Pesticide Analytical Manual Volume I

9/96 Revisions

The following pages contain corrections or changes for PAM I. Print these pages and use them to replace the same current pages in PAM I 3rd edition (published Jan., 1994).

Each set of two pages is intended to appear on two sides of the same paper, but Acrobat Reader does not offer a feature that facilitates printing on both sides of the page. It may be necessary to print one page at a time and turn the paper over to print the second page on the reverse side.

Material Transmitted	PAM I Page	Page of this File
1. Title Page and Masthead	i, ii (not numbered on page)	2, 3
2. Introduction	v, vi	4, 5
3. Section 102, pages 3 and 4	102-3, 102-4	6, 7
4. Section 105, pages 3 and 4	105-3, 105-4	8, 9
5. Section 204, pages 7 and 8	204-7, 204-8	10, 11
6. Section 404, pages 11 and 12	404-11, 404-12	12, 13
7. Section 605, pages 5 and 6	605-5, 605-6	14, 15
8. Appendix II, Report Form C	Appendix II-17 and Appendix II-18	16, 17

Explanations of changes:

Title and masthead reflect this current revision.

Introduction includes information about the FDA web site and the electronic files now available.

Table 102-b, page 102-4, contains a new sentence to clarify that brine is considered inedible and that it and other inedible media are discarded during preparation of processed food.

Table 105-a, page 105-4, corrects a previously inaccurate limit of quantitation (Lq) for MBC.

Paragraph 5, page 204-7, corrects a previously inaccurate statement about the eluant in which malathion elutes from Florisil.

Figure 404-b, page 404-12, corrects a previously inaccurate label for thiabendazole in both “B” chromatograms.

Figure 605-c, page 605-6, corrects a previously inaccurate label for the excitation filter.

Reporting Form C, Appendix II-17, clarifies a previously confusing entry area.

PESTICIDE ANALYTICAL MANUAL

VOLUME I: Multiresidue Methods

U.S. Department of Health and Human Services • Public Health Service
Food and Drug Administration

PESTICIDE ANALYTICAL MANUAL VOLUME I

3rd Edition, 1994

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PESTICIDE ANALYTICAL MANUAL

INTRODUCTION

The Food and Drug Administration (FDA) is responsible under the Federal Food, Drug, and Cosmetic Act for enforcing tolerances established by the Environmental Protection Agency (EPA) for amounts of pesticide residues that may legally remain on food (including animal feed). In meeting this responsibility, FDA collects and analyzes food from commercial channels of trade for determining compliance with EPA tolerances. The residue data gathered under this regulatory monitoring program are also used for evaluating the extent and significance of pesticide residues in the food supply.

The Pesticide Analytical Manual (PAM) is published by FDA as a repository of the analytical methods used in FDA laboratories to examine food for pesticide residues for regulatory purposes.[1] The manual is organized according to the scope of the analytical methods:

Volume I contains multiresidue methods (MRMs) that are used by FDA on a routine basis, because of their efficiency and broad applicability, especially for analyzing foods of unknown pesticide treatment history.

Volume II contains methods designed for the analysis of commodities for residues of only a single compound (although some methods are capable of determining several related compounds). These methods are most often used when the likely residue is known to the chemist and/or when the residue of interest cannot be determined by common MRMs.

PAM is designed to be used by analysts experienced in trace residue analysis. All of the techniques employed are subject to potential interferences from reagents, apparatus, containers, contaminated air supply, and handling by personnel. The experienced analyst is alert for these possibilities and recognizes the need to confirm results by other techniques that measure different chemical or physical properties of the analyte.

Experienced residue analysts are aware that no report of validation in another laboratory can substitute for verification that the method does indeed work in the analyst's own laboratory. The analyst should verify method performance in each particular application by a trial of the method that includes examination of reagent and sample blanks and measurement of the recovery of added analyte. The editors invite analysts to report results of their experiences with PAM methods.

