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OPERATING MANUAL

OFFICE DIVISION  
JUL 15 1945  
THE KELLEX CORP  
FIELD ENG. OFFICE

OM 48 5 A



SPACE RECORDER

AND ITS USE

IN LIGHT DILUENT SYSTEM

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MAR 13 1982  
By: *RH*

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FIRST EDITION

July 9, 1945

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FOREWORD

INVENTORIED  
MAR 13 1952  
By *RH*

The following manual covers the Space Recorder, an analytical device used to supplement the Line Recorder in analyzing for 616.

The pages of this manual are consecutively numbered from 1 to 23 with Arabic numerals in the upper right hand corner of each page.

Drawings are all marked with two numbers, a Roman numeral and an Arabic numeral separated by a short dash. The Roman numeral represents the major section of the manual separated by a green index tab, and the Arabic numeral indicates the numerical order of a print in its section.

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I. INTRODUCTION

The problem of analyzing the gas in the purge cascade is complicated by the fact that the composition varies greatly from one end of the cascade to the other. Near the bottom of the cascade the stream consists of essentially pure 616, whereas at the top it consists of a light diluent, principally 74, containing only small traces of 616. The Line Recorder was designed for analyzing 616 which contained relatively small amounts of impurities. The method it employs is to measure the flow of gas to a mass spectrometer tube by means of a Pirani gauge flowmeter; the 616 is removed chemically before it reaches the spectrometer tube and the residual gases measured by means of the spectrometer tube. It is seen that the method has its highest precision for 616 containing only small amounts of contaminants. In the event that the device is used to analyze for 616 in relatively large amounts of 74, or other diluents, the accuracy drops sharply. The reason for this is obvious, inasmuch as the 616 content is found by subtracting the difference between the flow computed from the flowmeter and the 74 determined from the Mass Spectrometer tube reading. Only with careful calibration is it possible to determine either of these quantities with an accuracy of 1% or better. Accordingly, the device becomes practically useless for determining the composition of a mixture containing under 2% of 616. As was pointed out in the instruction manual for the Line Recorder, it is not possible to measure 616 in the Line Recorder tube directly since the decomposition of 616 sharply limits the life of the tube.

In order to supplement the Line recorder in the purge cascade, an instrument known as the Space Recorder was devised. Its principal component is an ionization chamber (hereafter referred to as "a" or "the" signal can), which measures the specific radioactivity of the gas present, and since 616 is an emitter of alpha particles, it provides a convenient method for measuring the 616 content of gas samples. With this device, it is possible to detect the presence of mol fractions of 616 in 74 or other gas mixtures of the order of  $10^{-6}$ . Its upper range is limited only by the radioactive contamination produced in the signal can by high concentration of 616 and if one is willing to clean the can frequently concentrations of 50 mol% or higher can be measured. It is thus seen that this device conveniently supplements the Line Recorder in the purge cascades.

## II. MANIFOLD MODIFICATIONS

In the main cascade of Section 300 most buildings contain only ten cells and hence the same number of sampling points. Only in Section 4 is the number of cells per building as great as 14. However, in the purge cascades there are 21 cells and in addition 5 other places at which analyses are required. From the standpoint of procurement of manifolds for the purge cascades, it was desirable to use manifolds which differ as little as possible from those being manufactured for use with the Line Recorders in the main cascade.

### (a) Change in Headers

Figure II-1 shows a flow diagram for a purge cascade manifold. A comparison with Figure II.1-13 of the Operating Manual, "Line Recorder and Mass Spectrometer", Volume XIX shows that most of the essential features have been retained. The principal difference lies in the manner in which the headers X, Y, C-1 and C-2 are employed. In the main cascade whenever an analysis is being made of the contents of a given cell, the gas flowing up to the manifold from the cell is returned to the same cell by means of a return line to that cell. In the purge cascade there are only a few points to which one can return gas. For the Line Recorders these are:

- (1) Purge net feed (through valves 7A and 14A)
- (2) Surge drum F-351 (through valves 21A and 28A)
- (3) Cell 4 (through valves 35A and 42A)
- (4) Cell 7 (through valves 49A and 56A)

For the space recorders these are (referring to Figure II-2)

- (1) Purge net feed (through valve 18)
- (2) Cell 12 (through valve 19)
- (3) Cell 17 (through valve 20)
- (4) 74 overhead product, before compression in the 50 cfm pumps (through valve 21)

In each case gas is returned to the point having most nearly the same  $\delta^{16}$  concentration.

The incoming lines from the various points to be sampled are connected to the four headers in the manifold as follows:

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Header X

Purge net feed  
Surge drum F-351  
Cell 3  
Cell 5  
Cell 7  
Cell 9  
Cell 11

Header Y

Actual feed to cascade  
Cell 2  
Cell 4  
Cell 6  
Cell 8  
Cell 10

Header C-1

Cell 12  
Cell 14  
Cell 16  
Cell 18  
Cell 20  
Cell 22

Header C-2

Cell 13  
Cell 15  
Cell 17  
Cell 19  
Cell 21  
74 overhead product

An examination of Figure II-1 shows that the two line recorders are connected to headers X and Y in a fashion similar to that employed in the main cascade. The dynamic calibration check system, the cross over system, the static calibration system and the sample removal equipment are identical to the corresponding units employed in the main cascade.

(b) Normal Connections to Line Recorders and Space Recorders

Under normal circumstances, the Line Recorders will be used for analyzing points containing one or more percent of 616. Under normal circumstances the space recorders will be used for analyzing gas from cells containing less than several percent of 616.

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From Figure II-1, it is easy to see that headers C-1 and C-2 connect most readily to the two space recorders. Thus under normal conditions it is possible to simultaneously operate two line recorders on the lower part of the cascade and two space recorders on the upper part. Under these circumstances it is important that the transfer line connecting valves 35, 21 and 28 be kept filled with 74. If this were not done, the possibility of leakage of 616 from headers X and Y, which contain high concentrations of this component, through valve 35 and 42 into the space recorder could take place. It is not hard to see that if the space recorder connected to valve 42 were analyzing gas containing a mol fraction of 616 of  $10^{-6}$ , even a slight leak in the seat of the valves could seriously contaminate the gas flowing to the space recorder.

(c) Connection of Line Recorder to Upper Part of Cascade

In the event that one wishes to employ the line recorder on the upper part of the cascade as might be the case if one were hunting for a leak, use is made of the transfer line connecting valves 35 with 21 and 28. In this way, the gas from any cell connected to header C-1 could be connected to either line recorder through either header X or header Y. Under these circumstances, it is apparent that one of the space recorders cannot be used.

(d) Connection of Space Recorder to Lower Part of Cascade

In some cases it may be desirable to analyze the contents of a cell from the lower part of the purge cascade with the space recorder. In this case, the flow of gas is again through the transfer line, this time in the opposite direction to what it was in the previously cited case. It is not anticipated that this will be a very frequent occurrence since the use of the space recorder for analyzing gas containing high concentrations of 616 is not encouraged because of its detrimental effect upon the life of the signal can.

