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PRODUCTION TEST IP-358-AC:

REPLACEMENT OF CARBON DIOXIDE WITH NITROGEN AS A CONSTITUENT OF THE K REACTOR ATMOSPHERE

11

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Verified By J. J. Allen 2-11-94

PRODUCTION TEST LP-358-AC:

REPLACEMENT OF CARBON DIOXIDE WITH NITROGEN AS
A CONSTITUENT OF THE K REACTOR ATMOSPHERE

MASTER

OBJECTIVE

To investigate the reactivity and temperature effects and the associated operating problems, if any, resulting from the use of nitrogen instead of carbon dioxide as a constituent of the reactor atmosphere.

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BASIS AND JUSTIFICATION

Compensation for the positive long-term reactivity transient associated with Hanford reactors may be accomplished in two ways: the addition of a poisonous material (rods, splines, etc.) to the reactor, or cooling the moderator by changing the gas composition. During extended operating periods, the use of rods alone for compensation tends to cause flux distortions and difficult-to-control distributional flux cycling during periods of high residual exposure. Use of either splines or the poison column control facility (PCCF) introduces poison in particular locations in discrete increments which also hinders smooth control, particularly at high residual exposures. Lowering the graphite temperatures by changing the gas composition can be applied in a manner to almost exactly compensate for the uniform increase in reactivity in the control zone from long-term gains. However, because of high operating levels and restrictions on the use of CO₂ at high temperatures from graphite burnout considerations, operating periods start with about 90-95% He, thus limiting compensation

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for long-term gains by increasing the helium concentration. Since nitrogen is a relatively inert gas, its use in place of CO₂ should result in a lower graphite burnout rate at comparable graphite temperatures, permitting a higher graphite temperature limit; thus with nitrogen it will be possible to start an operating period with about 80 percent helium or less. The use of variable concentrations of N₂ rather than CO₂ should permit a significant degree of control flexibility to be available through variance of the reactor atmosphere composition at graphite temperatures above the present standard limits.

Nitrogen can be useful for control purposes because the effect of its relatively large neutron absorption cross-section (compared either to helium or to carbon dioxide, see Appendix 1) is small compared to the temperature-reactivity effects it can cause.^{/1/} Nitrogen has approximately the same thermal conductivity as carbon dioxide^{/2/} (see Figure 1); hence where nitrogen is used to replace the carbon dioxide in a helium-carbon dioxide mixture, the conductivity of the gas is relatively unaffected. This is an important feature, for it means that no significantly different power coefficients of reactivity should be introduced if nitrogen is used to replace the carbon dioxide, and that control techniques (using the gas system) with nitrogen-helium mixtures will be similar to those presently used for carbon dioxide-helium mixtures.

Nitrogen is a relatively inert gas which should be suitable for reactor use. In-pile studies made with nitrogen demonstrated that the presence of nitrogen did not increase the corrosion of aluminum or steel^{/3/}. In those tests aluminum and steel foils with a high surface-to-volume ratio were exposed in the reactor in the presence of graphite to water-saturated nitrogen in one case and to water-saturated helium in the other. Under these severe conditions, the corrosion rates of the metals were concluded to be functions of gas moisture content and independent of the nominal atmosphere. Under normally dry atmosphere operating conditions corrosion rates would be negligible, but in the presence of water vapor corrosion would take place irrespective of the gas carrier. Mass spectrometer analyses of the effluent gas were taken and showed that no oxides of nitrogen were formed. It was concluded that nitrogen in the pile atmosphere would not increase the corrosion of metallic components in the reactor even though water leaks existed.

TEST DETAILS

This production test is to be carried out at the KE reactor, where PT-IP-312-A^{/4/} is currently being performed. If this test demonstrates that 1) no significant problems exist during operation with nitrogen in the gas system, and 2) it is both feasible and desirable to operate with nitrogen in the gas system, either a revision in standards will be made or a Process Improvement Transition Authorization will be issued to permit both K reactors to operate with nitrogen instead of carbon dioxide in the reactor atmosphere.

