

Volatile Organic
Carbon (VOC):

Organic carbon evolved from the filter punch in a He-only atmosphere between ambient and 120°C (the first organic carbon peak).

1.8 Related Procedures

SOP's related to carbon analysis activities and other manuals which should be reviewed in conjunction with this document are:

- DRI SOP #6-001.1 Shipping and Mailing Procedures.
- DRI SOP #6-009.1 Field and Laboratory Safety Procedures.
- DRI SOP #4-001.1 Creation, Revision, Distribution, and Archiving of Standard Operating Procedures.
- DRI SOP #2-106.3 Pre-Firing of Quartz Filters for Carbon Analysis

The DRI Carbon Analyzer Model 3000 Owner's Manual, revised 6/90.

The DRI Carbon Analyzer Model 3000 Maintenance and Troubleshooting Manual, revised 7/94.

2.0 APPARATUS, INSTRUMENTATION, REAGENTS, AND FORMS

2.1 Apparatus and Instrumentation

2.1.1 Description

The components of the DRI thermal/optical carbon analyzer are depicted in Figures 2-1 and 2-2; the complete gas flow schematic is shown in Figure 2-3. The programmable combustion oven is the heart of the carbon analyzer and includes loading, combustion, and oxidation zones in a single quartz "oven" as depicted in Figure 2-4.

In addition to the DRI thermal/optical analyzer connected to a Pentium compatible computer, the following items are needed for routine carbon analysis:

- Stainless steel punching tool: 0.516 cm² area for removing small sample punches from quartz filters. This punching tool must be kept clean and sharp. If the punching tool is sharpened, the punch area must be reverified.

