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August 25, 1995

Walter E. Demick, P.E.  
Section Chief  
Division of Hazardous Remediation  
New York Department of  
Environmental Conservation  
50 Wolf Road  
Albany, New York 12233-7010

RE: Hudson River - Enhanced River Monitoring Project

Dear Mr. Demick:

Enclosed for your review and approval is a work plan prepared by O'Brien & Gere Engineers that describes a program for testing the ability of the existing river monitoring system to estimate the impacts of potential PCB sources on loading to the Hudson River in the vicinity of the remnant deposits.

As part of this test, we are still evaluating the use of dense plastic beads for simulating the movement of PCB oils. However, the work plan does describes how these beads might be used and provides information on their composition.

Before we proceed with the use of dye or plastic beads in the river we will need the NYSDEC approval. The work plan contains background information on these materials. Assuming NYSDEC approval, we are planning on performing the first round of sampling on September 7, 1995.

Please contact me as soon as possible with any concerns or questions.

Sincerely,

John G. Haggard  
Engineering Project Manager

Enc:

309025

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cc: Bill Ports, NYSDEC  
Bob Montione, NYSDOH  
Douglas Tonchuk, U.S. EPA  
Victor Bierman, LimnoTech  
Al D'Bernardo, TAMS  
Wiley Lavigne, NYSDEC - Region 5

**Hudson River Project  
River Monitoring Test**

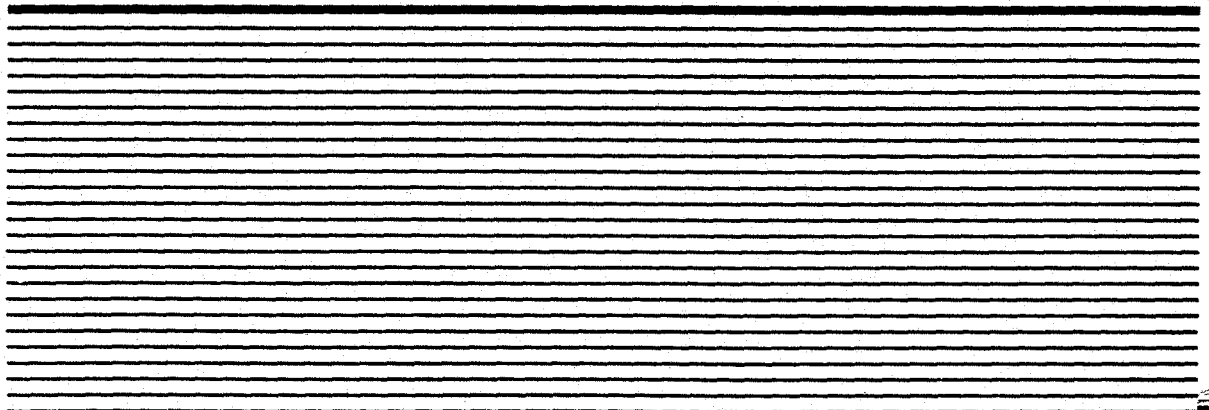


**General Electric Company  
Corporate Environmental Programs  
Albany, New York**

**August 1995**



**O'BRIEN & GERE  
ENGINEERS, INC.**



**Sampling and Analysis Plan**

**Hudson River Project  
River Monitoring Test**

*General Electric Company  
Corporate Environmental Programs*

**August 1995**

**O'Brien & Gere Engineers, Inc.  
Syracuse, New York**

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## 1. Introduction

This Sampling and Analysis Plan has been developed by O'Brien & Gere Engineers, Inc. (O'Brien & Gere) on behalf of the General Electric Company (General Electric). The principal purpose of the river monitoring test described herein is to collect water column PCB data to facilitate an assessment of current water column monitoring approaches, and to develop a more thorough understanding of the origin, fate, and transport of PCBs in the upper Hudson River.

### 1.1. Background

Water column sampling and high resolution capillary column PCB analysis has been conducted on the upper Hudson River between Hudson Falls and Thompson Island Dam (Figure 1) weekly or biweekly since April 1991 (O'Brien & Gere 1993a, 1993b, 1994a). In 1991 and again in 1992, PCB levels were observed to increase in the river upstream of the Thompson Island Pool sediments. The principal source of these loadings was discovered within an abandoned 150-year old mill in the vicinity of Bakers Falls in Hudson Falls, New York (the Bakers Falls source(s); O'Brien & Gere 1994b). Interim remedial measures were implemented in 1993 and 1994 to control these sources (O'Brien & Gere 1994b). Water column data collected before, during, and after the event provided new insights into the origin, fate, and transport of PCBs in the upper Hudson River (O'Brien & Gere 1994a).

Following implementation of the interim remedial measures, low concentrations of PCBs (<50 ng/l) in the water column have persisted (O'Brien & Gere 1994a). A number of potential sources of these PCBs have been identified, but current sampling methodologies were designed to monitor the effectiveness of the containment of capped sediments along the river banks (remnant deposits) not to isolate other potential sources.

This sampling and analysis plan (SAP) describes a research program to evaluate the current monitoring program's ability to quantify PCB flux to the Hudson River from the capped remnant deposits. Additionally, the results of the program will be used to quantify the relative magnitude of other potential sources of PCBs to the river.

## 1.2. Potential sources of PCBs

Six potential sources of PCBs have been identified which may be contributing to the low levels of PCBs currently found in the upper Hudson River:

*Upstream of Hudson Falls.* An intermittent source of PCBs is detected at the background sampling station located at Hudson River Mile 197.0 (HRM 197.0<sup>1</sup>; O'Brien & Gere 1993a, 1993b, 1994a). Sediments containing PCBs have been identified approximately 10 miles upstream of this station in Queensbury, (Engineering-Science 1995; O'Brien & Gere 1995a). These sediments may be the source of intermittent PCB detections at the background station.

*Hudson Falls plant site and vicinity.* On-going investigations at the Hudson Falls plant site and vicinity (HRM 196.9) continue to indicate that this area may be contributing PCBs to the river (O'Brien & Gere 1995b). This source was partially characterized and remediated in 1993 and 1994 (O'Brien & Gere 1994b). Subsequent investigations on the plant site have found dense non-aqueous phase liquid (DNAPL) in bedrock fractures beneath the site (Dames & Moore 1994). This DNAPL source may contribute to low level PCB concentrations that persist in the water column downstream of the source area (O'Brien & Gere 1994a).

*Fort Edward 004 area.* Soil and sediments in the vicinity of the General Electric Fort Edward facility 004 outfall located at approximate HRM 196.1 are currently being investigated (O'Brien & Gere 1994c). Elevated levels of PCBs have been detected in soils and sediments adjacent to and downstream of the outfall.

*Remnant deposits.* Removal of the Fort Edward dam in 1973 by Niagara Mohawk dropped water levels in the dam pool and left an estimated 1.5 million cubic yards of PCB containing remnant deposits along the banks of the river between HRM 196.4 and HRM 194.7.

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<sup>1</sup>The north-south orientation of the river provides a convenient location reference. Hudson River Mile 0.0 (HRM 0.0) is located at the Battery in New York City and river mile increases travelling north up the river.



In-place containment of the remnant deposits was performed by General Electric in the fall of 1990 (JL Engineering 1992) in accordance with the 1984 U.S. Environmental Protection Agency's (USEPA) Record of Decision for the site.

River monitoring is performed pursuant to a Consent Decree (90-CU-975) between General Electric and the United States. The current monitoring program, the Post-Construction Remnant Deposit Monitoring (PCRDMP), was initiated in 1992 following completion of the in-place containment measures (O'Brien & Gere 1992a, 1993b, 1994a). The results of the PCRDMP have identified increases in water column PCB concentrations through the remnant deposit region of the river (O'Brien & Gere 1994a). As discussed below, this increase may be due to the influences of other sources in the region or sampling anomalies.

*Thompson Island Pool sediments.* As the first pooled area downstream of Hudson Falls and Fort Edward, Thompson Island Pool is a sink for sediment particles and, potentially, for PCBs originating from sources upstream. Water column data collected at Thompson Island Dam (O'Brien & Gere 1993a; General Electric Company 1993, 1994, 1995) indicate that water column concentrations increase through Thompson Island Pool. Additionally, the PCB composition shifts from one that resembles Aroclor 1242 at the Fort Edward sampling station to one that is altered by the enrichment of mono- and di-chlorobiphenyls. The increase in PCB concentrations through the Thompson Island Pool may be attributable, at least in part, to sources upstream or sampling anomalies, as discussed in Section 1.3 and 1.4.

*Thompson Island Pool dredge spoil sites.* For navigational purposes the New York State Department of Transportation (NYSDOT) dredged the river in Thompson Island Pool during the 1950s through the 1970s (Malcolm Pirnie 1980). As a result, several NYSDOT dredge spoil sites containing sediments with PCBs are located adjacent to the Hudson River in Thompson Island Pool. In addition the Fort Miller dredge spoil site is located adjacent to the Moses Kill (HRM 191.5), a tributary to the Hudson River. Ground water discharge through these dredge spoils sites may be contributing PCBs to the river.

### 1.3. PCB dynamics

Water column PCB monitoring over the past several years has identified several PCB fate and transport phenomena:

*Consistent increases in PCB concentrations between HRM 196.8 and HRM 194.2 (reach 9) have been observed.* Since initiation of the PCRDMP in 1992, increases in PCB concentrations from upstream to downstream of the remnant deposits have been observed (O'Brien & Gere 1993b, 1994a). PCB concentrations downstream of the remnant deposits (HRM 194.2) have consistently been approximately twice that of concentrations upstream (HRM 196.8). In 1994, geometric means for these two stations were 31 and 17 ng/l, respectively.

*Little or no changes in PCB composition occur as water flows through reach 9.* The composition of PCBs in the water column are consistent over time between upstream and downstream stations in reach 9 (O'Brien & Gere 1993b, 1994a). PCB composition data from the remnant deposit sediments are different from water column data in that the PCBs have been dechlorinated, although composition data are limited. If the remnant deposits were responsible for the apparent PCB loading, then a noticeable shift in PCB composition would be expected to occur as the river passed by the remnant deposits. This does not occur.

*Upstream and downstream loadings in reach 9 appear correlated.* The apparent PCB mass loading from the area of the river between HRM 196.8 and HRM 194.2 (e.g., the remnant deposits including outfall 004 area) varies as the mass loading from upstream varies (between HRM 197.0 and 196.8). Generally, as upstream source loading increases, the apparent loading from the remnant deposits increases (O'Brien & Gere 1994a,c). Given the current understanding of PCB loading from the upstream sources, it is not clear physically why increases in PCB inputs in the area of the remnant deposits would occur at the same time increases at the upstream sources occur. The sources would be expected to behave independently. This suggests that the remnant deposits are not the source of the PCB loading.

*Anomalous loading from Thompson Island Pool.* Summer low flow PCB loadings from the Thompson Island Pool increased following the upstream loading events of 1991 and 1992. The summer low flow loadings from Thompson Island Pool decreased in 1994 following control of the Bakers Falls source(s) (O'Brien & Gere 1993a; General Electric Company 1993, 1994, 1995). However, Thompson Island Pool PCB loadings remain elevated compared to those observed prior to the upstream loading events (O'Brien & Gere 1993a). These observations suggest a causal link between the elevated Thompson Island Pool loads and upstream loading. It has been hypothesized that DNAPL originating from the Hudson Falls plant site area enters the river during the loading events and is transported undetected by the monitoring program as part of the bed load to the Thompson Island Pool. Once in the Thompson Island Pool these DNAPL PCBs may become incorporated within surface sediments. These "fresh" deposits of PCBs are later released to the water column producing the observations at Thompson Island Dam.

If the PCB inputs into Thompson Island Pool are underestimated, then the amount of PCBs estimated to come from the old buried sediments will be overestimated by the current monitoring program.

#### **1.4. Potential limitations of current monitoring program**

The existing monitoring program is limited in its ability to monitor PCB flux to the river from numerous potential PCB sources. As noted previously, the program was designed to quantify the potential impact of the remnant deposits on Hudson River water column PCB concentrations. The sampling design included quantification of water column PCB concentrations both upstream and downstream of the remnant deposits (see section 1.1). However, the program was not designed to specifically characterize flux from other potential sources in this region of the river:

- Upstream of the Hudson Falls site
- Hudson Falls site
- Fort Edward outfall 004 area
- Remnant deposits
- Thompson Island pool
- Thompson Island Pool dredge disposal site.

As such, the existing monitoring does not include enough sampling stations to differentiate the potential impact of the individual source(s) present.

It is difficult to obtain a representative sample in the remnant deposit reach of the river. In this section of the river, shallow rapids limit accessibility except by boat. Additionally, the shoreline along the upstream sections of this reach consists of a 70 to 100 feet high bluff. When the 1991 PCB increase showed the presence of a PCB source(s) between the background station and the remnant deposits, it was desired to establish another sampling station to monitor the activity of the suspected source areas separately. Unfortunately, access difficulties precluded composite sampling at the center of the channel. Therefore, a shoreline station was established to collect a grab sample along the west shore, upstream of the remnant deposits at a location referred to as the Canoe Carry (HRM 196.8). Based on our understanding of the potential PCB sources to the Hudson River (as described above), the current river monitoring program could have a number of potential limitations, as described below.

Grab samples collected from the west shore at HRM 196.8, which is 0.1 mile downstream of the Bakers Falls source(s), may not accurately represent mass loading from that source(s). Due to the proximity of this sampling station to the Bakers Falls source(s) (HRM 196.9), incomplete lateral mixing of PCBs originating upstream of HRM 196.8 would produce high or low bias in PCB concentrations at this location, depending on where the sample was collected. This could produce the false observation of increased PCB concentrations through the remnant pool, as described above, since samples are routinely collected from the western shore at HRM 196.8.

PCB oil may be present in the river and moving below sampling devices. Based on current understanding of PCB sources near the Hudson Falls plant site, it is possible that PCBs are entering the river from this source in the form of DNAPL. Given PCB's low affinity for water, such a source would likely not immediately dissolve, and may settle and be incorporated as part of the bed load. Such a lack of vertical mixing of PCBs may contribute to the observed increases in PCB through the remnant deposits.

There is also concern that the sample collected at Thompson Island Dam may not be representative of overall water column PCB concentrations in this region of the river. Grab sampling data collected at the west shore of Thompson Island Dam (HRM 188.5) may have limitations similar to the remnant deposit region of the river for use in characterizing source(s) upstream from the Thompson Island Pool. The difficulty in routinely obtaining a depth integrated composite sample from the center of the channel increases the uncertainty associated with the data collected at the Thompson Island Dam sampling station.

### 1.5. Program objectives

The sampling described in this SAP will provide data to assess the adequacy of the current PCRDMP to meet the project objective of quantifying PCB flux from the remnant deposits. The results of this monitoring program will be used to facilitate redesign of the PCRDMP, if required. Corollary objectives include:

- Evaluate contributions of PCBs from Hudson Falls, Fort Edward 004 area deposit, and the remnant deposits.
- Confirm the existence of the anomalous Thompson Island Pool PCB loading.
- Evaluate the impact of the new Bakers Falls hydroelectric power plant on PCB loading and dynamics in that region of the river.

### 1.6. Approach

The Hudson River Research and Monitoring Program will provide data to quantify and characterize potential sources of PCBs upstream of Rogers Island, and evaluate the lateral and vertical mixing regimes in Reach 9 and within Thompson Island Pool.

Three events, consisting of water column sampling and analysis across transects that are perpendicular to river flow, will be conducted. The transects will be located downstream of suspected source areas, as discussed above. Several stations across each transect will be sampled for PCB and TSS.

After receiving approval from the New York State Department of Environmental Conservation (NYSDEC), rhodamine WT dye will be continuously added from a suspected PCB source area during the sampling events. Discrete water samples will be collected from the sampling stations located across each transect, and analyzed for dye concentration. The dye will be used to simulate the behavior of a continuous dissolved phase source and will be used to interpret the PCB data.

Two events will include dye releases to the Bakers Falls plunge pool, before and after hydropower construction is complete (early September and October, respectively). The other event (late September) will include dye release at outfall 004 before the hydropower construction is complete. The actual timing of the studies will be contingent on NYSDEC approval and the date that the new hydrostation becomes operational.

The feasibility of using dye-impregnated plastic resin particles to simulate DNAPL behavior is currently under investigation. If feasible, one transect sampling event will include the addition of resin particles at the Bakers Falls plunge pool to simulate dispersion of a DNAPL. Data on particle dispersion and transport collected as part of this study will be used to evaluate the plausibility of the hypothesis that PCB DNAPL is contributing to PCB bed load through Reach 9 and into Thompson Island Pool. Particles and dye will be discharged into the river from the same location. The transport and dispersion of the particles will be monitored and evaluated with that of the dye.

The routine PCRDMP sampling will be conducted on the same day that the transect study is performed to allow comparison of the representativeness of PCRDMP sampling methods in characterizing PCB mass loading.

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## **2. Methodology**

The transect studies referenced in the preceding sections will consist of dye injection, resin particle injection (if feasible), hydrographic profile construction, and TSS, PCB, dye and resin particle sampling and analysis. These studies will be conducted across transects perpendicular to river flow and downstream of suspected source areas (Figures 1 and 2).

### **2.1. Dye injection**

Transect studies will include the continuous release of Rhodamine WT dye over a 6-hour period to simulate the behavior of a dissolved phase PCB source, and to assess the lateral mixing regime in downstream portions of the river. Dye will be injected into the Bakers Falls plunge pool during the first and third events (before and after startup of the Bakers Falls hydroelectric facility). During the second event, dye will instead be injected into the river from the Fort Edward outfall 004 remnant area. Dye loading will be calculated based upon the analytical detection limit of Rhodamine WT dye (0.1 ug/L) and the instantaneous river flow on the day of sampling. A material safety data sheet for Rhodamine WT dye to be used in this investigation is presented in Appendix A.

### **2.2. Plastic resin injection**

If feasible, one transect study will include the injection of dye-impregnated plastic resin particles to the river. This activity would be conducted from a location along the east shore of Bakers Falls to simulate the behavior of a DNAPL source of PCB from this area. The plastic particles have a specific gravity consistent with that of Aroclor 1242 (1.37). A material safety data sheet for the fluorescent particles being considered for this investigation is provided in Appendix B.

### 2.3. Hydrologic profile construction

Before dye release and sampling begins, hydrologic profiles of each transect will be constructed. Water depths will be measured across the transects to generate a bathymetric profile. River velocities will be measured using a Marsh-McBirney Model 201 velocity meter. These data will be used to calculate PCB, dye, and resin particle mass loadings within the individual parcels along each transect (Figure 2).

### 2.4. Sample locations

The transect studies will consist of the collection and analysis of water samples from four transect sampling locations across the Hudson River (Figure 1):

- near HRM 196.8 (Canoe Carry)
- approximately 500 feet downstream of Ft. Edward outfall 004 (outfall 004)
- upstream of the northern end of Rogers Island (Rogers Island)
- upstream of Thompson Island Dam (Thompson Island Dam).

In addition, a 6-hour composite sample will be collected from the background station at the Route 27 bridge located adjacent to the old Fenimore Bridge in Hudson Falls.

At the transects, analyses will include PCBs, TSS, dye and, potentially, plastic resin particles, as described in Section 2.4. Samples will be collected at six stations along each transect location (Figure 1). The stations will be spaced to maximize the spatial resolution of sampling in suspected PCB mass loading parcels along each transect. At the background station, the sample will consist of a composite of depth-integrated composites collected over a 6-hour period. This background station sample will be analyzed for PCB and TSS.

*Transect PCB and TSS samples.* There will be six sampling stations along each transect. Two samples will be collected at each sampling station, one near the surface of the water column (approximately 0.2



times the total depth), and one near the bottom of the water column (approximately 0.8 times the total depth). Sample composites will be made up of aliquots collected hourly over the 6-hour sampling period. The aliquots collected near the surface at each station will form one composite sample, and the aliquots collected near the bottom at each station will form a separate composite sample.

To summarize, the PCB and TSS sample collection will include:

- four transects
- six stations per transect
- six surface composite samples at each station
- six bottom composite samples at each station
- total of twelve samples per transect.

*Dye and plastic resin samples.* Samples for dye and dye-impregnated plastic resin particle analysis will be collected in the same manner and at the same frequency as the PCB/TSS samples; however, the dye and particle samples will not be composited.

The dye and resin particle sampling will include:

- four transects
- six stations per transect
- six surface samples collected at each station
- six bottom samples collected at each station
- total of seventy-two samples per transect.

### 2.5. Sample collection procedures

Samples will be collected at each station (water depth permitting) with a Kemmerer Bottle sampler. The areas where water depths are too shallow to permit use of a Kemmerer bottle, a grab sample will be collected with a stainless steel bailer. Composites will be formed by discharging a portion of the contents of the Kemmerer Bottle directly into a sampling container. The sample containers will be pre-marked in approximately one-sixth increments to guide preparation of the composite. Dedicated sampling equipment will be used for each transect. Sampling equipment will not be decontaminated between stations due to the close proximity of the sampling stations, expected low-level concentrations of PCBs, and

field logistics. However, the sampling equipment will be rinsed several times with river water prior to sampling at each station.

