

State of California
California Environmental Protection Agency
AIR RESOURCES BOARD

**Final Statement of Reasons for Rulemaking,
Including Summary of Comments and Agency Response**

**PUBLIC HEARING TO CONSIDER THE AMENDMENT AND ADOPTION OF STATIONARY
SOURCE TEST METHODS**

Public Hearing Date: September 26, 1996
Agenda Item No.: 96-7-2

I. Introduction:

On September 26, 1996, the Air Resources Board (the "Board") conducted a public hearing to consider the amendment and adoption of seven stationary source test methods.

At the public hearing, the Board adopted Resolution 96-46 to adopt and to amend regulations and to incorporate by reference the new and amended test methods. The revised regulations are Title 17, California Code of Regulations (CCR), Sections 94105, 94107, 94114, 94135, 94141, and 94143. Title 17, CCR, Section 94161 was also adopted. The incorporated test methods are ARB Methods 5, 7, 100, 425, 429, 431, and 436.

After consideration of formal comments received during the 45-day public comment period prior to the hearing, the Board directed staff to modify the regulations and provide a further 15-day period for public comment on these modifications. The modified regulations were made available to the public for a 15-day comment period between April 1, 1997 and April 15, 1997, pursuant to Government Code Section 11346.8(c). The "Notice of Public Availability of Modified Text" was mailed with the modified text of the regulations by March 31, 1997.

A Staff Report was prepared as the Initial Statement of Reasons for the proposed rulemaking. The Staff Report was released on August 9, 1996 and is incorporated by reference herein. The Final Statement of Reasons updates the Staff Report by explaining why the proposed test methods were modified, as well as summarizing the public comments received and the Board's responses to the comments.

The Board has determined that this regulatory action does not impose a mandate on local agencies or school districts.

The Board has further determined, for the reasons set forth in the Initial Statement of Reasons, that no alternatives considered by the agency would be more effective in carrying out the purpose for which the regulatory action was proposed or would be as effective and less burdensome to affected private persons, than the action taken by the Board.

II. Need for Adoption of New and Amended Test Methods

Since 1983, the Air Resources Board (ARB) has adopted 47 test methods for determining emissions from nonvehicular, or stationary, sources. The new test method and the six revised test methods are part of the Board's ongoing effort to provide the most updated and accurate procedures for measuring emission of air pollutants. Data collected from source tests are used to determine compliance with local air pollution control regulations, to evaluate the effectiveness of air pollution control equipment, and to inventory the emissions of both criteria and toxic pollutants. The August 9, 1996, staff report provides the background and the reasons for revision of each of the seven new and amended source test methods.

III. Changes to the Originally Proposed Test Methods

Some modifications have been made to the test methods proposed in the August 9, 1996 staff report. Most of the modifications were in response to the comments received during the 45 days prior to the September 26, 1996 public hearing. Modifications of the source test methods which are in response to public comment are explained in Sections V and VI, below.

Staff also proposed several modifications to Method 436 which were included in the 15-day package. Staff initiated changes to Method 436 to reflect the most recent version of EPA Method 29 for determination of metals emissions from stationary sources. These modifications are: (1) the addition in Figure 13 of quality assurance and quality control requirements for analyses conducted using inductively coupled plasma mass spectroscopy and (2) changes in the sample recovery procedure for impingers 5 and 6.

Finally, staff made editing changes to the proposed test methods to correct errors and improve readability. These editing changes are non-substantive and have no regulatory effect.

IV. Summary of Comments and Agency Responses

Ten comment packages were received during the 45 days prior to the September 26, 1996 public hearing. Those comments are summarized in Section V of this report, together with staff's response to each of the comments. No oral testimony was given at the Board meeting. Three additional comment packages were received during the 15-day comment period between April 1, 1997 and April 15, 1997. Section VI is a summary of comments received during the 15 day comment period and staff response to those comments.

V. Summary of Comments received during the 45-day comment period

Ten comment packages were received during the 45 days prior to the public hearing. Summarized below are the comments and recommendations for modifying the test methods, as well as staff response to these comments. Staff's additions to the methods in response to comments are noted by underline and deletions are noted by ~~graphic screen~~.

Comments and staff response are organized into the following categories:

- A. General comments affecting some or all of the proposed test methods
- B. Method 5
- C. Method 7
- D. Method 100
- E. Method 425
- F. Method 429
- G. Method 431
- H. Method 436

A. General comments affecting some or all of the proposed test methods

1. Comment by Mr. Michael Wang of Western States Petroleum Association (WSPA) regarding all seven of the proposed new and modified methods: ARB should provide guidance on what constitutes a test method modification which would require the ARB Executive Officer approval.

Agency Response: ARB staff is currently working with United States Environmental Protection Agency (USEPA) to develop guidance on classification of test method modifications and the corresponding level of approval required. Until this guidance is approved, the ARB considers all modifications of ARB test methods subject to Executive Officer approval unless specifically allowed in the method. This is important to maintain statewide consistency in applying test methodologies. No changes to the methods were made.

2. Comment by Mr. Wang regarding all seven of the proposed new and modified methods: The ARB should provide a simple administrative process that incorporates the updates to references such as the Federal Methods and American Society for Testing Materials (ASTM) methods.

Agency Response: We agree that references should be reviewed periodically and updated where appropriate. This can be done within the current method amendment process. No change to the methods was made.

3. Comment by Mr. Wang regarding Methods 425, 429, and 436: The ARB should provide a consistent approach to the determination of the method detection limit in Methods 425, 429 and 436.

Agency Response: Scientific experts continue to disagree on the best way to calculation method detection limits. It should be noted that the methods cited use a variety

of different analytical techniques, including ion chromatography, gas chromatography-mass spectrometry, and atomic emission spectroscopy. In addition, the ARB methods were developed to be consistent with the corresponding USEPA methods. For these reasons, a variety of calculation methods for method detection limits are included in the methods. No change to the methods was made.

B. Comments on Method 5, Determination of Particulate Matter Emissions from Stationary Sources

1. Comment by Mr. Jim Steiner of Steiner Environmental, Inc. regarding Section 2.1.5: This section specifies a borosilicate glass frit filter support and allows other materials with Executive Officer approval. Mr. Steiner recommended allowing Teflon, 316 stainless, and Teflon-coated 316 without Executive Officer approval, citing cleaning problems with the glass frit.

Agency Response: As currently worded, EPA Method 5 and ARB Method 5 are consistent in requiring approval for materials other than glass. Allowing the options as recommended would complicate the method since other options would still require approval. Written approval for routine use of appropriate alternative filter supports may be obtained by any testing organization. Therefore, no change to this section was made.

2. Comment by Mr. Steiner on Section 3.1.5: This section calls for acetone-insoluble, heat-stable silicone grease, or screw on connectors with Teflon sleeves, allowing alternate greases with Executive Officer approval. Mr. Steiner recommended specifically allowing use of Dupont "Krytox" perfluoroalkylether / polytetrafluoroethylene grease without Executive Officer approval, noting that this material has low solubility in methylene chloride.

Agency Response: Our experience is that grease of any sort presents some risks of contamination of the sample and that this contamination is best avoided using grease-free connector systems. Methylene chloride solubility is not an issue in the context of the current draft since grease is not to be used if methylene chloride is a rinse solvent. The current wording is consistent with EPA Method 5 and revision as recommended would introduce complexity to the method with small rewards resulting from it. No revision was made.

3. Comment by Mr. Steiner on Section 3.2: Mr. Steiner recommended that reagent grade methylene chloride be specified.

Agency Response: The current proposed draft already specifies that methylene chloride be reagent grade and meet solids residue requirements. No modification was made.

4. Comment by Mr. Steiner on Section 4.1.3: This section addresses methods for sealing the probe joints at the nozzle and filter holder. Mr. Steiner advocated allowing graphite ferrules in addition to the sealing methods currently allowed, i.e., Viton A O-rings, asbestos string gaskets, steel ferrules and teflon ferrules. He noted that graphite ferrules reduce the risk of breakage due to differential expansion and contraction in exposure to elevated temperatures.

Agency Response: Use of graphite ferrules may be reasonable and possibly slightly less hazardous than use of asbestos string gaskets. The current wording of this section of the method matches EPA Method 5. We have no experience with graphite ferrules to date. Breakage and oxidation of the ferrules might conceivably be a problem. Allowing graphite ferrules would complicate the method somewhat. The issue should be given further study and no changes were made at this time.

5. Comment by Mr. Steiner on Section 4.1.4.1: Mr. Steiner noted that although a pretest leak check is mandatory, no leak check is mandatory before restarting the train after replacing a component (for instance a filter or impinger assembly). He recommended pre-restart leak tests.

Agency Response: Historically, pretest leak checks were not mandatory, simply highly advisable. Pre-restart leak checks are also desirable, and are not prohibited, but they introduce a delay and disturb the continuity of sampling which is not the case with a pretest leak check. Note that leak checks after the test are required, and leak checks before replacement of a component are required. These checks are more important because they provide assurance that no leaks developed during testing, not simply that the train was leak-free before the test. Pre-restart leak checks were not added as a requirement to the method.

6. Comment by Mr. Steiner on Section 4.2: This section describes sample recovery and refers to a "cleanup area" which should be clean and protected from the wind to avoid sample contamination. Mr. Steiner advocated allowing cleanups of the probe and nozzle on the stack using special equipment.

Agency Response: While use of special apparatus could allow cleanups of the probe and nozzle on the stack without exposure to wind and dust, and while such apparatus could be considered a "cleanup area" to the extent that it provided and delimited such a region of protection from the elements, description of apparatus adequate for this purpose could complicate the method substantially. Mr. Steiner in fact does not provide a detailed description of the apparatus he advocated. Our experience is that as much or more breakage occurs when a train is raised as occurs when it is lowered. The current wording of the section for probe recovery matches EPA Method 5 closely. No specific provision for on-stack probe recovery was added.

7. Comment by Mr. Steiner on Section 4.3.1.2: This section refers to mason jars used as sample containers, while section 2.2.3 refers to "glass sample storage containers," noting that narrow mouth bottles are less leak-prone. Mr. Steiner recommended changing terminology in Section 4.3.1.2 from "mason jar" to "glass bottle."

Agency response: The intent of Section 2.2.3 is to recommend, but not require, narrow mouth bottles except when polyethylene containers are used. Wide mouth glass jars are allowed and have certain advantages in the field although extra care is advisable in sealing them. To make Section 4.3.1.2 consistent with the terminology of Section 2.2.3, the words "mason jar" in section 4.3.1.2 were replaced with "sample container."

8. General comment by Mr. Higuchi, South Coast Air Quality Management District (SCAQMD) and Mr. Wang of the Western States Petroleum Association (WSPA):

It was noted that acetone and methylene chloride present hazardous waste disposal problems and that the ARB should look for alternative solvents that pose a lower risk to sampling and analysis personnel and to the environment.

