalt-60 gamma radiation. This

investigation is primarily directed toward the identification of the copolymers and homopolymers formed in a mixture of the monomers. A mixture of a number of proportions of monomer with ethylene gas pressurized up to 10,000 psi is irradiated in a Co-60 field and the pressure allowed to fall during polymerization. Evaluation of the polymer is made on the basis of composition, infrared spectral analysis of cast films, preferential solubility and fractional precipitative methods, crystalline melting point, density, melt-index and molecular weight measurements by osmotic pressure, light scattering, and intrinsic viscosity techniques. Positive identification of a copolymer of ethylene with most of the monomers listed in Table 1 have been obtained⁽¹³⁾. It has not been possible, as yet, to establish identification of a copolymer of ethylene with butadiene or styrene.

Figure 12 shows the IR Spectra for a sample of a radiation produced polyethylene and for a copolymer of ethylene with CO showing a definite change in structure of the original homopolymer.

An interesting preliminary observation has been observed in the system, ethylene-chlorotrifluoroethylene (CTFE). When this system is irradiated in the proportion of approximately 7 parts of ethylene gas at 9600 psi to 1 part of liquid CTFE at 20°C, after a total dose of approximately 50,000 rads at a dose rate of 308,000 rad/hr, the system explodes violently with the formation of carbon. Some white polymer powder is also found after the explosion. Under similar conditions but at a lower dose rate of 230,000 rad/hr, the system polymerizes normally with no evidence of an explosive reaction. This would indicate that a threshold radiation intensity exists for this system, which initiates a runaway polymerization leading to a thermal decomposition and explosion in the ethylene gas phase. Further study is necessary for an identification of the activating species

TABLE 1

COBALT-60 GAMMA COPOLYMERIZATION OF ETHYLENE

Copolymer Characteristics

Run No.	Ethylene Copolymer	Initial Mole Ratio <u>Ethylene Comonomer</u>	Final Mole Ratio <u>Polyethylene Copolymer</u>	Density gms/cc	Crystalline Melting Point °C
1	Styrene	7.4	-	1.000	Non Cryst.
2	Divinyl benzene	21.2	-	-	-
3	Methyl methacrylate	17.5	3.6	0.956	125
4	Methyl acrylate	31.6	2.2	0.955	123-126.5
5	Vinyl acetate	5.5	7.1	0.963	105-106.5
6	Allyl acetate	6.5	93.2	0.939	122
7	Isobutylene	8.98	-	0.908	103-107.5
8	Trans 2-butene	8.2	-	0.954	103-107.5
9	Cis 2-butene	6.7	-	0.963	125-126
10	1-butene	10.8	-	0.912	102
11	Isoprene	18.7	-	0.956	122
12	Propylene	5.5	-	0.910	95
13	Acrylonitrile	3.8	0.24	1.120	Non Cryst.
14	Chlorotrifluoroethylene	27.8	13.2	1.004	122
15	Vinyl chloride	7.3	1.3	-	Non Cryst.
16	Carbon monoxide	7.9	1.95	1.090	112.5
17	Vinyl pyrrolidone	14.4	27.7	1.008	120.5
18	Methyl vinyl ketone	10.9	51.7	0.942	125

involved in this violent decomposition which takes place in a radiation field.

A survey program is under consideration on inducing exothermic reactions by Co-60 radiation. This includes; (a) partial oxidation reactions, e.g., ammonia oxidation for nitric acid and benzene oxidation for phenol production; (b) metal deposition reactions, e.g., from metal carbonyls, and organo-metallic compounds; and (c) radio-catalytically induced reactions. In the latter case, it has been found that a purified sodium alumino-silicate molecular sieve is capable of increasing the G-value for hydrazine formation from ammonia adsorbed on the sieve, by a factor of 10 and the ratio by a factor of 3 over that obtained by irradiating pure liquid ammonia⁽¹⁴⁾.

It is generally realized that the use of isotopic radiation sources awaits the development of the technology and demonstration of its use in a chemical process system. For the purpose of developing this much needed radiation engineering technology, BNL has designed a High Pressure Gamma Loop Facility⁽¹⁵⁾. The loop will consist of a stirred 12" diameter by 3'6" long reactor as shown in Figure 14, to contain up to 30,000 curies of Co-60, circulator, heat exchangers for temperature adjustment, and an analytical system as shown in Figure 13. The loop is primarily designed to handle the polymerization of ethylene at pressures up to 10,000 psig, at a circulation rate of 1740 lbs/hr of ethylene and polymer production at the rate of 15 lbs/hr. The facility, however, is designed to be versatile so that equipment changes can be readily made allowing the study of a variety of radiation-induced process systems. By designing the facility for handling high pressure ethylene, a maximum degree of safety precautions is primarily established. Much thought has been given to the safety aspect of this facility. Figure 15 indicates the arrangement of the equipment,

and the containment building needed to prevent spread of cobalt activity and to reduce the hazard due to possible combustion and explosion reactions. With this facility it should be possible to demonstrate a continuous radiation process under controlled conditions, and to produce sufficient quantities of material to carry out proper physical, chemical, and commercial tests and evaluations.

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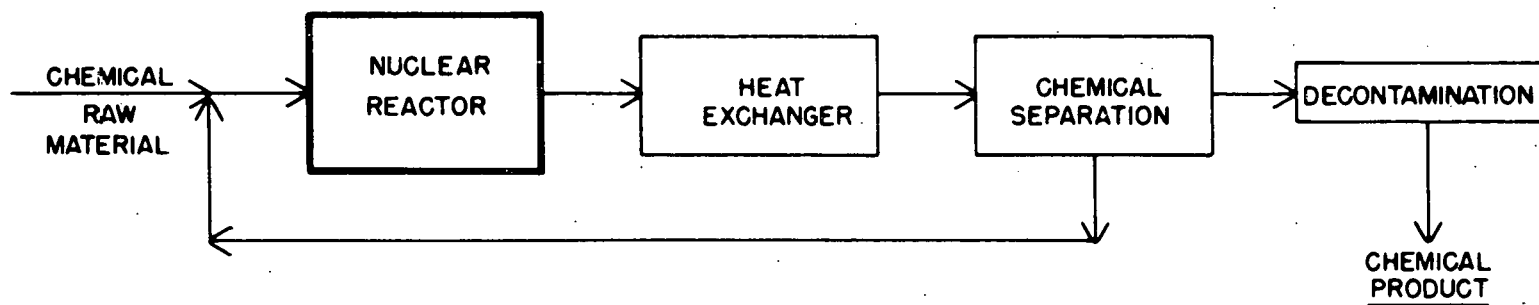
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I. RADIOCHEMONUCLEAR REACTORS