*Sample collection at Canoe Carry transect.* The upper reaches of the study area are difficult to access. Samples will be collected by installing a guide rope across the river at a transect location and marking approximate sampling stations on the rope. The guide rope will be secured to trees or other immovable objects. The sampling stations will be accessed by a small boat, which will be tethered to the rope. The guide rope will be removed following completion of sampling. For confirmation purposes, a continuous detection field fluorometer will be used at this transect to monitor dye dispersion characteristics and adjust transect sampling stations, if required.

*Sample collection along the outfall 004, Rogers Island, and Thompson Island Dam transects.* These transects are located in areas with lower current velocities than that at the Canoe Carry. Therefore, sampling locations will be identified by anchoring buoys at the desired stations and will be accessed by boat.

The Rogers Island transect will be located upstream of the northern tip of Rogers Island and downstream of the former Fort Edward Dam location. Time of travel from Bakers Falls to Fort Edward is approximately 2 hours at summer low flow conditions of approximately 2,500 cfs. Sampling will be initiated at this station approximately 2 hours after dye injection.

A continuous detection field fluorometer will be used to detect the dye front and then initiate sampling along the Thompson Island Dam transect. Time of travel from Bakers Falls to Thompson Island Dam is approximately 12 hours at summer low flow conditions of approximately 2,500 cfs.

## 2.6. Sample handling

Upon collection, the samples will be placed in appropriate containers. PCB and TSS samples will be chilled to approximately four degrees C and dye samples will be stored in the dark. The PCB and TSS samples will be transported to Northeast Analytical, Inc. (NEA) for analysis. Each sample will be assigned a unique sample designation, identifying sample location, and date and time of sample collection. Samples will be identified by a two part designation system consisting of sample location and station identification. Dye/particle samples

will include a designation for sampling round (1 through 6). Sample location identification will include one of the following designations:

CC = Canoe Carry sample  
004 = 004 sample  
FED = Fort Edward sample  
TIP = Thompson Island Pool sample

Sample station descriptions will be labelled 1 through 6 from west to east (Figure 2). As an example of the sample identification scheme, sample CC2 would be the transect sample collected at the second station from the west shore at the Canoe Carry transect sampling location. Sample containers required for this program are summarized below.

Analysis	Bottles	
	Type	Size
PCBs	glass	1-liter
TSS	plastic	250-ml
Dye/particles	plastic	1.9-liter (1/2 gallon)

Chain of custody procedures to be utilized for the program are presented in the PCRDMP QAPP (O'Brien & Gere Engineers, Inc. 1992a).

One-liter water samples will be filtered using a 0.5 um filter. Samples will be shipped to University of Southern California for analysis of particle fluorescence.

## 2.7. Analytical testing program

### 2.7.1. PCBs and TSS

Analytical methods for PCBs and TSS will be the same as those used in the PCRDMP. PCBs will be analyzed by capillary column using method NEA608CAP and TSS will be analyzed by USEPA Method 160.2. Both types of analyses will be performed by NEA. Copies of the methods are provided in the PCRDMP QAPP (O'Brien & Gere

1992b). Standard laboratory packages will be required. Detailed data packages of PCB results, Contract Laboratory Program (CLP) type, will not be included. The laboratory will maintain support data should CLP type data packages be required for complete validation of data in the future.

#### 2.7.2. Dye

Dye samples will be analyzed in the field or in the laboratory following procedures provided in the standard operating procedure for the fluorometer provided in the addendum to the QAPP (Appendix C).

#### 2.7.3. Particles

Particle filter samples will be analyzed by the University of Southern California using special fluorometry equipment which measure the mass of fluorescent particles on each filter. If the fluorescent resin particle study is feasible, a methodology for particle analysis will be forwarded for review.

### 2.8. Quality assurance/quality control

Quality Assurance/Quality Control issues are addressed in the Quality Assurance Project Plan developed for the PCRDMP (O'Brien & Gere 1992b) and the addendum to the QAPP provided in Appendix C. For each round of sampling, QA/QC samples will consist of collection of matrix spike, blind duplicate, and equipment blank samples as specified in the table below.

Analysis	QA/QC Sample Frequency		
	MS	DUP	EQBL
PCBs	1/20	1/20	1/location
TSS	NA	1/20	NA
dye	NA	1/20	NA
particles	NA	1/20	NA

Note: NA = not applicable

### 2.9. Health and safety

Health and safety issues are addressed in the Health and Safety Plan developed for the PCRDMP (O'Brien & Gere 1992c) and the addendum to the health and safety plan provided in Appendix D.

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### **3. Data management**

#### **3.1. Data management**

O'Brien & Gere will utilize an electronic data management system for water column data collected during the 1995 Hudson River Project research program. The system will be used to store and transfer raw field and laboratory data generated by O'Brien & Gere and laboratory subcontractors. The data management system is a computer software program that can search, segregate, and summarize the data according to users needs. The data generated by this program will be organized into a compilation of laboratory and field generated data in both bound and electronic file format. Bound data files will be developed and will include field notes, chain of custody forms, and laboratory results.

#### **3.2. Evaluation**

The PCB concentration, flow velocity, and cross sectional area information collected during the transect studies will be used to calculate PCB mass transport for each of the twelve water parcels along each transect (Figure 2). Differences in the vertical and lateral PCB mass transport data between the different parcels will be used to assess the nature of PCB loading from upstream sources. Additionally, differences in total PCB mass transport between upstream and downstream transects will be used as a quantitative measure of PCB loading from the potential source(s) located between the two transects.

Dye and resin particle data will be used to assist in the interpretation of PCB mass transport and loading data. The dye simulates the behavior of a continuous dissolved phase source of PCB entering the river at one potential source area. The extent to which resin particles represent PCB DNAPL that may be entering the river is limited. Nonetheless, particle data will be used as a reference for the manner in which DNAPL PCB entering the river continuously from one potential source area may behave.

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#### **4. Project organization, schedule, and deliverables**

The project organization and schedule are presented in Figures 3 and 4, respectively. The transect sampling will be performed as three transect sampling events. The first and third sampling events will include dye addition to evaluate the Bakers Falls source before (first event) and after (third event) hydropower construction is complete. The second sampling event will include dye addition to evaluate the outfall 004 source before the hydropower construction is complete.

The data obtained by the Hudson River Research and Monitoring Program will be reported in a data summary report. The report will include tabulated results and field documentation.

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## Figures



**O'BRIEN & GERE**  
ENGINEERS, INC.

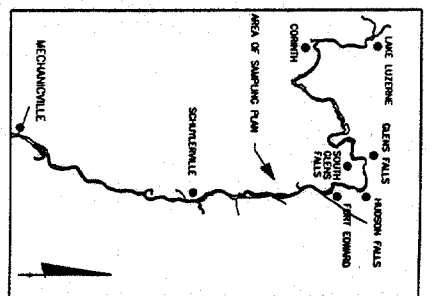


FIGURE 1

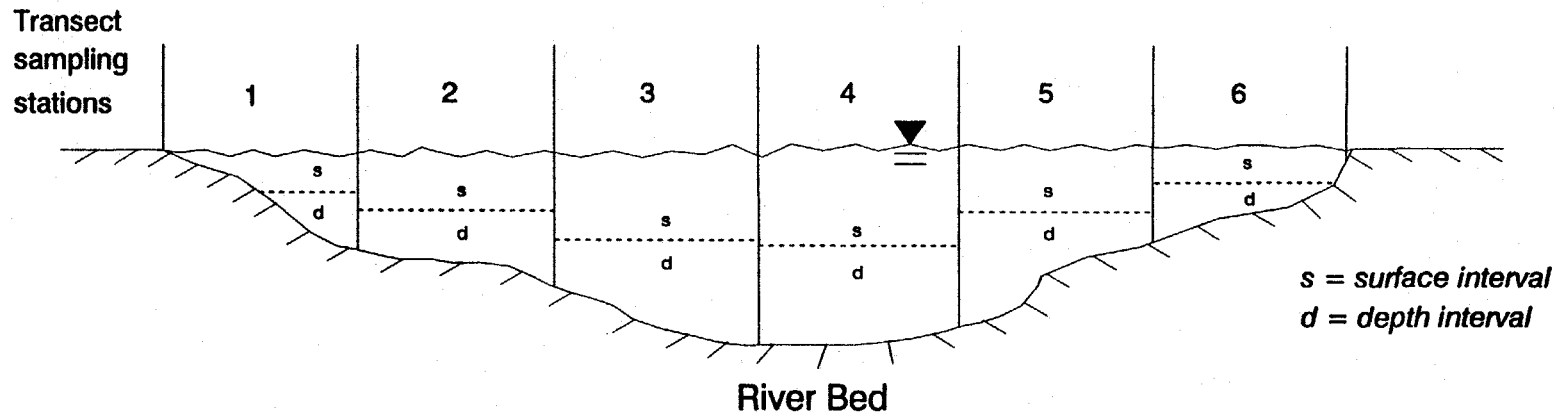
4000 0 4000  
APPROX. SCALE IN FEET



**D'O'BRIEN & GERE**  
ENGINEERS, INC.

- ◆ ROUTINE PCRDMP SAMPLING LOCATION
- TRANSECT SAMPLING LOCATION
- ✕ TRANSECT SAMPLING STATION
- ② REMNANT AREA
- (CC) SAMPLE LOCATION IDENTIFICATION

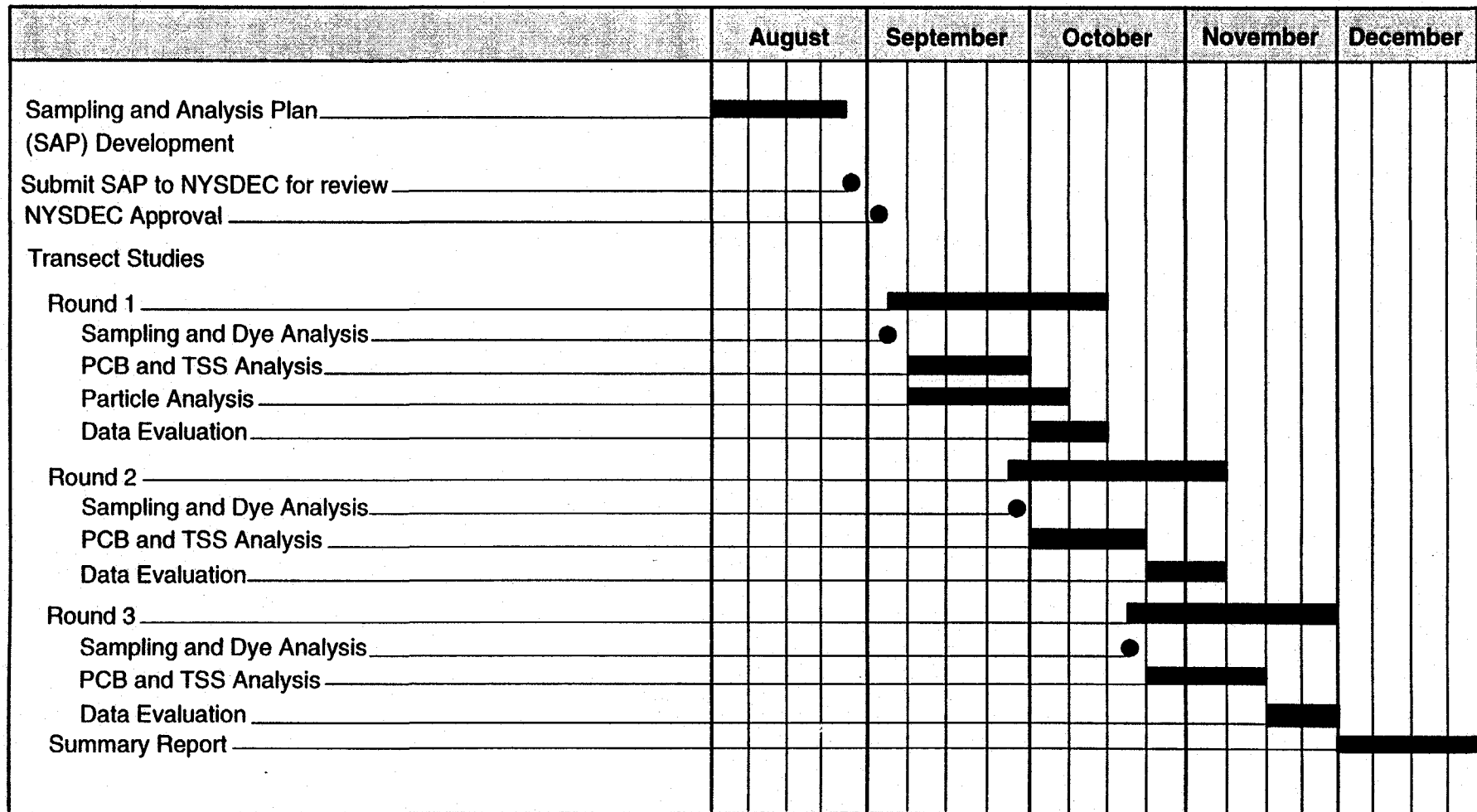
Figure 2  
Hudson River Project  
River Monitoring Test  
Transect Mass Loading Profile



c:\hudson\trnsprof.drw

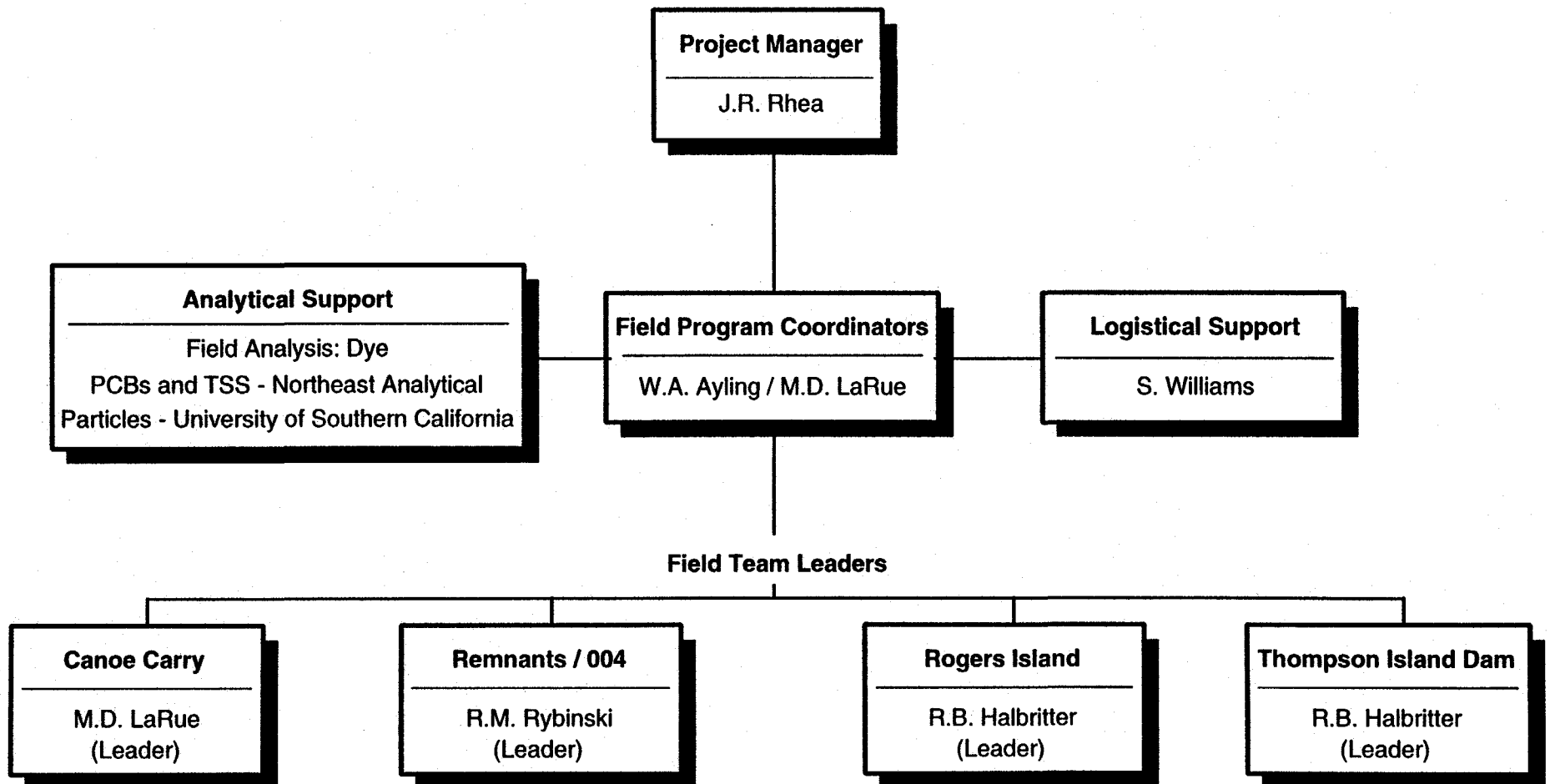
**Figure 3**  
**PROJECT SCHEDULE**

**Hudson River Project**  
**River Monitoring Test**



309053

**Figure 4**  
**PROJECT ORGANIZATION**  
**Hudson River Project**  
**River Monitoring Test**



## Appendices



**O'BRIEN & GERE**  
ENGINEERS, INC.

**APPENDIX A**

**Material Safety Data Sheet for Rhodamine WT Dye**



MATERIAL SAFETY DATA SHEET  
FWT RED 200 LIQUID  
PAGE 1 OF 4

-----  
MSDS PREPARATION INFORMATION  
-----

PREPARED BY: D. C. POTTER  
(513) 773-0600  
DATE PREPARED: 3/15/95

-----  
PRODUCT INFORMATION  
-----

MANUFACTURED BY: FORMULABS, INC.  
9676 NORTH LOONEY ROAD  
PIQUA, OHIO 45356  
(513) 773-0600 BUSINESS  
(800) 424-9300 CHEMTREC (24-HR EMERGENCY CONTACT)

CHEMICAL NAME . . . . . NOT APPLICABLE  
CHEMICAL FORMULA . . . . . NOT APPLICABLE  
CHEMICAL FAMILY . . . . . XANTHENE DYE

-----  
HAZARDOUS INGREDIENTS  
-----

HAZARDOUS INGREDIENT	%	T.L.V.	C.A.S. #
=====	=====	=====	=====
TRIMELLITIC ACID	~5.0	NONE	528-44-9

HAZARDOUS INGREDIENT	LD/50, SPECIES	LC/50, SPECIES
=====	=====	=====
TRIMELLITIC ACID		
ORAL (MOUSE)	2500 mg/kg	NOT AVAILABLE
DERMAL (RABBIT)	NOT AVAILABLE	NOT AVAILABLE

-----  
PHYSICAL DATA  
-----

PHYSICAL STATE . . . . . LIQUID  
APPEARANCE AND ODOR . . . . . DARK RED LIQUID WITH MILD ODOR  
SPECIFIC GRAVITY . . . . . ~ 1.15  
VAPOR PRESSURE (mm Hg @ 25 deg. C) . . . . . NOT DETERMINED  
VAPOR DENSITY (AIR = 1) . . . . . NOT DETERMINED  
EVAPORATION RATE (Butyl Acetate = 1) . . . . . NOT DETERMINED  
BOILING POINT . . . . . ~ 100 deg. C (212 deg. F)  
FREEZING POINT . . . . . ~ -10 deg. C (14 deg. F)  
PH . . . . . 10.4 - 10.8  
SOLUBILITY IN WATER . . . . . SOLUBLE

MATERIAL SAFETY DATA SHEET  
FWT RED 200 LIQUID  
PAGE 2 OF 4

-----  
FIRE OR EXPLOSION HAZARD  
-----

CONDITION OF FLAMMABILITY . . . . . NON-FLAMMABLE.  
MEANS OF EXTINCTION . . . . . WATERFOG, CARBON DIOXIDE, DRY  
CHEMICAL. WEAR SCBA.  
FLASH POINT AND METHOD . . . . . NOT APPLICABLE.  
UPPER FLAMMABLE LIMIT . . . . . NOT APPLICABLE.  
LOWER FLAMMABLE LIMIT . . . . . NOT APPLICABLE.  
AUTO-IGNITION TEMPERATURE . . . . . NOT APPLICABLE.  
HAZARDOUS COMBUSTION PRODUCTS . . . . . BURNING MAY PRODUCE OXIDES OF CARBON  
AND NITROGEN.  
UNUSUAL FIRE HAZARD . . . . . NOT APPLICABLE  
EXPLOSION DATA  
SENSITIVITY TO STATIC  
DISCHARGE . . . . . NOT APPLICABLE  
SENSITIVITY TO MECHANICAL  
IMPACT . . . . . NOT APPLICABLE

-----  
REACTIVITY DATA  
-----

PRODUCT STABILITY . . . . . STABLE  
PRODUCT INCOMPATIBILITY . . . . . DO NOT MIX WITH ACIDS  
CONDITIONS OF REACTIVITY . . . . . NOT APPLICABLE  
HAZARDOUS DECOMPOSITION PRODUCTS . . . . . SEE HAZARDOUS COMBUSTION  
PRODUCTS

-----  
TOXICOLOGICAL PROPERTIES  
-----

SYMPTOMS OF OVEREXPOSURE FOR EACH POTENTIAL ROUTE OF ENTRY:

INHALATION, ACUTE . . . . . TRIMELLITIC ACID MAY CAUSE IRRITATION.  
INHALATION, CHRONIC . . . . . NOT KNOWN.  
SKIN CONTACT . . . . . WILL TEMPORARILY COLOR THE SKIN.  
TRIMELLITIC ACID MAY CAUSE SKIN  
IRRITATION.

