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Operational Guide for the Chemical Analysis Module/ OSU Rate of Heat Release Calorimeter: The Combined Hazards Index

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16. Abstract

This technical note explains the operation, maintenance, and trouble shooting procedures needed to collect and reduce data for interior aircraft material evaluation using the Combined Hazards Index mathematical model in the Ohio State University Rate of Heat Release Apparatus.

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EXECUTIVE SUMMARY

The objective of this report is to provide instructions for the operation of the Chemical Analysis Module (CAM) of the Ohio State University (OSU) Rate of Heat Release Calorimeter. In addition to heat and visible smoke release rates, continous gas measurements are taken for the following six thermal decomposition products: carbon monoxide, carbon dioxide, oxygen, hydrocarbons, nitrogen oxides, and hydrogen cyanide. Equipment description, system schematics, and general maintenance is discussed as well as instructions for software use and data filing.

INTRODUCTION

BACKGROUND.

The Ohio State University (OSU) Rate of Heat Release Calorimeter is a standardized test method to measure heat and visible smoke release rates at various incident heat fluxes, sample orientations, and ignition modes. It has been accepted by the American Society for Testing and Materials (ASTM) (reference 1) and is currently being considered by the Federal Aviation Administration (FAA) as a regulatory test (reference 2) for aircraft interior materials.

To better understand the combustion phenomena, the FAA has undertaken a project to measure the release rates of toxic thermal decomposition gases, in addition to heat and smoke (reference 3). The Combined Hazard Index (CHI) (reference 4) is a mathematical model which integrates the toxic gas concentrations, on the basis of individual gas toxicity, and heat stress contribution to predict an incapacitation time. This provides an analytical means of ranking the total fire threat of many various types of materials on the basis of human survivability.

DISCUSSION

The OSU may be used to establish material fire performance over a wide range of heat fluxes. The FAA has found good correlation between 2-minute heat release values, obtained at 3.5w/cm^2, and material behavior tested under full-scale, postcrash cabin fire conditions. While this is the most appropriate point for testing, heat fluxes may be chosen that are higher or lower, depending on the scenario of interest. Due to irregular ignition properties at low flux, both impinging and non-impinging pilots are recommended for reproducible fire test results. Figures 1 and 2 show OSU design modifications effected by the FAA that are different from ASTM-E906. Consult references 1 and 2 for operational details. The remaining discussion concerns the Chemical Analysis Module operation, maintenance, and trouble-shooting.

DATA ANALYSIS SYSTEM. Prior to usage of any tape, data structure sizes must be allocated through a mark statement (mrk A, B) where A is a variable denoting the number of files and B is the file size. Before marking, the tape must be rewound (rew), and the least confusion will result if the entire tape is marked sequentially for each of the two tracks (trk 0, trk 1). The convention used has been to put all program files at the beginning of track 0. Generally, five program files are all that are needed to make each tape a self-contained system: i.e., one program for data acquistion, one for graphic output, one for calibration, one for system temperature checks, and one for software development. No program is longer than 5,000 bytes, and plenty of room exists for program expansion. Each data file requires space for sample identification, test date, and the data array for a 10-minute burn, with a sample every 2 seconds for eight data channels. The data files require 20,000 bytes. The beginning of the second track contains the thermopile baseline corrector files for the assorted heat fluxes which may be used. Each baseline corrector file requires 2,500 bytes, with the remainder of the tape marked for 20K-byte data file.