A. DIRECT USE OF REACTOR

1. FISSION FRAGMENT
2. NEUTRON GAMMA



B. INDIRECT USE OF REACTOR

1. FUEL, F.P. OR ISOTOPE IRRADIATOR
2. HIGH-VOLTAGE MACHINE

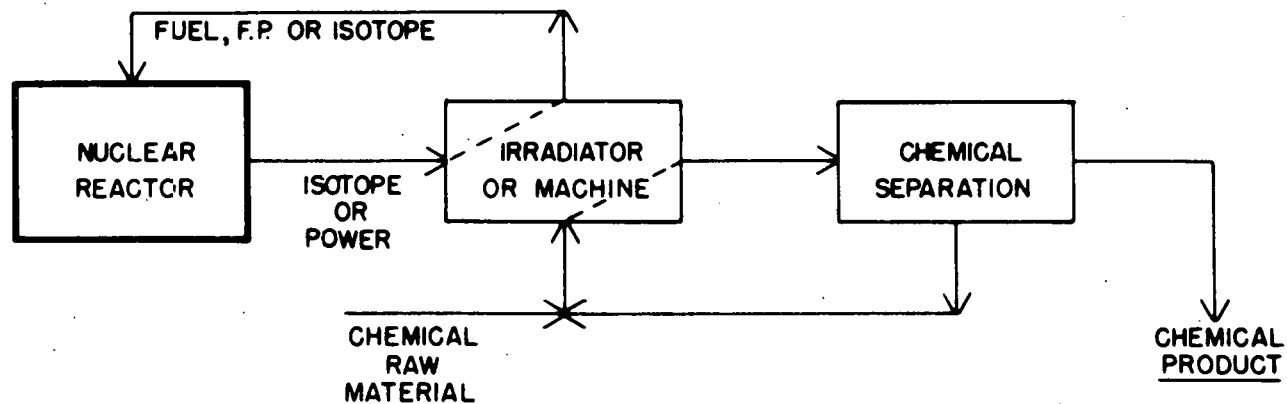
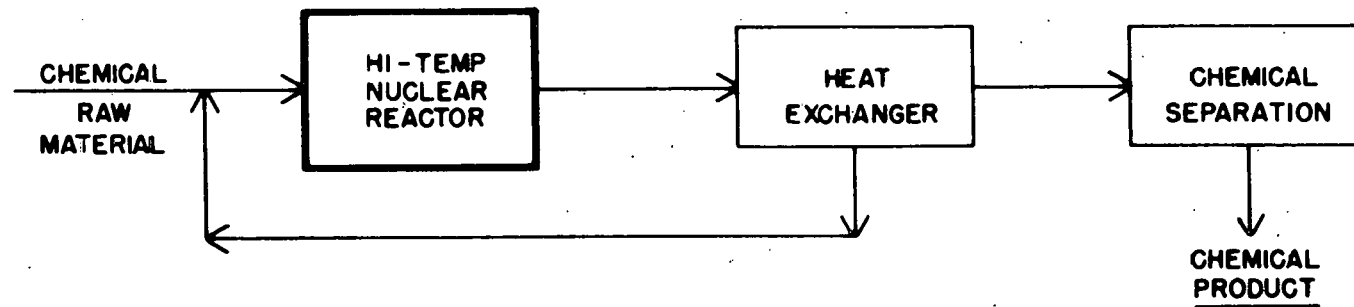


FIG. I

II. THERMOCHEMONUCLEAR REACTORS

A. DIRECT USE OF REACTOR

1. HIGH TEMPERATURE



B. INDIRECT USE OF REACTOR

1. PROCESS STEAM OR GAS

2. F.F. REACTOR AND CHEMICAL BURNER

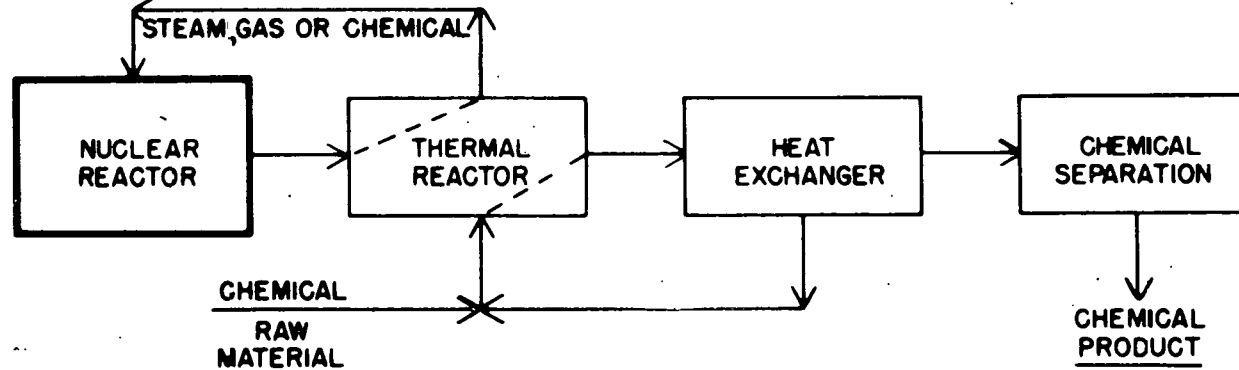
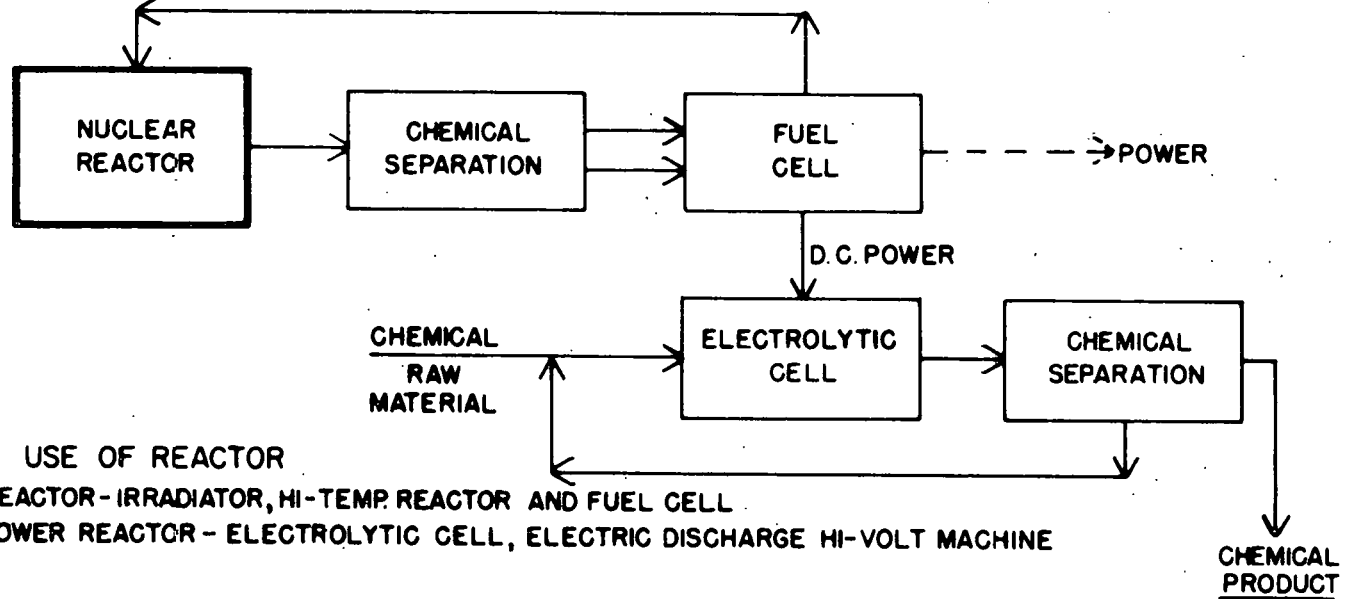


FIG. 2

III. ELECTROCHEMONUCLEAR REACTORS

A. DIRECT USE OF REACTOR

1. F.F. OR HI-TEMP AND FUEL CELL
2. THERMIONIC AND FISSION ELECTRIC



B. INDIRECT USE OF REACTOR

1. REACTOR-IRRADIATOR, HI-TEMP REACTOR AND FUEL CELL
2. POWER REACTOR - ELECTROLYTIC CELL, ELECTRIC DISCHARGE HI-VOLT MACHINE