(e) Analysis of Waste 74 in Holding Drum F-353

In addition to the changes already noted, the purge cascade manifold contains one additional feature not found on the regular manifold. This is the integrating volume 211 mounted on the outside of the air conditioned region. Gas from holding drum F-353 flows continuously through valves 201 and 203 past the open end of the adjustable leak 275. Volume 211 has been previously evacuated through valves 278 and 282, and leak 275 has been previously adjusted

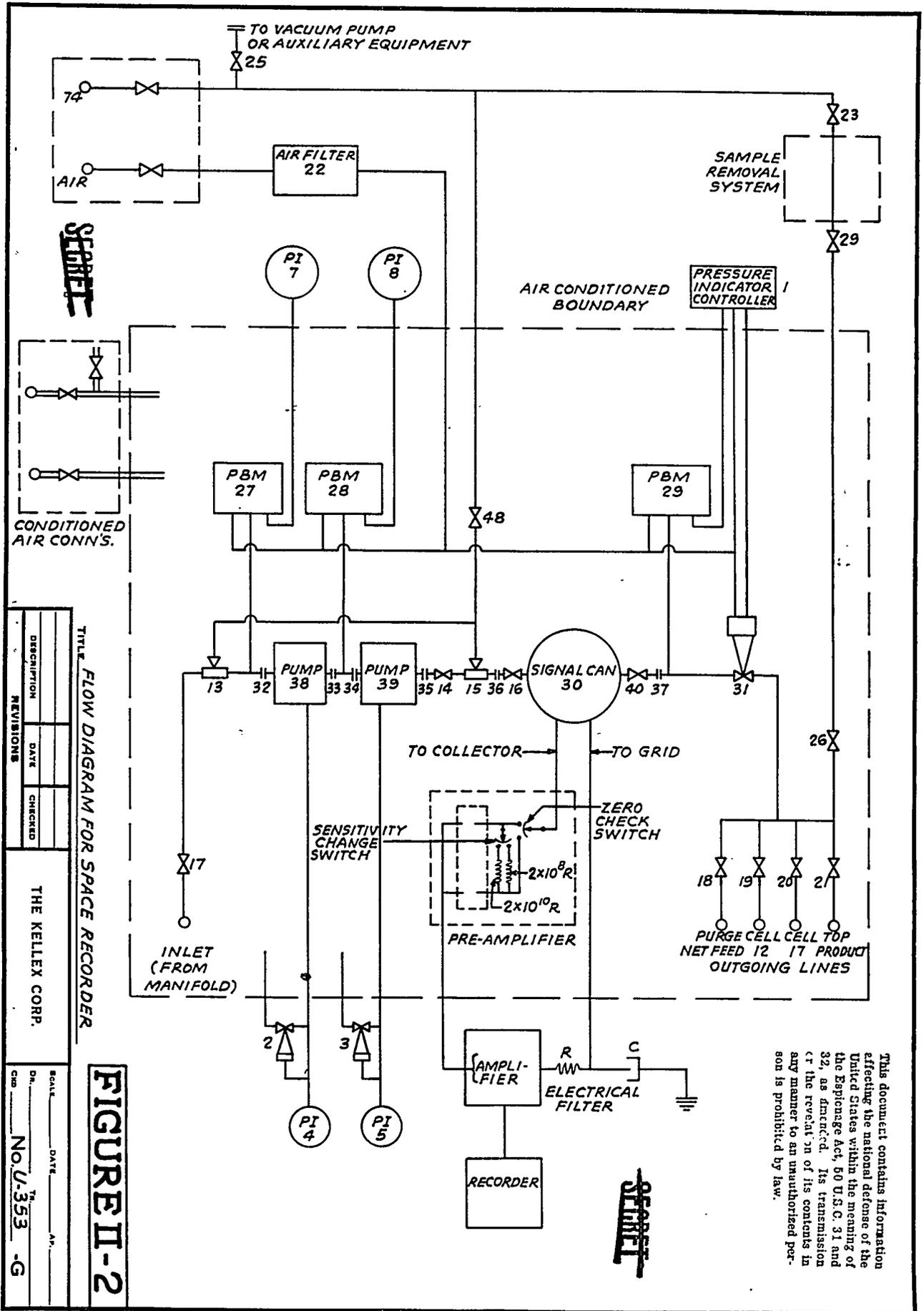
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so that in some arbitrary period, such as 8 hours, sufficient gas will flow into volume 211 so that the pressure will rise to a convenient value such as 1 psia. Then by connecting 211 to either of the adjustable leaks 73 or 74 used in connection with the standard sample bottles 115 and 116 it is possible to analyze the contents which have accumulated in 211. In the event that it is decided to analyze the contents of holding drum F-353 at any instant, it is possible by opening valves 202 and 204 to transfer the gas stream to either header C-1 or C-2 and hence make an analysis with either the space recorder or line recorder.

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TITLE: **FLOW DIAGRAM FOR SPACE RECORDER**

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**FIGURE II-2**

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III. CONSTRUCTION OF SPACE RECORDERS(a) Flow Diagram

The flow diagram for one of the space recorders is shown in Figure II-2. Gas enters the unit from the manifold through valve 17, passes through valves 13, 14, 15 and 16 into the signal can 30. The siphon type B-4 pumps 38 and 39 raise the pressure to whatever value is desired in the signal can. The pressure is controlled by the opening of valve 31 which is operated automatically by the controller 1. The gas then returns to the plant through one of the four lines shown. In the event that it is decided to remove a sample for external study, this can be accomplished by attaching a suitable sample removal system to the point indicated and opening valves 26 and 29.

(b) Signal Can

The radioactivity of 616 consists of the emission of high energy alpha particles at a definite uniform rate. This rate depends upon the relative isotopic constitution, since all three isotopes emit alpha particles at a different rate. These alpha particles as emitted have a very definite range of travel, which is inversely proportional to the pressure. In the gas samples, at standard temperature and pressure this range of travel is approximately 3cm.

While travelling this distance the energy of the emitted particle is expended by the production of approximately 130,000 ions which result from the collision of the particle with the gas molecules.

The collection of these ions and a measurement of the resultant electrical current constitutes a determination of the number of particles present and hence a determination of the 616 concentration.

In order to detect low concentrations of 616 it is necessary to employ a signal can having an internal diameter of 12" and an inside length of 24". Although the sensitivity could be improved by using a higher pressure, it was decided that it would not be practical to employ a pressure much higher than 10 psia with the pumps available. Because of the corrosive nature of 616 and the consequent danger of a high background, the signal can was made of the least corrosive materials. All metal surfaces are of nickel and KTF-10 plastic is used as an insulator.

The collector wire, 0.025" in diameter, is mounted along the axis of the can and connects to the pre-amplifier mounted on the cover of the can after passing through a vacuum-tight plastic disk. This disk has 10 concentric grooves cut along its inner surface in order to increase the electrical leakage path.

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A grid structure consisting of a cage approximately 20" long and 8" in diameter is mounted concentric with the collector wire. It is constructed entirely of nickel and every effort has been made to hold the surface area to a minimum. For example, the cylindrical surface of the grid structure consists of wires 0.0031" in diameter and one inch apart. Whereas the can operates at ground potential and the collector wire's potential never rises more than a few millivolts above ground, the grid structure is connected to a source potential of several hundred volts. Thus all positive ions formed within the grid structure are drawn to the central collector wire. If the can is operated at a pressure of more than 10 psia none of the alpha particles, originating from corrosion products on the inside wall of the can itself, have sufficient range to penetrate the grid assembly and hence the background ionization current in the device is due only to alpha particles originating on the grid structure or collector wire and to beta particles.

All internal parts are attached to the cover of the signal can which is gasketed with an aluminum wire seal. Gas circulation in the signal can is through two Crane 1/2" SM globe valves which are mounted on the side of the can near the top and bottom respectively. Connection to the rest of the flow system is through aluminum gasketed flanges, 36 and 37, shown in Figure II-2.