Agency Response: Because laboratories and testing organizations should be equipped to handle such wastes routinely, this is not a major problem. Mr. Wang's suggestion that less toxic and environmentally detrimental alternative solvents be sought is reasonable. ARB Method 5 currently allows such alternative solvents, as Mr. Wang noted. No change to the method was made.

9. General comment by Mr. Higuchi, SCAQMD: The SCAQMD procedure differs from the ARB procedure to allow for the capture of sulfur dioxide (SO_2) in the front half of the impinger so that it is considered as particulate.

Agency Response: It is acceptable that the SCAQMD procedure differs from the ARB procedure, as ARB methods are intended for use by districts which choose not to develop their own methods for district rules. Staff notes that condensation of sulfuric acid is a function of concentration as well as temperature, not temperature alone as suggested by the comment. No change was made to the method.

10. General comment by Mr. Higuchi: Mr. Higuchi noted that ARB Method 5 calls for a glass frit filter support and suggested that other materials offer advantages over this.

Agency Response: Section 2.1.5 of Method 5 allows the use of alternate materials. No change was made to the method.

11. General comment by Mr. Higuchi: Mr. Higuchi suggested that SO_2 may react with high-sodium glass filters, producing a weight gain.

Agency Response: Staff notes that EPA Method 5 specifies use of filters selected for immunity to such reactions only in testing sources with emissions containing SO_2 or SO_3 (sulfur trioxide). Few sources in California have substantial SO_2 and SO_3 emissions. Revision of the filter specifications in Section 3.1.1 to require quartz fiber filters or other filters immune to reaction with SO_2 and SO_3 may be desirable in the future. No change was made to the method.

12. General comment by Mr. Wang of WSPA: Mr. Wang recommended that provisions be added for correction of results for ammonium sulfate dihydrate which may be formed as a consequence of use of excess ammonia in emissions control systems.

Agency Response: Ammonium sulfate dihydrate should be retained in the source test results because the sampling conditions and reactions which may occur in the sampling train are representative of what might occur in the atmosphere. Further, this is a matter which has limited applicability and can be better resolved by site specific arrangements than changes to the test method. No revisions to the method were made.

13. Comment by Mr. Wang on Section 4.3: Mr. Wang noted that the definition of "constant weight" does not guarantee that constant weight will be achieved in a finite number of trials.

Agency Response: This is correct but the circumstances which might cause this situation would be unusual and sufficient to invalidate the test sample. No change in the section was made.

14. Comment by Mr. Wang on Section 4.3.1.3: Mr. Wang suggested enhancement of this section to provide guidance for breaking emulsions which may form in solvent extraction of the back half catch and drying the solvent extract.

Agency Response: It is not clear that formation of emulsions is a frequent or serious problem given the moderate agitation specified and the use of a cotton plug for separation of water droplets from the solvent. It is imperative that soluble salts not be used in contact with the aqueous phase since it is analyzed for non-volatile residual. Trace moisture in the extract is not critical since it does not impact the nonvolatile material extracted and will evaporate with the solvent. No change in the section was made.

15. Editorial comments by Mr. Wang for Sections 4.1.5, 4.3.1.1, 4.3.1.2, 5.3, 6.1, 6.11.1, Figure 5.3, Appendix: Mr. Wang suggested editorial changes to these method sections.

Agency Response: Editorial changes were made to the method as appropriate.

C. Method 7, Determination of Nitrogen Oxide Emissions from Stationary Sources

1. Comment by Mr. Higuchi, South Coast Air Quality Management District (SCAQMD): Mr. Higuchi noted that the SCAQMD method differs from the ARB method in respect to requiring audit standards.

Agency Response: The differences in the SCAQMD method are acceptable. Please see the staff response to comment 9 on ARB Method 5. No change to ARB Method 7 was made.

2. Comment by Mr. Higuchi: Mr. Higuchi noted that separate specification of phenol was deleted from the ARB method.

Agency Response: This is intentional and the wording in Section 3.3.6 with respect to phenol is sufficient. No change was made.

3. Comment by Mr. Higuchi: Mr. Higuchi suggested that the intermediate sample recovery described in Section 4.2 be eliminated and combined with analytical procedures in Section 4.3.

Agency Response: Section 4.2 is identical to EPA Method 7 Section 4.2 and provides an appropriate procedure where multiple day test programs are conducted at a location remote from laboratory facilities. No change was made.

4. Comment by Mr. Wang of Western States Petroleum Association on Sections 3.3.8 and 4.4.1: Mr. Wang inquired whether ARB has any assurance that audit samples will continue to be available from USEPA.

Agency Response: The ARB has no assurance regarding the audit samples. Section 3.3.8 provides for the use of audit samples from other sources with Executive Officer approval. No change was made to the method.

5. Comment by Mr. Wang on Section 5.2.3: Mr. Wang inquired whether the 7 percent (7%) deviation of calculated concentration from actual concentration is absolute or relative.

Agency Response: The allowable deviation should be calculated as 7% of the "actual" concentration since it is clear that this is the standard of comparison and no indication to the contrary is given. No change to the method was made.

6. Comment by Mr. Wang on Section 5.6: He inquired whether the "standard weights" referred to are "type S" or "type P."

Agency Response: The standard weights used must be those which support the performance specification for the analytical balance, i.e., weighings accurate to within 0.1 mg. No change was made.

7. Editorial comments by Mr. Wang for Sections 2.1, 2.1.10, 2.3.2, 3.1.1, 5.2.1.1, and 8: Mr. Wang suggested editorial changes to these sections.

Agency Response: Editorial changes were made to the method as appropriate.

D. Comments on Method 100, Procedures for Continuous Gaseous Emission Stack Sampling

1. General Comment by Mr. Alan Bahl of Red Star Yeast: Mr. Bahl wrote regarding his concern that ARB Method 100 would impose inappropriate requirements for the permanently installed monitoring system at the Red Star Oakland, California facility.

Agency Response: Although Method 100 is titled "Procedures for Continuous Gaseous Emission Stack Sampling," the method does not apply to continuous emissions monitoring systems which are permanently installed at a facility. Mr. Bahl's concern was addressed when he learned that Method 100 would not apply to the monitoring installation at the Red Star facility in Oakland. No change to the method was made.

2. Comment by Mr. John Higuchi of the South Coast Air Quality Management District (SCAQMD) on Section 1.5.1: He recommended that in order to accept data at 10% of an analyzer scale, the analyzer should undergo a four-point calibration, rather than a three-point calibration, with the low range calibration gas between 10 and 20 percent of the analyzer range.

Agency Response: Multi-point calibrations are performed semiannually for the ARB's Compliance Division gas analyzers. Data covering several years of these calibrations show that the analyzers have a linear response at concentrations near 10% of the analyzer scale. Although not included in Method 100, professional quality assurance practice for a source test team would include performance of at least a semiannual multi-point calibration of each gas analyzer scale at 5 or 10 evenly spaced points along the calibration curve. A point near 10% should be included, and no test data should be obtained at concentrations lower than the lowest calibration point on the curve. No change was made to the method.

3. Comment by Mr. Higuchi on Sections 1.5.5 and 1.5.6: Mr. Higuchi also recommended that the underlined text in the following definition be replaced: "*The difference between the concentration . . . after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.*" Replacement text should be "each test run."

Agency response: The definitions of bias and drift checks in Sections 1.5.5 and 1.5.6 were written to accommodate test runs of varying lengths. Normally test runs for Method 100 range between 20 minutes and one hour. It would then be appropriate to conduct bias and drift checks after each test run, as outlined in Section 6 for post test performance checks. However, Method 100 may also be run simultaneously with a five- to eight-hour toxic compound test method, such as Method 429 for polycyclic aromatic hydrocarbons. As stated in Section 5, paragraph 2, bias checks are required every two hours for a multi-hour test run. The definitions as currently written in Sections 1.5.5 and 1.5.6 were retained.

4. Comment by Mr. Higuchi on Section 2.1.7: Mr. Higuchi stated that the language appears to apply to a wet analyzer system.

Agency Response: Mr. Higuchi is correct that the conditions described in Section 2.1.7 are for a wet analyzer system. Such conditions would be inappropriate for measurement of carbon dioxide or carbon monoxide. However, this option was included for measurement of other pollutants under specific conditions. For example, a heated trace line with no condenser could be used for the measurement of nitrogen oxides in an exhaust stream which contains ammonia salts. Condensed ammonia salts would damage an analyzer. No change was made.

5. Comment by Mr. Higuchi on Section 2.2.4: Mr. Higuchi further commented that continuous analyzers for VOC should be used only when the species of hydrocarbons are known so that the analyzer can be correctly analyzed.

Agency Response: Section 2.2.4 was modified to read as follows:

An analyzer using a flame ionization detector (FID) or a nondispersive infrared analyzer (NDIR) is acceptable. The hydrocarbon species should be characterized prior to the source test to determine that the detector will respond predictably to the organic compounds present. Further, compound-specific calibration curves must be determined for use of either the FID or the NDIR analyzer to measure specific

organic compounds. [Underlined text is wording added to the regulation and noticed during the 15-day notice period.]

6. Comment by Mr. Jim Steiner of Steiner Environmental, Inc. on Section 2.2.8:

Mr. Steiner recommended that the use of a strip chart recorder should be mandatory, regardless of whether or not an electronic-data-acquisition system is used. The strip-chart recorder should be specified as 0 to 100 divisions with 0.5 divisions, 10 inches wide, with no more than two channels per recorder.

Agency Response: Due to concerns for safeguarding the integrity of electronic data, staff modified the method to require the use of a strip chart recorder regardless of the use of an electronic data-acquisition system. Staff will continue discussions with the source test community on ways to protect source test data and to insure that district staff has access to real-time data. If outstanding concerns regarding electronic data acquisition can be resolved, staff will recommend elimination of the requirement of a strip-chart recorder at a future time. The text below requires that the strip chart has a resolution of 0.5 %, which satisfies the proposed requirement of 100 divisions, with 0.5 resolution (if the commenter intended 0.5 resolution rather than 0.5 divisions). The proposed requirement for a 10-inch-wide recorder with no more than two channels per recorder would not provide an improvement in the protocol commensurate with the added cost.

The method was revised as shown below:

Section 2.2.8 Data Recorder/Data Acquisition System

Provide a permanent record of gas analyzer data using a strip chart recorder. a data logger or other electronic data acquisition system. If a data recorder is not used, a real-time hardcopy of test data must be provided upon request. A data logger or other electronic data acquisition system may also be used. Any electronic data acquisition system must be capable of integration at a ten second interval. Any data acquisition system The strip chart, as well as the data accession system, must have a resolution of 0.5 of the analyzer range. Data reporting includes
[Underlined text is wording added to the regulation and graphic-screened text is wording deleted from the regulation and noticed during the 15-day notice period.]

