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INSTRUMENTATION FOR ANALYSIS
OF THE INERT GASES USED IN FFTF

By

G. B. Barton

Chemistry Research Department
Chemistry and Metallurgy Division

March 1969

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G. B. Barton

ABSTRACT

Inert gas monitoring needs of the FFTF are reviewed in this document, and the selection criteria for the required instrumentation is defined. Current technology and commercially available on-line gas analytical instrumentation are reviewed and compared to FFTF requirements. Results of this study conclude that FFTF cell blanket gas analytical needs can be met in almost all instances by commercially available instrumentation. Current technology is inadequate to meet sodium cover gas monitoring needs and a development effort is required. Gas chromatography and possibly mass spectrometry appear to hold the greatest promise of meeting sodium cover gas monitoring needs.

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INSTRUMENTATION FOR ANALYSIS OF THE INERT GASES
USED IN FFTF
G. B. Barton

INTRODUCTION

A necessary feature of FFTF will be the provision of a blanket of appropriate inert gas for various reactor locations to guard against atmospheric attack upon the sodium coolant, and upon sensitive structural components. It will be very important that the quality of these inert gas blankets be ensured during the reactor operation. To provide such assurance, the impurity content of the blanket gases will have to be monitored routinely.

The cover gas impurities requiring routine determination are water, oxygen, hydrogen, nitrogen, carbon dioxide, carbon monoxide, and methane. (Other impurities--higher hydrocarbons, other types of organic compounds, and nitrogen oxides--may also be present on occasion and require occasional checking.) The analytical sensitivities required will depend upon the function of the gas blanket in the particular reactor location being monitored. Required sensitivities will range from the high sensitivities required for monitoring the argon (or helium) cover gas for the sodium coolant with the maximum impurity limits of the order of 10 ppm to the relatively low sensitivities needed for monitoring the nitrogen to be used where prevention of fire is the chief concern.

This report lists the various gas applications as outlined in the Conceptual System Design Description (CSDD)⁽¹⁾ for the inert gas receiving and processing system, summarizes the existing analytical instrumentation technology as it pertains to each application, describes the selection bases and criteria for instrumentation for each application, suggests promising instrumentation for many applications and, finally,

mentions briefly some of the research and development programs which will be required to ensure the availability of proven gas analyses instrumentation for the FFTF.

CONCLUSIONS

The two classes of inert gas application of concern in FFTF, i.e., the cell blanket gas and sodium cover gas, require a different set of instrumentation. The cell blanket will require analysis for oxygen and hydrogen. It will probably be desirable to measure water and nitrogen oxides also. For these analyses the following instruments are recommended:

Oxygen	- Beckman Paramagnetic oxygen analyzer
Hydrogen	- M.S.A. Thermatron
Water	- Panametrics hygrometer
Nitrogen oxides	- Laboratory chemical method using grab samples unless need for continuous analysis is established.

The sodium cover gas will require analysis for hydrogen, oxygen, nitrogen, methane, carbon monoxide and the capability to detect other unknown impurities. Basically these substances can be measured by either a gas chromatograph or a mass spectrometer. However, there may be interference from sodium vapor. Additional work is needed to define the magnitude of this interference and the advantages and disadvantages of both types of instruments when applied to this service before a recommendation is made.

APPLICATIONS OF INERT GAS BLANKETS

The Conceptual System Design Description (CSDD) documents⁽¹⁾ list the potential applications of inert gases and locations where analyses may be needed. When this information is considered it becomes apparent that the analytical requirements will be divided into two classes: 1) the cell blanket

gas (nitrogen) used to exclude high (>5%) oxygen concentrations from spaces where sodium leaks could conceivably occur, and 2) the sodium cover gas (argon or possibly helium) used to prevent contamination of sodium by reactive gases.

Information on the impurities found in the cover gas of the sodium cooled reactors that have been operated to date was summarized by H. P. Maffei.⁽²⁾ The data he has collected together with estimates of impurity contents of the gases as they should be received from suppliers and some estimates of the amounts of impurities that may be added to the gases by leaks (by indiffusion of air components, etc.) have been combined to prepare the estimates of the levels of impurities that the analytical instruments will have to be designed to measure. These estimated impurity levels are presented in Table 1. The location and number of sampling points are given in Table 2. The data given in Table 1 for argon and helium indicate the impurity levels expected to be observed in the sodium cover gas and in the fuel transfer and examination cell applications listed in Table 2. The data for nitrogen apply to the remaining cell applications listed in Table 2.

ANALYTICAL REQUIREMENTS

Sodium Cover Gas

The impurities of concern in the sodium cover gas are nitrogen, methane, hydrogen, oxygen, carbon monoxide and carbon dioxide. Consideration of reactions between sodium and these various impurities lead to the conclusion that it is very unlikely that oxygen, carbon dioxide or water will be measurable in a sample of the cover gases that has equilibrated over molten sodium. These substances react rapidly with sodium and will therefore be removed before they can be measured. It will be difficult to detect small leaks into the sodium cover

TABLE 1. *Estimated Range of Impurity Levels in Inert Gases*

Major Gas	Impurity Concentrations, ppm									Nitrogen Oxides
	H ₂ O	H ₂	O ₂	N ₂	CO	CO ₂	CH ₄	Ar	He	
N ₂	100 - 5000	0 - 40,000	1000 - 50,000	NA ^(a)	0 - 50	0 - 50	0 - 10	0 - 15,000 ^(b)	0 - 10 ^(b)	0 - 100
He	0 - 5	0 - 100	0 - 5	0 - 20 ^(c)	0 - 100	0 - 10	0 - 100	Trace ^(b)	NA	0
Ar	0 - 5	0 - 100	0 - 5	0 - 100 ^(c)	0 - 100	0 - 10	0 - 100	NA	Trace ^(b)	0

a. NA = Not applicable

b. Innocuous impurities

c. Concentration will depend upon use of nitrogen removal equipment.
In the absence of nitrogen removal equipment, ranges up to 10,000 ppm
or more are possible.

TABLE 2. *Inert Gas Applications in FFTF
that Will Require Analysis*

Argon	Number of Sampling Points	
	Shown in CSDD	Suggested
Primary Argon Supply	1	4-5
Primary Gas Recycle (Tanks TK 1-2 and TK 1-3)	2	2
Reactor Primary Heat Transport System	5	6
Reactor Primary Sodium Receiving and Processing System	2	3
Closed Loop Primary Heat Transport System	12	12
Closed Loop Primary Sodium Receiving and Processing System	6	6
Reactor Secondary Heat Transport System	4	4
Reactor Secondary Sodium Receiving and Processing System	2	3
Closed Loop Secondary Heat Transport System	6	6
Closed Loop Secondary Sodium Receiving and Processing System	2	2
Fresh Sodium Purification and Storage	2	2
Contaminated Sodium Processing and Storage	2	2
Interim Fuel Decay Storage	1	1
Fuel Transfer and Examination Cell	2	6
Fuel Handling Machine	1	1
<u>Nitrogen</u>		
Closed Loop Cell	0	6
Reactor Primary Sodium Purification and Storage Cell	0	1
Reactor Primary Heat Transport Equipment Cell	0	4
Contaminated Sodium Processing Cell	0	1
Radioactive Maintenance Cell	0	1
Reactor Access Space	0	1
Reactor Hot Cell (alternative)	0	1
Reactor Cavity	0	1
Primary Nitrogen Supply	1	5-6

gas system by monitoring either the oxygen or nitrogen content of the gas. It is anticipated that oxygen will be removed by the sodium before it can be measured. Past reactor operating experience has shown cover gas nitrogen contents to be high under normal operating conditions. Small increases in the already significant amount of nitrogen present will be difficult to detect. The other anticipated impurities (hydrogen, methane and carbon monoxide) react more slowly with sodium and therefore may be present in measurable amounts in the cover gas.

It will be necessary to verify that the supply of cover gas is of high purity in order that information can be collected about the system behavior from the analyses of the gas in contact with the sodium. One of the goals of the FFTF Development Task SG-6 is to determine whether the chemistry of impurities in the sodium can be inferred from the analysis of the cover gas. These various objectives indicate the desirability of having analytical instrumentation that is capable of measuring the impurities in the sodium cover gas at the one part per million level with the possible exception of nitrogen. Because nitrogen has been found in the cover gas of most reactors operated to date and its removal may not be required, the analytical sensitivity requirement is not expected to be as high for nitrogen.

It is expected that the sodium cover gas will be under pressure at most sampling points. Where the gas is not pressurized a pump will have to be provided to induce the sample gas flow. The large number of sample points listed in Table 2 indicates a need for multiple instruments on the sodium cover gas system.

Cell Blanket Gas

The data in Table 1 indicate that the range of impurities expected is about the range that is found in many protective atmosphere applications so the instruments required are available. The major problem is to select the ones most applicable to FFTF needs. Sampling the gases from the various cell enclosures should not prove to be a particularly difficult problem. The schematic diagrams of the various cell systems given in the CSDD indicate that there are blowers in each cell which should provide an adequate source of sample for measurement of the composition of the gases in these spaces.

Sampling

Any analysis is truly valid only for the sample on which the measurement is made. In order that the results of an analysis may be related to the source from which it is taken, the sample must be truly representative of the whole when separated and must remain unchanged by the separation or collection procedure. These requirements are particularly difficult to satisfy in the sodium cover gas systems for two reasons. The first factor is related to inhomogeneities within the cover gas. Significant concentrations of sodium vapor will exist in the gas space above the sodium in the various places of gas containment. If there is a leak at one point in the vessel permitting air to enter slowly, the oxygen and water vapor contained in the entering air will react rapidly with the sodium vapor until they are consumed. If the leak is remote from the sampling point and the gas is not being rapidly mixed, it is unlikely that any oxygen gas will survive long enough to be detected, whereas a leak near the sampling point may be detected. It has also been reported that stratification according to gas density has been found in the cover gas of

some operating reactors.⁽³⁾ A second factor obstructing valid sampling of a sodium cover gas is potential deterioration of sample validity in the sample lines. An example of this problem occurs in the case of hydrogen. The sample would generally originate from a gas at high temperatures and would contain significant sodium vapor. If the gas sample also contained hydrogen, a reversible reaction is possible since sodium and hydrogen can react to form sodium hydride which is decomposed by heat.⁽⁴⁾ Condensation of a vapor also often results in gettering a gaseous component. Thus, we can have removal of components from the gas space by chemical or physical processes.