### (c) Current Amplifier and Recorder

The small ion current to the collector wire is measured by means of a negative feed-back amplifier having an identical circuit to that employed in the line recorder. Although the amplifier is interchangeable with those employed in the line recorder, certain modifications were made on the pre-amplifier. An electrically operated zero check switch is included. This is operated automatically from a timer which should be set so that for approximately two minutes in each 30, the ion current does not flow through the grid resistor of the pre-amplifier. A shorting type switch is used for changing the contact and merely connects the collector wire from one end of the grid resistor to the other. The reason for employing a shorting type switch is to prevent the collector from acquiring the potential of the surrounding grid during switching and hence imparting a large transient pulse to the amplifier when it is connected back to the high end of the grid resistor.

In order to change the range of the instrument a second switch is provided to reduce the sensitivity by a factor of 100. This is done by connecting a resistor having a resistance of  $2 \times 10^8$  ohms in parallel with the normal grid resistor of  $2 \times 10^{10}$  ohms.

The output of the amplifier may be read either on the regular output meter or on the single point recording potentiometer which is connected to it. As in the case of the line recorder, the Leeds and Northrup recorder reads 200 mv full scale. It is equipped with alarm contacts and an extra slide wire for operation of a remote recorder.

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(d) Booster Pumps

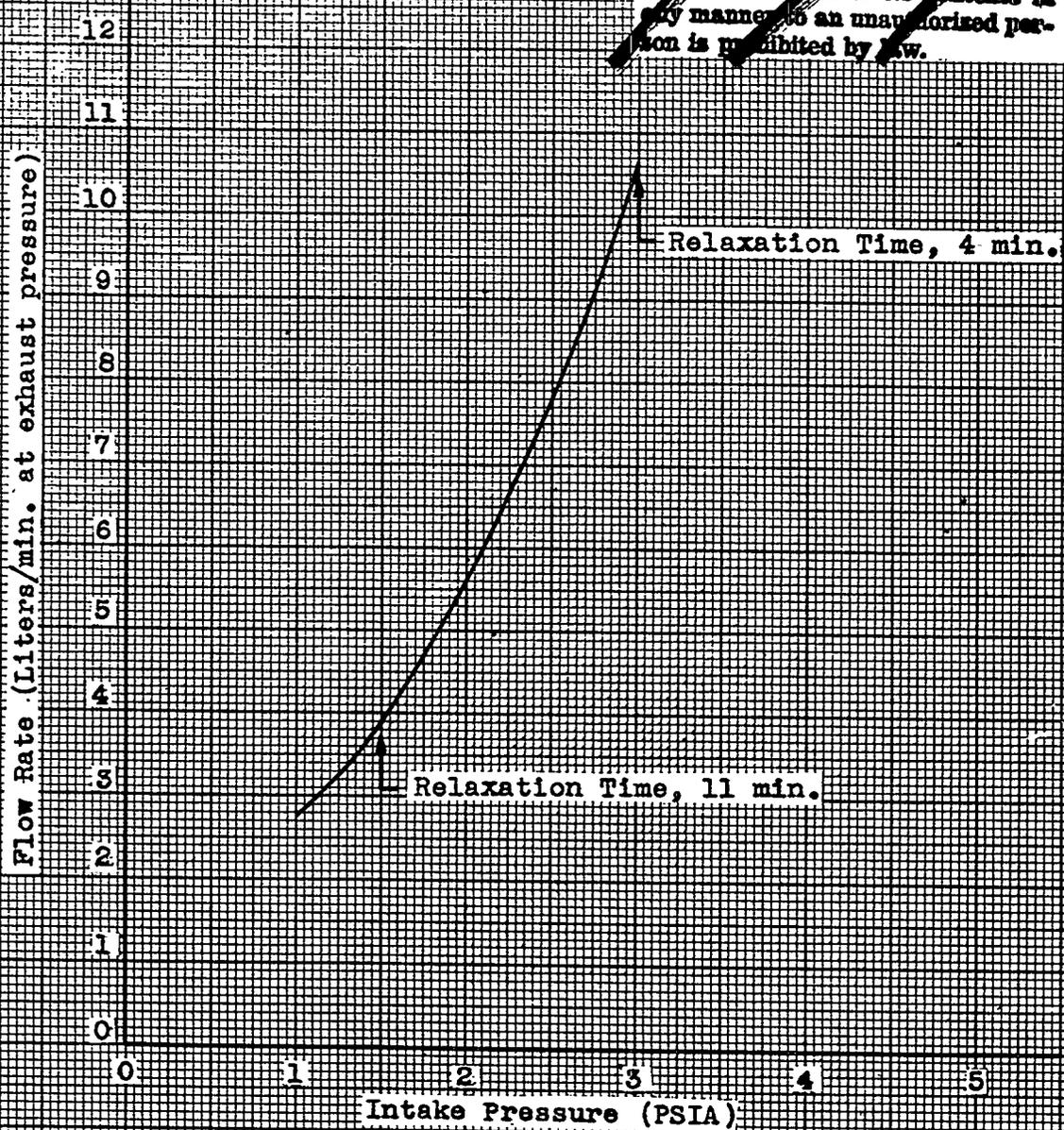
Two type B-4 pumps connected in series, 38 and 39, in Figure II-2 raise the pressure from a plant value of one to three psia to 10 psia, the normal operating pressure of the signal can. Figure III-1 shows the estimated flow rate in the signal can as a function of the inlet pressure to pump 38. Figure III-2 shows the variation of pressure between the two pumps as well as the average bellows pressures as a function of the inlet pressure to pump 38. Control valves 2 and 3 determine the pressure behind the bellows of the two pumps. For maximum bellows life each should be set at a value which is the mean of the respective inlet and outlet pressures.

(e) Voltage Supply

The potential applied to the grid structure of the signal can can be set at either 300 or 750 volts positive with respect to ground. A simple rectifier is employed and the voltage is held constant by the use of VR type voltage regulator tubes. A filter consisting of a resistance of 5 megohms and a capacity of 2 microfarads eliminates fluctuations. It is important that the potential of the grid structure should not vary since the variation would induce charges on the collector wire and hence result in instability of the amplifier output.