Title: Thermal/Optical Reflectance Carbon Analysis of Aerosol Filter Samples

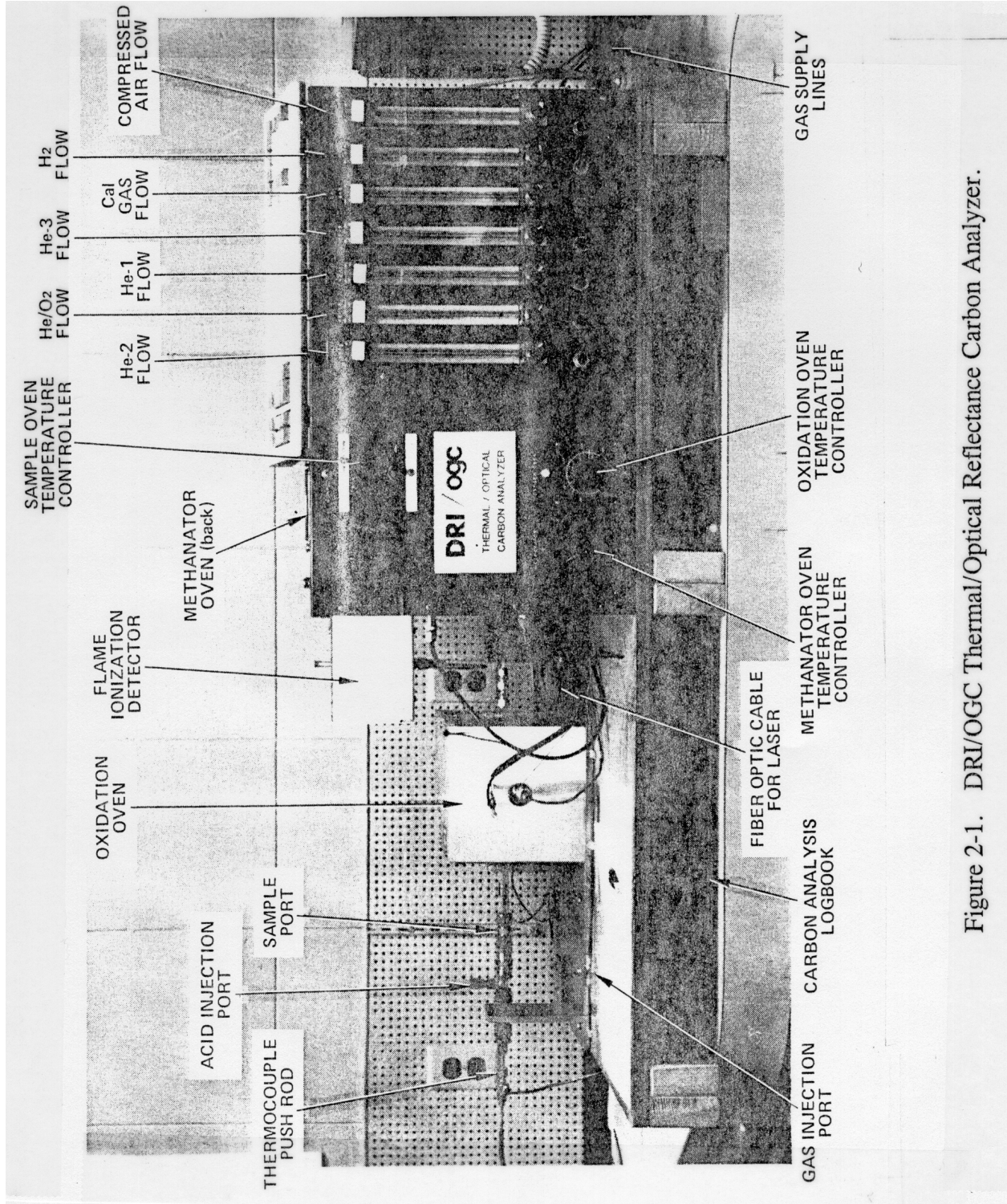


Figure 2-1. DRI/OGC Thermal/Optical Reflectance Carbon Analyzer.