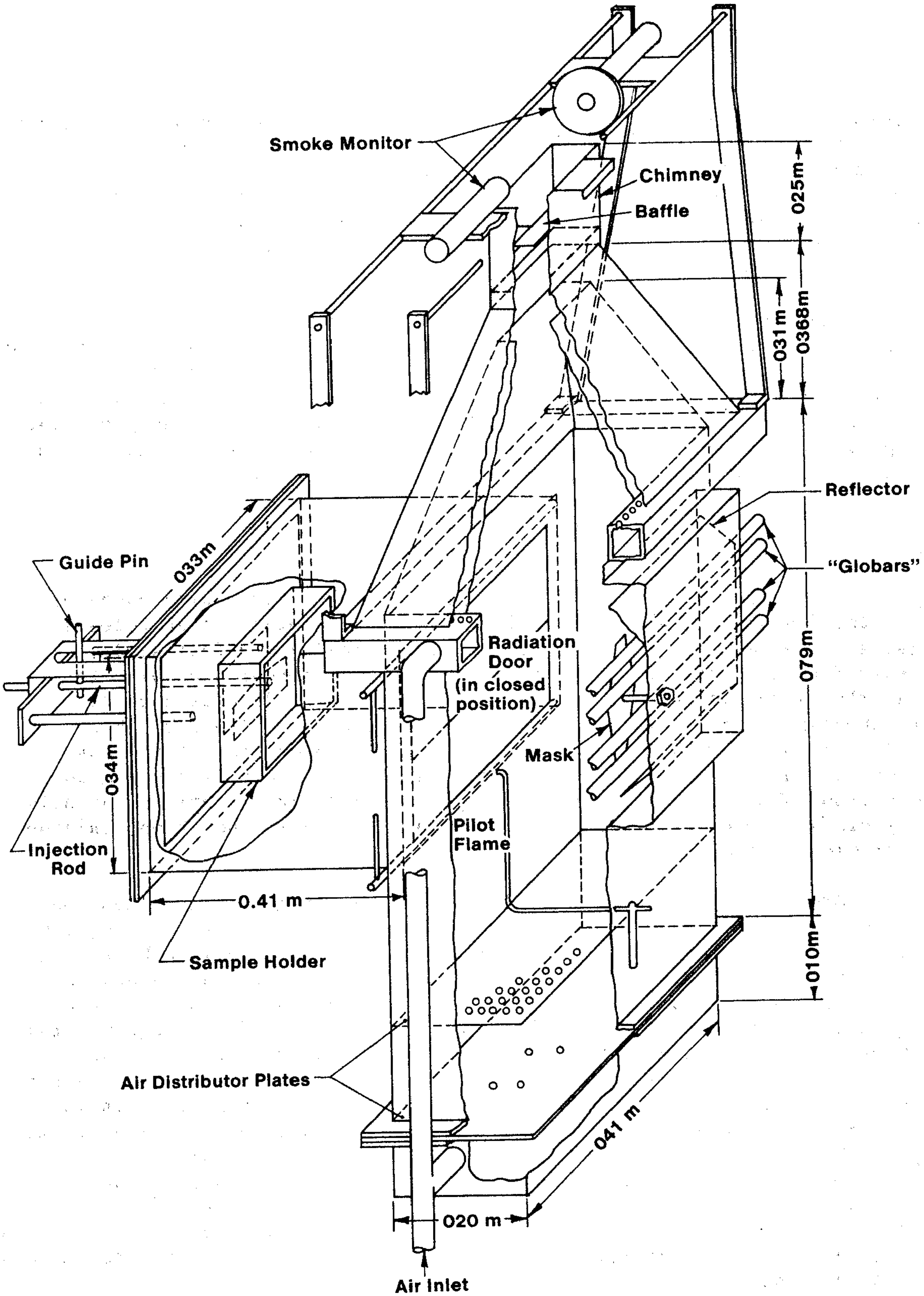
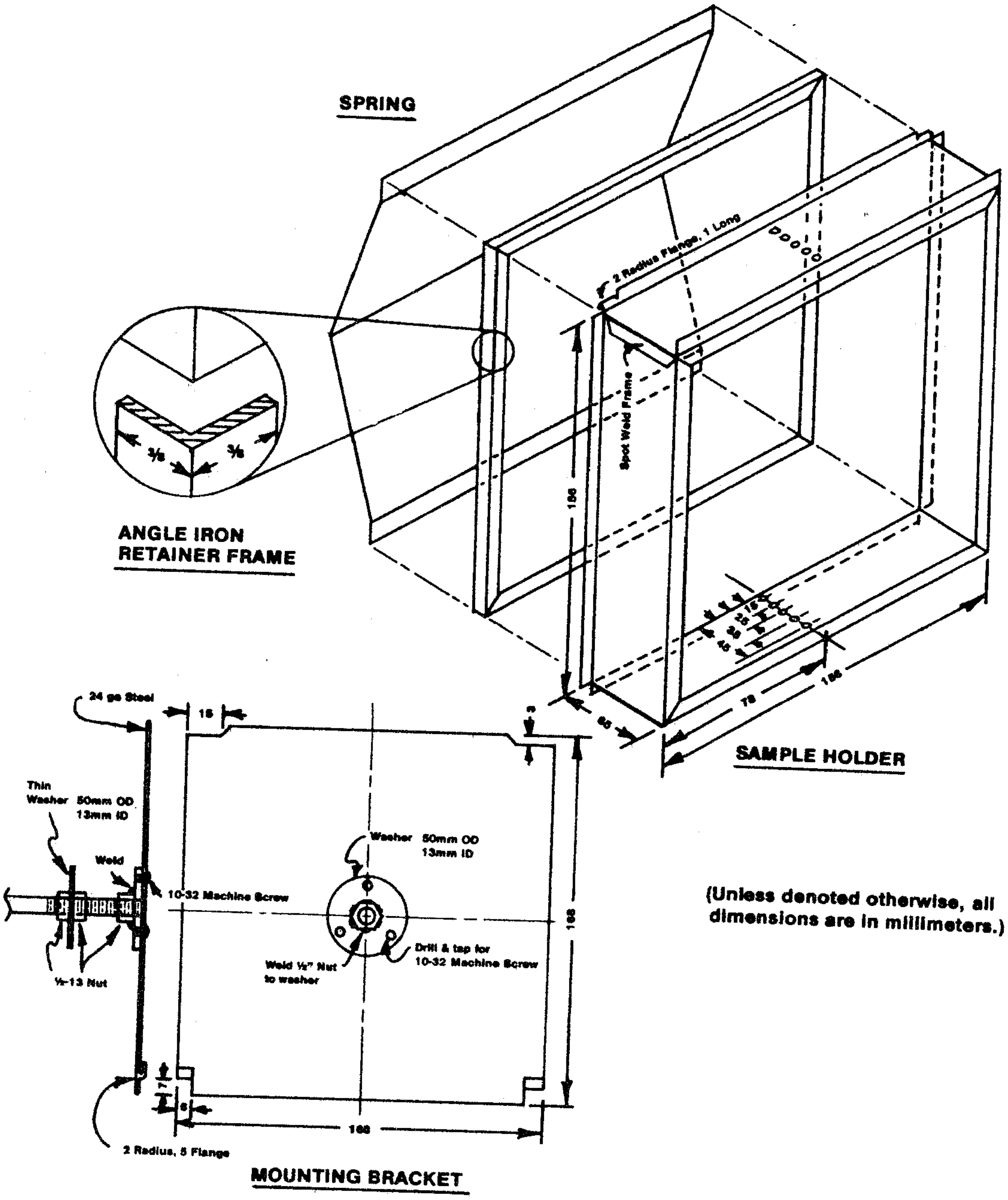