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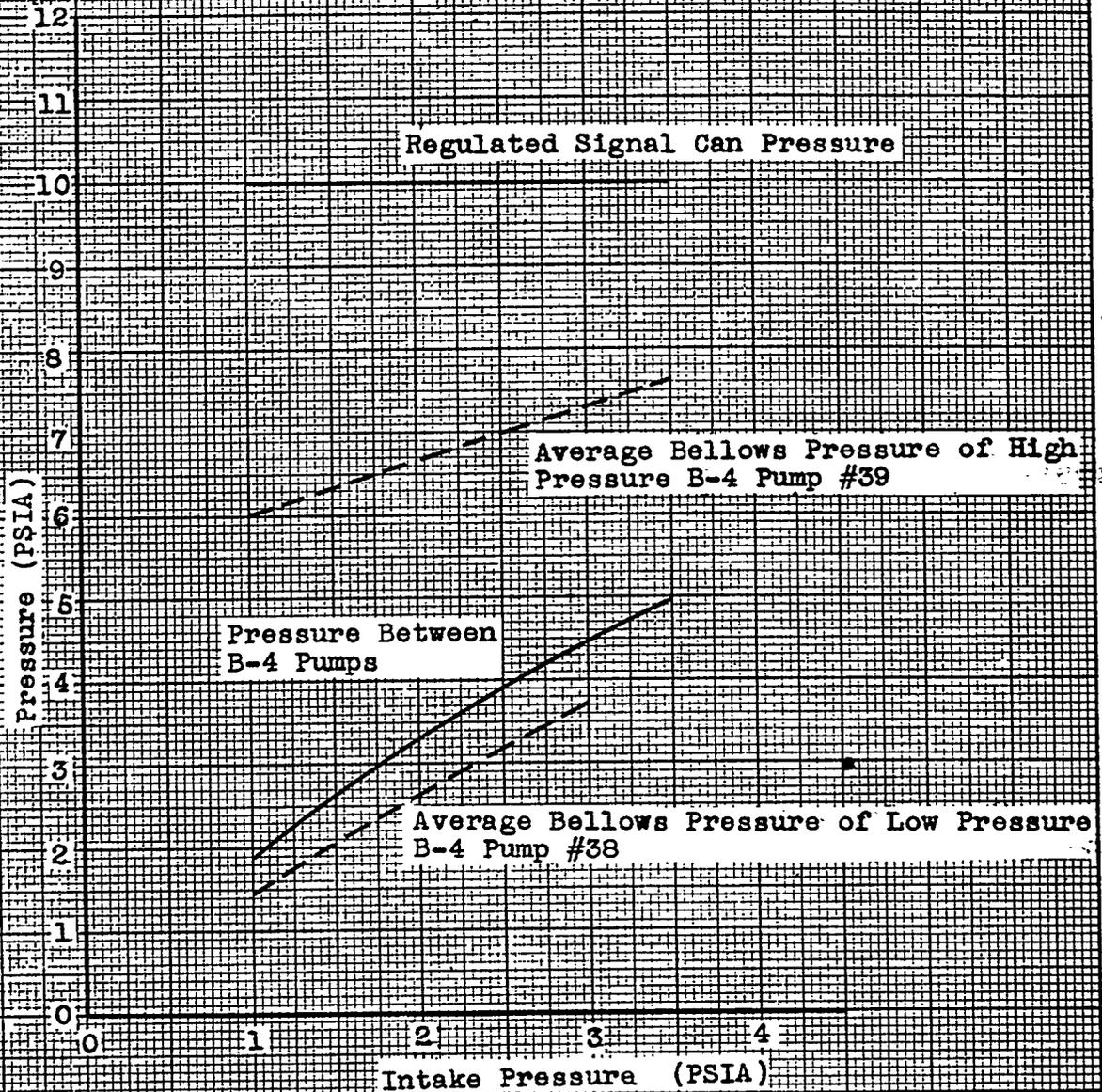
FLOW RATE VERSUS INTAKE PRESSURE USING TWO B-4 PUMPS

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FIGURE III-1

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PRESSURES OF B-4 PUMPS VERSUS INTAKE PRESSURE

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**FIGURE II-2**

IV. CHARACTERISTICS OF SIGNAL CAN

(a) Sensitivity

It has already been stated that the high energy alpha particles produced by the radioactive disintegration of 616 produce ions in their collisions with the gas present in the signal can. From the isotopic concentration and data on the radioactivity of the T isotopes (Table I) it is possible to compute the relationship between the concentration of 616 and the magnitude of the ion currents.

TABLE I

<u>Isotope</u>	<u>Decay Constant</u>	<u>Energy of Alpha Particle</u>	<u>Range in Air</u> (under std. conditions)	<u>No. ions formed in 74 gas by 1 particle</u>
T <sub>28</sub>	$4.86 \times 10^{-18}/\text{sec}$	4.21 MEV	2.7 cm	120,000
T <sub>25</sub>	$3.10 \times 10^{-17}/\text{sec}$	4.58	3.1	131,000
T <sub>24</sub>	$8.16 \times 10^{-14}/\text{sec}$	4.76	3.3	136,000

The contribution from the T<sub>25</sub> isotope activity is small compared to the total activity not only in normal 616 but for any enriched material since the T<sub>24</sub> enriches as least as much as T<sub>25</sub>. Thus the T<sub>24</sub> enrichment value must be known with the same accuracy as is desired for the 616 concentration measurement. The T<sub>25</sub> enrichment is not important except as it gives a rough indication of T<sub>24</sub> enrichment when the latter is not known. Table II readily illustrates this, by showing the relative rates of alpha particle emission of the three isotopes for normal 616 and for an arbitrary isotope mixture in which both T<sub>24</sub> and T<sub>25</sub> have been enriched to 10 times the concentration in normal 616 using as a standard 100 for the rate from T<sub>28</sub> in normal 616.

TABLE II

	<u>T<sub>28</sub></u>	<u>T<sub>25</sub></u>	<u>T<sub>24</sub></u>
Normal	100	4.6	100
Enriched 10 times 93		46	1000

Thus negligible error is made computing the concentration of 616 from the ion current and T<sub>24</sub> enrichment assuming the T<sub>25</sub> enrichment the same as the T<sub>24</sub> even though it may not be. The calculated expression given below has been checked by measurement with normal 616 and the agreement is better than 5%. In the calculation it was assumed that the can contains mostly 74 at a pressure of 10 psia and a temperature of 27°C. For other temperatures and pressures correction must be made according to the gas laws.

$$\text{Mol\%} = \left( \frac{1.86}{E + 0.86} \right) \times 1.9 \times 10^9 \times \frac{(T + 273)}{300} \times I$$

$$= \left( \frac{1.86}{E + 0.86} \right) \times 1.9 \times 10^9 \times \frac{(T + 273)}{300} \times V/R$$

- I = ion current (amp)
- R = grid resistor (ohms)
- V = signal (volts)
- E = T<sub>24</sub> isotope enrichment
- T = temperature in °C

Table III shows typical values obtained at 10 psia and 50°C for normal 616 and also for material in which the T<sub>25</sub> and T<sub>24</sub> have been increased by an amount such that the specific molar ionization is 10 times that of normal material. For the highest concentration a slight correction must be applied as discussed below.

TABLE III

<u>Mol% 616</u>		<u>Ion Current</u>	<u>Voltage Signal</u>	
<u>Normal</u>	<u>Enriched</u>		<u>Amplifier Resistor 2 x 10<sup>10</sup> ohm.</u>	<u>Amplifier Resistor 2 x 10<sup>8</sup> ohm.</u>
10	1.0	4.9 x 10 <sup>-9</sup>		0.98 v
1.0	10 <sup>-1</sup>	x 10 <sup>-10</sup>	9.8 v	98 mv
10 <sup>-1</sup>	10 <sup>-2</sup>	x 10 <sup>-11</sup>	0.98 v	9.8 mv
10 <sup>-2</sup>	10 <sup>-3</sup>	x 10 <sup>-12</sup>	98 mv	
10 <sup>-3</sup>	10 <sup>-4</sup>	x 10 <sup>-13</sup>	9.8 mv	

As the concentration of 616 in the can increases with a resulting increase in the ion current, a larger and larger proportion of the positive and negative ions form and recombine before they reach the electrodes. Below currents of 5 x 10<sup>-10</sup> amperes the error caused by the recombination is less than 10% for a grid voltage of 300 volts and less than 5% if the grid voltage is 750 volts. Figure IV-1, shows the percentage of ions collected as a function of the ion current. It is seen, for example, if the ion current is 10<sup>-9</sup> amperes and a 750 volt potential is applied to the grid, then 92.5% of the ions formed are actually measured. Our formula above can be corrected with the use of

Figure IV-1. For signals above  $6.5 \times 10^{-9}$  amperes, corresponding to a mol concentration of 20% normal (or 2% of ten times as radioactive enriched material)  $^{61}\text{Lr}$  in the signal can, the effect of recombination becomes too great to permit accurate measurements. Since the ion current is proportional to the partial pressure of  $^{61}\text{Lr}$ , it is recommended that for such high concentrations the signal can be operated at a much lower pressure. For example at 2 psia analyses could be made on 100% normal  $^{61}\text{Lr}$  and 10% enriched material. (Enriched material which will be frequently referred to from now on has been arbitrarily chosen for purposes of illustration to mean a specific alpha activity 10 times that of normal. In the plant the specific activity at any time will have to be determined by the alpha counting method). At pressures below 5 psia the range of the alpha particles becomes comparable to that of the signal can and there will be a loss in sensitivity due to the fact that some of the alpha particles will hit walls before they have fully dissipated their energy by forming ions in the gas within the grid. Figure IV-2 shows this effect and may be used for correction purposes in the event low pressures are ever employed.