Operating Procedure

1. No N₂ is to be added to the system for the first few days of operation. During this time an equilibrium He-CO₂ atmosphere is to be established, splines are to be used to improve the pile flattening, and the control rods are to be manipulated into what is thought to be an optimum configuration.
2. Once the reactor is at equilibrium, well flattened, and with a "good" rod pattern (about 5 days after startup) the test is to be initiated by stopping the flow of CO₂ into the reactor and introducing the nitrogen, utilizing splines as needed to

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maintain optimum conditions. For purposes of administering graphite temperature limits, each per cent of N_2 is to be treated as a per cent of He. The following information has been used to calculate the rate at which N_2 is to be introduced into the system:

- a. The total gas system capacity is assumed to be approximately 35,000 cubic feet.
- b. Total gas losses are assumed to average about 10,000 cubic feet per day. The N_2 shall be added to the pile atmosphere in the N_2 concentration from 0% to about 25% over and eight-day period. The following tentative gas makeup schedule is indicative of the flow rates required.

<u>Day of</u> <u>N_2 Addition</u>	<u>Ratio N_2/He</u> <u>in Makeup</u> <u>Stream</u>	<u>N_2 Flow Rate</u>	<u>% N_2 at</u> <u>End of Day</u>
1	1/8 - 1/7	35-60 ft ³ /hr	~ 3%
2	1/6 - 1/5	50-75	~ 6%
3	1/5 - 1/4	65-90	~ 9%
4	1/4 - 1/3	80-105	~ 12%
5	1/3 - 2/5	95-120	~ 15%
6	2/5 - 1/2	110-135	~ 18%
7	~ 1/2	125-140	~ 21%
8	~ 2/5	105-130	~ 24%
9	~ 1/3	~ 105	~ 25%
10	~ 1/3	~ 105	~ 25%

The actual makeup schedule shall be determined by the KE Pile Physicist with due regard for current operating conditions and ease of control. Once the CO_2 content is shown to be less than 1%, the nitrogen concentration may be varied in such a manner as to improve flexibility of control. (In general, this corresponds to a decrease of about 1 to 1.5 per cent per day). Prior to introducing any N_2 into the reactor atmosphere, the CO_2 must be completely shut off, and isolated from the gas system, to insure against inadvertent introduction of CO_2 into the reactor atmosphere.

Subsequent Startups and Operation - For the duration of the test, CO_2 is not to be used as a constituent of the reactor atmosphere. The control techniques utilizing the gas composition will remain essentially unchanged. The nitrogen content may be increased to as much as 100%, providing graphite temperature limits are not exceeded. In all cases the maximum permissible nitrogen concentration will be determined by the 700°C graphite temperature limit specified in PT IP-312-A which includes the "Emergency Conditions Only" temperature overrun provision of Section 40.01 of Process Standard C-040. With these limits the maximum graphite temperature will be limited to 800°C. Under no circumstances is the measured or calculated graphite temperature at any point in the reactor to exceed 800°C.

3. Orsat analyses and gas conductivity measurements are to be obtained and recorded each shift. Samples for mass spectrometer analyses should be taken daily just prior to adjusting the flow and makeup rates. Rod configurations and the spline status are to be recorded at the time these samples are taken for subsequent analysis.
4. The water from the driers shall be sampled each time they are regenerated, and will be analyzed for nitrate ion content. If the increase in nitrate ion content above that found in process water is 70% or more (on a mole/liter basis) of the bicarbonate ion content of process water, the test will be suspended and nitrogen purged from the gas system. These sampling procedures will continue until it is determined that the criteria for test suspension will probably not be realized. Process and Reactor

Development Operation will specify subsequent sampling procedures and frequency requirements. In addition, at least three gunbarrels shall be removed from the reactor and inspected, and a photographic record of the condition of the gunbarrels shall be made. The particular tubes, the exact number of gunbarrels, and the date of inspection shall be determined by KE Processing in conjunction with planned tube replacement so that no extra outage time is incurred. These same gunbarrels shall be removed upon termination of the test, or as soon thereafter as is convenient, if additional inspection is deemed necessary by both Research and Engineering and Manufacturing to assure that gross corrosion has not occurred as a result of the use of nitrogen in the reactor atmosphere.

Nitrogen Supply and Instrumentation

1. The nitrogen is to be injected into the makeup line at a controlled rate from one of the low pressure CO₂ tanks which has been filled with nitrogen. The present flow rate instrumentation will be adequate, provided it is calibrated for a nitrogen atmosphere. A supply of approximately 100,000 cubic feet of nitrogen should be available before the test. (It is estimated that 35,000 cubic feet of nitrogen will be used during the first two weeks of the test, and subsequently nitrogen will be used at a rate of about 18,000 cubic feet per week during equilibrium operation.
2. A special monitoring system for obtaining the gas composition is to be initiated. Routinely the gas composition can be monitored as follows: a) CO₂ content can be determined by an Orsat analysis; b) Helium content can be determined by a thermal conductivity cell by assuming N₂ and CO₂ have the same properties (or some weighted average, if necessary), and c) N₂ content can be calculated by differences. In addition to this method of estimating gas composition, daily samples should be obtained during the test for accurate mass spectrometer analysis.