FIGURE 1. OHIO STATE UNIVERSITY RATE OF HEAT RELEASE CALORIMETER



(Unless denoted otherwise, all dimensions are in millimeters.)

FIGURE 2. SAMPLE HOLDER

The typical marking for a tape is done by live keyboard statements, i.e., each statement is followed by pressing the EXECUTE key.

Statement	Function
trk 0	set the tape track to zero
rew	rewind the tape
mrk 5,5000	mark for program files
mrk 10,20000	mark for data files

Error message 48 is displayed. This is expected because there is not room for 10 data files, but the tape is fully marked on track 0.

Statement	Function
trk 1	set the tape to track one
rew	rewind the tape
mrk 3,2500	mark for three different thermopile heat fluxes.
mrk 10,20000	mark the remainder of track 1

The tape is now ready for use, but blank. Transfer the desired programs from other tapes. This is done by selecting the track on which the program is stored and using the load statement (ldf A), with A as the file number. Exchange tapes in the drive and load the program, which is now in memory, onto the new tape using the record statement (rcf A), where A is the new file number. For example, to load a program on track 1, file 3 on one tape to track 0, file 1 on another, insert tape where the desire program resides.

```
trk 1
ldf 3
(exchange tapes in the drive)
trk 0
rcf 1
```

Program transfer is now complete.

Data transfer is more complicated and must be done under program control. While the large data files never need to be transferred, the thermopile baseline corrector files do. A simple program to accomplish this is listed below. However, after each statement press STORE, not EXECUTE. Because a new program is being created, one must not exist in memory. Place the tape in the drive which has the baseline file to be moved.

```
1 - dim T[301]
2 - trk 1
3 - ent "file desired", A
4 - ldf A, T[*]
5 - ent "put it where," B
6 - ref B, T[*]
7 - end
```

Press run and "file desired?" will appear on the display. Enter the appropriate number, and press CONTINUE. The tape drive will get the file and stop with "put it where?" displayed. Change to the new tape and enter the file number, press CONTINUE. The file is now transfered.

