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**INFRARED DETECTION OF NUCLEAR
REACTOR COOLANT IMPURITIES
INTERIM REPORT**

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FEBRUARY, 1967

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INTRODUCTION

Water vapor content of reactor coolant gases is an important parameter which may serve as an indicator of leaks in a reactor system and often needs to be minimized to reduce corrosion and other undesirable effects. The most sensitive type of instrument practical for monitoring water vapor content of gases is the absorption hygrometer. Infrared absorption hygrometers measure the loss of energy in a water vapor absorption band from a beam of infrared radiation which has passed through a fixed volume of gas bearing the water vapor.

The developmental instrument described in this report has many applications other than measurement of water vapor content of gases. Instruments of similar design can be used as pyrometers⁽¹⁾ and as detectors of impurities other than water.⁽²⁾ Such applications are of importance to nuclear reactor technology.

Instruments have been described⁽³⁾ which utilize water vapor absorption bands centered at 1.38, 1.87 and 2.6 μ . Absorption bands at wavelengths greater than 2.6 μ are beyond the range of transmission of ordinary glass or quartz optics and beyond the range of sensitivity of lead sulfide cells. Absorption is the strongest in the 2.6 μ band, making this the most sensitive spectral region for appropriate

operation. Furthermore, present capabilities in thin film art are such that multilayer dielectric filters are readily designed and fabricated according to desirable specifications in the near infrared region.

SUMMARY

An experimental absorption hygrometer was developed to measure the water vapor content in a flowing stream of gas. The indicated accuracy of the instrument, in terms of dew point, is ± 2 °F over a range of dew points from -46 °F to +56 °F. To facilitate appropriate calibration of the hygrometer, a variable pressure saturator was developed and utilized. Based upon extensive tests of the initial hygrometer, the development and fabrication of a second generation system, termed Mark II, were undertaken with the expectation of improved accuracy and reliability.

PRINCIPLE OF OPERATION

Figure 1 shows a portion of the infrared absorption spectrum of water vapor. The absorption band at 2.6 μ is the strongest and, hence, the most desirable for use in hygrometry. However, if this band is to be used, the hygrometer filters must exclude the effect of the nearby strong carbon dioxide absorption band. Let two passbands be defined, one centered at 2.55 μ and

FIGURE 1. Water Vapor Absorption Spectrum

one at 2.3μ , with each being 0.1μ wide. If a beam of infrared radiation is transmitted through a cell containing water vapor, the transmitted energy in the 2.55μ band will depend upon the amount of water vapor in the cell; whereas, the transmitted energy in the 2.3μ band will not. Therefore, the energy transmitted in the 2.55μ band, if it effectively excludes the carbon dioxide absorption band, is a measure of the amount of water vapor present in the cell, and the energy in the 2.3μ band serves as a reference.

An instrument comparing the energy in two radiation bands is shown schematically in Figure 2. A tungsten ribbon filament lamp (GE 18AT10/IP-6V) serves as the source of infrared radiation. A rotating filter wheel, used to "chop" the radiation, is assembled (Figure 3) with four multilayer filters deposited in four quadrants on a glass substrate. One pair of opposing quadrants transmits only in the 2.3μ band, while the other pair transmits only in the 2.55μ band. An 1800 rpm synchronous motor is used to drive the wheel; thus, radiation passing through the filter alternates between 2.3 and 2.55μ in wavelength at a 60 Hz rate. The beam then traverses an absorption cell and is focused upon a lead sulfide detector by a 6 in. focal length lens made of Corning 2550 glass.