To enable the analysis to represent the original condition truly, the sample would: a) have to be delivered directly--at temperature--to an instrument capable of receiving it and measuring it, b) have to undergo known reproducible reactions whose products could be measured, or c) not change in composition with the changing conditions. Of the major methods currently available, only mass spectrometry offers promise of being able to handle gases directly at temperatures approaching those expected in FFTF. The effects of sodium vapor on this instrument are unknown. Gas chromatography of the anticipated gaseous mixtures has not been extended to the elevated temperatures expected. The equilibria and kinetics of the possible reactions are not known well enough to make it possible to cool the sample to a lower temperature, analyze it, and correct for the reactions occurring during cooling. These considerations of the effects of sodium vapor on a) the sample composition as the temperature is changed, b) as the sodium is removed, or c) on the instrument, also apply to instruments other than the gas chromatograph and mass spectrometer that can measure the trace impurities. The present method of removing sodium vapor is by condensation in a trap. The effect of this trapping on gas sample composition remains to be quantitatively defined.

In addition to the problems with sample composition as it may be affected by sodium, we have the considerations of sample collection or distribution from the points of origin to the instrument, whether this flow should be continuous or intermittent, and whether the gas volume is of uniform composition. If only an occasional analysis is required, then intermittent sampling may be adequate. However, in general it will be necessary to have continuous flow of the gas stream from the desired point of origin to the instrument where the sample is being analyzed. (The use of "grab" samples of these high purity gases is so unreliable as to be excluded from consideration.) There are a large number of points listed in Table 2 for the various systems that employ the high purity gases as sodium covers. Continuous collection of these gases of course can be accomplished in two ways, using either a sample line for each instrument and an instrument for each space to be analyzed, or a number of sample lines feeding to a selector valve and manifold from which the sample is fed to a single instrument. In order to select the proper approach, we must consider the required frequency of analyses. Most analyses can be on an intermittent basis from a continuously flowing sample stream and the instrumentation is recommended on this basis. The analyses of the various gases that are under consideration here serve more for quality assurance than for control.

INSTRUMENTATION STATE-OF-THE-ART

The instruments for analysis of the gases described above may be divided into two general classes, multicomponent and single component. The class of multicomponent instruments consists of gas chromatographs and mass spectrometers. The second class contains one or more types of instruments for

each component. These individual instruments will be discussed and the bases on which they were selected for examination will be given.

MULTICOMPONENT INSTRUMENTS

Gas Chromatograph

Basically, a gas chromatograph consists of a volumetric sampling system, a carrier gas supply, the chromatographic column, and a detector. In the chromatograph, the components of the gas mixture are separated from each other by virtue of the different value of free energy of absorption of each gas on an adsorbent. A given gas is adsorbed and desorbed from the carrier gas stream many times as it passes through the column. A strongly adsorbed substance goes through this cycle more slowly than a weakly held one and thus emerges from the column at a later time. An adequate column will separate the original mixture of gases into a series of binary mixtures of carrier gas and individual component. The detector senses the difference in properties of the gas mixture from the pure carrier with the magnitude of the difference being proportional to the concentration of component in the mixture. By proper selection of the sample size, column packing, and detector, it is possible to measure the required impurity concentrations to less than 1 ppm in helium. As will be further discussed below, argon presents a less favorable case and sensitivities are restricted to 5 to 10 ppm for impurities in argon. Further detector improvement may increase this sensitivity.

The column packings found most useful for separation of the fixed gases are molecular sieves (either type 5A or 13X). These packings readily separate hydrogen, oxygen, nitrogen, methane, and carbon monoxide. The molecular sieves have a strong affinity for water and carbon dioxide, retaining them

until the column is saturated or is heated to an elevated temperature (greater than 125 °C) so these substances are not measured. Eventually they saturate the column requiring its regeneration or discard. If the measurement is necessary, carbon dioxide can be isolated with a silica gel column and both water and carbon dioxide can be isolated with a Poropak⁽⁵⁾ column.

Several detectors have been used for the impurities of interest in cover gas monitoring. The most sensitive is the micro ion cross section detector using ultra pure helium as a carrier. Development of this detector has been described by Shannon.⁽⁶⁾ An instrument operating on this principle has been used for several years to measure the quality of helium in a high temperature loop. With this instrument it is possible to detect less than 0.2 ppm of oxygen, nitrogen, methane, and carbon monoxide. It is sensitive to 1 ppm of water, hydrogen and carbon dioxide. This detector has not been used routinely for water and carbon dioxide. If helium is used for the sodium cover gas, this ion cross section detector will be a strong contender. However, since argon is to be used, the instrument does not appear nearly as attractive since its sensitivity falls off by a factor of from 10 to 20. The argon detector is less sensitive because argon's ionization potential is lower than that of many of the impurity gases. This leads to a high background from the argon ions generated and to a small number of ions from the impurity molecules. With helium the opposite condition prevails, because of the higher ionization potential for helium than for any other fixed gas. A second sensitive detector is the flame ionization detector. It is insensitive to all of the above impurity gases except methane. With it, 0.5 ppm of methane in argon can be detected. A third type of detector is the thermal conductivity detector. It is sensitive to all the fixed gases. Its

operation depends upon the difference in thermal conductivity of the pure carrier and the gas mixture. Helium and hydrogen have much higher thermal conductivities than other gases so there is a greater difference between the thermal conductivity of the carrier and that of the mixture when using helium carrier. It appears that the thermal conductivity detector and the ionization detector in argon are of approximately equal sensitivity or more properly the signal-to-noise ratio (which determines the sensitivity limit of these detectors) is approximately the same. (A limiting sensitivity of 5 to 10 ppm was found for methane in argon.) Gas chromatographic detectors are continually being improved and more suitable ones may be available by the time equipment for FFTF is purchased.

Mass Spectrometers

A mass spectrometer is a device that separates ions of different masses (properly different mass to charge ratio, M/e) and then detects the relative number of ions of each mass. There are a number of different methods of separating the ions as well as several methods of generating the ions from the initial sample. The usual method of generating the ions is by bombarding the sample with an electron beam. This generates positive ions by knocking electrons off the molecule. For use on sodium cover gas analyses this is the preferred method of ion generation since it is the simplest and has had the most development.

The most common commercially available types of mass spectrometers are: the magnetic sector, time-of-flight, and RF-DC mass filter. The magnetic sector instrument separates a beam of ions by deflecting the flight path of the ions with a magnetic field. Ions of different mass are brought to the detector (usually an electron multiplier) by varying the magnetic field or the accelerating voltage that imparts the

initial energy to the ions. The time-of-flight instrument operates by virtue of the difference in velocities of ions of different masses when accelerated to the same energy. The RF-DC mass filter instruments are made in monopole and quadrupole configuration. In a combined DC-RF field only a single ion mass will follow a trajectory that will carry it from the ion source to the detector. By varying the DC and RF fields the mass range of interest can be swept.

The magnetic instrument requires the use of a system of slits to define the ion beam that is sent through the magnetic field. Neither of the other systems require the alignment slits which somewhat simplifies their ion source construction. Models of each type instrument are available that will operate at somewhat elevated temperatures (400 to 500 °F). Whether or not these temperatures are adequate for FFTF cover gas analysis is under investigation. Models of each type mass spectrometer are available with concentration sensitivities of 10^{-6} , i.e., 1 ppm for the favorable cases. Whether or not this will hold for the mixtures of concern in the sodium cover gas has not been established.

Selection of the optimum mass spectrometer will require experimental evaluation. Workers at LASL are investigating the application of a quadrupole type instrument to analyze sodium cover gas.⁽⁷⁾ To avoid duplication of effort and because of the simplicity of construction of the time-of-flight instrument, it was deemed desirable to examine a small time-of-flight instrument at BNW. By the time it is necessary to specify the instruments for FFTF, additional experimental data will be available for both mass spectrometers and gas chromatographs.

INSTRUMENTS FOR INDIVIDUAL COMPONENTS OF THE BLANKET GASESOxygen

Instruments for the determination of oxygen may be divided into two groups. First, those which depend upon the paramagnetic properties of oxygen and, second, those which utilize an electrochemical cell in which oxygen is involved. Those instruments which utilize the paramagnetic properties of oxygen depend either on a magnetic field causing the oxygen to move or on the paramagnetic properties of the oxygen concentrating the magnetic field to induce a mechanical force on a sensing body. Those instruments that utilize the movement of the gas in the magnetic field use a thermal conductivity cell to sense the change in properties of the gas mixture that has been brought about by the action of the magnetic field moving the oxygen.

There are at least four instruments available commercially that utilize the paramagnetic susceptibility of oxygen. The first of these is the Beckman analyzer. This utilizes the movement of a vane in a magnetic field as the oxygen content of the gas causes this magnetic field to be concentrated or diminished. The deflection of the vane is offset electrostatically to produce a null type instrument and the current required to return the vane to the null point is a measure of the oxygen concentration. This instrument has linear response with oxygen concentration. It can be designed to operate over a variety of ranges from as low as 1000 ppm (full scale) to 0-100% oxygen. Since it depends upon mechanical movement of a vane it is subject to noise from vibration. Although it is designed to resist this effect the possibility of being subject to vibration interference still exists.

Three commercial instruments utilize the paramagnetic properties of oxygen to cause a gas flow around a filament in a thermal conductivity cell. This can be calibrated in terms of oxygen concentration.

In the cells that utilize the chemical properties of oxygen, there are two types--first, those utilizing an aqueous system; second, those utilizing a solid electrolyte.