7. Comment by Mr. Wang of WSPA on Section 3.1: Mr. Wang recommended that ARB consider permeation systems as an alternative to certified calibration gases or dilution systems.

Agency Response: Method 100 was revised to allow the use of permeation tubes as specified in EPA Method 16, CFR 60 Appendix A. EPA Method 16 allows the use of permeation tubes as the standards for a number of sulfur compounds. Critical parameters include: (1) operation of the permeation tube at constant temperature, such as in a constant temperature bath, (2) operation at a constant flowrate, verified with a calibrated flowmeter or mass flow controller, and (3) operation at equilibrium, demonstrated by checking samples at one-hour intervals. Method 100 was modified by the addition of the following text to the first paragraph of Section 3.1:

... A permeation tube may be used as a calibration standard in place of the calibration gas, provided the following requirements of EPA Method 16 (CFR 60 Appendix A) are met for use of permeation tubes: Section 5.5 for the calibration system, Section 6.5 for the calibration gases, Sections 8.2 and 8.3 on pretest calibration, and Sections 10.2 and 10.3 on post-test calibration. [Underlined text is working added to the regulation and noticed during the 15-day notice period.]

8. Comment by Mr. W. A. Brommelsiek of Chevron on Section 4.5:

Mr. Brommelsiek proposed the addition of two paragraphs to Section 4.5 of Method 100: (1) The first proposed paragraph would specifically allow single point testing if it is demonstrated that the concentration at the single point is within 10% of the mean concentration determined from traverses performed downstream, but prior to any fuel gas processing. The data for one piece of equipment could be used to allow single-point testing on similar pieces of equipment with similar test ports. (2) The second proposed paragraph allows single point testing if the accuracy of the data is maintained within 10% of the mean concentration of the gas.

Agency Response: ARB's Method 100 allows single-point testing if the data are provided to show that the single-point results are within 10% of the mean concentration for sampling traverses conducted according to ARB Methods 1 and 2 for velocity traverses. A District representative responsible for determining compliance with a test-method regulation would be responsible for determining the validity of the data used to justify single-point testing.

Mr. Brommelsiek's paragraph (1) addressed a specific application of Method 100 to test-oil-production-steam generators for compliance with the San Joaquin Valley Unified Air Pollution Control District Rule 4305 for NOx and CO from boilers, steam generators, and process heaters. Most of the hundreds of oil production steam generators owned by Chevron do not have properly sited ports for performing stack testing according to ARB Methods 1 and 2. Mr. Brommelsiek proposed to sample a steam generator which is equipped with stack and sampling ports configured according to the requirements of ARB Methods 1 and 2. The concentration traverses would be conducted at this port and compared with sampling conducted at a single point located upstream at the "hog trough." If the hog trough concentration was within plus or minus 10 percent ($\pm 10\%$) of the mean of the downstream multi-point sample, Mr. Brommelsiek proposed that single point sampling at the hog trough could then be conducted for other steam generators which are configured and operated in the same manner as the generator which was tested.

ARB is opposed to including this proposal for testing steam generators in Method 100. Instead, the district staff responsible for approving source test results should examine, on a case-by-case basis, data presented to justify that single-point test results are within 10% of the mean concentration for a multi-point traverse. For the situation described by Mr. Brommelsiek, the district representative should also examine the source-test data and data regarding the configuration and operating parameters of the individual steam generators to determine if the test results can also be extended to other equipment. No change to the proposed method was made.

It is likely that the commenter intended paragraph (2) to clarify the text of Section 4.5 of Method 100. However, the concentration at any traverse point, rather than the accuracy of the data at that point, must be less than 10% different from the mean to allow single-point testing. No change to the method was made.

Comments 9 through 16 below all address Appendix 100.1 of Method 100, on verification of gas-dilution systems for field instrument calibrations:

9. General comment made both by Mr. Robert Wright of Research Triangle Institute and by Mr. Bruce Shroyer of Calibrated Instruments: The gas dilution protocol exhibits the same bias in favor of mass flow controllers and the same technical deficiencies as does EPA's Method 205. The method should be rewritten to accommodate the operational characteristics of gas dilution systems utilizing other technologies, including positive-displacement pumps.

Agency Response: ARB agrees that the method as written exhibits bias in favor of mass flow controllers and that the method should be rewritten to accommodate gas-dilution systems using other technologies. A possible certification procedure could use multi point characterizations of undiluted and diluted gas. The slopes of the regression lines for the diluted and undiluted gases could then be compared to determine the reliability of the diluter. At this time, we have not performed the laboratory studies necessary to include such a procedure in the method. Staff of the National Institute of Standards and Technology (NIST) has also acknowledged that work is needed to characterize the reliability of gas-dilution systems, but has not identified resources to carry out the needed investigations. In the interim, we have proposed to allow source testers to use the adopted EPA Method 205, despite its flaws. It is our view that any gas dilution system which meets the field evaluation protocol of Section 3.2 of the USEPA method should provide gas standards adequate for field work.

Method 100, Appendix 100.1 was deleted and Section 3.2 of Method 100 was revised as follows:

3.2 GAS DILUTION SYSTEM

An approved gas dilution system which meets the requirements of EPA Method 205, Verification of Gas Dilution Systems for Field Instrument Calibrations, CFR 40, Part 51, Appendix M, may be used to provide low-level calibration gases from a high-level calibration gas. The calibration gas used with a gas dilution system must should be an EPA Protocol gas. Alternately the gas used with a diluter must be certified to an analytical accuracy of ± 1 percent, NIST-traceable, and recertified annually. See Appendix 100.1 for the performance specifications of a gas dilution system. An approved gas dilution system which meets the requirements of EPA Method 205 may be used for all analyzer calibrations and sampling system bias checks. [Underlined text is wording added to the regulation and graphic screened text is wording deleted from the regulation and notice during the 15-day notice period.]

10. Comment by Mr. Wright on Appendix 100, Sections 3.4.1 and 3.4.2 and Method 100 Section 3.2: Mr. Wright also commented that the analytical accuracy specifications for high-level and mid-level calibration gases are unrealistic. Calibration gases certified at $\pm 1\%$ uncertainty should not be allowed unless the USEPA protocol or other accepted analytical protocol for gas certification is used. Also annual recertification of calibration gases is overly restrictive and should be changed to the certification periods for USEPA protocol gases.

Agency response: ARB agreed with Mr. Wright's concern that a gas manufacturer's claim of a level of analytical accuracy is not an indication of the actual reliability of the gas standard. Instead, the reliability of a gas standard is dependent on the certification protocol. Accordingly, the reference to $\pm 1\%$ analytical accuracy gases was deleted from Section 3.2 of the method, as shown above in the agency response to Comment 1 on the Appendix. Only USEPA Protocol gases, if available, should be used in a gas-dilution system.

Other comments regarding Appendix 100.1 are listed below as comments 11 through 16. Agency response to these comments was the deletion of the appendix at this time and the referral to EPA's adopted method, as outlined in the response to comment nine on the gas-dilution appendix. The comments listed below highlight the need for further evaluation of gas-dilution systems before ARB adopts a protocol of its own for certification and verification of gas-dilution systems. We will consider these recommendations when we complete our own evaluation of gas-dilution systems.

11. Comment by Mr. Wright on Appendix 100.1, Section 1.2: Mr. Wright commented that the gas analyzer used for field verification of the gas dilution system should be calibrated over its full range using four or more undiluted EPA protocol gases and the measured concentrations may differ from the predicted values no more than $\pm 5\%$.

12. Comments by Mr. Wright on Appendix 100.1, Section 3.1: Mr. Wright further stated that the $\pm 2\%$ specification is unrealistic. Error propagation analysis should be conducted to calculate the overall uncertainty associated with the proposed verification procedure. The ARB should also specify an overall uncertainty that is acceptable for gas-dilution systems, and it should specify a method for calculating uncertainty associated with each step in the calibration chain back to NIST primary gas and flow standards.

13. Comments by Mr. Wright on Appendix 100.1, Sections 3.2 and 4: Mr. Wright stated that the proposed amendment should be strengthened to include more detailed performance specifications and field evaluation procedures. The gas-dilution systems need to be evaluated more extensively in both the laboratory and the field than is specified in the method. He indicated also that the ARB needs to determine if gas dilution systems will meet the proposed specifications on a routine basis.

14. Comments by Mr. Wright on Appendix 100.1, Section 3.2: Mr. Wright included four comments on the annual certification procedure: (1) The method should be changed to require that mass-flow controllers be calibrated to NIST-traceable standards prior to each field test. (2) The proposed specification could be replaced by one based on the

statistical analysis of the multi-point-calibration data and the multi-point-dilution data. Specifically, determine the 95% confidence interval for concentrations predicted from the linear regression of the multi-point-calibration data. (3) Also, the yearly recertification of a gas-dilution system with primary flow standards is a significant cost which should be included in the economic analysis of the method. (4) Finally, Mr. Wright stated that the term NIST-traceable primary flow standard should be defined more clearly.

15. Comment by Mr. Shroyer on Appendix 100.1, Section 3.2: Mr. Shroyer proposed that a symmetry test may be performed to calibrate the positive displacement-pump system.

16. Comment by Mr. Shroyer on Appendix 100.1, Section 4: Mr. Shroyer stated that the verification of the accuracy in the field for a positive displacement pump (PDP) can be conducted without regard to the accuracy of the gas standard. He concluded that the use of a mid-level undiluted calibration gas as a check on the performance of the dilution system is not needed for a PDP.

E. Method 425, Determination of Total Chromium and Hexavalent Chromium Emissions from Stationary Sources

1. Comment by Mr. John Higuchi of the South Coast Air Quality Maintenance District (SCAQMD) regarding Section 2.4: Mr. Higuchi recommended that text in the section for Limit of Detection read, "*The LOD is based on the absolute value of the x-intercept of the calibration plot . . .*," to be consistent with the equation.

Agency Response: The method was revised as follows:

The LOD is based on the absolute value of the x-intercept of the calibration plot for absorbance versus concentration adjusted by three times the standard deviation of the absorbance for a mid-point concentration. [Underlined text is wording added to the regulation and noticed during the 15-day notice period.]

2. Comment by Mr. Higuchi regarding Section 4.1: Mr. Higuchi recommended that specifying hold-time limits and storage conditions would be helpful in the section for sample instability. Recovery within 24 hours, refrigeration, and a storage time of two weeks are recommended by Mr. Higuchi.