Time and Termination

This test will continue for one year from the date of approval except that the test may be terminated at any earlier date upon issuance of an appropriate Process Standard, or mutual concurrence of Research and Engineering and KE Reactor Operation. The test may be terminated for any of the following reasons:

1. Potential excessive process tube corrosion (as determined by the drier and dripleg water analysis).
2. Excessive graphite burnout and/or contraction.
3. Excessive radiation levels attributable to the use of N₂.
4. Operating problems (associated with the use of nitrogen) which restrict power level.

Process Standards

No relaxations in graphite temperature limits are authorized, other than those specified by PT IP-312-A which will remain in effect. In administering the temperature limits, the nitrogen is to be treated as helium. Process Standard B-010 shall be relaxed to permit: a) Use of nitrogen as a constituent of the reactor atmosphere operation with b) Nitrogen concentrations in excess of 4% with daily sampling for mass spectrographic analysis. Figure 1, Process Standard B-010 shall be superseded by a new calibration for helium-nitrogen mixtures which shall be obtained by KE Processing Operation. Nitrogen analysis shall be:

N ₂	99.5% min.	O ₂	0.2% max.
CO	0.2% max.	H ₂	0.2% max.
Other	< 0.1%		

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Monitoring and Priority

These shall remain the same as specified in PT IP-312-A which remains in effect, with the exception that two of the four tubes authorized by PT IP-25-A (originally PT-105-532-E) /10,11/ shall be used for graphite burnout monitoring.

Costs

Although nitrogen is roughly twice as expensive as carbon dioxide, the cost of gas should remain relatively unchanged due to the decreased helium consumption.

Potential Problems and Hazards

Although no significant hazards are expected to be associated with the test, there are certain precautions which shall be taken. Nitrogen has never been used in a Hanford reactor to such an extent before, and it is possible that unforeseen problems might arise. For example, the production rate of carbon 14 is expected to be enhanced manyfold as nitrogen replaces carbon dioxide. Although this is not expected to create radiation problems, /5,6,7/ the magnitude of the change is so large that the radiation effects shall be followed closely in both the reactor and gas buildings and in the environment. Another possible hazard is the formation of noxious gases or products. The mass spectrometer analyses can be used to find whether or not this hazard is created and what extent.

While the test results referred to in the "Basis and Justification" indicate that nitric acid formation in the stack will not result from the use of nitrogen, the seriousness of damaging the reactor requires that this potential problem be monitored.

The precautions outlined in the procedure are believed to be adequate to insure that reactor damage due to nitric acid will not occur. A discussion of this corrosion problem is presented in Appendix II.

Nitrogen is used in this test as part of the reactor control system and as such must be handled carefully. The combination of helium and carbon dioxide has been used for reactor control for several years; therefore, the use of nitrogen does not significantly alter the problems which already exist in this respect. As long as gas composition changes are made slowly and at a governed rate no new control problems should be created.

No potential hazards are foreseen from the use of nitrogen from the standpoint of nuclear safety.

The intentional introduction of nitrogen into the reactor atmosphere precludes the use of nitrogen as an indicator of an air leak into the gas system. Measuring the argon concentration and noting changes which are unexplainable from consideration of gas composition changes is a method which could be successfully employed to indicate air leaks. This will be followed with the daily mass spectrometer analysis required by this test. In addition, the possibility of employing a multichannel analyzer to obtain a continuous and "on site" readout of the argon concentration will be investigated by K Reactor Operation.

Responsibility

K Reactor Operation

KE Processing shall be responsible for 1) production continuity and safety of the reactor, 2) appropriate procurement and installation of the nitrogen supply and of a nitrogen

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monitoring system, 3) the controlled introduction of the nitrogen into the reactor, 4) obtaining gas analysis information, 5) recording required control data, and 6) scheduling the manometer traversing (with Irradiation Testing Operation and by Non-metallic Materials Development Operation, Hanford Laboratories) as called for in this test.

Research and Engineering

Operational Physics shall be responsible for 1) assistance in selecting the methods of introducing and monitoring the nitrogen in the system, 2) establishing the rates at which nitrogen and other gases are introduced with the system, 3) over-all coordination of the test, and 4) analyzing and interpreting the reactivity data which are obtained. - G. F. Bailey.

Process and Reactor Development shall be responsible for analyzing and interpreting all data other than reactivity and temperature data.

A final report presenting conclusions and appropriate recommendations will be jointly authored by the authors of this test.

Operational Physics
Research and Engineering
IRRADIATION PROCESSING DEPARTMENT

Process and Reactor Development
Research and Engineering
IRRADIATION PROCESSING DEPARTMENT

GF Bailey/RW Benoliel:gs

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APPENDIX I (1)

REACTIVITY LOSS RESULTING FROM THE LARGER NEUTRON ABSORPTION CROSS-SECTION OF N₂

The reactivity loss effected by replacing carbon dioxide with nitrogen in the reactor atmosphere can be estimated by assuming that the neutron absorption cross-section of the graphite is modified slightly, thereby affecting the relationship between neutrons lost to the moderator and those absorbed in uranium. The equation for this relation is:

$$\frac{dk}{k} = \frac{(\delta \Sigma_g)}{\Sigma_u \frac{V_u \bar{\phi}_u}{V_g \bar{\phi}_g} + \Sigma_g}$$

where: Σ = absorption cross-section, cm⁻¹,

$(\delta \Sigma_g)$ = variation in graphite cross-section due to the gas

V = volume

$\bar{\phi}$ = average flux

and the subscripts u and g refer to uranium and graphite. The values of the flux and volume ratios are assumed to remain constant; for K piles the following values are used:

$$V_u/V_g = 0.0285$$

$$\bar{\phi}_u/\bar{\phi}_g = 0.452$$

$$\Sigma_u = 0.364$$

$$\Sigma_g = 27 \times 10^{-5} \text{ (no gas in pile)}$$

$$\Sigma_g = 27 \times 10^{-5} + (\delta \Sigma_g) \text{ with gas in pile}$$

The variation in the graphite cross-section due to the presence of gas in the pile can be calculated in the following manner. The graphite stack has several void regions for rods, test holes, etc. These voids are assumed to be homogeneously distributed throughout the reactor. In addition to these voids the graphite bars contain interstitial voids. The interstitial void volume can be calculated from the difference in density of crystalline graphite (2.27 grams per cc) and reactor graphite (approximately 1.65 grams per cc). The voids are likewise assumed to be uniformly distributed throughout the stack. When these voids are filled with gas the apparent neutron absorption cross-section of the graphite is increased ($\delta \Sigma_g$). For the present test it is of interest to determine the reactivity effect of changing the gas from a 90% He - 10% CO₂ mixture to a 75% He - 25% N₂ mixture. The absorption cross-sections of the gases at standard temperature and pressure are assumed to be:

$$\Sigma_{\text{He}} = 0.019 \times 10^{-5}$$

$$\Sigma_{\text{CO}_2} = 0.009 \times 10^{-5}$$

$$\Sigma_{\text{N}_2} = 10.10 \times 10^{-5}$$

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It is further assumed that at a maximum graphite temperature of 625°C the flux weighted average temperature is 450°C. At this temperature and for atmospheric pressure the reactor contains only 0.4 as much gas as under standard conditions. For a total effective void volume of 30 per cent the effect of the He-CO₂ mixture is:

$$\begin{aligned}(\delta \Sigma_g) &= (30\%) (0.4) [0.9 (0.019) + 0.1 (0.009)] 10^{-5} \\ &= 0.00216 \times 10^{-5}\end{aligned}$$

Under the same reactor conditions the effect of the He-N₂ mixture would be:

$$\begin{aligned}(\delta \Sigma_g) &= (30\%) (0.4) [0.75 (0.019) + 0.25 (10.1)] 10^{-5} \\ &= 0.305 \times 10^{-5}\end{aligned}$$

The value of dk/k for this change in gas is -0.06% or approximately 60 c-mk.

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APPENDIX II

THE CORROSION PROBLEM (8)

If water containing dissolved nitrogen and either dissolved hydrogen or oxygen is in a reactor flux field, one of the following reactions can be expected (9).

