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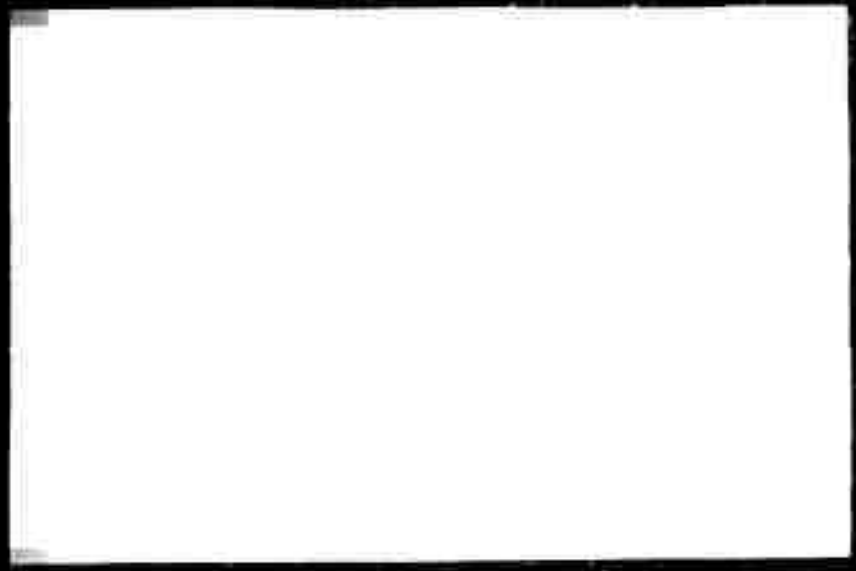
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**A PURITY ANALYSIS SURVEY OF  
CAPE CANAVERAL LIQUID OXYGEN**

**W. L. Ent and C. J. Sterner**

**AIR PRODUCTS, INCORPORATED  
Allentown, Pennsylvania**

**A. P. I. Research Project No. 03-9-2882**

**Contract No. AF 33(616)-6730**

**APRIL 1961**

**Directorate of Rocket Propulsion  
AIR FORCE FLIGHT TEST CENTER  
AIR RESEARCH AND DEVELOPMENT COMMAND  
UNITED STATES AIR FORCE  
Edwards Air Force Base, California**



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## ABSTRACT

Liquid oxygen, as generated and stored at Cape Canaveral, Florida, has been sampled and analyzed regularly for a period of three months. Samples were procured with approved cryogenic samplers from the liquid oxygen generation facility and from six pre-selected launch complex storage tanks. Analysis of the samples was performed at the Air Products Research and Development Laboratories in Allentown, Pennsylvania, and on occasion at the Patrick Air Force Base Laboratory.

Results of analyses indicate: a negligible build-up in contaminant concentration between the generation facility and the launch complex storage tanks; a loss of purity due to nitrogen pressurization at the launch complex storage tanks; and the presence of carbon dioxide above its solubility limit in all of the samples procured.

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## I. INTRODUCTION

The United States missile and space program requires the use of large quantities of liquid oxygen as a rocket propellant. Although commercial producers have been supplying liquid oxygen to various users for a number of years, the missile requirements for a high order of reliability have focused attention on the contaminants of liquid oxygen. An investigation of the nature and extent of contaminants in liquid oxygen was authorized by Contract No. AF 33(616)-6730, July, 1959, and has continued from that date.

LOX is handled considerably from the time of leaving the production plant to the time of its ultimate use. The extent of the increase in contaminant concentration in LOX in the course of its handling was not known. In order to obtain a more clear picture of the effect of the handling system on contamination, a program of sampling and analysis of liquid oxygen in the field was approved as part of the above contract. Accordingly, an investigation was undertaken at Cape Canaveral to sample and analyze the liquid oxygen in six ICBM complexes (four Atlas and two Titan) and at the LOX production facility at intervals during a three-month period.

The three-month period covered January, February, and March 1961.

## II. LOX HANDLING PRACTICE - CAPE CANAVERAL

The liquid oxygen used at Cape Canaveral is usually supplied by the LOX production facility operating at the Cape. This facility consists of two independent plants (1160 and 1161), each capable of producing 75 tons (15,700 gal.) of LOX per day, and four plant storage tanks, each with a capacity of 28,000 gallons. Generally, both plants operate at the same time, although during times of low demand, or when the plant storage tanks are full, one plant may operate while the other is either standing by or is being maintained. Occasionally, at times of peak demand, LOX may be brought in from an outside source.

When liquid oxygen is required at one of the complexes, tank trailers are driven to the production facility and loaded with LOX which is filtered as it is pumped from the plant storage tanks. Four trailers may be loaded at one time from the filter manifold, each trailer having a capacity of 3800 gallons. When the trailers are filled, they are driven to the complex area and unloaded into the pad storage tank, which has a capacity of 28,000 gallons. The trailers are unloaded by pressurizing the trailer tank with oxygen gas and forcing the contents into the pad tank through a portable filter unit.

Activities associated with the LOX storage tanks may consist of launches, static firing tests, incompletes or recycled countdowns, missile tanking tests, dumping the storage tank contents, filling and topping the storage tank, and taking samples for analysis. When a missile is scheduled to be launched, one of the last operations to be performed is loading the missile with LOX. About 30 minutes before the launch, the LOX storage tank is pressurized with nitrogen gas to about 30 psig to provide pump suction head and the liquid oxygen is pumped through filters into the missile tank. The missile tank is kept full by adding subcooled liquid oxygen to compensate for boil-off. If the count is held up after the LOXing operation, the missile tank is topped with LOX until the count is either continued, recycled, or cancelled. If the count is seriously delayed or cancelled, the LOX in the missile tank is either returned to the storage tank or discarded. Static firing tests follow the same procedure as the actual launches. Tanking tests are designed to test the LOX handling system by filling the missile tank. After a tanking test, the LOX in the missile tank may be returned to the storage tank or discarded. After a storage tank is dumped, generally because the LOX does not meet purity or particle count specifications, the tank is usually purged afterward with gaseous nitrogen. When a storage tank is topped, it is generally filled until the LOX overflows from the tank vent line.

### III. EXPERIMENTAL PROGRAM

Samples of liquid oxygen were taken periodically from the LOX storage tanks of four Atlas complexes (Pads 11, 12, 13, and 14) and two Titan complexes (Pads 19 and 20), as well as from the process stream of the LOX production facility at Cape Canaveral. These samples were then shipped to Air Products, Incorporated (API) at Allentown, Pennsylvania where they were analyzed for a variety of components. At times, duplicate samples were taken by Pan-American Airways Pad Engineering and analyzed at the Patrick AFB Laboratory. In this way, cross-checks were made of the validity of the analytical methods. Analyses at Allentown were performed on all samples by at least two different instruments. The first instrument was a dispersive-type infrared spectrophotometer (Beckman IR-4) which was used to determine carbon dioxide, methane, nitrous oxide, water vapor, and acetylene. The second instrument was a Multi-Contaminant Analyzer (MCA) developed by Air Products for WADD under Contract No. AF 33(616)-6747 and made available for the present study. This instrument used separate non-dispersive (Luft Principle) infrared optical benches for measuring the various contaminants, and a paramagnetic analyzer for measuring oxygen concentration. The contaminants determined by the MCA were total hydrocarbons (THC), carbon dioxide, carbon monoxide, acetylene, butane plus hydrocarbons, and water. Details of the analytical techniques are described in Section VII, "Discussion of Sampling and Analyses."

The sampling apparatus and sampling technique used in this study are discussed in Section VII and described in Appendix A of this report. The sampling apparatus used is capable of taking true liquid samples provided the sampling period is no longer than ten minutes. In order to cool the sampling lines and sampler quickly enough to stay within the time limit, it was necessary to pressurize the LOX storage tanks to 20 psig with gaseous nitrogen.

This requirement not only introduced additional nitrogen to the bulk of the LOX as a contaminant, but also complicated the taking of a sample since the vent valve and pressurization controls were located in the block-house of the pad.

The samples at the pads were taken from the LOX sampling line normally used to take routine samples. Until the present program, routine sampling had consisted of draining liquid oxygen from the storage tank via the

sampling line into an open-mouthed stainless steel vacuum bottle (4). In order to distinguish between samples taken with open flasks and those taken with the closed sampler, the former are called liquid samples and the latter are called cryogenic samples. Duplicate cryogenic samples were taken by splitting the sample stream through a tee connection and filling two identical samplers simultaneously. After a cryogenic sample was taken and the sampler valves were closed, the trapped sample was allowed to warm and vaporize. At ambient temperature, the pressure inside the sampler generally reached about 1000 psig.

No formal sampling schedule was followed, but samples were taken depending on the activities at the pads and the availability of support personnel. Efforts were made to obtain samples before and after particular activities such as missile tanking tests, launches, and storage tank toppings. It proved impossible, with the equipment available, to obtain successful samples from the trailers used to carry LOX from the production facility to the complexes.

#### IV. HISTORY OF ACTIVITIES ON COMPLEXES

In order to interpret the data obtained during the course of the study period, it is necessary to consider each pad separately. The following descriptions are intended to provide a general background for each pad without going into much detail on individual samples.

##### A. Pad 11 - Convair

At the start of the test program, this complex had been inactive for a number of months due to modifications. During this inactive period, the LOX storage tank had been cleaned and inspected, remaining in clean condition until February 2, 1961 when the tank was filled with liquid oxygen. From that date until the completion of the sampling program (March 28), activities concerning the LOX storage tank consisted of one missile tanking test and ten topping loads of LOX added to the storage tank. From February 16 through March 28, eight samples were taken from the tank and subsequently analyzed. Of these eight samples, one was taken one hour after completion of the tanking test, one was taken less than one hour after topping, and one was taken about 14 hours after topping. In the cases of the other five samples, there were no particular activities at the storage tank for at least 16 hours before taking samples.

##### B. Pad 12 - Convair

After a period of considerable activity, a missile was launched on January 23 and the complex was shut down for modifications. The liquid oxygen which remained in the storage tank after the launch (approximately 11,000 gallons) was allowed to boil away by normal heat leak during the remainder of the test period, reaching a residual level of approximately 1400 gallons on March 29. Five samples were taken at the LOX storage tank during this inactive two-month period. During the active period, one sample was taken on a normal day and one sample was taken seven hours before the launch. The storage tank was topped three times during the active period.

**C. Pad 13 - Convair**

This complex was the most active of the six pads studied; four missiles were launched during the course of the study period. This activity is reflected by the 16 LOX tank loadings which occurred during the test period. Altogether, ten samples were taken from the LOX storage tank and analyzed. Of these samples, two were taken about five hours after a tank topping and one was taken about one hour after topping. No storage tank activities took place at least 24 hours before taking any of the other seven samples.

**D. Pad 14 - Convair**

Only one missile was launched from this complex during the three-month period. Other activities at the LOX storage tank during this period included one missile tanking test and seven loads of LOX delivered to the storage tank. Also, the contents of the LOX storage tank were discarded once, since the LOX did not meet specifications. A 24-hour purge with nitrogen gas followed the dumping of the LOX. Ten samples were taken during the study period and analyzed. Of the ten samples, two were taken about one hour after topping the storage tank. The other eight were taken at times of no particular activity.

**E. Pad 19 - Martin**

This complex was almost as active as Pad 13; three missiles were launched and one static firing test was conducted. The LOX tank was loaded on 12 occasions and nine samples were taken during the course of the study period. Of the nine samples, one was taken about one hour after topping, one was taken about three hours after topping, and one was taken 18 hours after a launch. There was no particular activity at least 24 hours before taking any of the ten other samples.

**F. Pad 20 - Martin**

Although more active than Pad 14, this complex was not as active as Pad 19, launching two missiles during the three-month period. Eight loads of LOX were added to the storage tank and eight samples were taken. No particular activities were associated with any of the eight samples.

### **G. LOX Plant**

With the exception of some problems associated with mal-functioning of the CO<sub>2</sub> filters and adsorbers in these plants, both plants operated at normal production rates as controlled by the available plant site storage capacity.

## **V. RESULTS**

The chronology of tank loadings, samples, and special activities during the three-month study period is presented in Table I. In this table, the left-hand column under each pad number presents the individual sample numbers. The center column indicates special activities which are represented by letters; "L" standing for launches, "T" for tanking tests, "D" for dumping LOX, and "S" for static firing. The right-hand column presents LOX tank loadings in thousands of gallons. Under the designation LOX Plant, the left-hand column refers to the sample numbers and the number in parentheses indicates the LOX plant which was sampled.

Three of the 68 samples were completely unsuccessful. The complete analytical data of the 65 analyzed samples are presented in Appendix B, Tables B-1 through B-7. For the sake of clarity, the accumulated data presented in Appendix B have been interpreted and condensed into Table II. In this table, the CO<sub>2</sub> data were obtained from the multi-contaminant analyzer.

Table III gives the analytical results of the samples which were taken shortly after some activity involving the sampled storage tank had taken place.

Table IV presents the results of all of the duplicate samples which were taken. Sample 59 in Table IV is the single sample which was analyzed by both Air Products and Patrick AFB Laboratories.



TABLE I - CHRONOLOGY OF STORAGE TANK ACTIVITIES

(Continued)

Date	LOX Plant	PAD 11			PAD 12			PAD 13			PAD 14			PAD 19			
		Samp. No.	Spec. Act.	Load. gal/1000	Samp. No.	Spec. Act.	Load. gal/1000	Samp. No.	Spec. Act.	Load. gal/1000	Samp. No.	Spec. Act.	Load. gal/1000	Samp. No.	Spec. Act.	Load. gal/1000	
2/3	20(1161)																
2/6						33.7											
2/7																	
2/8																	1.1
2/9																	
2/10																	0.5
2/13																	
2/14	25(1161)			12.2													
2/15																	
2/16																	
2/17	31(1161)																
2/20																	
2/21																	
2/23	36(1160)																
2/24																	
2/27	39(1161)																
3/1																	
3/2																	
3/3																	
3/6																	

TABLE I - CHRONOLOGY OF STORAGE TANK ACTIVITIES

(Continued)

Date	LOX Plant	PAD 11		PAD 12		PAD 13		PAD 14		PAD 19		PAD 20	
		Samp. No.	Spec. Act.	Samp. No.	Spec. Act.	Samp. No.	Spec. Act.	Samp. No.	Spec. Act.	Samp. No.	Spec. Act.	Samp. No.	Spec. Act.
3/7						9.7		42					
3/8				44				43					
3/9	45 (1161) 46		8.6										
3/10						3.4							
3/11						32.6							
3/13		48	3.8		L			47					
3/14			7.7							49		50	
3/15	52(1160)	51				25.9					1.8		
3/16		59		54									
3/17			5.4					55					15.2
3/20	56(1160)	57				32.9							
3/21										60			
3/22			8.5										
3/23											1.5		
3/25					L(?)								
3/27			1.5										
3/28		63				26.1			10.8	61			
3/29	66(1161)			65				68				67	
3/31											L		