To determine what is on a tape, select the track, rewind the tape, type (tlist), and press EXECUTE. The internal printer will type the track, then file number followed by three columns of numbers. The first is the data code, the second the number of bytes of data, the third the number of bytes allocated for the data. A data code of 6 tells you its a program, and 3 is data only. The number of bytes in a program will help you identify which program is on tape. A code of 0, followed by 0 data bytes tells you the file is empty.

All programs are set up to display the program name when RUN is pushed. To get a new program push STOP, and ldf A where A is the file number. Each program gives instructions to the user. The aquisition program will ask for sample name, number, burn number, heat flux, etc. No knowledge of the workings of the program is needed to run the program.

The only software maintenance required is changing the calibration gas constant when a calibration gas tank is changed on the module, or changing the thermopile calibration constant to reflect a more recent calibration or change of the heat flux at which the specimen is tested. The appropriate calibration gas constants are:

C[1] - CO part per million (ppm) concentration
C[2] - CO_2 ppm
C[3] - Oxygen percent
C[4] - Methane ppm
C[5] - NO_2 ppm
C[6] - HCN ppm

The values are assigned in statement 6

C[10] is the thermopile calibration factor and is found in statement 82. It is the differential heat output in kilowatts (ΔkW) produced by a methane calibration flame divided by the differential thermopile millivolts (ΔMV) generated and the 6-inch by 6-inch sample (6" x 6" = .02323 square meters) area in square meters.

$$C[10] = \frac{\Delta kW}{\Delta MV\ (.02323)}$$

Many of the procedures above apply, even if gas data are not collected. Calibration of the thermopile, or aquisition of smoke and heat only, will run without supporting the module. Because the structure is larger than needed, most variables will be filled with zeros wasting data space, but because the structure is the same, no new plot or aquisition software is needed. A go to (gto) statement is needed to avoid the mandatory calibration routines, inserted at statement 27 and directed to statement 58.

CHEMICAL ANALYSIS MODULE. The Chemical Analysis Module (figure 3) is a self-contained unit capable of continuous analysis of six gases; carbon monoxide (CO), carbon dioxide (CO_2), oxygen (O_2), hydrocarbons as methane (CH_x), nitrogen oxides (NO_x), and hydrogen cyanide (HCN). It contains a sample delivery system, including pumps, filters, valves, and pressure monitoring devices, as well as calibration and detector fuel gases. The data analysis system, driven by the Hewlett-Packard 9825T desktop computer, is built-in and includes a digital voltmeter (Hewlett-Packard 3490 DVM), scanner (Hewlett-Packard 3495), flat-bed plotter (Hewlett-Packard 9862A), and printer (Hewlett-Packard 9871).