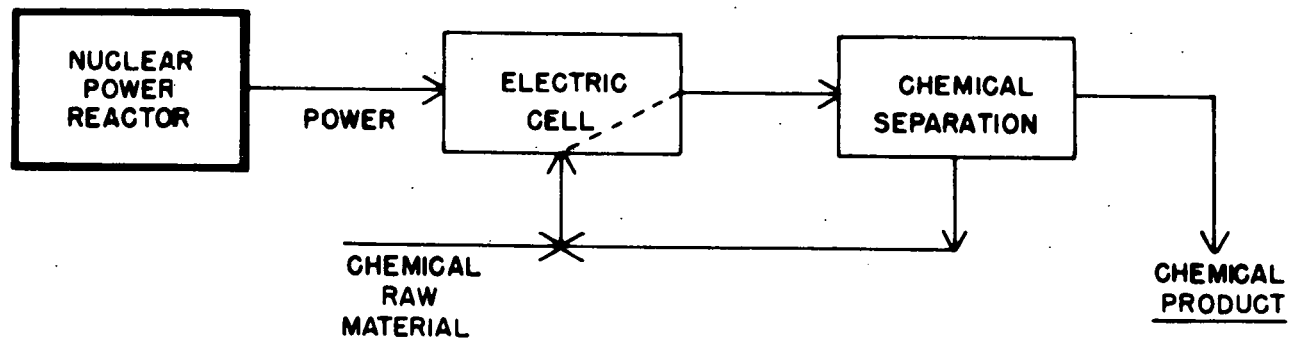
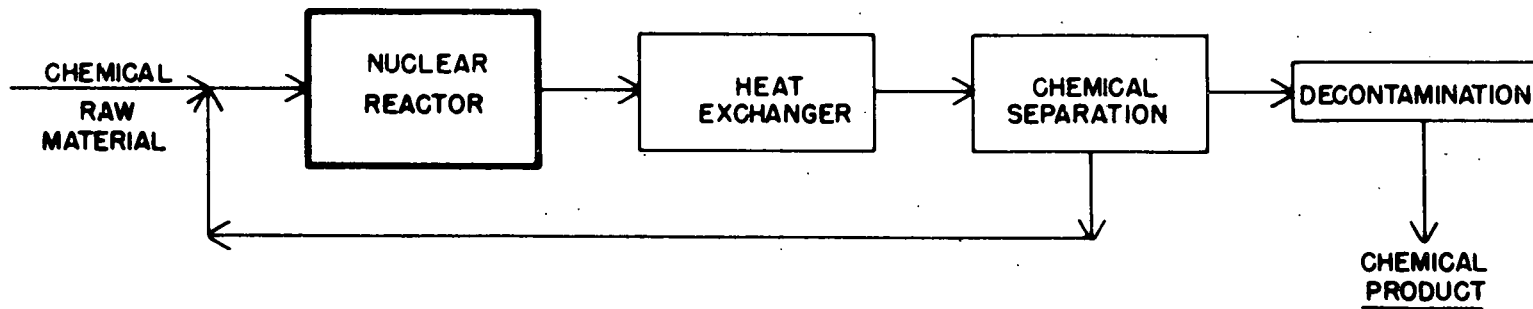