Flows of gas through the signal can of less than 12 liters/minute (measured at the pressure of the signal can) may be used without cutting the sensitivity (at any concentration of  $^{61}\text{Lr}$ ) by more than 5% if the grid voltage is as low as 250 v. Figure IV-3 shows the effect of flow upon the size of the signal. If higher grid voltage is employed, the effect is even less, as may be seen in the graph.

#### (b) Background Ion Current in Signal Can

The ever present residual ion current puts a low limit on the concentration of  $^{61}\text{Lr}$  which can be measured in the signal can. This background is due to a number of causes;

- (1) All materials contain traces of radioactive elements. The materials from which the signal cans are constructed are no exception to this rule.
- (2) The passage through the signal can of gamma rays from the surroundings and cosmic rays produces a small residual ionization current.

The combined effect of (1) and (2) produces in general an output signal of less than 2 mv. (Voltage signals given are for a grid resistor of  $2 \times 10^{10}$  ohms unless otherwise stated). The presence of a tank containing 150 lbs of normal  $^{61}\text{Lr}$  placed 9 inches away from the signal can gave a signal of 5 mv and when moved four feet away, 0.5 mv. Thus the presence of large amounts of  $^{61}\text{Lr}$  in the plant should have little effect.

- (3) The radioactive disintegration of T results in the

formation of actinon and radon. It can be shown that the amount of actinon present in the signal can will be entirely negligible. On the other hand, the concentration of radon will gradually increase with time. At the end of the first year of operation of the K-25 plant, the concentration in the purge cascade will be such as to produce a signal of less than 20 mv. After a number of years of operation, the concentration might rise sufficiently high to cause some trouble.

- (4) The admission of 616 to the signal can results in a deposit of radioactive compounds on the internal surface of the can, which gives rise to the most serious source of background. The chemical reaction of 616 with the surfaces of the can or with any substances occluded on the surfaces, such as water vapor, produces non-volatile T compounds which, of course, emit alpha particles. Although the grid structure minimizes the effect of this source, it does not remove it completely. Also, the radioactive disintegration of the T in 616 results in the formation of non-volatile beta and gamma emitters which deposit on the walls of the can.

Figure IV-4 shows the manner in which the background depends on the exposure of the can to normal 616. Exposure is arbitrarily defined as the integrated concentration of 616 in mol percent, multiplied by the number of hours of running. It is found that the background varies with the pressure, and is greatest at 2.5 psia. At this pressure the background consists mainly of alpha particles, originating from the T reaction products on the large surface of the can itself, which have a range extending beyond the grid into the collecting region. The curve for the background at 2.5 psia indicates that after about 0.3 hour percent exposure in a well treated can, the reaction of 616 with the walls and occluded substances essentially stops as indicated by the fact that the curve levels off at about this exposure. However, at the normal operating pressure of 10 psia the background continually rises even though the chemical reaction of 616 has apparently ceased. It is reasonable to assume that this rising background at 10 psia is due to beta particles, gamma rays, or both, coming from the successive radioactive disintegrations of  $TX_1$  (the decay product of  $T_{28}$ ). Beta particles and gamma rays coming from material on the walls have a much greater range than the alphas and will enter the collecting region at the normal operating pressure of 10 psia. This assumption also indicates how the background at 10 psia can continue to build up after the T reaction products cease being deposited.

The alpha background is proportional to the enrichment and at the normal operating pressure of 10 psia is less than 1 mv for normal 616 and less than 10 mv for enriched material. All studies indicate that as far as this sort of background is concerned, the useful life (time it takes for background to become equal to signal for  $10^{-3}$  mol percent normal 616) of the can is possibly 100 hour percent or more. To increase the useful life, the signal can should be well baked out and fluorinated; also, exposure of 616 should be minimized in one of the two signal cans for the purge cascade by using only at the lowest concentrations.

It is our guess that the background due to beta rays, gamma rays, or both, will not rise more rapidly for enriched 616 than for normal; on the contrary, it will rise at a slightly slower rate and will not exceed 1 mv at 1 hour percent exposure; 20 mv at 100 hour percent; and 100 mv at 1000 hour percent. Even if this background is proportional to enrichment, it will not exceed 10 mv at 1 hour percent, which will correspond to six months useful life for one of the signal cans operated as recommended (see discussion on operating conditions).

HF1

(c) Effect of OA on Signal Can

The principal contaminant in the 74 at the top of the purge cascade will probably be OA. A signal can at 70°C was run for several weeks at varying 616 concentrations with 2-4% OA present the majority of the time.

Under these conditions the background curve shown in Figure IV-4 was the same as when no OA was present, showing that the presence of OA has no adverse effect on the background. There is a measurable leakage resistance of between  $10^{10}$  and  $10^{11}$  ohms across the MFP-10 insulators for 2 to 5% OA. However, the use of a 100% feed back amplifier minimizes the effect of this leakage, giving no apparent loss of sensitivity, instability, or zero shift of the amplifier even with an  $8 \times 10^{10}$  ohms input resistance. At 5-10% OA the leakage does become sufficient to make measurements impossible; however, it is not anticipated that concentrations of this magnitude will normally be reached.

(d) Effect of Temperature on Signal Can

It has been shown that the signal can may be operated at a temperature as high as 70°C without producing harmful effects such as

- (1) Abnormal rate of increase of background due to increased corrosion rate.
- (2) Flow of plastic gaskets producing inward gas leakage.
- (3) Poor operation of preamplifier (only noticeable effect was a small amplifier zero temperature coefficient).

(e) Accuracy

The absolute accuracy of the space recorder will be limited by our knowledge of the following factors:

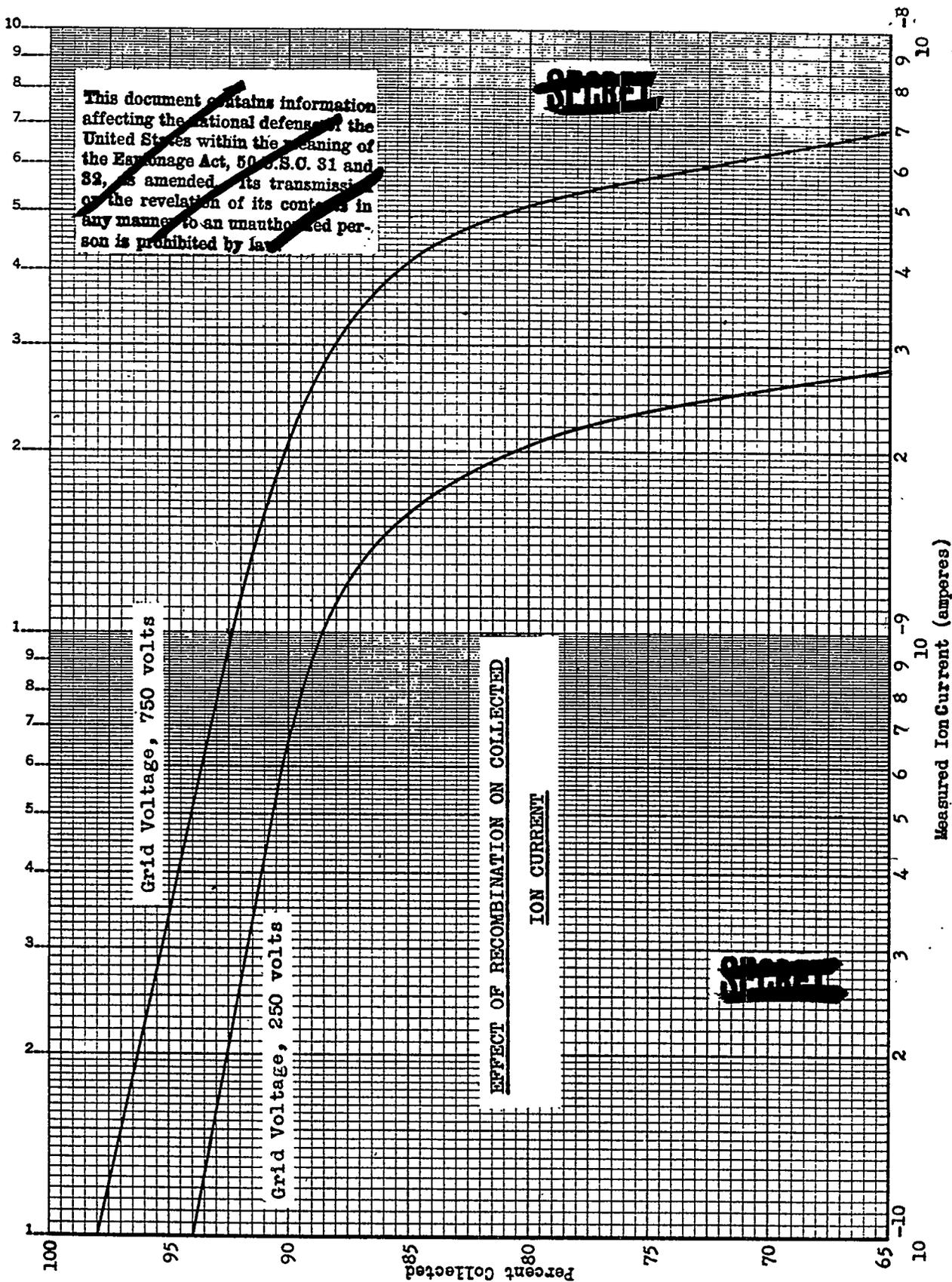
- (1) Isotope enrichment of the 616. It is only necessary to know increase in T<sub>24</sub> which must be determined by other methods.
- (2) Value of the preamplifier grid resistor. The specifications

require that the absolute resistance value of this resistor be known to within 5% and that the ratio of the resistance of the  $2 \times 10^8$  ohm resistance to the  $2 \times 10^{10}$  ohm be known to 2%. Rather than employ the formula given in this report for the sensitivity, it is possible to make a direct calibration by introducing a definite mixture of 616 in 74 into the can.

- (3) Amount of recombination. With the use of the experimentally determined recombination curve shown in Figure IV-1, the error from this factor should not exceed several percent.
- (4) Pressure and temperature of gas. The error due to inexact measure of these quantities should be far less than the others mentioned.

The emission of alpha particles occurs at a random rate causing some fluctuations of the signal with the smaller signals showing a greater percentage fluctuation. For a signal of 10 mv, the smallest signal which we can measure conveniently, this variation will be  $\pm 10\%$ . There should be no trouble in reading the mean of the fluctuations to within 25% of the total variation, giving in the worst case a possible accuracy of 5% in the reading of the total signal.

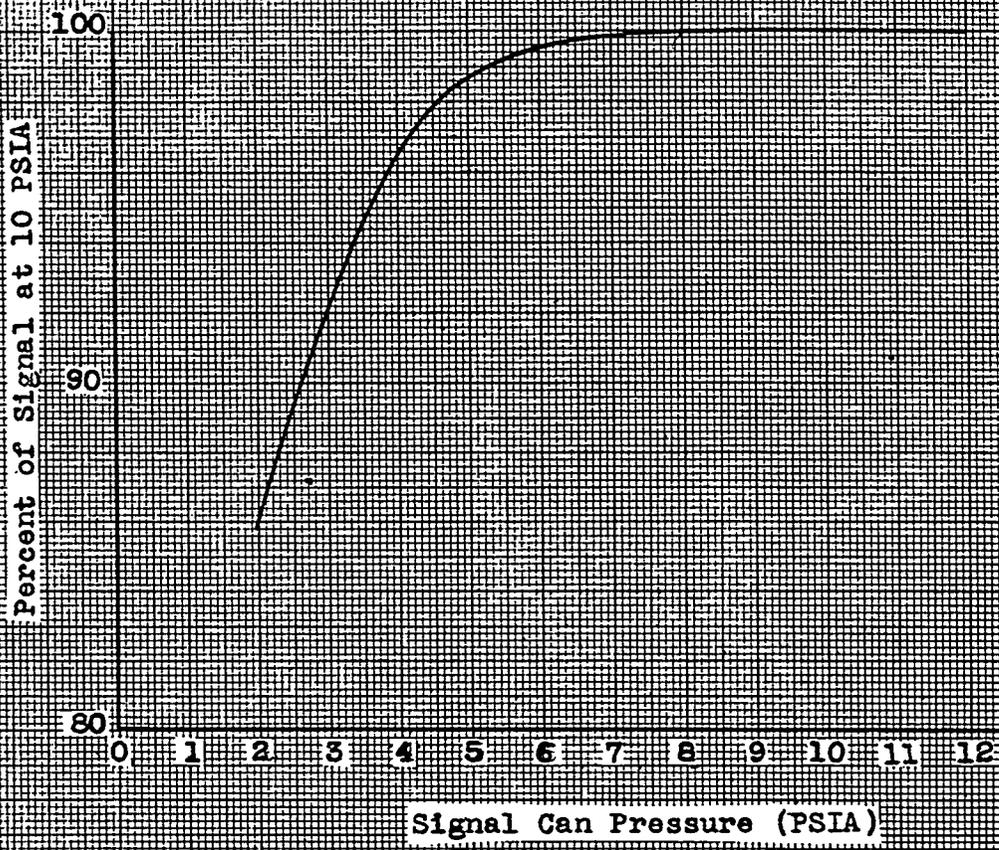
By far the limiting factor as far as accuracy is concerned is the size of background relative to signal due to gas. In the next section recommendations are given for keeping the background in the worst case not greater than the minimum signal which will be expected. Even under such conditions where the background is as large as the signal it should be possible to determine the signal to within 10% if the background is checked at sufficiently frequent intervals. Perhaps once per week will suffice for this measurement when the background is high.