Revisions

Starting with transmittal 96-1 (9/96), revisions of PAM I will be issued in two ways: (1) changes in most manual sections will be distributed as hard (paper) copies, with symbols ▶ or ◀ marking lines that have been changed, and (2) updates to

[1] 40 CFR 180.101 (c)

the tables in Chapters 3 and 4, Appendix I, and the indices to methods, names, and CAS Registry numbers will be issued only *via* Internet. No hard copies will be distributed for the latter updated sections, but updates will be available more frequently than in the past.

Chapter tables of contents will include the date on which each section within the chapter was transmitted; dates associated with those sections distributed only electronically will reflect the most recent version at the time the table of contents issued.

Internet Access to PAM I Files

PAM I is now available *via* Internet as Adobe Acrobat "portable document format" (pdf) files. Pdf format permits the user to read and print the document from any computer using appropriate free software.

To obtain a copy of PAM I files, use one of these routes:

1) Go to the World Wide Web site at: http://vm.cfsan.fda.gov. Follow these links: Center for Food Safety and Applied Nutrition/Pesticides and Chemical Contaminants/ Pesticide Analytical Manual. A link on the resulting page displays the PAM I Table of Contents with links to currently available files. Follow the instructions for downloading.

2) FTP to ftp.cfsan.fda.gov. Go to the directory public/pam. Download ReadMe.txt and other file(s) as desired; use ascii protocol during download of ReadMe.txt and binary protocol for pdf files.

Adobe Acrobat Reader is required to view and print pdf files. (ReadMe can be read with any word processor.) Download a copy of this free software from Adobe's web site at http://www.adobe.com/acrobat/readstep.html. A link to that site is provided on the PAM I page. Choose the version of Acrobat Reader appropriate to your own computer system.

Peanuts	Whole peanut meat (kernel) after removing hulls.
Peanut hulls	Whole commodity after removing peanut meat.
Dates and olives	Whole commodity after removing and discarding stems and stones or pits.
Pineapples	Whole commodity after removing and discarding crowns (leaves at top of fruit).
Avocados and mangoes	Whole commodity after removing and discarding stones.
Bananas	Whole commodity including peel after removing and discarding crown tissue and stalk.
Miscellaneous raw fruits and vegetables not previously included	Whole commodity after removing and discarding obviously decomposed or withered leaves, stems, stones or pits, shells or husks; if commodity has adhering amounts of soil, remove by lightly rinsing in running water.
Almond hulls	Whole commodity after removing shell and nutmeat.
Cereal grains group	Whole commodity (grain) except for fresh corn (including sweet corn). Include kernels plus cob after removing and discarding husk.
Eggs	Whole commodity after removing and discarding shells.
Fish	Edible portion of the commodity after removing and discarding heads, tails, scales, fins, viscera, bones (if inedible), and skin (if inedible).
Crab (hard shell)	Edible portion of commodity after removing and discarding shells, gills, and viscera.
Crab (soft shell)	Edible portion of commodity after removing and discarding gills.
Shrimp and crayfish	Edible portion of commodity after removing and discarding heads, shells, and inedible tails of shrimp.
Lobster	Edible portion of commodity including tomalley (liver) after removing and discarding shells and stomachs (hard sac near head).
Oyster, clam, and other shellfish	Edible portion of commodity including the liquor, after removing and discarding shells.
Rabbits and other game	Edible portion of commodity after removing and discarding bones.

Processed Foods

In the absence of EPA regulations, FDA also developed the instructions listed in Table 102-b on the portion of processed food to be analyzed for tolerance enforcement purposes. These instructions, like the ones for raw agricultural commodities, ensure uniformity and consistency in FDA analysis of processed food for pesticide residues. The instructions take a practical approach for sample preparation of processed food; *e.g.*, fruit juice concentrates that are normally reconstituted before consumption are also reconstituted prior to analysis for pesticide residues. Therefore:

- Follow the directions in Table 102-b to prepare test samples of processed foods.