Agency Response: The method was revised as follows:

Chromium is subject to changes in valence state during the time between sampling and analysis recovery. Take all reasonable precautions, some of which are required in various sections of this method, to minimize all influences which may change the valence states of chromium in each sample. Factors which influence such changes are hold time, pH, and other chemical species.

Recovery of trains shall take place within 24 hours of sampling. Storage between recovery and analysis shall be at or below 4°C and shall be limited to two weeks. [Underlined text is wording added to the regulation and graphic screened text is wording deleted from the regulation and noticed during the 15-day notice period.]

3. Comment by Mr. Higuchi regarding Section 6.2: A range of 0.5 ug/50 mL to 3.0 ug/50 mL is unnecessarily restrictive for the hexavalent chromium manual colorimetric (Cr6 M-C) range. The range can be expanded to 0.5 to 50 ug/mL, provided that the residuals are less than 10%.

Agency Response: The method was revised as follows:

A straight line response curve was obtained in the range 0.5 μg Cr6 /50 mL to 3.0 μg Cr6 /50 mL (10 to 60 ng/mL). The range can be expanded to 0.5 to 50 μg /mL, provided that the residuals are less than 10%. For a minimum analytical accuracy of 100 ± 10 percent, the lower limit of the range is 2 μg /100mL. [Underlined text is wording added to the regulation and notice during the 15-day notice period.]

4. Comment by Mr. Higuchi regarding Section 7.3.2.4: Mr. Higuchi stated that it is not clear what the hot plate will be used for in the Cr6-M-C analysis.

Agency Response: The hot plate is used in sample preparation, Section 16.1.2 of the method. No change was made.

5. Comment by Mr. Higuchi regarding Section 9.3.2: Mr. Higuchi stated that there appears to be some missing text in section 9.3.2 Water "*Use of ASTM Type I reagent water . . .*"

Agency Response: The method was revised as follows:

Use of ASTM Type I reagent water shall be an acceptable alternative. [Underlined text is wording added to the regulation and notice during the 15-day notice period.]

6. Comment by Mr. Higuchi regarding Section 9.3.5: Mr. Higuchi stated that specifying the recommended maximum storage time and conditions would be helpful in the section for hexavalent chromium (Cr6) stock solution.

Agency Response: The method was revised by the addition of the following statement to the regulation, which was notice during the 15-day notice period:

Storage shall be at or below 4°C and shall be limited to four weeks.

7. Comment by Mr. Higuchi regarding Sections 11.3 and 11.4: Mr. Higuchi stated that unlike the Cr6 M-C and Cr GF-AA calibration procedures, the Cr6 IC-C Analytical

Calibration Procedure does not contain a "zero standard." It also does not require a check standard. Both are recommended additions. The number of standards for the calibration curve is not mentioned in the Cr6 M-C Calibration Procedure. There is also no check standard. This information should be added.

Agency Response: The method was revised as follows:

11.3 Cr6 IC-C Analytical Calibration Procedure

Inject a series of Cr6 calibration standards which brackets the sample concentrations. Also, use a zero standard. Typically, 4 to 6 calibration...

The following statement was also added to Sections 11.3 and 11.4(5):

Inject a check standard prior to the tenth run of the instrument since its last calibration run.

11.4 Cr6 M-C Analytical Calibration Procedure . . .

(5) Inject a check standard prior to the tenth run of the instrument since its last calibration run. [Underlined text is wording added to the regulation and notice during the 15-day notice period.]

Finally, after the 15-day notice of the availability of modified text, a statement was added to 11.4(3) to specify that 4 to 6 calibration standards are needed to plot the calibration curve.

8. Comment by Mr. Higuchi regarding Section 15.2: Mr. Higuchi stated that filtration is recommended to be optional for the manual colorimetric (M-C) analysis because the samples are often clear and additional sample handling may introduce errors.

Agency Response: The method was revised as follows:

(1) Filtration shall be an option for the analyst at this point, depending on the turbidity of the prepared sample. The analyst shall filter the preparation for clarity at this point. [Underlined text is wording added to the regulation and graphic screened text is wording deleted from the regulation and notice during the 15-day notice period.]

9. Comment by Mr. Higuchi regarding Section 17.2.1: Mr. Higuchi stated that in order to make the spike recovery work without volume correction in the calculations, there has to be an instruction in the section entitled "Check for Matrix Effects" on making the sample and matrix spike.

Agency Response: The requested instructions are already included in Section 17.2.1. Volume correction is not necessary since two equal volume aliquots of the sample solution are used. No change will be made.

10. Comment by Mr. Higuchi regarding Sections 17.2.2 and 19.1.1: Mr. Higuchi stated that it is not clear in Sections 17.2.2 and 19.1.1 whether blanks are referred to as zero standards or reagent blanks.

Agency Response: The method was revised by the addition of the following statement in Sections 17.2.2 and 19.1.1:

These are zero standards. [Underlined text is wording added to the regulation and noticed during the 15-day notice period.]

11. WSPA comment on 425.20.3: WSPA stated that this section contains a source reporting limit (SRL). The commenter noted that 425.20.1 addresses detected and not detected chromium 6 or [total?] chromium, but does not report NOT DETECTED results. The commenter stated that the reporting needs to be clarified.

Agency Response: If total chromium or chromium 6 was not detected above the limit of detection (LOD), the final reporting of the sample results is "NOT DETECTED" at source-reporting limit. No change to the method was made.

12. Comment by Mr. Wang of Western States Petroleum Association (WSPA) regarding Method 425: Mr. Wang stated that ARB should include in the scope of the method the staff's interpretive guidance on the maximum hexavalent chromium in total chromium measurements. The interpretive guidance was that hexavalent chromium was no greater than 5% of total chromium. The interpretive guidance is useful when the hexavalent chromium determinations are spurious.

Agency Response: Such data are anecdotal and are not necessarily valid for all source categories. No change to the method was made.

13. Comment by Mr. Wang regarding Section 22(1): Mr. Wang state that USEPA intends to withdraw Method 7191 as discussed by O. Fordham (EPA-OSW) at the USEPA 12th Annual Solid Waste Analysis and Quality Assurance Conference, July 1996. There are significant problems with the chromium 6/alkaline medium as discussed by R. Vitale, et al., at the USEPA 11th Annual Solid Waste Analysis and Quality Assurance Conference, July 1995.

Agency Response: The problem cited by R. Vitale for solid waste samples is not as applicable to analysis of the cleaner air samples collected according to Method 425. However, reference to the SW-846 method has been omitted from the final regulation, as noticed in the 15-day notice of availability of modified text.

14. Comment by Mr. Wang regarding Sections 3.4.1 and 17.1.1(2): Mr. Wang stated that the discussion of limit of detection (LOD) herein is different from a method detection limit (MDL); however, the LOD is used to establish a reporting limit (RL) [3.4.2]. He stated that the LOD applies to calibration deviation, and does not account for variation in the method.

Agency Response: The above comment is unclear. However, if the commenter is referring to data that is greater than the Limit of Detection (LOD), but less than the reporting limit (RL), the data should not be quantitated. No change to the method was made.

15. Comment by Mr. Wang regarding Section 11.5(6): Mr. Wang states that the reporting guidance does not make sense for air samples; it is guidance for solid waste type samples.

Agency Response: The reporting guidance in Section 11.5(6) was deleted from the final regulation, as noticed in the 15-day notice of the availability of modified text.

16. Editorial comments by Mr. Wang for Sections 7, 7.3.1, 9.3, 9.3.3, 11.4, 11.4.1, .2, .5, 17.1.1, .2, 22. Mr. Wang suggested editorial changes to these sections.

Agency Response: Editorial changes were made to the method as appropriate.

F. Method 429, Determination of Polycyclic Aromatic Hydrocarbon (PAH) Emissions from Stationary Sources

1. Comment by Mr. Gene Riley of Triangle Labs on Section 4.1: Mr. Riley commenting on Sealing Materials stated that a number of testers are currently using "O" rings to seal the filter holder and the ball/socket joints as well as the "O" ring for sealing the impinger joints (45/50). Some testers elect to use a viton material "O" ring which in the measurement of PAHs could possibly contaminate the collected sample when rinsed with the solvents. To allow for flexibility, "O" rings should be addressed, however, with the stipulation that the material be of Teflon which is inert.

Agency Response: After 15-day notice of the availability of modified text, the following changes to the final regulation were made to prevent the use of materials that might be reactive:

- (a) Section 4.1. ARB added the following statement: "All surfaces which may come in contact with the sample or recovery solvents shall be of quartz, borosilicate glass, or Teflon."
- (b) Section 4.1.2. ARB changed "should" to "must" in the first sentence so that it reads as follows: "The probe liner must be made of quartz, borosilicate glass, or Teflon." Also, ARB deleted the second sentence: "The liner of the probe is to provide an inert surface for the PAH in the stack gas." ARB added this replacement sentence: "Other inert materials may be used only if they have been approved by the Executive Officer."
- (c) Section 4.1.4. ARB added the following statement to specify the sealing materials for the filter holder: Whenever "O" ring seals are used, they shall be of Teflon or Teflon coated.

- (d) Section 4.1.8. ARB added the following statement to specify the sealing materials for the impingers: Whenever "O" ring seals are used, they shall be of Teflon or Teflon coated.

2. Comment by Mr. Riley on Section 5.1.1: Mr. Riley, commenting on the Probe Nozzle Brush, stated that ARB may want to insert "teflon" to replace "inert bristle with stainless steel wire handle." Teflon brushes with teflon handles are now available and being widely used.

Agency Response: After the 15-day notice, the following change was made to the final regulation at Section 5.1.1: ARB deleted the current requirement: "Inert bristle brush with stainless steel wire handle." ARB amended the requirement to read, "Teflon brush with Teflon handle."

3. Comment by Mr. Riley on Section 5.2.1: Mr. Riley stated that most testers refer to this as high-performance liquid chromatography (HPLC) water grade.

Agency Response: Section 5.2.1 of Method 429 defines reagent water as "deionized (DI), then glass distilled, and stored in hexane and methylene chloride-rinsed glass containers with TFE-lined screw caps." This M429 definition is consistent with the EPA Method 23 (M23) water requirement for the chromic acid cleaning solution for the sampling train glassware and for sample recovery.

M23 does contain two requirements for HPLC water but they are not applicable to Method 429. HPLC water is used in the early steps of the XAD-2 resin cleaning procedure described in Method 23. These are steps that would be performed by the manufacturer of a pre-cleaned resin. As the ARB method requires that the resin be purchased precleaned by the manufacturer and then cleaned again with methylene chloride, there is no need for HPLC water for the cleaning of the resin.

HPLC water is also used in the M23 impingers but the impinger contents are not recovered for analysis. We considered the use of HPLC water rather than reagent water to prepare the impinger solution for the M429 trains and decided that the change was not necessary. The results of blank analyses do not indicate a need for HPLC water.