SKIN ABSORPTION . . . . . NO HARMFUL EFFECTS EXPECTED.  
EYE CONTACT . . . . . TRIMELLITIC ACID MAY CAUSE IRRITATION.  
INGESTION . . . . . NOT KNOWN.

EFFECTS OF ACUTE EXPOSURE . . . . . CONTACT WITH TRIMELLITIC ACID MAY CAUSE  
IRRITATION TO THE SKIN, EYES AND  
RESPIRATORY TRACT.

EFFECTS OF CHRONIC EXPOSURE . . . . . NOT KNOWN.

THRESHOLD LIMIT VALUE . . . . . NOT AVAILABLE.

CARCINOGENICITY . . . . . NOT LISTED BY IARC, NTP OR OSHA.

TERATOGENICITY . . . . .

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MATERIAL SAFETY DATA SHEET  
FWT RED 200 LIQUID  
PAGE 3 OF 4

MUTAGENICITY . . . . . THERE IS CONFLICTING EVIDENCE AS TO THE  
MUTAGENICITY OF THE DYE CONTAINED IN  
THIS PRODUCT.

TOXICOLOGICALLY SYNERGISTIC  
PRODUCTS . . . . . NONE KNOWN

PREVENTIVE MEASURES

PERSONAL PROTECTIVE EQUIPMENT:

GLOVES . . . . . RUBBER.  
RESPIRATORY . . . . . NONE REQUIRED UNDER NORMAL  
CONDITIONS.  
EYE PROTECTION. . . . . GOGGLES.  
CLOTHING . . . . . PROTECTIVE CLOTHING WHERE SKIN  
CONTACT IS UNAVOIDABLE.  
OTHER . . . . . HAVE ACCESS TO AN EYEWASH.  
ENGINEERING CONTROLS . . . . . LOCAL VENTILATION.  
SPILL OR LEAK RESPONSE . . . . . WEAR APPROPRIATE SAFETY EQUIPMENT.  
CONTAIN AND CLEAN UP SPILL  
IMMEDIATELY, PREVENT FROM ENTERING  
FLOOR DRAINS. CONTAIN LIQUIDS  
USING ABSORBENTS, SWEEP POWDERS  
CAREFULLY MINIMIZING DUSTING.  
SHOVEL ALL SPILL MATERIALS INTO  
DISPOSAL DRUM, FOLLOW DISPOSAL  
INSTRUCTIONS. SCRUB SPILL AREA  
WITH DETERGENT, FLUSH WITH COPIOUS  
AMOUNTS OF WATER.  
WASTE DISPOSAL . . . . . DISPOSE OF WASTE IN ACCORDANCE  
WITH FEDERAL, STATE AND LOCAL  
REGULATIONS.  
HANDLING PROCEDURES AND EQUIPMENT . . . . . NO SPECIAL REQUIREMENTS.  
STORAGE REQUIREMENTS . . . . . STORE AT ROOM TEMPERATURE BUT  
ABOVE THE FREEZING POINT OF WATER.  
SHIPPING INFORMATION . . . . . KEEP FROM FREEZING.

FIRST AID MEASURES

FIRST AID EMERGENCY PROCEDURES:

EYE CONTACT . . . . . FLUSH EYES WITH WATER FOR AT LEAST  
15 MINUTES. GET IMMEDIATE MEDICAL  
ATTENTION.

SKIN CONTACT

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MATERIAL SAFETY DATA SHEET  
FWT RED 200 LIQUID  
PAGE 4 OF 4

-----  
INHALATION . . . . . IF INHALED, MOVE TO FRESH AIR. IF  
BREATHING IS DIFFICULT, GIVE  
OXYGEN AND GET MEDICAL ATTENTION.

INGESTION . . . . . IF SWALLOWED, GIVE VICTIM WATER  
AND INDUCE VOMITING. GET  
IMMEDIATE MEDICAL ATTENTION.  
NEVER GIVE FLUIDS OR INDUCE  
VOMITING IF PATIENT IS UNCONSCIOUS  
OR HAS CONVULSIONS.

-----  
SPECIAL NOTICE  
-----

All information, recommendations, and suggestions appearing herein concerning this product are based upon data obtained from the manufacturer and/or recognized technical sources; however, Formulabs, Incorporated makes no warranty, representation or guarantee as to the accuracy, sufficiency or completeness of the material set forth herein. It is the user's responsibility to determine the safety, toxicity and suitability of his own use, handling, and disposal of the product.

Additional product literature may be available upon request. Since actual use by others is beyond our control, no warranty, express or implied, is made by Formulabs, Incorporated as to the effects of such use, the results to be obtained or the safety and toxicity of the product, nor does Formulabs, Incorporated assume any liability arising out of use by others of the product referred to herein. The data in the MSDS relates only to the specific material designated herein and does not relate to use in combination with any other material or in any process.

-----  
END OF MATERIAL SAFETY DATA SHEET  
-----

**APPENDIX B**

**Material Safety Data Sheet for Flourescent Resin Particles**

## SECTION I - PRODUCT IDENTIFICATION

Manufacturer: DAY-GLO COLOR CORP  
ENVIRONMENTAL HEALTH & SAFETY  
4515 ST CLAIR AVENUE  
CLEVELAND OH 44103

Information Phone: 216-391-7070  
Emergency Phone: 800-424-9300

Product Class: SYNTHETIC ORGANIC COLORANT  
Trade Name : SATURN YELLOW ZQ PIGMENT  
Product Code : ZQ-17N  
C.A.S. Number: MIXTURE  
Prepared By : SCOTT A. FLEMING  
Title : REGULATORY CHEMIST

Hazard Ratings: Health - 1  
! none -> extreme Fire - 1  
! 0 ---> 4 Reactivity - 0  
!  
!

## SECTION II - HAZARDOUS INGREDIENTS

Ingredients	CAS #	Weight %	Exposure Limits	VP
			ACGIH/TLV OSHA/PEL	mm HG

(No hazardous ingredients known at this time.)

## SECTION III - PHYSICAL DATA

Boiling Range: None  
Evap. Rate: Non Volatile  
Volatiles vol % 00.00 Wgt% 00.00  
Appearance: Colored powder  
V.O.C.: See Section IX

Vapor Density: Non Volatile  
Liquid Density: Heavier than Water.  
Wgt per gallon: 10.00 Pounds.

## SECTION IV - FIRE AND EXPLOSION HAZARD DATA

Flammability Class: NA Flash Point: None F LEL: None UEL: None

**-EXTINGUISHING MEDIA:**

Based on the NFPA guide, use dry chemical, water or other extinguishing agent suitable for Class A fires. For large fires, use water spray or fog, thoroughly drenching the burning material.

**-SPECIAL FIREFIGHTING PROCEDURES:**

Clear area of personnel. Approach upwind. Wear self-contained breathing apparatus.

**-UNUSUAL FIRE & EXPLOSION HAZARDS:**

Improper handling may lead to dust cloud formation which, as with any organic compound, may be an explosion hazard.

---

SECTION V - HEALTH HAZARD AND PERSONAL PROTECTION INFORMATION

---

**-FIRST AID:**

- EYES:** Flush with water for at least 15 min. while holding eyelids open.
- SKIN:** Practice good industrial hygiene, wash with soap and water.
- INGESTION:** Give water, do not induce vomiting. Call a physician.
- INHALATION:** Remove to fresh air. Treat symptoms. Call a physician.

**-TOXICOLOGY INFORMATION:**

No toxicity studies have been conducted on this product.

**-PRIMARY ROUTE(S) OF EXPOSURE:**

- EYE CONTACT:** May cause slight irritation
- SKIN CONTACT:** May cause slight irritation
- INHALATION:** Treat as a nuisance dust. Avoid breathing.

**-SYMPTOMS OF EXPOSURE:**

A review of available data does not identify any symptoms from exposure.

**-CHRONIC:**

- CARCINOGENICITY:** NTP? (N) IARC MONOGRAPHS? (N) OSHA REGULATED? (N)
- Long term exposure may result in dermatitis for sensitive individuals.

**-AGGRAVATION OF EXISTING CONDITIONS:**

Respiratory allergies and diseases may be aggravated in extreme exposures.

**-RESPIRATORY PROTECTION:**

A dust mask or NIOSH approved respirator with a dust filter.

**-VENTILATION:**

General ventilation for comfort conditioning is usually enough to maintain the dust within the nuisance limit of 5 mg/cu.m.

**-PROTECTIVE EQUIPMENT:**

- GLOVES:** Required only for sensitive individuals.
- EYE PROTECTION:** Glasses or goggles are recommended.
- RESPIRATORY PROTECTION:** Use a NIOSH approved dust respirator.

---

SECTION VI - REACTIVITY DATA

---

**STABILITY:** ☐ Unstable ☒ Stable

**HAZARDOUS POLYMERIZATION:** ☐ May occur ☒ Will not occur

**-INCOMPATIBILITY:**

Avoid contact with strong oxidizers (eg. chlorine, peroxides, chromate, nitric acid, perchlorates, concentrated oxygen, permanganates) which can generate heat, fires, explosions and the release of toxic fumes.

---

SECTION VI - REACTIVITY DATA (cont.)

---

**-CONDITIONS TO AVOID:**

Avoid excessive dust in vicinity of electrical or other spark generating equipment. Avoid extreme heat.

**-HAZARDOUS DECOMPOSITION PRODUCTS:**

The fumes and smoke released contain oxides of carbon and nitrogen which are highly toxic. Do not breath smoke or fumes. Wear suitable protective equipment.

---

SECTION VII - SPILL OR LEAK PROCEDURES

---

**-SPILL CONTAINMENT AND RECOVERY:**

This product is not defined as a hazardous waste under EPA 40 CFR 261. Sweep up & dispose of as any dust or dirt.

**-DISPOSAL:**

Same as above.

---

SECTION VIII - REGULATORY INFORMATION

---

**-FEDERAL REGULATIONS:**

OSHA HAZARD COMMUNICATION RULE, 29 CFR 1910.1200: See Section II for hazardous ingredients as defined.

**-CERCLA/SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT (TITLE III)**

This is not a regulated material under 40 CFR 117, 302. Notification of spills not required.

**-SECTIONS 311 AND 312 - MATERIAL SAFETY DATA SHEET REQUIREMENTS:**

Our hazard evaluation has found this product to be non-hazardous.

**-SECTION 313 - LIST OF TOXIC CHEMICALS (40 CFR 372):**

See Section X.

**-TOXIC SUBSTANCES CONTROL ACT (TSCA):**

All components in this product are listed, or are excluded from listing, on the U.S. Toxic Substances Control Act (TSCA) 8(b) Inventory.

**-FEDERAL WATER POLLUTION CONTROL ACT, CLEAN WATER ACT, 40 CFR 401.15:**

This product contains no ingredients regulated by this Act.

**-CLEAN AIR ACT, 40 CFR 60, SECTION 111, 40 CFR 61, SECTION 112:**

This product contains no ingredients regulated by this Act.

**-STATE REGULATIONS:**

MICHIGAN CRITICAL MATERIALS: This product does not contain ingredients listed on the Michigan Critical Register.

**-CONEG-COALITION OF NORTHEAST GOVERNORS:**

This product is in compliance with the CONEG (Conference of Northeast Governors) requirements thru 1/1/1994 (ie total cadmium, chromium, lead and mercury less than 100 ppm). The detection limits of the test method used (in ppm) indicated by < and also the analytical test results for the pigment are as follows:

ANTIMONY (Sb)	<4	ARSENIC (As)	<4
BARIUM (Ba)	<0.50	CADMIUM (Cd)	<0.25
CHROMIUM (Cr)	<0.50	COPPER (Cu)	1.3
LEAD (Pb)	<1.0	MERCURY (Hg)	<0.05

(cont.)



-----  
SECTION VIII - REGULATORY INFORMATION (cont.)  
-----

## -CONEC-COALITION OF NORTHEAST GOVERNORS: (cont.)

NICKEL (Ni) &lt;0.75

SELENIUM (Se) &lt;4

SILVER (Ag) &lt;0.50

ZINC (Zn) 38,500

In other words Zinc and Copper were the only element found in our pigments.

## -TRANSPORTATION-49 CFR 172-101:

This product is not regulated by DOT.

## -FDA-21 CFR:

DAY-GLO Color Corp.'s products are not listed by the FDA for use under 21 CFR, since potential applications are so numerous that specific applications must be submitted to the FDA for inclusion in the 21 CFR FDA listing.

## -CLEAN AIR ACT AMENDMENTS OF 1990

No DAY-GLO product contains an ozone depleting substance (ODS) nor are any of our products manufactured with them.

-----  
SECTION IX - PRECAUTIONARY & LABEL INFORMATION  
-----

## -HMIS LABEL STATEMENT:

## ZQ-17N SATURN YELLOW PIGMENT

HEALTH - 1      FLAMMABILITY - 1      REACTIVITY - 0

PRECAUTIONS: Can cause respiratory irritation. Avoid breathing dust. Use & store with adequate ventilation. Dust explosion hazard with ignition source.

FIRST AID: EYES: Flush with water for 15 minutes. SKIN: Wash with soap and water. INGESTION: Give water, do not induce vomiting. Call a physician.

FIRE FIGHTING USE: Water spray, dry chemical, foam or CO2 (Toxic fumes emitted on burning).

SPILL CONTROL: Sweep up & dispose according to local, state and federal regulations.

## CONTAINS:

RESIN

ALBERTA YELLOW

C&gt;14 ALCOHOL

## CAS NO. OR NJ TSN:

80100023-5027-P

80100023-5004-P

71750-71-5

TARGET ORGANS: NO ORGANS AFFECTED.

COATING V.O.C. : NONE

MATERIAL V.O.C.: NONE

## -OTHER PRECAUTIONS:

None

-----  
SECTION X - ADDITIONAL REGULATORY INFORMATION  
-----

## -SARA TITLE III SECTION 313:

This product contains the following toxic chemicals subject to the reporting requirements of section 313 of the Emergency Planning and Community Right To Know Act of 1986 and of 40 CFR 372:

CAS#	Chemical Name	Percent by Weight
-----	-----	-----
	None	

## -PROP 65 (CARCINOGEN):

WARNING: This product contains a chemical known to the state of California to cause cancer.

CAS#	Chemical Name
-----	-----
	None

## -PROP 65 (TERATOGENIC):

WARNING: This product contains a chemical known to the state of California to cause birth defects or other reproductive harm.

CAS#	Chemical Name
-----	-----
	None

## -PROP 65 (BOTH CARCINOGEN AND TERATOGENIC):

WARNING: This product may contain a chemical known to the state of California to cause cancer or birth defects or other reproductive harm

CAS#	Chemical Name
-----	-----
	None

Page: 6

DAY-GLO COLOR CORP

Material Safety Data Sheet for: SATURN YELLOW ZQ PIGMENT(ZQ-17N)

-----  
SECTION X - ADDITIONAL REGULATORY INFORMATION (cont.)  
-----

**-DISCLAIMER:**

The information contained herein is believed to be accurate, but is not warranted. Nothing contained herein constitutes a specification nor is it intended to warrant suitability for the intended use.

## **APPENDIX C**

### **Addendum to the Quality Assurance Project Plan for the Post-Construction Remnant Deposit Monitoring Program**

**GENERAL ELECTRIC COMPANY  
1995 HUDSON RIVER PROJECT  
PCRDMP QUALITY ASSURANCE PROJECT PLAN  
ADDENDUM**

**Introduction**

This Quality Assurance Project Plan (QAPP) addendum has been prepared to describe quality assurance/quality control (QA/QC) procedures to be used by O'Brien & Gere employees while conducting field activities on the Hudson River during 1995 research investigations. The 1995 research program will follow the QAPP developed for the Post-Construction Remnant Deposit Monitoring Program (PCRDMP). However, some of the research activities are not explicitly included in the PCRDMP Field Sampling Plan (FSP; O'Brien & Gere 1992a) or the QAPP (O'Brien & Gere 1992b) developed for the PCRDMP. This QAPP addendum specifically addresses QA/QC procedures to be followed while performing research investigations in 1995 which consist of dye studies and transect investigations as described in the Field Sampling Plan (O'Brien & Gere 1995). This QAPP addendum is not intended as a stand alone document, rather it must be used in conjunction with the QAPP developed for the PCRDMP (O'Brien & Gere 1992).

**1. Project description**

This QAPP addendum provides the quality assurance/quality control (QA/QC) criteria for work efforts associated with surface water sampling and related analytical tasks outlined in the sampling and analysis plan developed by O'Brien & Gere Engineers, Inc. for the 1995 Hudson River monitoring program. The principle objective of this sampling and analysis program is to investigate mass loading of PCBs in the region of the river between Hudson Falls and Thompson Island dam. The program is designed to address sampling concerns identified by the PCRDMP. Results of this investigation will be used to recommend future PCRDMP sampling needs. Additional site background details are presented elsewhere (O'Brien & Gere 1994).

**2. Project organization and responsibility**

<u>Project Officer</u>	J. Kevin Farmer, P.E.
<u>Project Manager</u>	J Rhea, Ph.D.
<u>Field Program Coordinator</u>	W. Ayling
<u>Quality Assurance Officer</u>	M. Caputo
<u>Data Validator</u>	J. Smith
<u>Site Environmental Technicians</u>	no changes

## 2. Project organization and responsibility (continued)

### Laboratory Quality Assurance Coordinators

Northeast Analytical Inc.	W. Kotas
University of Southern California	B. Jones

### Laboratory Sample Custodians

Northeast Analytical Inc.	M. Kaskel
University of Southern California	B. Jones

## 3. Data quality objectives

The analyses and reporting level required in this program are summarized below.

Analysis	Analytical Method	Analytical Reporting Level
PCBs	NEA608CAP	III
TSS	USEPA 160.2	III
dye	fluorometer	II
fluorescent resin particles	fluorometer	II

## 4. Sampling procedures

Sampling locations are presented in Section 2 of the sampling and analysis plan.

Decontamination of the equipment will be as follows:

- 1) rinse equipment with tap water and scrub with brush to remove particulates which may be present
- 2) rinse with acetone
- 3) rinse with hexane
- 4) air dry
- 5) rinse with distilled water

## 5. Sample custody

No changes.

## 6. Calibration and frequency

No changes for PCB and TSS analyses. Dye analysis calibration will be performed according to the instrument manual provided as an attachment to this addendum.

## 7. Analytical Procedures

Analytical procedures for capillary column analysis of samples for PCBs, and TSS are presented in the QAPP. Analytical procedures for dye analysis are presented in the instrument manual provided as attachment to this addendum.

## 8. Data reduction, validation, and reporting

Northeast Analytical will perform PCB and TSS analyses. Dye samples will be analyzed by field technicians and fluorescent resin particle analyses will be performed by the University of Southern California.

PCB analyses will be reported using standard laboratory reporting methods. A data validatable package will not be provided by the laboratory, but will be maintained by the laboratory should validation be required at some future time.

## 9. Quality control checks

For dye samples collected during Phase 1 sampling, field duplicates will be included at a frequency of 1/20 as quality control samples.

Quality control samples will be collected for each round of Phase 2 sampling as summarized below.

Parameter	Samples/ transect	Samples/ round	Matrix spike	Field duplicate	Equipment blank
PCBs	12	48	3	3	3
TSS	12	48	NA	3	NA
dye	72	288	NA	15	NA
fluorescent resin particles	72	288	NA	15	NA

Notes: NA = not applicable

## 10. Performance and systems audits

No changes.

**11. Preventive maintenance**

No changes.

**12. Data assessment procedures**

No changes.

**13. Corrective action**

No changes.

**14. Quality assurance reports to management**

No changes.



OPERATING AND SERVICE MANUAL

Model 111 Fluorometer

Printed Circuit Type\*

\*Some units between Serial No. 1398 and 1763. All units above Serial No. 1763. Identified by the letters "P.C." following the serial no. on the rear screen.

G. K. TURNER Associates  
2524 Pulgas Avenue  
Palo Alto, California 94303

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makers of medical and technical instruments



2524 PULGAS AVENUE • PALO ALTO, CALIFORNIA

## INTRODUCTION

The Turner Model 111 Fluorometer has been created to add the dimension of time or position to fluorescent measurements. With this instrument and its accessories you will be able to record fluorescence or turbidity on any sample. Applications include:

1. Individual samples
2. Automated chemistry samples
3. Scanning of chromatograms
4. Scanning of electrophoresis strips
5. Scanning of thin layer chromatographic plates
6. Continuous monitoring of chromatogram column effluents
7. Tracing water flow

A complete Price List is included, which describes the many accessories presently available.

We are developing new techniques and methods, enlarging our file of fluorescent methods, and developing new accessories. Upon receipt of the registration card fastened to the back of the instrument, we will place you on our mailing list for new information and procedures. If, after reading our manual and going over your analysis problems, you have questions on possible techniques or accessories, we will be pleased to send you information from our extensive file.

Sincerely,

G. K. TURNER ASSOCIATES

A handwritten signature in cursive script that reads 'George K. Turner'.