Those utilizing an aqueous electrolyte are based on the familiar Hersch cell. The Hersch cell consists of a silver cathode, an active metal anode such as lead or cadmium, and an alkaline electrolyte such as potassium hydroxide. The oxygen is converted to hydroxyl ions at the surface of the silver with the consumption of electrons. The electrons are supplied from the active metal anode where the metal is converted into its oxide or hydroxide. The output of the cell (current) is proportional to the quantity of oxygen in the gas that is flowing through the system. The mechanical arrangement of the cell, the cell electrode design and the choice of anode are all variable, and several cell designs are therefore possible. Several instruments are available commercially based on slight cell modifications. In general these instruments are most useful for measuring trace concentration of oxygen in the 10 to 100 ppm range rather than at the approximately 1% level that is most likely to be in the cell atmosphere gas. By using a membrane such as teflon or polyethylene to separate the gas sample from direct contact with the aqueous solution, cells of lower sensitivity are produced. These are applicable over a 0 to 25% range.

The second electrochemical cell approach is based on the use of a solid electrolyte (calcia stabilized zirconia) to transfer oxygen ions from gas of one concentration to gas of another concentration. In so doing an electrode potential is

set up that is proportional to the difference in oxygen pressure. The fact that the potential is dependent upon the partial pressure of oxygen makes it possible to use air with its known and constant oxygen content as the reference gas. The sample stream to be measured is then passed on the other side of the electrochemical barrier. These cells are applicable over an extremely wide range of oxygen concentrations in the sample gas from parts per billion to pure oxygen. However, these cells have a disadvantage in that the gas must be heated to approximately 800 °C. If the gas contains a material that can be oxidized at this temperature, the indicated oxygen content will be reduced by the amount of oxygen consumed by the oxidation of this foreign substance. For example, oil vapors, hydrogen, or similar materials will reduce the indicated oxygen concentration.

So far the only oxygen measuring instruments for the cell blanket gas applications that have been evaluated at BNW are the ones of the solid electrolyte type. A paramagnetic type instrument has been ordered for installation on one of the BNW test loop enclosures and will be evaluated as it is used in this application. Several of the instruments utilizing the aqueous electrochemical cell have been tested here for use in the monitoring of sodium cover gas. (See comments in Appendix, page A-6)

Water

The measurement of water in gases has been the subject of many investigations because of its large influence on properties of many materials. Much of the work has been done in the field of meteorology because of the prime importance of the water vapor content of air to weather prediction.

A wide variety of instruments have been developed ranging from automated laboratory type instruments such as the

psychrometer and the dew point devices to instruments which depend upon changes in properties of substances such as hair. The meteorologists have also developed a sensor based on lithium chloride impregnated in a porous media which changes resistance with the amount of moisture absorbed. Of the various instruments mentioned here, only those based on measurement of dew point are applicable to the low moisture levels we expect in the cell blanket atmosphere. One promising type of hygrometer is based on the use of an alumina film to separate an aluminum metal base from an evaporated gold layer to form a leaky capacitor. Its capacitance and resistance both vary with the moisture content of the alumina film. This sensor can be made into a probe type instrument which does not require gas flow. With the proper electronics, the measurement of the change in capacitance and resistance of this sensor as a function of moisture content is possible over a wide range. The commercially available version senses moisture levels equivalent to dew points from -110°C to 10°C , or from approximately 0.01 ppm to over 10,000 ppm of moisture in the gas. These sensors can be calibrated to about $1\frac{1}{2}$ to 2°C dew point. They have not been on the market for a long enough period to be certain of the life of the sensing elements. The manufacture guarantees them for six months and our experience in laboratory testing has indicated that they will normally have a life of at least six months. Some of ours have been in service for more than a year.

Another instrument for the measurement of water vapor is based on the use of a piezo electric crystal coated with a hygroscopic substance. As the water vapor content of the gas surrounding the crystal changes, the amount absorbed on the hygroscopic surface changes. The resulting change in mass shifts the frequency of oscillation of the crystal. This frequency shift can be measured readily. Because of the time

lag involved in reaching equilibrium in the hygroscopic coating on the crystal, it is necessary to use a reference flow of dry gas and to reverse the gas flow so that alternately the sorption-desorption of moisture on the detector is measured.

Another widely used type of moisture monitor depends on the sorption of moisture from the gas streams into a P_2O_5 coating that has been deposited between platinum electrodes. An electric current is continually passed through the resulting phosphoric acid to electrolyze the absorbed water. Measurement of the required current gives a measure of the amount of moisture that it absorbed from the gas stream. With a continuous flow of gas at a known rate it is possible to calculate the moisture content of the gas. There are several commercially available versions of this instrument differing in cell geometry and in the electronic circuitry that is used to supply and measure the current. These instruments are most generally used to monitor gases containing less than 1000 ppm water.

The water content of gases can also be measured by infrared spectrophotometers. Instruments based on this principle are capable of measuring moisture from as low as $-100^{\circ}F$ dew point or about 1 ppm of water up to saturation.

Any system that is used to measure water content of the gas must contend with the problem of sorption of moisture on surfaces. If the gas flows through a sample line, there is a lag in the adsorption or desorption of moisture from the surface of the sample line. The following example will illustrate this effect. A 25 ft length of 1/4 in. stainless steel tubing was dried with a gas of approximately 2 ppm moisture until equilibrium had been established. The gas flow was approximately $200\text{ cm}^3/\text{min}$. When the moisture content of the gas was increased to 600 ppm, a time delay of approximately eight minutes was observed between switching the gas flow and

detection of the increased moisture content at the sensing instrument. (The gas flow delay is about 40 sec in this length of tubing.) Therefore, if a rapid response is desired, it is necessary to go to a very high flow rate or to use a probe type sensor. On their Dragon reactor the British⁽⁸⁾ tested both a probe type instrument and a pressurized electrolytic type in which the cell was enclosed in the flowing gas stream and the sample was allowed to exit from this gas stream directly through the sensing cell. They reported the pressurized electrolytic cell type instrument performed satisfactorily with a reasonably rapid response going from less than 2 ppm to 100 ppm in 4 min with initial response in 30 sec. The reverse change was slower going from 100 ppm to less than 5 ppm reading in about 15 min. The probe type instrument they tested would not maintain its calibration. The sensor was of a different design than the Panametrics unit manufactured in this country.

Hydrogen or Combustibles

A significant source of hydrogen exists in an equipment cell atmosphere arising from the reaction of sodium with water or water vapor. The possible occurrence of explosive mixtures exists if sufficient hydrogen is accumulated and later mixed with air. It may therefore be necessary to provide instrumentation to detect the presence of high concentrations of hydrogen or combustibles in the cell atmosphere. The simplest way to detect hydrogen in either nitrogen or argon is to measure its effect on the thermal conductivity of the mixture. Hydrogen has a much higher thermal conductivity than either of the other gases and it is therefore possible to detect very small concentrations of hydrogen. However, since the desire here is to protect against explosive mixtures it should not be necessary to measure very small quantities of hydrogen but only

concentrations approaching the explosive limit which is 4% in air, or approximately 7% when mixed with 5% oxygen (the minimum flammable mixture).⁽⁹⁾

It is possible to detect combustible substances in a gas mixture by passing the gas over a heated filament, usually platinum or other materials which have a catalytic effect, and measuring a temperature increase resulting from the combustion. If a sample of the inert gas blanket is diluted with air or oxygen and passed over such a filament, the presence of significant quantities of combustible materials can be detected.

Nitrogen

In the cells where argon is to be used as the inert blanket, it may be desirable to detect nitrogen if work now in progress demonstrates that high concentrations of nitrogen will lead to the nitriding of the stainless steel or other structural materials to an undesirable extent. There is one commercially utilized method that is quite sensitive for the detection of nitrogen in such gases. It is based on the emission of ultraviolet light from nitrogen when excited in an electric field. By proper selection of detector and filters, it is possible to measure nitrogen concentrations over a rather wide range (1 ppm to low percentages).

Nitrogen Oxides

The possibility exists that if a mixture of nitrogen and oxygen are present in some of the areas (such as the reactor cavity where they are subject to a radiation field) nitrogen oxides will be generated. The primary oxide will be the dioxide. This is of concern if materials of construction are used that are sensitive to nitric acid, since the combination of nitrogen dioxide with water produce nitric acid. Nitrogen dioxide is one of the noxious impurities in polluted air so there has been considerable effort reported⁽¹⁰⁾ to develop

methods of measuring nitrogen oxides. Generally the pollution chemist is interested in measuring only trace quantities below the ppm level and most of the instruments that have been developed to date are of the automated chemical reaction type. A survey of literature indicates that at least one system⁽¹¹⁾ has been developed that utilizes the ultraviolet adsorption of light by nitrogen dioxide to measure its concentration in air. It would appear possible to develop an instrument for use in FFTF based on this principle if it is deemed desirable and necessary to determine the concentration of nitrogen dioxide in the blanket gases in the cell, where high radiation fields exist. However, until a need is demonstrated, it would appear satisfactory to rely on the chemical method (either automated or laboratory) since it is easy to set up for spot checks of the concentration of nitrogen oxide.

Carbon Monoxide and Carbon Dioxide

It is possible to measure carbon monoxide and carbon dioxide in gas mixtures by infrared spectrophotometry. Commercial instruments, known as nondispersive infrared analyzers, are available which operate on a differential absorption principle and do not require filters, prisms, or gratings to isolate a specific infrared energy band characteristic of the substance being measured. Rather, the detector is sensitized to the substance to be measured and responds to changes in concentration of this substance in the sample gas.

Carbon dioxide has many strong absorption bands in the infrared and can be measured down to a few parts per million. Standard ranges available on commercial instrumentation are 0 to 100 and 0 to 1000 ppm with lower ones available. Carbon monoxide is not as strongly absorbing. The minimum range instrument commercially available is 0 to 100 ppm.

SELECTION CRITERIA

The following are the important criteria in selecting the instruments to perform the analyses required in FFTF gas systems.