Title: Thermal/Optical Reflectance Carbon
Analysis of Aerosol Filter Samples

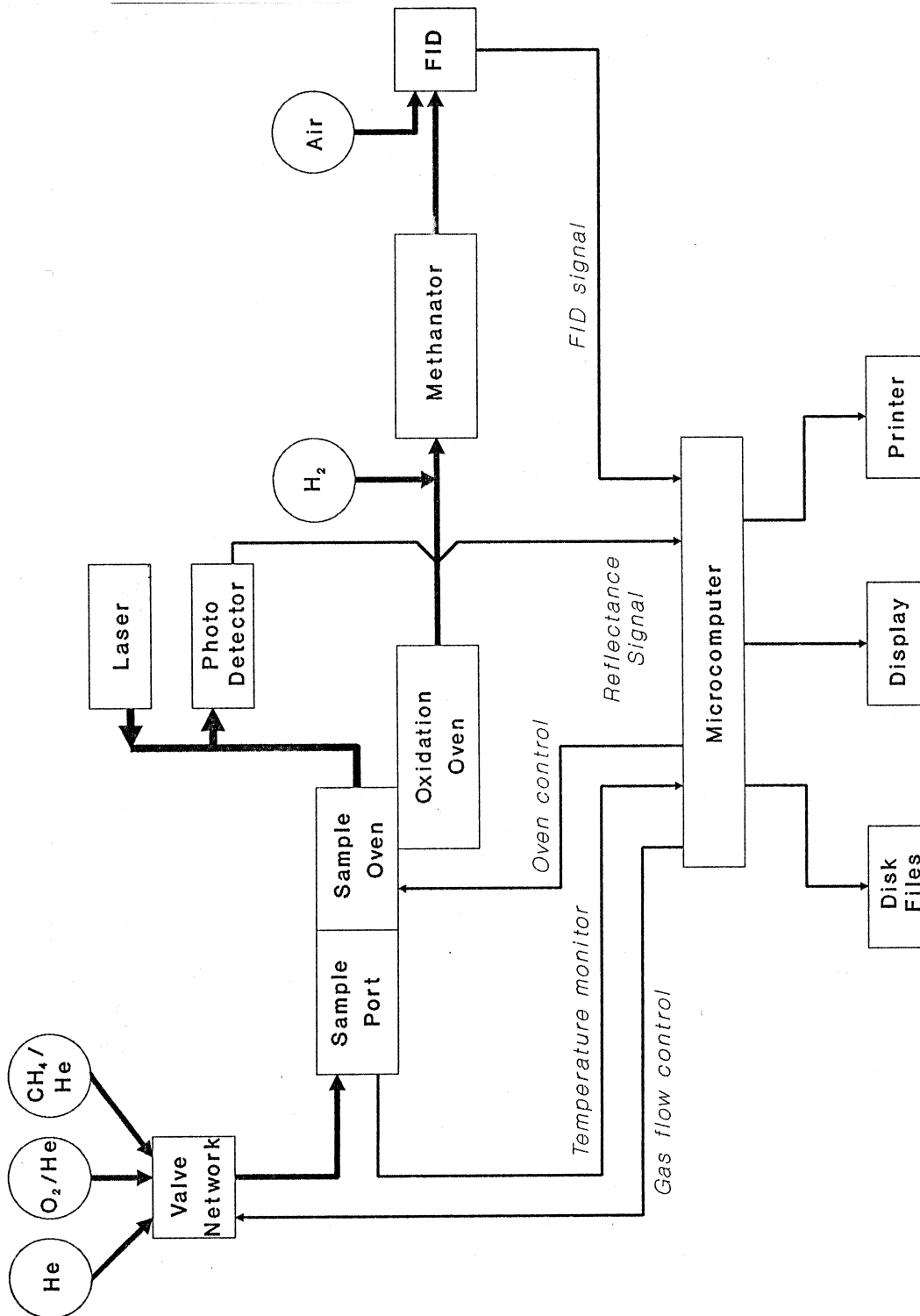


Figure 2-2. DRI/OGC Thermal/Optical Reflectance Carbon Analyzer Block Diagram.