**FIGURE IV-1**

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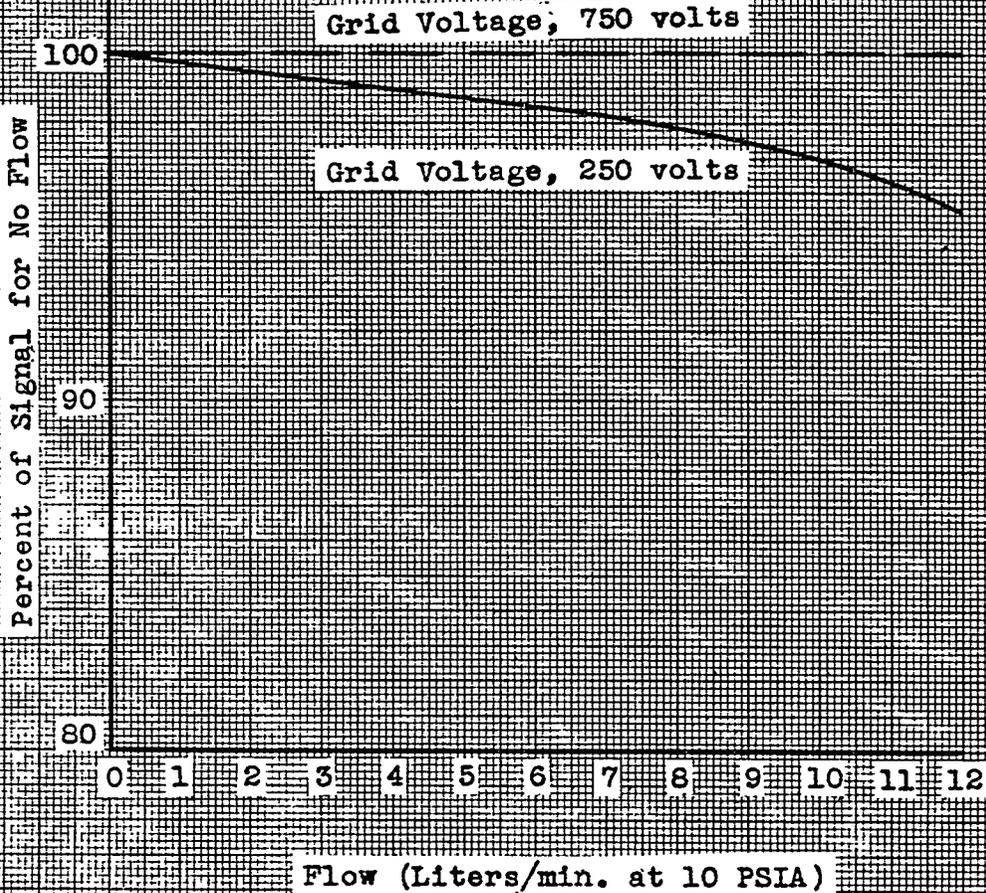
RELATIVE SIGNAL VERSUS TOTAL SIGNAL CAN PRESSURE  
FOR CONSTANT 616 PARTIAL PRESSURE

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**FIGURE IV-2**

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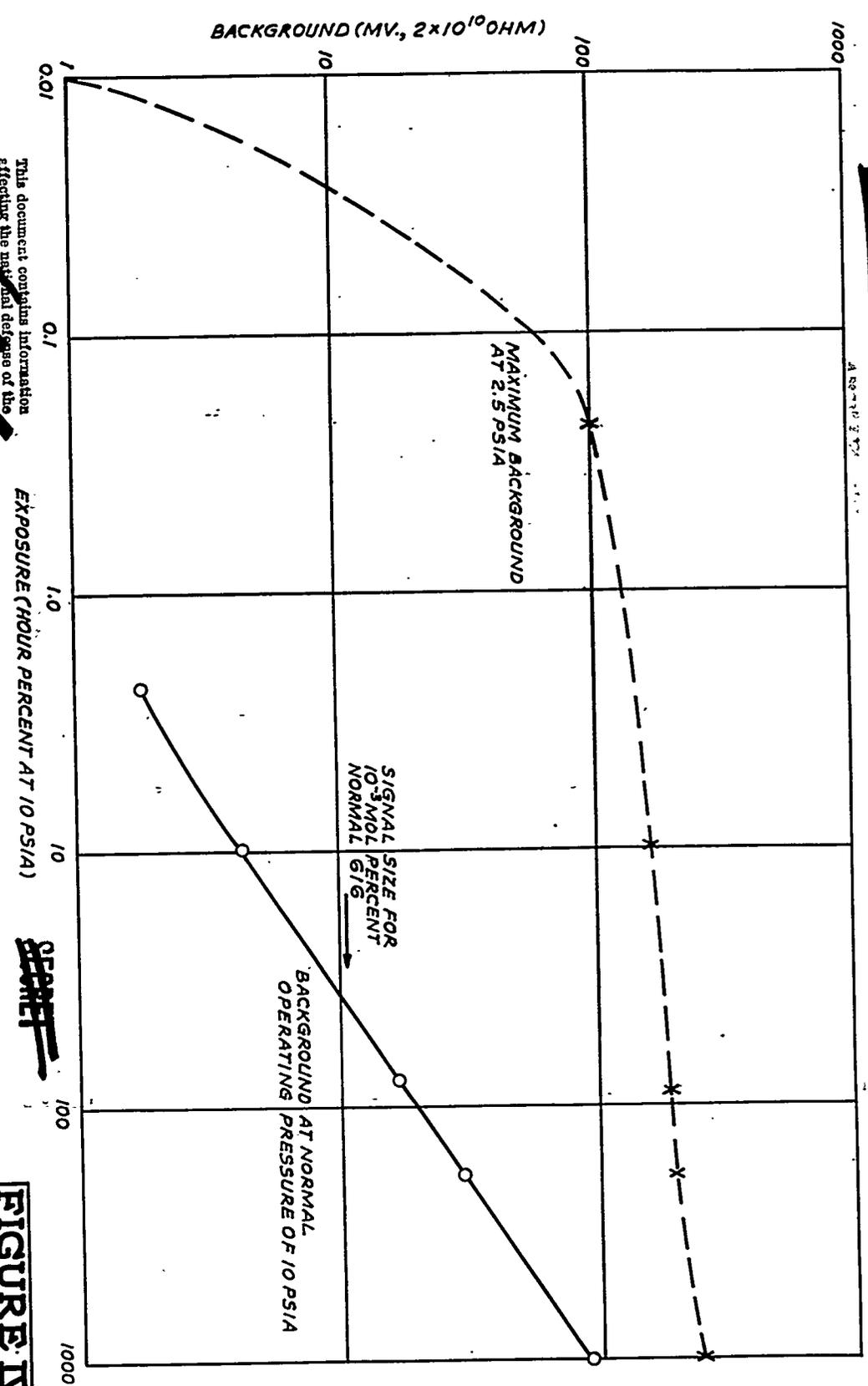
RELATIVE SIGNAL VERSUS FLOW

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**FIGURE IV-3**

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48-110 377



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EXPOSURE (HOUR PERCENT AT 10 PSIA)

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TITLE BACKGROUND VERSUS EXPOSURE

FIGURE IV-4

DESCRIPTION	DATE	CHECKED	REVISIONS

THE KELLEX CORP.

JOB NO. \_\_\_\_\_  
 SCALE \_\_\_\_\_ DATE \_\_\_\_\_  
 DR. \_\_\_\_\_ No. \_\_\_\_\_  
 -G

V. RECOMMENDED OPERATING PROCEDURES

(a) Pressure

It is recommended that the space recorder be normally operated at a pressure of 10 psia. Higher pressure decreases a little the background current due to alpha particles arising from corrosion products, but will cut the flow slightly and hence lengthen the time response. With a flow of 4 liters per minute measured at the pressure of the signal can, the relaxation time of the can will be approximately 11 minutes.

(b) Temperature

It is recommended that the temperature of the signal can and preamplifier should not exceed 60°C.

(c) Grid Voltage

For low concentrations of 616 a grid voltage of 300 volts should be used. Recombination effects are negligible in this range and the background ionization current is somewhat less than it is at 750 volts. For higher concentrations when the amplifier signal exceeds 100 millivolts using the  $2 \times 10^8$  ohm amplifier resistor the grid voltage should be 750 volts in order that the effect of recombination be minimized as much as possible.

(d) Exposure of Signal Can to 616

Inasmuch as the rate of increase of ionization current background in a signal can depends upon the exposure to which the can has been subjected, it is desirable to hold the exposure to a minimum if one intends to use the can for detecting the presence of low concentration of 616. It is suggested that the range of analyses in the purge cascade be divided between the two signal cans and that the time of running at any concentration be roughly inversely proportional to the concentration. Thus if one chamber is never used for concentrations of normal material above 0.1 mol%, and the times of running recommended in Table II followed, the background should not exceed 20 millivolts in six months.

