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PRODUCTION TEST IP-725
INCREASED GRAPHITE TEMPERATURE LIMIT -
F REACTOR

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PRODUCTION TEST IP-725
INCREASED GRAPHITE TEMPERATURE LIMIT -
F REACTOR

By

A. Russell

December 1, 1964

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PRODUCTION TEST IP-725
INCREASED GRAPHITE TEMPERATURE LIMIT - F REACTOR

I. OBJECTIVE

The fundamental objective of the graphite temperature limit is to prevent excessive oxidation of the graphite moderator blocks with carbon dioxide and water vapor in the reactor atmosphere. Laboratory tests have shown that 10 per cent uniform oxidation of graphite results in a loss in strength of approximately 50 per cent.¹ This value of a strength loss of 50 per cent has been arbitrarily established as the limit to graphite oxidation over the lifetime of the reactor stack. This limit has been further arbitrarily interpreted as an oxidation rate of not more than two per cent per thousand days of reactor operation. At this rate limit, the reactor stack could be operated for 15-20 years before the graphite strength was reduced 50 per cent.²

As with any chemical reaction, the rate of reaction of graphite with oxygen, carbon dioxide, and water vapor increases with increasing temperature. While all three oxidants are present to some degree in the reactor gas system, the reaction between graphite and oxygen at the present temperatures is so rapid that all practical precautions must be taken to exclude it from the gas system. In addition, reaction with water vapor is sufficiently low at present temperatures and concentrations that it does not significantly contribute to the over-all stack oxidation. The graphite temperature limits presently in effect at the smaller reactors were therefore primarily established to hold the reaction rate between graphite and carbon dioxide at two per cent per thousand operating days and were based on laboratory and in-reactor experiments which are presently believed to have been partially contaminated with oxygen from air leakage. Additional laboratory tests have been conducted which indicate that the reaction of graphite with carbon dioxide could be considerably lower and would therefore permit an increase in graphite temperature without compromising the present oxidation limit of two per cent per thousand operating days.³

It is therefore the objective of this production test to investigate the effects of higher graphite temperatures on the stack oxidation rate. Specifically, this production test authorizes an increase in the graphite temperature limit of 50 C at F Reactor. Should the graphite oxidation rates obtained during this test warrant additional testing at even higher temperatures, such tests will be authorized by supplements to this production test.

It should be noted that even if the oxidation rates observed during the test period are substantially in agreement with the laboratory data, it cannot necessarily be concluded that operation at higher graphite temperatures will be acceptable under all reactor operating conditions. For example, while a temperature increase of 50 C may be acceptable from the standpoint of the graphite-CO₂ reaction, the graphite-water vapor reaction rate may be unacceptably high during periods of relatively wet operation. Thus, the results of this test will serve only as a partial basis for any revisions to the graphite temperature limits.

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II. BASIS AND JUSTIFICATION

The process standard graphite temperature limit is based on laboratory and in-reactor measurements of oxidation monitoring samples. Additional laboratory tests conducted on the graphite-CO₂ reaction show the reaction rate to be considerably lower than that currently used as the basis for the process standards limit, and suggest that both the laboratory and in-reactor experiments were partially contaminated with air. Figure 1 shows the laboratory and in-reactor test results which served as the basis for the process standards temperature limits. Note that the curve which serves as the basis for the present temperature limits contains a certain amount of conservatism or safety factor when compared to the laboratory data. Figure 1 also shows the results of additional tests which were carefully controlled to exclude air from the experimental apparatus.

On the basis of these additional tests, the sliding graphite temperature limit in the process standards which vary with helium percentage were changed to a uniform temperature limit of 650 C, independent of helium percentage by PITA-26-I and are presently in effect at all reactors with a He-CO₂ atmosphere.⁴ The purpose of this test is to measure the stack oxidation rate by raising the temperature limit to 700 C (790 stringer temperature), independent of helium percentage as shown in Figure 2.

Two additional factors which suggest that the revised graphite temperature limit of 650 C currently in effect may be conservative are the recent in-reactor oxidation monitoring samples and the measurement of carbon monoxide (reaction product of the graphite-CO₂ reaction) in the reactor gas atmosphere. Since the tightening of the permissible air in-leakage limits, the in-reactor oxidation monitoring samples have shown oxidation rates of about 0.5 per cent per thousand operating days. While the temperature limit in the reactor stack is 650 C, the monitor samples are placed in uncooled graphite channels which operate at maximum temperatures of approximately 735 C. Note from the carefully controlled laboratory tests shown in Figure 1 that the calculated oxidation rate of graphite in 100 per cent CO₂ at 735 C is approximately 0.9 per cent per thousand operating days. This is in reasonably good agreement with the observed oxidation rate of 0.5 per cent at the percentage of CO₂ found in the reactor gas system. In addition, the monitor samples generally operate at a temperature slightly less than the temperature limit. If the reaction rate data which serves as the basis for the graphite temperature limits in the process standards were correct, the oxidation rates of the monitor samples should have been in excess of 100 per cent per thousand operating days. An additional indication that the over-all stack oxidation rate may be low is the concentration of carbon monoxide in the gas system. Figure 3 is an approximation of the carbon monoxide concentration in the gas system as a function of daily gas loss and graphite oxidation rate. If the graphite oxidation rate were really at the present limit of two per cent per thousand operating days at the present operating graphite temperature, the carbon monoxide concentration should be in the order of ten per cent. Gas composition analysis, however, shows the carbon monoxide concentration at the old reactors generally to be 0.5-1.0 per cent.