Table 102-b: Portion of Processed Food to be Analyzed for Pesticide Residues

Processed food consisting of one ingredient and sold in a ready-to-eat form (*e.g.*, canned fruits packed in syrup or their own juice, canned vegetables packed in water or brine, or frozen fruits or vegetables, dried fruits, single-strength juices, catsup)	Analyze the whole processed commodity including any liquid or other edible media in which the commodity is packed. Discard inedible media, *e.g.*, brine.
Processed food consisting primarily of one ingredient and sold in a form requiring further preparation before it is ready to eat (*e.g.*, fruit juice concentrates, dehydrated vegetables, and powdered potatoes)	Analyze the whole processed commodity after compensating for or reconstituting to the commodity's normal moisture content.
Processed food in a form not ready to eat, used as an ingredient or component of other food (*e.g.*, flour, tomato concentrates such as paste, and citrus oils)	Analyze the whole processed commodity on an "as is" basis.
Cheese	Analyze the whole commodity including natural cheese rind after removing and discarding waxed or oiled rinds.
Frozen seafood (*e.g.*, fish or shrimp)	Analyze the edible portion after thawing; discard water.
Canned seafood	Analyze the edible portion including edible liquor and media, such as oil, broth, or sauces in which commodity is packed. Discard media that is not edible.
Frog legs	Analyze the edible portion of commodity after removing and discarding bones.

1) Determinative step sensitivity to any particular residue. A distinct Lq applies to each residue determinable by a particular MRM, because the sensitivity of the determinative step to each compound may be different.

2) Limited detector sensitivity. Not all individual detectors are capable of reaching the sensitivity specified; in such cases, the Lq will be higher than targeted.

3) Greater detector sensitivity. Directions here recommend sensitivity at which detectors should be operated, even though some are capable of greater sensitivity. However, operation at conditions that produce recommended sensitivity may sometimes be precluded by other disadvantages in detector performance. For example, many models of ^{63}Ni electron capture detectors are not linear at conditions that produce sensitivity of 50% FSD to 1.5 ng chlorpyrifos, as is recommended for other detectors; most are linear, however, at conditions that produce 50% FSD to 0.15 ng chlorpyrifos. The rules in Section 105 C specify that, in this situation, the laboratory should operate at the greater sensitivity in order to work in a linear range, then proportionately reduce the weight of sample equivalent injected in order to maintain Lqs consistent with those achieved by other laboratories.

4) Other improvements that affect determinative step. Wide bore capillary GLC columns (Section 502 C) permit analytes to elute in a tighter band than was possible with packed column chromatography. When detector response is measured in terms of peak height, use of capillary columns results in an apparent improvement of response. Injection of a smaller amount of equivalent sample, as directed in Section 105 C, is appropriate and, at the same time, beneficial to the longevity of the column.

5) Excessive interferences from sample co-extractives. Interferences from sample co-extractives raise the Lq of a method by masking the detector response to the residue or by preventing injection of the specified sample equivalent without undesirable damage to the system. Additional procedures to clean up the sample extract prior to determination may improve the Lq by removing these interferences.

Table 105-a: Examples of Method Specifications Used to Calculate Lqs

PAM I Method[1]	Recommended Mg Injected	Recommended Sensitivity[2]	Lq (marker compound)[3]
302 E1+DG2 (FPD-P)	20 mg	1.5 ng chlorpyrifos	0.015 ppm chlorpyrifos
302 E3+C1+DG3 (ElCD-X)	20 mg	1.5 ng chlorpyrifos	0.015 ppm chlorpyrifos
302+E1+C3+DL1	116 mg	10 ng carbofuran	0.017 ppm carbofuran
303 E1+C1+DG1 (EC)	20 mg 2 mg	1.5 ng chlorpyrifos 0.15 ng chlorpyrifos	0.015 ppm chlorpyrifos 0.015 ppm chlorpyrifos
304 E4+C2+DG1 (EC)	10 mg (cheese with 30% fat)	1.5 ng chlorpyrifos	0.03 ppm chlorpyrifos, whole product basis
401 E1+C1+DL1	200 mg	10 ng carbofuran	0.01 ppm carbofuran
402 E1+C1+DG3 (fatty foods)	5 mg Eluate 1	1.5 ng chlorpyrifos (0.2 ng PCP methyl ether)	0.008 ppm PCP methyl ether
	10 mg Eluate 2	1.5 ng chlorpyrifos (0.5 ng 2,4,5-T methyl ester)	0.01 ppm 2,4,5-T methyl ester
402 E2+C1+DG3 (nonfatty foods)	10 mg Eluate 1	1.5 ng chlorpyrifos (0.2 ng PCP methyl ether)	0.004 ppm PCP methyl ether
	20 mg Eluate 2	1.5 ng chlorpyrifos (0.5 ng 2,4,5-T methyl ester)	0.005 ppm 2,4,5-T methyl ester
403 E1+C1+DL3	800 mg	40 ng diuron	0.01 ppm diuron
▶ 404 E1+DL5	125 mg	62.5 ng MBC	0.1 ppm MBC
404 E1+DL7	125 mg	6.25 ng thiabendazole (fluorescence detector)	0.01 ppm thiabendazole

