A. Scope of the Procedure

Method 3M 0222 is designed as an alternate test procedure for currently approved EPA Method 608. The EmporeTM disk is used in place of liquid-liquid extraction. This method is being promulgated as an alternative procedure for the determination of nineteen specified organochlorine pesticides and seven PCBs listed below: Aldrin alpha-BHC beta-BHC Chlordane delta-BHC Dieldrin Endosulfan I Endosulfan II Endosulfan sulfate Endrin Endrin aldehyde gamma-BHC PCB-1254 Heptachlor Heptachlor epoxide Methoxychlor Toxaphene 4,4'-DDD 4,4'-DDE 4,4'-DDT PCB-1016 PCB-1221 PCB-1232 PCB-1242 PCB-1248 PCB-1254 PCB-1260

The parameters listed in the table can be determined by gas chromatography using Method 3M 0222. When the method is used to analyze unfamiliar samples for any or all of the compounds listed, compound identifications should be supported by at least one additional qualitative technique. The method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column.

B. Summary of the Methods

A measured volume of sample, approximately 1–L, is extracted using a 90 mm Empore[™] disk. The disk is eluted with acetone followed by methylene chloride. The eluant is dried by pouring through anhydrous sodium sulfate and exchanged to hexane during concentration to a volume of 10-mL or less. The eluant is separated by gas chromatography and the analytes are then measured with an electron capture detector.

The method provides a Florisil column cleanup procedure and an elemental sulfur removal procedure using activated copper powder to aid in the elimination of interferences that may be encountered.

C. Technical Justification for Approved *Procedure*

The approval of this procedure is based on Agency review of the supporting information and data submitted by the applicant, 3M Corporation. EPA is approving the method based on the method description in EPA's Environmental Monitoring Management Council format, comparative analyses using the proposed and approved procedures, and EPA's technical and statistical reviews of each data package.

3M Corporation provided test data comparing the proposed procedure with appropriate approved procedure. The results from the proposed alternate method were compared to the approved EPA Method using liquid-liquid extraction/gas chromatography procedures. EPA statisticians and chemists conducted independent reviews of the data. The recovery and precision of all the submitted data for both the approved and proposed methods were also compared to the recovery and precision acceptance criteria derived for EPA Method 608 from Performance Evaluation Studies WP 18 and 23.

The Agency has judged the currently approved Method 608 method to be acceptable in the evaluation of the proposed procedure. EPA's Environmental Monitoring Systems Laboratory in Cincinnati, Ohio (EMSL-Cincinnati) thoroughly reviewed and evaluated the supporting data submitted by the 3M Corporation. The comparability reviews indicated that the analyses afforded comparable recovery and precision in the recommended concentration ranges for the listed organochlorine pesticides and PCBs. EPA proposed approval of the EmporeTM disk procedure and sought public comment on the suitability of this method as an alternate procedure for use in the determination of the parameters listed in 59 FR 65878 (December 21, 1994). The administrative record is on file at EMSL-Cincinnati, 26 W. Martin Luther King Dr., Cincinnati, Ohio 45268. The record is available for public inspection. The approved procedure is also available from 3M Corporation, 3M Center Building 220– 9E-10, St. Paul, MN 55144-1000.

Based on EMSL-Cincinnati's review, and pursuant to 40 CFR Section 136.5, EPA has approved the 3M Corporation's "Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk" method as an acceptable alternative procedure for nationwide use. Specifically, the method exhibits sufficient precision and recovery to establish (1) its acceptability under Part 136 and (2) its comparability to the approved procedure for analysis of the specified organochlorine pesticides and PCBs. As an approved alternate test procedure, this procedure is acceptable for use by any person required to test for these parameters.

IV. Public Comments and Response to Most Significant Comments

The Agency requested comments on the proposal to approve the 3M method for pesticides and PCB's. Comments were received from 5 individuals/ organizations. All commenters favored approval of disk extraction as an acceptable alternate procedure (ATP). The most significant comments were as follows:

Comment: Other companies produce extraction disks on inert surfaces, so all references in the method to the disk in the 3M method should be generic in nature so that other commercial products can be used by the analyst. Commenter supports feasibility of generic approach by noting the method includes initial quality control demonstrations that can demonstrate applicability of the alternative vender's product, and that EPA used general product description language in the comparable method approved in 40 CFR 141 for drinking water analyses.

Response: EPA's limited resources are not sufficient to fully evaluate all new technologies that may be applicable to monitoring programs under the Clean Water Act. The nationwide alternate test procedure (ATP) program was established 40 CFR Part 136.4 to allow developers of new commercial instruments, product or supplies to demonstrate the efficacy of the measurement technology to measure pollutant concentration levels. The ATP program is expensive for the applicant as applicability to a broad variety of wastewaters must be demonstrated. The Agency does not require this applicant to demonstrate that the extraction technology can be made to work using competitor's products. The use of a competitive product in this method would require additional method development to optimize solvents, flow rates, and other features of the method. After these procedures have been standardized, a suitable demonstration of applicability is required. Because of the diverse nature of wastewaters under this regulation, a general statement of applicability could be made only if a number of different wastewaters are tested. Limited use approval could be obtained on a case-by-case basis by demonstrating applicability to an individual discharger's wastestream.