- a. Range: The range of the instrument should be such that the expected readings or alarm limit is a large fraction of full scale.
- b. Accuracy: This should be such that deviation of the indicated reading from the true value by any instrument inaccuracy will not introduce a safety hazard or a serious error in interpreting test results.
- c. Reliability: This should be such that the instrument is available for a large fraction of the operating time.
- d. Selectivity: The degree of selectivity or specificity required is dependent on the individual application.
- e. Sample Requirements: Flow rate, temperature, or pressure may be important in selecting between competing instruments. It will be desirable to withdraw little gas from the sodium cover spaces to minimize adding contaminants via the cover gas. The need for calibration and zero gases (pure standards) may also be considered under this heading.
- f. Engineering Parameters: Such items as space required by the instrument, its mounting and sample system, location, need for shielding, cost, and maintenance are items that may determine the selection in lieu of technical restrictions.
- g. Sample Multiplexing: A criteria that may become important is the capability to accept multiple samples. Since there are many sampling points to be monitored economy will dictate feeding as many samples to a single instrument as possible. If two instruments are otherwise equally

applicable, the one with the shorter response time (time to equilibrate) would be preferable.

- h. Data Processing: Since it is anticipated that much of the data from the various instruments will be processed by computer, another criteria for selection may be the suitability of the instrument for such coupling.
- i. Operator Attention: If the instrument requires frequent standardization or adjustment of operating controls or interpretation of the results it may prove too demanding for economical application. (Information for this criteria will be accumulated from test loop and prototype system operation and consequently will not be discussed below.)

CELL BLANKET MONITORS

Oxygen Analyzers

- a. Range: The primary requirement for oxygen control in the cell blanket gas is to maintain the oxygen concentration below the rapid combustion level of approximately 5%. An instrument range of 0 to 5 or 6% will therefore be adequate for the application. If an additional and more restrictive criterion of control of oxygen concentration for inhibition of nitriding is established then it may be desirable to provide an additionally more sensitive range such as 0 to 1%. An additional range of 0 to 25% for cells where people may enter is also needed.
- b. Accuracy: Precise determination of oxygen concentration is not necessary on cell gas applications, and an accuracy of 5% will be adequate.
- c. Reliability: Since this is a safety device, the reliability should be high enough to preclude the development of a hazard because of an instrument malfunction. Most of the instruments suitable for this use require a

calibration gas for final adjustment of the sensing circuits. This provides a check on the instrument performance.

- d. Specificity: The instrument reading should be unaffected by the presence of other substances in the gas stream. For example, the solid electrolyte analysis is not satisfactory. This is because the mixture of hydrogen and oxygen would indicate no oxygen when heated in the instrument, but the mixture would still be a source of oxygen to the sodium.
- e. Sample Requirements:
 - (1) Flow: The flow rate of gas from this system should be of small concern in ranges required for most commercial oxygen meters (0.1 to 2.5 scfh).
 - (2) Temperature: It is expected that the gases in the cells will generally be above ambient temperature (100 to 125 °F). Temperatures are not expected to be high enough to offer any instrument problems.
 - (3) Pressure: The pressure drop through the instruments will be a major item of concern. Pressure head available from the gas circulator in the cell will be small. If the instrument has an appreciable pressure drop through it, then it will be necessary to provide a pump to produce the required gas flow. Probe type sensors which do not require a gas flow would be desirable.
 - (4) Calibration and Zero Gases: Most of the instruments applicable to this range of analyses have adjustable parameters in their circuits to compensate for operating variables. It is necessary to provide both calibration and zero gases to permit proper instrument calibration and adjustment.

- (5) Sensor Location: Since the cells will contain radioactive materials, the sensors and electronic components can be subjected to radiation fields that may be intense enough to influence their operation. Therefore it will be advantageous to select an instrument with a sensor which is insensitive to moderate radiation fields. Maintenance of the electronic circuitry will be easier if a remote sensor is used permitting the location of the electronic portion of the instrument in an uncontaminated zone. The gases themselves are not expected to contain enough radioactive substances to affect the instruments.

For the oxygen analyzers it is anticipated that specificity and sample requirements will be the most important criteria in making the final instrument selection.

Water or Moisture Analyzers

- a. Range: It will be desirable to have some water present in the cell blanket gases rather than use the dry nitrogen that is obtained by evaporation of liquid nitrogen. An instrument capable of indicating moisture levels above 40 ppm (-57 °F frost point) will therefore be required. The incoming nitrogen will be extremely dry (less than 1 ppm) and actual addition of water may be required. A suitable moisture monitor range is -60 to 0 °F frost point (30 to 1300 ppm).
- b. Accuracy: Assuming the primary purpose of the measurement is the maintenance of sufficient moisture in the gas for proper operation of electric motor brushes, an accuracy of 5 °F around -50 °F should be adequate. If other criteria are used for setting moisture levels they may also dictate the accuracy with which the moisture levels must be known.

- c. Reliability: Reliability requirements are difficult to define for this application.
- d. Selectivity: The dew point/frost point instruments that measure the temperature at which condensation occurs on a mirror respond to any condensible substance that may be present in the gas. It is not expected that there will be anything but water in the cell blanket gases that would condense and re-evaporate as would water on the automatic dew point instrument.
- e. Sample Requirements:
 - (1) Flow Rate: The electrolytic instruments require a fixed flow rate of $100 \text{ cm}^3/\text{min}$ while the other instruments vary in their requirements. The requirements of each are listed in the Appendix. Most require so little flow that this will not be a controlling factor in selection.
 - (2) Temperature: The samples are expected to be at ambient temperature when they reach the instrument sensor so temperature capability should have little bearing on instrument selection.
 - (3) Pressure: As with the oxygen meters only sufficient pressure to overcome internal resistance is needed, however, this may require as much as 20 psi for some instruments. There is one probe type sensor available that does not require gas flow.
 - (4) Calibration and Zero Gases: This class of instruments are precalibrated and do not require frequent adjustment so calibration and zero gases are not needed.
 - (5) Sensor Location: Most of these instruments are available with the sensor separable from the electronic components. In all cases they can be used to measure a sample brought to them. However, water is readily adsorbed on most sample line surfaces.

Significant delay in instrument response can be anticipated as a result of sample line adsorption and desorption. Thus a probe in direct contact with the gas at the point of interest is the best tool for measuring moisture level changes.

Hydrogen or Combustibles Analyzers

- a. Range: The lower flammability limit for hydrogen in air is 4 vol%. The lower flammability limit for hydrogen in nitrogen which is later mixed with air (the cell gas case) is 7%. Thus if the hydrogen is held below 7% in the cell gas, safety is ensured. An alarm at 4% would provide a safety factor even if the cell atmosphere were air. A recommended range for the cell gas monitor is therefore 0 to 5%.
- b. Accuracy: Two percent of full scale would be adequate.
- c. Reliability: As with the oxygen analyzer the hydrogen monitor is safety instrumentation. Generation of sufficient hydrogen to approach the flammability limit by a sodium water reaction appears a remote possibility that would be accompanied by other indications of trouble. The measurement of hydrogen therefore would not be the only safeguard and the degree of reliability will not have to be as high as for some of the other instruments.
- d. Selectivity: It may not be necessary to have a specific measure of hydrogen concentration but rather of total combustibles. At least two considerations point to hydrogen as the prime candidate for measurement. First, a ready source is present since it can be generated by the reaction of sodium with water. Any other common combustible gas would have to be piped in. Second, in a mixture of inert and fuel gases to which oxygen is being added the hydrogen-oxygen reaction will become self

propagating at much lower oxygen concentrations than with other combustibles such as methane or other hydrocarbons.

e. Sample Requirements:

- (1) Flow: The flow rate of gas from this system should be of little concern in the ranges required for most commercial analyzers (0.1 to 2.5 scfh).
- (2) Temperature: Ambient temperatures should be adequate. If a sample is provided through a pipe line it will probably be at ambient temperature.
- (3) Pressure: Pressure drops as low as a few inches of water are common in instruments adequate for this measurement. If the instrument selected requires a drop greater than this it will be necessary to provide a sampling pump.
- (4) Calibration and Zero Gases: These are required for all of the commercially available instruments for measuring hydrogen.
- (5) Sensor Location: It will be advantageous to consider instruments permitting remote location of the sensor so the electronics may be outside the high radiation fields which may be present in some of the cells.

For the hydrogen or combustibles analyzers, selectivity and sample requirements are the most important criteria in making the instrument selection.

Nitrogen Oxides

Selection criteria for instrumentation to measure nitrogen oxides in cover gas systems cannot be defined at this time since the need for nitrogen oxide measurements has not yet been established.

COMPARISON BETWEEN SELECTION CRITERIA AND AVAILABLE INSTRUMENTATION

The commercially available instruments considered for measuring the impurities in the cell blanket gases are listed in Table 3. The instruments for a particular analysis such as oxygen are grouped together and their capability to satisfy the more important selection criteria is indicated. In addition, notes on particular features are appended. The list price and cabinet size is also tabulated. More detailed data concerning each commercially available instrument are given in the Appendix.

If the listed instrument is judged to be capable of satisfying the selection criteria a plus sign is shown in the criteria column; if it cannot, a minus sign is shown and any limitations are discussed in a footnote. Under the heading "Sample Requirements" a minus sign is given if any of the following situations exist: the instrument 1) requires what is judged to be excessive flow rate of sample, 2) is too limited in the temperature range in which it will operate, or 3) requires a higher sample pressure than is available from the system (or will be readily available from a sample pump).

In the cases of oxygen and moisture there are several commercially available instruments listed in Table 3. The ones which appear to meet FFTF selection criteria best are:

Oxygen Analyzers - Beckman Instruments (Paramagnetic) primarily because of its specificity for oxygen. The sensors of the Teledyne Analytical Instruments (membrane Hersch cell) are probe type and thus would require no sample gas flow. If they require only infrequent calibration they may be very attractive. The only known technical objection to any of the listed oxygen analyzers is the interference by fuel gases in the solid electrolyte type instruments.