TABLE IV

<u>Signal</u> <u>(<math>2 \times 10^{10}</math> ohm)</u>	<u>% Enriched</u>	<u>% Normal</u>	<u>% of Operating</u> <u>Time Permitted</u>
10 mv	$10^{-4}$	$10^{-3}$	100%
100 mv	$10^{-3}$	$10^{-2}$	40%
1000 mv	$10^{-2}$	$10^{-1}$	4%

The other signal can would be used for the higher concentrations and if the can were never needed for low concentrations it could be run for very long periods without necessary cleaning. Needless to say, the background in the high concentration can would be too great to permit the use of this can for checking the low concentration can, at the lowest concentration. As long as the 616 is approximately of normal enrichment, the rate of rise of background for long exposures is known. However, for enriched 616 it is not certain just how fast the background will rise especially for exposures above 1 hour percent. If the supposition described previously for the nature of high backgrounds at high exposures proves to be correct, then we should expect the background given in Table V for various exposures. This table arbitrarily assumes that the enrichment is such as to give a 10 fold increase of ionization current.

TABLE V

Mol concentration of 616 (enriched)	Time of Continuous Operation			Background ( $2 \times 10^{10}$ ohms grid resistor)
	0.1%	1.0%	10%	
<u>Exposure (hour percent)</u>				
100	1 month	1/2 week	1/2 day	30 mv
1000	2.5 years	1 month	1/2 week	100
10,000	25.0 years	2.5 years	1 month	400

Thus the instrument could be run for at least six months at almost any concentration and the background would remain small enough to give accurate analyses at concentrations above  $10^{-2}$  mol percent of enriched 616 (signals above 1.0 volt) and approximate checks on the other can at intermediate concentrations.

In the event that the explanation is not entirely correct, the background may rise more rapidly with exposure to enriched 616. Table VI shows that the maximum expected background would be for tenfold enriched 616 if the background rise is proportional to the ionization current.

TABLE VI

Mol Concentration of 616 (enriched)	Time of Continuous Operation			Background ( $2 \times 10^{10}$ ohms grid resistor)
	0.1%	1.0%	10%	

TABLE VI (Continued)

<u>Exposure (hours percent)</u>	<u>Time of Continuous Operation</u>			<u>Background</u> ( $2 \times 10^{10}$ ohms grid resistor)
	<u>0.1%</u>	<u>1.0%</u>	<u>10%</u>	
100	1 month	1/2 week	1/2 day	300 mv
1000	2.5 years	1 month	1/2 week	1 volt
10,000	25.0 years	2.5 years	1 month	4 volt

(e) Measurement of Background

The background is measured by flushing out and filling signal can with pure dry 74 to 10 psia (see next section for instructions). In general the longer the exposure the less frequently the background need be measured. In the case of the instrument used for low concentrations, the background at the start should be measured every day or two until it ceases to rise rapidly upon further exposure, which may take several weeks. Then measurements once a week should be sufficient. Likewise the background of the instrument for higher concentrations should be measured every day or two for the first several weeks. Whatever the conditions are when the signal can is operating on a cell line, namely pressure and grid voltage, they should be the same for background measurements. The 74 used for background measurements should be dried to a dew point of  $-70^{\circ}\text{F}$ . to prevent contamination of signal can walls with water vapor which will react with 616 and increase background. By no means should 74 containing hydrogen such as is used for a reducing atmosphere in high temperature soldering be used.

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## VI. MAINTENANCE

### (a) Installing and Removing Signal Can

After the initial installation, all parts of the manifold in which there has been moist air and through which the gas stream flows must be evacuated and flushed with dry 74 several times. To begin with the inlet and outlet valves 17, 18, 19, 20 and 21 (see Figure II-2) and valves 16 and 40 on the signal can are closed. After the B-4 pumps and signal can are in place and connected and valves 14 and 31 open, the section between valves 17 and 16 is evacuated through the lines containing valves 13, 15, 48 and 25; that between valve 40 and the outlet valves is evacuated through the lines containing the valves 26, 29, 23 and 25. A fore pump is all that is necessary for this evacuating. These parts of the system are flushed several times by admitting dry 74 to atmospheric pressure and pumping out. Then valves 13, 15, 48, 26, 29 and 23 are closed, 16 and 40 opened, and the 74 in the signal can and manifold allowed to flow out through one of the outlet valves 18-21. The B-4 pumps may then be turned on, inlet valve 17 opened, and pressure controller set to the desired operating pressure of 10 psia. Whenever the B-4 pumps are on, exhaust valves 14, 16, 40, 31, and any one of 18 through 21 must absolutely be open. Neglecting to do so may burst a B-4 pump bellows.

In subsequent installations all lines will have been previously filled with dry 74 up to connections 36 and 37 and capped; the signal can then need only be connected, the 74 emptied into the plant through 14, 16, 40, 31 and one of 18-21, and the procedure from then on continued as directed above.

Before removing the signal can, it should first be filled with the lowest concentration of 616 from purge cascade. Then valve 17 is closed and system emptied to as low pressure as is possible through one of the outlet valves 18-21. The controlling valve, 31, must be set open for this. The B-4 pumps are then turned off and outlet valve and valve 14 closed. The system between 14 and the outlet valves including the signal can is then flushed several times admitting 74 to atmospheric pressure through 15 and 48 and draining into plant. Finally this part of the system is filled with 74 to 1 psig, valves 16, 40, and 15 closed, the signal can removed, and the connections 36 and 37 on the manifold capped. The above flushing reduces the 616 concentration remaining in the signal can to less than 1 per cent of the concentration of the last gas admitted to the can from the plant. This same flushing procedure is to be used for obtaining background measurements except the final pressure of 74 in the can is set at 10 psia.

The replacement of a B-4 pump involves no more complicated procedure than that for removing and installing a signal can. After the system is emptied to low pressure by closing 17 and the pumps

turned off, valve 16 is closed and the system isolated between 17 and 16 flushed with dry 74 several times. Because of the high resistance to flow of the B-4 pumps when not running, the 74 is admitted through both 13 and 15 and emptied back through 17 and also through 16. After the pump is removed the connections 32 and 33 or 34 and 35 are capped.

(b) Pretreatment of Signal Can

If precaution is not taken to pretreat the signal can at the beginning and after cleaning, and to always use thoroughly dried 74 in making background measurements, the backgrounds obtained will be abnormally higher than predicted and will even prevent measuring low concentrations.

At the beginning and after every cleaning the signal can must be treated by baking to remove as much absorbed water vapor as possible and by exposure to hot 216 to make the surface passive to 616. This treatment should be done on an auxiliary manifold which includes an oven into which the signal can can be easily placed, fore pump, diffusion pump with a speed of at least 5 liters/sec., liquid 74 trap, and means for admitting and pumping out 216 and admitting dry 74. Needless to say, for this large can the trap and pumping lines must also be large. The 216 must be pumped or flushed out with dry 74 through separate lines to a chemical trap or hood.

Baking should be done at 70°C (not above since the plastic gaskets then start to flow) and for at least 10 hours while continuously pumping to a pressure less than  $10^{-4}$  mm. The can is then treated with 216 at 70°C, using a mixture of 25% 216, 75% 74 at atmospheric pressure. A treatment of 40 hours was found satisfactory in tests made in the laboratory. After removing the 216 the can should be filled with dry 74 to 1-2 psig for transferring to the main manifold. See Section V-a for installation instructions.

(c) Cleaning Signal Can

When the background has become too high to permit accurate analysis at the lowest concentrations, the can should be removed as described in Section V-a and washed thoroughly by circulating water through it for an extended time.