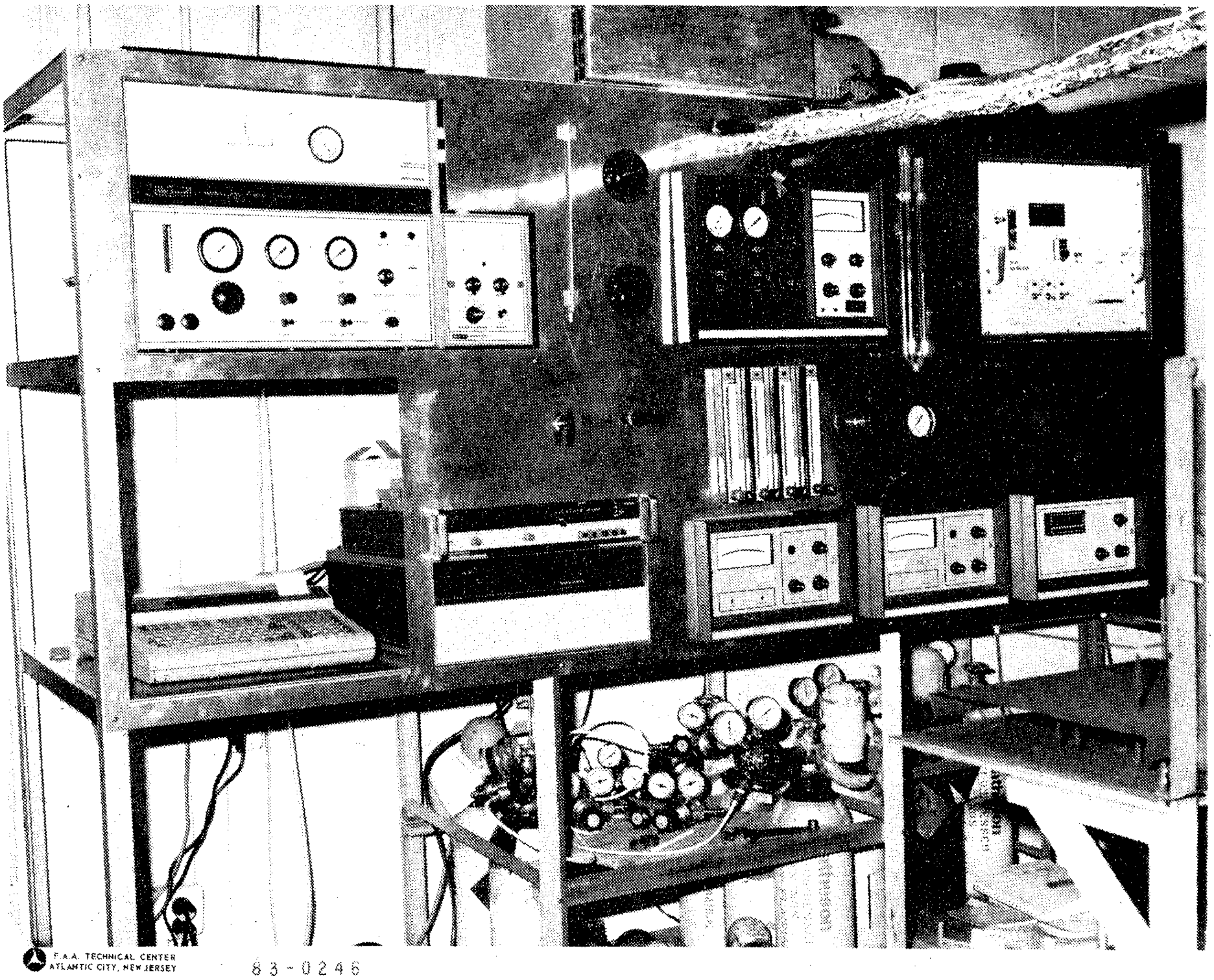


FIGURE 3. CHEMICAL ANALYSIS MODULE

The instrumentation consists of the following: Beckman 864 CO and CO_2 Analysers, a Beckman OM-11EA Oxygen Analyser, a Beckman 402 Hydrocarbon Analyser, a Beckman 905 NO_x Analyser, and a Kin-Tek CN-80 Hydrogen Cyanide Analyser. Analog output is set to 1 VDC full-scale for all analysers but the Kin-Tek, which is non-selectable. Thermopile signal ranges from approximately 10 to 20mv, the smokemeter from 0 to 0.5 VDC.

System schematic is shown in figure 4. The sample is drawn from the inner pyramidal section of the OSU, in the geometric center, crossing the short dimension of the exhaust stack, parallel with the plane of symmetry and 50 mm below the recombination point of the OSU airflow. The sample is drawn into the system by a positive displacement type pump, Bellows MB-18HT. The sample passes through two filters, one coarse filter consisting of 3mm glass beads and coarse fiberglass batt and a 1.2 μm mesh, 7.0 cm fiberglass filter disc. A mercury-filled manometer measures the differential pressure between the filters and pump to detect filter or probe clogging. The main system switching valve (Whitey 43x-F4) allows sample probe or calibration gases to be introduced to the system. After the pump, the sample is manifolded to the analysers with CO, CO_2, and oxygen in series, and with the others in parallel. The HCN and hydrocarbon analysers have their own pump, while adjustable back pressure valves control the flow to the others. The analysers are supplied with 2 liters/minutes of sample, except for the hydrocarbon analyser which draws 4 liters/minutes. The system draws 22 liters/minutes total flow, at the normal operating pressure of 2 psi and 90 mm Hg manometer reading. The excess sample is exhausted to minimize lag time for sample delivery to the analyser. Experimentation has shown that the oxygen analyser, which has the longest line, has a 6-second response time.

An alternative method of heat release measurement is determined by measuring the drop in oxygen concentration, as described by NBS (reference 5). The oxygen analyser is protected with an ascarite/drierite filled trap, 120 milliliters capacity, to scrub out water, CO_2, and acid gases. This is done because the analyser measures the partial pressure of oxygen, so the concentration of oxygen would be artificially lowered by the presence of combustion products. The oxygen analyser is calibrated simultaneously with the thermopile. Oxygen concentration is measured during the baseline flow, much like the thermopile millivolt reading. When the methane flow is step changed, a measurement of the depleted oxygen concentration is made, developing the proportionality constant for heat release per percent oxygen concentration change. It should be kept in mind that methane burns to CO_2 and water vapor only and is a gaseous fuel. When oxygen is consumed to produce CO_2 and water, the greatest amount of energy is liberated, this makes the calibration constant higher than for a real sample, which would consume oxygen while producing carbon monoxide, aldehydes, phenols, and other oxygenated species that release less energy. Also, energy is consumed in vaporizing solids or liquids, which does not occur in methane combustion. Oxygen depletion is then a more theoretical measure of energy liberation and will always give higher heat release values than the thermopile.