TABLE 3. *Comparison of Commercially Available Instruments with the Selection Criteria for Measurement of Impurities in Cell Blanket Gases*

Manufacturer and Type	Range	Accuracy	Sensitivity	Sample Requirements			Supporting Gas Required?		Can Sensor Be Remote?	Cost, \$	Size, in.			
				Flow	Upper Temp.	Pressure	Calibration	Zero			W	H	D	
Oxygen Analyzers														
Beckman Instruments (Paramagnetic, Electromechanical)	+	+	+	+	+	+	Yes	Yes	No	1,750	15	18	12.5	
Leeds & Northrup (Paramagnetic, Thermal Conductivity)	+	+	+	+	+	+	No	No	Yes	1,350	11.5	9.5	18.5	
Mine Safety Appliances (Magnetic Wind Cooling)	+	+	+	+	+	+	Yes	Yes	Yes	1,350	19	12	13	
Hays Corp. (Magnetic Wind Cooling)	+	+	+	+	+	+	No?	No?	Yes	1,630	16	10	9	
Hays Corp. (Membrane, Hersch Cell)	+	+	+	+	+	+	Yes	Yes	No	464	10.5	13.5	7	
Teledyne Analytical Instruments, Inc. (Membrane, Hersch Cell)	+	+	+	+	+	+	Yes	No	Yes	1,200	10.5	9	5	
Westinghouse (Solid Electrolyte)	+	Note a	+	+	+	+	Yes air	No	No?	1,950	17	8.5	9.5	
Thermo Labs (Solid Electrolyte)	+	Note a	+	+	+	+	Yes air	No	Yes	980	8 3	6 4	6 8	Control Sensor
Moisture Analyzers														
Cambridge Systems (Automated Dew Point)	+	+	+	+	+	+	No	No	No	2,740	19	10	12	
Panametrics (Alumina Film Hygrometer)	+	+	+	+	+	+	No	No	Yes	1,195	12.5	10	6.5	
Beckman Instrument Corp. (Electrolytic-P ₂ O ₅)	Note b	+	+	+	+	+	No	No	Yes	1,285	11	13	8	
Consolidated Electro-dynamics Corp. (Electrolytic-P ₂ O ₅)	Note b	+	+	+	+	+	No	No	Yes	555	11	6	8	
Kahn & Co. (Electrolytic-P ₂ O ₅)	Note b	+	+	+	+	+	No	No	?	495	12	6.5	7	

TABLE 3. (contd)

Manufacturer and Type	Range	Accuracy	Sensitivity	Sample Requirements			Supporting Gas Required?		Can Sensor Be Remote?	Cost, \$	Size, in.				
				Flow	Upper Temp.	Pressure	Calibration	Zero			W	H	D		
<u>Moisture Analyzers (contd)</u>															
Manufacturers Engineering and Equipment Co. (Electrolytic-P ₂ O ₅)	Note b	+	+	+	+	+	+	No	No	?	?				
E. I. duPont de Nemours (Quartz Oscillator)	+	+	+	(-) Note c	+	+	+	Yes	Yes	Yes	3,850	17	5	13	
Mine Safety Appliances (Nondispersive Infrared)	+	+	+	+	+	+	+	Yes	Yes	Yes	3,600	12	13	19 to 68	
Beckman Instruments (Nondispersive Infrared)	+	+	+	+	+	+	+	Yes	Yes	Yes	3,500	6 6.5	6 8.5	18 29 to 61	Amplifier Analyzer
<u>Nitrogen</u>															
Mine Safety Appliances (Spectral)	+	+	+	+	+	+	+	Yes	Yes	No	6,500	28	84	25	
<u>Nitrogen Oxides</u>															
Technicon Controls (Automated Chemical Analysis)	Note d	+	+	+	+	+	+	No	No	No	5,000				
<u>Hydrogen</u>															
Mine Safety Appliances (Thermal Conductivity)	+	+	+	+	+	+	+	Yes	Yes	No	700	11	8	8	
<u>Combustibles</u>															
Mine Safety Appliances (Temp. Rise)	+	+	+	+	+	+	+	Yes	Yes	Yes	1,100	19	9	20	
<u>Hydrocarbons</u>															
Teledyne Analytical Instruments (Hydrogen Flame Ionization)	+	+	+	+	+	+	+	Yes	Yes	No	2,500	16	17	9	

Notes:

- The solid electrolyte oxygen meters measure the oxygen in excess of that which will combine with a fuel material such as hydrogen or hydrocarbons, so the accuracy of the oxygen measurement is dependent on the absence of such materials from the gas stream.
- The upper limit of 1000 ppm (-5 °F dew point) for the electrolytic moisture monitors may be insufficient for use on the Cell Blanket gases if the decision is made to add moisture to them.
- The sample flow rate of 1500 cm³/min is much higher than required by any other moisture analyzer but for the Cell Blanket application this may not be an excessive amount.
- This instrument is designed for air pollution studies. Whether or not the Cell Blanket gas, nitrogen oxide concentration will fall within the range of this instrument is not known.

Moisture Analyzers - Panametrics (alumina film hygrometer) primarily because it is a probe type sensor and thus the problem of sampling system buffering of changes in the moisture content of the gas is eliminated.

The other analyses (N_2 , H_2 , NO_x , CH_x) may be covered by the single instrument listed for each. The major advantage listed for the recommended oxygen and water analyzers does not eliminate final selection of other instruments if other criteria are weighted more heavily in favor of the alternative instrument. For the cell blanket applications most of the listed instruments would provide the required analyses.

SELECTION CRITERIA FOR SODIUM COVER GAS MONITORS

The expected components of the sodium cover gas are listed in Table 1. Impurity concentrations will normally be low, hopefully near the instrument detection level. Only during periods when the cover gas is being contaminated, will appreciable quantities of impurities be detected by the measuring instruments.

The following are the suggested criteria for selection of the instrumentation to analyze the sodium cover gas for impurities.

- a. Sensitivity and Range: Indications are that all impurities except nitrogen will be present in the sodium cover gas in very low concentrations. An important criterion for instrument selection then will be high sensitivity. Nitrogen may be present to nearly 1% so a wide range will be needed for it.
- b. Accuracy: The minimum accuracy at high sensitivity should be 10% of full scale with 5% accuracy at higher concentrations.

- c. Selectivity: A high degree of selectivity or specificity is needed to isolate the individual components of the mixture particularly if unknown substances occur that need to be identified.
- d. Sample Requirements: The monitoring instrument should require a reasonably small sample flow. Ideally it should be able to accept the sample, with its burden of sodium vapor at the maximum operating temperature of the cover gas spaces (up to 1400 °F).
- e. Reliability: The instruments should operate with a minimum of down time to provide the continuity of measurement desired to protect against the consequences of introduction of impurities to the sodium from the cover gas.
- f. Multiplexing: Because of the large number of sample points indicated for the system there will be a need for multiple instruments. Therefore, another criterion is the adaptability of the instrument or modules of it to accept multiple samples.
- g. Data Display and Computer Interfacing: The output signal from the instruments should be compatible with the computer data handling system.

COMPARISON BETWEEN SELECTION CRITERIA AND COMMERCIALLY AVAILABLE INSTRUMENTS

The commercially available instruments considered for measuring the impurities in the sodium cover gas are listed in Table 4. There are some major unresolved problems so a recommendation is not being made at this time.

- a. Sensitivity and Range: As pointed out in the State-of-the-Art discussion earlier, the gas chromatograph, when applied to an argon cover gas, may have limiting sensitivity of 5 to 10 ppm. This is not as good as is desirable but may be improved by development of the proper

TABLE 4. Comparison of Commercially Available Instruments with Selection Criteria for Measuring Impurities in Sodium Cover Gas

Manufacturer and Type	Range	Accuracy	Sensitivity	Sample Requirements			Supporting Gas Required?		Can Sensor Be Remote?	Cost, \$	Size, in.			
				Flow	Upper	Pressure	Calibra- tion	Zero			W	H	D	
					Temp.									
<u>Gas Chromatograph</u>														
Bendix Corp. Greenbriar Div. (Ionization Detector)	+	+	Note a	+	Note b	+	Yes	No	No	12,000	27 16 14	42 22 12	18 27 16	Analyzer Controller Recorder
Thermo Instrument Corp. (Thermal Conductivity Detector)	+	+	Note c	+	Note b	+	Yes	No	No	7,250	38	78	18	
<u>Mass Spectrometer</u>														
Bendix Corp. (Time-of-Flight)	+	+	Note d	+	Note e	+	Yes	No	No	16,000	20 27 18	26 51 9	18 27 7.5	Electronic Cabinet Vacuum Cabinet Recorder
Aero Vac Corp. (Magnetic Sector)	+	+	Note d	+	Note e	+	Yes	No	No	18,500	48	60	24	
Electronic Associates, Inc. RF-DC (Quadrupole)	+	+	Note d	+	Note e	+	Yes	No	No	23,500	22	2 racks, 72	each 20	
General Electric Co. RF-DC (Monopole)	+	+	Note d	+	Note e	+	Yes	No	No	24,500	22 22	84 72	24 28	Controller Analyzer
<u>Oxygen Analyzers</u>														
Beckman Instruments (Hersch Cell)	+	+	+	+	Note f	+	No	No	No	2,280	16	18.5	11	
Engelhard Industries (Hersch Cell)	+	+	+	+	Note f	+	No	No	No	2,100	19	20	9.5	
Lockwood & McLorie, Inc. (Hersch Cell)	+	+	+	+	Note f	+	No	No	No?	1,200	13	15	8.5	
Teledyne Analytical Instruments (Hersch Cell)	+	+	+	+	Note f	+	No	No	No	2,045	16	19	10	

TABLE 4. (contd)