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The incentive for increasing the graphite temperature limit for this test is to pilot higher moderator temperatures which may be desired for production of alternate products such as Pu-240. An additional incentive is the possible reduction in helium loss. Numerous reactor gas loss data are available which demonstrate that helium loss rate increases exponentially with helium percentage in the reactor gas. This occurs for two reasons. First, the greater the percentage of helium in the gas, the greater will be the quantity of helium lost for each cubic foot of reactor gas which leaks out of the system, and secondly, the greater the percentage of helium in the gas, the less dense will be the gas mixture and the greater will be the gas loss rate for any given size opening and gas pressure. Thus, relatively small reductions in helium percentage can result in relatively large savings in helium. As an illustration, Figure 4 shows the approximate relative helium loss as a function of graphite temperature at constant reactor power. This curve must be qualified by stating that the reduction in helium loss is achieved provided the temperature is increased by reduction in helium percentage rather than by increasing reactor power. Obviously, if the temperature is increased only by raising reactor power, the helium loss would remain unchanged. Figure 4 does not indicate the actual helium loss since this depends upon reactor power level and will vary from summer to winter. It does, however, illustrate that substantial savings in helium can be achieved by a modest increase in graphite temperature if the associated increase in graphite oxidation rate is acceptable.

It should be noted that even though an increase in graphite temperature may be feasible from the standpoint of graphite oxidation, it may not necessarily be economically justified because of metal exposure or metal availability. An increase in the graphite temperature limit, if it were expanded to include all He-CO₂ reactors, would probably require a reduction in metal exposure to maintain the Pu-240 content within the present product specification of six per cent. If the present product specification were relaxed or if higher quantities of Pu-240 were desired as an alternate product, the incentive for raising the graphite temperature by reducing helium percentage would be enhanced. Thus, the incentive for increasing graphite temperature to save helium hinges largely upon the optimum balance between reduced helium loss and increased metal costs and will not be completely resolved by the production test.

III. TEST DETAILS

A. Procedure

Upon approval of this production test, the graphite temperature limit at F Reactor will be increased by 50 C. For the duration of this test, the graphite stringer temperature limit will be 790 C (corresponding to a maximum graphite filler block temperature of 700 C). It is estimated that the graphite temperature can be increased 50 C by a reduction in helium percentage of approximately 15-20 per cent.

The interim normal nitrogen limit presently in effect during equilibrium operation at F Reactor represents a slight relaxation of air entry control and was considered justifiable on the basis that the reactor was soon to be

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permanently shut down.⁵ For the duration of this test, however, the normal nitrogen limit will be revised downward to .1%, consistent with the limits at B, C, and D Reactors. This limit of .1% is considered desirable to reduce the contamination of the oxidation monitor samples with air.

B. Instrumentation

1. Graphite oxidation monitoring samples presently in tube channels 1067 and 2580 will be charged out and replaced with new samples.
2. The presently installed chromatograph is calibrated to measure carbon monoxide concentrations up to 10 per cent. Although there is the prospect that this concentration could be exceeded, the present chromatograph can be adequately re-ranged and calibrated with present equipment.
3. The increase in graphite temperature will increase the reaction rate between graphite and the water vapor in the gas atmosphere. This reaction can be monitored by measurement of one of the reaction products, hydrogen. A hydrogen analyzer with adequate sensitivity and range is available and will be temporarily installed for this purpose.

C. Data Required

1. Graphite oxidation which has occurred during the test will be measured at the conclusion of the test by analysis of the oxidation monitoring samples.
2. Oxidation during the test will be monitored by analysis of the reaction products, carbon monoxide and hydrogen, in the reactor gas system. The continuously recording chromatograph and hydrogen analyzer will be used for this purpose.
3. Gas loss information will be available from operational records.

IV. HAZARDS

The oxidation rate data shown in Figure 1 represents the best laboratory data presently available on the graphite-CO₂ reaction. There is always the prospect, however, that the oxidation rate may be higher than anticipated. In addition, data on the graphite-water vapor reaction, as it applies to the stack geometry, are very limited. There is, therefore, the hazard that at the higher temperature authorized for this test, these reactions may proceed at a rate which is unacceptable for prolonged reactor operation. One of the primary purposes for monitoring the carbon monoxide and hydrogen reaction products in the gas system is therefore to provide early warning of excessive oxidation rates if they should exist.

