26

THERMAL STABILITY OF LX-09
CHEMICAL REACTIVITY TESTING

A. C. Teter

QUALITY DIVISION

SEPTEMBER 1971
FINAL REPORT

Normal Process Development
Endeavor No. 222

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Mason & Hanger-Silas Mason Co., Inc.

Pantex Plant

P. O. BOX 647
AMARILLO, TEXAS 79105
806-335-1581

operated for the
ATOMIC ENERGY COMMISSION
under

U. S. GOVERNMENT Contract DA-11-173-AMC-487 (A)

DISCLAIMER

**Portions of this document may be illegible
in electronic image products. Images are
produced from the best available original
document.**

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately-owned rights.

THERMAL STABILITY OF LX-09
CHEMICAL REACTIVITY TESTING

A. C. Teter

QUALITY DIVISION

The purpose of this project was to evaluate the thermal stability of LX-09.

Final Report
September 1971
Endeavor No. 222
Acct. No. 22-2-34-14-402

Section W

THERMAL STABILITY OF LX-09

CHEMICAL REACTIVITY TESTING

ABSTRACT

Several CRT tests were performed with LX-09 using sample containers obtained from LLL. These containers were used to help eliminate differences that were inherent in the design of the various sample containers used by the different laboratories. Initial experiments showed poor agreement in test data between LLL and Pantex containers until Pantex containers were fitted with different valves. Later experiments show no significant differences in test data between the two types of containers after the modification. Fillers of stainless steel (used in LLL containers) and of glass (used in Pantex containers) were interchanged without any significant differences in results. Results of tests with PBX 9404 and with LX-04-1 are included.

INTRODUCTION

The original purpose of this project was to evaluate the thermal stability of LX-09. Information on thermal stability has been obtained at LLL and these efforts have been directed toward obtaining uniform results of chemical reactivity testing (CRT) between LLL and Pantex. Previously, CRT testing between these laboratories has not been as uniform as desired. In some instances specifications of CRT requirements had to be spread somewhat in order to encompass the spread in results obtained from these laboratories. LLL requested that Pantex investigate CRT testing particularly with LX-09 and make recommendations to ensure the best possible agreement between the laboratories.

EXPERIMENTAL

The experiments were run under standard procedures outlined in RM 251080 using sub-ambient temperature programming and a Poropak Q Column 10 feet x 1/4 inch. A brief outline of this procedure is as follows:

One quarter gram of HE is weighed into a glass or stainless steel capsule and inserted into the sample container. A filler of glass rod or stainless steel rod is added to reduce the dead space volume inside the sample container. The air is then removed by evacuating, followed by a helium purge through the container for a few minutes before final sealing under about 30 psig helium pressure.

The sample container is then partially immersed in a temperature bath maintained at 120 C for 22 hours. The sample container is immersed to well above the sample capsule area.

When the heating period ends, the container is removed from the bath and placed on a special chromatograph inlet system designed for this purpose. Air is evacuated from the inlet system and the sample is introduced as a loop in the helium gas stream used as a carrier gas for the chromatograph.

The chromatograph oven is previously cooled to -90 C, using liquid nitrogen as a coolant. The oven heat is programmed at 20 degrees/minute to 200 C during the analysis. The peak areas were measured using a Datex digital integrator. Calibration was accomplished by checking the chromatographic response by the introduction of known amounts of respective high purity gases.

RESULTS AND DISCUSSIONS

Table I is a summary of results from testing of LX-09 using LLL sample containers. These data, collected over a period of several months, were considered good.

Table II shows data obtained from regular Pantex containers and indicates high nitrogen, oxygen content, and considerable fluctuations of other values. This was apparently caused by trapped air in the teflon sealed diaphragm valves being used at the time.

Table III shows results obtained with the Pantex containers after the diaphragm valves were replaced with Kel F^a seated needle valves. Tables I and III compare very favorably in quantity of gas evolved and the precision of the tests appear acceptable.

Table IV is the result of the same comparison tests with LX-04-1, again showing good agreement between the two types of containers.

Table V contains values obtained with PBX 9404 which are included for comparison purposes.

A series of experiments was performed to indicate variations that might be expected under less rigid temperature control. LX-09 samples were run at the temperature indicated using a Benco-Grant^b constant temperature bath with a temperature control range of ± 0.02 C (Table VI). The temperature control specifications were confirmed using a Hewlett Packard quartz thermometer with recorder. The table shows that a variation of the few degrees makes a considerable difference in the quantities of gases evolved.

The effects of enclosing the sample in the container under either higher or lower pressure than 30 psig are shown in Table VII. PBX 9404 was used as its larger gas evolution would tend to show greater differences than other PBX's.

It appears from the data that this parameter did not significantly affect values.

Finally the stainless steel inserts in the LLL containers were replaced with glass inserts (Pantex containers use glass) without any apparent change in results. Stainless inserts were also tried in the Pantex containers with the same results. The container configurations are shown in Fig. 1.

^a Whitey OKF-2 stainless steel needle valve

^b Benco-Grant high temperature thermostatic bath Model HB3X,
Osborn Engineering, Inc., Tulsa, Oklahoma

Fig. 1. Sample Containers

CONCLUSIONS AND RECOMMENDATIONS

It is now apparent that the cause of differences in CRT analyses between LLL and Pantex was the Teflon diaphragm valves being used on the Pantex sample container. No significant differences in values were obtained by changing space fillers and vials from glass to stainless steel. Very small differences were noted when the helium sample pressure was varied from 10 to 50 psig.