George K. Turner, President

## I. FLUOROMETRIC TECHNIQUES

### A. GENERAL

The popular concept of fluorescence is a visible luminescence arising from the action of ultraviolet light. In practice, fluorescence due to the action of visible light is very common; it is simply difficult for the eye to sort out the fluorescent color without the aid of optical filters. Fundamentally, fluorescence is the emission of radiation from a molecule or atom following absorption of radiation. Of particular interest is fluorescence arising from the electronic absorption spectrum (100 m $\mu$  to 800 m $\mu$ ). In this range the absorption of a photon of light raises one electron of the molecule to a higher energy state. The return to ground state is accompanied by the emission of a photon. In solution there is generally a loss of energy before the return so that the emitted photon has less energy (is of longer wavelength) than the absorbed photon.

From an analytical standpoint, the intensity of fluorescent light emitted by a sample under constant input light intensity is directly proportional to the concentration of the fluorescent compound. An illuminating discussion on why fluorometric analysis techniques are so sensitive and specific will be found in a reprint appended to this manual: Fluorescence Analysis by George K. Turner, Med. Elect. News, 3, No. 2, 16-17 (Jan. 1963). The early chapters of Fluorescence Assay in Biology and Medicine, S. Udenfriend, Academic Press (1962) discuss theory and instrumentation and the later chapters treat methodology.

### B. BASIC METHODS

#### DIRECT

The compound to be analyzed for is itself fluorescent. The fluorescent compound is separated by chemical and/or physical means from other fluorescent materials present. Its fluorescence is a direct measure of its concentration. A typical example is Quinidine in Plasma, Turner Manual of Fluorometric Clinical Procedures.

#### CONVERSION

The compound to be analyzed for is converted into a fluorescent compound quantitatively. The resultant fluorescence is a direct measure of its concentration. A typical example is Catecholamines in Urine, Turner Manual of Fluorometric Clinical Procedures.

#### COUPLING

The compound to be analyzed for forms a fluorescent complex. The resultant fluorescence is a direct measure of its concentration. A typical example is Serum Calcium, or Magnesium, Turner Manual of Fluorometric Clinical Procedures.

#### DESTRUCTION

The compound to be analyzed for can destroy the fluorescence of another compound in a quantitative manner. The reduction in fluorescence is proportional to the amount of the unknown present. A typical example is "Determination of Parts per Billion Iron by Fluorescence Extinction," Block, J., and Morgan, E., Anal. Chem., 34, 1647-1650 (1962).

### C. SOME SUGGESTIONS

Our experience has been that considering the remarkable sensitivity of fluorescent assay, very little trouble will be encountered, especially in the normal working range which is 10 to 100-fold more dilute than spectrophotometric (absorption) methods. The sensitivity of fluorescence, however, is capable of almost indefinite extension and it is possible in many instances to determine quantities of a nanogram (0.001  $\mu$ g) or less. For the privilege of analyzing such minute samples one must pay a penalty, and that penalty is care and cleanliness.

One of the most common limits on sensitivity is set by blank fluorescence, that fluorescence arising from the reagents or other sources separate from the compound of interest. The guiding rule should be to have fluorescence of the "blank" small, or at least very consistent, compared to the fluorescence of the material being analyzed. This may be checked conveniently using Method II, Section IV of this manual.

## REAGENTS

Generally speaking, little difficulty will be encountered with commercially available reagent-grade chemicals. This is particularly true in the clinical laboratory where the majority of assays are done with visible light activating the fluorescence of the sample. Remember, that in order to fluoresce, a compound must absorb the incident light. You can prove this to yourself. Put in the general purpose filters (7-60 and 2A) and compare the fluorescence of tap-water and distilled water using Method II, Section IV. You will probably find a whopping difference. Now put in the filters for cholesterol assay (see the Manual of Fluorometric Clinical Procedures accompanying the instrument). You will probably not be able to distinguish the two as both will be nearly zero even at maximum sensitivity as will even the quinine solution of the Trial Kit.

It is under ultraviolet activation at high instrument sensitivity that high "reagent blank" fluorescence will probably be encountered. Even spectro-quality solvents sometimes contain traces of impurities fluorescent under ultraviolet light. Simple distillation in all-glass apparatus frequently remedies the problem. There are also several companies marketing fluorescent-grade reagents for those who must work at very low concentration in the ultraviolet region.

## STORAGE AND HANDLING

A frequent source of contaminants fluorescent under ultraviolet light are the plasticizers in some plastics and rubber. For dispensing distilled water, an all polyethylene or glass system is recommended. A short piece of rubber tubing on the end (for clamping) is all right if the water is allowed to run for a few seconds before use. Water standing in rubber may develop quite high fluorescence with the general purpose or shorter wavelength filters.

Polyethylene or borosilicate glassware is recommended for general storage of reagents. Test tubes or flasks may frequently be capped with polyethylene, Saran Wrap,\* or Parafilm\*\* for mixing and storage.

It is recommended that the suitability of any container material be checked. Solvents can even pick up fluorescence from scrupulously cleaned and rinsed borosilicate glassware. Though this is negligible for most work, it may be necessary to condition glassware for analyses of very low levels of materials where the general purpose filters or quartz U. V. excitation are used.

One thing which must not be forgotten is that at ultimate fluorescence sensitivity, the concentration of unknown involved is sometimes so low that it could disappear as a monolayer adsorbed on the walls of the vessel. Standards should generally be stored as concentrated solutions.

## CLEANLINESS

The degree of cleanliness required will depend on the wavelengths employed. Generally, in the clinical laboratory, no special precautions other than normal good laboratory technique will be required. In water tracing work using rhodamine B one may dabble ones hands in the sample if desired, since, though the instrument will detect 0.1 ppb of the dye, it can scarcely distinguish between distilled water and sewage.

In analyzing for traces of NADH (DPNH) or other materials where U. V. activation is used, it is necessary to observe a scrupulous level of cleanliness. At top instrument sensitivity, tipping the cuvette against a clean finger may raise fluorescence ten or more divisions. Household detergents contain "brighteners" -- violently fluorescent materials. used to make clothes look whiter under the ultraviolet component of sunlight. Laboratory detergents do not usually contain brighteners, but thorough rinsing is still required.

## PHOTOSENSITIVITY

Many materials are destroyed by the light they absorb. This is the main reason that the Turner Fluorometer uses such low light levels and resorts to such extreme photomultiplier sensitivity.

## DILUTION

Fluorescence is sensitive to pH and in some cases, concentration of ions other than the one of interest. Also, in some cases, the fluorescence of the reagent blank will be appreciable. For this reason, unless careful checks are made, dilution must always be made with reagent solution, not distilled water or other simple solvent.

\*Saran Wrap -- Trademark, Dow Chemical Company

\*\*Parafilm -- Trademark, American Can Company

## TEMPERATURE

Fluorescence is commonly more temperature sensitive than absorption. Although not all fluorescent materials exhibit marked sensitivity, there are instances where there is as much as a 2% reduction in fluorescence for every degree centigrade rise in temperature. The Turner Fluorometer is unique in its low sample compartment temperature rise, and consequent low rate of change of sample temperature. Standards, reagent blanks and samples should, of course, be at the same temperature before readings are started.

Experience has shown that even highly temperature-sensitive materials may be left in place for 1/2 minute or longer before drift occurs. This is adequate time to take readings on several ranges.

For kinetic studies where controlled and constant temperatures are required over extended periods of time, we recommend use of the Temperature Stabilized Door #110-655. See Price List for details.

## DELAYED FLUORESCENCE

A very few materials require a noticeable length of time to develop full fluorescence. This is presumably due to the presence of some phosphorescence, which is common in solids but unusual in solution except at very low temperature.

D. FILTER SELECTION - See pages 29-33.

E. CONCENTRATION RANGE

In colorimetry (absorptiometry) it is necessary, for accuracy, to operate in a narrow range of sample concentrations, preferably between 30% and 70% transmission (density 0.52 - 0.15). No such restriction exists in fluorometry. One merely adjusts the internal range selector to allow measurement of any concentration range desired. Very dilute solutions (even having 99.999% transmission) may be measured with the same accuracy as more concentrated solutions. The lower limit of concentration which can be measured is usually set by the fluorescence of the "blank". The upper limit is set by absorption of the exciting light, with consequent deviation from linearity. These limits are usually several orders of magnitude apart. The first time through a calibration curve will readily tell you if you are in the linear range.

In some cases it may be necessary to work at higher concentrations than covered by the internal range multiplier. A neutral density filter, such as #110-823 (1%) may be installed in addition to the regular secondary filter to further reduce sensitivity. A 6 to 12-fold increase in sensitivity may be obtained by installing the #110-865 High Sensitivity Conversion Kit, or the #110-655 Temperature Stabilized Door.

## II. PUTTING YOUR FLUOROMETER INTO OPERATION

### A. UNPACKING

Remove the "Trial Kit," just in front of the instrument, and the small test tube rack which holds it in place.

Using the handhold directly beneath the "Trial Kit," pull the entire instrument and its packing forward, until it is clear of the shipping carton.

The top of the instrument is protected by a piece of cardboard, with two foam pads glued to it. Fold these two pads out, and lift off. Place on a table top, foam up. Remove the following items from cut-outs in the right foam pad:

1. Spare general-purpose lamp, #110-850
2. General purpose Primary Filter, color spec. 7-60, #110-811
3. General purpose Secondary Filter, color spec. 2A, #110-816

Remove the dust cover from the top of the instrument.

Lift the instrument straight up, and place on your laboratory bench. Be sure that nothing is under it which will interfere with the small cooling fan.

Install the Interchangeable sample holding door which is appropriate to your application. See the Price List at the end of this manual for details of the many sample holding doors which are available. These interchangeable doors are installed by:

1. Holding the door with the outer surface (Green) to your left, and the latch towards you.
2. Engaging the hinge pins at the rear of the door with the hinge sockets, at the extreme left of the instrument.
3. Closing the door.

### B. INSTALLING FILTERS

Open the sample holder door by depressing the latch on the right side of the door knob. With the sample holder door open, you will see a vee-shaped compartment in the instrument. This is the sample compartment. The filter holders consist of a small bracket and spring on each side of the sample compartment. The primary filter is the almost black piece of glass marked 7-60. It should be slid into place diagonally from your right until it hits the filter stop in such a way that it rests on the right filter holder bracket, and is retained by the spring. The inscription 7-60 should be towards the right front of the instrument, and down.

The secondary filter is the slightly yellowish piece of glass marked 2A. It should be slid into place diagonally from your left until it hits the filter stop in such a way that it rests on the left filter holder bracket and is retained by the spring. The inscription 2A should be towards the center of the instrument and down.

Install other filter combinations in a similar manner; primary filter (or filters) on the right filter holder bracket, secondary filter (or filters) on the left filter holder bracket. If you have not done so already, we strongly recommend that you purchase our #110-838 FILTER KIT, containing our seventeen most widely used filters. See our Price List for details.

### C. CONTROLS

1. **POWER switch (front panel).**  
This switch turns power on and off to the fluorometer, and all accessories plugged into the RECORD and SAMPLE outlets on the rear of the instrument. On is up. Off is down.
2. **START switch (front panel).**  
After the POWER switch is turned on, it is still necessary to actuate a momentary circuit to start the ultraviolet lamp. The START switch actuates the lamp start circuit when held in the up position. It will return to its normal position when released.

Note: This switch should always be held in the "up" position for three or four seconds, before it is released.

3. **SAMPLE switch (front panel).**  
This switch turns power on and off to any accessories plugged into the SAMPLE power outlet (rear panel) when the POWER switch is on. On is up. Off is down.

4. RECORD switch (front panel).  
This switch turns power on and off to any accessories plugged into the RECORD power outlet (rear panel), when the POWER switch is on. On is up. Off is down.
5. BLANK knob (near FLUORESCENCE dial).  
This knob controls the amount of light introduced in the rear light path to compensate for the fluorescence of reagents (blank). See Section IV, DATA REDUCTION NOTES, part B, for details.

To operate, turn the lever actuated lock counterclockwise and turn the BLANK knob as required. After adjustment, turn the lever actuated lock clockwise.

6. RECORDER terminals (rear panel).  
Three terminals are provided for connection to the measuring circuit of your recorder. See Section VI, SERVICE INFORMATION, under the RECORDER COUPLING CIRCUIT, for details.
7. SPAN control (rear panel).  
This control is provided so that you may make the reading of your recorder agree with the reading on the FLUORESCENCE dial.

Adjustment of the SPAN control may be carried out as follows:

- a. Turn the Fluorometer on by following the OPERATING INSTRUCTIONS, Section III, below.
  - b. Install a fluorescence blank and turn the ZERO knob until the FLUORESCENCE dial reads zero.
  - c. Install a fluorescent solution, choosing instrument range and solution concentration to yield a reading of about 80 on the FLUORESCENCE dial.
  - d. Install the fluorescence blank and reset the ZERO knob until the FLUORESCENCE dial again reads zero.
  - e. Adjust the zero control on your recorder until the recorder reads zero. (On the Turner #111-801 recorder, this is a screwdriver adjustment under a round, removable button on the front panel of the recorder.)
  - f. Install the fluorescent material. Adjust the SPAN control until the recorder reads the same as the FLUORESCENCE dial.
  - g. Repeat steps d, e and f until both conditions are met, without further adjustment.
8. RANGE SELECTOR (may be seen by opening the sample holder door, and locating the knob just under the primary filter bracket).  
To select the desired range, pull the RANGE SELECTOR knob out slightly and move it until it is over the desired range. Release it and move it slightly from side to side until it drops into place. The variety of cuvettes which may be used with your instrument preclude its accurate calibration. The numbers 3X, 10X and 30X indicate the approximate increase in sensitivity which is obtained over the least sensitive or 1X position.

With the primary filter removed, you may observe the various apertures, introduced by the RANGE SELECTOR.

9. REG. and H.V. controls (rear panel).  
These controls are factory set and will not require adjustment. Their purpose is covered in section VI., SERVICE INFORMATION.

#### D. POWER

Plug the power cord into any convenient 115 volt 60 cycle per second power source (Model 111-000), or 50 cycles (Model 111-050). Note that your instrument is equipped with a three-prong plug meeting the latest Underwriters' Laboratory specification. An adapter with a grounding wire for use with the older type two-pin wall sockets is supplied for your convenience. We recommend that this wire be grounded, for your protection.

For applications in the field, battery powered inverters or portable generators yielding a sinusoidal output waveform may be used. Inductive switching transients must be avoided. Please request the latest information from the factory.



#### E. ACCESSORY POWER OUTLETS

Your fluorometer is equipped with two separately controlled rear panel accessory power outlets. Power is removed from both of these outlets when the POWER switch is in the down position. When the POWER switch is in the up or on position, the RECORD and SAMPLE switches control power to the RECORD and SAMPLE outlets respectively. These circuits are not fused. (They should not be used for highly inductive loads, unless suppressors are added.) They are rated at five amperes each.

We recommend that a suitable grounding wire and a three-prong power plug meeting the latest Underwriters' Laboratory specifications be used with all accessories.

### III. OPERATING INSTRUCTIONS

#### HOW TO TURN THE FLUOROMETER ON.

1. Turn the switches marked "SAMPLE," "RECORD," AND "POWER" to the off (down) position.
2. Plug power cord into receptacle.
3. Move the POWER switch to the up or on position.
4. Hold the START switch in the up or on position for three or four seconds. Release.
5. Open the sample holder door.
6. Check that the U.V. source is on by observing through the door latch opening.
- \*7. After about 30 seconds warm-up, the FLUORESCENCE dial will wander. This is normal. The servo system which drives the FLUORESCENT dial is inoperative until the sample holder door is closed. After two minutes warm-up the instrument is ready for operation.
8. To turn on the recorder, turn the front panel switch marked RECORD to the on (up) position.
9. To actuate electrically driven or controlled sample systems, turn the front panel switch marked SAMPLE to the on (up) position.

#### HOW TO OPERATE THE FLUOROMETER

Refer to the instruction for the specific sample holder door used. In general there are five basic steps:

1. Check the primary filters, secondary filters, range selector, sample holder and cuvette against your data sheet for the particular measurement being made.
2. Introduce the reagent blank.
- \*\*3. Release the lock on the BLANK knob by moving counter-clockwise. Turn the BLANK knob until the FLUORESCENCE dial (or recorder) reads zero. The FLUORESCENCE dial will move in the opposite direction from the direction that you turn the BLANK knob. Tighten the lock on the BLANK knob.
- \*\*\*4. Introduce the standard. After the FLUORESCENCE dial (or recorder) has ceased moving, note its reading.
- \*\*5. Introduce the unknown. After the FLUORESCENCE dial (or recorder) has ceased moving, note its reading.

#### HOW TO TURN THE FLUOROMETER OFF.

Turn the POWER switch to off (down).

#### \*\*\*HOW TO REDUCE THE DATA.

1. Divide the concentration of the known sample by the reading obtained on the known sample.
2. Multiply by the reading obtained on the unknown sample. This yields the concentration of the unknown.

---

\*When your fluorometer has been subjected to heavy vibration, as it might receive in shipment, let it warm up 15 minutes before making critical measurements. Once the mercury in the ultraviolet lamp has redistributed itself, 2-minute warm-up will suffice.

For activation with the visible mercury lines, 405 m $\mu$ , and 436 m $\mu$ , and 546 m $\mu$ , a longer warm-up is recommended. The instrument may be used at these wavelengths essentially immediately but frequent standardization is desirable for about 45 minutes, after which time results should stabilize and compare with previous days' readings.

\*\*After reaching its final position the FLUORESCENCE dial (or recorder) will continue to move in a random manner, approximately  $\pm 1/4$  dial division.

\*At the extremely low concentrations where fluorescent techniques are used, we always recommend standards, as a check on cleanliness, recovery, absorption on glassware, etc. The unique stability of the Turner fluorometer will allow you to repeat readings week after week, and thereby provides a valuable check on your standards. Calibration will be changed whenever the ultraviolet lamp is removed from its socket, however.

\*\*\*For exploratory work or other special cases, please go over the Data Reduction Notes, section IV of this manual.

#### IV. DATA REDUCTION NOTES

##### A. INTRODUCTION

The Turner Model 111 Fluorometer has the unique property of giving repeatable readings over long periods of time, regardless of light source aging, variation in line voltage, or photomultiplier drift or fatigue. This is due to the inherent stability of its optical bridge design.

The controls have been engineered to allow the user to take the maximum advantage of this property, as a check on his standards, and to minimize the need for frequent calibration.

Flexibility and operating simplicity have not suffered. Method I below will be used for most routine work. Method II, while slightly more complicated, is preferred for exploratory work, where reagent blank is subject to variation and should be measured, and where concentrations of the unknown may vary over as wide a range as six decades.

##### B. WHAT THE CONTROLS DO.

The controls involved are the FLUORESCENCE dial, the BLANK knob (both on the front panel) and the RANGE SELECTOR, (in front of the ultraviolet lamp).

The BLANK knob allows the amount of light leaving the sample which is represented by ZERO on the FLUORESCENCE dial to be continuously adjusted. It is used to compensate for the fluorescence of the reagent blank.

The FLUORESCENCE dial gives a reading proportional to the amount of light leaving the sample, which is in excess of the light compensated for by the BLANK knob.

The RANGE SELECTOR sets the amount of ultraviolet light falling on the sample to one of four values. The RANGE SELECTOR positions are marked 1X, 3X, 10X and 30X. The 1X range gives the least light, 3X about three times as much, etc.

##### C. WHAT THE SAMPLE DOES.

The light leaving the sample consists of two components. One component is due to the fluorescence of the unknown. This is proportional to the amount of the unknown present. The second component is due to such effects as fluorescent impurities in the reagents used, interfering substances, and, for extremely delicate measurements, fluorescence of the cuvette itself. Both of these components are proportional to the amount of ultraviolet light falling on the sample.

##### Method I.

Method I should be used for routine measurements, where the range of concentrations encountered may be conveniently read on one setting of the RANGE SELECTOR, and where the reagent blank is low, or known to be consistent.

*REAGENT WATER*  
A reagent blank, and a standard are required.

The Fluorometer is turned on, and lamp started, as detailed in Section III of this manual. Filters are installed, according to the application.

The reagent blank is inserted. The RANGE SELECTOR is set to the desired range. The sample door is now closed, and the FLUORESCENCE dial brought to ZERO, by rotating the BLANK knob.

The standard is inserted, and the sample door closed. Wait until the FLUORESCENCE dial reaches its final reading. Record the reading. This is  $R_s$ .

The unknown is inserted, and the sample door closed. Wait until the FLUORESCENCE dial reaches its final reading. Record the reading. This is  $R_u$ .

---

Since one of the signs of a deteriorated or contaminated reagent may be a change in the fluorescence of the blank, it is recommended that the reagent blank be measured daily as described under Method II, and its reading recorded before proceeding with the method of measurement described under Method I.

The concentration of the unknown may be found by a single ratio:

$$\text{Unknown concentration equals } \frac{R_u}{R_s} \text{ times Standard Concentration}$$

If the RANGE SELECTOR setting is changed, the BLANK knob must be reset on a reagent blank as outlined above. This is because changing the range alters the amount of light which is suppressed by the BLANK knob. Ranges may be precalibrated, as long as the BLANK knob is reset, using a reagent blank on each range, prior to reading the standard and unknowns.

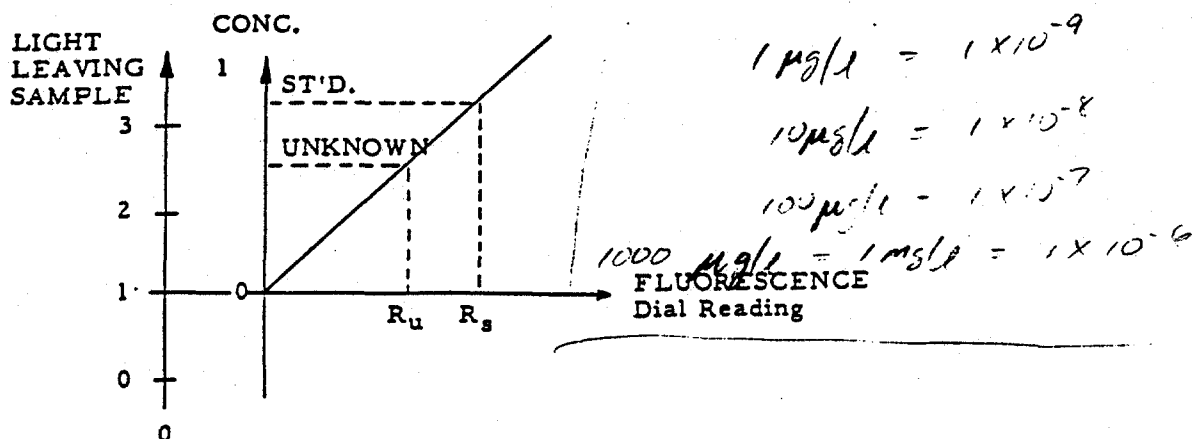


Fig. 1 - Routine Operation

## Method II

Method II should be used for exploratory work, where unknowns vary over a wide range of concentrations, or where information on the stability of the reagent blank is required.

A reagent blank, and a series of standards covering the range of unknowns is required.

The fluorometer is turned on and lamp started, as detailed in Section III of this manual. Filters are installed, according to the application.

Our dummy cuvette (#110-940) is inserted.\* The setting of the RANGE selector is not important. The sample door is now closed, and the FLUORESCENCE dial brought to ZERO, by rotating the BLANK knob.

The reagent blank and appropriate standard are read on each range in turn, without moving the BLANK knob. This information may be conveniently tabulated as shown in Figure 2. The notation ND 1% refers to the use of our range extension filter, #110-823 color specification ND 1%.

Range	X30	X10	X3	X1	X30+ND 1%	X10+ND 1%
Reagent blank	30	10	3	1	0	0
1 µg/liter	90	30	9	---	---	---
10 µg/liter	---	---	63	21	6	---
100 µg/liter	---	---	---	---	60	20

Fig. 2 - Data Tabulation For Exploratory Work.

The unknown concentration may be found by first finding the range on which the FLUORESCENCE dial moves well up scale, and recording range and reading. This is  $R_u$ . Referring back to the table for that range, find the reading for the reagent blank, a convenient standard reading, and the standard concentration. These values are  $R_b$ ,  $R_s$  and  $C_s$ . The unknown concentration is equal to  $\frac{R_u - R_b}{R_s - R_b} \times C_s$ .

\*Note: A piece of heavy black paper may be placed over the primary or secondary filter instead. The object is to reduce to zero the light reaching the photocell from the sample.

For example, assume a reading of 78 on the X3 range in the table, Figure 2. The unknown concentration is  $\frac{78 - 3}{63 - 3} \times 10$  or 12.5  $\mu\text{g/liter}$ .

Note that the BLANK knob is not moved after it is initially set.

Method II is graphically presented in Figure 3, below:

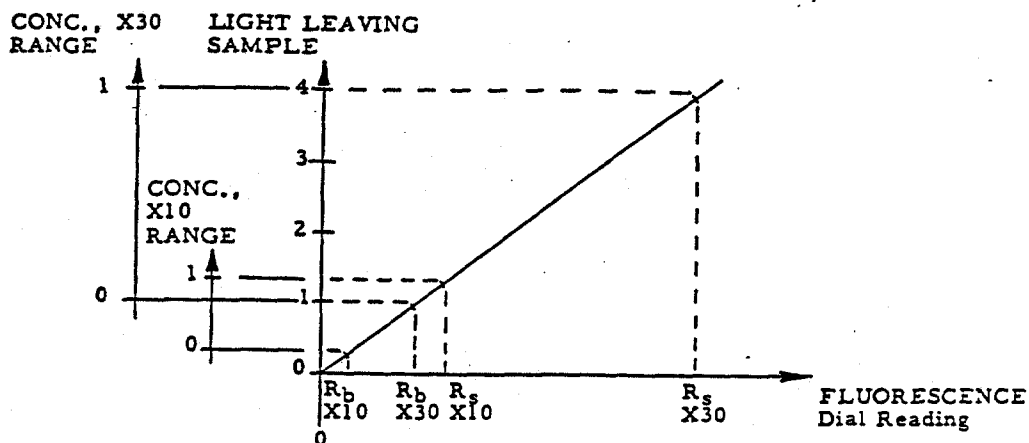


Fig. 3 - Exploratory Operation

Note that the curve relating FLUORESCENCE dial reading to light leaving the sample has not been changed. As the RANGE SELECTOR is moved towards lower numbers (less sensitivity) the concentration scale is compressed, and moved towards zero.

#### D. GRAPHICAL METHODS.

All of the above methods may be handled graphically, as suggested by the figures. The linear relationship between concentration and fluorescence makes it possible to draw up calibration curves knowing the reading on either one standard and a reagent blank, or two standards on any range used.

#### E. NONLINEARITY.

Only three reasons for deviations from linearity have been found. These are:

1. Extremely high concentration of the fluorescent material, leading to self-absorption of light. This concentration quenching effect frequently occurs at concentrations above about 1 ppm and should be checked.
2. High concentration of a material in the reagents which absorbs either the exciting ultraviolet light, or the emitted light.
3. Non-linearity in a chemical reaction which is used to convert the unknown to a fluorescent material, or non-linear recoveries.

Generally, problems 1 and 2 above can be cured by dilution. Calibration curves must be resorted to in the infrequent situation of case 3.

## V. MAINTENANCE AND TROUBLESHOOTING

### A. INTRODUCTION.

Reliability has been stressed in the design of your fluorometer. It should provide you with years of trouble-free operation. As far as we can determine, it is fail-safe. If a failure does occur, the instrument will give no answer. If correctly operated, it will not give a wrong answer.

The bulk of the failures which may occur are connected with tubes or light-sources. These troubles may be corrected by reference to the trouble-shooting list below.

Where the simple measures outlined in this section do not correct a failure, we recommend that this instrument be sent to your scientific instrument dealer for repair. It is mandatory that this manual accompany the instrument if any repairs are required.

Factory repair service, including complete calibration and test is available. Should you desire to use this service, we recommend that you procure a shipping carton for your instrument before returning it. See the price schedule, below. Shipment should be prepaid and made in such a way that the instrument will be delivered directly to our plant. The manufacturer assumes no responsibility for damage in transit from the customer but does guarantee safe and satisfactory return. Return shipments are to, Palo Alto, California.

Filter Fluorometer, Model 110	
Repair, test, calibrate, each . . . . .	\$31.25
(Price does <u>not</u> include parts used)	

Filter Fluorometer, Model 111	
Repair, test, calibrate, each . . . . .	\$40.00
(Price does <u>not</u> include parts used)	

Shipping carton for Model 110 or Model 111 Fluorometer	
(Model must be specified), each . . . . .	\$ 7.50

### B. ROUTINE MAINTENANCE.

No routine maintenance is required. All moving parts are either life-time lubricated, or require no lubrication.

### C. PARTS.

Wherever possible, standard components have been used throughout. While we recommend local purchase of all readily available components, we do maintain all parts on a same-day AIR-EX basis, and we are pleased to supply them.

Naturally, manufacturing changes have occurred over the years. To avoid confusion, therefore, we have adopted the policy of shipping parts only after we have received the Serial Number of the instrument and the date at the lower left-hand corner of the schematic diagram. Mechanical parts should be described. We prefer to have these returned so that we may examine them to determine the cause of failure.

### D. TROUBLESHOOTING (Instruments containing a printed circuit board, as shown in Fig. 6, p. 25.)

Caution: This instrument contains a high-voltage power supply. Voltages as high as 2000 volts may be encountered when the rear cover, or electronic assembly is removed.

#### 1. GENERAL.

Prior to carrying out any of the following procedures, the power line voltage should be checked. It must be between 105 and 130 volts A.C., at the frequency (60 or 50 cycles) specified on the nameplate affixed to the rear of the instrument.

Where operating on portable power sources, the frequency must be checked, with all normal loads applied. It should be within  $\pm 1$  cycle of that specified on the nameplate. Where operating on portable power sources, the waveform must be checked with all normal loads applied. It should be a reasonably good sine wave. Proper operation will not be obtained if the waveform approaches a square wave or has excessive "peaking".

2. ULTRAVIOLET LAMP DIFFICULT TO START, OR WILL NOT START.

Caution: If the instrument is equipped with the far-ultraviolet lamp, in which the ultraviolet lamp appears to be made of clear glass, or with the #110-855 (U.V.), wear eyeglasses. Serious and painful eye irritation will result from direct observation of these lamps.

Replace lamp with a spare. Refer to Section VI., SERVICE INFORMATION, Part F, for the lamp replacement procedure. While these lamps have a normal life of several thousand hours, they are not generally available. A new spare should be ordered immediately. Be sure to specify the type used, by reference to the price list. Two bent or hairpin-shaped lamps are available. These are #110-850 (normally supplied with the instrument), which has an opaque white interior coating and #110-851, which is clear. Three straight lamps are available for use with the #110-856 lamp adaptor. These are #110-853 (Blue), #110-854 (Green) and #110-855 (U.V.). Remember-whenever it is necessary to look at lamp #110-851 or #110-855, eyeglasses should be worn.

Should there be any question of type, please return the faulty lamp with your order.

3. PILOT LIGHT DOES NOT COME ON.

Check fuse. Replace if faulty. The pilot lamp is a neon lamp, which has extremely long life, and is probably not at fault.

If the pilot light does not come on immediately after power is applied, remove power and refer to Section VI., SERVICE INFORMATION for possible power supply short circuits.

4. FLUORESCENCE DIAL GOES TO FULL C.W. (JUST OVER 100) WITH SAMPLE IN PLACE.

Use a less sensitive range, place one of our #110-823, color specification ND 1%, range extension filters in the secondary filter holder (in addition to the normal secondary filter), or dilute the sample.

5. FLUORESCENCE DIAL GOES TO FULL C.C.W. (JUST UNDER 0) WITH SAMPLE IN PLACE.

BLANK knob is incorrectly adjusted. Turn C.C.W.

6. FLUORESCENCE DIAL DOES NOT MOVE, OR MOVES WITHOUT RESPONDING TO BLANK KNOB SETTING.

- a. Be sure that there is nothing obstructing the cooling fan under the instrument, and that it is turning properly.
- b. Check to see if the ultraviolet lamp is lit.
- c. Check all tubes on the electronic chassis and replace (if required).
- d. Check and replace (if required) the reference light source, V05. See Section VI, SERVICE INFORMATION, Part G, for lamp test and replacement procedure.
- e. Sample extremely fluorescent, overloading circuits. Install black rod, or otherwise prevent light from reaching photomultiplier. If operation is normal, instrument is operating correctly. Sample must be diluted, or neutral filter must be added.

7. FLUORESCENCE DIAL JITTERY, USUALLY AT 1-2 CYCLES PER SECOND.

Lamp emission may be erratic. This is infrequent but, when occurring, may be confirmed by removing the primary filter and setting the range selector to 30X. **WARNING:** If the instrument is equipped with the far ultraviolet lamp, #110-851, in which the lamp appears to be made of clear glass, or with the #110-855 (U.V.) lamp, do not observe it unless you wear eyeglasses. Painful and serious eye irritation will result from direct observation of these lamps.

This erratic condition will be most likely to occur when the instrument is cold, as in cold room operation. It may usually be cured by turning the instrument off for about a minute and restarting as usual. Be sure to hold the START switch up for three or four seconds. If the condition is not corrected, replace the lamp with a spare, following the procedure on page 21. Order a new lamp as described in part 2 above.

FLUORESCENCE DIAL UNUSUALLY SLUGGISH OR JITTERY - see Section VI, SERVICE INFORMATION, under part E. SERVO DAMPING CIRCUIT, for specifications. Some materials, quinine for example, require a finite time to develop full fluorescence. The dial may thus approach its final reading slowly. Check that instrument used is Model 111-000 for 60 cycle operation, or Model 111-050 for 50 cycle operation, as specified on the nameplate affixed to the rear of the instrument.

Operation will be more sluggish with the far ultraviolet lamp, #110-851 and the lamp #110-855 (U.V.) than with other lamps.

9. EXCESSIVE SENSITIVITY TO LINE VOLTAGE.

When the instrument is used for luminescence measurements, external line voltage regulation must be provided! Referring to Section VI-B, it may be seen that the optical bridge is not effective when luminescence measurements are being made. Please request the manufacturer's reference on "BIOLUMINESCENCE" for further data.

If the sample is improperly centered, as it may be if the Sample Door is damaged in shipment, or modified, line voltage sensitivity may also be encountered. Please request further information from the factory, forwarding the Serial Number of the instrument and the types of sample holding doors (and modifications, if present) which are being used.



## VI. SERVICE INFORMATION

### A. GENERAL.

This section is divided into the following subsections.

- B. OPTICAL SYSTEM
- C. ELECTRONIC SYSTEM
- D. OPTICAL COMPARATOR AND PHOTOMULTIPLIER
- E. ELECTRONIC CIRCUITS
- F. DISASSEMBLY
- G. ADJUSTMENT AND CALIBRATION

Sections B and C are recommended reading for those who would like to understand the overall operation of their fluorometer. The balance of this section is written to aid the serviceman, who is unable to repair the instrument with the simple tests listed under Section V., MAINTENANCE AND TROUBLESHOOTING.

### B. OPTICAL SYSTEM (See Fig. 4).

This fluorometer is basically an optical bridge which is analogous to the accurate Wheatstone Bridge used in measuring electrical resistance. The optical bridge detects the difference between light emitted by the SAMPLE and that from a REAR LIGHT PATH. A single photomultiplier surrounded by a mechanical light interrupter sees light alternately from the SAMPLE and the REAR LIGHT PATH. Photomultiplier output is alternating current, permitting a drift-free A.C. amplifier to be used for the first electronic stages. The second stage is a phase-sensitive detector whose output is either positive or negative, depending on whether there is an excess of light in the SAMPLE or REAR LIGHT PATH, respectively. Output of the phase detector drives a servo amplifier which is in turn connected to a servo motor. The servo motor drives the LIGHT CAM (and the FLUORESCENCE dial) until equal amounts of light reach the photomultiplier from the SAMPLE and from the REAR LIGHT PATH. The quantity of light required in the REAR LIGHT PATH to balance that from the SAMPLE is indicated by the FLUORESCENCE dial. Each of this dial's 100 divisions adds equal increments of light to the REAR LIGHT PATH by means of the LIGHT CAM.

Light in the REAR LIGHT PATH may also be operator-adjusted with the BLANK control which sets the REAR LIGHT PATH to be equal to the residual fluorescence of a blank with the FLUORESCENCE dial set at zero.

Note: When the instrument is used for luminescence measurements in which light emitted by the sample is independent of the intensity of the light source, photomultiplier gain changes still cancel out—but light source changes do not. External voltage regulation will be required. Please request the reference on "BIOLUMINESCENCE" for further data.

Light-source variations do not affect the light balance. Such variations are caused by aging of the ultraviolet source and by line-voltage and frequency changes. Because these affect both the light on the SAMPLE and light in the REAR LIGHT PATH proportionately, light balance is not changed. Variations in photomultiplier sensitivity cancel for the same reason.

Dark current is not a problem because the photomultiplier sees interrupted light and the electronic circuitry detects only the difference in light from the REAR LIGHT PATH and from the SAMPLE.

Persistence of fluorescence does not affect the unit because the light falling on the SAMPLE is steady. Only the light emitted from the fluorescent sample is interrupted.

FLUORESCENCE dial sensitivity is always the same, even when high-concentration work is being done with neutral-density filters in the secondary-filter holder.

The FORWARD LIGHT PATH is included so that there is always light reaching the photomultiplier, even when sample fluorescence is zero. This ensures that the servo system will operate correctly both above and below zero, with non-fluorescent blanks.

### C. ELECTRONIC SYSTEM (See Fig. 5)

The output of the photomultiplier is an A.C. signal at a frequency of about 400 cycles per second and is proportional to the light unbalance. Its phase changes 180°, with respect to the light-interrupter position, when the light in the CALIBRATED LIGHT PATH changes from larger to smaller than the light from the SAMPLE plus the FORWARD LIGHT PATH.

This signal is amplified, and applied as the signal to the phase detector.

The reference lamp and reference photocell generate an A.C. signal whose instantaneous value depends on the position of the light interrupter. This signal is amplified and applied as the reference to the phase detector.

The phase detector generates a D.C. signal proportional to the signal applied to it, and whose polarity depends on the phase of the applied signal with respect to the reference.

This D.C. signal is filtered, combined with a signal proportional to the speed at which the FLUORESCENCE dial is moving, developed primarily by C30 and the output potentiometer, and applied to the chopper. The chopper converts this D.C. signal to a 60 (or 50) cycles per second A.C. error signal, which is then amplified and used to drive the servo motor.

#### D. OPTICAL COMPARATOR and PHOTOMULTIPLIER (See Fig. 4)

This assembly mounts directly behind the secondary light filter and contains the photomultiplier, reference photocell and light source, light interrupter, motor, and the acrylic light pipes. Replacement or adjustment of these elements should be carried out only by the manufacturer.

Due to the careful selection and special aging procedures used at the factory, photomultiplier failures are practically nonexistent. Time between failures has proven to be in excess of 500,000 hours! Even if you suspect the photomultiplier, carefully check all other possibilities first.