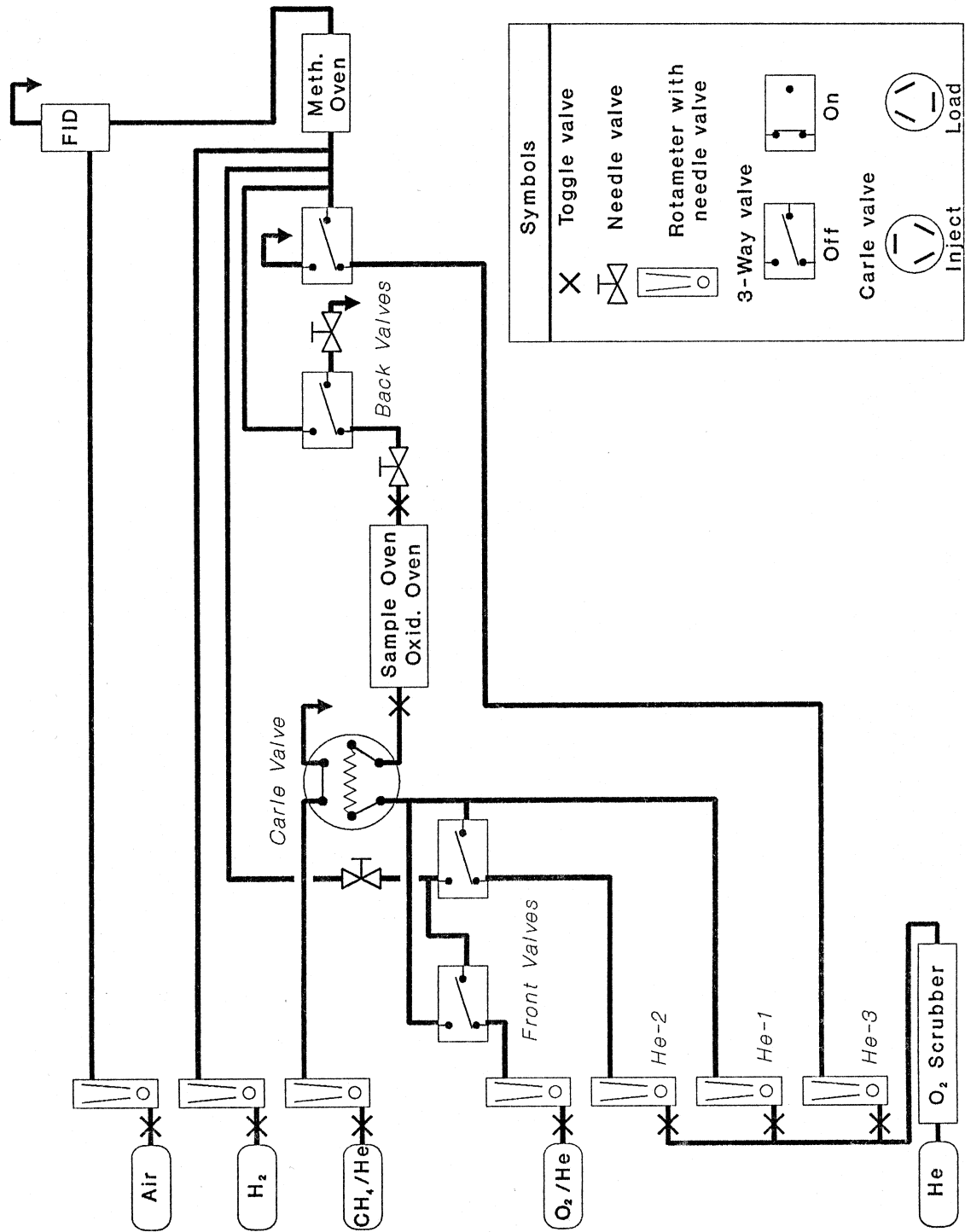


Figure 2-3. DRI/OGC Thermal Optical Reflectance Carbon Analyzer Flow Schematic.