TABLE II

SUMMARY OF ANALYTICAL RESULTS

Location (Pad or Plant)	Date Sampled	Sample	CO <sub>2</sub> (ppm)	THC (ppm)	CH <sub>4</sub> (ppm)	C <sub>2</sub> H <sub>2</sub> (ppm)	C <sub>4</sub> <sup>+</sup> (ppm)	Purity (Mol %)	LOX Volume (gallons)
11	2/16	1	17	14	15	-	-	99.75	F *
11	2/23	2	39	17	17	-	-	99.78	F
11	3/6	3	25	20	22	-	-	99.55	26,500
11	3/13	4	11	16	15	trace	-	99.45	F
11	3/15	5	10	16	16	-	-	99.53	F
11	3/16	6	15	22	19	-	-	99.45	F
11	3/20	7	10	17	16	-	-	99.35	F
11	3/28	8	12	20	20	-	-	99.45	F
12	1/10	1	14	13	18	-	-	99.70	-
12	1/23	2	13	20	17	-	-	99.40	F
12	2/2	3	8	13	15	-	-	98.95	8,500
12	2/16	4	14	18	21	-	-	99.45	4,600
12	3/8	5	11	24	27	-	-	99.73	2,200
12	3/16	6	7	31	31	-	-	99.73	2,100
12	3/29	7	10	43	45	-	-	99.60	1,400

\* (F denotes Full Tank)

TABLE II (continued)

Location (Pad or Plant)	Date Sampled	Sample	CO <sub>2</sub> (ppm)	THC (ppm)	CH <sub>4</sub> (ppm)	C <sub>2</sub> H <sub>2</sub> (ppm)	C <sub>4</sub> <sup>+</sup> (ppm)	Purity (Mol %)	LOX Volume (gallons)
13	1/16	1	13	16	17	-	-	99.30	F
13	1/23	2	14	19	18	-	-	99.30	F
13	1/26	3	15	19	16	-	-	99.40	Low
13	2/9	4	9	16	14	-	-	99.40	22,700
13	2/14	5	11	16	15	-	-	99.40	22,300
13	2/20	6	43	16	16	-	-	99.50	22,000
13	2/27	7	15	15	16	-	-	98.40	5,000
13	3/8	8	17	14	16	trace	-	99.50	F
13	3/15	9	25	21	23	-	-	99.77	F
13	3/20	10	13	14	15	-	-	99.75	F
14	1/17	1	24	54	30	-	-	99.40	--
14	1/25	2	35	32	25	-	-	99.60	F
14	2/1	3	14	19	21	-	-	99.60	F
14	2/9	4	22	22	20	-	-	99.60	21,800
14	2/17	5	15	16	15	-	-	99.70	F
14	2/28	6	14	17	17	-	-	99.50	5,000
14	3/7	7	20	16	16	trace	-	99.70	23,000
14	3/13	8	14	16	16	trace	-	99.60	21,500
14	3/17	9	20	20	20	trace	-	99.60	20,500
14	3/24	10	11	17	15	-	-	99.70	F

TABLE II (continued)

Location (Pad or Plant)	Date Sampled	Sample	CO <sub>2</sub> (ppm)	THC (ppm)	CH <sub>4</sub> (ppm)	C <sub>2</sub> H <sub>2</sub> (ppm)	C <sub>4</sub> <sup>+</sup> (ppm)	Purity (Mol %)	LOX Volume (gallons)
19	1/18	1	18	24	20	-	-	99.50	F
19	1/25	2	16	15	13	-	-	99.40	F
19	2/3	3	11	13	15	-	-	99.50	27,200
19	2/13	4	11	18	15	-	-	98.60	5,500
19	2/16	5	25	10	13	-	-	98.60	27,800
19	2/21	6	37	19	16	-	-	98.90	10,300
19	3/14	7	12	18	16	0.25	-	99.40	27,200
19	3/21	8	10	19	17	0.13	-	99.50	F
19	3/28	9	12	18	17	0.16	-	99.50	F
<hr/>									
20	1/19	1	20	14	18	-	-	99.70	--
20	1/27	2	17	19	17	-	-	99.40	--
20	2/2	3	11	13	13	-	-	99.50	F
20	2/13	4	12	22	18	-	-	98.60	<4,000
20	2/20	5	16	20	20	-	-	98.80	<4,000
20	2/28	6	23	15	16	-	-	98.00	25,000
20	3/14	7	16	17	17	-	-	98.6	7,600
20	3/29	8	17	16	17	-	-	99.50	25,000

TABLE II (continued)

Location (Pad or Plant)	Date Sampled	Sample	CO <sub>2</sub> (ppm)	THC (ppm)	CH <sub>4</sub> (ppm)	C <sub>2</sub> H <sub>2</sub> (ppm)	C <sub>4</sub> <sup>+</sup> (ppm)	Purity (Mol %)	LOX Volume (gallons)
1160	1/6	1	3	10	11	-	-	99.80	--
1160	1/19	2	61	7	12	-	-	99.70	--
1160	1/27	3	63	7	15	-	-	99.70	--
1161	2/3	4	110	3	11	-	-	99.60	--
1161	2/14	5	74	80	22	-	-	99.70	--
1161	2/17	6	52	16	12	-	-	99.70	--
1160	2/23	7	36	15	14	-	-	99.90	--
1161	2/27	8	>100	15	15	-	-	99.70	--
1161	3/9	9	25	12	13	-	-	99.60	--
1161	3/9	10	24	12	13	-	-	99.60	--
1160	3/15	11	47	13	16	-	-	99.70	--
1160	3/20	12	50	13	14	-	-	99.50	--
1161	3/29	13	10	12	12	-	-	99.60	--

**TABLE III**

**SAMPLES ASSOCIATED WITH STORAGE TANK ACTIVITIES**

Location (Pad)	Date Sampled	Activity	Sample Time *	Analysis		
				CO <sub>2</sub> (ppm)	Methane (ppm)	Purity (Mol %)
11	3/6	Tanking Test	1	25	22	99.55
11	3/13	Topping	1	11	15	99.45
11	3/15	Topping	14	9.5	16	99.53
13	1/16	Topping	5	13	17	99.30
13	3/15	Topping	1	25	23	99.77
13	3/20	Topping	5	13	15	99.75
14	2/1	Topping	1	14	21	99.55
14	2/17	Topping	2	14.5	15	99.65
19	1/18	Topping	1	17.5	20	99.45
19	1/25	Topping	3	15.5	13	99.40

\* Hours after activity.

TABLE IV

COMPARISON OF DUPLICATE SAMPLES

API\* and PAFB\*\*

Location (Pad)	Date Sampled	Sample No.		CO <sub>2</sub> (ppm)		Methane (ppm)		Purity (Mol %)	
		API	PAA***	API	PAFB	API	PAFB	API	PAFB
14	2/1	16	294	14	16	21	21	99.55	99.60
20	2/2	17	307	10.5	11	13	9	99.5	99.55
14	2/9	21	365	22	1.5	20	7	99.55	99.3
14	2/17	30	496	15	4	15	4	99.65	99.65
13	3/8	43	650	17	6	15.5	4.5	99.5	99.4
11	3/13	48	691	11	0	15	4	99.45	99.6
11	3/15	51	726	9.5	5	16	7	99.53	99.4
13	3/15	53	734	25	12	22.5	0	99.77	99.7
11	3/20	57	775	10	4	16	4	99.35	99.4
13	3/20	58	778	13	11	14.5	4	99.75	99.75
11	3/16	59	743	15	5	19	3	99.45	99.5

\* Air Products, Incorporated

\*\* Patrick Air Force Base

\*\*\* Pan American Airways

## VI. DISCUSSION OF RESULTS

### A. Carbon Dioxide

As can be seen in Table II of the results, in practically every sample the  $\text{CO}_2$  was found to be in quantities well above its solubility limit of 4.2 ppm in liquid oxygen. This indicates that solid  $\text{CO}_2$  was suspended in the LOX and was taken as part of the liquid sample. The  $\text{CO}_2$  concentrations found in the samples from the complexes were in the range from 10 to 40 ppm with the average around 20 ppm. In contrast, the  $\text{CO}_2$  found in the samples from the LOX production unit averaged higher than 50 ppm, with a few samples out of the range of the analytical instruments.

The high  $\text{CO}_2$  obtained from the LOX production unit can be traced to some extent to damaged filters in both LOX plants. These filters were in the process of being repaired during this program and the success of the repair effort can be seen in the last two samples from the 1161 LOX plant, with  $\text{CO}_2$  contents of 9.5 ppm and 24 ppm. (See Table B-7).

Although the sampling of suspended solids is far from exact, the relative uniformity of the  $\text{CO}_2$  analytical results indicate that the suspended solid  $\text{CO}_2$  must have been distributed quite uniformly in the bulk of the oxygen liquid. However, it must also be pointed out that the  $\text{CO}_2$  results given in Table II should not be taken as a true measure of the  $\text{CO}_2$  inventory, since considerable solid  $\text{CO}_2$  probably settled in the tanks. This point is illustrated by Figure 1 where the  $\text{CO}_2$  concentration and the residual liquid oxygen volume at Pad 12 are plotted as functions of time. The LOX in this storage tank was allowed to boil away undisturbed for a period of 65 days. Although there was no chance for any significant amount of the original  $\text{CO}_2$  to leave the system, the apparent  $\text{CO}_2$  concentration hardly changed while the volume of liquid oxygen decreased by a factor of eight. This indicates that the excess solid  $\text{CO}_2$  settled to the bottom of the tank.

### B. Hydrocarbons

The results from the hydrocarbon analyses, which included total hydrocarbons (THC), methane, acetylene, and butane and heavier hydrocarbons ( $\text{C}_4+$ ), present a picture of relatively uniform and almost insignificant hydrocarbon contamination, both at the LOX plants and at the active pads. With the exception of acetylene, the only other hydrocarbon detected was methane, the least hazardous of the hydrocarbons. There was only a

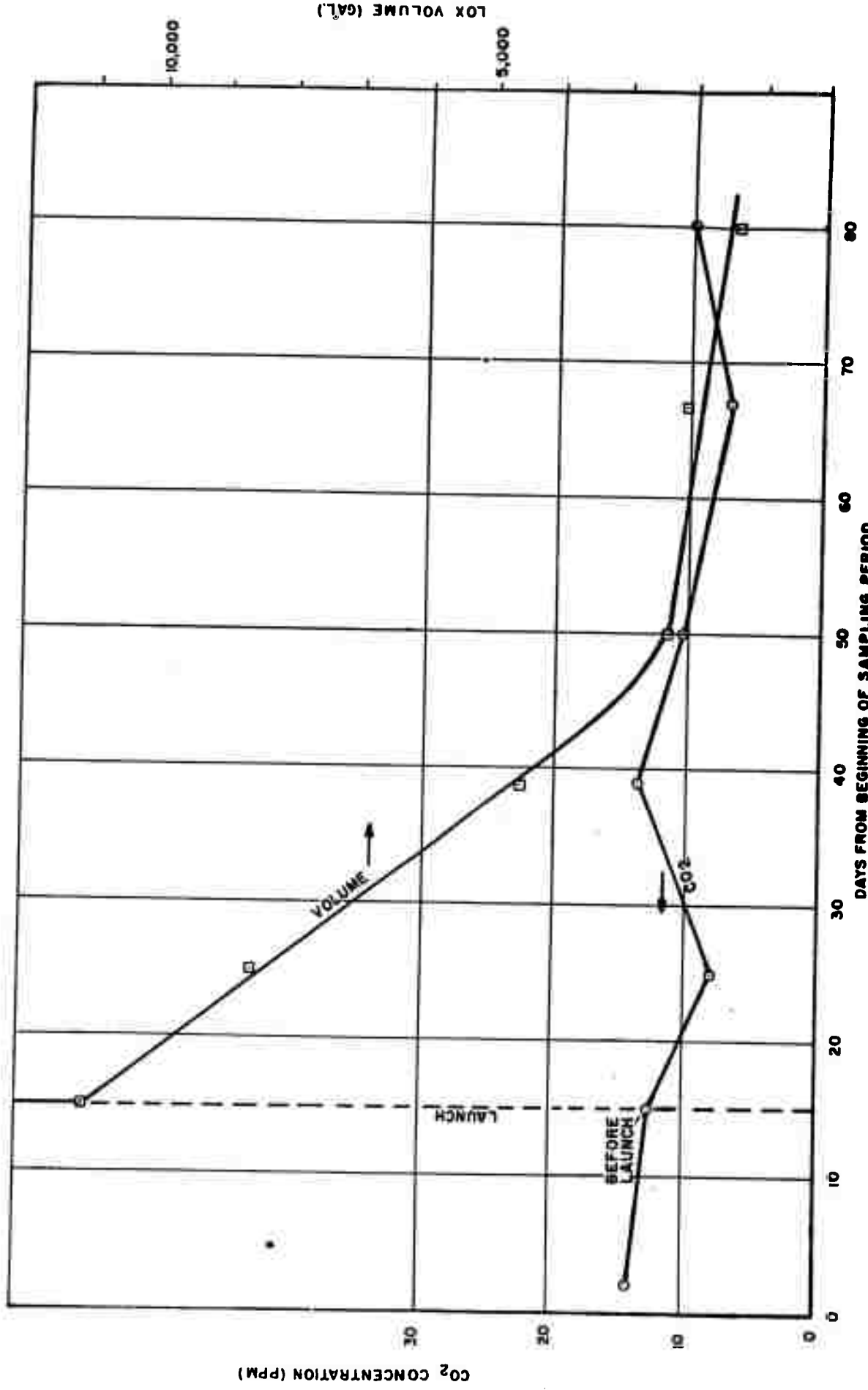


FIGURE 1. CO<sub>2</sub> CONCENTRATION PAD 12

CO<sub>2</sub> CONCENTRATION (PPM)

VOLUME ↑

LAUNCH

BEFORE LAUNCH

CO<sub>2</sub>

small (approximately 25%) increase of the methane concentration from the production plant to the final use of the LOX in a missile. The average concentration of methane was 13.5 ppm from the plant and 17.1 ppm from the active pads.

However, this was not the case for the inactive storage tank at Pad 12, as shown in Figure 2. In this figure, the hydrocarbon concentration and the residual liquid oxygen volume are plotted as a function of time. The last five samples were taken while the LOX was boiling off undisturbed. During this period, the volume of LOX decreased by a factor of eight, while the concentration of hydrocarbon (methane) increased only by a factor of three. Although a simple extrapolation of the hydrocarbon curve would lead one to expect that the methane concentration would not reach dangerous levels by the time all of the LOX was evaporated, the upward break toward the end of the curve shows that the methane rate of concentration may increase rapidly toward the end of the evaporation period. This would mean that the last few gallons of LOX to boil away could contain dangerous concentrations of methane, as has been predicted in a previous report (1).

With the exception of acetylene, no evidence was found of any hydrocarbons heavier than methane in the samples. It is not likely that any of the heavier hydrocarbons such as lubricants would be detected in the samples even if they were present in the LOX since the solubilities of these hydrocarbons in liquid oxygen are so slight as to be undetectable by the analytical techniques used. There is no simple technique currently available for monitoring insoluble hydrocarbons in liquid oxygen.

### C. Purity

There was no conclusive pattern to the results obtained from the purity analyses of the samples as shown in Table II. All samples from the LOX production unit proved better than specifications (99.5%). In comparison, half of the samples from the complexes were below specifications for purity. It is reasonable to assume that the impurity was principally nitrogen and that it resulted from the technique of pressurizing the LOX tanks with nitrogen gas. It is interesting to note that Pad 14 was consistently above specifications, while Pad 20 was consistently below specifications.

Figure 3 shows the pattern of oxygen purity in the tank at Pad 12 as the LOX slowly boiled away. It is probable that the residual oxygen purity in the tank shortly after launch was below specifications due to nitrogen pressurization; this was followed by preferential nitrogen evaporation during the long boil-off period. This pattern has also been predicted in a previous report (2).