#### DETAILS OF THE OPTICAL DESIGN

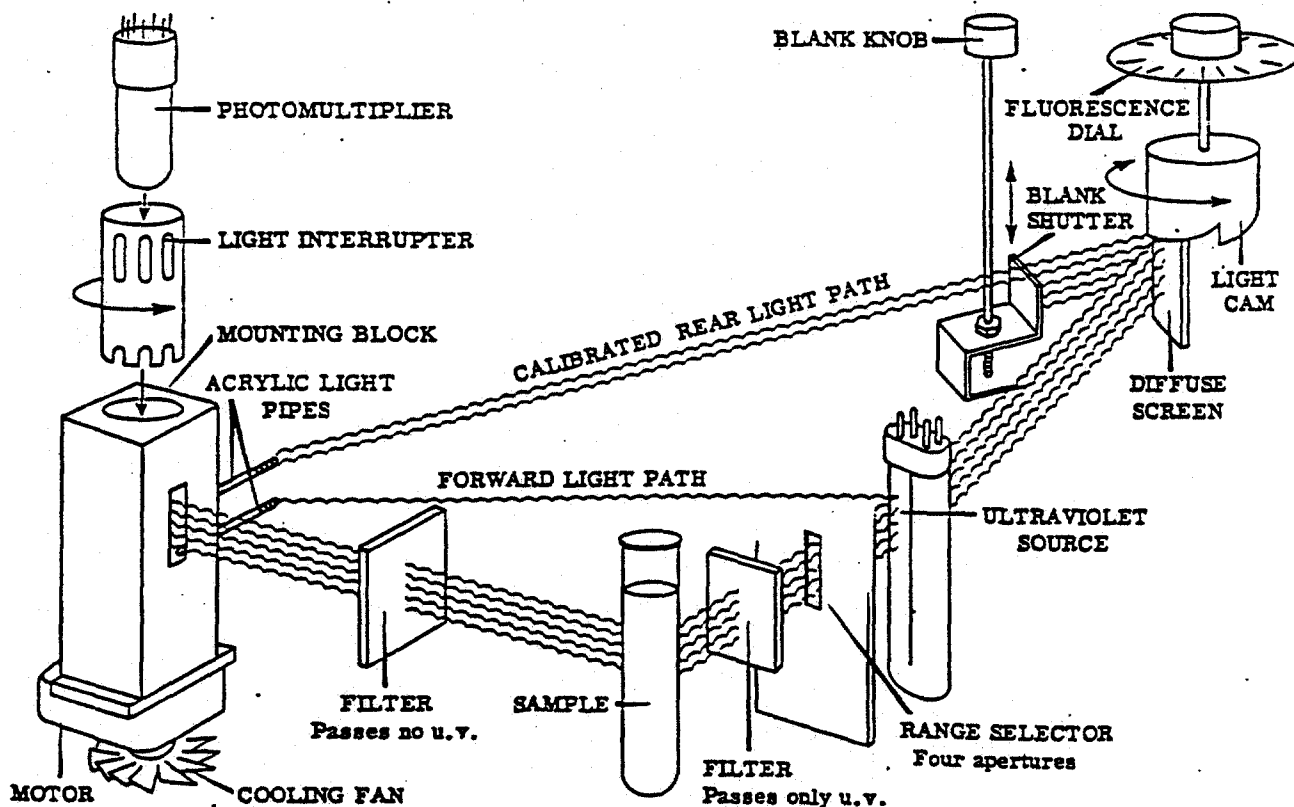


Fig. 4 - Optical System

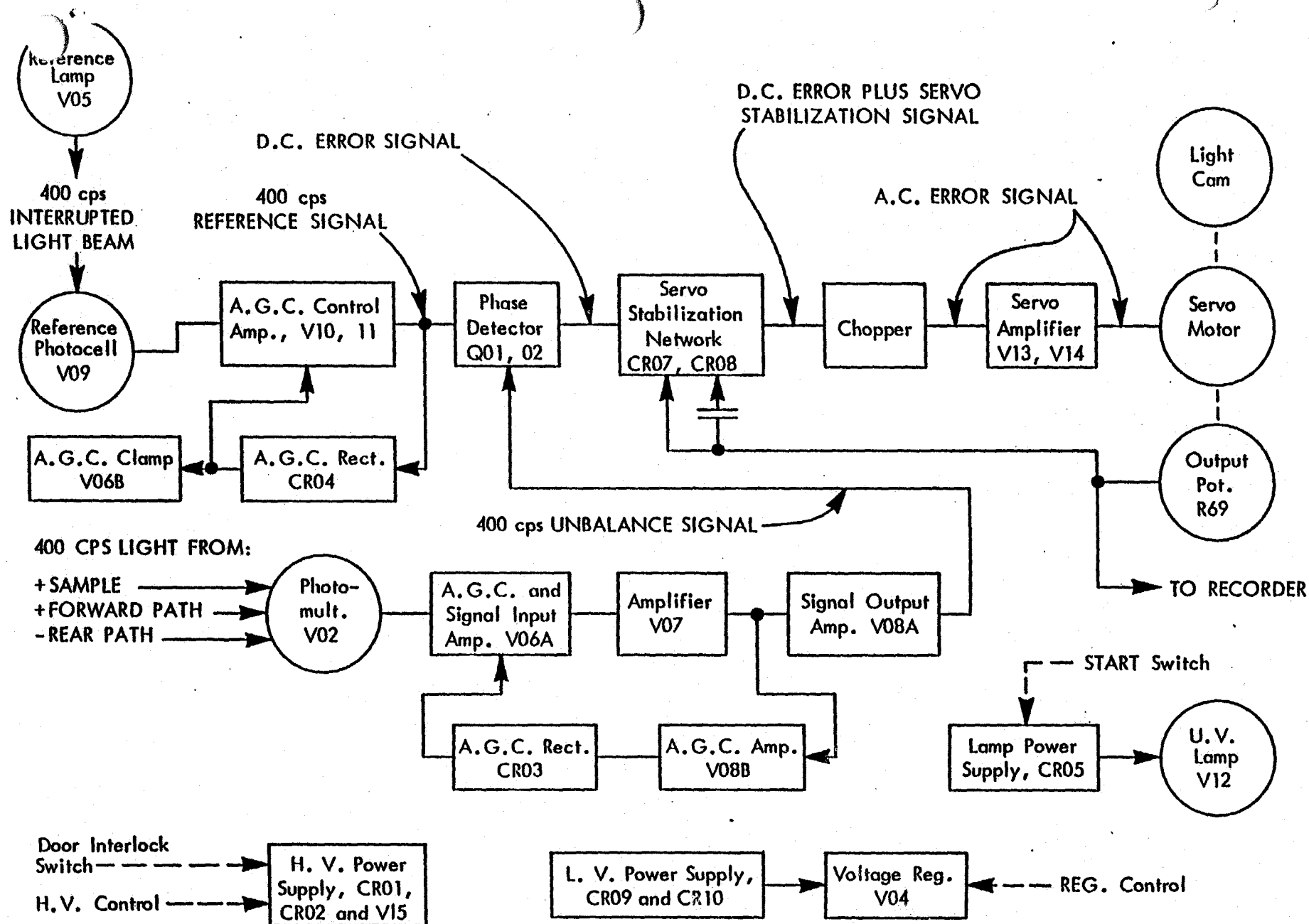


Fig. 5. Electronic System

## REFERENCE GENERATOR

The reference generator is physically a part of the optical-comparator assembly. It consists of a light source, V05 (a type #44 pilot light), the special filter glass around it, which passes only infrared light, a red-sensitive photocell V09 (a type 927 gas phototube), and the lower set of slots in the light interrupter.

The output of the 927 gas phototube is a very irregular waveform, with a fundamental frequency component whose phase is constant with respect to the phase of the photomultiplier output. The phase of the 927's output may be adjusted by sliding light source V05 in or out. Adjustment is described in Part G of this section.

## SUMMARY

The optical comparator described above combines the FORWARD LIGHT PATH, REAR LIGHT PATH and light from the SAMPLE, and develops an A.C. error signal. Adjustment is critical as the various light paths must add or subtract quite precisely, which means that they must be passed by the light interrupter in the correct time relationship. Adjustment is a factory procedure. The output signal from the photomultiplier is only a few millivolts, and it is so masked with noise that it is difficult to observe directly with an oscilloscope and may not be measured with a voltmeter.

The REFERENCE GENERATOR is mounted on the optical comparator. Its adjustment is described in part G of this section.

## E. ELECTRONIC CIRCUITS

The electronic circuits in the Model 111 Recording Fluorometer are not straightforward. Please read the following circuit description carefully before attempting repair.

### PHOTOMULTIPLIER AMPLIFIERS (See Fig. 5 and 8)

The signal from the photomultiplier consists of:

1. Dark current.
2. Noise, over a wide frequency range.
3. An A.C. voltage whose fundamental frequency component is dependent on the number of dial divisions the FLUORESCENCE dial is off balance. The phase of this voltage changes from 0° to 180°, as the FLUORESCENCE dial goes from below balance to above balance.

Dark current is eliminated by use of an A.C. amplifier.

Noise is gradually reduced by limiting both the low frequency and high frequency response of the amplifier. This is done by proper selection of coupling capacitors and bypass capacitors in the A.C. amplifier and by the tuned circuit consisting of T02 and C24. Final noise reduction is handled in the Servo Damping Circuit, described below.

The signal is amplified in turn by V06A, V07 and V08A. The output of V08A is coupled to the PHASE DETECTOR, described below, by transformer T02. The amplification factor of V06A is set by the A.G.C. voltage applied via R30 to its grid.

An A.G.C. system is required since the signal available from the photomultiplier, per dial division of unbalance varies widely, and the gain of the PHOTOMULTIPLIER AMPLIFIER stages may also vary widely. Choice of ultraviolet lamp affects the photomultiplier output, and line voltage and tube changes affect the gain of the amplifier.

The A.G.C. system operates to maintain the high frequency component of the residual noise from the photomultiplier constant. Note—the unbalance signal from the photomultiplier could not be used, since it goes to zero at balance! The high frequency noise component required for A.G.C. control is separated from the unbalance signal by the network consisting of C12 and R33. It is amplified by V08B. When it exceeds a threshold value established by R36 and R37, it is rectified by CR03. After filtering, this A.G.C. signal is applied to the grid of V06A, as described above.

The signal-to-noise ratio is so low that until the amplifier bandwidth is reduced by the resonant circuit consisting of T02 and C24, the signal is very difficult to observe on an oscilloscope. The waveform to be expected at T02 is given on the schematic, Fig. 8.

## REFERENCE AMPLIFIER

This amplifier converts the output of the reference photocell, V09, to a reasonably good sine wave, required by the phase detector.

The signal is amplified by V10 and V11, and applied to the tuned output transformer, T03. When the signal exceeds the bias set up by R48 and R49, it is rectified by CR04, and the A.G.C. voltage developed is used to reduce the gain of the A.G.C. tube, V10.

Tube V06B, diode connected, with cathode grounded, prevents the reference amplifier A.G.C. control voltage from going positive during warm-up, and blocking the reference amplifier.

## PHASE DETECTOR

Transistors Q01 and Q02 act like switches. When the reference voltage out of T03 is at such a point in its cycle that R54 is positive and R53 is negative, the base of Q02 goes positive, while the base of Q01 remains at ground potential since the base to emitter junction of Q02 is reverse biased, while Q01 is forward biased. The collector of Q02 is now open circuited, since no current is flowing in the emitter circuit. The collector of Q01 is essentially short circuited to ground since current is flowing in the emitter circuit. When the reference voltage out of T03 reverses, Q02 becomes short circuited, while Q01 becomes open circuited.

The action of Q01 and Q02 is thus equivalent to a SPDT switch whose arm is grounded, and whose contacts are alternately connected to the "COM" tap and "16Ω" tap of T02 depending on the polarity of the reference signal.

If the 16 ohm tap of T02 is more positive than the 4 ohm tap, while Q01 is short circuited, a negative voltage is developed across R55. If the 16 ohm tap of T02 is more negative than the 4 ohm tap, while Q01 is short circuit, a positive voltage is developed across R55. Following this reasoning through for all combinations will show that a voltage will be developed across R55 which is proportional to the signal voltage, and whose polarity depends on whether the signal and reference voltages are in or out of phase.

This is the desired error voltage. Its magnitude is proportional to the error from correct position of the FLUORESCENCE dial. Its polarity is dependent on whether the FLUORESCENCE dial is above or below balance.

## RECORDER COUPLING CIRCUIT

Potentiometer R69 is a three-turn precision wirewound potentiometer, geared to the FLUORESCENCE dial. The end corresponding to zero on the FLUORESCENCE dial is grounded. The end corresponding to 100 is supplied with approximately 105 volts D-C, regulated by the regulator tube, V04. The voltage at the arm of this potentiometer varies from about 2 volts to 93 volts, as the FLUORESCENCE dial is rotated from 0 to 100. The slight offset at zero is due to potentiometer end resistance, and the necessity of having the FLUORESCENCE dial move through zero smoothly.

The potentiometer arm connects to the upper red recorder binding post (marked 0-1 MA) through R70 and the SPAN control, R71. The total resistance of R70 and R71 may be varied from 91K to 111K ohms, so that the output current into a low-impedance load, such as a milliammeter or galvanometer type recorder may be set to vary from 0.02 milliamperes, with the FLUORESCENCE dial at zero, to 1.02 milliamperes with the FLUORESCENCE dial at 100.

The middle red binding post (marked 10 MV SHUNT) is connected to ground through a precision 10 ohm resistor, R72. When the two red binding posts are connected together and the SPAN control is properly adjusted, the voltage across R72 will vary from 0.2 to 10.2 millivolts, as the FLUORESCENCE dial moves from zero to 100.

## STANDARD RECORDER CONNECTIONS

For 0-1 milliampere recorders, 5,000 ohm maximum impedance.

1. Connect the recorder positive terminal to the upper (red) recorder binding post (marked "0-1 MA") on the rear of the fluorometer.
2. Connect the recorder negative terminal to the lower (black) recorder binding post (marked "GROUND").
3. Be sure that there is no jumper wire between the upper and center binding post.
4. Adjust zero and SPAN controls per Section II-C-7, page 5.

For high impedance 0-10 millivolt recorders (1000 ohm minimum impedance).

1. Connect a jumper wire between the upper (red) recorder binding post (marked "0-1 MA") and the center (red) recorder binding post (marked "10 MV SHUNT").
2. Connect the recorder positive terminal to the center (red) recorder binding post.
3. Connect the recorder negative terminal to the lower (black) recorder binding post (marked "GROUND").
4. Adjust zero and SPAN controls per Section II-C-7, page 5

#### NON-STANDARD RECORDER CONNECTIONS

For millivolt recorders:

1. The recorder must have a range of under 1,000 millivolts, and an impedance at least 100 times its range.
2. Connect a resistor whose resistance in ohms equals the recorder range in millivolts between the upper (red) recorder binding post (marked "0-1 MA") and the lower (black) recorder binding post (marked "GROUND").
3. Connect the recorder positive terminal to the upper (red) recorder binding post.
4. Connect the recorder negative terminal to the lower (black) recorder binding post.
5. Be sure that there is no jumper wire between the upper and center binding posts.
6. Adjust zero and SPAN controls per Section II-C-7, page 5

For microampere recorders:

1. The recorder must have a range of under 1000 microamperes and an impedance of under 5,000 ohms.
2. Connect a resistor in series with the positive terminal of the recorder. The resistor should have a value of about:
$$R \text{ (in megohms)} = \frac{95}{\text{recorder range, microamperes}} - 0.1$$
3. Connect the free end of the resistor to one end of a potentiometer whose resistance is about 20% of the value of the resistor.
4. Connect the arm of the potentiometer to the upper (red) recorder binding post (marked "0-1 MA") on the rear of the fluorometer.
5. Connect the recorder negative terminal to the lower (black) recorder binding post (marked "GROUND").
6. Be sure that there is no jumper wire between the upper and center binding posts.
7. Set the control near the recorder terminals on the fluorometer marked "SPAN" to its approximate center settings.
8. Adjust zero and SPAN controls per Section II-C-7, page 5 , except use the potentiometer added in place of the "SPAN" control.

#### SERVO DAMPING CIRCUIT

It is necessary that the Model 111 Fluorometer respond slowly to changes in fluorescence of the sample. This is because the photomultiplier circuit is so sensitive that the noise from the photomultiplier is high, and must be averaged over a long period of time.

This is accomplished by feeding two inputs into the servo amplifier (described in detail below). One input is proportional to the error from correct position of the FLUORESCENCE dial, and comes from the phase detector, described above. It is so phased that it causes the FLUORESCENCE dial to move towards its correct position. The second input is proportional to the speed of the FLUORESCENCE dial, and so phased that it tries to cause the FLUORESCENCE dial to stand still.

The servo amplifier drives the FLUORESCENCE dial in the appropriate direction and at the proper speed to cause these two inputs to be equal. Thus the combination of these two inputs, one proportional to error, one proportional to speed, causes the FLUORESCENCE dial to approach its final reading at a speed which is proportional to its error from

the final reading. Components are so chosen that errors under about 10 dial divisions will be reduced to one tenth in about five seconds.

#### VELOCITY CIRCUIT

As described above, the voltage at the arm of potentiometer R69 varies about one volt per dial division. The voltage levels at the ends of C29 and C30 away from the potentiometer arm are low by comparison, due to servo action. Therefore, as the FLUORESCENCE dial moves, currents proportional to the rate of motion of the FLUORESCENCE dial are developed in these two capacitors.

The current through C30 flows through the servo stabilizing lead network consisting of C31 and R58, where it joins the error current through R57 at the servo amplifier input. Action is as described under "SERVO DAMPING CIRCUIT," above.

The current through C29 is shorted to ground by CR07 and CR08, when over 0.4 volts is developed across these silicon rectifiers. Hence, for large errors, the current through C29 is ineffective, allowing rapid response when large errors from the final balance point exist. When near balance, CR07 and CR08 become essentially an open circuit, and further damping starts due to current flowing in C28.

#### SERVO AMPLIFIER

The servo amplifier may be considered to start with the input resistor to the chopper, R60. The chopper alternately short circuits and open circuits the voltage out of R60, at a 60 (or 50) cycles per second rate. This generates an A.C. square wave signal, proportional to the voltage into R60 and whose phase depends on the polarity of the input voltage. This A.C. signal is amplified by the voltage amplifier, V13, and the power amplifier, V14. Output power is coupled to the servo motor control windings by the tuned autotransformer, T04. The field winding of the servo motor is powered directly from the power line.

The servo amplifier will drive the servo motor at full speed for an input of under 40 mv D.C.

#### PHOTOMULTIPLIER POWER

Unregulated photomultiplier power is supplied by the full-wave voltage doubler circuit consisting of CR01, CR02, C06 and C07. Approximate regulation is obtained by use of V15, a corona type voltage regulator. The rear-panel control S02, marked H.V., is factory set so that the photomultiplier gain is optimum. The A.G.C. action of the photomultiplier amplifier compensates for normal line voltage variation. The photomultiplier voltage divider string returns to filtered B+, on capacitor C03C. Switch S01, actuated by the front-door latch, short-circuits the photomultiplier voltage when the door is open. Ample series resistance (R02 and R03) protects the power supply. This shorting switch protects the photomultiplier from excessive light, when the dynode voltages are applied.

#### LAMP POWER

The ultraviolet lamp operates on direct current, so that if the light interrupter happens to rotate very close to a multiple of the line frequency, no interaction results. Line power is rectified by CR05 filtered by C26 and CH01. Current is limited by resistor R52. CH01 also serves to supply a starting surge of voltage, when the starting switch, S06 is first opened. The normal D-C voltage drop across resistor R52 is about 75 volts. R51 is a bleeder resistor.

#### CIRCUIT POWER

Circuit power is supplied by a full wave center-tapped rectifier circuit, utilizing CR09 and CR10. R75 is used to limit current surges into the input capacitor, C03A.

#### F. DISASSEMBLY

CAUTION! This instrument contains a high-voltage power supply. Voltages as high as 2000 are used.

#### ELECTRONIC ASSEMBLY

The electronic assembly may be detached by removing the two screws on either side of the instrument, and pulling the assembly back. Four connectors, two on cable ends, two on the electronic chassis may be disconnected to free the electronic chassis completely.

The cables are long enough to allow operation with the electronic assembly partially removed, and accessible for test. The most convenient position is with the electronic assembly lying on the side panel nearest the POWER switch. Use a pad or clean cloth to protect the finish.

Do not attempt careful calibration in the disassembled position, except in a darkened room, since room light may now reach the acrylic light pipes in the optical comparator.

#### FORWARD COVER

The cover over the forward part of the instrument -- which carries the instrument name -- may be removed by first removing the BLANK and FLUORESCENCE knobs, then the shaft lock assembly under the BLANK knob, and, finally, four screws up from the base. This may only be done after the electronic assembly is removed, and then only for very good reason. Realignment of the FLUORESCENCE knob is covered in Part G of this section.

#### ULTRAVIOLET LAMP REPLACEMENT, "U" TYPE

**CAUTION!** If the instrument is equipped with the far-ultraviolet lamp #110-851, in which the ultraviolet lamp appears to be made of clear glass, or lamp #110-855 (U.V.), do not observe it in operation unless you wear eyeglasses. Painful and serious eye irritation will result from direct observation of these lamps.

Turn the fluorometer off. Open the sample compartment door. Remove and carefully set aside the primary (right) optical filter. This filter consists of one or more pieces of 2" x 2" colored glass. Remove the two screws on the right edge of the flat plate on which the optical filter was mounted. Slide this whole plate straight up, then out. The lamp is now exposed. Lift up the "U"-shaped wire retainer which holds the ultraviolet lamp in place. Remove the lamp by pulling straight down. When putting a new lamp in place, remember that the two large pins should be toward you, and parallel to the plate which was removed.

**WARNING:** Be sure to replace the "U"-shaped wire retainer, as this secures the lamp in its mounting. Slide the filter mounting plate in and down. Its back edge must be behind the machined bar which separates the two filters. Replace the two screws and primary filter.

#### LAMP REPLACEMENT, STRAIGHT TYPE

Straight lamps require use of the Lamp Adaptor, #110-856. Follow the directions which accompanied your Lamp Adaptor.

#### G. ADJUSTMENT AND CALIBRATION

##### GENERAL

On rare occasions, it may be necessary to replace the reference light source. (See Troubleshooting.) When the new lamp is installed, readjustment may be required.

#### REFERENCE LIGHT SOURCE TEST AND REPLACEMENT

(Work should be done in a room which can be darkened.)

1. Unplug the instrument.
2. Remove the two screws on each side of the instrument.
3. Place a pad on the work bench, to protect the finish.
4. Slide the electronic assembly straight back, and lay on the pad, side panel nearest the POWER switch down.
5. Move the electronic assembly as far to the left (as viewed from the rear) as possible, without disconnecting the various connectors. **Warning!** This instrument contains a high voltage power supply. Voltages as high as 2000 are used.
6. Plug the power cord in, and turn the instrument on.
7. Find the black glass filter on the optical comparator just over the motor. This is located directly under the G.K. TURNER ASSOCIATES symbol on the front cover.
8. If a purple light can be seen through this filter, the reference lamp source is operating properly.

If the reference lamp source is operating properly, look for trouble elsewhere.

If the reference lamp is out, go on to the next step.



9. Unplug the instrument.
10. Remove the screw just above the right end (as viewed from the rear of the instrument) of the black glass filter. This screw holds the cable clamp and filter in place. **DO NOT REMOVE THE LAMP SOCKET!**
11. Pull filter and clamp together off to the left. The reference lamp should now be exposed.
12. Remove the lamp by pushing it in slightly, and rotating a fraction of a turn counterclockwise. Replace with a new type #44 pilot light - obtainable at any radio store. NOTE only a type #44 will work.
13. Replace the filter by reversing steps 10 and 11 above.
14. Set the range selector to the least sensitive range. Be sure both a primary and secondary optical filter are in place. Remove the cuvette, if present.
15. Referring to the OPERATING INSTRUCTIONS, Section III, plug the instrument in, darken the room, and carry out steps 1-7 under "HOW TO TURN THE FLUOROMETER ON" and steps 1-3 under "HOW TO OPERATE THE FLUOROMETER," as if a reagent blank were in place. If operation is satisfactory, unplug the instrument, and reassemble. If not, go on to the next steps.
16. Install the general purpose primary and secondary filters supplied with your instrument (color specification #7-60 and #2A respectively). Do not install a cuvette. Set the RANGE SELECTOR to the X3 range.
17. Unplug V13, a type 12AX7 tube.
18. Connect the ground lead of an oscilloscope to the chassis. Warning. High voltage! Connect the sync. lead of the oscilloscope to the 500 ohm tap of T03. Connect the oscilloscope input to terminal 12 on the printed circuit board, or the lower lead of R55.  
  