Process water is nearly saturated with dissolved oxygen and more oxygen is formed in the reactor by radiolytic decomposition of water. Therefore, if nitrogen is used as an appreciable percentage of the reactor gas, the formation of nitric acid in water which has leaked into the stack is not improbable.

It is possible that in addition to the above aqueous reactions, the formation of NO_2 or NO would occur in the gas phase. Subsequently these gases may be absorbed by water to give nitric acid by:

It is necessary to insure that any nitric acid so formed does not result in significant additional damage to reactor components when water leaks do occur. Of course, considerable damage to process tubes and other components does occur even in the absence of nitrogen in the reactor gas.

The nitrate ion itself will not cause damage. The danger from nitric acid formation is that the water in the stack will attain such a low pH that corrosion is accelerated.

Fortunately, a considerable amount of nitric acid can be absorbed without a major change in pH because the process water is buffered by the equilibrium between dissolved CO_2 and bicarbonate ion:

As nitric acid is added to this system, most of the added hydrogen ions will combine with bicarbonate ions and be removed until the bicarbonate ions are nearly "used up". Acid additions beyond this point will result in a higher hydrogen ion concentration and, hence, lower pH's.

It is, of course, not possible to determine the degree of absorption of nitric acid in situ. Indirect measurement can be made. If a water leak is sufficient in size to result in water collection in the drip legs, the situation is relatively straightforward. One can assume that the nitrate content of water in the drip legs is quite similar to that of water in the stack. Analysis of the nitrate ion of water sampled

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from the drip legs can be compared to the nitrate content of plant process water. The difference, in moles/liter, is equivalent to the amount of absorbed nitric acid in moles/liter. If this pick-up is found to be, say no more than 70 percent of the bicarbonate ion content (both on the same mole basis) one can be assured that so little nitric acid was picked up that no extra damage is occurring.

If a leak is occurring and water is not being collected in the drip legs, the situation is less clear cut. In this case the water is removed by vaporization along with any "excess" nitric acid and no direct means for assessing the situation are available. However, an indirect, conservative approach is available. Conditions are much more conducive for the absorption of nitrogen dioxide in water in the silica gel driers than in the reactor. The driers are packed with silica gel crystals which absorb water in quite thin layers (perhaps a few molecules thick) with a resulting extensive exposed surface area of water. The degree of absorption of oxides from the reactor gas in this case will be more complete than in the reactor where the exposed water surface is lower. Therefore, if the concentration of nitrates in moles/liter in the condensate from the driers is no more than 70 percent of the bicarbonate ion content in the process water (in moles/liter) there is essentially nil likelihood that extra reactor damage is occurring.

A final case to be considered is the simultaneous occurrence of two or more leaks. Even if water collection in the drip legs is occurring, one may be monitoring only the larger leak if one depends on sampling from the drip legs. Therefore, even if water collection in the drip legs is occurring, a daily collection of 24 or more gallons per day of condensate from the driers should be a signal to relay on the more conservative approach, and monitor samples from the gas driers.

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Note: $\frac{1}{T}$ is a partial measure of the temperature sustained by the graphite at the corresponding per cent Helium

$\frac{1}{T}$ Thermal Resistivity, vs Per Cent Helium
at 500°K
(Data from HW-28766)

Figure 1

APPROVALS

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STEEL TEST COUPONS IN
NITROGEN-HELIUM REACTOR ATMOSPHERE

Ref: HW-66973 - PT-IP-358-AC - Replacement Of Carbon Dioxide
With Nitrogen As A Constituent Of The K Reactor Atmosphere,
by GF Bailey and RW Benoliel, dated October 3, 1960.

During the reactor outage of November 29, 1960, a graphite boat containing eight 1" x 1" x 1/16" thick steel coupons was placed in the 2-C test hole of the KE reactor. The graphite boat and samples are exposed directly to the reactor atmosphere. They are positioned in the test hole close to the side thermal shield.

The samples were inserted by KE Processing to provide an additional means of monitoring the nitrogen-helium atmosphere for its potential corrosion effect on steel. The nitrogen-helium atmosphere is now being tested at KE reactor in place of the carbon dioxide atmosphere now normally used at the other production facilities.

The initial test coupon will be removed during the first outage after January 18, 1961. Thereafter, coupons will be removed for examination at approximately two-month intervals until all coupons have been removed or until completion of the helium-nitrogen test, whichever comes first.

C. A. Mansius

C. A. Mansius
Assignment Supervisor
KE Processing Operation

CAM bf

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END