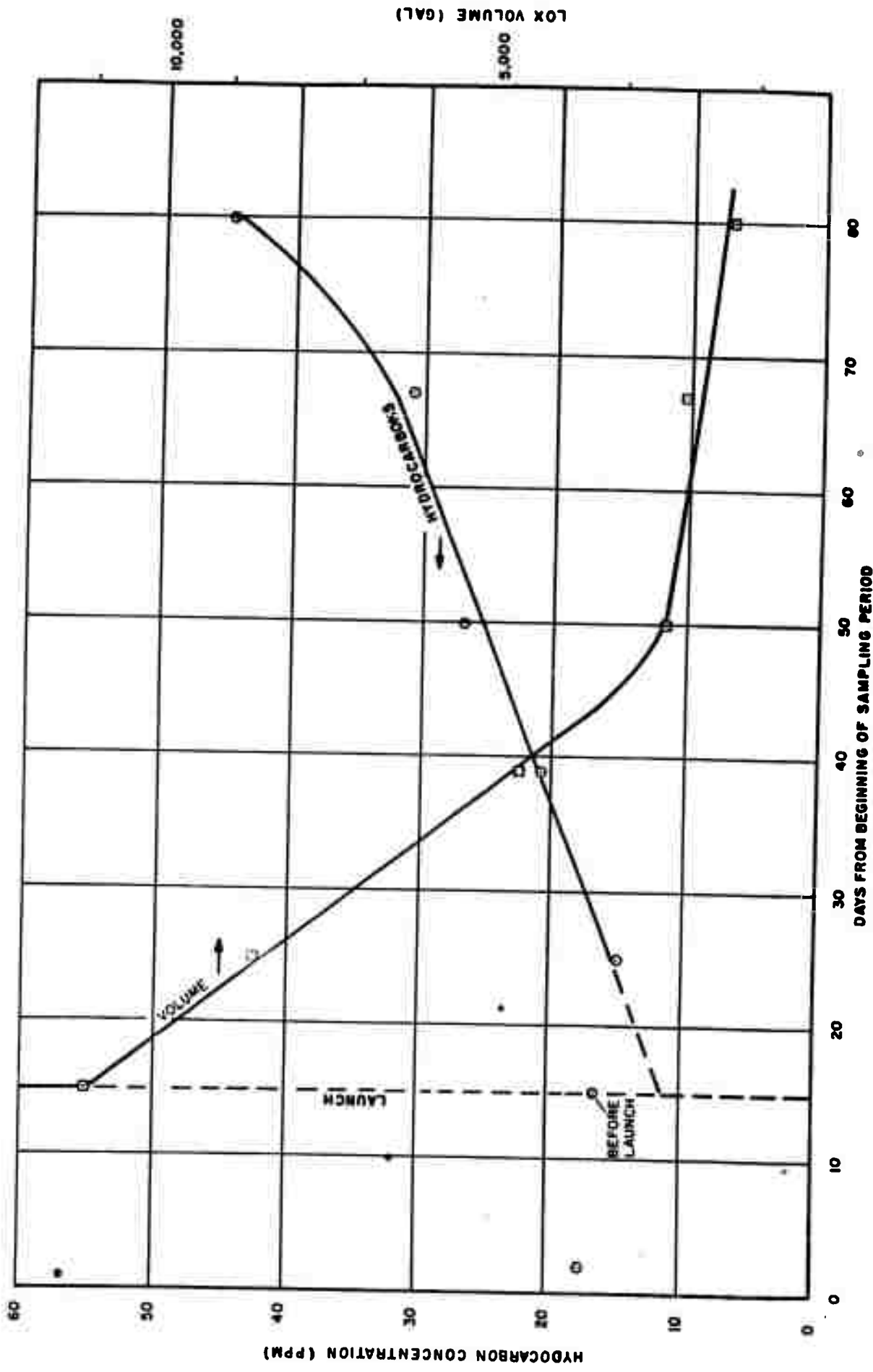


FIGURE 2. HYDROCARBONS & VOLUME PAD 12

(179) 3MHT07A 07

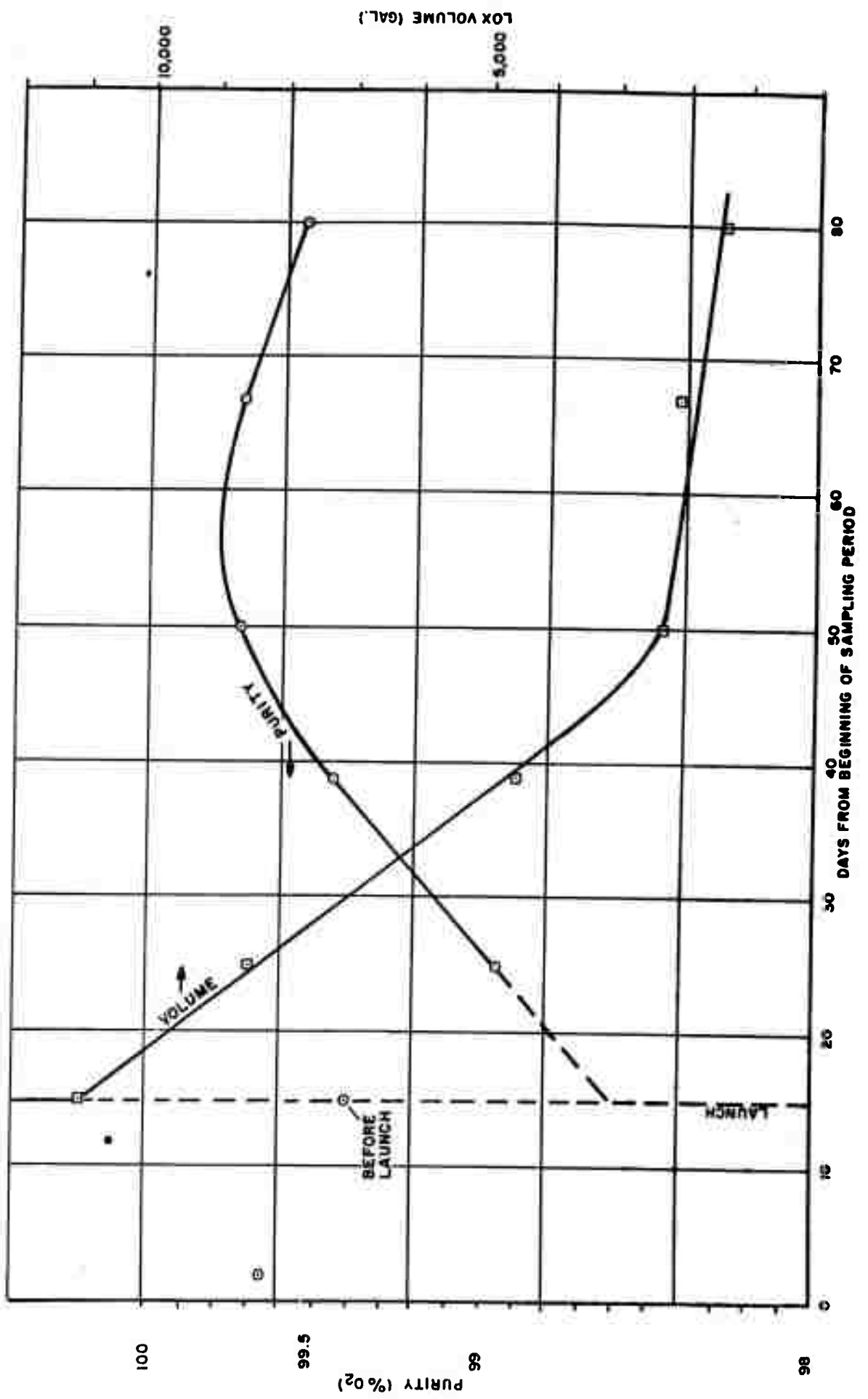


FIGURE 3. PURITY & VOLUME PAD 12

**D. Effect of Activities - Storage Tanks**

Relatively few samples were obtained following activities which involved the storage tanks. Examination of the results (Table III) shows little difference between these samples and the normal run of samples. However, a slight statistical trend shows that the LOX in a storage tank will be at its highest purity shortly after topping the tank.

**E. Comparison of Analytical Results**

Table IV presents direct comparisons between the results obtained at Air Products and the results obtained with duplicate samples at Patrick AFB Laboratory. The last sample (API No. 59) is the single sample which was analyzed by both laboratories. In general, the results between the two laboratories disagreed considerably in the analyses of CO<sub>2</sub> and methane. However, there was fairly good agreement between the purity analyses of the two laboratories.

## VII. DISCUSSION OF SAMPLING AND ANALYSES

### A. Sampling Techniques

The samples were procured with the model A-3-A cryogenic liquid batch sampler described in Appendix A. This sampler operates on the trapped liquid-vaporization technique (4) and was developed by Air Products, Incorporated for the purpose of sampling and vaporizing cryogenic liquids from a process stream or storage container. The Air Force (Edwards Air Force Base Laboratories) has also developed a cryogenic liquid batch sampler and prior to the commencement of this program, it had been suggested that the A-3-A sampler be compared with the Air Force sampler at the Edwards Laboratories. In December 1960, the A-3-A sampler was tested at Edwards AFB and it was found that the results obtained with this sampler were equivalent to the results obtained with the Air Force sampler, provided sufficient pressure could be exerted on the liquid in the tank to fill the sampler to overflowing within ten minutes. In subsequent tests, the necessary tank pressure was found to be approximately 20 to 25 psig.

All of the samples collected from the tanks and from the plant at Cape Canaveral were collected in accordance with this pressurization provision. In the plant, samples were removed in all cases from the product oxygen being transferred from the plant to the plant storage facilities. The solid matter present in this liquid stream is well dispersed because of turbulent flow. In contrast, the degree of mixing of the liquid in the pad storage tanks depended on heat leak of the tank, previous activity, and other factors (4). Gaseous nitrogen was, in most cases, used for pressurizing the pad storage tanks. On the basis of previous tests and studies (2), as well as the present test program, it can be concluded that the LOX purity at the pad tanks was lower than the purity of the plant product due primarily to this nitrogen pressurization.

### B. Analytical Techniques

The results of the analyses as performed in the API laboratory are depicted in Appendix B, Tables B-1 through B-7. Analyses were performed by both an infrared spectrophotometer with a long path (10 meter) cell and with the Multi-Contaminant Analyzer developed for the Wright Air Development Division under Air Force Contract AF 33(616)-6747 (5) and utilized on this portion of this contract at the discretion of WADD.

The samplers which were shipped by Air Express from Cape Canaveral arrived with the sample pressure generally above 1000 psig. Normally, the initial analysis of the sample at the Air Products Laboratory was performed on the infrared spectrophotometer. After proper purging or evacuation procedures, a portion of the sample was introduced to the 10-meter cell at a pressure of 10 atmospheres (approximately 135 psig). The sample was then scanned in the infrared range from 15 to 2 microns and the spectrum was compared with calibration spectra to determine qualitatively and quantitatively the components present. The spectrophotometer was calibrated quantitatively for carbon dioxide, nitrous oxide, acetylene, and methane. The sensitivities of these components at this particular pressure and sample cell length were 1.0 ppm for carbon dioxide, 0.2 ppm for nitrous oxide, 0.05 ppm for acetylene, and 2.0 ppm for methane.

The samplers were then moved to the laboratory where the MCA was located, and an independent analysis was obtained for the constituents as listed under MCA in the tables. These constituents were: total oxygen content, carbon dioxide, acetylene, water vapor, total hydrocarbons, carbon monoxide, and butanes-plus fraction of the total hydrocarbons. A complete description of the MCA is contained in ASD Technical Report 61-197 (5). Briefly, the oxygen purity is determined by a para-magnetic instrument contained within the MCA. Carbon dioxide, acetylene, water vapor, carbon monoxide, and butanes-plus are determined in Luft Principle infrared optical benches. The total hydrocarbons in the oxygen stream are determined by converting them to carbon dioxide by means of a catalyst furnace and analyzing for the carbon dioxide in a Luft Principle infrared optical bench. The determination of butanes-plus hydrocarbons requires a vapor phase chromatographic column to accomplish the C<sub>3</sub>- to C<sub>4</sub>+ separation prior to this determination in the infrared optical bench. Sensitivities for the various analyses in this analyzer depend on the available sample pressure. However, with typical operation at a 44 psig sample pressure, the sensitivities were: carbon dioxide, 0.25 ppm; acetylene, 0.06 ppm; water vapor, 0.5 ppm; total hydrocarbons, 0.25 ppm; carbon monoxide, 0.5 ppm; and butanes plus hydrocarbons, 0.25 ppm. the accuracy of the para magnetic unit in the 95 to 100 per cent range is  $\pm 0.05$  mole per cent.

As the course of the analyses by these two different techniques progressed, the results showed several differences. As these differences became evident, investigations were undertaken to determine the reasons for these differing results. It was not possible to re-analyze the samples to study any differences

in the results of analyses by the two techniques, since only a total of 20 samplers were used in the program. In order to obtain the maximum number of samples, these samplers remained in the laboratory only for a period of time sufficiently long for analyses to be performed. The samplers were purged, cleaned, pressurized, and returned to Cape Canaveral to be used in obtaining additional samples.

In order to better explain these analytical differences, a discussion follows of each component which was determined and the analytical problems which were involved in its determination.

### 1. Oxygen Content (Purity)

No difficulties were encountered with this analytical technique. The MCA was standardized and zeroed with the appropriate span and zero gases prior to analyzing each batch of samples. As a check, the analyzer was spanned and zeroed at the conclusion of each series of analyses. Span and zero gases were checked and analyzed using a standard Orsat apparatus with a specially calibrated pipette for increased accuracy and sensitivity.

When it was initially determined that some of the analyses from the pad tanks had oxygen purities below the 99.5 mole per cent requirement, check analyses were performed. Several samples which had indicated low purity when analyzed with the MCA were checked with a standard Orsat apparatus and with a separate paramagnetic instrument. The analytical results from these three techniques agreed within 0.05 mole per cent.

### 2. Carbon Dioxide

The analysis for carbon dioxide was performed by the MCA and infrared spectrophotometer. During the course of analysis for this component, two problems were discovered which were associated with the infrared spectrophotometric technique.

- a. In early analyses, concentrations indicated by the infrared spectrophotometer were consistently lower than those indicated by the MCA. It was determined that nitrogen absorbs infrared radiation at high pressure (10 atmospheres) due to the energy of inter-molecular reactions or collisions (3). Figures 4 and 5 show two spectra of high

Infrared Absorption Spectrum of High Purity N<sub>2</sub>; Cell Length = 10 m  
Sample Pressure = 10 Atm.