Two wedge filters, one adjusted manually and one positioned by a servomotor, are used to modify the spectral content of radiation from the lamp and cause equal effects upon the lead sulfide photoresistor from the 2.3 and 2.55μ passbands. The manually positioned filter is used to affect a

1. Lamp
2. Servo Driven Filter Wedge
3. Manually Adjusted Filter Wedge
4. Rotating Filter Wheel
5. Synchronous Motor
6. Mirror
7. Lens
8. Absorption Cell
9. Photo Cell
10. Pre Amplifier
11. Servo Amplifier
12. Servo Motor

FIGURE 2. Schematic of Radiation Ratio Hygrometer

FIGURE 3. Filter Wheel

zero adjustment in the readout. Any increase in the water vapor content of the absorption cell reduces the energy content in the $2.55\ \mu$ band and thus produces an alternating current signal across the lead sulfide cell. This signal is amplified, phase detected, and used to energize a servo-motor. The servo-motor moves the wedge filter to decrease the energy in the $2.3\ \mu$ passband until the photocell can no longer detect a difference in energy content in the two beams. Similar null balancing is achieved for a decrease in water vapor content. The wedge filter position, which is the measure of the water vapor content of gas in the absorption cell, is read out electrically by a ten-turn potentiometer driven by the screw that moves the servo-positioned wedge filter.

FILTER CHARACTERISTICS

The rotating filter wheel consists of four quadrants, each made up of multilayer stacks having the form

HLHLH on a glass substrate. Here H represents a quarter-wave optical thickness of germanium, and L represents a quarter-wave optical thickness of silicon dioxide at the wavelength of peak transmission. The four quadrants form two opposing pairs of filters (Figure 3), where one pair has a bandpass peaking at $2.55\ \mu$ with a width at half-maximum transmission of 85%. Details of the transmission spectrum may be seen in Figure 4.

The two wedge filters have the form HLHLH on a glass substrate, and the optical thickness of these layers varies linearly from one end to the other. The result is that, with proper adjustment of the manually positioned filter, the other filter can always be positioned by the servo-motor so its absorption in the $2.3\ \mu$ passband, will compensate for any degree of absorption by water vapor in the sighting path.

Transmission characteristics of the wedge filters are shown in Figure 5. The two spectra shown were taken, one

FIGURE 4. Rotating Filter Wheel Transmission Spectrum

at each end of a 2 in. long wedge. The actual physical wedge, existing only in the five layer dielectric stack, amounts to about 5 millionths of an inch in 2 in. and is, therefore, imperceptible except in its effect on transmission characteristics.

ELECTRONIC ARRANGEMENT

Electronic circuits developed to energize a readout meter and recorder, bias the photocell, amplify the signal from the photocell, and provide synchronous detection are shown in Figure 6. A lead sulfide detector energized by a

*FIGURE 5. Transmission Spectra Taken,
one at Each End of Wedge Filter*

9.4 V mercury battery serves to convert the infrared signal to an equivalent electrical signal. The four quadrant rotating filter wheel (previously described) "chops" infrared radiation from the lamp to produce a

60 Hz signal in the output of the photocell if there is any deviation from a fixed energy ratio between the 2.3 and 2.55 μ bands. The 1800 rpm synchronous motor used to drive the filter wheel is energized from a 115 Vac, 60

FIGURE 6. Electrical Infrared Absorption Hygrometer

Hz line through a phase changing network composed of relay K and two limit switches. Activation of either limit switch causes the relay to reverse the phase of the motor. If the synchronous motor should lock in on line frequency 180° out of phase, the servo loop will cause the wedge filter to be driven to one end of its travel, where it will activate one of the limit switches and change the phase of the synchronous motor.

MECHANICAL ARRANGEMENT

Figure 7 shows the mechanical arrangement of the system. The servo-motor

is connected to a 0.5 in. diam, 40 turns/in. screw through a slip clutch to protect against damage, such as that caused by driving the wedge filter to a mechanically limited position. A 10-turn potentiometer is coupled to the end of the screw opposite the servo-motor.

The manually adjusted wedge filter is not shown in this picture, but it is normally mounted between the servo driven wedge filter and the lamp compartment.

A cover seals the compartment in which all of these parts are mounted. The compartment is purged with dry

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FIGURE 7. Mechanical Arrangement

nitrogen and further protected against change in water vapor level by a bag of desiccant placed in the compartment.