FIG. 3

IV. PHOTOCHEMONUCLEAR REACTORS

A. DIRECT USE OF REACTOR

I. NUCLEAR PHOTO EFFECT



B. INDIRECT USE OF REACTOR

I. PHOTOCELL

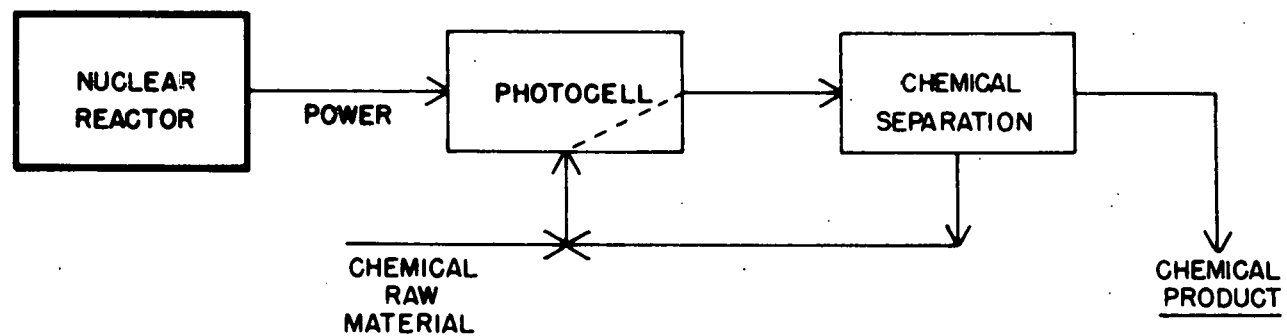


FIG. 4

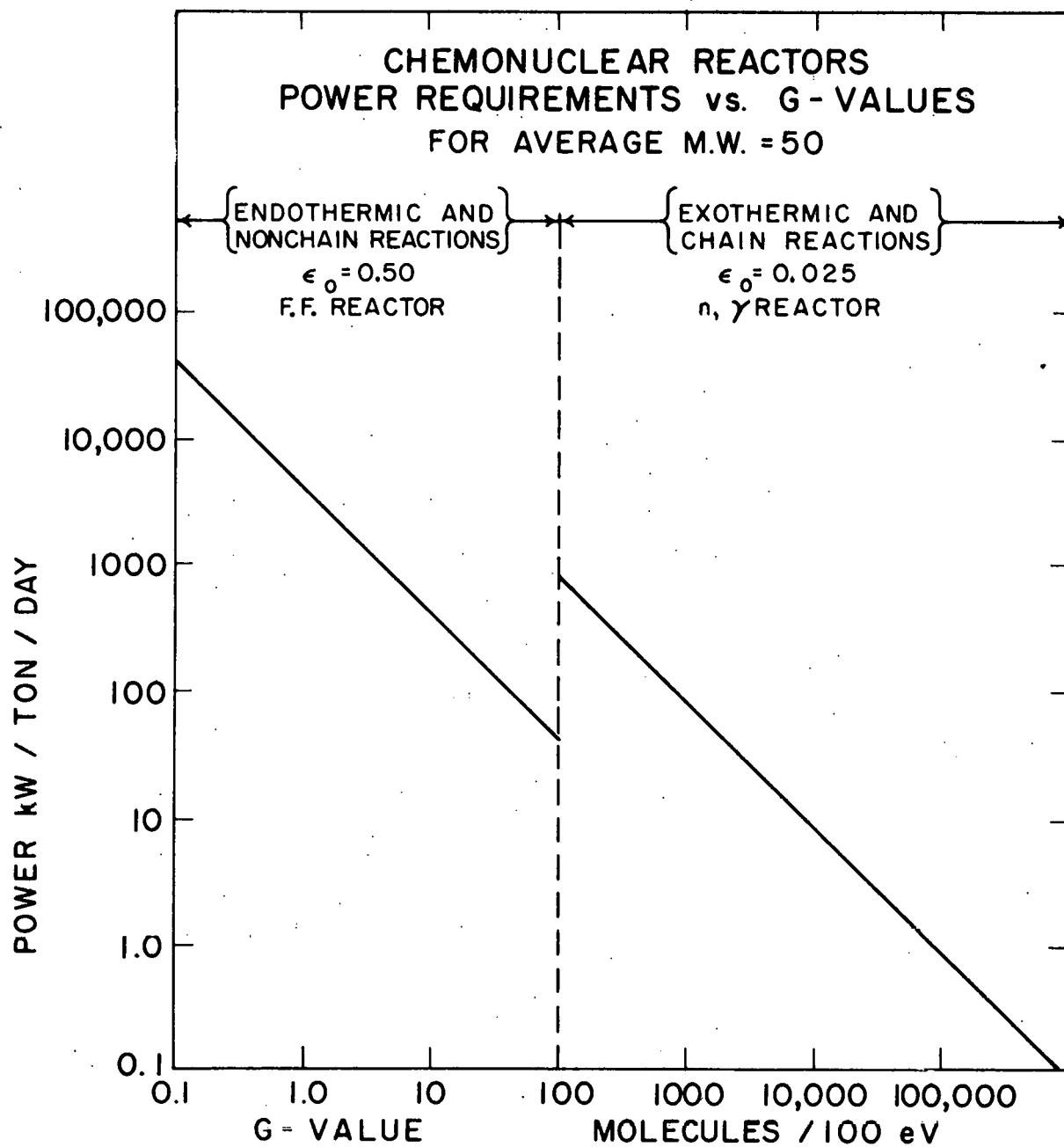


FIG. 5

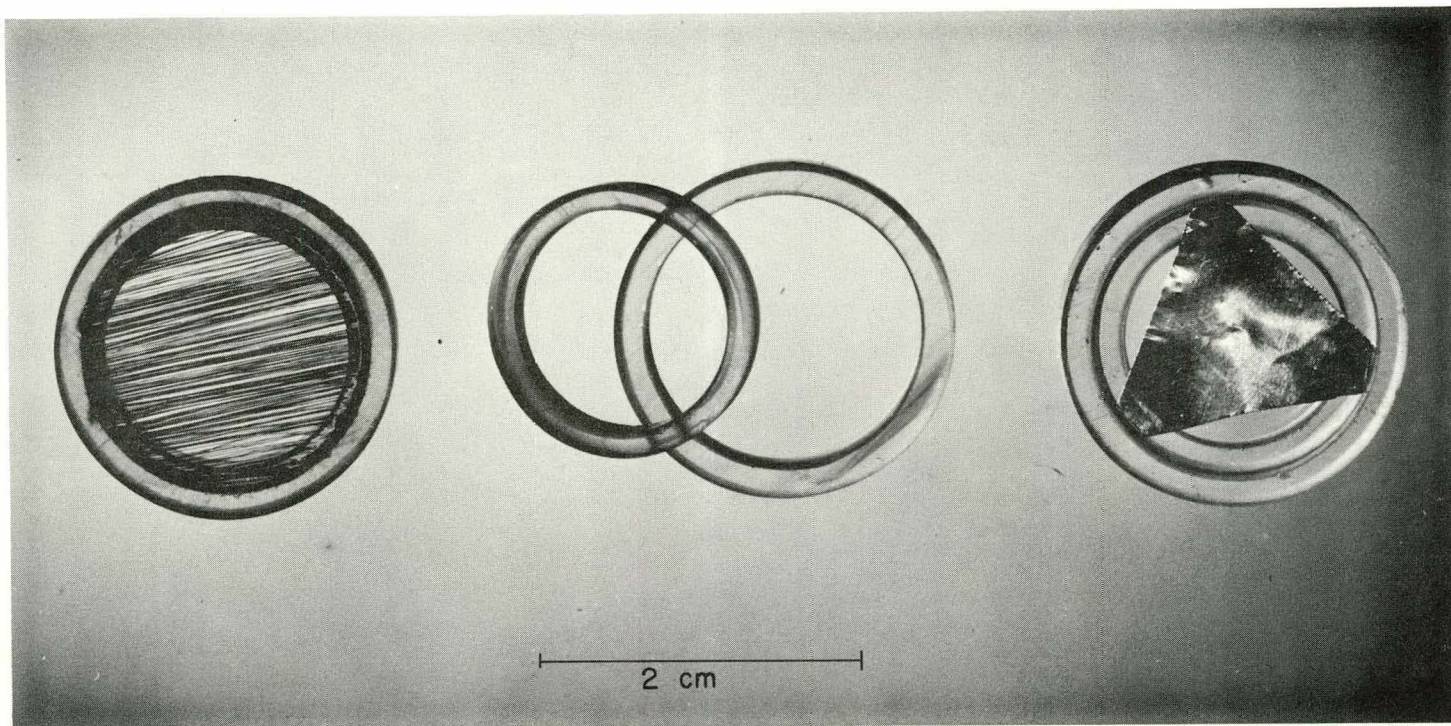


FIG. 6

ENERGY DEPOSITION
 EFFICIENCY vs WT LOADING OF U-GLASS
 FIBER ELEMENT. FIXED ARRAY OF MULTIPLE
 STRAND U-GLASS 5 STRAND-3 μ TWISTED FIBER

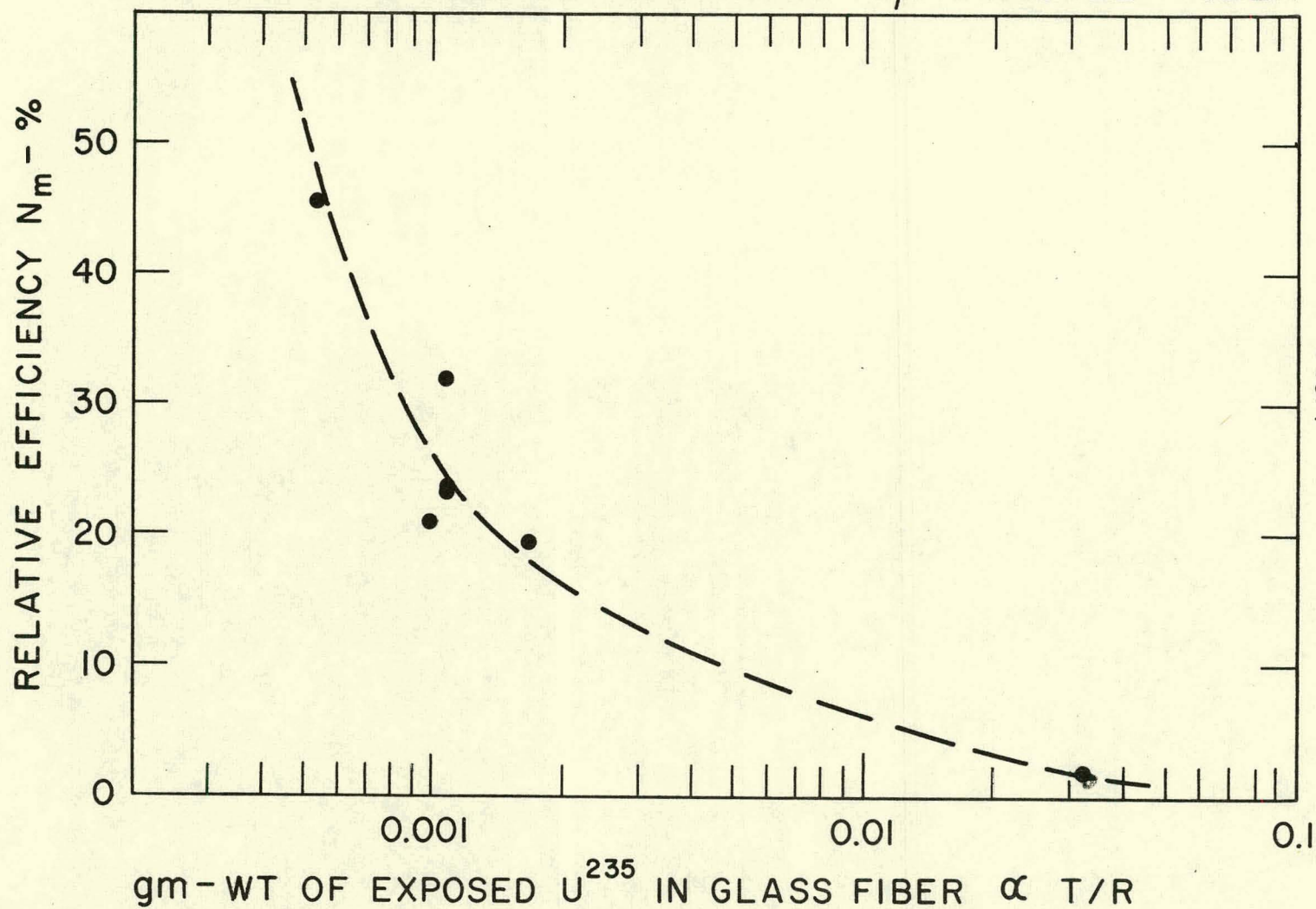


FIG. 7

RELATIVE EFFICIENCY FOR AN ORDERED ARRAY OF INFINITE SLABS

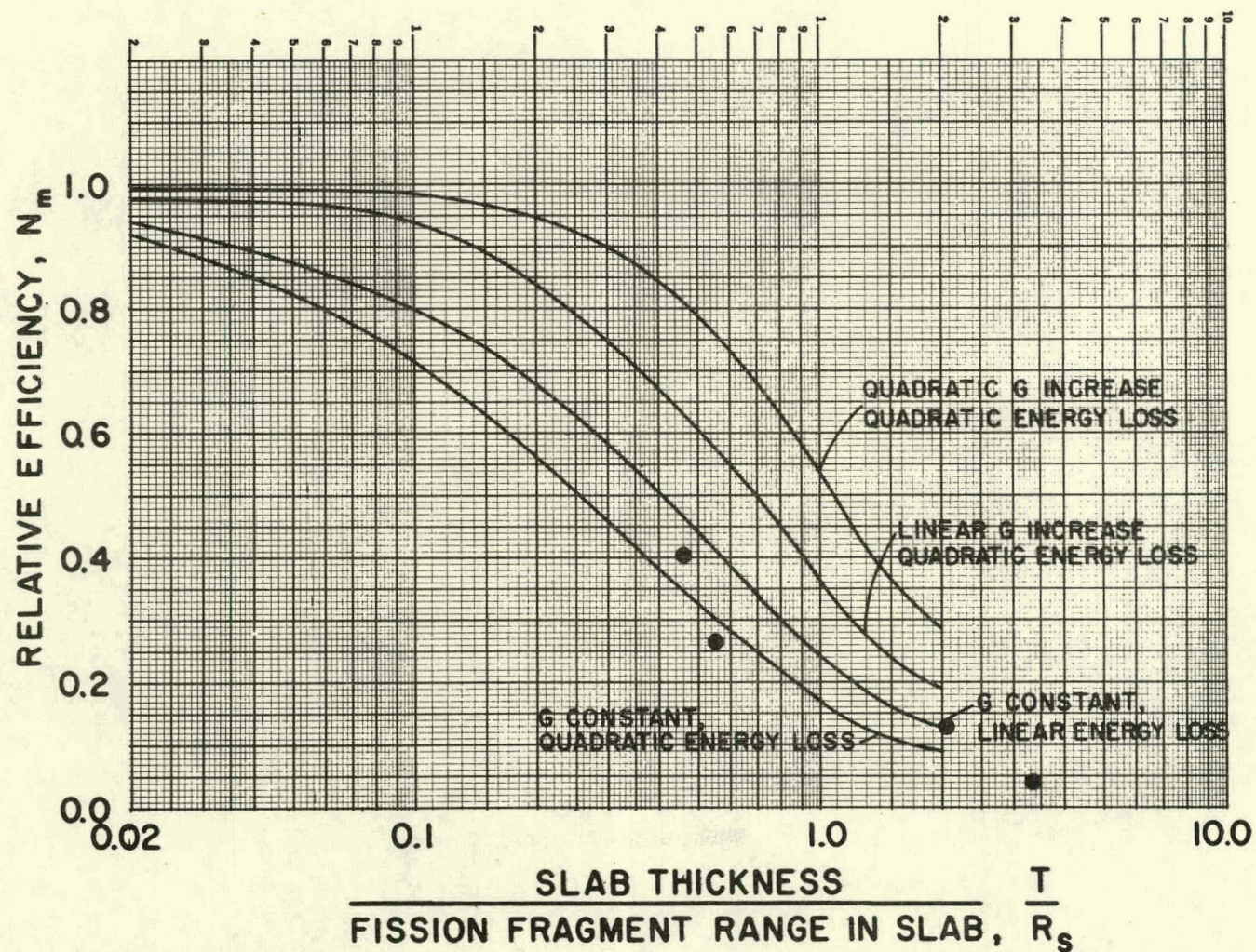
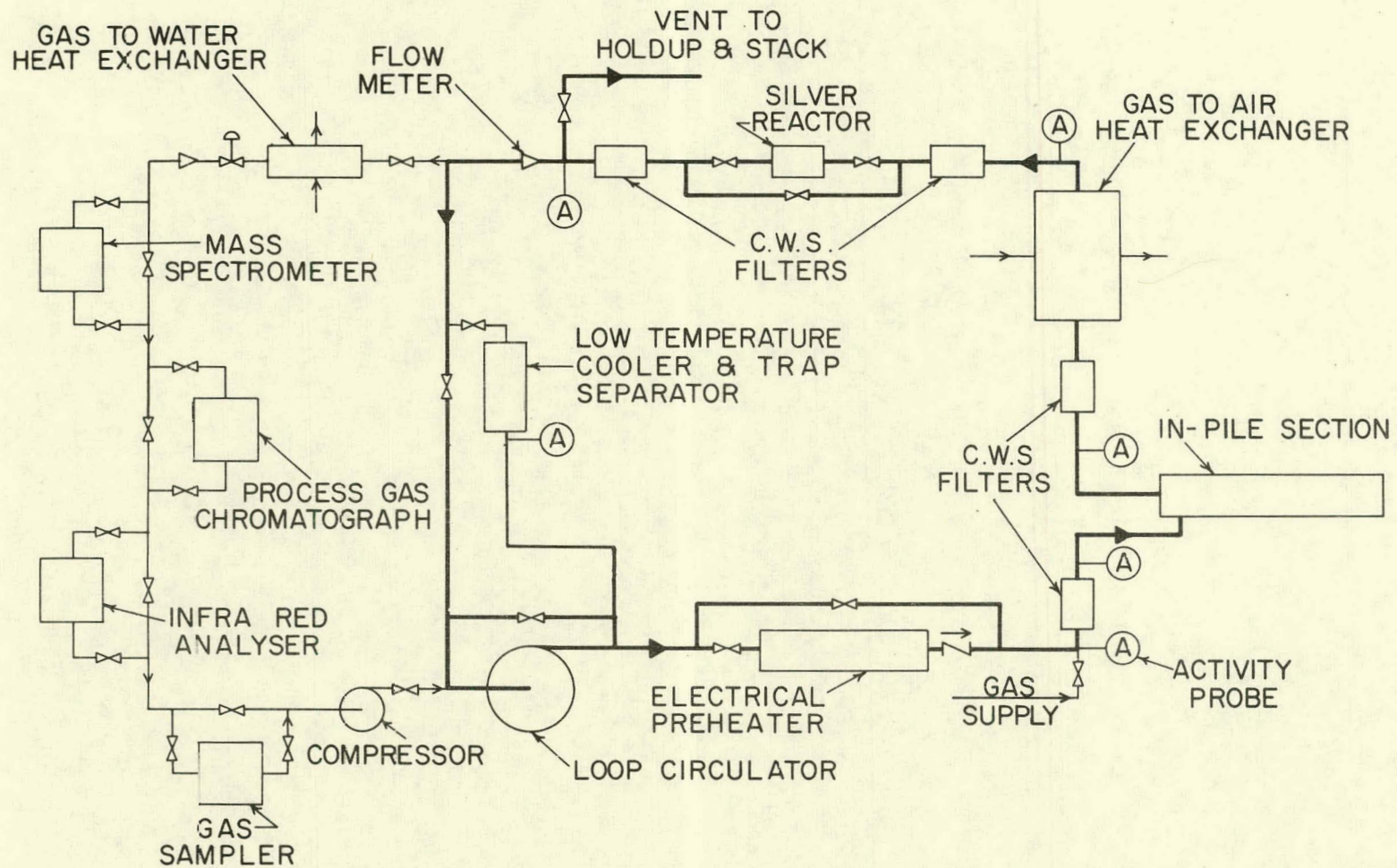
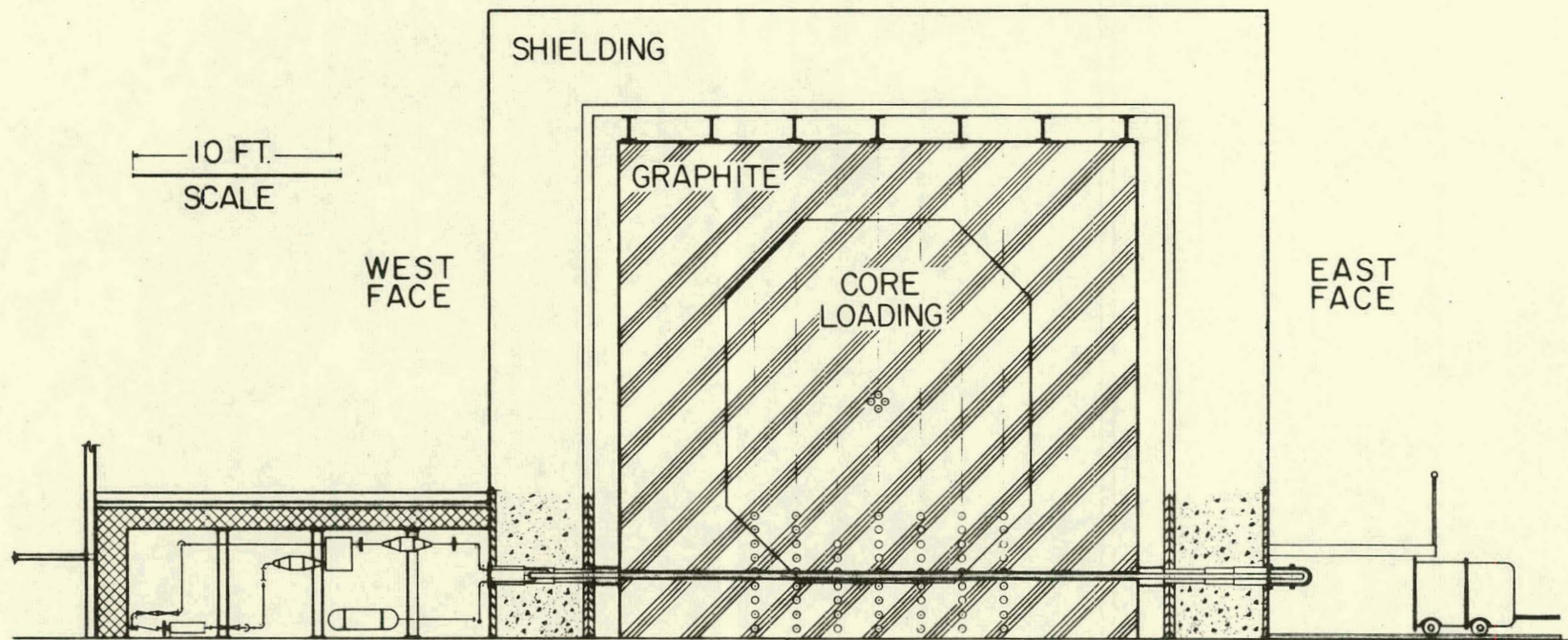