The largest problem was apparently due to entrapped air in the sample container system. Reproduction of the analyses shown should not be difficult when all sources of entrapped air are eliminated from the sample container system.

Variations of values overlapped when the temperature of the constant temperature bath was varied ± 3 C from the specified 120 C.

It is apparent, however, that best results should be obtained when the temperature is controlled to the ± 1 C required by the test specifications.

TABLE I. LX-09 WITH LLL SAMPLE CONTAINERS
(cc-STP)

<u>Container No.</u>	<u>N₂ + O₂</u>	<u>CO + NO</u>	<u>CO₂</u>	<u>N₂O</u>
30	0.011	0.026	0.015	0.006
32	0.010	0.023	0.016	0.006
30	0.005	0.017	0.013	0.006
32	0.011	0.015	0.012	0.004
32	0.007	0.022	0.014	0.005

TABLE II. LX-09 WITH STANDARD PANTEX CONTAINERS
(cc-STP)

<u>Container No.</u>	<u>N₂ + O₂</u>	<u>CO + NO</u>	<u>CO₂</u>	<u>N₂O</u>
3	0.016	0.015	0.018	0.005
9X	0.024	0.016	0.039	0.009
3	0.017	0.014	0.020	0.006
9X	0.016	0.006	0.014	0.005

TABLE III. LX-09 WITH MODIFIED PANTEX CONTAINERS
(cc-STP)

<u>Container No.</u>	<u>N₂ + O₂</u>	<u>CO + NO</u>	<u>CO₂</u>	<u>N₂O</u>
18	0.001	0.016	0.013	0.005
	0.005	0.012	0.011	0.004
	0.006	0.013	0.013	0.005
	0.006	0.011	0.012	0.004
	0.008	0.021	0.015	0.006
19	0.001	0.016	0.015	0.005
	0.007	0.015	0.016	0.005
	0.010	0.016	0.016	0.005
	0.006	0.020	0.016	0.005
12	0.011	0.024	0.021	0.007
	0.008	0.014	0.015	0.005
	0.007	0.019	0.016	0.005
	0.006	0.011	0.012	0.004
	0.005	0.015	0.015	0.005
	0.010	0.013	0.020	0.007

TABLE IV. LX-04-1
(cc-STP)

	(1)	(2)	(3)	(4)	LLL
N ₂	0.002	0.003	0.003	0.004	0.005
NO	-	-	0.002	-	-
CO ₂	0.009	0.003	0.005	0.002	0.006
N ₂ O	0.003	0.004	0.003	0.007	0.006
Total	0.014	0.010	0.013	0.013	0.017

TABLE V. PBX 9404 STANDARD
(cc-STP)

N ₂	0.018	0.019	0.024	0.022
CO	0.031	0.028	-	0.039
NO	0.204	0.198	0.221	0.219
CO	0.143	0.143	0.171	0.156
N ₂ O	0.017	0.018	0.025	0.024
Total	0.412	0.406	0.479	0.460

TABLE VI. LX-09 VARIATION OF TEMPERATURE

115 C, 22 Hrs. (cc)

	(1)	(2)	(3)	(4)	X	Range
N ₂	0.003	0.002	0.002	0.003	0.003	0.001
CO	-	-	-	-	-	-
NO	0.007	0.003	0.003	0.007	0.005	0.002
CO ₂	0.009	0.006	0.007	0.011	0.008	0.002
N ₂ O	0.002	0.002	0.002	0.002	0.002	
Total	0.021	0.013	0.014	0.023	0.018	0.004

117 C, 22 Hrs. (cc)

N ₂	0.006	0.014	0.005	0.006	0.008	0.004
CO	-	-	-	-	-	-
NO	0.022	0.018	0.031	0.009	0.020	0.008
CO ₂	0.010	0.018	0.018	0.008	0.013	0.004
N ₂ O	0.004	0.003	0.005	0.004	0.004	0.001
Total	0.042	0.053	0.057	0.027	0.045	0.011

123 C, 22 Hrs. (cc)

N ₂	0.009	0.011	0.007	0.009	0.009	0.001
CO	-	0.001	T	0.003	0.002	0.001
NO	0.024	0.023	0.016	0.022	0.02	0.003
CO ₂	0.016	0.021	0.017	0.017	0.018	0.002
N ₂ O	0.006	0.006	0.006	0.006	0.006	
Total	0.055	0.062	0.046	0.057	0.055	0.006

125 C, 22 Hrs. (cc)

N ₂	0.007	0.007	0.008	0.019	0.012	0.005
CO	0.001	0.001	0.001	0.002	0.001	0.001
NO	0.035	0.036	0.029	0.061	0.040	0.012
CO ₂	0.025	0.026	0.024	0.032	0.027	0.003
N ₂ O	0.009	0.009	0.007	0.010	0.009	0.001
Total	0.078	0.079	0.076	0.124	0.089	0.020

TABLE VII. PBX 9404 VARIATION WITH PRESSURE
(cc-STP)

	50 psig	50 psig	10 psig	10 psig
N ₂	0.016	0.010	0.011	0.012
CO	0.029	0.027	0.027	0.027
NO	0.188	0.183	0.187	0.184
CO ₂	0.138	0.130	0.134	0.129
N ₂ O	0.016	0.016	0.016	0.013
Total	<u>0.387</u>	<u>0.366</u>	<u>0.375</u>	<u>0.365</u>

REFERENCES

1. Interim Progress Report, Process Development Report No. 0012,
2. Thermal Stability of LX-09, October, 1970.
3. RM 251808 issue G., Chemical Reactivity Test.