Manufacturer and Type	Range	Accuracy	Sensitivity	Sample Requirements			Supporting Gas Required?		Can Sensor Be Remote?	Cost, \$	Size, in.			
				Flow	Upper Temp.	Pressure	Calibration	Zero			W	H	D	
Carbon Monoxide, Carbon Dioxide, Methane Analyzers - Note g														
Mine Safety Appliances (Nondispersive Infrared)	+	+	+	+	Note h	+	Yes	Yes	Yes	3,600	12	13	19 to 68	
Beckman Instruments (Nondispersive Infrared)	+	+	+	+	Note h	+	Yes	Yes	Yes	3,500	6 6.5	6 8.5	18 29 to 61	Amplifier Analyzer

Notes:

- If helium is used as the cover gas a sensitivity of less than 1 ppm can be reached, but if argon is the cover gas this limit cannot be reached with present detectors.
- The upper temperature limit for operation of the gas chromatograph has not been determined. As discussed in the text, this may be one of the deciding factors in selecting the usable instrument.
- This instrument uses thermal conductivity detectors which appear to be unable to detect concentrations as low as 1 ppm.
- It has not been established that any type mass spectrometer will have sufficient sensitivity for the analyses to be made on the sodium cover gas. Some appear capable of measuring less than 10 ppm.
- All the listed mass spectrometers can operate at elevated temperatures but, as discussed in the text, one of the unresolved problems is the effect of temperature changes on the composition of the gas due to chemical reaction with sodium or to gettering by condensing sodium.
- These instruments operate with an aqueous cell so they are limited to sample gas temperatures only slightly above ambient.
- These substances cannot be analyzed by a single instrument of the type listed but can be analyzed in individual instruments sensitized for the specific substance of interest.
- The general problem of elevated temperature analysis of sodium cover gas has been discussed in the text. These instruments operate at near ambient temperatures.

detector. (This is an active line of investigation and more sensitive detectors may be available commercially by the time selection for FFTF is made.) If helium were the cover gas, then the instrument would have a sensitivity of less than 1 ppm of the impurities listed in Table 1.

The manufacturers of mass spectrometers indicate a sensitivity ratio of maximum to minimum pressure for a gas of about a million. This would correspond then to 1 ppm of one gas in another. Whether this will be realizable with the expected cover gas impurities is under investigation. Both gas chromatographs and mass spectrometers have sufficient dynamic range to measure from the part per million level to the low percent level. The other analyzers listed in Table 4 are available with ranges down to 0 to 5 ppm for oxygen and normally 0 to 100 ppm for the other impurities (CO, CO₂, H₂O). They are available as dual range instruments to increase the range by a factor of 5 or 10 which should be adequate for any application they may be used for on this system.

- b. Accuracy: At the limiting sensitivity, the accuracy of the gas chromatographs examined to date has been well within 10% of the full scale range. The accuracy of the mass spectrometer is still under investigation. The other instruments listed that have been examined to date meet this criterion.
- c. Selectivity: One of the major virtues of the gas chromatograph is its capability to almost completely isolate the expected impurities from each other. Water and carbon dioxide are retained by the molecular sieve columns used for separating the other listed impurities. They can be separated on a Poropak column. Such unlikely impurities as higher hydrocarbons or other organics would not be separated on the proposed column and thus would not be

detected. The mass spectrometer separates the various ions that are formed from the molecules introduced into it. When ions have the same mass but different chemical identity they are not separated by the low resolution mass spectrometers proposed for this use. Thus carbon monoxide and nitrogen appear at mass 28. However, if no other major source of these ions is present the quantity of carbon monoxide can be deduced from the quantity of mass 12 and 16 ions produced by the cracking procedure. This principle can be extended to more complex mixtures with loss of accuracy. Any substance yielding overlapping ions, such as from background of sample system or spectrometer system, will decrease sensitivity for small concentrations of impurities. All gaseous substances that enter the mass spectrometer will yield some ions so substantial quantities of an unknown substance would not go undetected. Selection of the mass spectrometer would dictate that a separate instrument for carbon monoxide be employed.

The other listed instruments are highly selective for the individual substance they are designed to measure.

- d. Sample Requirements: The absolute volume of sample required by the mass spectrometer is very small. The gas chromatograph requires about 10 cm^3 and the other instruments from 50 to 300 cm^3 . However, all are designed as flow instruments and the flow rate largely determines the speed of response. Nominal flows of $100 \text{ cm}^3/\text{min}$ are adequate for all except the carbon monoxide analyzer if a speed of response of the order of one minute is adequate. The carbon monoxide analyzer would require a flow between 400 and $500 \text{ cm}^3/\text{min}$ to attain this response speed.

One of the major unresolved problem areas is the effect of sodium vapor removal on the remaining composition of the gas stream. At 1200 °F the gas, if at equilibrium, will be of the order of 6% sodium vapor. If this cannot be fed to the instrument then it needs to be removed in such a manner that it does not take the components of interest with it. Two areas requiring study are evident: 1) the definition of the maximum temperature or maximum quantity of sodium vapor the instrument can handle, and 2) the effect on composition of removing sodium vapor in various type traps. The outcome of these studies may determine whether the gas chromatograph or the mass spectrometer is the preferred instrument. The remaining instruments listed cannot accept samples containing significant concentrations of sodium vapor.

- e. Reliability: This will vary with the individual product but it appears that all the listed commercial instruments will operate continuously for extended periods of time.
- f. Multiplexing: The capability to accept multiple samples may be controlled by the memory effects in the sample system as well as in the instrument itself. Cycle time may be important in determining how many instruments are required to furnish the data required. Data is not available on the mass spectrometer system yet. It appears a gas chromatograph would have a cycle time of about ten minutes.
- g. Data Display and Computer Interfacing: The output of both the gas chromatograph and the mass spectrometer has been coupled to computers, however, automatic computer control of the mass spectrometer is more complex than that of a chromatograph. The other instruments should be relatively simple to adapt to computer readout.

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APPENDIX

This appendix contains a description of the commercially available instruments that have been considered for the various gas analyses required in the inert gas systems of the FFTF. It consists of manufacturers' data with comments from comparisons and test data where available.

OXYGEN

Paramagnetic Type Meters

Instruments which utilize the paramagnetic properties of oxygen include:

1. Beckman Instruments, Inc., Model F3

This instrument utilizes a test body whose displacement is proportional to the change in volume magnetic susceptibility of the surrounding gas. An electrostatic system is used to counterbalance the displacing force and the potential required for this is measured. The restoring potential is a linear function of the oxygen concentration.

Ranges: 0 to 1% to 0 to 100%. Dual range available.

Accuracy: 1% of full scale.

Sample Requirements: Temperature 50 to 110 °F; flow 50 to 250 cm³/min; pressure 0 to 20 lb/in.² with less than 1 in. drop across instrument.

Case Dimensions: 15 w × 18 3/8 h × 12 1/2 d (in.).

Output: 5 mV standard, others available. Indicating meter.

Cost: \$1750.

Span gas required? Yes. Zero gas required? Yes.

Special Characteristics: Measurement essentially unaffected by other expected gases.

2. Leeds & Northrup Company, Model 7803-G Oxygen Analyzer

This instrument measures the change in thermal conductivity of a gas containing oxygen by the flow of oxygen induced by a magnetic field.

Ranges: 0 to 5% to 0 to 25%. Single range.
 Accuracy: 0.15% O₂ or 1.5% of range, whichever is greater.
 Sample Requirements: Temperature 40 to 100 °F; flow 1 to 45 ft³/hr; pressure 0.5 to 2 psig.
 Output: Millivolt.
 Span gas required? No, uses air. Zero gas required? No.
 Cost: \$1350.
 Case Dimensions: 11 1/2 w × 9 1/2 h × 17 1/2 d (in.).
 Special Characteristics: Background gas compensation required. No moving parts in sensing system.

3. Mine Safety Appliances Company, Model 802 Magnetic Oxygen Analyzer

This instrument measures the oxygen content of the gas by measuring the change in temperature of a filament caused by a change of flow of oxygen which is induced by a magnetic field.

Range: 0 to 1% to 0 to 100%. Three adjustable ranges.
 Accuracy: 2% of range.
 Sample Requirements: Temperature 40 to 110 °F; flow 0.5 to 2.5 ft³/hr; pressure drop 6 in. water.
 Output: 10 mV (adjustable).
 Span gas required? Air can be used.
 Zero gas required? Yes.
 Cost: \$1350.

Case Dimensions: 19 w × 12 h × 12 3/4 d (in.).
 Special Characteristics: Background gas compensation required. No moving parts in sensing system.

4. Hays Corporation, Model 632-II

Measure change in temperature of a heated nickel coil by the flow induced by a magnetic field.

Ranges: 0 to 1% and 0 to 10%, or other combinations between this dual range.
 Accuracy: 2% of range.
 Sample Requirements: Temperature up to 120 °F; flow 0.2 ft³/hr; pressure within ±15% of calibration.
 Span gas required? No. Zero gas required? No.

Output: 0 to 50 mV or mA.

Case Dimensions: 16 w × 9 3/4 h × 9 1/4 d (in.).

Cost: \$1630.

Comments on Group

The Leeds & Northrup and Mine Safety Appliances instruments differ in cell design with the MSA appearing to be more complex. There does not appear to be a technical reason for choosing one above the other of these instruments.

Hersch Cell Type Meters

The Hersch cell is one in which oxygen is reduced to hydroxyl ions at a silver electrode by the acquisition of electrons. The electrons flow from an anode of lead or cadmium where the metal is oxidized to form the oxide. The alkaline electrolyte is not consumed, only the anode. The four instruments listed below differ in mechanical arrangement of the cell components and anode.

1. Beckman Instruments, Inc., Model 80

Cathode: Silver wire.

Anode: Sheet lead separated by a porous diaphragm.

Electrolyte: 27.5% KOH.

Range: 0 to 5 to 0 to 500 ppm dual ranges at 5:1 ratio.

Accuracy: 5% when calibrated every 24 hr.

Sample Requirements: Temperature 50 to 110 °F;
flow 100 cm³/min; pressure 2 to 30 psig.

Span gas required? No, uses internal calibrator, but a span gas is more convenient.

Zero gas required? No, uses sample.

Output: 0 to 5 mV.

Case Dimensions: 16 w × 18 1/2 h × 11 d (in.).