FIG. 8



FLOW SHEET
BROOKHAVEN CHEMONUCLEAR IN-PILE TEST LOOP

FIG. 9



SECTION THRU
BROOKHAVEN CHEMONUCLEAR IN-PILE TEST LOOP

FIG. 10

Co^{60} RADIATION POLYMERIZATION OF ETHYLENE RATE CURVES

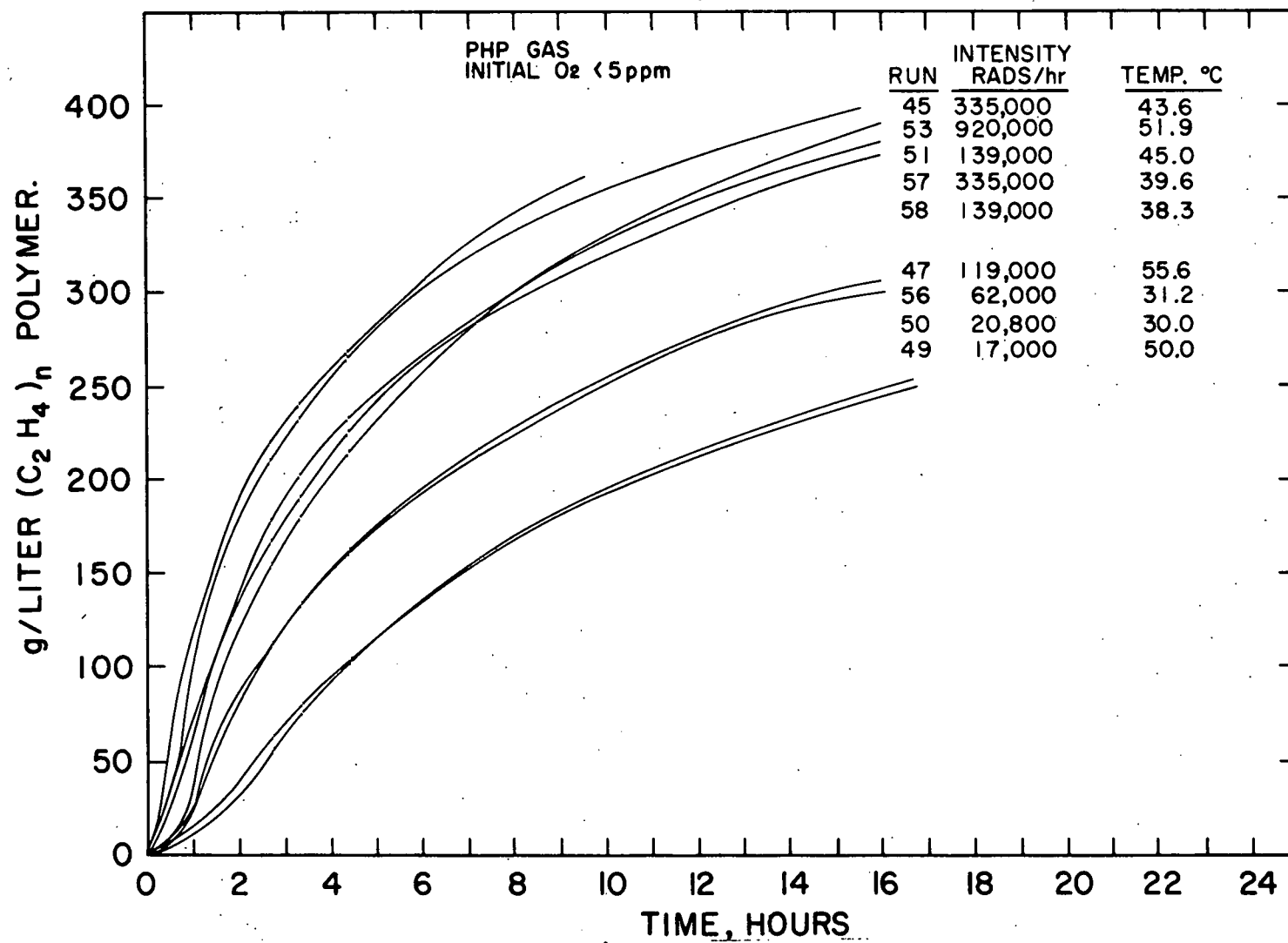


FIG. II

IR SPECTRA FOR POLYETHYLENE HOMOPOLYMER AND
ETHYLENE-CARBON MONOXIDE COPOLYMER PRODUCED
BY Co^{60} GAMMA RADIATION

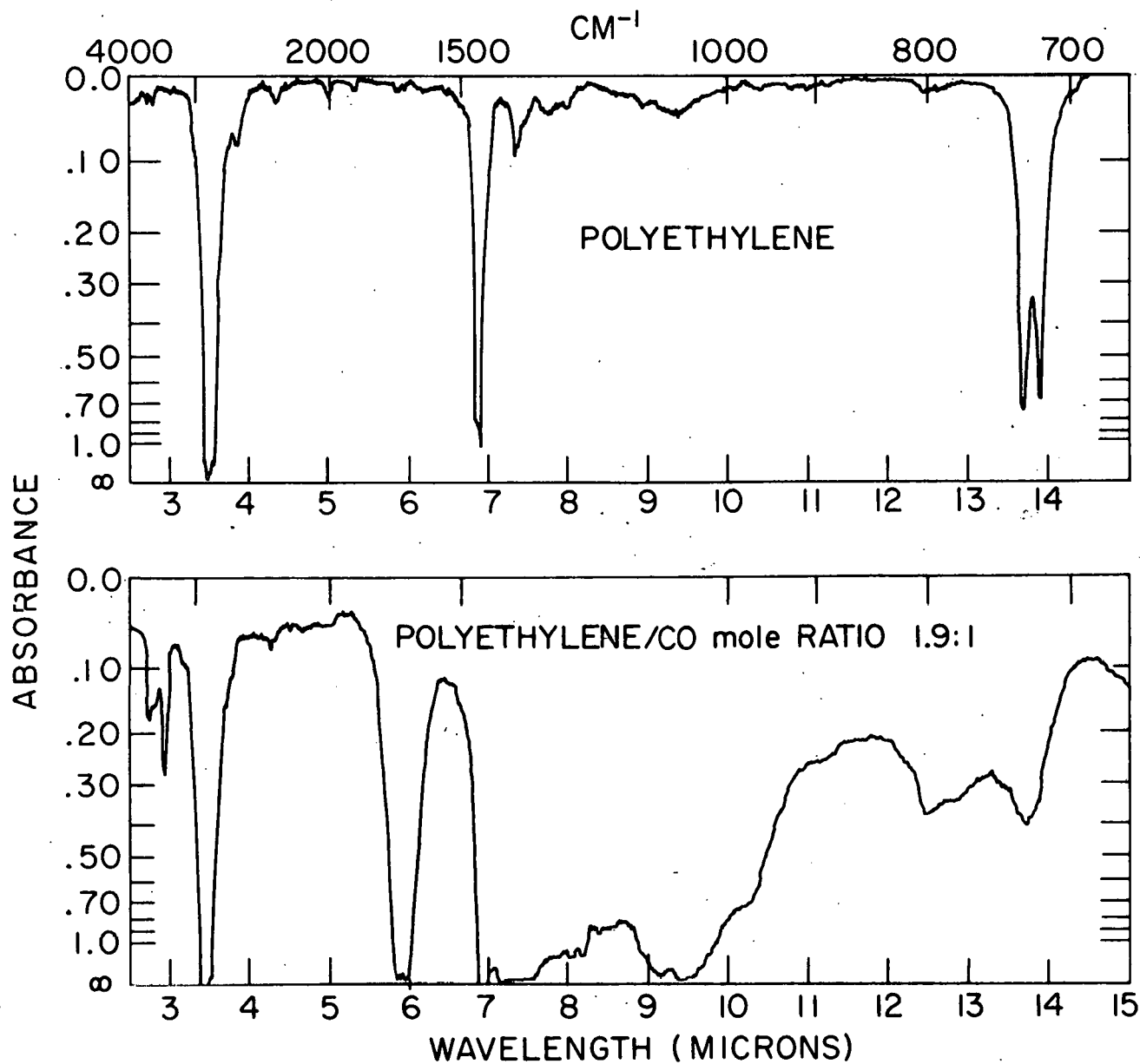


FIG. 12

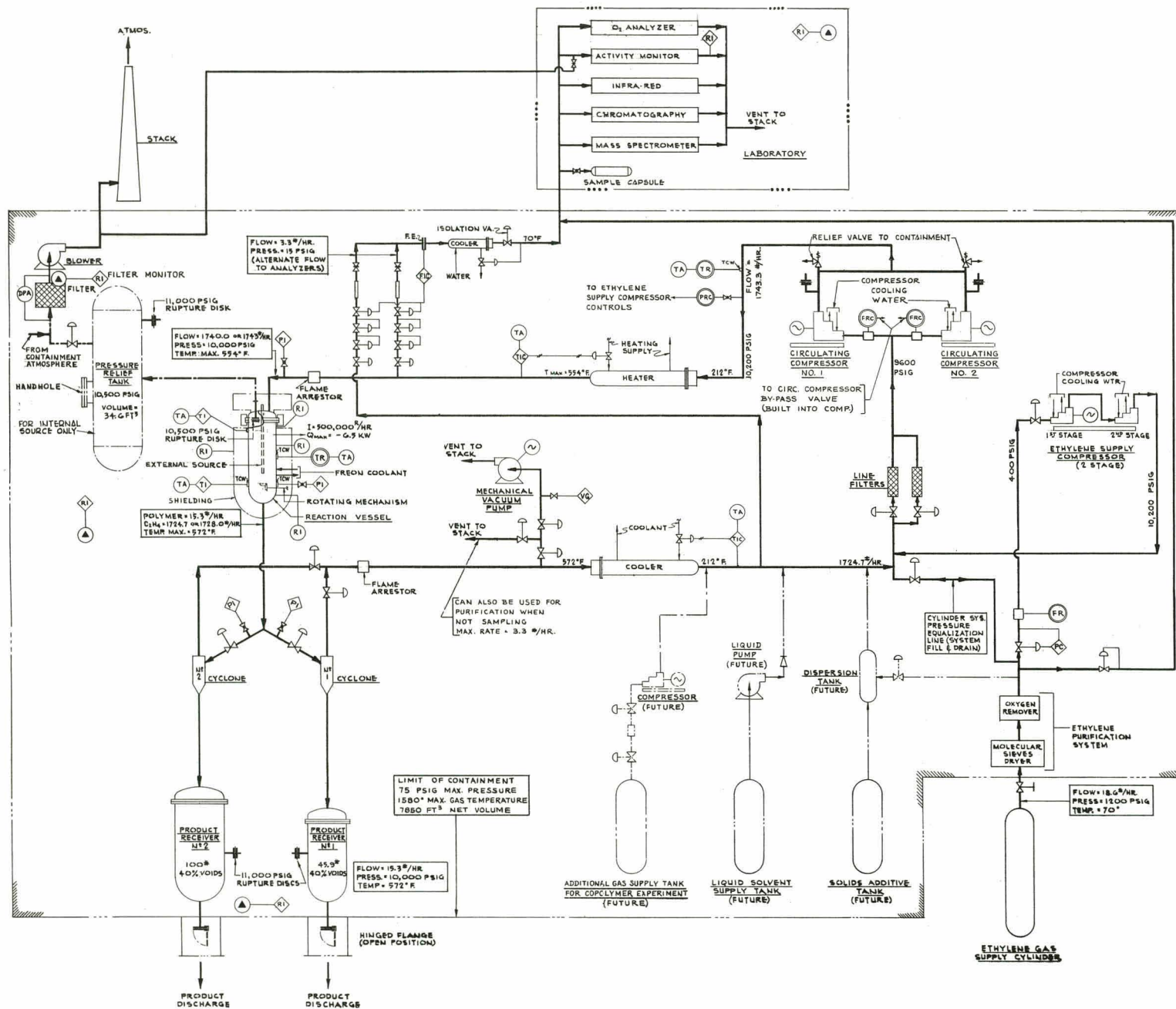
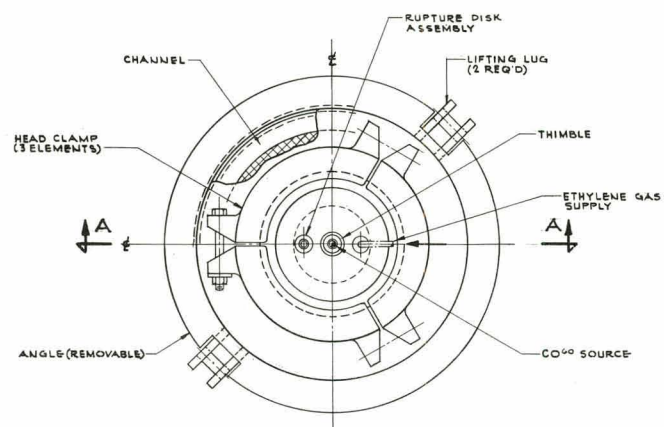
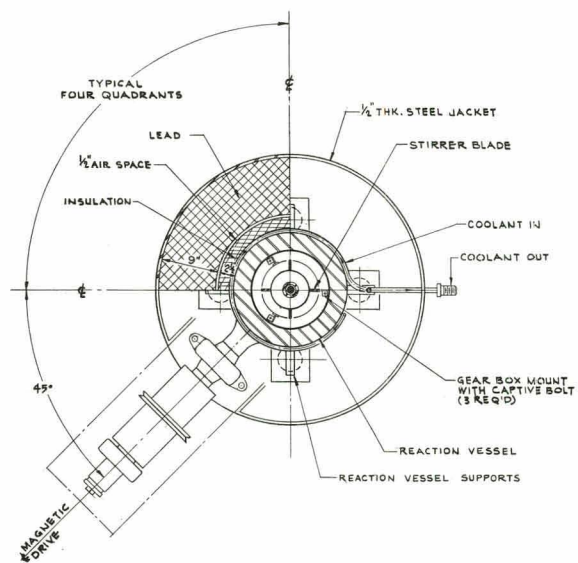