Title: Thermal/Optical Reflectance Carbon
Analysis of Aerosol Filter Samples

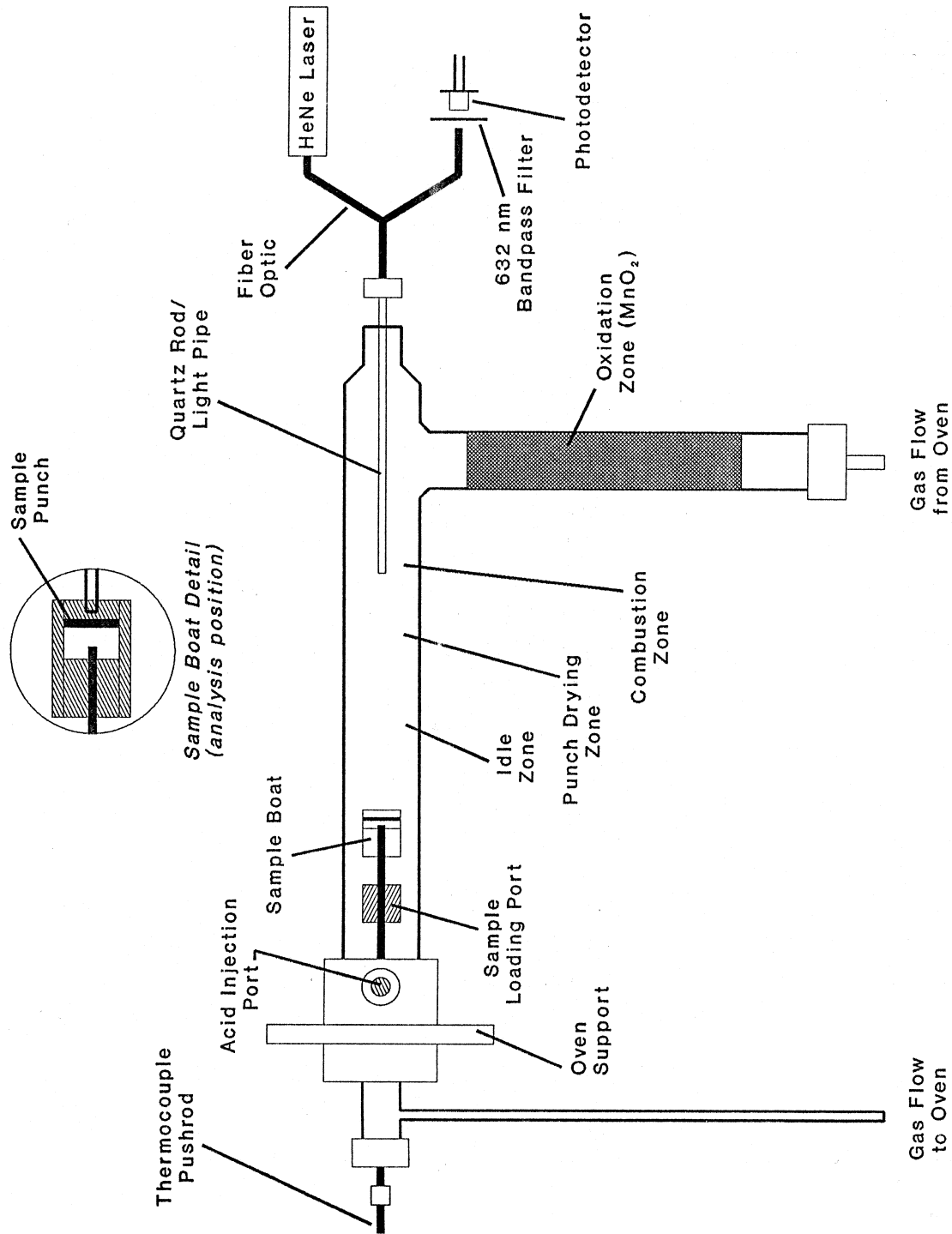


Figure 2-4. DRI/OGC Thermal/Optical Reflectance Carbon Analyzer Combustion Oven.

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- Syringes: Hamilton gas-tight 1000 and 2500 ml syringes for calibration injections; 25 ml syringe for carbonate analysis and for analyzer calibration.
 - Quartz filters: Pallflex 2500 QAT-UP or equivalent.
 - Tweezers.
 - Glass petri dish.
 - Logbook/notebook.
 - Transparent tape.
 - Kimwipes.
 - Small cooler.
 - Blue ice.
 - Butane lighter to light the FID.
 - Small adjustable wrench.
 - A copy of CARBON.EXE (the analysis program), version 4.0 or later, and CARBON.DAT (the analysis parameter file), version 4.0 or later.

2.1.2 Instrument Characterization

The DRI carbon analyzer is program-driven and data is stored automatically to disk via an Pentium compatible computer. Response times and signal lag times are built into the parameter file which is loaded when the analysis program begins. The program is event driven; that is, when the FID signal returns to its baseline after a minimum of 80 seconds at one analysis condition, the program will advance to the next temperature or carrier gas mixture. A maximum time limit per analysis condition is also established to prevent a slight baseline drift from holding the analyzer in one condition indefinitely. This method requires no sample pretreatment, requires between 15 and 70 minutes of analysis time per sample, requires at least one 0.516 cm² punch per filter, and destroys the sample punch.

Operator concerns for correct routine operation of the instrument include the following (refer to section 4 for more details):

- Insure that the sample port is tight after loading a sample punch.
- Remember to push the sample in when the computer tone sounds (Section 4.3); DO NOT leave the room until the analysis begins.

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- Insure that the thermocouple is physically decoupled from the sample boat after pushing in the sample to prevent oven temperature from influencing the laser reflectance signal.
 - Check the graphical printout after each analysis run to insure that the FID, temperature, and laser signals are behaving as expected (Section 4.3). Report any anomalies to the lab supervisor immediately.
 - The quartz oven is susceptible to breakage, especially at the sample port. Care should be taken to avoid exerting tangential pressure on the oven when manipulating the sample port fitting.

2.1.3 Maintenance

Regular maintenance for the analyzer involves daily checking of compressed gas supplies, cleaning the punching tool and tweezers between each sample with dry KimWipes (Kimberly-Clark Corporation), and backing up data files on a daily basis. Checks of laser adjustments (physical and electrical) are made at least monthly; analyzer calibrations are performed every six months. All calibrations and repairs must be recorded in the log book. Additionally, all repairs must be recorded in the maintenance log book.

Refer to the maintenance and troubleshooting guide for additional information.