There were no changes made.

4. Comment by Mr. Riley on Section 5.3: Mr. Riley, commenting on "No smoking allowed," stated that smokers should not be allowed to recover the samples unless they have thoroughly washed their hands or are wearing non-contaminating gloves. Also he asked if protective surgical gloves should be addressed.

Agency Response: After 15-day notice, the following change was made to the final regulation:

Section 4.4.4 ARB added "No smoking is allowed" to the beginning of the description of the procedure for operating the sampling train. The method already contains a similar prohibition in the sample recovery procedure (Section 5.3).

5. Mr. Wang of the Western States Petroleum Association (WSPA) discussed method detection limits: Mr. Wang stated that the practical quantitation limit (PQL) and the reporting limit (RL) are used interchangeably in methods 425, 429 and 436. The method detection limit (MDL) process must be used consistently from method to method.

Agency Response: The Method 429 procedure for determining MDLs is consistent with the procedure adopted by the EPA (40 CFR 136, Appendix B). However, we are not aware of an EPA method that could serve as a model for reporting PAH emissions from stationary sources. The EPA Method 23 does not consider the reporting requirements addressed by Method 429. The reporting requirements proposed in Method 429 were the result of discussions during method development workshops. No change was made.

6. Comment by Mr. Wang on Section 8.3.1: Mr. Wang stated that the theoretical method quantitation limit (TMQL) is confusing: the MDL is five times the TMQL in 429.8.3.1 and 429.A1.1(e), but elsewhere the MDL is 2.5 - 5.0 the S/N, as in 429.A1.1(a).

Agency Response: See the response to Comment #20 on Method 429. It appears that the instructions of the method were misunderstood. Values equal to five times the TMQL or 2.5 to 5 times the S/N were not proposed as different definitions of the MDL. They were proposed as options for an initial estimate of the MDL and the level to be used to spike the XAD-2 resin for the MDL determination. This approach is consistent with 40 CFR 136 Appendix B which offers four options. Method 429 differs by adding a fifth option, the TMQL. No change was made.

7. Comment by Mr. Wang on Section 2.4.2: Mr. Wang stated that ARB needs to provide guidance on data handling procedure for averaging of field blank/sample blank corrected data reduction [sic].

Agency Response: This comment is addressed in the response to Comment #21, below, on Method 429.

8. Comment by Mr. Wang on the use of methylene chloride: Mr. Wang stated that ARB should review the use of methylene chloride and look for alternative solvents that pose a lower risk to sampling and analysis personnel and to the environment.

Agency Response: The use of methylene chloride as an extraction solvent is consistent with EPA Method 8270 requirements. A consequence of this is its use for cleaning of glassware and recovery of the sampling train. There was no change.

9. General Comment by Mr. Wang on Method 429: Mr. Wang stated that ARB should accept SW-846 Method 8270a in selective ion monitoring (SIM) as an alternative to dilution isotope mass spectrometry. Method 8270a SIM is a cost-effective alternative to

the more expensive ARB method. Method 8270a SIM is recognized by EPA as a performance-based method alternative.

Agency Response: The ARB will not approve Method 8270A as an alternative analytical procedure because it is not equivalent to the isotope dilution mass spectrometry procedure required by Method 429. The ARB used EPA Method 8270 until there were enough deuterated standards available to develop the improved procedure required by Method 429. Also, judging from the number of inquiries that we have received since the adoption of Method 429 in 1989, we believe that Method 429 is being used for a significant portion of PAH testing conducted throughout the country.

The isotope dilution technique required by Method 429 requires quantitation of fourteen of the nineteen target PAH compounds by their deuterated analogues. This means that similarity assumptions are required for only the five target analytes that are quantified by PAH other than their deuterated analogues. In contrast, Method 8270 requires similarity assumptions for fourteen target analytes because the method requires only five deuterated standards for quantitation of the nineteen target analytes. The isotope dilution technique required by Method 429 is the superior procedure because of the larger number of labeled standards and the need for fewer similarity assumptions than Method 8270.

In Method 429, the 15 deuterated internal standards are added to the sample prior to extraction. Quantitation is therefore based on internal standards that have been through the same sample preparation steps as the analyte in the sample. Consequently, Method 429 provides an automatic correction for extraction and cleanup losses. Quantitation using Method 8270 is based on five deuterated standards that are added to the sample extract just prior to analysis. Therefore, Method 8270 must include an added procedure for adjusting for losses during sample preparation and cleanup. This is accomplished by adding three surrogate standards (d_{14} -terphenyl, 2-fluorobiphenyl and d_8 -nitrobenzene) to the sample before extraction. These standards are not PAH. Nevertheless, Method 8270 allows the use of the recoveries of these compounds to demonstrate the performance of the procedure and also to adjust the concentrations of the 19 target PAH. Method 8270 assumes that these three compounds behave the same as the target PAH. Since Method 429 does not rely on such assumptions, we believe that it is the superior method.

The cost of standards for Method 429 may be higher than that for Method 8270, but the difference is small. The initial cost of acquiring standards is considerable but the cost of labeled standards per sample is a small fraction of that initial cost. In 1991, we estimated that the total cost of labeled and unlabeled standards for a single calibration run is less than \$2.50. Our estimate of the total cost of standards for one sample and one five-point calibration curve was less than \$10.00. We consulted two commercial laboratories about our estimate and both agreed that it was realistic.

10. Comment by Mr. Wang on Section 2.3.1: Mr. Wang stated that blank contamination needs further discussion.

Agency Response: The method contains adequate instructions for the pretest determination of blank contamination of the sampling media (filters and resin). Section

2.3.1 contains references to (1) Sections 4.2.1 and 4.2.2, which describe the procedures to be used to clean, analyze and store the sampling media, and (2) Sections 10 and 10.1, which contain the reporting requirements. Also Section 2.5 describes how the results of the blank contamination checks are to be used in the estimation of sampling parameters. No changes were made.

11. Comment by Mr. Wang on Sections 2.3.2 and 2.3.3: Mr. Wang stated that the requirement for the MDL could be misinterpreted as meaning to be performed with every batch. Whereas, the intent is that a current MDL be available. An MDL per every experimental set would be prohibitively expensive. Mr. Wang requested that ARB see 8.3.1 and General Comments.

Agency Response: ARB believes the requirements of this section are clearly stated. The first statement assigns the responsibilities for the MDL determination, the second establishes limits on the applicability of the MDL procedure, and the third refers to the sections (8.3 and Appendix A) that describe the required MDL procedure.

Section 8.3.2 contains the performance criteria to be used by the analyst to decide how frequently the MDL must be determined. These performance criteria were established in consultation with the representatives of the commercial laboratories that participated in the ARB test method workshops. No change was made to the method.

12. Comment by Mr. Wang on Section 4.2.1: Mr. Wang asked ARB to check the reference year and to see his General Comments.

Agency Response: The following change was made to make the method consistent with EPA Method 5.

ARB added "(Reapproved 1978)" after "ASTM standard Method D2986-71."

13. Comment by Mr. Wang on Section 4.2.2.1: Mr. Wang asked that ARB define "giant" Soxhlet.

Agency Response: After 15-day notice of the availability of modified text, the following change was made to the final regulation:

The word "giant" was deleted. This term is not necessary as this section already states that the procedure must be carried out in a Soxhlet extractor "which will hold enough XAD-2 for several sorbent traps, method blanks and QC samples."

14. Comment by Mr. Wang on Section 4.2.2.4: Mr. Wang asked that ARB define accept criteria for XAD-2 contamination.

Agency Response: The acceptance criteria are defined in the second and third paragraphs of Section 4.2.2.4 and corrective action is described in the fourth paragraph. No change to the method was necessary.

15. Comment by Mr. Wang on Section 4.2.7: Mr. Wang asked that ARB define acceptable drying time as 0.5-1 hour.

Agency Response: The method was not changed to define an acceptable drying time. However, the following change was made to the cleaning procedure in the final regulation after 15-day notice of the availability of modified text:

The current section was deleted and replaced with "Clean by methylene chloride Soxhlet extraction for 16 hours. Air dry in a clean container in a clean hood. Store in methylene chloride washed glass jar with TFE lined screw cap."

16. Comment by Mr. Wang on Section 4.2.8: Mr. Wang asked that ARB develop an alternative to chromic acid.

Agency Response: The chromic acid requirement is consistent with the EPA Method 23 requirement for cleaning glassware for dioxin and furan emissions testing. Also, Section 4.3.2 of Method 429 allows other cleaning procedures "as long as acceptable blanks are obtained." Acceptance criteria for blanks are defined in Section 8.2. No change to the method was necessary.

17. Comment by Mr. Wang on Section 4.3.1: Mr. Wang stated that the ARB should refer to EPA QA Handbook for Air Pollution Monitoring Systems 600/4-79-027 a, b, c Volume II with updates.

Agency Response: The reference (APTD-0576) was not changed. This is a standard reference used in EPA Method 5.

18. Comment by Mr. Wang on Section 4.3.2: Mr. Wang stated that the reference 11.4 was superseded and asked that ARB see his comment on 4.2.8.

Agency Response: This reference for the glassware cleaning procedure is the same one referenced by EPA Method 23 for the preparation of sampling trains for dioxin and furan emissions testing. See also the ARB's response to WSPA's Comment #12 on Section 4.2.8. No change to the method was made.

19. Comment by Mr. Wang on Section 6.0: Mr. Wang stated that this section does not address demonstration of proficiency such as running acceptable blind quality control samples and achieving EPA requirements.

Agency Response: Section 6.0 refers to the sections (7.3, 8.2.6, and 8.3.1) that address the demonstration of the ability to generate acceptable results. No changes were made.

20. Comment by WSPA on Section 8.3.1: WSPA state that the TMQL concept is flawed. WSPA directed ARB to see the discussion in its General Comments on MDL.

Agency Response: Section 8.3.1 was changed as indicated below, but the theoretical method quantitation limit (TMQL) concept is retained. The TMQL concept was proposed in method development workshops and the commenter has not proposed a better approach. The TMQL is not used to define the method detection limit (MDL); it is used only to establish an initial spike level for the MDL determination. The analyst may use the TMQL or any of the other four approaches suggested in Appendix A for determining the initial spike level. Those four were taken from the EPA procedure included as Appendix B to Title 40, Part 136 of the Code of Federal Regulations (40 CFR 136).

After the 15-day notice of the availability of the modified text, the following was deleted from Section 8.3.1: "A suggested initial spike level for the MDL determination is 5 times" and was replaced with "The analyst may use any of the five approaches described in Appendix A (A1.1) to estimate an initial spike level for the MDL determination. One of the suggested approaches is based on"

21. Comment by Mr. Wang on Section 9.2.4.2: Mr. Wang state that ARB needs to provide guidance on data handling procedure for averaging of field blank/sample blank corrected data reduction [sic].