[1] Parenthetical codes indicate the detector used in the GLC determinative step.
[2] Ng marker compound that causes detector response of 50% FSD; where residues targeted by the method are different from the marker compound, weight of example target that caused 50% FSD is also listed.
[3] Calculated by formula in Section 105 B; note that sensitivity is divided by 5 to produce ng causing 10% FSD.

- Elute each column with 200 mL 6% ethyl ether/petroleum ether. (Collect rinses with this eluate.)

- Change receivers; elute each column with 200 mL 15% ethyl ether/petroleum ether.

- Change receivers; elute each column with 200 mL 50% ethyl ether/petroleum ether.

- Concentrate each eluate, dilute to volume with hexane, and inject about 5 µL into appropriate GLC systems to determine recoveries. Dilute 1.0 mL each standard solutions A and B to 10 mL and use diluted solution as GLC reference standard.

- Consider Florisil lot acceptable if one of three columns permits complete recovery of test compounds and exhibits proper elution pattern (heptachlor, heptachlor epoxide, chlorpyrifos, and fonofos in 6% eluate; dieldrin, endosulfan I, parathion-methyl, and pirimiphos-methyl in 15% eluate; malathion and endosulfan sulfate in 50% eluate; and endosulfan II in both 15 and 50% eluates). Acceptable recovery is >80% for all compounds except heptachlor, and 60-90% for heptachlor. In subsequent use of lot of Florisil, use same weight as that in column with acceptable elution. ◀

- If none of the three columns exhibits proper elution but a consistent relationship exists between weight and elution, test additional columns of weights 3 g above or 3 g below that calculated using LA Value. If these columns also do not exhibit proper elution, it is best to use a different lot of Florisil.

If acceptable weight of Florisil is determined, test that column size further with following procedures:

- Repeat elution tests above, using 1.0 mL each solutions C and D. Elute column with 250 mL petroleum ether, followed by 6, 15, and 50% ethyl ether/petroleum ether eluants; collect each eluate separately. Determine recoveries of pesticides and verify accuracy of elution pattern using gas chromatographic measurement.

- Transfer each eluate quantitatively to separate tared 20 mL beaker. Evaporate solvent on steam bath or hot plate until constant weight is attained to measure amount of butterfat recovered in each eluate. Acceptable lots of Florisil typically permit about 0.3 mg (range 0-1.7 mg) butterfat to elute in petroleum ether eluate, 0.1 (0-0.4) mg in 6% ethyl ether/petroleum ether, 82 (40-135) mg in 15%, and 105 (60-172) mg in 50%.

- Repeat elution tests above, using 1.0 mL each solutions A and B and eluting with Eluants 1, 2, and 3 instead of ethyl ether/petroleum ether eluants.

It is acceptable, once the Florisil lot has been tested and appropriate weight of Florisil determined, to measure and record height of column produced by specified weight; subsequent columns may then be prepared by measuring height rather than weight.