2.1.4 Spare Parts

The following spare parts must be kept on hand to insure minimal interruptions in carbon analysis:

- Quartz rods: 3 mm nominal diameter, Homosil optical quality rod, (GM Associates, Oakland), cut to 9³/₄" lengths and polished on both ends using a lap polisher.
- Quartz ovens: specially built ovens by Adams & Chittenden Scientific Glass, 1414 Fourth St., Berkley, CA 94710, voice: (510) 524-9551, fax: (510) 524-955, contact person: Tom Adams. Or by Glasstech, La Plata, MD, voice: (301) 392-0723, contact person: Mike Trembly.
- Quartz boats: made at DRI from scraps of broken ovens.
- Thermocouple rods: 12" length by 1/8" OD, type K ground isolated with 316 stainless steel sheaths (Omega, Part # TJ36-CAIN-18U-18).
- FID flame tips: for Gow-Mac #12-800 FIDs (Gow-Mac, #132-117). Gow-Mac Instrument Co., P.O. Box 25444, Lehigh Valley, PA 18002-5444, voice: (610) 954-9000, fax: (610) 954-0599.
- Septa: 1/4" and 1/8", for injection ports.

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- Replacement needles for syringes.
 - Replacement oxygen/moisture trap (R&D Separations, Model OT3-2).
 - Replacement hydrocarbon trap (R&D Separations, Model HT200-4).
 - Replacement indicating oxygen trap (Chromatography Research Supplies, Model 202223).
 - Stainless steel wire: for forming "ears" to hold the sample boat in position and for wrapping the "ears" onto the thermocouple push rod (Rocky Mountain Orthodontics, #RMO E-19, 0.914 mm).
 - Quartz wool: for repacking the oxidation oven (Alltech Associates, #4033).
 - Teflon ferrules: Parker or Swagelok style, 1/2" ID, for the sample port fitting.
 - Teflon ferrules: 1/2" OD by 1/8" ID, for the thermocouple rod at the back of the oven.
 - Heating element for oven: custom made 650 W coiled heater (Marchi-Norman Associates #SDH175). Marchi-Norman Associates, Inc., 630 Price Ave., Redwood City, CA 94063, voice: (415) 364-3411, fax: (415) 364-4138.
 - FID battery: 300 VDC (EverReady #495).
 - Printer paper.
 - Printer toner cartridge.
 - Computer super disks, LS-120.

2.2 Reagents

The following chemicals should be reagent grade or better:

- Potassium hydrogen phthalate (KHP), for calibration use (Fisher, #P-243).
- Sucrose, for calibration use (EM Science, #SX1075-1).
- Manganese dioxide (MnO_2), crystalline, as an oxidizer in the oxygen oven (Nurnberg Scientific, #C5162).
- Nickelous Nitrate [$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], crystalline, used as a reducer in the methanator (Fisher Scientific, cat # N62-500, CAS 13478-00-7).

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- Chromosorb 60/80 mesh, used as a support for nickel catalyst in methanator (Supelco, catalog # 2-0165).
 - Hydrochloric acid (HCl), 0.4 molar solution, for use in cleaning punch and quartz ovens, and for use in carbonate analysis.
 - Distilled deionized water (DDW): total carbon background should be 6 ppm or less (determined as described in Section 5.1).

2.3 Gases

The following compressed gases should be industrial grade or better:

- Helium for a carrier gas, regulated to 15-20 psi with a metal diaphragm regulator. The higher pressure is required due to the pressure drop across the oxygen/moisture and hydrocarbon traps.

The laboratory supervisor will determine the minimum delivery pressure necessary for proper helium flows.

- 5% methane by volume in helium for calibration injections and calibration peaks; regulated to 10 psi by a metal diaphragm regulator, NIST traceable.
- 5% carbon dioxide by volume in helium for calibration injections; regulated to 10 psi by a metal diaphragm regulator, NIST traceable.
- 10% oxygen by volume in helium as a carrier gas, regulated to 10 psi by a metal diaphragm regulator.

In addition, the following gases are required:

- Hydrogen for the FID flame, regulated to 17 psi with a metal diaphragm regulator.
- Compressed air to supply oxygen to the FID, regulated to 10 psi by a metal diaphragm regulator

At least one backup cylinder per gas type should be kept on hand at all times. The calibration gases typically last for one year. The hydrogen, helium, and O₂/He mixture are typically replaced every four to six weeks. The compressed air is replaced every 4 to 5 days. All gases are replaced when the cylinder pressure drops below 500 psi.