Agency Response: Mr. Wang described the treatment of the results from two field blank runs, but only one is required by the method. The commenter's proposal for adjusting the field results is not acceptable because it allows the reporting of values lower than the MDL of the method. No change to the method was made. As currently required by the method, the field blank train will be used to establish the reporting limit for each emissions test.

22. Comment by Mr. Wang on 4.3.5: Mr.Wang state that the text should read, "Teflon-lined."

Agency Response: "Teflon lined" was replaced with "Teflon-lined."

23. Comment by Mr. Wang on 4.4.3: Mr. Wang stated that either "Leak Check" or "Leak-Check" should be used with consistency in use throughout this section. He stated, "Li = leak check."

Agency Response: The method was revised so that "Leak-Check " is used consistently.

24. Comment by Mr. Wang on 4.5.5: Mr.Wang stated that K2 = m3/ml or ft3/ml. He suggested superscripts.

Agency Response: The subscripts were changed to superscripts (m³ or ft³).

25. Comment Mr. Wang on 6.1: Mr. Wang stated that Material Safety Data Sheets is correct terminology.

Agency Response: After the 15-day notice of the availability of the modified text, the following change was made to the final regulation: ARB added "safety" before data and deleted "handling."

26. Comment by Mr. Wang on 11.2 and 11.3: Mr. Wang stated that Bibliography 11.2 and .3 are out of date.

Agency Response: References 11.2 and 11.3 are the cleanup procedures current in the most recent update of SW846. Section 6.6 of Method 429 also describes cleanup procedures and performance requirements. It also contains the following statement that allows some flexibility in the performance of the cleanup requirements of the method: "Acceptable alternative procedures may also be used provided that the analyst can demonstrate that the performance requirements of Sections 8.1.3.1 and 8.2.6 can be met." No changes were made to the method.

27. Comment by Mr. Wang on Appendix A: Mr. Wang asked that ARB revise Appendix A after review of the limits. He also asked ARB to see his General Comments.

Agency Response: Staff determined that there was no need to change Appendix A. See the response to Comment 20 on Method 429.

G. Method 431, Determination of Ethylene Oxide Emissions from Stationary Sources

The following comments on Method 431 are all by Mr. Clinton Cooney, San Diego Air Pollution Control District (APCD). Page numbers refer to the August 9, 1996 staff report.

1. Comment on Appendix A (page 3): Mr. Cooney stated that the title of Appendix A should not reference sterilizers, but rather should reference control units.

Agency Response: Changing the title of Appendix A would not alter the effectiveness of the method. No change was made.

2. Comment on Appendix A, Stack Gas Moisture (page 3): Mr. Cooney stated that a moisture measurement should not be required for catalytic units, rather allow a wet/dry bulb measurement of ambient moisture.

Agency Response: After the 15-day notice of the availability of the modified text, the method was revised in the final regulation as the commenter suggested.

3. Comment on Appendix A, Stack Gas Moisture (page 3): Mr. Cooney stated that for hydrolytic units, require only a wet bulb/dry bulb humidity measurement of ambient air and the temperature of the outlet stream.

Agency Response: After the 15-day notice of the availability of the modified text, the method was revised in the final regulation as the commenter suggested.

4. Comment on Appendix A, Stack Gas Moisture (page 3): Mr. Cooney stated that the above comments apply even when flow measurements are required.

Agency Response: The method was revised as discussed in agency responses to comments 2 and 3.

5. Comment on Appendix A, Hydrolytic scrubber type units (page 3):
Mr. Cooney stated that the Method should not require measuring the moisture of the inlet stream for hydrolytic scrubbers, due to the health hazard of high concentrations of ethylene oxide.

Agency Response: After the 15-day notice of the availability of the modified text, the method was revised in the final regulation to omit the term "measured" from the discussion of stack gas flow rate measurement for hydrolytic scrubbers in Appendix A. Inlet estimation procedures are discussed in Appendix B.

6. Comment on Appendix A, Hydrolytic scrubber type units (page 3):
Mr. Cooney suggested that ARB insert the following: For the Joslyn system there is no flow rate, so there would be no requirement to measure the flow.

Agency Response: Appendix K on the testing procedures for Joslyn recovery type control units was added to the method. The revision was noticed during the 15-day notice period.

7. Comment on Appendix A, Determination of Ethylene Oxide Concentration at the Inlet of Control Units (page 5): Mr. Cooney stated that the subtitle, Determination of Ethylene Oxide Concentration at the Inlet of Control Units, contradicts the main title of the appendix.

Agency Response: Changing the title or subtitle of this appendix in the manner suggested would not have altered the effectiveness of this method. No change was made.

8. Comment on Appendix A, Option 1, Inlet Estimation (page 5): Mr. Cooney asked what sites where inlet estimation is necessary do not have water ring seal pumps.

Agency Response: Because no change was proposed by the commenter, the method was not revised.

9. Comment on Appendix A, Option 2, Inlet Measurement (page 5): Mr. Cooney asked why does the method state that the sterilization cycle should not be aborted for a test of the aeration cycle. He stated that this procedure is an economic burden on a hospital.

Agency Response: The commenter did not explain how this would impose an economic hardship on a hospital. Furthermore, this condition allows the hospital to include a normal load in the sterilizer and thus would not result in any additional cost. No change was made.

10. Comment on Appendix A, Option 2, Inlet Measurement (page 5): Mr. Cooney stated that documenting the position of the probe by making extra measurements is burdensome.

Agency Response: Because the commenter has not recommended a change to the method, no change was made.

11. Comment on Appendix A, Tedlar bag sampling/analysis procedure (page 6): Mr. Cooney stated that it is too difficult to plan a test to obtain an adequate sample in a Tedlar bag.

Agency Response: The commenter did not explain why he has difficulty planning a test to obtain an adequate sample. No change was made.

12. Comment on Appendix A, Tedlar bag sampling/analysis procedure (page 6): Mr. Cooney stated that using Tedlar bags for ethylene oxide sampling is hazardous to laboratory personnel and results in a disposal problem.

Agency Response: Tedlar bag sampling for ethylene oxide and other toxic compounds has been accepted by a number of test groups as a safe procedure if appropriate safety procedures are carried out. Safety concerns are covered in the method, Appendix I, Section 5.11. No change was made.

13. Comment on Appendix A, Tedlar bag sampling/analysis procedure, third paragraph (page 6): Mr. Cooney stated that requirements for cat-ox and acid scrubbers are a contradiction to the Rule. [The "Rule" refers to San Diego APCD Rule 1203.]

Agency Response: The requirement for integrated bag sampling of acid scrubbers after the first evacuation was deleted from Appendix A, Measurement Methods, Tedlar bag sampling/analysis procedure, Option 2. The revision was noticed during the 15-day notice period.

14. Additional comment on Appendix A, Tedlar bag sampling/analysis procedure, third paragraph (page 6): The commenter provided the information that he has tested air washes.

Agency Response: Because no suggestion for changing this method was given, the method was not revised.

15. Comment on Appendix A, Tedlar bag sampling/analysis procedure, seventh paragraph (page 6): Mr. Cooney stated that if only one sterilizer is connected to the control device, and the first two subtests show the unit to be overwhelmingly passing, do not require a third test.

Agency Response: This suggestion would violate the federal National Emission Standards for Hazardous Air Pollutants (NESHAPS) for ethylene oxide commercial sterilization and fumigation operations (40 CFR Parts 9 and 63). Therefore, no change was made.

16. Comment on Appendix A, Direct Interface Sampling Analysis (page 6):

Mr. Cooney stated that sampling frequency of less than two minutes for catalytic units is too restrictive. Similarly, a sampling frequency of less than one minute for hydrolytic scrubbers is also too restrictive.

Agency Response: This suggestion is contradictory to the EPA NESHAPS (see response to comment 15). No change was made.

17. Comment on Appendix A, Inlet Estimation, paragraph two (page 6):

Mr. Cooney stated that the requirement that the wash period be monitored for emissions contradicts [San Diego APCD] Rule 1203.

Agency Response: This procedure is only applicable when the inlet estimation technique is used in conjunction with monitoring at the control unit inlet. As the inlet estimation technique is a mass balance approach, the entire ethylene oxide (EtO) emission from the sterilization chamber must be monitored at the control unit outlet. When actual monitoring is conducted at the control unit inlet (as required by Rule 1203), as opposed to using the inlet estimation technique, then only the "entire first evacuation" must be monitored. The test method was not changed.

18. Comment on Appendix A, Direct Interface Sampling Analysis, paragraph three (pages 6 and 7): Mr. Cooney stated that it is incorrect to require only the evacuation be tested for catalytic units, but require the evacuation and air washes be tested for hydrolytic units. This also contradicts Rule 1203.

Agency Response: After the 15-day notice of the availability of the modified text, the method was revised in the final regulation to delete this statement.

19. Comment on Appendix A, Direct Interface Sampling Analysis, last paragraph (page 7): Mr. Cooney stated that three test runs are burdensome.

Agency Response: See response to Comment 15. No change to the method was made.

20. Comment on Appendix B (page 8): Mr. Cooney stated that water-ring-seal pumps are often used on units where inlet estimation is needed.

Agency Response: The commenter has provided information only, and has not recommended a change to the method. No change to the method was made.

21. Comment on Appendix B (page 8): Mr. Cooney stated that the method should allow for expressing the pressure P in units of atmospheres.

Agency Response: The method was revised to allow chamber pressure to be expressed in atmospheres. The revision was noticed during the 15-day notice period.

22. Comment on Appendix C (page 9): Mr. Cooney stated that to be consistent with requirements in Appendix A, the term "should" should be changed to "cannot" in the following expression:

"The exposure stage should not be shortened . . ."

Agency Response: The proposed change from "should not" to "cannot" was made. The revision was noticed during the 15-day notice period.

23. Comment on Appendix D (page 10), points 1 through 3, top of page: Mr. Cooney stated that this procedure will expose test personnel to extremely high levels of ETO and is expensive.

Agency Response: The referenced procedure was deleted. The revision was noticed during the 15-day notice period.

24. Comment on Appendix D (page 10): Mr. Cooney stated that the correct positioning of a probe can be determined by a detailed engineering drawing.

Agency Response: It has been the experience of ARB staff that in years of working at industrial sites, that elusive "detailed engineering drawing" most often does not exist. Many times the available engineering drawings are original design plans that may not adequately represent the modified configuration of an operating unit. Further, no engineering drawing can provide information regarding flow rate or concentration stratification. No change was made.

25. Comment on Appendix D (page 10), second group of three points: Mr. Cooney stated that the Inlet Estimation technique is subject to serious error due to pressure gauge readings. Requiring inlet estimation as part of a measurement method seems contradictory.