Set up to observe a signal of several volts, peak to peak at about 400 cycles per second. Be sure that the oscilloscope is set up for external sync.
19. Be sure the ultraviolet lamp is on.
20. Close the sample door and put a finger over the hole leading to the upper acrylic light pipe in the optical comparator (see fig. 4). As your finger is removed and replaced, the waveform on the oscilloscope should change. The signal will be quite noisy. Adjust the BLANK knob until similar waveforms, but of opposite polarity, are observed when the finger is in or out. Gently turn the FLUORESCENCE dial if sufficient adjustment may not be had with the BLANK knob.
21. The reference lamp socket and its wires may be handled with impunity. Six volts is the highest voltage present. Loosen the screw to the right (as viewed from the rear of the instrument), which holds the lamp socket in place.
22. Slide the lamp socket along, alternately opening and closing the hole leading to the upper acrylic light pipe, with your finger. The waveform will be a series of sections of sine waves (see schematic fig. 8). Find the setting where the sine wave sections are all in one direction with the finger in, and all in the other direction with the finger out, and where the half sine waves are of the polarity shown on the schematic, with your finger out. Tighten the reference lamp mounting screws.
23. Remove all test leads. Replace V13, and go back to step 14. Remember that you have the BLANK knob badly misadjusted.

#### FLUORESCENCE DIAL ALIGNMENT

1. With power off, turn the FLUORESCENCE shaft full counterclockwise.
2. Set the FLUORESCENCE dial so that it is about two divisions beyond 0 on its scale. Set the dial so that its top surface is level with the top of the lucite pointer. Tighten the two set screws.

#### H. V. CONTROL ADJUSTMENT

This is normally a factory adjustment, and should be done only if there is reason to believe that the setting has been changed. It should be carried out with the line voltage at the lowest value anticipated.

Turn the H.V. control counterclockwise to slow response, reduce jitter, and increase dead zone. Turn the H.V. control clockwise to speed response, increase jitter and decrease dead zone.

TABLE I. ELECTRONIC PARTS LIST, Model 111  
(PRINTED CIRCUIT INSTRUMENTS)

Part No.	Description (1)	Location on Printed Cir- cuit Board, unless noted	Part No.	Description (1)	Location on Printed Cir- cuit Board, unless noted
C01	0.1 @ 2000 v oil	Fig. 7 (inside)	R21	10 K	
C02	0.1 @ 2000 v oil	Fig. 7 (inside)	R22	2.0 K 10W W.W.	Fig. 6 (outside)
C03	40-40-20 @ 450 v electrolytic	Fig. 6 (outside)	R23	2.0 K 2W W.W. VAR. "REG"	Fig. 6 (outside)
C04	Deleted	---	R24	4.7 M	
C05	Deleted	---	R25	470 K	
C06	0.27 @ 1000 v	Fig. 7 (inside)	R26	56 K	
C07	0.27 @ 1000 v	Fig. 7 (inside)	R27	2.2 K	
C08	500 PF ceramic		R28	470 K	
C09	.001 or .002 ceramic (2)		R29	820	
C10	25 @ 25 v electrolytic		R30	4.7 M	
C11	0.005 ceramic		R31	470 K	
C12	65-320 PF (NOISE ADJ)		R32	470 K	
C13	0.5 @ 200 v		R33	470 K	
C14	0.01 @ 600 v		R34	100 K	
C15	500 PF ceramic		R35	470 K	
C16	0.005 ceramic		R36	100 K	
C17	25 @ 25 v electrolytic		R37	560 K	
C18	0.005 ceramic		R38	220 K	
C19	25 @ 25 v electrolytic		R39	470 K	
C20	0.5 @ 200 v		R40	4.7 M	
C21	0.01 @ 600 v		R41	470 K	
C22	0.01 @ 600 v		R42	100 K	
C23	Deleted	---	R43	2.2 K	
C24	0.002 to .047 @ 600 v (3)	Fig. 6 (outside)	R44	470 K	
C25	0.022 to .047 @ 600 v (3)	Fig. 6 (outside)	R45	470	
C26	200 @ 200 v electrolytic	Fig. 6 (outside)	R46	470 K	
C27	2.0 @ 200 v		R47	470 K	
C28	0.5 @ 200 v		R48	2.2 M	
C29	1.0 @ 200 v		R49	100 K	
C30	0.05 to 0.1 @ 600 v (2)		R50	22 5W W.W.	Fig. 7 (inside)
C31	0.022 @ 600 v		R51	47 K 2W	Fig. 7 (inside)
C32	0.01 ceramic		R52	750 20W W.W.	Fig. 6 (outside)
C33	Not present to 25 $\mu$ l @ 25 v (4)		R53	1.5 K	
C34	Deleted	---	R54	1.5 K	
C35	0.1 @ 400 v	---	R55	10 K	
C36	Deleted	---	R56	470 K	
C37	0.01 ceramic		R57	470 K	
C38	25 @ 25 v electrolytic	Fig. 7 (inside)	R58	4.7 M to 15 M (5)	
C39	0.5 to 2.7 @ 600 v (3)	Fig. 7 (inside)	R59	220 K	
C40	100 PF ceramic		R60	470 K	
C41	100 PF ceramic		R61	470 K	
C42	0.005 or .01 ceramic (2)		R62	470 K	
C43	Not present or 0.47 @ 200 v (4)	Early units, across R66 On U.V. Lamp Socket	R63	4.7 K	
C44	0.01 @ 1000 v ceramic		R64	470 K	
CHOPPER	Airmax 175 or eq.		R65	470 K	
CH01	Choke, 2.5 H @ 130 ma	Fig. 6 (outside)	R66	4.7 K	
CR01	1N1732 or eq.	Fig. 7 (inside)	R67	470 K	Early units, on socket of V-14
CR02	1N1732 or eq.	Near CR01			Fig. 7 (inside)
CR03	1N459 or eq.		R68	470 2W	Servo Gear Train
CR04	1N459 or eq.		R69	Variable, W.W. 5K 3T, linear (Servo Potentiometer)	
CR05	GEA70 (General Electric)	Fig. 7 (inside)	R70	90.9 K $\pm 1\%$	
CR06	Deleted	---	R71	Variable 20 K 2W linear "SPAY"	Fig. 6 (outside)
CR07	1N459 or eq.		R72	10 $\pm 1\%$	Fig. 7 (inside)
CR08	1N459 or eq.		R73	100 30W (V.I. 16)	Fig. 6 (outside)
CR09	1N2564 or eq.	Fig. 7 (inside)	R74	470 K (7)	
CR10	1N2564 or eq.	Fig. 7 (inside)	R75	300 20W W.W.	Fig. 7 (inside)
F01	Fuse, 2 amp, Slow Blow	Fig. 6 (outside)	S01	GKT #110-304A (8)	
J01	Connector, Female, 9 Pin	Fig. 7 (inside)	S02	Centralab #2502 "H.V."	Fig. 6 (outside)
J02	Connector, Female, 12 Pin	Fig. 7 (inside)	S03	SPST Toggle, 15A @ 125 v A.C. "SAMPLE"	Control Panel
J03	Connector, Female, 4 Pin	Cable end to optical comparator	S04	SPST Toggle, 15A @ 125 v A.C. "RECORD"	Control Panel
		Cable end to Ref. Photo- cell, V09	S05	SPST Toggle, 15A @ 125 v A.C. "POWER"	Control Panel
		Optical Comparator	S06	SPST Toggle, 15A @ 125 v A.C. "START"	Control Panel
J04	Connector, Female, 3 Pin			Spring return, normally open.	
M01	Motor, Light Interrupter, GKT #110-906		T01	Main Power, GKT #111-221	Fig. 6 (outside)
M02	Motor, Servo, GKT #111-104		T02	Triad S-78Z or eq. (9)	Fig. 6 (outside)
P01	Connector, Male, 9 Pin	Servo Gear Train	T03	Triad S-78Z or eq. (9)	Fig. 6 (outside)
P02	Connector, Male, 12 Pin	Mates J01	T04	Triad S-51X or eq. (9)	Fig. 6 (outside)
P03	Connector, Male, 4 Pin	Mates J02	TB01	3 connection terminal strip, center grounded	Fig. 7 (inside)
		Optical Comparator	TB02	Power distribution terminal board	Near T01
Q01	Transistor 2N217 or eq.		TB03	Mounts R-75	Fig. 7 (inside)
Q02	Transistor 2N217 or eq.		V01	Lamp, neon, NE-2	Near U.V. Lamp Socket
R01	100 K	On U.V. Lamp Socket	V02	Photomultiplier, GKT #110-346A	Optical Comparator
R02	1.2 M 2W	Fig. 7 (inside)	V03	Deleted	---
R03	100 K 2W	Fig. 7 (inside)	V04	OB2	
R04	1.2 M	Near R02	V05	Pilot Lamp, #44, 6.3 v @ 0.25A	Optical Comparator
R05	1.2 M	Near R04	V06	12AX7 (10)	
R06	1.3 M	Near R05	V07	6AU6	
R07	1.8 M	Near R06	V08	12AU7	
R08	100 K 2W	Fig. 7 (inside)	V09	Phototube, 927	Optical Comparator
R09	470 K $\pm 10\%$ 1/4 W	Top of Optical Comparator	V10	6AU6	
through			V11	12AU7	
R18			V12	Ultraviolet Lamp (11)	
R19	750 10W W.W.	Fig. 6 (outside)	V13	12AX7 (10)	
R20	6.8 K 1W	Fig. 7 (inside)	V14	6V6	
			V15	GV3A-900 or GV3A-1000 (12)	Mounted on S01

Note 1. Unless otherwise specified, all capacitors are tubular and in microfarads, and all resistors are in ohms, 1/2 w  $\pm 10\%$ .

Note 2. Replace with value installed, if required.

Note 3. Value selected to resonate associated transformer. Replace with value installed, if required.

Note 4. Chosen to properly phase servo amplifier. Replace with value installed, if required.

Note 5. Chosen to minimize servo overshoot. Replace with value installed, if required.

No.

Note 7. On underside of P.C. Board, early printed circuit instruments.

Note 8. Seal switch actuated by Sample Door Latch.

Note 9. Value of associated resonating capacitor will change, if transformer is damaged.

Note 10. Occasionally, a tube exhibiting high grid current will test O.K. but not operate satisfactorily.

Note 11. A number of lamps are available. See price list for details.

Note 12. Corona type voltage regulator, available from G. K. Turner Associates. Specify voltage rating! Chosen to match photomultiplier. Estimated life, indefinite.

**SELECTED TEST POINT VOLTAGES  
TURNER MODEL 111 FLUOROMETER  
(PRINTED CIRCUIT INSTRUMENTS)**

**Test Conditions:**

1. Line voltage, set at 115 volts.
2. General purpose U.V. lamp, #110-850, installed and operating.
3. Primary filter, color spec. #7-60, catalog #110-811, installed.
4. Secondary filter, color spec. #2A, catalog #110-816, installed.
5. Dummy cuvette, #110-840, installed. Blank knob set so fluorescence dial reads 20.
6. Sample door closed..
7. Instrument completely assembled except rear screen removed.
8. Voltmeter used which has a 20,000 ohms per volt D.C. or greater and 1,000 ohms per volt or greater A.C. sensitivity. VTVM where specified must have 10 megohm input impedance or higher.
9. All voltages are measured with respect to chassis ground.
10. A Test Adapter Set, such as Vector #T-789C, is used for voltage measurements on tubes.

Only a single adapter is installed at any one time. (Multiple adapters can lead to oscillation and false readings.)

**Test Points:**

1. All points called for are available on the printed circuit board, or at tube sockets.
2. Numbers with circles refer to corresponding numbers around the periphery of the printed circuit board.
3. All other numbers refer to tube pin numbers.

TABLE II. VOLTAGE CHART, Model 111

Test Point	Reading (Volts)		Test Point	Reading (Volts)	
①	2.8 to 3.2 A.C.		V10-1	-1.5 to -2.6 D.C.	VTVM
②	+190 to 210 A.C.		-5	+105 to 136 D.C.	
③	2.8 to 3.2 A.C.		-7	+0.3 to 1.5 D.C.	
⑥	+19 to 25 D.C.		V11-1	+190 to 210 D.C.	
	FLUORESCENCE dial <u>must be</u> at 20		-2	0 to -3.0 D.C.	VTVM
⑫	average under 0.1 D. C. Noisy. VTVM		-3	+6 to 8 D.C.	
⑳	+103 to +113 D.C.		V13-1	+50 to 105 D.C.	
㉑	+140 to 165 D.C.		-2	under 0.1	VTVM
㉓ Near 20	+65 to 78 D.C.		-3	+0.3 to 1.0 D.C.	
			-6	+55 to 92 D.C.	
V06-1	+105 to 145 D.C.		-7	under 0.1	VTVM
-2	-1 to -2.3 D.C.	VTVM	-8	+0.4 to 1.1 D.C.	
-6	-.5 to -2.1 D.C.		V14-3	+235 to 255 D.C.	
V07-1	under 0.1		-4	+250 to 275 D.C.	
-5	+95 to 115 D.C.		-5	under 0.1	VTVM
-7	+2.5 to 3.0 D.C.		-8	+13 to 16 D.C.	
08-1	+190 to 210 D.C.				
-2	under 0.1				
-3	+6.0 to 7.5 D.C.				
-6	+32 to 50 D.C.				
-7	-.4 to -1.8 D.C.	VTVM			

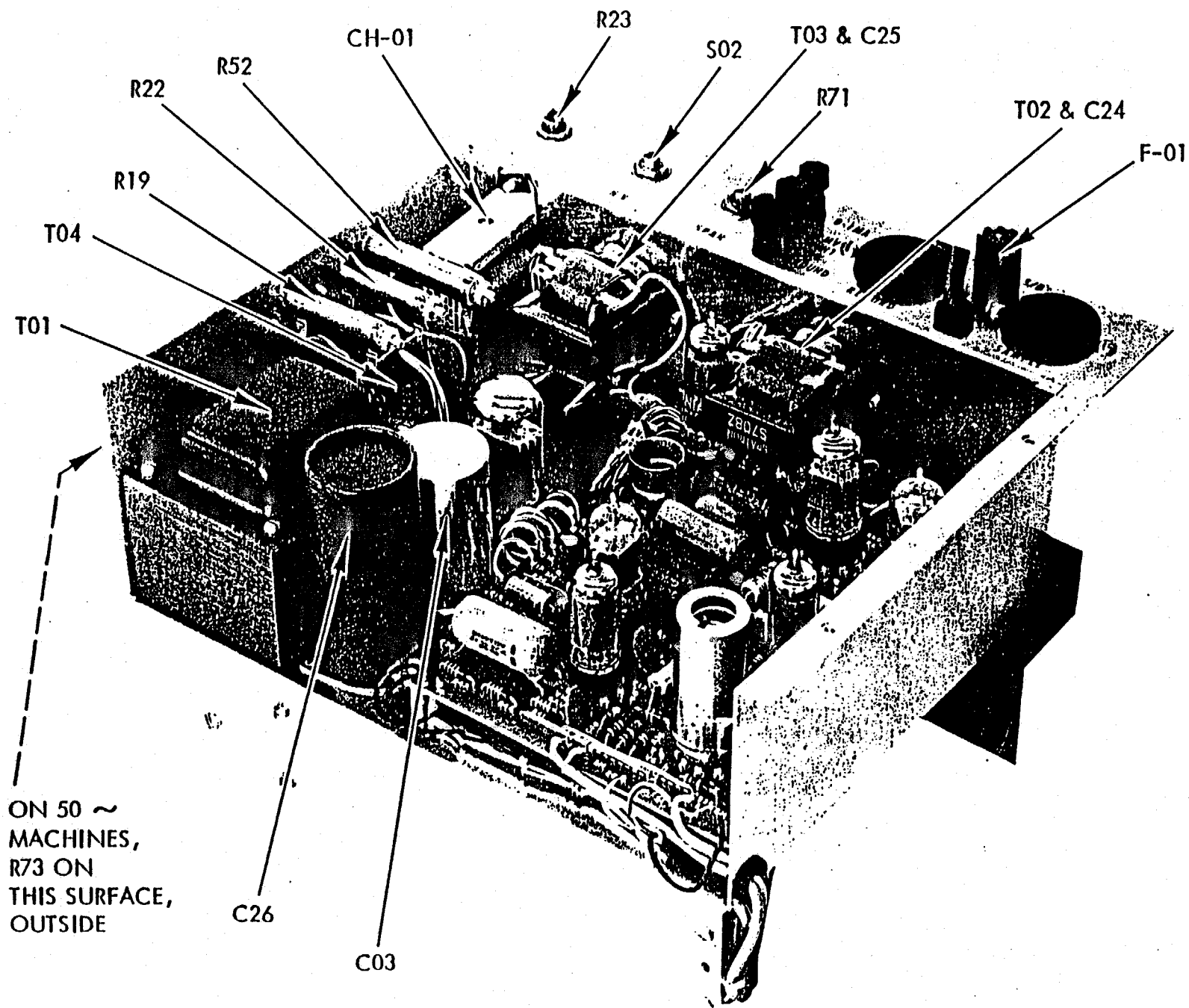


Fig. 6. Outside View, Electronics Chassis

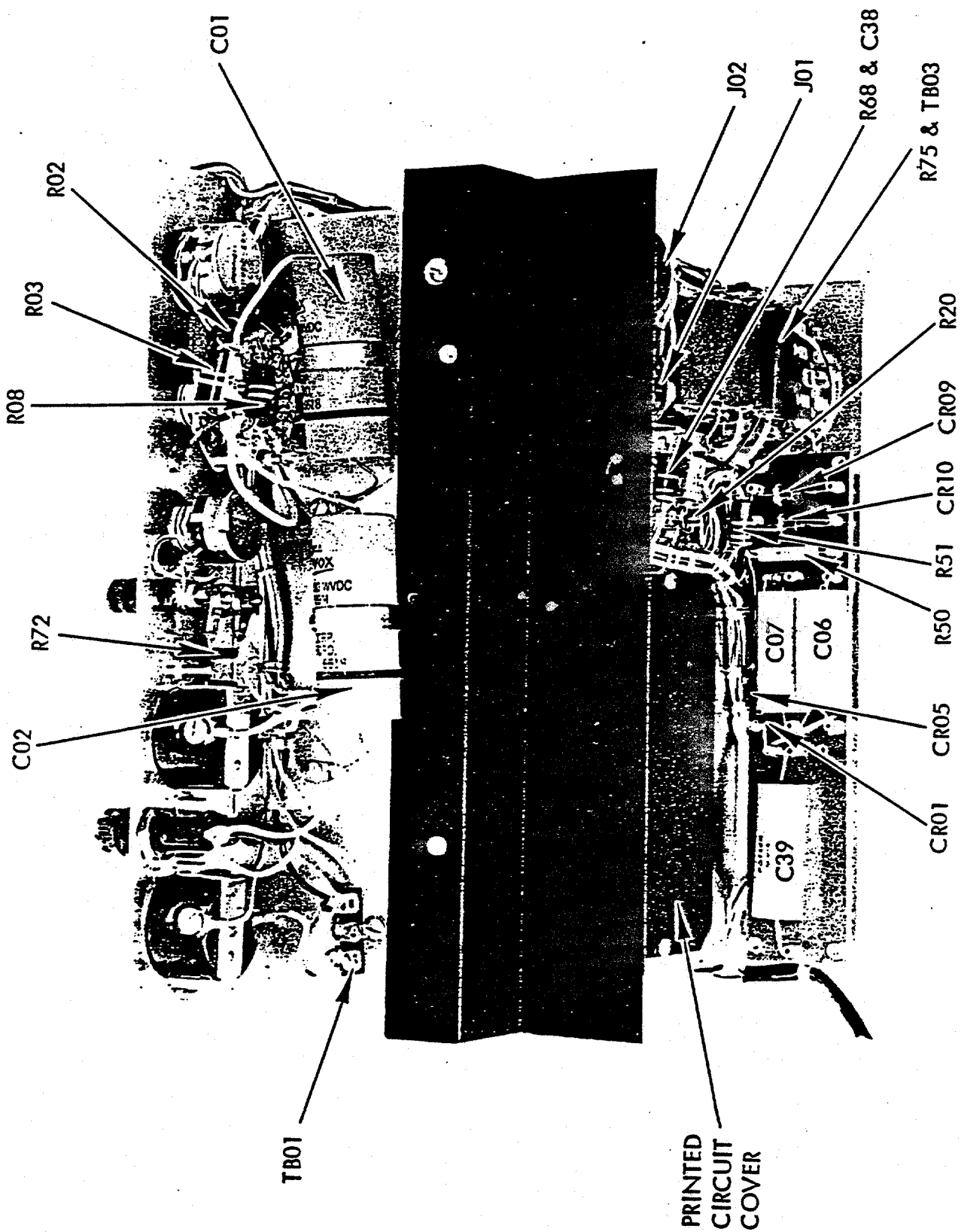


Fig. 7. Inside View, Electronics Chassis

## VII. SELECTION OF FILTERS AND LIGHT SOURCE

Analysts are frequently and justifiably confused about the proper choice of color filters in fluorometric analysis. It is not uncommon for several otherwise identical procedures to call for entirely different filters. This is not as impossible as it sounds. All of the combinations may be quite satisfactory. It happens occasionally, however, that very poor choices are made. These poor choices sometimes limit the practical sensitivity of the assay by accenting the fluorescence of the reagent blank compared to that of the sample. Frequently the filters were chosen solely on the basis of ready availability and are needlessly inefficient. A more serious error occurs when the primary and secondary filters overlap in their transmission characteristics, causing "cross-talk". The seriousness of this and its avoidance is discussed later.