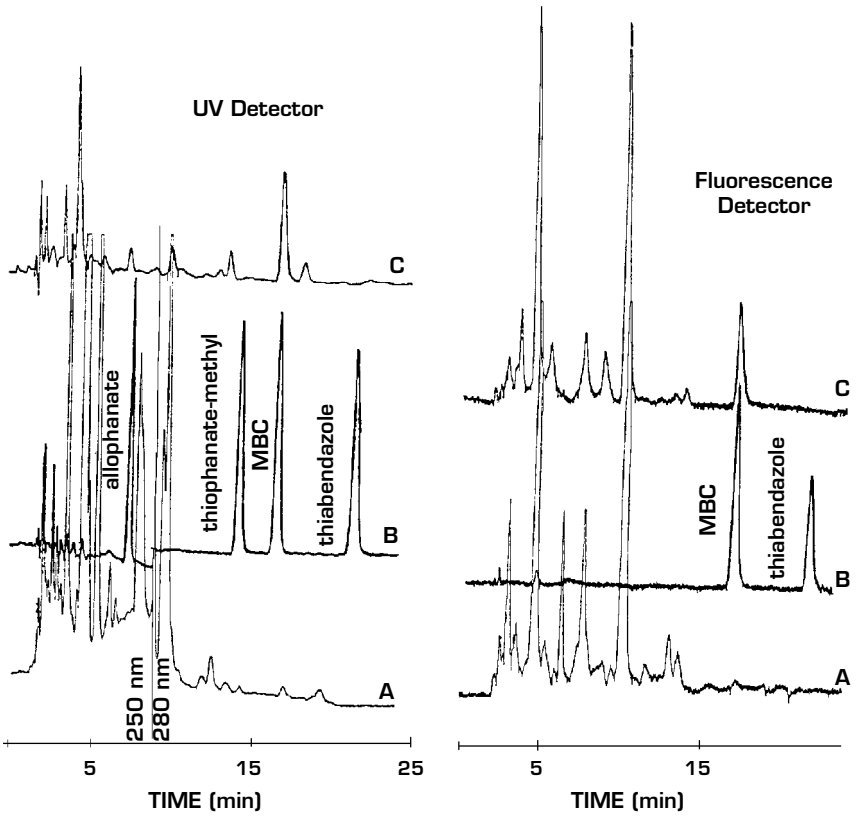
- Inject at least three 25 µL portions 2.5 ng/µL mixed standard solution. Determine following parameters:
  1) retention time and peak height for each peak; relative standard deviations (RSD) for repetitive retention times and peak height measurements
  2) column efficiency (N) for thiabendazole peak
  3) asymmetry factor (As) for thiabendazole peak
- Inject each of three different concentrations of mixed standard solutions (10-100 ng/25 µL injection). Plot peak height vs. amount injected.
- HPLC systems adequate for analysis for benzimidazoles will meet following minimum criteria: retention times of about 8, 15, 18, and 23 min for allophanate, thiophanate-methyl, MBC, and thiabendazole, respectively (retention times may vary among columns but should remain constant for particular column); RSD <0.5% for retention times and <3% for peak heights of individual peaks in three consecutive chromatograms; N>12,000 and As <1.3 for thiabendazole peak.
- Examine systems not meeting these criteria for problems, using various troubleshooting sections of Chapter 6. Correct problems uncovered by troubleshooting until system meets criteria defined above.
- System will typically respond linearly to 10-100 ng of each compound, but linear range may vary among systems. Perform quantitative analyses only within calculated linear range of system as determined above. Dilute sample extracts as needed to permit injection of analyte level within linear range. Adjust amounts injected so that peak heights of analyte and reference standard do not differ >25% from one another.

Directions

See Figure 404-b for typical chromatograms produced by HPLC system.

- To extract from E1-E3 (dissolved in 4.0 mL methanol), add 6.0 mL ion pairing solution; mix. Residue *must* be dissolved in methanol prior to adding ion pairing solution.
- Filter through 0.45 µm porosity membrane; filter will plug as solution is applied, so filter only volume needed for HPLC determination, about 1 mL.
- Inject 25 µL sample solution and chromatograph as described in System Operation.
- Compare chromatographic response (peak retention times, heights, and/or areas) with that of standard solution and calculate residue amount.
- If further dilutions are necessary, use mixture of 4:6 methanol:ion pairing solution as diluent.
- To convert calculated MBC (MW 191.2) to equivalent benomyl (MW 290.4), multiply by 1.52.
- To convert calculated MBC to equivalent thiophanate-methyl (MW 342.4), multiply by 1.79.
- Peaks of 50% FSD at conditions established for screening analysis are equivalent to about 0.5 ppm each of thiophanate-methyl, allophanate, and thiabendazole; MBC peak of 50% FSD at these conditions represents about 0.3 ppm.