Agency Response: Appendix B of the method specifies that a calibrated differential pressure gauge be used. There should be no reason to suspect that pressure measurements would be less accurate than other types of test measurements. The estimation procedure is necessary to provide data to indicate that the probe is positioned in a manner to correctly measure inlet flow. No change was made to the method.

26. Comment on Appendix D (page 10), second group of three points: Mr. Cooney stated that the requirement to perform two velocity traverses simultaneously is burdensome for a one-man source test company.

Agency Response: The process of obtaining correct measurement of ethylene oxide concentrations and flow rates should not be compromised to accommodate a one-person test company. No change to the method was made.

27. Comment on Appendix D (page 11), Part 5: Mr. Cooney stated that the requirement to perform this procedure three times is burdensome.

Agency Response: See response to Comment 15. No change was made.

28. Comment on Appendix E, Section 4.1.1: The method should clearly indicate if one of the calibration standards is zero gas.

Agency Response: Mr. Cooney stated that the method clearly states that the low end concentration is not zero gas, but five times the limit of detection. No change was made.

29. Comment on Appendix E, Section 4.1.3: The commenter asked for clarification regarding how standard deviations are determined.

Agency Response: The method was revised to state that the standard deviation of the gas chromatograph area counts is calculated at each level of the multi-point calibration and must be included in the analytical report. The revision was noticed during the 15-day notice period.

30. Comment on Appendix E, Section 4.1.5: Mr. Cooney requested further explanation of the term S in the determination of the analytical limit of detection.

Agency Response: The method was revised to specify how S, the standard deviation of replicate determinations of the lowest calibration standard, is calculated.

31. Comment on Appendix E, Section 5: Mr. Cooney stated that the term "Laboratory" should be inserted in the title of this section.

Agency Response: Adding the term "Laboratory" to the title of Section 5 would not alter the effectiveness of the method. No change was made.

32. Comment on Appendix E, first paragraph of Section 5: Mr. Cooney stated that daily calibration should not be required due to travel time needed for the source tester to reach a site.

Agency Response: The term "daily" was deleted. The revision was noticed during the 15-day notice period.

33. Comment on Appendix E, Sections 5.1, 5.2, 6.3, and 7.4: Mr. Cooney stated that the eight-hour calibration checks should be revised to accommodate personnel who work 10-hour shifts.

Agency Response: The eight-hour calibration checks were revised to ten-hour calibration checks in Sections 5.1, 5.2, and 7.4.

34. Comment on Appendix E, Section 8: Mr. Cooney stated that in-field calibration checks should be performed after each subtest.

Agency Response: Section 8.5 already contains the requirement requested by the commenter. No change was made.

35. Comment on Appendix F: Mr. Cooney stated that the description of ethylene oxide calculations is unwieldy and better wording is needed.

Agency Response: No specific change was proposed by the commenter; no change was made.

36. Comment on Appendix F, direct GC approach, page 18: Mr. Cooney stated that the one- and two-minute sampling intervals are unrealistic.

Agency Response: See response to Comment 16. No change was made.

37. Comment on Appendix G: Mr. Cooney stated that the reporting requirements are discussed previously in Appendix E, Section 7.

Agency Response: A reference in Appendix G was added to alert the reader to the more detailed discussion of analytical reporting requirements specified in Appendix E.

38. Comment on Appendix H, Tedlar Bag Sampling: Mr. Cooney suggested that ARB omit the requirement for use of a continuous FID in conjunction with the Tedlar bags as the tester can determine the end of the cycle by the equipment indicator dials.

Agency Response: The requirement of simultaneous use of a continuous monitor with Tedlar bag sampling was deleted. The revision was noticed during the 15-day notice period.

39. Comment on Appendix H, On-Site GC: Mr. Cooney suggested that ARB omit the comment that on-site GC is more expensive than container sampling because that comment may reflect merely a personal attitude.

Agency Response: The statement which the commenter cited was omitted from the method. The revision was noticed during the 15-day notice period.

40. Comment on Appendix H, Inlet Estimation, top of page 22: Mr. Cooney stated that the requirement for determining the concentration of the site ethylene oxide cylinder should be eliminated due to the expense and the safety hazards associated with the use of several high concentration ethylene oxide cylinders in such a procedure.

Agency Response: The method was revised to include the determination of the concentration of the site ethylene oxide cylinder only at the discretion of the District. The revision was noticed during the 15-day notice period.

41. Comment on Appendix H, Inlet Estimation, middle of page 22: Mr. Cooney stated that the estimation method should not include the use of pressure gauges, as these gauges are usually not accurate and are difficult to calibrate.

Agency Response: The commenter is incorrect to state that pressure gauges are categorically inaccurate and difficult to calibrate. For example, depending on the pressure range, gauges can easily be calibrated against a primary standard mercury manometer. No change was made.

42. Comment on Appendix H, Acid Scrubber, bottom of page 22 and Comment on Appendix H, Acid Scrubber, bottom of page 23: Mr. Cooney stated that the method should not disallow use of the estimation method for systems with water-ring-seal pumps.

Agency Response: Discharge of EtO in the water used as the working seal fluid in flow-through-water-ring-seal pumps is specifically prohibited by the Statewide ATCM. Use of recirculating-water-ring-seal pumps to evacuate a sterilization chamber may cause a shift of EtO transfer which may invalidate the mass balance approach of the inlet estimation technique. No change was made to the method.

43. Comment on Appendix H, Acid Scrubber, bottom of page 22: Mr. Cooney stated that the redundant quality control checks of the method result in testing personnel being exposed to high concentrations of ethylene oxide.

Agency Response: The commenter did not indicate what procedures of the method he considers to be unsafe and redundant. Therefore, no change was made.

44. Comment on Appendix H, Acid Scrubber, top and middle of page 23: Mr. Cooney stated that the one minute and two minute restrictions should not be stipulated for hydrolytic scrubbers or for catalytic oxidation units.

Agency Response: No change was made. See response to comment 16.

45. Comment on Appendix H, Acid Scrubber, middle and bottom of page 23: Mr. Cooney stated that proper probe placement can be determined from engineering drawings.

Agency Response: No change was made. See response to comment 24.

46. Comment on Appendix I, Section 3: Mr. Cooney suggested that ARB clarify what is needed to demonstrate equivalency.

Agency Response: ARB staff is currently working with USEPA to develop guidance on classification of test method modifications and the corresponding level of approval required. Until this guidance is approved, the ARB staff considers all modifications of ARB test methods subject to Executive Officer approval unless specifically allowed in the method. This is important to maintain statewide consistency in applying test methodologies. No change to the method was made.

47. Comment on Appendix I, Section 5.1: Mr. Cooney stated that a wet/dry bulb measurement should be sufficient.

Agency Response: The option for a wet/dry bulb measurement was provided in revisions to Appendix A, as described in responses to comments 2 and 3. No additional changes in Appendix I were made.

48. Comment on Appendix I, Section 5.3: Mr. Cooney stated that impingers containing liquids should not be used in the sampling train.

Agency Response: Guidelines regarding the use of impingers in the sampling train were omitted from the method. The revision was noticed during the 15-day notice period.

49. Comments on Appendix I, Sections 6.1.1 and 6.2.2: Mr. Cooney stated that repetition of sampling requirements here is redundant; a field spike should not be required.

Agency Response: Emphasis of quality control requirements is important and deserves reiteration in this section. Field spikes are an essential part of quality control. No changes to the method were made.

50. Comment on Appendix I, Section 6.2.2: Mr. Cooney stated that nitrogen should not be allowed as a diluent for the field spike.

Agency Response: Staff disagrees with the comment that the use of nitrogen as a diluent gas would interfere with EtO quantitation on gas chromatography/flame ionization detection (GC/FID). No change was made.

51. Comment on Figure 1: Mr. Cooney stated that the rotameter should not be placed between the probe and the Tedlar bag, but rather it should be placed between the can and the sample pump.

Agency Response: The procedure shown in Figure 1 is identical to the procedure detailed in ARB Method 422, "Standard Operating Procedure for the Sampling of Volatile Organic Compounds in Emissions from Stationary Sources into Tedlar Bags." No change was made.

52. Comment on Figure 4: Mr. Cooney stated that the calculations in Appendix F use total flow, not average flow. Figure 4 requires average flow, which seems inappropriate.

Agency Response: The calculations in Appendix F require calculation of total volume, not total flow. As stated in the method, the volume is determined by integration of flowrate versus sampling time. No change was made.

53. Comment on Appendix I, Section 7: Mr. Cooney stated that analysis and disposal of Tedlar bags which contain ethylene oxide will result in a safety hazard.

Agency Response: See response to Comment 12. No change was made.

H. Method 436, Determination of Multiple Metals in Emissions from Stationary Sources

1. Comment by Mr. Higuchi of the South Coast Air Quality Management District regarding reporting limits: Mr. Higuchi stated there are too many options for establishing reporting limits.

Agency Response: The method is clear in indicating that reporting limits are based upon the field blank sample train or the field reagent blanks. Reporting limits used for test planning are solely for the purpose of estimating the required sample volume and have no bearing on the final reporting limit. No change to the method was made.

2. Comment by Mr. Higuchi regarding sample train configuration (Section 4.1): Mr. Higuchi stated that the location of the filter in the sample train introduces unnecessary sample divisions, additional contamination, and more stringent sample digestion.

Agency Response: The location of the filter in the sample train was selected to maintain consistency with EPA Method 29. Alternative sample train configurations may be approved after demonstrating statistical equivalency based upon completion of EPA Method 301 validation testing. No change to the method was made.

3. Comment by Mr. Higuchi on Section 5.1.2: Mr. Higuchi stated that requirements were needed for pre-cleaning all glassware that the sample comes in contact with.

Agency Response: Section 5.1.2 of Method 436 was modified to include cleaning of all previously used funnels, flasks, beakers and storage containers. The revision was noticed during the 15-day notice period.

4. Comment by Mr. Higuchi regarding blank preparation: Mr. Higuchi stated that sample blanks should be reserved under Sample Train Assembly rather than Sample Recovery.

Agency Response: Blank samples are addressed under Section 5 "Sample Collection and Recovery Procedures" to maintain consistency with other ARB Methods. No change to the method was made.

5. Comment by Mr. Higuchi: Mr. Higuchi stated that the sample pH should be tested during Sample Recovery rather than during Analytical Preparation.

Agency Response: Section 5.2 and Section 9.4.1 of Method 436 were modified to include testing and adjustment of sample pH during Sample Recovery. The revision was noticed during the 15-day notice period.

6. Comment by Mr. Higuchi regarding reagent blanks: Mr. Higuchi stated that reagent blanks should be referred to as field reagent blanks.