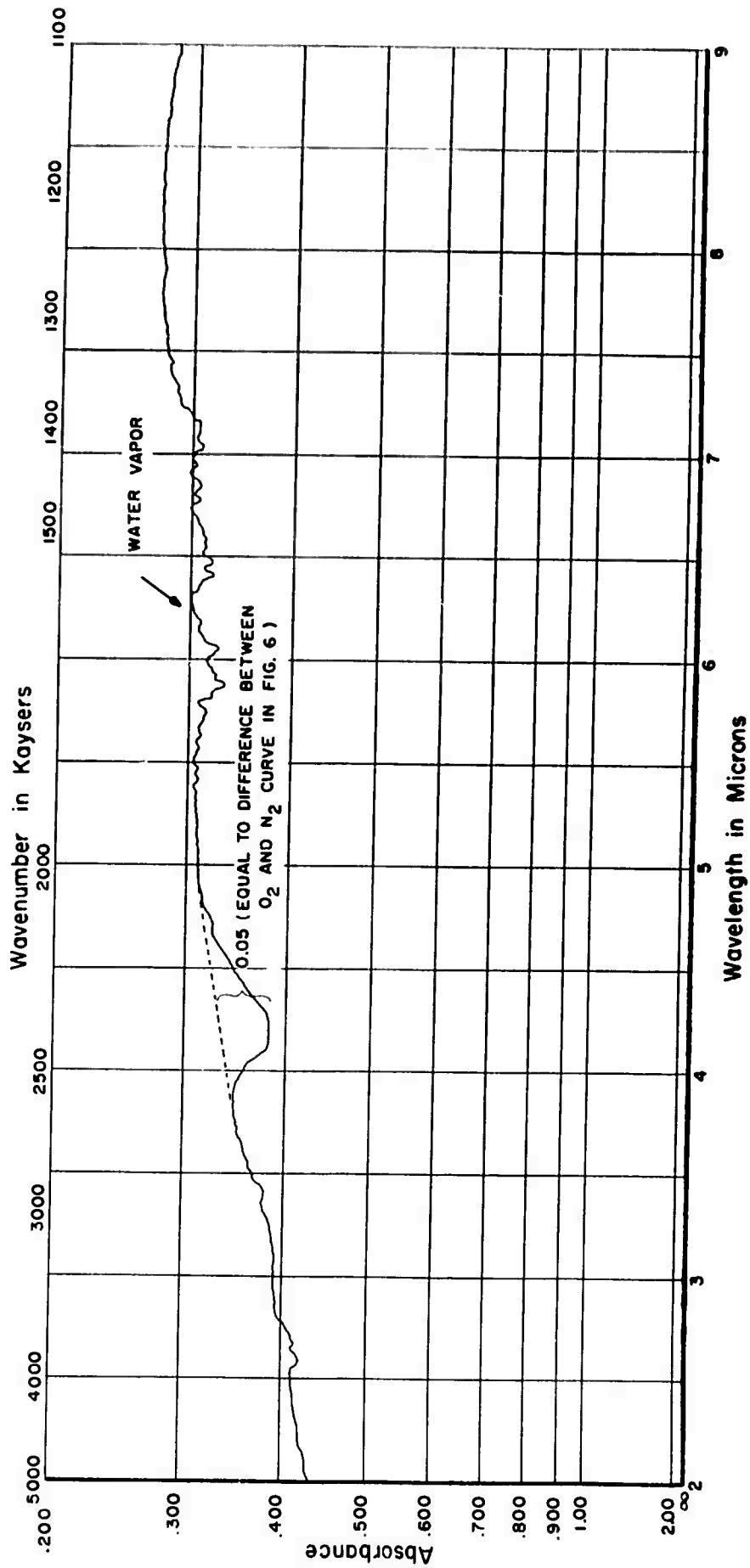


Figure 4

Infrared Absorption Spectrum of High Purity N<sub>2</sub>; Cell Length = 10 m  
Sample Pressure = 1 Atm.

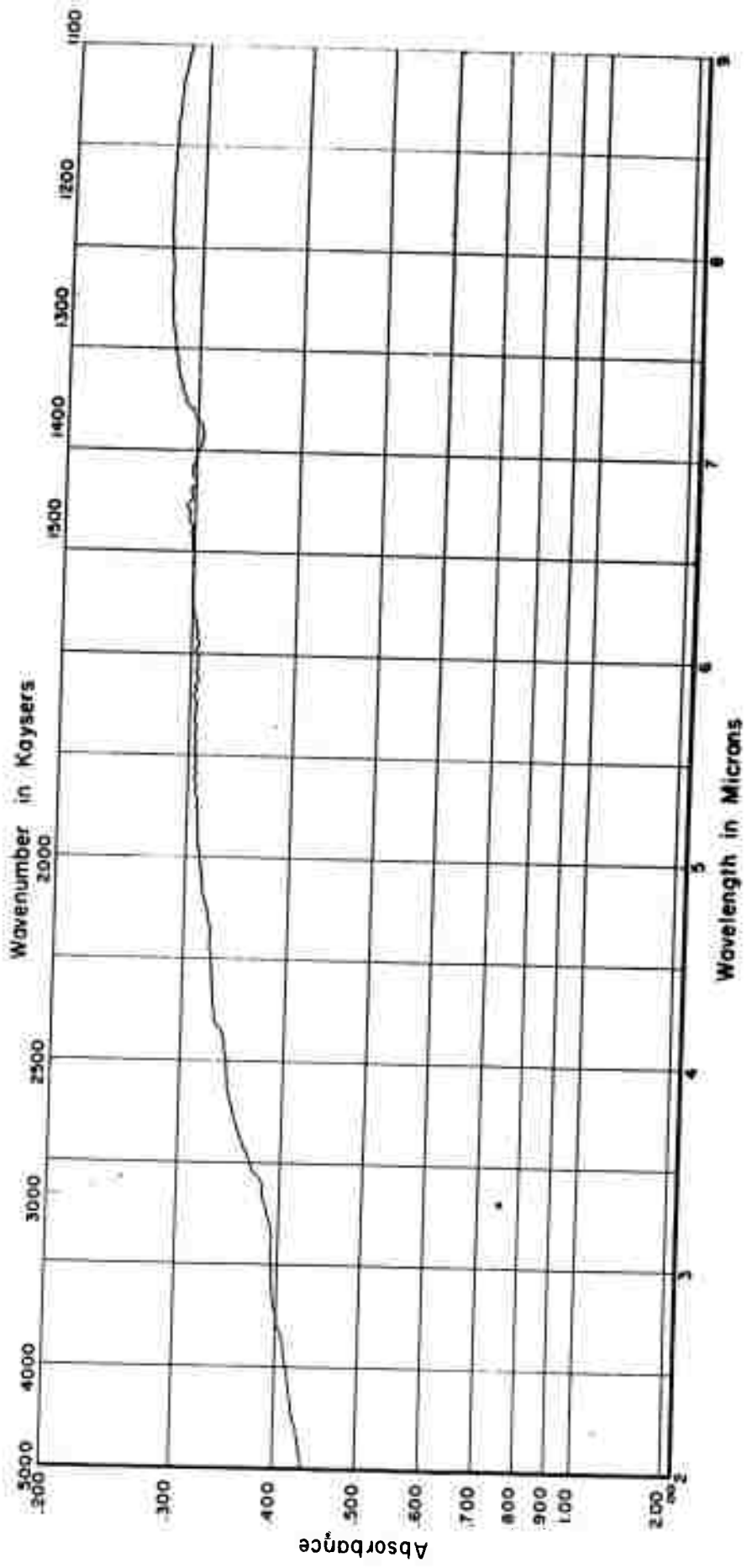


Figure 5

purity nitrogen, one at zero psig and the other at 135 psig (10 atmospheres). The nitrogen absorption band is apparent at 4.22 microns. The standards for the calibration of the infrared spectrophotometer for carbon dioxide analysis were prepared as CO<sub>2</sub> in nitrogen and the instrument calibration curve was prepared at 10 atmospheres pressure. The infrared absorption at each concentration of CO<sub>2</sub> in nitrogen on the calibration curve is due to three factors: the absorption of the CO<sub>2</sub> molecule itself, the absorption due to the interactions between the CO<sub>2</sub> and nitrogen molecules, and the absorption due to the interactions between nitrogen molecules. It was presumed that these three factors would be additive in contributing to the absorption at the 4.22 micron peak. To prove this theory, standards of CO<sub>2</sub> in oxygen were prepared (high pressure oxygen has a broad absorption band at 6.7 microns). The difference in the indicated analysis using CO<sub>2</sub> in oxygen standards was approximately equal to the amount of absorption due to the nitrogen intermolecular collisions (forces). This is illustrated in Figures 4 and 6. Since the original calibration curve was prepared with standards using CO<sub>2</sub> in nitrogen, and the analysis was performed for CO<sub>2</sub> in oxygen. The results obtained with the infrared spectrophotometer were originally consistently lower than those obtained with the MCA.

The calibration curves were corrected for the nitrogen absorption and the corrected data using these new curves appeared to agree favorably with the results obtained with the MCA.

- b. On the next batch of samples analyzed with the infrared spectrophotometer and the MCA, a disagreement of the results in the analysis of carbon dioxide by the two techniques occurred again.

This batch of samples was analyzed approximately one week after the calibration curve had been corrected for the absorption due to the intermolecular collisions (forces) of the nitrogen at high pressures. The results from the infrared spectrophotometer fell considerably below the results from the MCA. It had been reported by the manufacturer of the infrared spectrophotometer that an inert gas purge should be placed on the optical path in the instrument when analyzing for contaminants which could also be present in the laboratory atmosphere. Since CO<sub>2</sub> is an atmospheric constituent, an investigation was undertaken to determine if this contamination might be the cause for the low readings.

# CALIBRATION CURVE

CO<sub>2</sub> in N<sub>2</sub> vs CO<sub>2</sub> in O<sub>2</sub> using Beckman IR-4

Path ————— 10 m.  
Sample Press ——— 10 Atm.  
Scan Speed ——— 1  $\mu$ /min.  
Sill ————— 0.80 mm at 10  $\mu$ (Sel)  
SB-DB Ratio ——— 1:1  
 $\lambda$  ————— 4.22  $\mu$

⊙ CO<sub>2</sub> Standards Prepared in N<sub>2</sub>  
△ CO<sub>2</sub> Standards Prepared in O<sub>2</sub>

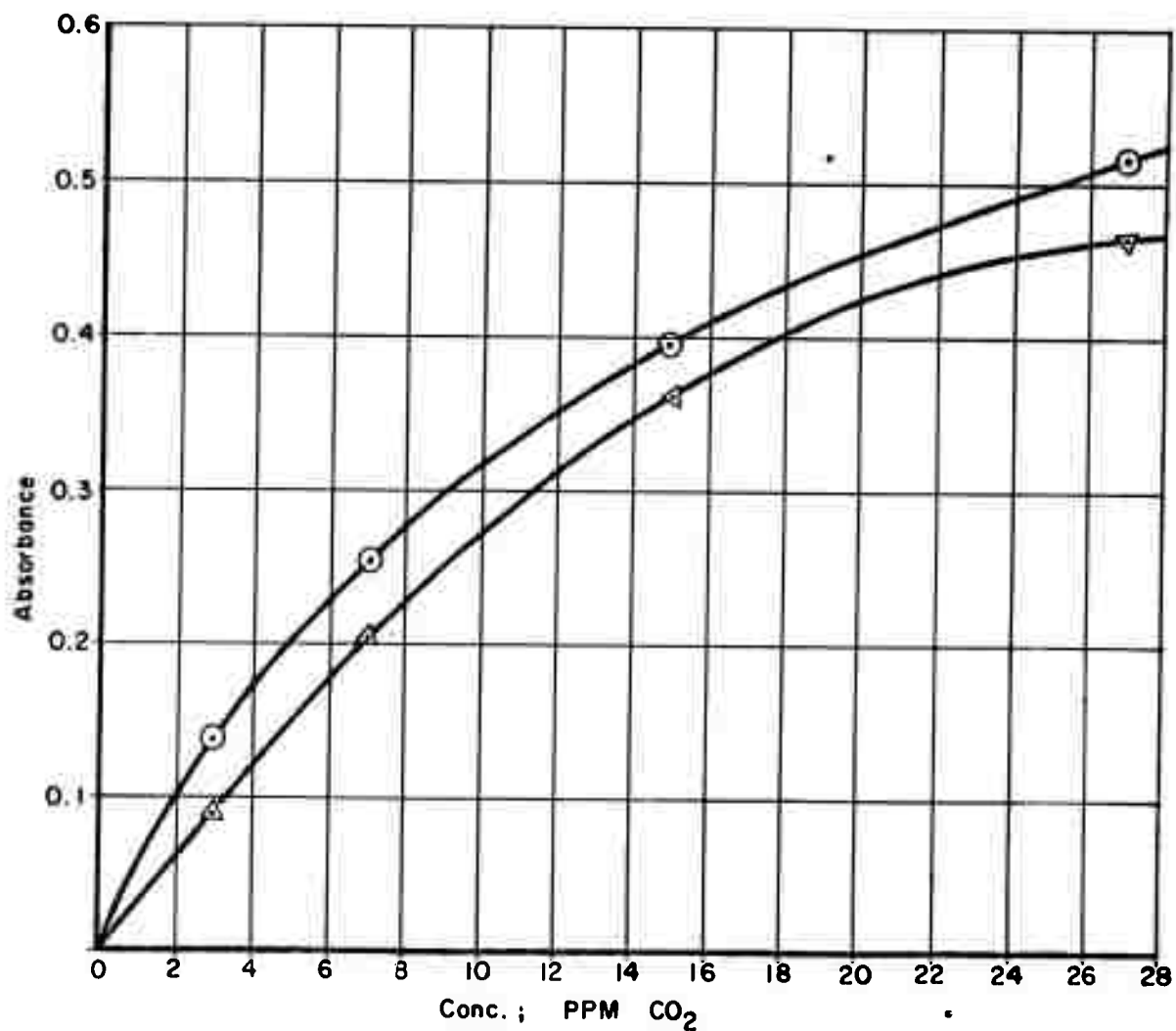


Figure 6

A series of analyses were performed, scanning only the 4.22 micron carbon dioxide absorption peak, with varying amounts of a high purity nitrogen purge in the analyzer cabinet. It was determined that there was a relationship between the amount of atmospheric CO<sub>2</sub> in the optical path of the instrument and the amount of energy transmitted by the instrument through the sample cell at this particular wave length. Lowering the amount of atmospheric CO<sub>2</sub> in the optical portion of the instrument increases the energy transmitted through the cell.

Since it was impossible to accomplish a complete purge of the instrument without extensive remodification, a procedure was undertaken which allowed for the calibration of the instrument prior to each gas analysis with one of the calibration mixtures which had been used to plot the calibration curve. If this point fell off the original curve, either a correction was applied to the curve for the analysis, or the atmosphere was changed within the instrument so that the calibration point would fall on the curve. Even with these time consuming techniques, the agreement between the two CO<sub>2</sub> analytical procedures was not completely satisfactory throughout the first portion of the program. Samples procured subsequent to mid-February showed satisfactory agreement.

Since Luft Principle infrared analysis is not influenced by the surrounding atmosphere, the carbon dioxide data as determined with the MCA which utilized the Luft Principle technique, can be presumed to be more valid.

#### 4. Acetylene

Acetylene was determined by the infrared spectrophotometer and the MCA. There were no problems associated with the analytical techniques involved in these analyses. The infrared spectrophotometer was the more sensitive of the two techniques for this contaminant, since the scale expansion adapter could be readily utilized. Acetylene was only detected in a few of the samples analyzed and in most cases it was less than 0.1 ppm. The three samples procured in March from Titan Pad 19 (Table B-5) showed the highest concentrations of acetylene which were detected. The reason for the decreased concentration after the original concentration of 0.25 ppm on March 14

is probably due to the continued addition of acetylene-free LOX to the tank. In each case, acetylene was detected only with the infrared spectrophotometer, and was below the minimum detectable limit of the MCA at the sample pressure being utilized.