Cost: \$2280.

2. Engelhard Industries, Mark II

Cathode: Helix of silver discs surrounded by porous plastic tube.

Anode: Cadmium with supplementary platinum electrode to extend life of cadmium.

Ranges: 0 to 10, 0 to 100, 0 to 1000 ppm. Triple range.

Accuracy: 5% when calibrated every 8 hr.

Sample Requirements: Temperature 60 to 75 °F; flow 8 liters/hr; pressure 1 to 250 psig.

Span gas required? No, uses internal calibrator.

Zero gas required? No, deoxygenates sample.

Output: 0 to 5 mV.

Case Dimensions: 18 3/4 w × 20 h × 9 1/2 d (in.).

Cost: \$2100.

3. Lockwood & McLorie, Inc., Model G.P.S. 1

Cathode: Porous silver.

Anodes: Cadmium and platinum.

Electrolyte: 25% KOH.

Ranges: 0 to 20, 0 to 100, 0 to 200 ppm.

Accuracy: 5% or 0.1 ppm, whichever is greater.

Sample Requirements: Temperature 0 to 50 °C; flow 50 to 200 cm³/min; pressure 15 to 30 psig.

Span gas required? No, measure is absolute for oxygen by coulometry.

Zero gas required? No, background current less than 0.07 ppm oxygen.

Output: millivolt, adjustable to match recorder.

Case Dimensions: 13 1/4 w × 15 h × 8 1/2 d (in.).

Cost: \$1200.

4. Teledyne Analytical Instruments, Model 306

Cathode: Silver gauze.

Anode: Lead immersed in electrolyte with no separating diaphragm.

Ranges: 0 to 5 to 0 to 1000 ppm with widest up to ten times narrow range; dual.

Accuracy: 2% of primary range when calibrated every 24 hr.

Sample Requirements: Temperature 30 to 120 °F;
flow 150 cm³/min; pressure 1 to 150 psig.

Span gas required? No, uses internal calibrator.

Zero gas required? No, adds oxygen to sample.

Output: 0 to 5 mV.

Case Dimensions: 16 w × 18 1/4 h × 10 d (in.).

Cost: \$2045.

Comments on Group

The Lockwood & McLorie cathode design is such that they claim 100% efficiency in converting oxygen to hydroxyl at the electrode surface. The other instruments all require calibration to compensate for the incomplete reaction of oxygen. They are about 10% efficient which would be expected to reduce their sensitivity at low oxygen levels. Because of the 100% efficiency the Lockwood & McLorie instrument requires accurate flow control whereas the other instruments are diffusion controlled and so are nearly independent of flow above a lower limit (about half that specified above for the instrument).

All these instruments require periodic replacement of the alkaline electrolyte and scrubber solutions with the Lockwood & McLorie probably being most frequent (every 2 weeks on its scrubber).

Diffusion Membrane Limited Hersch Cell Meters

A change in cell design so oxygen admission to the cell is by diffusion through a semipermeable membrane produces an instrument that is more nearly a probe-type analyzer.

1. Hays Corporation, Model 623.02

Polyethylene membrane.

Ranges: 0 to 5, 0 to 10, 0 to 25%. Triple range.

Accuracy: 3% of full scale.

Sample Requirements: Temperature 30 to 120 °F;
flow 0 to 10 ft³/hr; pressure -2 to +5 psig.

Span gas required? No. Zero gas required? No.

Output: 25 mV.

Case Dimensions: 10 5/8 w × 13 1/4 h × 6 3/4 d (in.).

Cost: \$464.

Special Characteristics: Cell is in instrument case so sample must be brought to it. Not affected by CO₂ and other common gases. Large concentrations of SO₂, Cl₂ or H₂S can interfere.

2. Teledyne Analytical Systems, Model 326 (Also makes multichannel systems)

Teflon membrane.

Range: 0 to 1 to 0 to 100%. Dual range available.

Accuracy: 1% of full scale.

Sample Requirements: Temperature 30 to 125 °F; independent of flow; pressure not specified.

Span gas required? Air. Zero gas required? No.

Output: 0 to 5 mV.

Cost: \$1200.

Special Characteristics: Cell can be mounted remote from instrument. Cell must be selected for CO₂ concentration of sample and is poisoned by acidic gases.

Comments on Group

The probe type sensor could be very convenient for cell installation, if it is not radiation sensitive or is in a shielded area.

Solid Electrolyte Meters

The operation of this class of instruments is based on the conduction of oxygen as ions through calcia stabilized zirconia at elevated temperatures. This makes it possible to set up a concentration cell whose voltage is a function of the ratio of partial pressures in a sample and reference gas. Air is usually used as the reference gas.

1. Westinghouse, Model 207E

Ranges: Triple range. 0.8 to 150 and 80 to 15,000 ppm, and 0.8 to 150% (a reading of 150% means that the pressure of oxygen in the sample side of the cell is 1.5 atm when air at atmospheric pressure is the reference gas).

Accuracy: 2% on meter, 1% on cell output. (Percent of full scale.)

Sample Requirements: Temperature -50 to +650 °F; pressure 1/4 in. water to 15 psig; flow 2 scfh.

Output: 0 to 10 mV.

Case Dimensions: 16 3/4 w × 8 1/2 h × 9 1/2 d (in.).

Cost: \$1950.

Special Characteristics: The model tested did not have provision for collection of the vented sample gases so would not be suitable for use with radioactive gas. Sample sensing cell and furnace mounted in instrument cabinet.

2. Thermo-Lab Instruments, Inc.

Range: 0.002 to 209,000 ppm oxygen (logarithmic).

Accuracy: 2% of full scale (meter deflection).

Output: 0 to 10 mV.

Sample Requirements (unspecified) may be used as a probe in a gloved box.

Case Dimensions: 8 × 6 × 6 cabinet of control-readout, sensor 8 × 4 × 3 (in.).

Cost: \$980.

Special Characteristics: Designed so the sensor can be used in a gloved box without pumping sample gas through it. Requires reference air flow.

Comments on Group

This class of oxygen sensor measures the excess of oxygen over possible fuels in the gas stream. There are a number of applications in FFTF where oxygen concentrations need to be measured at least potentially in the presence of a combustible substance, notably hydrogen. In these applications other types of oxygen meters are preferable.

MOISTURE

Automated Dew Point Instruments

Cambridge Systems, Model 992

This instrument measures the temperature of a mirror as dew begins to form on it. The mirror is cooled with a thermo-electric cooler to 100 °F below ambient temperature. For lower dew points, auxiliary cooling is necessary.

Range: 100 °F below ambient temperature. With auxiliary cooling -100 °F. With sensor preheating, to +200 °F.

Accuracy: 1 °F down to -20 °F; below this errors increase linearly to 2 °F at -100 °F.

Sample Requirements: pressure, vacuum to 20 psig; flow 60 to 600 cm³/min.

Output: Copper-Constantan thermocouple. Other outputs available on order.

Case Dimensions: Fits 19 in. relay rack.

Cost: -40 °F range, \$2740; -100 °F range, \$3995.

Special Characteristics: This is a primary measurement of condensable vapor and does not require calibration.

Alumina Film Hygrometer

The thickness and conductivity of a specially deposited alumina film varies with the moisture it absorbs from the gas surrounding it. The film is deposited on aluminum by an anodizing process and is overlain with a thin gold film by evaporation to form a condenser whose properties can be measured electrically.

Panametrics, Model 1000 .

Range: -110 to 10 °C.

Accuracy: 1 °C at 10 °C to 3 °C at -110 °C.

Sample Requirements: Probe does not require flowing sample; operates to 60 °C.

Case Dimensions: 12 1/2 w × 6 1/2 h × 10 1/8 d (in.).

Special Characteristics: Each probe requires individual calibration by the manufacturer. Probe life is still under investigation. Guaranteed for 6 months. Probe can sense changes in moisture content of gas directly without the delay introduced when sampling lines are necessary. One electronic chassis will handle five probes (six on automatic model).

Cost: \$1195. Replacement probes: \$195.

Electrolytic Hygrometers

These instruments absorb moisture from a gas stream into a coating of phosphorous pentoxide on the cell walls. This results in the formation of phosphoric acid which is subsequently electrolyzed to convert the water to hydrogen and oxygen. The electrolysis current is a measure of the water being electrolyzed, so by flowing gas at a constant rate the current is a measure of the moisture content of the gas.

1. Beckman Instrument Corporation, General Purpose Model

Ranges: 0 to 10, 0 to 30, 0 to 100, 0 to 300, 0 to 1000 ppm.

Accuracy: 5% of full scale.

Output: 0 to 10 and 0 to 50 mV.

Sample Requirements: Temperature to 80 °C; flow 100 cm³/min bypass flow to decrease response time lag 0 to 2 ft³/hr; pressure 10 to 100 psig. (Pumped model available for lower pressures.)

Case Dimensions: 11 w × 13 h × 8 d (in.).

Cost: \$1285.

2. Consolidated Electrodynamics Corporation, Model 26-303

Ranges: 0 to 10, 0 to 30, 0 to 100, 0 to 300, 0 to 1000 ppm.

Accuracy: 5% of full scale.

Output: Adjustable 0 to 100 mV.

Sample Requirements: Temperature to 50 °C; flow 100 cm³/min; pressure 20 to 100 psig.

Case Dimensions: 11 1/8 w × 5 5/8 h × 7 3/4 d (in.).

Optional in rack panel 8 3/4 h.

Cost: \$555. Replacement cells: \$150.

3. Kahn and Company, Inc., Model No. 141-060

Ranges: 0 to 10, 0 to 30, 0 to 100, 0 to 300, 0 to 1000 ppm.

Accuracy: 5% full scale.

Output: To potentiometric recorder.

Sample Requirements: Temperature to 80 °C;
flow 20 to 140 cm³/min; pressure 10 to 400 psig.

Case Dimensions: 12 w × 6 1/2 h × 7 d (in.).

Cost: \$495.