Figure 404-b
Chromatograms of Benzimidazole Compounds



Chromatograms of: (A) peach extract partitioned from the acidic phase of 404 E1, (B) standard solution, (C) peach extract partitioned from basic phase of 404 E1. HPLC operation as directed in DL5. Sample contains 0.14 ppm field-incurred MBC.

ALTERNATIVES:

DL6 HPLC, CONCENTRATED ION PAIR MOBILE PHASE, UV AND FLUORESCENCE DETECTOR

Reference

Gilvydis, D.M., and Walters, S.M. (Aug. 1989) "Modification of LIB 3217 for Carbendazim (MBC) in Green and Roasted Coffee Beans," LIB 3353, FDA, Rockville, MD

Principles

Concentration of ion pairing reagent is increased eight times to increase k′ values of analytes and improve separation from early eluting co-extractives.

Additional Reagents

ion pairing solution, 32.7 mM 1-decanesulfonate, sodium salt. Pipet 7.0 mL phosphoric acid into 200 mL HPLC grade water; dissolve 8.0 g 1-decanesulfonate, sodium salt in this mixture. Pipet 10.0 mL triethylamine into solution and dilute to 1 L with HPLC grade water. Filter through <1 µm porosity membrane. (pH of solution should be about 2.4.)

Gradient elution is possible provided the solvents do not absorb. At very sensitive settings, changes in RI, as caused by gradient elution or pressure and flow changes, can produce baseline shifts with some types of detector cells.

The fixed wavelength detector is less versatile but is much less expensive and often gives less noise than the continuously variable wavelength spectrophotometric detector. As mentioned above, the great advantage of the variable wavelength detector is the ability to optimize sensitivity and/or selectivity for each analyte by detection at the most favorable wavelength.

Multichannel or Photodiode Array Detectors

In a photodiode array detector, polychromatic radiation is passed through the detector flow cell, and emerging radiation is diffracted by a grating so that it falls on an array of photodiodes. Each diode receives a different narrow wavelength band. The complete array of diodes is scanned by a microprocessor many times a second. The resulting spectra may be displayed on a cathode ray tube monitor and/or stored in the instrument for transfer to a recorder or printer. The detector is best used in conjunction with a computerized data station, which allows various post-run manipulations, such as identity confirmation by comparison of spectra with a library of standard spectra recalled from disk storage. Detection can be made at a single wavelength or at a number of wavelengths simultaneously, or wavelength changes can be programmed to occur at specified points during the run. Absorbance ratios at selected wavelengths (*e.g.*, 254 and 280 nm) can be displayed for each peak, which aids in determining identity and the presence of unresolved components.

Applications

The UV detector has been the most widely used for pesticide residue determination. Section 404 uses UV and fluorescence detectors to determine benzimidazole residues, whereas other references describe combinations of UV and photoconductivity [1-3]; the photodiode array is applicable to determining paraquat and diquat [4].

Problems, Maintenance, and Troubleshooting

Air bubbles in UV flow cells can produce a series of very fast noise spikes on the chromatogram, or pronounced baseline drift. Falsely high absorbance readings can be caused by impure or improperly prepared mobile phase, large air bubbles in the flow cell, a misaligned flow cell, or dirty end windows. Gas bubbles develop in the detector cell because they are pumped through the system or the solvent is degassed in the detector. Prevent bubbles from being pumped through the system by eliminating system leaks, expelling air from the pumping system, avoiding very volatile solvents, and not stirring the mobile phase reservoir too vigorously. Prevent solvent degassing in the sample cell by degassing the mobile phase prior to use. If the cell has no back pressure valve, raise cell pressure above atmospheric by attaching ≥10' spiral steel or Teflon tubing to the detector outlet to act as a flow restrictor, and placing the tubing outlet above the detector. The tubing must not shut off flow completely, as too great a pressure increase could shatter the cell windows.