2.4 Forms and Paperwork

All samples are logged into a receiving book and into a computerized database login file upon receipt at the laboratory. Refer to Figure 2-5 for the format of this logbook. A sample analysis list

will be prepared by the laboratory supervisor indicating which samples will be analyzed and any special instructions.

As individual samples are analyzed, entries are made in the "Carbon Analyzer Logbook", as shown in Figure 2-6. As each analysis run is completed, the sample analysis list is marked with the date and analyzer number, as shown in Figure 2-7.

Title: Thermal/Optical Reflectance Carbon
 Analysis of Aerosol Filter Samples

PROJECT	DATE REC'D	BY	DATE	SAMPLE ID	# SAMPLES	ANALYSIS	COMMENTS
IMPROVE ↓	030289	LLP ↓	9/89 ↓	Lot U ↓	481 NPS 14 NESC.	TOR ↓	25 mm Q ↓
IMPROVE ↓	040789	LLP ↓	9/89 ↓	Lot W ↓	470 NPS 27 NESC.	TOR ↓	25 mm Q ↓
SJVAS	041889	LLP	7/89	4 th Quarter assorted	353	XRF	47 mm T
IMPROVE ↓	042589	LLP ↓	9/89 ↓	Lot X ↓	492 NPS 18 NESC.	TOR ↓	25 mm Q ↓
AAAS	050389	LLP	5/31/89	89-11H - 89121	8	AC	8x10 GF

Figure 2-5. DRI Air Analysis Logbook Format.

Title: Thermal/Optical Reflectance Carbon
Analysis of Aerosol Filter Samples

Date: 6/2000

Number: 2-204.6

Revision: 6

12/27/95	MSM
1. BAKE OVEN	
2. CHANGED AIR (2200)	
3. LEAK TEST	
C11227-1/19.9/23828	
/945BLK	
SB1227-1	
/ZIRKEL.95/BATCH07	
GGGQ252-1	
GGGQ258-1	
GGGQ259-1	
HSGQ182-1	
HSGQ183-1	
HSGQ195-1	PUNCH FELL OVER DURING RUN
HSGQ196-1	
HSGQ197-1	
HSGQ198-1	
HSGQ199-1	
HSGQ201-1	
M11227-1/20.2/23693	
12/28/95	MSM
1. BAKE OVEN	
2. LEAK TEST	
M11228-1/20.4/23798	
/ZIRKEL.95/BATCH07	
HSGQ205-1	
HSGQ206-1	
HSGQ135-2	R FOR # 2
JUGQ332-1	
/SEPULVEDA.95/BATCH03	
SFTQ008-1	
SFTQ010-1	
SFTQ013-1	
SFTQ014-1	
SFTQ016-1	
SFTQ021-1	

Figure 2-6. DRI Carbon Analyzer Logbook Format.

Title: Thermal/Optical Reflectance Carbon
 Analysis of Aerosol Filter Samples

IMPROVE: Batch P3 Quartz

Date : 07/15/94
 From : F.Divita
 To : J.Chow
 C.Frazier
 B.Hinsvark
 Carbon Lab

Analysis: OC/EC by TOR : 300 samples, data in IMOETP3I.DBF

Sample Overview:

This analysis list covers samples from the NPS IMPROVE project. These are 300 PM2.5 samples on 25 mm Quartz filters, including no lab blanks and 3 field blanks. These samples were collected with an IMPROVE sampler.

Analysis Overview:

Sample deposit area: 3.8 cm²
 Analysis start date: After Lot03
 Analysis deadline : 6/16/94
 Sample location : Back freezer

Analysis Details:

Carbon analysis data will be stored in the D:\IMPROVE\LOTP3 directory.

Filter	Description	OC/EC
P59916	GRCA1040594C1P	Y _____
P59917	GRCA1040594C1S	Y _____
P59918	GRCA1040594C2P	Y _____
P59920	OKEF1040594C1P	Y _____
P59922	OKEF1040594C2P	Y _____
P59924	MEVE1040594C1P	Y _____
P59926	MEVE1040594C2P	Y _____
P59927	MEVE1040594C2S	Y _____
P59928	LAVO1040594C1P	Y _____
P59930	LAVO1040594C2P	Y _____

Figure 2-7. Example DRI Carbon Analysis List.