#### 4. Water Vapor

In the initial tests for this contaminant, only the infrared spectrophotometer and the MCA were utilized. Since water shows up in a series of sharp absorption bands on the infrared spectrophotometer and since oxygen at high pressures also absorbs infrared in the same region (3), it was difficult to determine the extent of water vapor contamination with this technique (See Figures 7 and 8). From previous experience in analyzing for water, it was presumed that in most cases water was present in quantities less than 1.0 ppm. However, during this period of analysis, the MCA indicated rather high concentrations of water which varied from 0 to 100 ppm. The calibration standard used was continuously verified against an electrolytic hygrometer type water vapor analyzer which continuously indicated the 30 ppm by volume value used in calibrating the infrared optical bench. To determine if there might not be better agreement of the water vapor analysis between the two analytical techniques, the 30 ppm standard was analyzed in the infrared spectrophotometer and gave no indication of water anywhere on the spectrum (this standard was prepared in nitrogen) (3). Because of this disagreement, the standard was then analyzed by an Alnor Dew Pointer, which is an instrument based on an entirely different analytical method. The standard analyzed by this technique was found to contain less than 0.5 ppm water. Following this analysis, the electrolytic hygrometer was found to have an intermittently shorting cell, which caused the high water vapor readings. Subsequent to this, all of the samples (provided there was sufficient pressure) were analyzed with the Alnor Dew Pointer instrument and were found to contain only trace quantities of water. This was verified with the MCA after it had been properly calibrated. This is shown by the results of the water vapor analyses in the samples procured during March.

There is an absorption band for water at 2.6 microns. This is a rather insensitive band and was evident only where there was a positive indication of water above the oxygen absorption in the 5 to 7.5 micron region. By a series of tests with varying concentration water vapor

Infrared Absorption Spectrum of High Purity  $O_2$  ; Cell Length = 10 m  
Sample Pressure = 10 Atm.

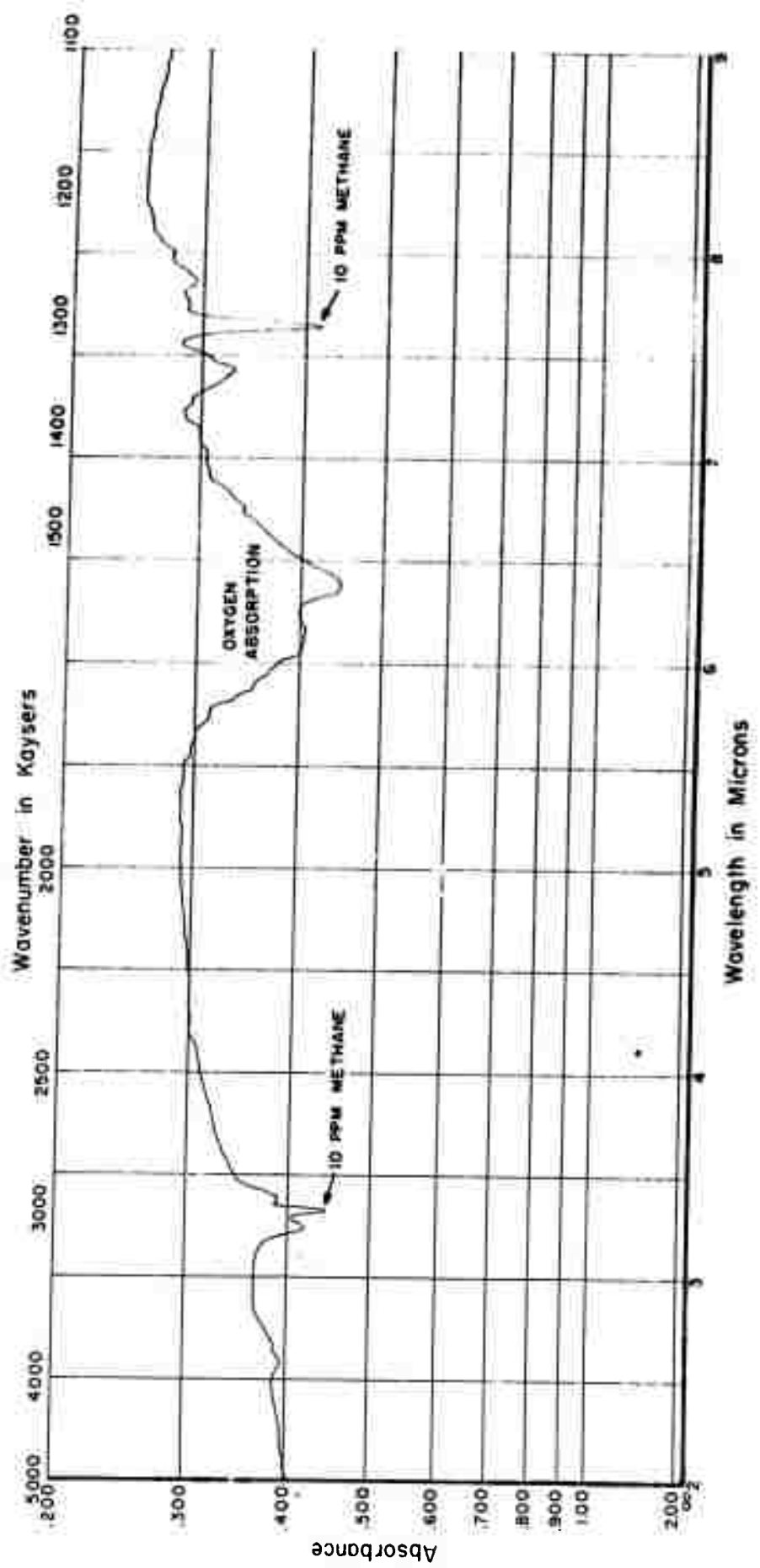


Figure 7

Infrared Absorption Spectrum of High Purity O<sub>2</sub>; Cell Length = 10 m  
Sample Pressure = 1 Atm.

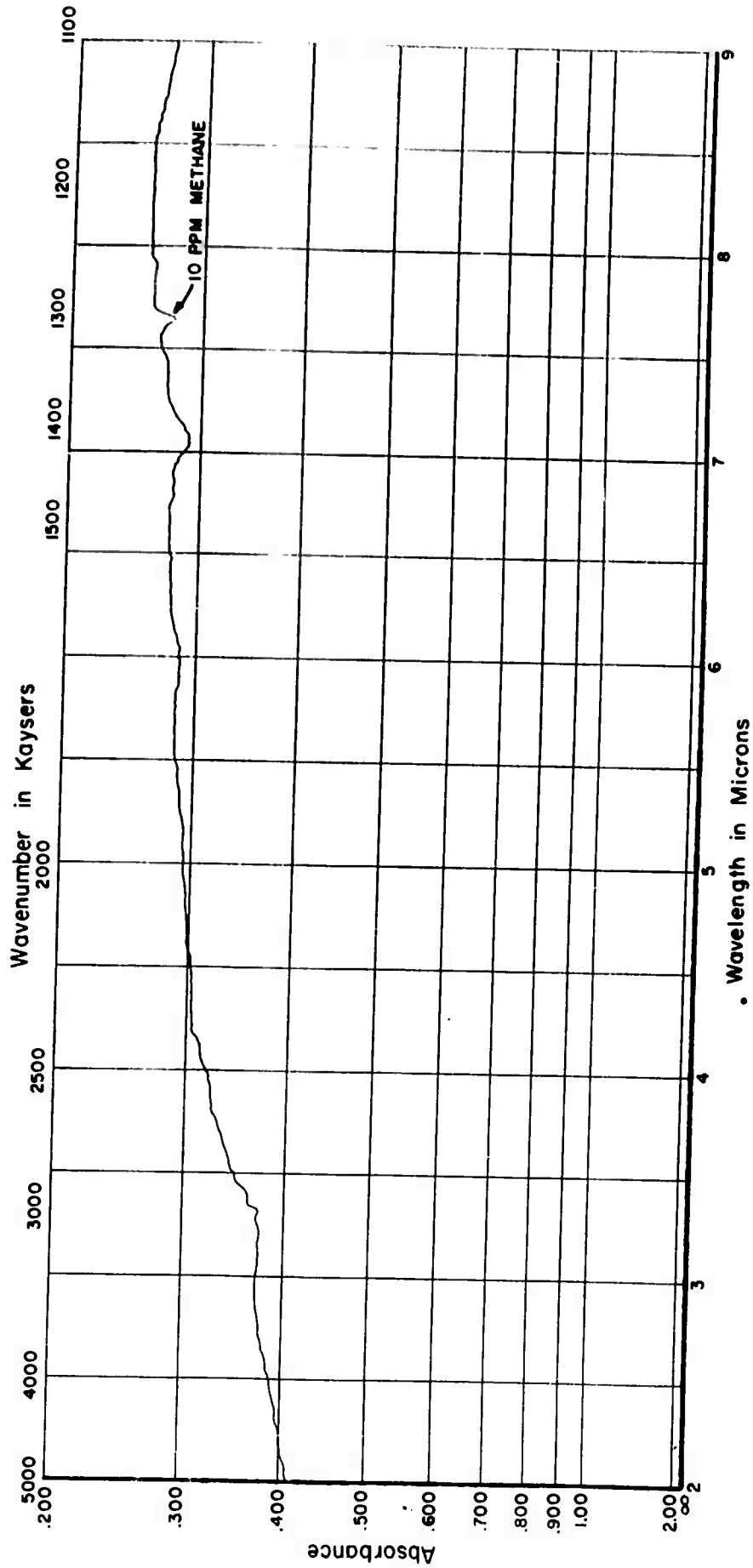


Figure 8

standards, it was found that this absorption peak was only discernable when the water vapor content was above 2.0 ppm. In all samples where this peak was seen, there was approximately the same magnitude of absorption. These samples are indicated in the tables under IR-4 - water vapor as present, approximately 2.0 ppm. All samples in which this 2.6 micron absorption was not seen are indicated as present less than 2 ppm.

#### 5. Total Hydrocarbons and Methane

With the exception of a minor difficulty with the infrared amplifier of the optical bench of the MCA, there is very good agreement between the infrared spectrophotometer methane values and the MCA total hydrocarbon values. These values should agree within the analytical accuracies of the two techniques, since the only volatile hydrocarbon which is present under normal plant operation and liquid handling techniques is methane.

The values for methane obtained from the infrared spectrophotometer are believed to be more valid because the problem with the MCA amplifier may have recurred throughout the whole series of analyses. These values also agree favorably with the results which would be expected, based on API's experience on the determination of the amount of methane in the product from liquid oxygen plants.

#### 6. Nitrous Oxide, Carbon Monoxide, and Butanes-plus Hydrocarbons

Nitrous oxide was present in trace quantities in all of the samples analyzed. This contaminant has a very strong absorption band on the infrared spectrophotometer at 4.45 microns. The sensitivity is approximately 0.1 ppm and the accuracy would be  $\pm 0.1$  ppm. Neither carbon monoxide nor butanes-plus hydrocarbons was detected in any of the samples analyzed.

## VIII. CONCLUSIONS

- A. Liquid oxygen, as produced in the liquid oxygen facility at Cape Canaveral, meets the specifications for purity, total hydrocarbons, moisture, and acetylene as required by procurement specification MIL-P-25508C (7 November 1960).**
- B. The liquid oxygen (LOX) sampled at the various pad storage tanks at Cape Canaveral meets the specifications for total hydrocarbons, moisture, and acetylene, but in a number of cases the LOX did not meet the specification for purity. The low purities may have been due to the technique of pressurizing LOX storage tanks with nitrogen gas.**
- C. The total hydrocarbon contamination found in the liquid oxygen was methane.**
- D. In all of the samples taken, carbon dioxide was found in quantities well above its solubility in LOX which indicates that solid CO<sub>2</sub> was present in the LOX. No conclusions can be drawn at this time as to the effects of solid CO<sub>2</sub> in liquid oxygen.**
- E. On the first samples tested, different analytical techniques were found to give different results for CO<sub>2</sub>. These differences were resolved by improving the methods used for standardizing and calibrating the infrared analyzer.**
- F. Some disagreement was found between the results as obtained by the Air Products Laboratory and those as obtained by the Patrick AFB Laboratory on equivalent samples. The disagreements have not been resolved at present but may be due to the standardizing technique used by the Patrick Laboratory for their infrared spectrophotometer.**

## IX. RECOMMENDATIONS

With the exception of the possible additional carbon dioxide requirement as outlined below, the Air Force should maintain MIL-P-25508C (Military Specification, Propellant, Oxygen) as it exists regarding the level of purity, and the amounts and types of impurities present in liquid oxygen.

It should be determined if the presence of solid carbon dioxide in a missile tank is detrimental to the operation of its liquid oxygen flow and flow control system.

Until such time as it is determined whether CO<sub>2</sub> is a hazard in a missile system, the procurement specification should be revised to limit the level of carbon dioxide to below its solubility in liquid oxygen. This value is approximately 5 ppm (molar) at the normal boiling point of liquid oxygen.

It is suggested that the Patrick Laboratory incorporate a calibration gas preparation procedure similar to that used at the Edwards Air Force Base Flight Test Center Laboratory. This apparatus utilizes a micro-manometer to measure the volumes of gas used in preparing the standardization mixtures, and the mixtures are prepared directly in the analyzer gas cell, eliminating potential errors from gas transfer or diffusion. (This standardization technique was used at Edwards Air Force Base in preparing a methane in oxygen calibration curve. The results of three analyses for methane on the Edwards 75 tons per day Plant liquid oxygen product showed an average of 13.5 ppm. The results of all of the analyses for methane from the identical 75 tons per day Plants at Cape Canaveral in this report also averaged 13.5 ppm.)

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**APPENDIXES**

## APPENDIX A

### AIR PRODUCTS, INCORPORATED CRYOGENIC LIQUID BATCH SAMPLER MODEL A-3-A

### OPERATING AND INSTRUCTION MANUAL

#### INTRODUCTION

The Air Products Batch Sampler is a new development in quality control technique for determining the purity of such cryogenic liquids as oxygen, nitrogen, and argon. It will sample and prepare for analysis any low-boiling point liquid which is non-toxic and non-corrosive with respect to the materials in the sampler.

It is ruggedly constructed of high-pressure, stainless steel pipe and caps fitted with high-pressure brass, oxygen-service valves. Each valve is fitted with a safety disc to assure the safety of the sampling personnel during sampling. It is designed for use by unskilled personnel carrying out simple instructions printed inside the cover of the carrying case. This manual is written to give further technical information on the sampler and as an addendum to the sampling instructions.

#### PRINCIPLE OF OPERATION

The sampler is based on the common principle of trapping a known volume of a cryogenic liquid within a high-pressure container and allowing the liquid to vaporize to a high-pressure gaseous sample by normal heat leak. In the acquiring of a sample the liquid to be vaporized to the high-pressure gas sample is contained in the inner cup. (See Figure A-1) By proper manipulation of the valves, the entire container is filled with the liquid being sampled. There is, therefore, a protective liquid blanket surrounding the sample liquid in the inner cup, which prevents vaporization or fractionation of the trapped liquid. This surrounding liquid blanket is removed at a rapid rate (approximately 30 seconds duration) and the inner sample vaporizes as stated above. This method

of insulating the trapped liquid by identical cold liquid is one of the unique features of this sampler (proprietary development - patent protection anticipated) and allows it to obtain a more representative sample than would be obtained by other "trapped liquid-vaporization techniques."