The practice of fluorometry is basically quite simple. The fundamentals to be remembered are:

1. Some compounds have the ability to discharge energy obtained when they absorb light, by re-emitting light of a longer wavelength. The efficiency of this process may be anywhere from a fraction of a percent to 100%. In fluorometry a selected portion of this re-emitted light is measured.
2. The intensity of light emitted is proportional to the amount of exciting light absorbed. At low concentrations the absorbed light may be considered proportional to the concentration and to the intensity of exciting light. In practice, therefore, the dial reading of the fluorometer is linear with concentration of the fluorescent molecule involved. The range between the lowest detectable sample and the point where significant non-linearity occurs is normally a factor of  $10^4$ - $10^5$  in concentration of the fluorescent molecule involved.
3. If a compound is fluorescent, any wavelength of light absorbed will cause it to fluoresce. The spectrum of the emitted light is normally quite broad and its shape and location are independent of the wavelength of the exciting light. The only variation is in intensity - where less light (actually energy) is absorbed, less light is emitted. Proportionality of fluorescence and concentration are maintained even when the exciting light and measured fluorescent light are far removed from the peaks.

Why, then, does it matter what filters are used? A little thought will show that the choice of activating wavelength and wavelength measured can markedly affect the sensitivity and the specificity. The correct choice of filters minimizes the fluorescence of reagents (blank) and other compounds present, while retaining high sensitivity. It is frequently possible to exercise almost unbelievable selectivity in fluorescent analysis. The choice of filters is thus an important decision, but unlike in colorimetry where a peak or plateau must be used, it may be an empirical choice.

As might be expected, many fluorometric procedures are based on the older colorimetric procedures. The discovery that fluorescence accompanies the absorption of light of a particular compound or complex immediately opens the possibility of 100-1000 fold greater sensitivity. It also allows greater specificity by virtue of the freedom to select two, rather than a single filter, and that these two filters need not be at the excitation and emission peaks. Even more important, many interfering colored materials will not be fluorescent.

Confronted with the problem of devising a fluorometric assay, one may proceed in a fairly simple manner. The absorption spectrum of the compound is of value in the first choice of filters. Excitation with the mercury line closest to the major absorption peak will normally provide the greatest sensitivity, but for most practical purposes, excitation with the longest wavelength mercury line for which the compound exhibits significant absorption will usually give adequate sensitivity and, more important, greater selectivity.\* The choices, and means of obtaining them with the Turner Fluorometer, are discussed in Table III.

Having chosen a tentative primary filter, it requires but a few minutes to run through a series of "sharp-cut" secondary filters compatible with the primary filter, comparing the ratio of sample reading to reagent blank (see Method II, Data Reduction Section of the Turner Model 110 or 111 Operating Manual). If the data thus obtained indicate a fairly sharply peaked emission curve, the possibility of a band-pass secondary filter may be considered. Usually there is little advantage to a narrow-pass secondary filter, though they are occasionally of value where the emission peak falls at a wavelength shorter than 500 mμ.

The optimum secondary filter will normally be independent of the primary (activation) filter chosen and may then be compared for sample-to-blank ratio with other possible primary filters, to arrive at the best final combination.

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\*In those rare cases where one of the mercury lines is not satisfactory, other light sources are available from which, with appropriate filters, nearly any desired wavelength may be obtained. See fig. 9.

In choosing potential filter combinations, one must always pay heed to compatibility of the filters. By compatibility, it is meant that the transmission of the primary and secondary filters do not overlap significantly, else scattered light from even slight turbidity of the sample will reach the detection unit and register as fluorescence "cross-talk." Another potential source of error is fluorescence of the filters themselves: Fluorescence of a primary filter combination may result in light entering the sample which is not suspected from the transmission curve. Since this will frequently be of a longer wavelength than the intended activation light, it may, when scattered, pass the secondary filter. By the same token a fluorescent secondary filter will cause a positive error in a turbid sample. It should be emphasized that very little turbidity may be required to cause quite large errors when improper filters are used. The filter holder in the Turner Fluorometer was expressly designed to accept any 2" x 2" filter without special, expensive mounting, so that the best available filters for the purpose may be used.

The filters in the Turner Price List are a carefully chosen series, designed to cover a wide range of applications and which, by proper combination, may be used for perhaps 95% of the assays in the literature. For those rare instances where a special filter combination may be required, Turner Associates stocks for immediate shipment most of the Kodak Wratten filters and Corning glass color filters as well as some selected Schott and Ilford filters.

Interference filters may be used; however, many interference filters transmit a small amount of light at all wavelengths. This small side-band transmission with resulting overlap with the secondary filter may be sufficient to cause errors in samples with variable turbidity. In purchasing interference filters for a fluorometer, so state, and specify the filter with which it is to be paired, requesting full attenuation of transmission in the wavelength range of the other filter.

The characteristics of the light source are as important as those of the filters, both from the standpoint of what wavelength is being directed at the sample and from the standpoint of how much overlap may be tolerated between the primary and secondary filters. Figure 9 gives the (normalized) emission curves of the various light sources standardly available for your Model 110 or 111 Fluorometer. Those lamps designated as T-5 require the #110-856 adaptor for installation.

The vertical bars in fig. 9 are the emission lines of a low-pressure mercury arc lamp (#110-851). The line at 254 mμ is not given to scale. About 95% of the emission is at this wavelength. The other lamps all contain a phosphor which absorbs the 254 mμ line, converting the emission to a broad band of light as shown. The emission of the #110-850, -853, and -854 lamps have the mercury lines at 365, 405, 436, 546, and 578 mμ superimposed on the smooth curves given (no 254 mμ). The #110-855 lamp has, in addition to these, a much attenuated emission at 254 mμ. Additional lamps with emission in the red are available on special request.

Examination of the curves indicates there would be no point in using (for example) a 470 mμ filter with the #110-851 lamp, and very little light would be obtained with the #110-850 lamp. If the filter were broad enough, the light obtained would be principally in the region of 436 mμ, regardless of the fact that the filter peaks at 470 mμ. The #110-853 lamp would be the correct choice. As a further illustration, consider the #110-810 (Corning 7-34) filter. This transmits from 230 to 420 mμ, with a broad peak at 320 mμ. With the #110-851 lamp, the excitation will be effectively at 254 mμ; with the #110-853 lamp, at 310 mμ; and with the #110-850 lamp, at 360 mμ.

It may be readily seen that filter overlap in the 500 mμ region, for example, would be of minor consequence when using the #110-851 lamp, somewhat troublesome with the #110-850 lamp, and disastrous with the #110-853 lamp.

To assist you in selection of filter combinations two tables are appended. Table III lists some of the more commonly used activation wavelengths and the appropriate primary filters and light sources. Table IV lists the compatibilities of some TURNER primary and secondary combinations, principally with the #110-850 and #110-851 lamps. No attempt has been made to describe all situations, and where a light source has strong continuous emission in the region of interest, some of the combinations listed as compatible may be questionable, e.g., the 546 mμ primary filter may be used with the #110-824 filter in conditions of high turbidity with the #110-851 lamp, modest turbidity with the #110-850 lamp, but only clear solutions with the #110-854 lamp.

For understanding of the tables, two definitions are desirable:

1. The term band-pass in a filter is self explanatory. The filter passes light only in a given region of the spectrum. The wavelength noted in the tables is that of the peak transmission.
2. A "sharp-cut" filter passes all light of longer wavelengths than a given value and blocks all light of shorter wavelengths. The transition from zero to full transmission (normally 80-90%) usually takes place over a region of some 40 mμ. The characteristic wavelength of a "sharp-cut" filter, the "cut," is defined as the wavelength at which its transmission is 37%. With most manufacturers, there is about a 10 mμ tolerance in location of the "cut."

The photomultiplier in the Turner Fluorometer has an S-4 surface. (A red-sensitive photomultiplier is available on special order.) It is sensitive to wavelengths from 300 to 700 mμ, but falls off very rapidly above 600 mμ. When using "sharp-cut" filters, the characteristics of the photomultiplier thus set the longest wavelengths of emitted light which may be measured.

The identifying numbers marked on Turner filters are the color specification numbers of the original manufacture. The complete characteristics of the filters may be had by reference to Corning Bulletin CF-3 or to Kodak publication B-3 (available at camera shops). An exception, is the Turner #110-812 (405) filter which is a composite of a Corning 7-51 (1/2 thickness) and a Wratten 2C filter, the combination of which provides a band-pass filter useful in isolating the 405 mμ mercury line. A second exception is the #110-815 filter, especially designed to pass 254 mμ, but block wavelength between 300 and 420 mμ, which is manufactured in our plant.

If you have a special measurement problem and wish assistance in selection of appropriate filters and light source, please do not hesitate to write. Our staff is here to serve you and help you make the best use of your instrument.

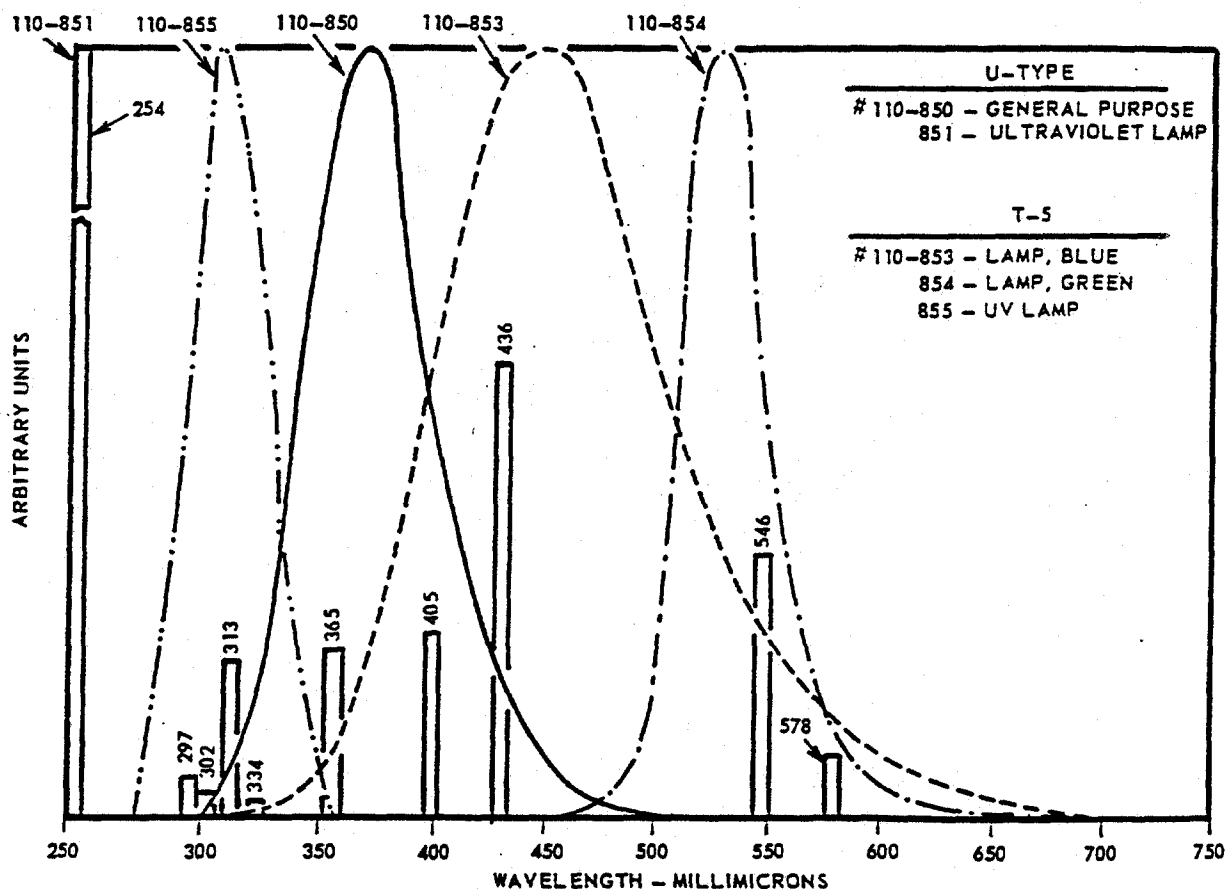


Fig. 9. Composite  $\lambda$  Range - Turner Light Sources



TABLE III. PRIMARY (ACTIVATION) FILTERS

<u>Wavelength (mμ)</u>	<u>Filter No.</u>	<u>Comments</u>
254	110-810 (7-54)	Passes also the 313,365 and part of the 405 mμ lines, which are relatively minor in the required 110-851 lamp. Quartz cuvettes (110-802) must be used.
254	110-810 (7-54) + 110-815	Provides pure 254 mμ activation when measurement in the near ultraviolet is required. Quartz cuvettes (110-802) and 110-851 lamp required. The #110-815 filter is short-lived. See Price List.
270-340	110-810 (7-54)	With #110-855 lamp, filter #110-810 isolates a band of light peaking at 310 mμ (compatible only with emission wavelengths larger than 420 mμ). Within the 270-340 mμ region an interference filter may be used to isolate any desired band of light from the #110-855 lamp. Quartz cuvettes (110-802) must be used below 325 mμ.
325	110-810 (7-54) + Wratten 34A (unmounted) 110-836	Gelatin held in place by the Corning filter. Used with the standard (110-850) lamp to provide a narrow band of activating light peaking at 325 mμ. With the 110-851 lamp it isolates the 313 mμ line. In neither case is the activating light very intense, the former being generally best, but it is suitable for a great deal of work. Pyrex cuvettes are generally satisfactory.
365	110-811 (7-60) or 110-834 (7-37)	The general purpose primary filter supplied with the Turner Fluorometers. Normally used with the standard (110-850) lamp and pyrex cuvettes. 110-834 recommended for solid samples or paper chromatogram door.
405	110-812 (405)	Should be used with the dark Corning 7-51 glass <u>away</u> from the light as the Wratten 2C is slightly fluorescent and must be blocked. Used with either 110-850 or 110-851 lamp (about 2-fold gain in sensitivity with latter) and pyrex cuvettes.
405 + 436	110-813 (47B)	Seldom used alone, but for some applications where either 405 or 436 may be used, provides increased sensitivity by providing both. As with the 405 filter it is normally used with 110-850 lamp, but the 110-851 lamp provides increased sensitivity. Used with pyrex cuvettes.
436	110-816 (2A) + 110-813 (47B)	The 2A is placed nearest the lamp. The 2A eliminates the 405 mμ line. Other comments as for 405.
470	110-827 (3) + 110-831 (48)	Used only with 110-853 blue lamp.
546	110-814 (1-60) + 110-822 (58) or 110-832 (546)	The 1-60 is placed nearest the lamp. Other comments as for 405. 110-832 recommended for tracer work with Rhodamine B or Pontacyl Brilliant Pink B or for best results with paper chromatogram door.

TABLE IV. COMPATIBILITIES OF FILTERS

	Secondary Filters	PRIMARY FILTERS						(See Table I)	(See Table I)	1-60 + 58 546 mμ
		7-54 254 mμ	7-54 + 815 pure 254 mμ	7-54 + 34A 325 mμ	7-60 365 mμ	7-37 360 mμ	405 405 mμ	47B + 2A 436 mμ	3 + 48 470 mμ	3 + 48 + 48 470 mμ
SHARP CUT SERIES	110-816 (2A) 415 mμ		X	X*	X	X				
	110-827 (3) 455 mμ	X	X	X	X	X	X			
	110-828 (4) 465 mμ	X	X	X	X	X	X			
	110-817 (8) 485 mμ	X	X	X	X	X	X			
	110-818 (2A-12) 510 mμ	X	X	X	X	X	X			X*
	110-826 (2A-15) 520 mμ	X	X	X	X	X	X	X	X*	X
	110-829 (16) 535 mμ	X	X	X	X	X	X	X	X	X
	110-819 (22)** 560 mμ	X	X	X	X	X	X	X	X	X*
	110-824 (23A) 570 mμ	X	X	X	X	X	X	X	X	X
	110-820 (25) 595 mμ	X	X	X	X	X	X	X	X	X
	110-810 (7-54) + Wratten 34A (110-936) 325 mμ		X							
	110-811 (7-60) 360 mμ		X							
	110-812 405 mμ		X	X*	X*	X				
	110-813 (47B) 430 mμ		X	X*						
	110-813 (47B) + 110-816 (2A) 435 mμ	X*	X	X*	X	X	X*			
	110-816 (2A) + 110-831 (48) 460 mμ	X*	X	X*	X	X	X*			
NARROW PASS SERIES	110-825 (65A) 495 mμ	X	X	X	X†	X†	X			
	110-825 (65A) + 110-817 (8) 510 mμ	X	X	X	X	X	X	X*		
	110-822 (58) 525 mμ	X	X	X	X	X	X	X		
	110-822 (58) + 110-818 (2A-12) 535 mμ	X	X	X	X	X	X			X*
	110-822 (58) + 110-826 (2A-15) 540 mμ	X	X	X	X	X	X	X	X	
	110-822 (58) + 110-814 (1-60) 545 mμ	X	X	X	X	X	X	X	X	
	110-833 590 mμ	X	X	X	X	X	X	X	X	X

X Indicates compatibility  
 \* Use with care as slight overlap exists  
 \*\* This filter slightly fluorescent  
 † Add #110-816 (2A) to 110-825 (65A)

**APPENDIX D**

**Addendum to Health and Safety Plan  
for the Post-Construction Remnant Deposit Monitoring Program**

**GENERAL ELECTRIC COMPANY  
1995 HUDSON RIVER PROJECT  
PCRDMP HEALTH AND SAFETY PLAN  
ADDENDUM**

**1. Introduction**

This Health and Safety Plan (HASP) addendum has been prepared to describe health and safety procedures to be used by O'Brien & Gere employees while conducting field activities on the Hudson River during the 1995 research and monitoring program. The 1995 program will follow the HASP developed for the Post-Construction Remnant Deposit Monitoring Program (PCRDMP). However, some of the activities are not explicitly included in the Field Sampling Plan (FSP; O'Brien & Gere 1992a) or the HASP (O'Brien & Gere 1992b) developed for the PCRDMP. This HASP addendum specifically addresses health and safety procedures to be followed while performing research investigations in 1995 which consist of dye studies and transect investigations as described in the Field Sampling Plan (O'Brien & Gere 1995). This HASP addendum is not intended as a stand alone document, rather it must be used in conjunction with the HASP developed for the PCRDMP (O'Brien & Gere 1992).

**2. Project personnel**

<u>Project Officer</u>	J.K. Farmer
<u>Project Manager</u>	J.R. Rhea
<u>Company Safety and Health Officer</u>	S.E. Wilson
<u>Site Safety and Health Coordinator</u>	W.A. Ayling

**3. Health and safety hazards**

No changes.

**4. Personal protective equipment**

Rubber gloves and eye protection will be worn during mixing and handling of the concentrated dye and fluorescent resin particle suspensions.

**5. Site activities and associated personal protective requirements**

No changes, except as described in item 4 above.

**6. Site air monitoring**

Air monitoring will not be conducted.

## **7. Action levels**

Decontamination procedures will be performed in a well ventilated area. Workers should be aware of vapor hazards and temporarily discontinue solvent use should vapor dispersion be insufficient.

## **8. Site access and control**

No changes.

## **9. Medical monitoring**

No changes.

## **10. Personnel training**

No changes.

## **11. Decontamination**

No changes.

## **12. Emergency response**

A summary of emergency contacts is provided in Table 1. This table will be retained in the field by the Field Coordinator.

## **13. Special precautions and procedures**

### Boat safety

Some of the activities at Thompson Island Pool will be conducted at night. During daylight hours before nighttime field activities begin, field personnel will familiarize themselves with the region of the river they will be working in. A guide light will be located at each shore. An on-board spot light will be used to assist boat navigation.

## **References**

O'Brien & Gere Engineers, Inc. 1992. Health and Safety Plan, Post- Construction Monitoring Program, Fort Edward Dam PCB Remnant Deposit Containment. June 1992.

O'Brien & Gere Engineers, Inc. 1992 Field Sampling Plan, Post-Construction Monitoring Program, Fort Edward Dam PCB Remnant Deposit Containment. June 1992.

### Table

#### **1. Emergency Contacts**

TABLE 1  
GENERAL ELECTRIC COMPANY  
1995 HUDSON RIVER PROJECT  
PCRDMP Health and Safety Plan Addendum

Emergency Contacts

Contact	Phone number
Emergency	911
Fire Department (Fort Edward)	518-747-3325
Hospital (Glens Falls)	911
Police (sheriff)	518-747-4011
General Electric Company (John Haggard)	518-458-6619
Poison Control Center	800-336-6997
Chemical Emergency Advice (CHEMTREC)	800-424-9300
NYS Thruway Lock 7 Fort Edward	518-747-4614
O'Brien & Gere Engineers, Inc.	315-437-6100
New York State Department of Environmental Conservation	518-668-5441