The additional stack oxidation which will occur during the test period will be arbitrarily limited to one per cent, corresponding to an oxidation rate limit of ten per cent per thousand operating days during the three month test period. The carbon monoxide and hydrogen concentrations in the reactor gas system which would result from this rate are shown in Figure 5 and will serve

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as a guide until the oxidation monitor samples can be removed and analyzed. Although it is not anticipated that the 50° increase in the temperature limit authorized by this test will result in an oxidation rate of ten per cent per thousands operating days, the test will be terminated by gradually reducing the temperature limit back to 650 C over a period of several days should the concentration of carbon monoxide in the gas system reach the values shown in Figure 5. The test will also be terminated should the concentration of hydrogen reach four per cent, not because of excessive oxidation, but to preclude the remote possibility of explosion. The explosive range of hydrogen in air is 4-75 per cent and while there is no danger of explosion from hydrogen leakage out of the reactor because of high air flow across the unit (approximately 40,000 cfm) there could be some concern when the unit is opened to atmosphere during shutdown if the hydrogen were greater than four per cent. It is unlikely that this four per cent limit on hydrogen will have any effect on the test since the 50 C increase in temperature will probably not increase the present concentration of hydrogen of .01-.03 per cent by more than a factor of 3-5.

V. PROCESS STANDARDS

All Process Standard limits with regard to over graphite temperature limit provisions and inlet dew point requirements will remain in effect except those specifically exempted by PITA-26-I, Supplement C.⁵

The reduction in helium percentage is expected to result in an increase in biological shield temperature in the order of 30°. Although 30° is not expected to increase the shield temperature to the 160° limit which would require reactor shutdown, it will likely drive the temperature up into the 110-160° range. Process Standards recommends charging fringe poison to maintain shield temperatures below 110°, however, for the duration of this test, maximum shield temperatures in the range of 110-160° will be considered acceptable without charging additional fringe poison.

VI. LIMITS

The various temperature and gas system limits for this test are summarized as follows:

- A. Graphite temperature limit - 700 C in graphite filler blocks (corresponds to 790 C on front to rear thermocouple stringers).
- B. Over graphite temperature limit provision - Process Standards, Section C-040.
- C. Normal nitrogen limit - .1%.
- D. Normal oxygen limit - .02%.
- E. Startup oxygen limit - 1% when power level exceeds 1 Mw.
- F. Carbon monoxide limit - See Figure 5.
- G. Hydrogen limit - 4%.

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H. Moisture content - Process Standards Section B-010.

I. Biological shield temperature - 160 C.

VII. TEST DURATION

This test is expected to last approximately three months but may be terminated early:

- A. When it is believed that meaningful oxidation results can be obtained from the oxidation monitor samples.
- B. If the concentration of carbon monoxide in the gas system exceeds the values shown in Figure 5 or if the concentration of hydrogen exceeds four per cent.
- C. If biological shield temperatures exceed the current Process Standards limit of 160.
- D. Upon mutual concurrence of the Managers of F Processing Operation, Process and Reactor Development Operation, and Process Technology Operation.

Senior Engineer
Reactor Engineering
Process and Reactor Development

A Russell/ba

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VIII. RESPONSIBILITIESA. Manufacturing Section

1. F Reactor Operation will be responsible for:
 - a. Providing sufficient outage time for charge-out of oxidation samples at the beginning and conclusion of the test.
 - b. Operation within the current Process Standard Limits except those specifically exempted by PITA-26-I, Supplement C and this production test.

Approved: [Signature]B. Research and Engineering Section

1. Process and Reactor Development Subsection will be responsible for:
 - a. Evaluation and interpretation of oxidation sample results.
 - b. Interpretation of reactor gas composition.
 - c. Recommending additional testing, if warranted.
 - d. Interpretation of gas analysis during the test to assure that termination of the test, if necessary, can be initiated before hazardous concentrations of hydrogen and carbon monoxide are reached.
 - e. Issuance of final report.

Approved: [Signature]

2. Process Technology Subsection will be responsible for auditing compliance with the provisions of this test.

Approved: [Signature]

3. Testing Subsection will be responsible for accomplishing charge-out of oxidation monitor samples.

Approved: [Signature]C. Facilities Engineering Section

1. Mechanical Development Unit will be responsible for temporary installation and operation of hydrogen analyzer.

Approved: [Signature] DLH per JOK

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4. R. E. Baars. PITA-26-I, Transition to High CO₂ Concentration and Revision of Gas Purity Limits and Controls, HW-79281. October, 1963.
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Figure 1
GRAPHITE OXIDATION WITH CARBON DIOXIDE

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Graphite Temperature

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Figure 2
GRAPHITE TEMPERATURE LIMITS

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MM-3300-074(3-60)
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Graphite Temperature

Figure 3

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CARBON MONOXIDE CONCENTRATION IN REACTOR GAS

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Figure 4

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APPROXIMATE RELATIVE HELIUM LOSS

Relative Helium Loss

BM-5300-074(3-60)
AS-42 12-10-60

Graphite Temperature

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Figure 5
CARBON MONOXIDE AND HYDROGEN CONCENTRATION IN
REACTOR GAS AT GRAPHITE OXIDATION RATE OF 10% PER
THOUSAND OPERATING DAYS

BR 100-100 (1-10)
 AR 100-100 (1-10)

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