### SAMPLING INSTRUCTIONS

The sampling instructions are contained within the lid of the sampler carrying case and are reproduced here as Figure A-2. In order for the operator\* to better understand what is occurring as he proceeds through the sampling instruction steps, comments and additions to the listed instructions are outlined below:

1. Place opened container in upright position as in the above sketch.  
**DO NOT BLEED THE PRESSURE FROM SAMPLE CYLINDER.**

The sampler is placed in upright position so that the liquid flowing into valve (A) will flow into the inner cup and then overflow into the outer, high-pressure container. In any position other than upright, the inner cup would not retain the proper volume of liquid for vaporization to a high-pressure sample.

The sampler should normally be shipped under high gas (oxygen or nitrogen) pressure; 500 to 800 psig is recommended. This oxygen or nitrogen is added to the sampler at the laboratory before shipment to the sampling site. Before this addition, the sampler is cleaned at the laboratory by acceptable "LOX-service" cleaning methods. The operator uses this gas to purge the sampling line, the valves and deflectors, the bleed valve, and the associated piping of moisture, dirt, and other contaminants. If the sampler arrives at the sampling site with less than 500 pounds pressure, the operator would know that the sampler had not been properly prepared at the laboratory or that some of the purging gas had leaked from the container indicating that the sampler is faulty.

2. Attach the copper tube liquid deflectors (D) to valves (B) & (C). Tighten the deflectors so that they point overboard on the left-hand side of the container.

\* (operator - person performing sampling.)

In order to have easy access to the connections on the valves, they must be pointed towards the operator. These deflectors are used as their name implies - to deflect the cryogenic liquid away from the operator during the manipulation of the valves in sampling.

3. Using appropriate adapters connect the sample line between bleed valve (F) and sample valve (A). **CAUTION: NEVER ATTEMPT TO SAMPLE A TANK OR SYSTEM WHICH HAS NOT HAD A BLEED VALVE (F) ATTACHED TO THE SAMPLE VALVE (G).**

A 3/8-inch O.D. insulated section of copper tubing is preferred for a sample line. The opening within this tube closely approximates the port openings in the valves. This decreases the chance of "metering" or "fractionation" during sampling. Bleed valve (F) is in the sample line as a safety measure to vent the sample line when valves (A) and (G) are closed in the sampling process.

4. Tighten all sample line connections and slowly open valve (A) allowing the sample line to be purged through valve (F).

This is a part of the purging procedure, which also purges bleed valve (F).

5. When 75% of the pressure in the sampler has been depleted, close valve (F).

At the conclusion of this operation, there should be approximately 200 psig residual pressure in the cylinder to be used in purging valves (B) and (C).

6. Purge valves (B) and (C) by alternately opening and closing these valves until the remaining pressure is depleted. Valves (B) and (C) should be closed at the end of this operation.

Valves (B) and (C) are alternately purged with "short spurts" of approximately two seconds duration until the cylinder charge gas is depleted. A long, slow purging on either of these valves could cause the valves to cool (due to the Joule-Thomson Effect)

condensing water on the surface which might freeze when the valves are further cooled during sampling. The short intermittent spurts of purge gas also accomplishes a more efficient job of purging foreign materials from the valve ports or seats. Valve (C) is closed at the end of this operation so that as sampling begins no vapor will purge from this valve.

7. Open valve (G); open valve (B). Valve (A) should be open from step 4. The sampler will begin to fill with liquid.

With valves (G), (A), and (B) open, and valve (C) closed, liquid will begin to flow from the cryogenic liquid tank (H). It will vaporize in the line (E), the vapor formed passing into the cylinder, purging the cylinder, and venting at valve (B). As this line becomes cold, liquid will pass through it, through the internal tube attached to valve (A), and into the sample cup. This liquid will also vaporize, purging the sample cup and portions of the large container, venting through valve (B). As the cup becomes cooled, the liquid collecting in it will overflow through the sample holes and the large slots in the cup into the large container. The liquid striking the large container will also vaporize, purging the large container, and again venting through valve (B). As the large container cools, it will fill with liquid which will finally vent through valve (B).

8. If possible at this time, pressurize cryogenic liquid tank, to approximately 10 to 15 psig.

In pressurizing the cryogenic liquid tank, observe precautions concerning tank safety discs, rated pressures, etc. In order to procure a representative sample, the liquid should begin flowing from the deflector on valve (B) in a minimum of 10 minutes. If liquid flow does not occur at this valve in this period of time, the flow through the sampler is too slow, and concentration of some of the warmer boiling contaminants could occur. The time required for this flow is a function of sample tank pressure and liquid "head".

9. When liquid starts to flow from the deflector on valve (B) and it is felt a representative sample has been obtained, perform the following valve manipulations in quick succession.

- a. Close valve (B)
- b. Close valve (A)
- c. Open valve (C)

When liquid is flowing from valve (B), the inner cup contains the last liquid to leave the tank being sampled. It is surrounded by liquid at its same temperature, preventing heat leak from the outside to the inner cup which in turn prevents excessive vaporization or fractionation. Only that liquid which fills the inner cup to the small holes is vaporized as the final sample. Therefore, the remaining liquid within the cylinder must be removed at a rapid rate to negate the possibility of heat leak affecting the trapped volume. This is accomplished by closing valve (B) which allows the sampler to "rock on the line" to the tank. Valve (A) is then closed and because of heat leak, the pressure immediately begins to build up within the container. To prevent a build-up which would rupture the safety discs on the valves, valve (C) is immediately opened. Vapor formed at the top of the container rapidly forces all of the liquid, except that which is trapped within the sample cup, out through valve (C). This flow of liquid should not take longer than 30 seconds. Therefore, valve (C) should be opened as wide as possible during this portion of the operation.

10. When liquid ceases to flow from the deflector on valve (C) - (gas should still be escaping), close valve (C).

Some gas will continue to escape from this valve due to the normal expansion of the vapor in the large cylinder container. When the operator is certain that all of the liquid is drained, valve (C) is closed. (Because of the "Coriolis Effect" - irrotational vortex or "Whirlpool", small portions of liquid will spurt from this valve after the majority of the liquid is drained. These last vestiges of liquid should be allowed to drain.) All of the valves - (A), (B), and (C) - are ruggedly

built for this type of service and sufficient pressure should be applied to assure a good valve seat seal.

11. Using proper precautions to prevent being "burned" by cold piping, open valve (F), and immediately close valve (G).

As pointed out above, when disconnecting the sampler from the tank, valves (A) and (G) must be closed. To prevent rupture of the sample line, valve (F) must be opened before valve (G) is closed.

12. Disconnect the sample line at valve (A) and lay the sampler container flat (lid side up) for 30 seconds.

The sampler is laid flat to allow the liquid to escape from the inner container into the large high-pressure container. If this is not accomplished, there is a slight possibility that fractionation could occur as the liquid in the inner cup vaporizes. This fractionation could cause the vapor sample to be inadequately diffused through the entire high-pressure container.

13. On warming to ambient conditions, the sample gauge should indicate a pressure of approximately 1300 psig.

The sampler when properly utilized should always attain a pressure at ambient conditions of  $1300 \pm 100$  psig. If the pressure attained is higher than 1400 psig., it would indicate that all of the liquid was not drained during step number 10. If the sample pressure is less than 1200 psig., the sampler should be immersed in water or a suitable liquid to check for leaks. The most common site of this type leak is in the rupture disc seal on the valves. These can usually be corrected by tightening the seal bonnet when the valve is slightly cooled.

14. The copper tube liquid deflectors should be removed and replaced in the sampler container before shipping.

In handling the liquid deflectors and the sample line, precautions should be taken to prevent excessive moisture from condensing on their inner surfaces after use.

### **SAFETY PRECAUTIONS**

Normal rules involving the safe handling of cryogenic liquids and/or oxidants should be applied in the use of this sampler. Specific rules for safe operation are listed below:

1. In sampling cryogenic liquids, protect the body from contact with the liquid or cold piping by using acceptable, approved gloves, a hood and a hard hat, if necessary. If protection is not provided for the face and eyes with a hood, a transparent face shield should be worn.
2. The major hazard involved in the use of this sampler is the possibility of the operator inadvertently trapping cold liquid in a closed section of piping. This possibility is protected against by the use of safety discs in the sampler itself. However, extreme care and caution should be exercised in the handling and manipulation of the sample line and associated piping to prevent trapping liquid in a closed line between the sampler and the sampled tank. Before closing any valve between the sampler and the sampled tank, the operator should assure himself that liquid will not be trapped. In reference to the sampling instructions and as a further safety precaution, valve (F) should also be fitted with a 300 psig rupture disc.
3. Normal care concerning smoking, open flames, static charges, etc. should be exercised when sampling liquid oxygen. Special care should be exercised after the operator has been in prolonged contact with oxygen vapor to assure that his clothing is well purged before exposing himself to an area where open flames, static charges, smoking, etc. might be prevalent.

## SPECIFICATIONS

Casing size - 25" x 12" x 8-3/4"

Weight - 30 pounds (including case)

Pressure Limits - Full vacuum to 2000 psig.

Pressure Gauge - 0 to 2000 psig.

Pressure Cylinder - Type 304 Stainless Steel seamless pipe. Walls 0.237" thick. Pressure cylinder rated at 3000 psig. Each valve equipped with an 1800 psig rupture disc. Valve seats and seals made of Kel-F.

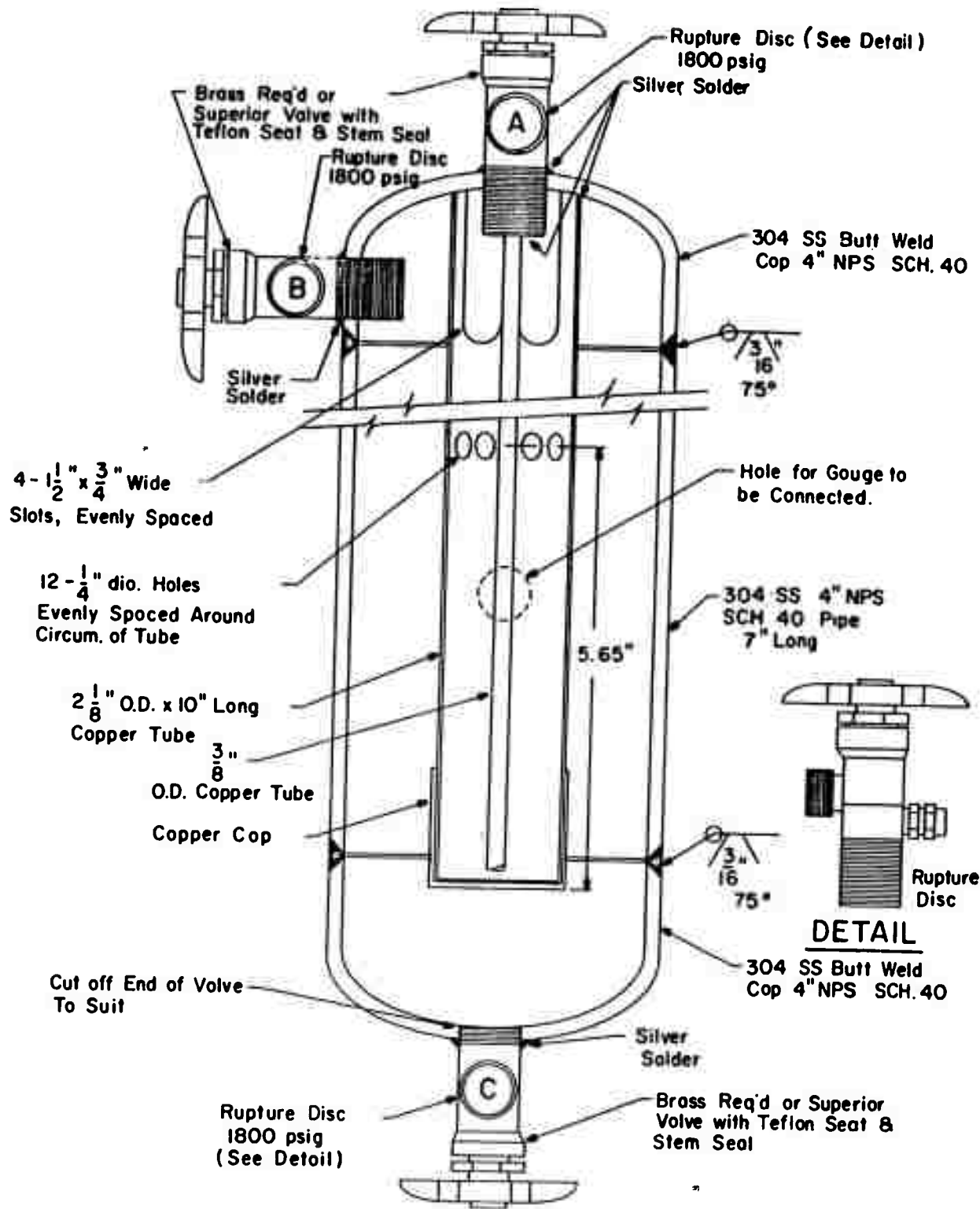
Valves - Brass.

Liquid Sample Size - 17.0 cubic inches.

Cylinder Displacement - Approximately 150 cubic inches.

Gaseous Volume (Oxygen) - 14,500 cubic inches at standard conditions.

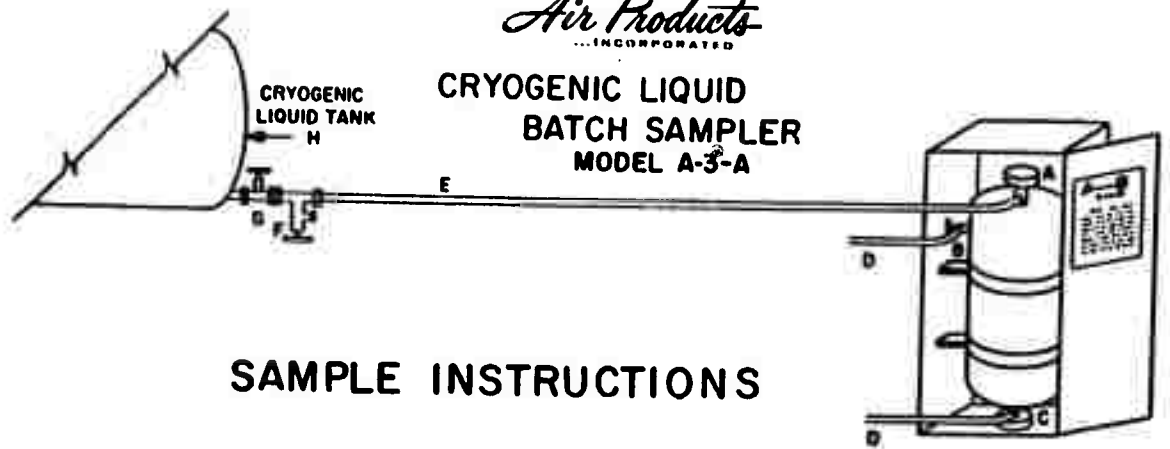
Final Sample Pressure (Oxygen) - 1300 psig.