4. Manufacturers Engineering & Equipment Corporation

Manufactures a similar instrument.

Comments on the Group

This type instrument has been widely used. On a clean gas stream it operates satisfactorily for long periods. Sodium vapor or sodium compounds such as the oxide or carbonate will inactivate the cell. In normal use the cells require occasional replacement.

Quartz Oscillator Hygroscopicity Balance

duPont, Moisture Analyzer, Model 510005

Range: 0 to 5, 0 to 25, 0 to 250, 0 to 2500, 0 to 25,000 ppm.

Accuracy: ±5% of full scale.

Output: 0 to 10 mV.

Sample Requirements: Temperature, ambient;
flow 1500 cm³/min; pressure 40 to 100 psig.

Case Dimensions:

Control Module: 9 w × 6 3/4 h × 14 1/4 d (in.).

Sample Module: 16 w × 19 1/4 h × 13 1/2 d (in.).

Cost: \$3850.

Carbon Dioxide, Carbon Monoxide, Methane, and Water Impurities

This group of impurities can be measured by their infrared absorption properties. Nondispersive infrared type instruments are available which do not require prisms, gratings, or filters

to select the wavelengths which are characteristic of the substance to be measured. These instruments depend on the selective absorption of the infrared energy in a detector filled with the substance being measured. A chopper in the light path causes the generation of an ac signal that can be filtered and amplified for accurate measurement. By proper choice of the detector filling gas, and filter gases, if necessary, the instrument can be made specific for individual gases.

1. Mine Safety Appliances Company, Lira Model 200

Ranges: 0 to 100, 0 to 1000 ppm (lower available for CO₂).

Accuracy: 1% of full scale.

Sample Requirements: Temperature 30 to 120 °F; flow controls speed of response. High flow rates increase pressure in cell increasing absorption for a given level of substance being measured. Cell volumes from 30 to 300 cm³, thus at 100 cm³/min response time would vary from approximately 0.5 to 5 min to equilibrium.

Output: Field adjustable 0 to 10, 0 to 25, 0 to 50 and 0 to 100 mV.

Case Dimensions: 12 w × 12 3/4 h × length depends on sensitivity requirements, varies from 19 to 68 in.

Cost: \$3300 to \$3600.

2. Beckman Instruments Corporation, Model 315A

Ranges: 0 to 100, 0 to 1000 ppm (lower available for CO₂).

Accuracy: 1% of full scale.

Sample Requirements: Temperature -20 to 120 °F; flow controls speed of response. Calibrate at operating pressure, normally atmospheric but may be higher.

Output: 0 to 10, 0 to 100 mV and 0 to 1 and 0 to 5 V.

Case Dimensions:

Amplifier Readout: 6 1/4 w × 6 1/4 h × 13 d (in.).

Analyzer: 6 1/2 w × 8 1/2 h × 29 to 61 d (in.).

Comments on Group

These instruments give a continuous reading of the impurity concentration in the sample stream on which they operate. They require both span and zero gases.

Nitrogen Impurity

Mixtures of nitrogen with argon or helium can be measured by exciting a glow discharge in the gas and measuring the ultraviolet light emitted from the nitrogen at 3371 Å.

Mine Safety Appliances Company, Spectro Analyzer

Range: 0 to 5 ppm up. (Upper limit not given in catalog sheet.)

Accuracy: 2% of full scale.

Sample Flow: 0.2 to 5 ft³/hr.

Output: 0 to 10 mV.

Span gas required? Yes. Zero gas required? No.

Case Dimensions: 28 3/8 w × 84 h × 24 3/4 d (in.).

Cost: \$6500.

Nitrogen Oxides

The chemical reactions that have normally been used to determine NO₂ have been automated for air pollution studies. Instruments for direct photometric determination of NO₂ are not known to be marketed but have been described in the literature.

Technicon Autoanalyzer

Range: 10 ppb to 10 ppm.

Accuracy: 10%

Sample Flow: 500 cm³/min.

Span gas required? No. Zero gas required? No.

Case Dimensions:

Analyzer: 18 w × 30 h × 14 d (in.).

Recorder: 18 w × 16 h × 14 d (in.).

Cost: \$5000.

Hydrogen Contamination

The determination of hydrogen by a specific method is not readily accomplished. The usual methods depend on measuring one of its properties and assuming it is the component of the mixture producing the effect. One of these properties is thermal conductivity which is higher than for any other gas. Helium is also high so one may be mistaken for the other if only thermal conductivity changes are measured. It is possible to design a multifilament thermal conductivity cell in which other gas properties participate in producing the observed effects so that the contribution from a single component is maximized and can thus be approximated. Combustion on a heated filament is another nonspecific measure that may indicate hydrogen. Combination of hydrogen with oxygen over a catalyst will produce water which can then be detected with a moisture monitor of some type. By proper selection of the catalyst it is possible to react the hydrogen without appreciable oxidation of organic materials such as methane, etc.

Thermal Conductivity

There are so many manufacturers of this type of equipment that only one example will be cited. This one is being tested on a prototype loop enclosure.

Mine Safety Appliances Company, Thermatron

Range: 0 to 4%.

Accuracy: 2% of full scale.

Sample Requirements: Flow 50 to 2000 cm³/min; pressure above 10 in. water.

Output: 0 to 10 mV.

Span gas required? Yes. Zero gas required? Yes.

Case Dimensions: 11 w × 8 h × 8 d (in.).

Cost: \$700.

Combustible Gas Analyzer

As with the thermal conductivity instrument there are many manufacturers of this type of equipment.

Oxygen Combination

This method is more applicable to trace levels of hydrogen than to the percent levels that are of concern in the cell blanket. Engelhard Industries manufacture an instrument of this operating principle.

Hydrocarbons

The hydrogen flame ionization detector is very sensitive to hydrocarbons and other organic materials. It is used in air pollution studies. Since this is an optional instrument only one manufacturer's product is listed as an example of what is available.

Teledyne Analytical Instruments, Hydrocarbon Analyzer

Ranges: 0 to 10, 0 to 100, 0 to 1000 ppm standard, others available.

Accuracy: 1% of full scale.

Sample Requirements: Flow 100 to 400 cm³/min; pressure up to 100 psig. Requires a fuel mixture of 40% hydrogen-60% nitrogen plus air or oxygen.

Span gas required? Yes. Zero gas required? Yes.

Output: 0 to 5 mV.

Case Dimensions: 16 w × 17 h × 9 d (in.).

Cost: \$2500.

Gas Chromatographs

The gas chromatographs suitable for sodium cover gas analyses will have to be process type instruments built to our specifications. Building of such instruments is a rapidly growing business. There are several known manufacturers and by the time we are ready to procure instruments for installation on FFTF there may be more. The one commercial system on

which experience has been accumulated was built by the Greenbriar Division of Bendix Corporation. It was built for analysis of trace impurities in helium. The impurities were hydrogen, oxygen, nitrogen, methane and carbon monoxide. It was found that it would reliably detect 1 ppm hydrogen and 0.5 ppm of the other impurities. An instrument built in BNW shops showed a greater sensitivity by a factor of two or three. The increased sensitivity was attained through the use of ultra high purity helium as a carrier and a micro cross section ionization detector. It has been reported that a laboratory instrument using the same type detector exhibited about tenfold greater sensitivity so additional improvements in the process type instrument may be possible.

Since instead of a helium cover gas it is necessary to analyze an argon gas, then the sensitivity of currently available instruments is such that the minimum detectable impurity levels will be about 10 to 20 times higher than for the same impurities in helium, i.e., about 5 to 10 ppm in the argon. The field of gas chromatography is a very rapidly growing one with many lines of investigation, one of these being detectors. If a suitable detector is developed it may become possible to detect impurities in argon at the same level as is presently possible with helium.

Known suppliers of process chromatographs:

Greenbriar Plant Division, The Bendix Corporation
Beckman Instruments, Inc.
Mine Safety Appliances
Process Analyzers, Inc.
Thermco Instrument Corporation

Greenbriar Division, The Bendix Corporation

Accuracy: 5% of full scale.

Cost: \$12,000 to \$15,000

Dimensions:

Analyzer: 27 w × 42 h × 18 d (in.).

Control: 11 w × 22 h × 27 d (in.).

Sample Requirements: Temperature to 120 °F;
flow 100 cm³/min; pressure 5 to 50 psig.

Span gas required? Yes.

Zero gas required? Carrier, pure helium or argon.

Mass Spectrographs

Many instruments of this class have been built using different methods of achieving the mass separation. After reviewing the literature on the various instruments available it was deemed advisable to investigate the performance of a time-of-flight instrument because of its mechanical simplicity and capability to operate at elevated temperatures. Additionally, a new, relatively inexpensive version of this instrument had been just introduced for which the manufacturer claimed sufficient sensitivity to be competitive with the gas chromatograph for the analyses of argon streams. This instrument is in the process of evaluation. An alternative form (quadrupole) of mass spectrometer is under evaluation at LASL for the same applications.

The three leading types of mass spectrometers for this use are:

- a. The quadrupole or its close relative the monopole, in which an rf field around the pole structure controls the flight path of a beam of ions so that only those of a given mass/charge arrive at the detector. This type instrument is capable of operation at elevated temperatures possibly higher than the other types.

- b. Time-of-flight (TOF), in which the various mass/charge ions separate because of their difference in velocity when accelerated to equal energies. When the ions are allowed to drift freely for a distance they become separated into discrete groups whose arrival time at the detector can be measured.
- c. Magnetic sector, in which the flight path of a beam of ions is controlled by passing them through a magnetic field. At a given ion energy and magnetic field only a given ion of a single mass/charge will arrive at the detector. Different masses are measured by varying the magnetic field or the ion energy. In general these instruments require more mechanical alignment maintenance than either of the other types listed.

Manufacturers include:

Quadrupole type:	Electronic Associates, Inc.	\$24,000
Monopole type:	General Electric Company	\$24,500
Time-of-Flight:	Bendix Corporation	\$16,000
Magnetic Sector:	Aero Vac Corporation	\$18,500

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