FIG. NO.13

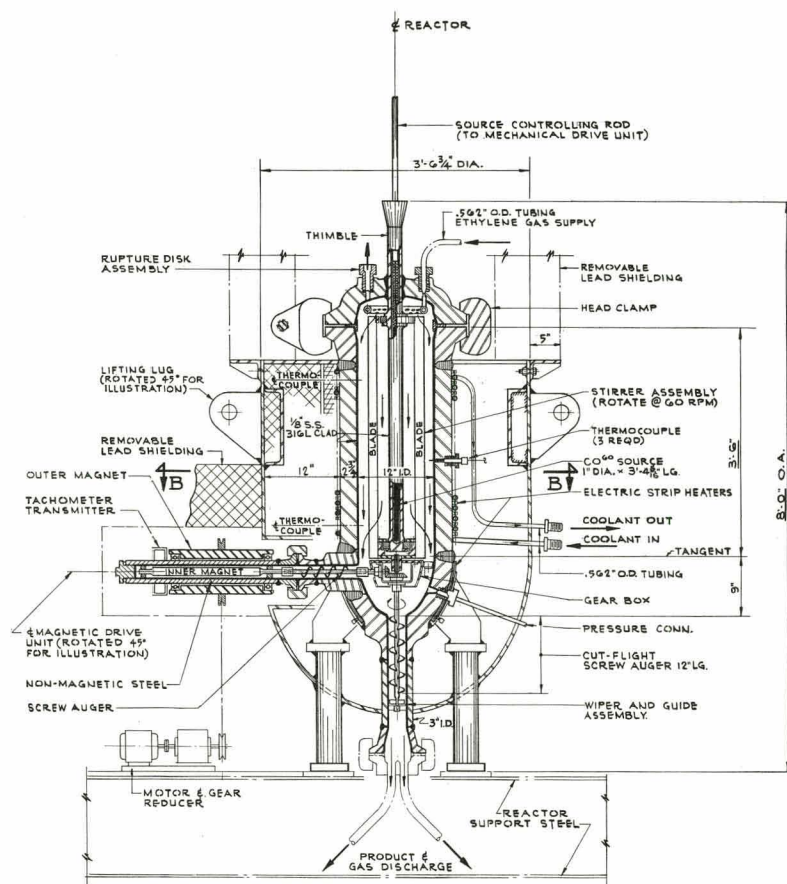
HIGH PRESSURE
GAMMA LOOP EXPERIMENT
FLOW DIAGRAM



PLAN



SECTION "B-B"



SECTION "A-A"

FIG. NO.14

HIGH PRESSURE
GAMMA LOOP EXPERIMENT
REACTION VESSEL ARRANGEMENT
WITH EXTERNAL SOURCE

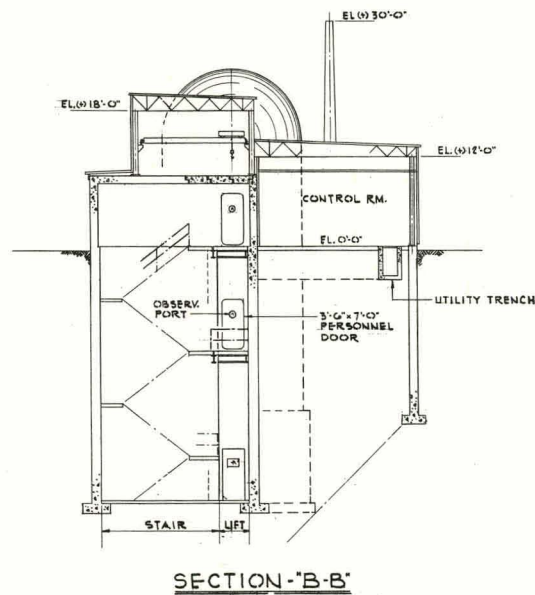
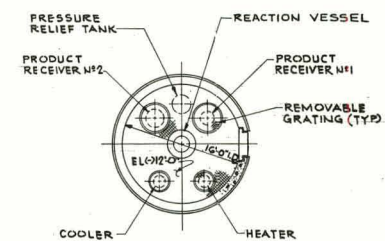
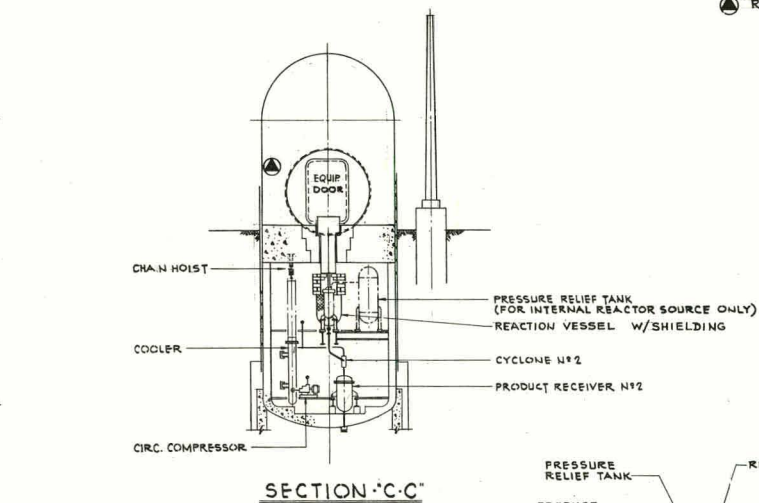
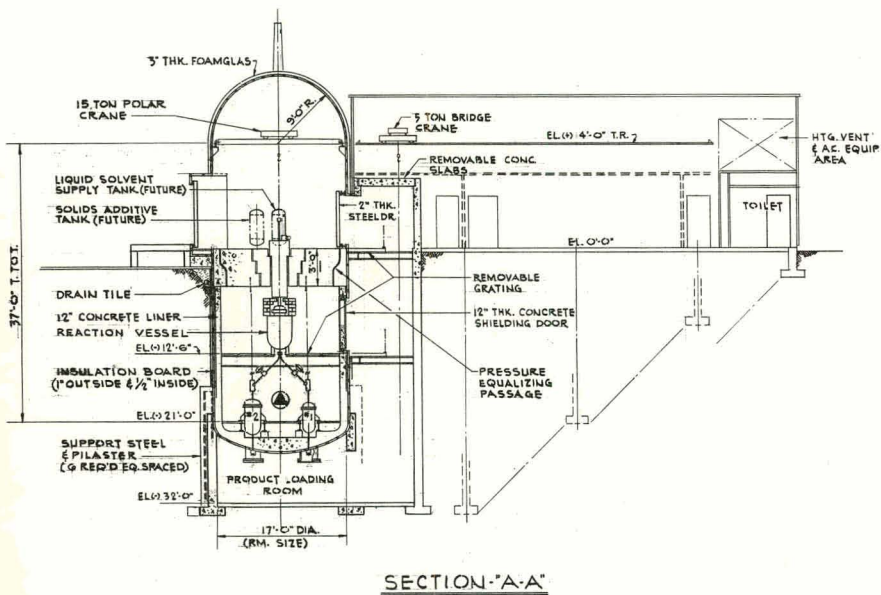
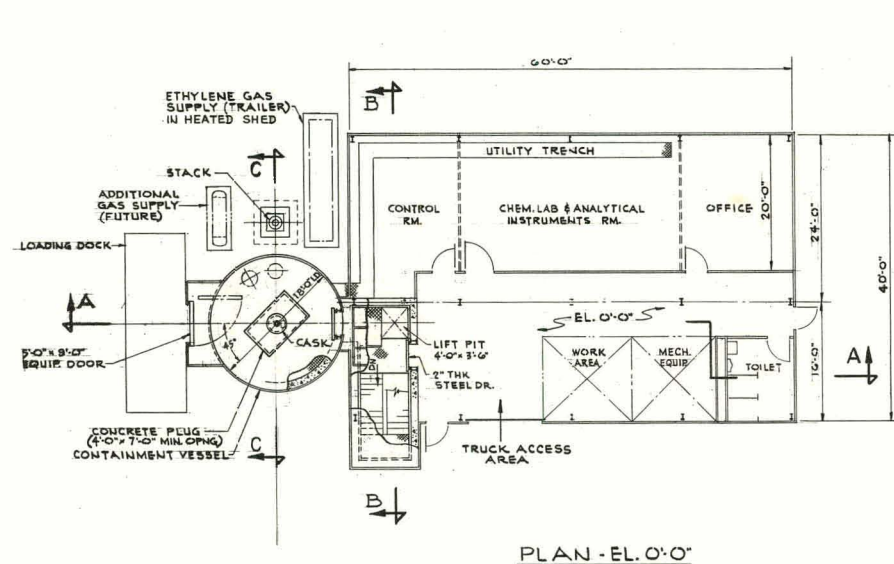


FIG. NO.15

HIGH PRESSURE
GAMMA LOOP EXPERIMENT
BUILDING ARRANGEMENT