# CRYOGENIC LIQUID BATCH SAMPLER

(MODEL A-3-A)

Fig. A-1



## SAMPLE INSTRUCTIONS

1. Place Opened Container in Upright Position as in the Above Sketch. **DO NOT BLEED THE PRESSURE FROM SAMPLE CYLINDER.**
2. Attach the Copper Tube Liquid Deflectors (D) to Valves (B) & (C). Tighten the Deflectors so that they Point Overboard on the Left Hand Side of the Container.
3. Using Appropriate Adapters Connect the Sample Line Between Bleed Valve (F) & Sample Valve (A).  
**CAUTION: NEVER ATTEMPT TO SAMPLE A TANK OR SYSTEM WHICH HAS NOT HAD A BLEED VALVE (F) ATTACHED TO THE SAMPLE VALVE (G).**
4. Tighten All Sample Line Connections and Slowly Open Valve (A) Allowing the Sample Line to be Purged through Valve (F).
5. When 75% of the Pressure in the Sampler has been Depleted, Close Valve (F).
6. Purge Valves (B) & (C) by Alternately Opening and Closing these Valves until the Remaining Pressure is Depleted. Valves (B) & (C) should be Closed At the End of this Operation.
7. Open Valve (G). Open Valve (B). Valve (A) Should be Open from Step 4, the Sampler will Begin to Fill with Liquid.
8. If Possible at this Time, Pressurize Cryogenic Liquid Tank to Approximately 10-15 PSIG.
9. When Liquid Starts to Flow From the Deflector on Valve (B) and It is Felt a Representative Sample has been Obtained, Perform the Following Valve Manipulations in Quick Succession.
  - a. Close Valve (B)
  - b. Close Valve (A)
  - c. Open Valve (C)
10. When Liquid Ceases to Flow from the Deflector on Valve (C)-(Gas Should Still Be Escaping), Close Valve (C).
11. Using Proper Precautions to Prevent Being "Burned" by Cold Piping Open Valve (F) and Immediately Close Valve (G).
12. Disconnect the Sample Line at Valve (A) and Lay the Sampler Container Flat (Lid Side Up) for 30 Seconds.
13. On Warming to Ambient Conditions the Sample Gauge Should Indicate a Pressure of Approx. 1300 PSI.
14. The Copper Tube Liquid Deflectors Should be Removed and Replaced In the Sampler Container Before Shipping.

Fig. A-2

# Sample Analysis Data AF 33-(616)-6730

ATLAS PAD NO. #	MCA O <sub>2</sub> Pur.	MCA CO <sub>2</sub>	IR-4 CO <sub>2</sub>	IR-4 N <sub>2</sub> O	MCA C <sub>2</sub> H <sub>6</sub>	IR-4 C <sub>2</sub> H <sub>6</sub>	MCA H <sub>2</sub> O vapor	IR-4 CH <sub>4</sub>	MCA THC	IR-4 CH <sub>4</sub>	MCA CO	MCA C <sub>2</sub> H <sub>6</sub>	Other	Remarks
Sample #1 Tank Full Sampled 2/15/61	99.75%	17.0 ppm	3.5 ppm	0.6 ppm	<0.25 ppm (MDL)	<0.05 ppm (MDL)	9.0 ppm	1.5 ppm	13.5 ppm	<1.2 ppm (MDL)	<1.2 ppm (MDL)	<2.5 ppm (MDL)		All Analyses on Molar Basis MDL = min. detect limit
Sample #2 Tank Full Sampled 2/23/61	99.78%	39.0 ppm	14.2 ppm	0.4 ppm	<0.25 ppm (MDL)	<0.05 ppm (MDL)	3.0 ppm	16.5 ppm	17.4 ppm	<1.2 ppm (MDL)	<1.2 ppm (MDL)	<12 ppm (MDL)		MDL = min. detect limit
Sample #3 Tank Full Sampled 3/16/61 After 100 tanking test	99.55%	25.0 ppm	24.4 ppm	0.7 ppm	<1.2 ppm (MDL)	0.5 ppm (Trace)	4.0 ppm (Ainar)	22.0 ppm	20.0 ppm	<0.6 ppm (MDL)	<0.6 ppm (MDL)	<12 ppm (MDL)		Analytical Difficulty (See Report Test)
Sample #4 Tank Full Sampled 3/30/61 After 100 tanking test	99.45%	110 ppm	11.2 ppm	0.5 ppm	<1.2 ppm (MDL)	0.5 ppm (Trace)	0.3 ppm (MDL)	16.0 ppm	16.0 ppm	<0.6 ppm (MDL)	<0.6 ppm (MDL)	<12 ppm (MDL)		Taken Simultaneously With PAA66C-69-31
Sample #5 Tank Full Sampled 3/15/61 After 100 tanking test	99.53%	95 ppm	11.0 ppm	0.5 ppm	<1.2 ppm (MDL)	<0.05 ppm (MDL)	4.0 ppm (Ainar)	16.0 ppm	16.3 ppm	<0.6 ppm (MDL)	<0.6 ppm (MDL)	<12 ppm (MDL)		Taken Simultaneously With PAA66C-72B-31
Sample #6 Tank Full Sampled 3/16/61 After 100 tanking test	99.45%	15.0 ppm	14.0 ppm	0.6 ppm	<1.2 ppm (MDL)	<0.05 ppm (MDL)	Inault	19.0 ppm	22.4 ppm	<0.6 ppm (MDL)	<0.6 ppm (MDL)	<12 ppm (MDL)		Sampled with PAA Sampler No 66C-74-31
Sample #7 Tank Full Sampled 3/20/61	99.35%	10.0 ppm	7.6 ppm	0.4 ppm	<1.2 ppm (MDL)	<0.05 ppm (MDL)	12.0 ppm (MDL)	16.0 ppm	17.2 ppm	<0.6 ppm (MDL)	<0.6 ppm (MDL)	<2.5 ppm (MDL)		Taken Simultaneously With PAA66C-775-31
Sample #8 Tank Full Sampled 3/28/61	99.45%	11.5 ppm	8.5 ppm	0.7 ppm	<1.2 ppm (MDL)	<0.05 ppm (MDL)	12.0 ppm (MDL)	20.0 ppm	19.5 ppm	<1.2 ppm (MDL)	<1.2 ppm (MDL)	<2.5 ppm (MDL)		

Table B-1

# Sample Analysis Data AF 33-(616)-6730

Atlas Pad No.12	MCA O <sub>2</sub> Sur.	MCA CO <sub>2</sub>	IR-4 CO <sub>2</sub>	IR-4 N <sub>2</sub> O	MCA C <sub>2</sub> H <sub>6</sub>	IR-4 C <sub>2</sub> H <sub>4</sub>	MCA H <sub>2</sub> O <sub>over</sub>	IR-4 H <sub>2</sub> O <sub>over</sub>	MCA THC	IR-4 CH <sub>4</sub>	MCA C <sub>2</sub> H <sub>2</sub>	MCA C <sub>2</sub> H <sub>4</sub>	Other	Remarks
Sample 1 One Sampled 1/23/61	99.7%	14 ppm	15 ppm	0.4 ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	49.5 ppm -58.7 ppm	Present 4.5 ppm	13 ppm	175 ppm	<0.8 ppm (MDL)	<12 ppm (MDL)		All Analytes at Major Basis
Sample 2 One Sampled 1/23/61 7 hrs. Before Launch	99.4%	12.5 ppm	10.5 ppm	0.4 ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	3.0 ppm -38.7 ppm	Present Approx. 2 ppm	20 ppm	16.5 ppm	<0.8 ppm (MDL)	<25 ppm (MDL)		MDL min detect
Sample 3 One Sampled 2/2/61 1 hr. After Topping Tank	98.57%	5 ppm	5.5 ppm	0.3 ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	5 ppm -11.7 ppm	Present Approx. 2 ppm	13 ppm	150 ppm	<0.8 ppm (MDL)	<25 ppm (MDL)		
Sample 4 One Sampled 2/16/61 Tank Had 4,600 Gal. Inactive Since 1/23/61	99.45%	13.5 ppm	3.0 ppm	0.4 ppm	<0.25 ppm (MDL)	<0.5 ppm (MDL)	16.5 ppm	Present <2 ppm	18.2 ppm	21.0 ppm	<1.2 ppm (MDL)	<25 ppm (MDL)		Analytical Diff. See Report Text 1
Sample 5 One Sampled 3/9/61 Tank Had 3200 Gal. Inactive Since 1/23/61	99.73%	10.5 ppm	12.2 ppm	0.5 ppm	<1.2 ppm (MDL)	<0.5 ppm (MDL)	<0.1 ppm Alnor	Present Approx. 2 ppm	24.0 ppm	27.0 ppm	<0.6 ppm (MDL)	<12 ppm (MDL)		
Sample 6 One Sampled 3/16/61 Tank Had 2100 Gal. Inactive Since 1/23/61	99.73%	7.0 ppm	8.4 ppm	0.6 ppm	<1.2 ppm (MDL)	<0.5 ppm (MDL)	<0.1 ppm Alnor	Present <2 ppm	31.0 ppm	31.0 ppm	<0.6 ppm (MDL)	<12 ppm (MDL)		
Sample 7 One Sampled 3/29/61 Tank Had 1400 Gal. Inactive Since 1/23/61	99.6%	10 ppm	6.0 ppm	0.9 ppm	<1.2 ppm (MDL)	<0.5 ppm (MDL)	<0.12 ppm (MDL)	Present 2 ppm	43.0 ppm	45. ppm	<1.2 ppm (MDL)	<25 ppm (MDL)		

Table B-2

# Sample Analysis Data AF 33-(616)-6730

ATLAS PAD No.3	MCA C, Pur	MCA CO <sub>2</sub>	IR-4 CO <sub>2</sub>	IR-4 N <sub>2</sub> O	MCA C <sub>2</sub> H <sub>6</sub>	IR-4 C <sub>2</sub> H <sub>6</sub>	MCA H <sub>2</sub> O <sub>ice</sub>	IR-4 H <sub>2</sub> O <sub>ice</sub>	MCA1 THC	IR-4 CH <sub>4</sub>	MCA CO	MCA C <sub>2</sub> H <sub>4</sub>	Other	Remarks
Sample 1 Can Sampled 1/18/61 5 Hrs. After Topping, (1) Tanker Used	99.3%	13 ppm	9.0 ppm	0.4 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	0.75 ppm (MDL)	16.5 ppm	16 ppm	16.5 ppm	<0.06 ppm (MDL)	<12 ppm (MDL)		All analysis in Miller Series
Sample 2 Can Sampled 1/23/61 30 Hrs. Before Lunch	99.3%	14 ppm	23 ppm	0.5 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	4.5 ppm (MDL)	18 ppm	18.5 ppm	18 ppm	<0.06 ppm (MDL)	<12 ppm (MDL)		MDL + min detect limit
Sample 3 Can Sampled 1/26/61 48 Hrs. After Lunch	99.4%	15 ppm	13 ppm	0.5 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	2 ppm (MDL)	16 ppm	18.8 ppm	16 ppm	<0.06 ppm (MDL)	<25 ppm (MDL)		
Sample 4 Can Sampled 2/9/61 Tank Had 22,700 Gals	99.4%	9 ppm	5.5 ppm	0.4 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	15 ppm (MDL)	16 ppm	16 ppm	14 ppm	<0.06 ppm (MDL)	<12 ppm (MDL)		1 Analytical Difficulty Issue Report Test
Sample 5 Can Sampled 2/14/61 Tank Had 22,300 Gals	99.4%	11 ppm	9.5 ppm	0.5 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	4.6 ppm (MDL)	16 ppm	16 ppm	14.5 ppm	<0.06 ppm (MDL)	<12 ppm (MDL)		
Sample 6 Can Sampled 2/20/61 Tank Had 22,000 Gals	99.5%	4.3 ppm	9.2 ppm	0.5 ppm	<0.25 ppm (MDL)	<0.05 ppm (MDL)	<0.25 ppm (MDL)	Present <2 ppm	16.1 ppm	16.2 ppm	<0.12 ppm (MDL)	<12 ppm (MDL)		
Sample 7 Can Sampled 2/27/61 Tank Had 5,000 Gals	98.4%	15 ppm	11.9 ppm	0.4 ppm	<0.25 ppm (MDL)	<0.05 ppm (MDL)	9 ppm (MDL)	Present <2 ppm	14.9 ppm	15.5 ppm	<0.12 ppm (MDL)	<25 ppm (MDL)		
Sample 8 Can Sampled 3/8/61 Tank Full	99.5%	17 ppm	17 ppm	0.5 ppm	<1.2 ppm (MDL)	Trace	<0.1 ppm (Alinor)	Present Aprox. 3 ppm	14 ppm	15.5 ppm	<0.06 ppm (MDL)	<12 ppm (MDL)		Taken Simultaneously with PMA 66C-65G-31
Sample 9 Can Sampled 3/15/61 1 Hr. after Topping	99.77%	25 ppm	25.4 ppm	0.8 ppm	<1.2 ppm (MDL)	<0.5 ppm (MDL)	<0.1 ppm (Alinor)	Present <2 ppm	21 ppm	22.5 ppm	<0.06 ppm (MDL)	<12 ppm (MDL)		Taken Simultaneously with PMA 66C-734-31
Sample 10 Can Sampled 3/20/61 5 Hrs. After Topping	99.75%	13 ppm	12.5 ppm	0.4 ppm	<1.2 ppm (MDL)	<0.5 ppm (MDL)	<0.1 ppm (Alinor)	Present <2 ppm	14 ppm	14.5 ppm	<0.06 ppm (MDL)	<25 ppm (MDL)		Taken Simultaneously with PMA 66C-778-31

Table B-3

# Sample Analysis Data AF 33--(616)-6730

ATLAS PAD No.14	MCA O <sub>2</sub> Pur.	MCA CO <sub>2</sub>	IR-4 CO <sub>2</sub>	IR-4 N <sub>2</sub> O	MCA C <sub>2</sub> H <sub>6</sub>	IR-4 C <sub>2</sub> H <sub>4</sub>	MCA H <sub>2</sub> O vapor	IR-4 † H <sub>2</sub> O vapor	MCA † THC	IR-4 CH <sub>4</sub>	MCA CO	MCA C <sub>2</sub> H <sub>2</sub>	Other	Ref: C:Ks
Sample 1 <small>Date: Sampled 1/17/61 One Day After Tanking Test</small>	99.4%	23.5ppm	100ppm ↑	0.4ppm	<06ppm (MDL)	<05ppm (MDL)	13.5ppm -73% P <sub>2</sub> O <sub>5</sub> 2ppm	Present Approx. 2ppm	54ppm	29.5ppm	<5ppm (MDL)	<25ppm (MDL)		All Analyses on Major Basis
Sample 2 <small>Date: Sampled 1/25/61 CO<sub>2</sub> Pressurized Full Tank</small>	99.6%	35ppm	295ppm ↑	0.8ppm	<12ppm (MDL)	<05ppm (MDL)	2ppm † -97% P <sub>2</sub> O <sub>5</sub> 2ppm	Present Approx. 2ppm	32ppm	24.5ppm	<05ppm (MDL)	<25ppm (MDL)		MDL = min. detect limit
Sample 3 <small>Date: Sampled 2/1/61 Immediately After Topping</small>	99.6%	14ppm	12ppm ↑	0.6ppm	<06ppm (MDL)	<05ppm (MDL)	10ppm † -108% P <sub>2</sub> O <sub>5</sub> 2ppm	Present Approx. 2ppm	18.5ppm	20.5ppm	<08ppm (MDL)	<12ppm (MDL)		Taken Simultaneously With PAA Sampler No. 66C-294-21
Sample 4 <small>Date: Sampled 2/9/61 With PAA Sample No.3 Tank Had 21,800 Gals.</small>	99.55%	22ppm	5ppm	0.5ppm	<06ppm (MDL)	<05ppm (MDL)	72ppm †	Present Approx. 2ppm	22ppm	20ppm	0.5ppm (MDL)	<12ppm (MDL)		Taken Simultaneously With PAA Sampler No. 66C-365-21
Sample 5 <small>Date: Sampled 2/17/61 Two Hours After Topping</small>	99.65%	14.5ppm	4.5ppm	0.4ppm	<25ppm (MDL)	<05ppm (MDL)	16.5ppm †	Present Approx. 2ppm	15.5ppm	15.0ppm	<12ppm (MDL)	<25ppm (MDL)		Taken Simultaneously With PAA No. 34C- 496-21
Sample 6 <small>Date: Sampled 2/28/61 Tank Had 5,800 Gals.</small>	99.5%	13.5ppm	11.9ppm ↑	0.5ppm	<25ppm (MDL)	<05ppm (MDL)	16.0ppm †	Present Approx. 2ppm	17.4ppm	17.0ppm	<12ppm (MDL)	<25ppm (MDL)		† Analytical Difficulty (See Report Text)
Sample 7 <small>Date: Sampled 3/7/61 23,000 Gals. in Tank</small>	99.65%	19.5ppm	23.5ppm ↑	0.5ppm	<12ppm (MDL)	0.08ppm (Trace)	<0.1ppm (Alnor)	Present Approx. 2ppm	15.6ppm	16.0ppm	<06ppm (MDL)	<12ppm (MDL)		
Sample 8 <small>Date: Sampled 3/13/61 Tank Had 21,500 Gals.</small>	99.60%	14ppm	15.6ppm	0.6ppm	<12ppm (MDL)	0.05ppm (Trace)	<0.1ppm (Alnor)	Present Approx. 2ppm	15.6ppm	15.5ppm	<06ppm (MDL)	<12ppm (MDL)		
Sample 9 <small>Date: Sampled 3/17/61 Tank Had 20,500 Gals.</small>	99.60%	19.5ppm	15.6ppm	0.6ppm	<06ppm (MDL)	0.05ppm (Trace)	<0.1ppm (Alnor)	Present Approx. 2ppm	19.5ppm	20.0ppm	<06ppm (MDL)	<12ppm (MDL)		
Sample 10 <small>Date: Sampled 3/29/61 Full Tank</small>	99.70%	11.0ppm	11.0ppm	0.5ppm	<12ppm (MDL)	<05ppm (MDL)	<0.1ppm (Alnor)	Present Approx. 2ppm	17.0ppm	15.0ppm	<12ppm (MDL)	<25ppm (MDL)		