Agency Response: Method 436 text was modified by changing reagent blank to field reagent blank as requested. The revision was noticed during the 15-day notice period.

7. Comment by Mr. Michael Wang of Western States Petroleum Association regarding Section 2.3: Mr. Wang stated that the laboratory should provide MDL's determined within the last year.

Agency Response: MDL values in Section 2.3 and Table 1 are estimated values only. Source test contractors should obtain MDL data from the laboratory performing analysis prior to performing Method 436. No change to the method was made.

8. Comment by Mr. Wang regarding Section 7.1: Mr. Wang stated that the HF matrix guidance for use of the alumina torch needed clarification.

Agency Response: The note in Section 7.1 states ". . . since all front half samples will contain HF, use an alumina torch." No change was made to the method.

9. Comment by Mr. Wang regarding Section 7.3: Mr. Wang commented that mercury analysis is problematic and biased.

Agency Response: Note 2 in Section 7.3 permits mercury analysis using automated analytical systems such as the Leeman Model PS200, Perkin Elmer FIAS systems and similar models. Use of these systems may reduce concerns expressed by Mr. Wang. No specific changes to the method were proposed by Mr. Wang; no change was made to the method.

10. Comment by Mr. Wang regarding Section 9.4.1: Mr. Wang stated that the sample holding time preservation requirements should be immediate.

Agency Response: The comment is unclear. However, the commenter may be referring to the same issue as that discussed by Mr. Higuchi in comment 5. See response to Comment 5 on Method 436.

11. Comment by Mr. Wang regarding Figure 13: Mr. Wang stated that the frequency of QA/QC is excessive in Figure 13.

Agency Response: Figure 13 was modified to reflect the quality assurance and quality control requirements in EPA Method 29. The revision was noticed during the 15-day notice period.

12. Comments by Mr. Wang regarding Sections 11.4.1, 11.4.2, 11.4.5, 13.3.1, 13.3.2, 17.1.1, 17.1.2 and 22.

Agency Response: These section numbers are not contained in Method 436.

13. Comment by Mr. Wang on the Method 436 references: Mr. Wang stated that ARB Method 436 cites the September 1988 version of the EPA Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846 Third Edition. The more current updates of these EPA methods should be cited.

Agency Response: The correct adopted revision date for each cited EPA method was added to the method references in Section 13. The revision was noticed during the 15-day notice period.

14. Comment by Mr. Wang on Section 2.3: Mr. Wang stated that the MDL discussion in Method 436.2.3 is actually the analytical detection limit.

Agency Response: The method detection limit (MDL) defined in Method 436, Section 1.3.4 is consistent with the MDL definition in the EPA's Test Methods for Evaluating Solid Waste, SW 846 Chapter 1, Revision 1. No change was made to the method.

15. Comment by Mr. Wang on Table 2: Mr. Wang suggested the table be renamed.

Agency Response: Renaming the table does not alter the effectiveness of the method. No change was made to the method.

16. Editorial comment by Mr. Wang on Section 9.5.3: Mr. Wang stated that the standard methods for water and wastewater cited in this section should be omitted.

Agency Response: The incorrect citation of Method 303F was replaced with the correct citation of Methods 245.1 and 245.2. The revision was noticed during the 15-day notice period.

17. Editorial comments by Mr. Wang on Sections 4.2.1, 4.5.37.2, 4.7.13, and Table 1 are unclear; therefore, no changes were made to the method.

18. Comment by Mr. Jim Steiner, Steiner Environmental, Inc., on Section 5.1.8: Mr. Steiner stated that since the field blank train is sealed immediately after leak check, there is no overwhelming reason to leave the sealed train in the test area for the duration of sampling.

Agency Response: Section 5.1.8 of Method 436 was modified to make it consistent with the other air toxics methods by eliminating any reference to the amount of time the blank train remains in the sampling area. The revision was noticed during the 15-day notice period.

19. Comment by Mr. Steiner on Section 5.2: Mr. Steiner suggested that ARB modify the section to specifically allow nozzle and probe cleaning on the stack.

Agency Response: The location where probe recovery is performed should be selected primarily on the cleanliness of the location, with due consideration to logistics, not its convenience to the tester. However, it is true that many sample platforms provide a suitable environment for probe recovery when compared to ground conditions in the sampling area. Since Section 5.2 of Method 436 requires "a cleanup area that is clean and protected from the wind and other potential causes of contamination," this section should not be modified by either adding or deleting text which may ultimately serve to limit the recovery options available to the tester. No changes to the method were made.

20. Comment by Mr. Steiner regarding Section 9.4.1: Mr. Steiner stated that five days is an insufficient time period between sampling and storage in pH 2 acid solutions.

Agency response: The method was modified to specify that the sample is acidified during sample recovery, as noted in the agency per response to Comment 5 (Mr. Higuchi). The five-day limit on holding time was also removed. The revision was noticed during the 15-day notice period.

21. Comment by Gene Riley of Triangle Labs on Section 13, Bibliography: Mr. Riley stated that several of the EPA/OSW Reference Methods have not been updated.

Agency Response: See agency response to comment 13 (Mr. Wang).

VI. Summary of Comments received during the 15-day comment period

During the 15-day comment period between April 1 and April 15, 1997, three additional sets of comments were received on the proposed methods. Two comment letters pertained to Method 100; a third letter included comments on Method 431. The comments are summarized below, together with agency response to these comments.

A. Comments on Method 100, Procedures for Continuous Gaseous Emission Stack Sampling

1. Comments by Ms. Suzanne Blackburn of the San Diego Air Pollution Control District regarding Sections 6.1, 6.2, and 7.2: Ms. Blackburn provided editorial corrections for the terms in equations 100-1, 100-2, and 100-3. She also suggested an alternative approach to determining sampling system bias.

Agency Response: Sections 6.1, 6.2, and 7.2 of Method 100 were not modified after August 9, 1996, and hence were not included in the 15-day notice. Although Ms. Blackburn's comments do not address the 15-day package, the proposed editorial corrections for Sections 6.1 and 6.2 were made. These editorial corrections clarify the terms of the equations, but are not substantive changes which alter the method or the method's effectiveness.

2. Comment by Mr. Jim Steiner of Steiner Environmental Services on Section 3.1: Mr. Steiner state that the method should be revised to require that calibration gases must be EPA Protocol 1 gases, or NIST $\pm 2\%$ only if an EPA Protocol gas is not available.

Agency Response: The text of Section 3.1 to which Mr. Steiner referred was not modified after August 9, 1996, and hence was not included in the 15-day package. No change to the method was made.

3. Comment by Mr. Steiner on Section 3.2: The commenter requested that the "should" be changed to "must" in the following statement from Method 100, Section 3.2, included in the 15-day package:

The calibration gas used with a dilution system should be an EPA Protocol gas.

Agency Response: EPA Protocol gases may not be available for a specific constituent in a necessary concentration range. In addition, the "should" in Section 3.2 of ARB Method 100 is consistent with the text in the U.S. EPA's Method 205, Section 2.2:

An EPA Protocol calibration gas is recommended, due to its accuracy, as the high-level supply gas.

For these reasons, no change was made to the ARB method.

B. Comments on Method 431, Determination of Ethylene Oxide Emissions from Stationary Sources

Mr. Daniel Kremer of Kremer Environmental Services submitted comments on Method 431 in response to the 15-day changes. Although much of Mr. Kremer's letter concerns portions of the method not included in the 15-day package, the following comments address the 15-day changes.

1. Comment by Mr. Kremer regarding page 11 of Appendix D from the 15-day changes: Mr. Kremer stated that the procedure to verify the position of the probe cannot be applied to aeration room testing, or to any system which utilizes a liquid sealed vacuum pump. The procedure should be changed to a physical/positional requirement for inlet probe placement which can be easily verified by the tester.

Agency Response: We agree that the procedure to verify the position of the probe cannot be applied to aeration room testing where the aeration room control system is physically separate from the sterilization chamber control system. However, most sterilization/aeration systems in California utilize shared control systems and so inlet probe positioning can be documented during a sterilization chamber run. The probe position would be identical for the shared aeration room test. The few cases where catalytic oxidation control systems are dedicated solely to aeration rooms must be reviewed and approved on a case-by-case basis.

We also agree that implementation of the procedure becomes complicated with a system which utilizes a liquid-sealed-vacuum pump. The previously proposed alternative procedure was deleted in the 15-day package due to safety concerns. We plan to address the need for an alternative procedure in the next revision of the method. In the interim, the method contains provisions for case-by-case approval of alternative procedures by the ARB Executive Officer (see Appendix H, first paragraph).

In addition, we agree that a physical/positional requirement would be a simpler procedure. However, we are not aware of valid physical/positional siting criteria for catalytic oxidation units. No inlet probe physical/positional siting criteria were proposed by the commenter. For these reasons, no change was made to the method.

The following comments concern Appendix K, which was new text in the 15-day package.

2. Comments by Mr. Kremer regarding Sections 1 and 2 under identified points of ethylene oxide emissions from the Joslyn system: Mr. Kremer stated: (1) The recovery "burps" may be discharged to either an acid or a thermal hydrolysis scrubber, (2) the 12/88 sterilant gas is no longer available, and (3) the chamber moisture is normally discharged only once per cycle, rather than discharged several times during the cycle.

Agency Response: The suggested corrections to Appendix K will not alter the effectiveness of Method 431. However, these comments may be considered when the method is revised.

3. Comment by Mr. Kremer regarding Section 3.5 on page 40: Mr. Kremer stated that no specific guidelines are given for the collection and analysis of water samples from the outlet of the chamber water jacket. How should the samples be collected? How many samples should be collected? When during the cycle should these samples be collected? How should the sample analysis be performed? Mr. Kremer also indicated that he has significant experience in testing liquid discharge streams from the Joslyn System and would provide test methods and data to the Air Resource Board.

Agency response: Staff agrees that specific guidelines may need to be included in this test method in order to produce consistent, valid data. However, Method 431 does not yet include water sampling/analysis procedures for ethylene oxide. Staff welcomes the receipt of test methods and data developed by Mr. Kremer for consideration as the water sampling procedures are developed for Method 431.

4. Comment regarding aeration/Detoxification-B emissions: Mr. Kremer stated that aeration/detox-B emissions are uncontrolled and are discharged directly to the dedicated ventilation system. Therefore, no inlet vs. outlet comparison can be made. In addition, it is not specified which Appendix A measurement methods are to be used, and at which points in the discharge stream the sampling is to be conducted.

Agency response: In order to conform to the Statewide ATCM for ethylene oxide sterilizers, the Joslyn recovery system described by the commenter would have to be modified by the addition of aeration control equipment for those facilities permitted for ethylene oxide use greater than 600 pounds per year. Again, staff agrees that specific guidelines should be included during the next revision of the method. No change was made to the method at this time.