CALIBRATION EQUIPMENT

Gases of known and controllable water vapor content are needed to calibrate the hygrometer. The adiabatic saturation process, used to introduce a known quantity of water vapor into a dry gas, may be understood by reference to Figure 8. The equipment consists of an inlet leading to a pressure reducing valve followed by a constant temperature bath. Water added to the gas at the constant temperature bath station produces a mixture having a dew point equal to the temperature of

the bath at the pressure indicated on the meter. Variation in humidity ratio W with meter pressure P_m is given by

$$W = \left(\frac{M_v}{M_g} \right) \left(\frac{P_v}{P_m - P_v} \right) \frac{\text{gm H}_2\text{O}}{\text{gm Gas}}$$

where M_v is the molecular weight of water vapor, M_g is the molecular weight of the gas, and P_v is the partial pressure of the water vapor. Thus, the water vapor content is readily controlled by changing the gas pressure P_m in the constant temperature bath.

Two methods of calibrating the variable pressure saturator are provided. First, the gas flow is metered and

FIGURE 8. Variable Pressure Saturator

passed through a desiccant, which is weighed before and after a measured quantity of gas has passed through. The difference in weight is the amount of water vapor in that quantity of gas. The other method of calibration is achieved by comparison to a calibrated dew point indicator which receives the water vapor carrying gas from the outlet of the variable pressure saturator. Calibration curves obtained by using the second method are shown in Figure 9.

A photograph of the variable pressure saturator, dew point indicator and hygrometer is shown in Figure 10.

FIGURE 9. Dew Point as a Function of Pressure

CALIBRATION

Calibration data plotted in Figure 11 were obtained by using the arrangement shown in Figure 10. Dry nitrogen (having a dew point of -55°F) was passed through the variable pressure saturator, through an Alnor dew pointer, and, finally, through the absorption hygrometer. Dew points from 20 to 55°F were calculated for pressures ranging from 57 to 5 lb in⁻² on the saturator pressure gauge, but dry nitrogen at -55°F dew point was off scale for the hygrometer. Calibration points between -55°F and 20°F could not be obtained without modifications. One major modification would be the rebuilding of the variable pressure saturator to permit the use of higher pressures.

Filters used for these calibrations completely eliminated the effects of carbon dioxide absorption. This was demonstrated by passing dry CO_2 through the hygrometer and noting that the hygrometer gave a reading equivalent to that obtained with dry nitrogen.

Some idea of the performance of the hygrometer at dew points below 20°F may be obtained from Figure 12, which is a plot of data taken with another set of filters. This set of filters was later replaced by a set designed to better eliminate sensitivity to carbon dioxide, which has an absorption band at $2.7\ \mu$ and which overlaps the water vapor band. In Figure 12, the dew point at -47°F is derived from an Alnor dew pointer reading of dry nitrogen, with no water vapor added by the variable pressure saturator.

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FIGURE 10. Calibration Equipment

The calibration data of Figure 11 demonstrate a ± 2 °F sensitivity and repeatability.

FUTURE DEVELOPMENTS

The second generation (Mark II) absorption hygrometer is presently under development. Its design includes

features which experience with the first system indicated would improve performance. Quartz is being used in place of Corning 2550 for the lens material because it is transparent in the visible region. This transparency facilitates optical system alignment necessary for focusing the lamp filament on

FIGURE 11. Calibration Curve

FIGURE 12. Calibration Curve-First Filter Set

the lead sulfide cell. Condenser lenses are used between the lamp and wedge filter to focus the lamp filament on the filter to promote more precise filter action; the wedge filter is made in circular form and mounted directly on the shaft of the servo-motor to eliminate improper mechanical movement. A multilayer stack in the form $(H/\frac{1}{2})LHLHL(\frac{H}{2})$ has been designed to reduce the "droop" in transmission on the long wavelength side of peak transmission, and better provisions are being made for sealing of the motor compartment from ambient atmosphere. These and many other improvements are expected to yield a hygrometer with improved accuracy and reliability.

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