Table B-4

# Sample Analysis Data AF 33-(616)-6730

TITAN PAD No.19	MCA O <sub>2</sub> Pur.	MCA CO <sub>2</sub>	IR-4 CO <sub>2</sub>	IR-4 N <sub>2</sub> O	MCA C <sub>2</sub> H <sub>6</sub>	IR-4 C <sub>2</sub> H <sub>4</sub>	MCA H <sub>2</sub> O <sub>vap</sub> or H <sub>2</sub> O <sub>liq</sub>	IR-4 † H <sub>2</sub> O <sub>vap</sub> or H <sub>2</sub> O <sub>liq</sub>	MCA THC †	IR-4 CH <sub>4</sub>	MCA C <sub>2</sub> H <sub>2</sub>	MCA C <sub>2</sub> H <sub>6</sub>	Other	Remarks
Sample 1 Date: Sampled 1/18/61 One Hr. After Filling With 5 Tankers	99.5%	17.5 ppm	12.5 ppm ↑	0.4 ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	13.8 ppm -73% <sup>Day</sup>	Present approx. 2 ppm	23.5 ppm	19.5 ppm	<5 ppm (MDL)	<25 ppm (MDL)		All Analyses on Molar Basis
Sample 2 Date: Sampled 1/25/61	99.4%	15.5 ppm	14 ppm ↑	0.4 ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	5.0 ppm -86% <sup>Day</sup>	Present approx. 2 ppm	15.3 ppm	12.5 ppm	<0.5 ppm (MDL)	<25 ppm (MDL)		MDL = min. detect. limit
Sample 3 Date: Sampled 2/3/61 Tank Nearly Full	99.5%	10.8 ppm	9.0 ppm ↑	0.4 ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	.5 ppm -112% <sup>Day</sup>	Present approx. 2 ppm	12.5 ppm	15 ppm	<0.8 ppm (MDL)	<25 ppm (MDL)		
Sample 4 Date: Sampled 2/13/61 Tank Had 500 gal.	98.6%	10.5 ppm	12.0 ppm ↑	0.6 ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	13.5 ppm	Present approx. 2 ppm	18 ppm	14.5 ppm	<0.6 ppm (MDL)	<12 ppm (MDL)		† Analytical Difficulty (See Report Text)
Sample 5 Date: Sampled 2/16/61 Tank Nearly Full	98.6%	25 ppm	8.0 ppm ↑	0.5 ppm	<2.5 ppm (MDL)	<0.5 ppm (MDL)	8 ppm	Present approx. 2 ppm	9.9 ppm	12.5 ppm	<1.2 ppm (MDL)	<25 ppm (MDL)		
Sample 6 Date: Sampled 2/21/61 14 Hrs. After Launch; Tank Had 50 lbs.	98.9%	37 ppm	11.4 ppm ↑	0.4 ppm	<2.5 ppm (MDL)	<0.5 ppm (MDL)	Ineff. sample	Present approx. 2 ppm	18.6 ppm	16 ppm	<1.2 ppm (MDL)	<12 ppm (MDL)		
Sample 7 Date: Sampled 3/14/61 Tank Contained 118"	99.4%	12 ppm	14.0 ppm	0.7 ppm	<2.5 ppm (MDL)	0.25 ppm	0.1 ppm	Present approx. 2 ppm	17.6 ppm	16 ppm	<0.6 ppm (MDL)	<12 ppm (MDL)		
Sample 8 Date: Sampled 3/21/61 Tank Contained 124"	99.5%	10.1 ppm	8.6 ppm	0.7 ppm	<2.5 ppm (MDL)	0.13 ppm	0.1 ppm	Present approx. 2 ppm	18.5 ppm	16.5 ppm	<1.2 ppm (MDL)	<12 ppm (MDL)		
Sample 9 Date: Sampled 3/28/61 Tank Contained 126"	99.52%	12 ppm	12.2 ppm	0.8 ppm	<2.5 ppm (MDL)	0.16 ppm	0.1 ppm	Present approx. 2 ppm	17.5 ppm	16.5 ppm	<1.2 ppm (MDL)	<12 ppm (MDL)		

Table B-5

# Sample Analysis Data AF 33-(616)-6730

TITAN PAD No. 20	MCA O <sub>2</sub> Fut.	MCA CO <sub>2</sub>	IR-4 CO <sub>2</sub>	IR-4 N <sub>2</sub> O	MCA C <sub>2</sub> H <sub>4</sub>	IR-4 C <sub>2</sub> H <sub>4</sub>	MCA H <sub>2</sub> O vapor	IR-4 H <sub>2</sub> O vapor	MCA† THC	IR-4 C <sub>2</sub> H <sub>6</sub>	MCA CO	MCA C <sub>2</sub> H <sub>6</sub>	Other	Remarks
Sample 1 Date: Sampled 1/19/61 Little Previous Activity	99.7%	20ppm	175ppm †	0.7 ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	22.5ppm -66% <sup>†</sup> H <sub>2</sub> O	Present Approx. 2ppm	14 ppm	17.5 ppm	<.5 ppm (MDL)	<.12 ppm (MDL)		All Analyses on Molar Basis
Sample 2 Date: Sampled 1/27/61 See Remarks	99.4%	16.5 ppm	12.5ppm †	0.5 ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	480ppm -18% <sup>†</sup> H <sub>2</sub> O	Present Approx. 2ppm	185ppm	17 ppm	<0.5 ppm (MDL)	<.5 ppm (MDL)		Sampler Gauge Damaged in Shipment Analysis Questionable
Sample 3 Date: Sampled 2/2/61 2 Days After Tapping	99.5%	10.5ppm	70 ppm †	0.3ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	<.19 ppm (MDL)	Present Approx. 2ppm	12.5 ppm	13 ppm	<0.8 ppm (MDL)	<.12 ppm (MDL)		MDL = min. detect. limit
Sample 4 Date: Sampled 2/13/61 3 Days After Launch <4,000 Gals.	98.6%	12ppm	7.5ppm	0.5ppm	<0.6 ppm (MDL)	<0.5 ppm (MDL)	12ppm †	Present <2ppm	22ppm	17.5ppm	<.06ppm (MDL)	<.12 ppm (MDL)		† Analytical Difficulty (See Report Text)
Sample 5 Date: Sampled 2/20/61 <4000 Gals.	98.75%	15.5ppm	60ppm	0.6ppm	<.25ppm (MDL)	<.05 ppm (MDL)	105ppm †	Present <2 ppm	199ppm	20.0ppm	<.12 ppm (MDL)	<.25ppm (MDL)		
Sample 6 Date: Sampled 2/28/61 22 Hrs After Tapping, Tank had 103	98.0%	23.0ppm	24.0ppm	0.6ppm	<.25ppm (MDL)	<.05 ppm (MDL)	50ppm †	Present <2 ppm	15 ppm	16 ppm	<.12 ppm (MDL)	<.25ppm (MDL)		
Sample 7 Date: Sampled 3/14/61 Tank Contained 40"	98.6%	16.0ppm	16.5ppm	0.6ppm	<.12ppm (MDL)	<.05 ppm (MDL)	10.1ppm (MDL)	Present <2 ppm	16.9ppm	16.5ppm	<.06ppm (MDL)	<.12 ppm (MDL)		
Sample 8 Date: Sampled 3/29/61 Tonk Contained 105"	99.45%	16.5ppm	22.4ppm †	0.7ppm	<.12ppm (MDL)	<.05 ppm (MDL)	<.12ppm (MDL)	Present Approx. 2ppm	15.5ppm	16.5ppm	<.12 ppm (MDL)	<.25ppm (MDL)		

Table B-6

# Sample Analysis Data AF 33-(616)-6730

LOX PLANTS P-1160-61 Valve O-188	MCA O <sub>2</sub> Pur.	MCA CO <sub>2</sub>	IR-4 CO <sub>2</sub>	IR-4 N <sub>2</sub> O	MCA C <sub>2</sub> H <sub>6</sub>	IR-4 C <sub>2</sub> H <sub>4</sub>	MCA H <sub>2</sub> O <sub>vap</sub> or -105°F or -64°F or -30°F	IR-4 † H <sub>2</sub> O <sub>vap</sub>	MCA † THC	IR-4 CH <sub>4</sub>	MCA CO	MCA C <sub>2</sub> H <sub>2</sub>	Other	Remarks
Sample 1 Date: Sampled 1/6/61 12 Hrs. on No. 2 Adsorber (P-1160)	99.78%	2.5 ppm	1.6 ppm †	0.2 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	1.0 ppm †	Present Approx. 2 ppm	10 ppm	11.0 ppm	<0.08 ppm (MDL)	<25 ppm (MDL)		All Analyses on Molar Basis
Sample 2 Date: Sampled 1/9/61 7 Hrs. on No. 2 Adsorber (P-1160)	99.7%	61 ppm	37.5 ppm †	0.3 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	<12 ppm † (MDL)	Present † <2 ppm	7 ppm	11.5 ppm	0.1 ppm (MDL)	<25 ppm (MDL)		MDL = min. detect. limit
Sample 3 Date: Sampled 1/27/61 26 Hrs. on No. 2 Adsorber (P-1160)	99.7%	63 ppm	>65 ppm †	1.2 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	25.5 ppm † -64°F or -30°F	Present † <2 ppm	7 ppm	14.5 ppm	<12 ppm (MDL)	<25 ppm (MDL)		
Sample 4 Date: Sampled 2/3/61 26 Hrs. on No. 2 Adsorber (P-1161)	99.6%	110 ppm	>65 ppm †	0.3 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	<12 ppm † (MDL)	Present † <2 ppm	2.5 ppm	10.5 ppm	<12 ppm (MDL)	<25 ppm (MDL)		
Sample 5 Date: Sampled 2/14/61 5 Hrs. on No. 1 Adsorber (P-1161)	99.7%	74 ppm	46 ppm †	0.3 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	60 ppm †	Present † <2 ppm	80 ppm	22 ppm	<12 ppm (MDL)	<12 ppm (MDL)		† Analytical difficulty (See Report Text)
Sample 6 Date: Sampled 2/17/61 22 Hrs. on No. 1 Adsorber (P-1161)	99.7%	52 ppm	22 ppm †	0.5 ppm	<0.06 ppm (MDL)	<0.05 ppm (MDL)	12 ppm †	Present † <2 ppm	16 ppm	12 ppm	<12 ppm (MDL)	<12 ppm (MDL)		
Sample 7 Date: Sampled 2/23/61 9 Hrs. on No. 2 Adsorber (P-1160)	99.85%	36 ppm	10.2 ppm †	0.3 ppm	<0.25 ppm (MDL)	<0.05 ppm (MDL)	100 ppm †	Present † <2 ppm	14.8 ppm	13.5 ppm	<12 ppm (MDL)	<25 ppm (MDL)		
Sample 8 Date: Sampled 2/27/61 12 Hrs. on No. 1 Adsorber (P-1161)	99.7%	>100 ppm	>100 ppm	0.8 ppm	<0.25 ppm (MDL)	<0.05 ppm (MDL)	Insuff. Sample	Present † <2 ppm	15.0 ppm	15.0 ppm	<12 ppm (MDL)	<25 ppm (MDL)		
Sample 9 Date: Sampled 3/9/61 15 Hrs. on No. 1 Adsorber (P-1161)	99.6%	2.50 ppm	25.0 ppm	0.9 ppm	<12 ppm (MDL)	<0.05 ppm (MDL)	<0.1 ppm (MDL)	Present † <2 ppm	12.4 ppm	12.5 ppm	<0.06 ppm (MDL)	<12 ppm (MDL)		
Sample 10 Date: Same as Sample No. 9	99.6%	24.0 ppm	24.0 ppm	0.8 ppm	<12 ppm (MDL)	<0.05 ppm (MDL)	<0.1 ppm (MDL)	Present † <2 ppm	11.6 ppm	12.5 ppm	<0.06 ppm (MDL)	<12 ppm (MDL)		Duplicate of Sample No. 9 taken 1 min. After Sample No. 9
Sample 11 Date: Sampled 3/15/61 14 Hrs. on No. 1 Adsorber (P-1160)	99.65%	470 ppm	42.5 ppm	0.3 ppm	<12 ppm (MDL)	<0.05 ppm (MDL)	<0.1 ppm (Alnor)	Present † <2 ppm	12.9 ppm	16.0 ppm	<0.06 ppm (MDL)	<25 ppm (MDL)		
Sample 12 Date: Sampled 3/29/61 9 Hrs. on No. 2 Adsorber (P-1160)	99.53%	500 ppm	51.5 ppm	0.4 ppm	<12 ppm (MDL)	<0.05 ppm (MDL)	<0.1 ppm (Alnor)	Present † <2 ppm	12.5 ppm	13.5 ppm	<0.06 ppm (MDL)	<25 ppm (MDL)		
Sample 13 Date: Sampled 3/29/61 7 Hrs. on No. 1 Adsorber (P-1161)	99.62%	9.5 ppm	8.8 ppm	0.2 ppm	<12 ppm (MDL)	<0.05 ppm (MDL)	<12 ppm (MDL)	Present † <2 ppm	11.5 ppm	11.5 ppm	<0.06 ppm (MDL)	<25 ppm (MDL)		

Table B-7

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