System Operation. Turn on all equipment (described in the respective owners manual). Computer, scanner, and digital voltmeter are required for aquisition. CO analyser range switch should be turned from "TUNE" to "3", with CO_2 analyser from "TUNE" to "2". O_2 analyser from "STANDBY" to "25%". NO_x analyser range switch on "250", mode switch to "NO_x Sample," making sure 30 psi air is supplied. Hydrocarbon analyser needs fuel gas and air on. To light the flame ionization detector (FID), switch from normal to overide and adjust fuel pressure to 30 psi,

CHEMICAL ANALYSIS MODULE

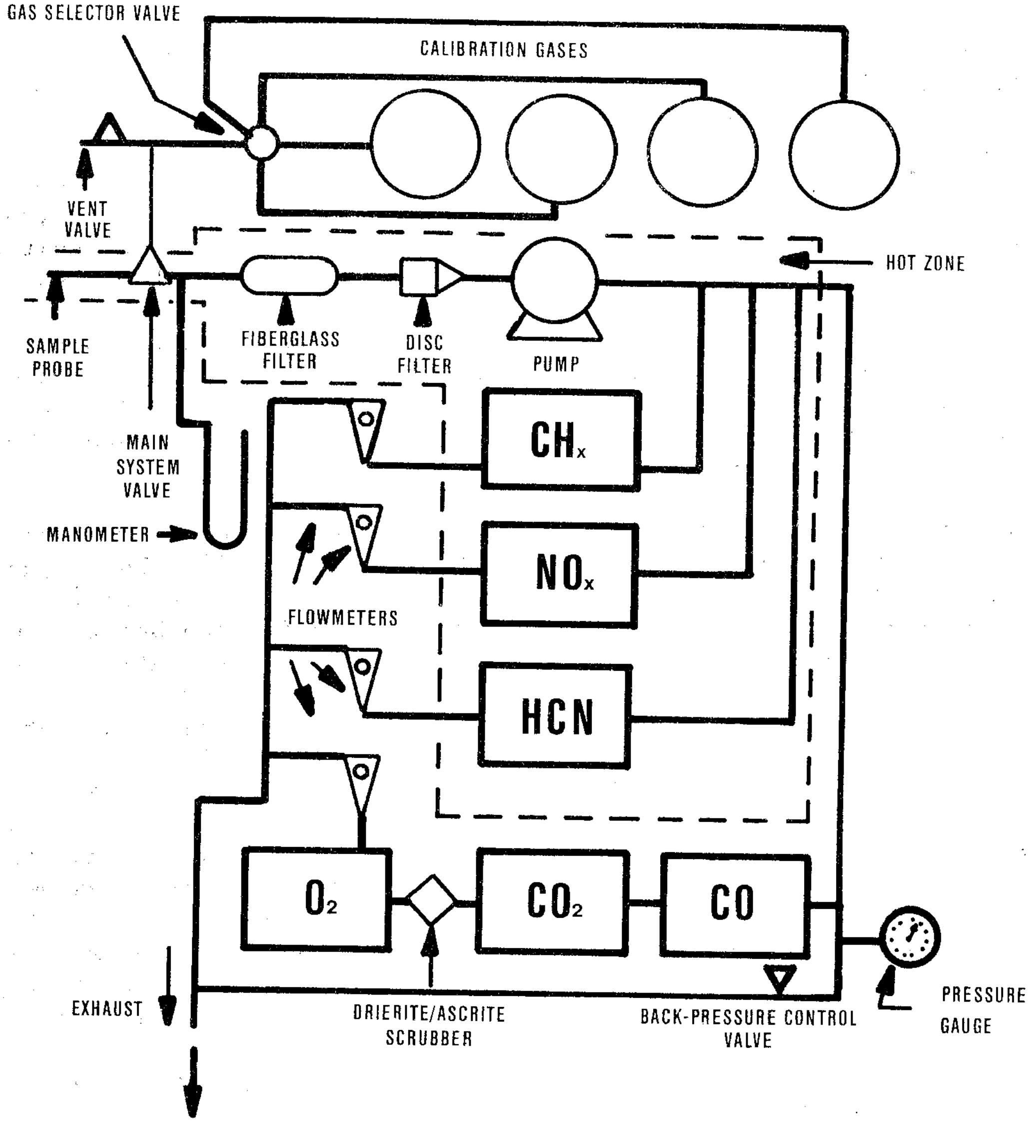


FIGURE 4. CHEMICAL ANALYSIS MODULE SCHEMATIC

and air to 5 psi. Momentarily turn ignite switch. Wait and repeat if flame out light remains flickering. When flame out light is off, raise air pressure to 15 psi, switch to "NORMAL" and turn on hydrocarbon sample pump. Turn on power to HCN analyser. Release pinch clamp on electrolyte reservoir return line. Turn on electrolyter pump, open electrolyte flow valve all the way. Begin to tighten pinch clamp until electrolyte flows to the cyanide detector cell. Close the electrolyte flow valve until the rate is one drop every 1 to 4 seconds. Turn on the sample pump to the HCN analyser.

Make sure a clean fiberglass filter pad is installed in the pump compartment. Switch the main system valve (just below pump compartment on face of the panel board) to calibrate, the gas selector valve to position 1, and the adjacent valve to vent. Turn on the main system pump. Manometer should read 45-50 mm (90 mm total differential pressure with ambient).

Adjust analyser flow rates to 60 flowmeter units (FMU), stainless steel ball, for all but the hydrocarbon analyser (far left). Start with the HCN analyser, as it has an independent pump. The flow adjust valve is located on the back of the HCN analyser, next to the sample input line. Increase back-pressure control valve on panel board to 60 FMU for the CO, CO_2, oxygen analyzers (far right). Adjust hydrocarbon analyser pressure regulator so that the panel board flow meter (far left) bounces between 120 and 150 FMU stainless steel ball. (Increasing hydrocarbon analyser pressure reduces flowmeter reading.) Adjust NO_x analyser pressure regulator to produce a 60 FMU reading (increasing pressure reduces flowmeter flow reading). Readjust main system back-pressure control valve to give 60 flowmeter units with the stainless steel ball for the CO, CO_2, O_2 analysers, and any other reading that may have changed. System pressure gauge should read between 2 and 3 psi. If any flowmeter float appears to stick, clean the flowmeter tube and balls. Note that the valve orifices have been removed from the flowmeter assemblies. Although the valves turn, they do not control flow.

Calibrate the Analysers. Load the aquisition software into the computer. (trk 0; ldf 1) Press RUN. "CHI Data Aquisition" is displayed. Enter the sample identification information as queried by the computer, pressing continue after each entry. When "Calibrate for CO" appears on the display, open the main tank valve. Increase tank pressure regulator to approximately 10 psi. Slowly begin to close the vent valve on the panel board, watching the manometer. Adjust the tank pressure regulator so that when the vent valve is completely closed, the manometer reading is the same as before initiation of calibration. Adjust gain control on the analyser. When the analyser is within 1 percent full-scale, the computer will accept the data and allow the next analyser to be calibrated. After the analyser is calibrated, open the vent valve, and close the main tank valve. Proceed in this manner until all analysers are calibrated for full-scale. With the pumps running, rapidly switch the main system valve from calibration to sample. If the valve is switched too slowly, mercury will be sucked from the manometer into the system. This can also happen if the calibration gas is off, and the vent valve is closed.

Zeroing the analysers is done with sample gas being drawn from the OSU, and allows offset from ambient gas concentrations to account for CO_2 production and oxygen depletion due to the pilot flames. All but oxygen and HCN analysers have zeroing potentiometers on the instrument faces. Absolute zero on the oxygen analyser is set internally, but can be checked during calibration of HCN or NO_x, which contain no oxygen. If the analyser shows more than 0.2 percent, check the system for leaks. Similarly the smoke meter has full-scale and zero controls and is checked by the computer. Oxygen baseline concentration is measured automatically by the computer, and "system ready" is displayed.

Sample Runs. The sample is wrapped with 0.001 aluminum foil and inserted into the holder assembly. When the OSU has stabilized, the plenum door is opened, sample is attached to the injection rod, the door is closed, and CONTINUE is pressed on the computer. A 1-minute hold period commences. At 40 seconds into this period the thermopile baseline output is measured. When the computer "beeps," the radiation doors are opened, the sample inserted into the test chamber, and the radiation doors closed behind the sample. The computer automatically sequences the scanner, measures the voltage of all instruments, mathematically manipulates the voltage into meaningful units, and stores these values in memory. The run lasts 10 minutes. Nothing needs to be done at this point but wait. After 10 minutes the data are transfered from memory to tape and the run is complete. The internal printer of the computer has also printed a "ticker tape" documenting the unique sample identification data, as well as the calibration gas concentrations, baseline voltages, and data tape location. Bookkeeping must be done by hand, because all data files contain the same number of bytes of data, although the data are different.

Data Output. To access the data, a plotting program is loaded from tape (trk 0, ldf 2 for release rates, trk 0, ldf 3 for Combined Hazard Index). The program will request the data location, as printed by the computer "ticker-tape" during aquisition. The computer will load the data from the tape, confirm sample identity, and request either the data channel if release rate data is desired, or sample loading parameters for the Combined Hazard Index. Channel 7 will plot smoke data, channel 8 thermopile heat release data, channels 1 through 6 gas data as previously mentioned. Channels may be plotted in any order, but each channel can be plotted only once because data are massaged during the plot routine, altering initial values. The program may be rerun as the data tape is never modified.

Routine Maintenance. After each run, the fiberglass disc filter must be replaced. The Drierite/Ascarite trap and the fiberglass/glass bead coarse filter must be replaced daily, or as often as required. The Drierite/Ascarite trap is oriented so that sample passes through the Drierite section first, then the Ascarite. (They cannot be mixed since they will react.) Eight-mesh indicator Drierite is used and turns from blue to pink when exhausted and replacement necessary. Twenty- to 30-mesh Ascarite is used, and must be interspersed with medium grade fiberglass. Ascarite used in pure form tends to fuse into a cement-like block and will cause flow problems. The fiberglass/glass bead filter needs to be cleaned when the manometer reading is 5 mm greater than normal. Retainer screens are cleaned with acetone, as are glass beads, while the fiberglass is replaced. Thermopile temperature sensing tips should be lightly brushed to remove soot at the start of each day.

Trouble Shooting. Flow problems will develop with any system, due to the highly corrosive sample nature, and will show up in two ways. Either the manometer will show excessive pressure differential, or there will be insufficient flow to the analysers.

Should the probe connecting tubing be clogged, disconnect it before the fiberglass/glass bead filter and the system will return to normal operating pressure as indicated on the manometer. Normally this is corrected by back-flushing the probe with trichloroethylene via a syringe adapted from Luerlok to Swagelok fitting connectors. If this doesn't work, remove the three-holed probe from the OSU chamber and clean it with a tubing brush and solvent.

Flow obstruction downstream from the pump is indicated by excessive pressure on the system gauge, and insufficient flow on the flowmeters. Often this will occur in the cold tubing between the analysers and the flowmeter. This can be checked by disconnecting the outlet tubing at the analyser, capping the tubing, and checking the analyser output with a clean flowmeter. Proceed sequentially upstream or downstream, depending on the results, with the separate flowmeter. The split point between the excess sample dump line and analyser line is another likely place to require cleaning as are the flowmeters. Use solvents, such as acetone, for cleaning. Avoid acids or bases due to the corrosive effects on the tubing. Due to the highly corrosive nature of the sample, the tubing can be eaten away to the point of leaking. This is demonstrated by manometer readings that are good, but low flow and low system pressure gauge reading when the back-pressure control valve is completely closed.

REFERENCES

1. American Society for Testing and Materials, Heat and Visible Smoke Release Rates for Materials and Products, ASTM Committee E-5 Fire Standards, Designation E-906, April 1984.

2. Federal Register Part II, Department of Transportation, 14 CFR Parts 25 and 121, Improved Flammability Standards for Materials Used in the Interiors of Transport Category Airplane Cabins; Notice of Proposed Rulemaking, Docket No. 24594; Notice 85-10, FR Vol. 50 No. 73, April 16, 1985.

3. Filipczak, R., Computerized Data Aquisition/Reduction for the Ohio State University Rate of Heat Release Calorimeter, DOT/FAA/CT-TN83/01, June 1983.

4. Filipczak, R., The Combined Hazard Index Calculated from Release Rate Data, DOT/FAA/CT-TN83/36.

5. Sensenig, D., An Oxygen Consumption Technique for Determining the Contribution of Interior Wall Finishes to Room Fires, Department of Commerce, NBS Technical Note 1128, July 1980.

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