To dissolve gas bubbles lodged in the cell, briefly increase cell back pressure by holding a piece of rubber septum over the detector outlet or by connecting a syringe to the outlet. With aqueous systems, it may be necessary to fill the cell with methanol and repeat application of back pressure.

Protect the detector from temperature fluctuations by placing the system away from direct sunlight and drafts, and regularly monitor flow rate and pressure for change.

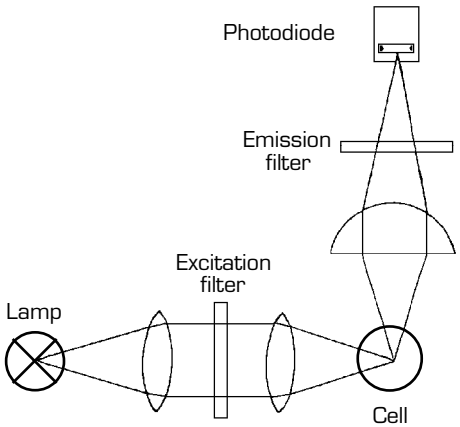
Detector response can drop because dirt in the cell or a bad source lamp reduces the level of radiation reaching the photocell. Some detectors have a meter that allows easy determination of light level. If it is low, clean the detector or change the source lamp. (Avoid eye damage by not viewing the light directly.) Consult the detector manual for the proper procedure for changing the lamp and cleaning the cell. The average life of a 254 nm lamp is approximately 5000 hr, but it should be replaced as soon as aging begins to cause significant intensity changes. Some cells can be taken apart, the optical components cleaned with a suitable solvent and dried, and the cell re-assembled. Others cannot be taken apart and are cleaned by flushing the cells with a series of solvents delivered from a 50 mL glass syringe, *e.g.*, acetone, 6 M nitric acid, distilled water, and acetone, then drying with a flow of clean, dry nitrogen before reconnection to the column. If necessary, allow 6 M nitric acid to stand in the cell overnight. To remove particles most effectively, draw nitric acid through the cell with a syringe in a direction opposite to the normal flow.

605 B: FLUORESCENCE DETECTORS

Fluorescence detectors provide two to three orders of magnitude more sensitivity than UV detection. Selectivity is also excellent because of the choice of excitation and emission wavelengths and the fact that only a small fraction of all compounds naturally fluoresce.

The simplest type of instrumentation is a fixed wavelength fluorometer with bandpass filters for both excitation and emission. More convenient and versatile fluorometric detectors can operate at variable wavelengths. These are equipped with monochromators to select excitation and emission wavelengths. Most compounds that fluoresce naturally have a rigid, planar conjugated cyclic structure. Nonfluorescent compounds can be detected if they are first converted to fluorescent compounds by pre- and post-column derivatization.

Figure 605-c
Fluorescence HPLC Detector



[Reprinted with permission of John Wiley and Sons, Inc., from Meyer, V.R. (1988) *Practical High Performance Liquid Chromatography,* Figure 5.10, page 74.]

Detector Design

Figure 605-c is a schematic diagram of a simple filter fluorometer detector. Light from a mercury lamp passes through a filter that selects the excitation wavelength. An interference filter providing a 10-20 nm

REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical * on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: *

Alternative Names:

Reference Standard (source and number):

Molecular Formula:

Structure:

Comments:

Results for DG module (Section 302): DG ____

Standard reference material dissolved in

Brief details about GLC system used:

Column:

Length:

id:

Film thickness:

Carrier gas:

Flow rate:

Makeup gas:

Flow rate:

Retention time (relative to _____) of _____ (marker compound): ◀

Detector:

Temperature:

Other conditions:

Detector response to ____ ng _____:_____% FSD

Behavior of * :

Retention time (relative to _____):

ng required for 50% FSD:

Information submitted by:

Address:

Phone: ( )

Date: