

California Environmental Protection Agency



Air Resources Board

STAFF REPORT

Initial Statement of Reasons for a Proposed Public Hearing to Consider the Adoption of a New Test Method for the Determination of Volatile Organic Compounds (VOC) in Consumer Products

October 4, 1996

Monitoring and Laboratory Division



Cal/EPA

California
Environmental
Protection
Agency



Air Resources Board

P.O. Box 2815
2020 L Street
Sacramento, CA
95812-2815



Pete Wilson
Governor

James M. Strock
Secretary for
Environmental
Protection

October 4, 1996

Dear Sir or Madam:

Over the last year the Air Resources Board (ARB or Board) has worked with the public and affected industries (stakeholders) to develop Method 310, Determination of Volatile Organic Compounds (VOC) from Consumer Products. The existing test methods do not provide a guide for integrating the various test procedures referenced in the anti-perspirants and deodorants, consumer products, and aerosol coating products regulations. Method 310 corrects this problem as well as other limitations.

We held four workshops to allow stakeholders the opportunity to provide comments to clarify and improve Method 310. As a result of those comments we have made a number of changes, such as stating the overall precision and accuracy of Method 310. Based on comments provided at the last workshop held on August 22, 1996, we have made two major changes to Method 310. These changes include the removal of the ASTM D2879-92 (isotenoscope procedure for determining vapor pressures) and the addition of a detailed explanation on the application of the method, and the steps taken if an initial determination by Method 310 finds a product does not comply with the established VOC standard. The removal of ASTM D2879-92 will allow for the completion of an inter-laboratory study to determine the variability of the method and review of the "degassing step" developed by the ARB staff to minimize the loss of VOC. In response to requests by stakeholders, Method 310 now includes a process that would be used by the Executive Officer for determining whether a product selected complies with the standard. If the initial determination by Method 310 shows that a product is non-compliant, the ARB Executive Officer will request the manufacturer to submit formulation data. The Executive Officer will take no enforcement action if testing verifies the formulation data and the product complies with VOC standards after adjusting the initial determination based on the formulation data. These changes are reflected in the October 4, 1996 version of Method 310 which is found in Appendix B of the enclosed Staff Report.

We are planning to have the Board consider adoption of the October 4, 1996 version of Method 310 at the November 21-22, 1996 meeting. To provide an opportunity for additional public review and comments on the October 4, 1996 version of Method 310 before the Board meeting, we will present a summary of the method and respond to questions at the next meeting of the Consumer Products Working Group (CPWG) scheduled for October 29 and 30, 1996. The time and location of the next CPWG meeting will be listed in the agenda that will be sent to you shortly. We realize that a full discussion of Method 310 may not be possible during the CPWG meeting. If there is interest to discuss Method 310 further, we will hold a workshop on October 31, 1996 following the conclusion of the CPWG meeting. The time and location will be announced at the CPWG meeting.

Sir or Madam

-2-

October 4, 1996

We appreciate the input provided by stakeholders during the development of Method 310. If you have questions or have further comments, please contact me at (916) 445-3742 or contact George Lew or Michael Spears at (916) 263-1630 and (916) 263-1627, respectively.

Sincerely,

A handwritten signature in black ink, appearing to read "William V. Loscutt". The signature is written in a cursive style with a large, prominent initial "W".

William V. Loscutt, Chief
Monitoring and Laboratory Division

Enclosure

TITLE 17. CALIFORNIA AIR RESOURCES BOARD

NOTICE OF PUBLIC HEARING TO CONSIDER THE ADOPTION OF A NEW TEST METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOC) IN CONSUMER PRODUCTS;

AND

THE ADOPTION OF AMENDMENTS TO THE TEST METHOD SECTIONS OF THE CALIFORNIA REGULATIONS FOR REDUCING VOC EMISSIONS FROM ANTIPERSPIRANTS AND DEODORANTS, CONSUMER PRODUCTS, AND AEROSOL COATING PRODUCTS

The Air Resources Board (the "Board" or "ARB") will conduct a public hearing at the time and place noted below to consider the adoption of a regulation establishing a new test method for the Determination of Volatile Organic Compounds (VOC) in Consumer Products. At the hearing the Board will also consider the adoption of amendments to the test method sections of the Regulation for Reducing VOC Emissions from Antiperspirants and Deodorants, the Regulation for Reducing VOC Emissions from Consumer Products, and the Regulation for Reducing VOC Emissions from Aerosol Coating Products.

Date: November 21, 1996

Time: 9:30 a.m.

Place: Air Resources Board
Hearing Room, Lower Level
2020 L Street
Sacramento, California

This item will be considered at a two-day meeting of the ARB, which will commence at 9:30 a.m., November 21, 1996, and may continue at 8:30 a.m., November 22, 1996. This item may not be considered until November 22, 1996. Please consult the agenda for the meeting, which will be available at least 10 days before November 22, 1996, to determine the day on which this item will be considered.

INFORMATIVE DIGEST OF PROPOSED ACTION AND PLAIN ENGLISH POLICY STATEMENT OVERVIEW

Sections Affected: Amendments are being proposed to sections 94506(a), 94515(a), and 94526, Title 17, California Code of Regulations (CCR), in order to incorporate by reference the new test method for the Determination of VOC in Consumer Products.

Background

Section 41712 of the California Health and Safety Code requires the ARB to adopt regulations to achieve the maximum feasible reduction in reactive organic compounds (ROCs) emitted by consumer products ("ROC" is equivalent to "VOC"). To date, the Board has adopted three regulations which establish VOC limits for various categories of consumer products. These regulations are the Regulation for Reducing VOC Emissions from Antiperspirants and Deodorants (the "antiperspirant and deodorant regulation"; sections 94500-94506.5, Title 17, CCR), the Regulation for Reducing VOC Emissions from Consumer Products (the "consumer products regulation"; sections 94507-94517, Title 17, CCR), and the Regulation for Reducing VOC Emissions from Aerosol Coating Products (the "aerosol coatings regulation"; sections 94520-94528, Title 17, CCR).

Each of these regulations contains a section specifying the applicable test methods. The test methods sections of the regulations are section 94506 (antiperspirants and deodorants), section 94515 (consumer products), and section 94526 (the aerosol coating products), Title 17, CCR. These sections incorporate by reference a number of different test methods. Some of the incorporated test methods are used to determine compliance with the applicable VOC limits, and some of the test methods are used for other compliance determinations.

Description of the Proposed Regulatory Action

The ARB staff is proposing the adoption of a regulation establishing a new Test Method 310 for the Determination of Volatile Organic Compounds (VOC) in Consumer Products (Method 310). Method 310 would be used to determine compliance with the applicable VOC limits, and would be incorporated by reference in the test methods sections of the ARB regulations for antiperspirants and deodorants, consumer products, and aerosol coating products.

Method 310 specifies that a number of tests be performed on consumer product samples. The initial testing procedure is different for non-aerosol and aerosol products. For aerosol products, the propellant is first vented and a small portion is collected in a tedlar bag to quantify the amount of exempt compounds. The amount of propellant remaining after subtracting the exempt compounds is considered to be a VOC. The VOC content of the remaining liquid portion of the aerosol product is then tested in the same manner as non-aerosol products are tested. These tests include the determination of total volatiles, ammonia, water, and exempt compounds.

Method 310 also specifies an equation for calculating the percent VOC content of both aerosol and non-aerosol products. Using the results of the various tests performed on the product, the equation essentially takes the total volatile content of a product and arrives at the percent VOC by subtracting the water, acetone, and other substances that are exempt under the regulations. If the initial determination of VOC content indicates that a product does not comply with the applicable VOC standards, Method 310 provides that the ARB Executive Officer will request the product manufacturer to supply formulation data. Further testing may then be performed as necessary to

verify the supplied formulation data. If there exists a discrepancy that cannot be resolved between the results of Method 310 and the supplied formulation data, then the results of Method 310 take precedence and may be used to establish a violation of ARB regulations.

Method 310 incorporates by reference some of the same testing procedures referenced in the current ARB regulations, but it also includes eight additional testing procedures. These additional procedures expand the applicability and flexibility of Method 310 and correct the limitations in the currently specified test methods. The test procedures incorporated in Method 310 are consistent with U.S. Environmental Protection Agency (U.S.EPA), American Society for Testing and Materials (ASTM) and National Institute for Occupational Safety and Health (NIOSH) test methods.

Comparable Federal Regulations

The U.S. EPA has published a proposed rule, *National Volatile Organic Compound Emission Standards for Consumer Products*, which appeared in the April 2, 1996 Federal Register (Vol. 61, No. 64, pages 14531-14543). The proposed rule specifies VOC standards for antiperspirants and deodorants and other consumer products (but not for aerosol coatings), and is similar to the ARB consumer products regulation. However, the U.S. EPA's proposed rule does not include a test method that is comparable to ARB Method 310. Instead, the U.S. EPA's proposed rule relies predominately on formulation information to demonstrate compliance.

In addition, Method 310 incorporates by reference a number of U.S. EPA, NIOSH, and ASTM test methods. Many of these same test methods are also incorporated by reference in the Code of Federal Regulations. However, the test methods incorporated in the Code of Federal Regulations are not used for the same purpose as these test methods are used in Method 310 (i.e., to determine compliance with VOC regulatory standards for consumer products).

AVAILABILITY OF DOCUMENTS AND CONTACT PERSON

The ARB staff has prepared an Initial Statement of Reasons (ISOR) for the proposed regulatory action which includes a summary of the environmental and economic impacts of the proposal and technical support documentation. Copies of the ISOR may be obtained from the ARB's Public Information Office, 2020 L Street, Sacramento, California 95814, (916) 322-2990, at least 45 days prior to the scheduled hearing. The ISOR contains the full text of the proposed action. The staff has also compiled a record which includes all information upon which the proposal is based. This material is available for inspection upon request to the contact person identified immediately below. The ARB has determined that it is not feasible to draft the regulation in plain English due to the technical nature of the regulation; however, a plain English summary of the regulation is available from the agency contact person named in this notice, and is also contained in the ISOR for this regulatory action.

Further inquiries regarding this matter should be directed to George Lew, Monitoring and Laboratory Division, Engineering and Laboratory Branch, P.O. Box 2815, Sacramento, California 95412, (916)263-1630.

COSTS TO PUBLIC AGENCIES AND TO BUSINESSES AND PERSONS AFFECTED

The determinations of the Board's Executive Officer concerning the costs or savings necessarily incurred in reasonable compliance with the proposed regulatory action are presented below.

The Executive Officer has determined that the proposed regulatory action will not create costs or savings, as defined in Government code section 11346.5(a)(6), to any state agency or in federal funding to the State, costs or mandate to any local agency or school district whether or not reimbursable by the State pursuant to Part 7 (commencing with section 17500), Division 4, Title 2 of the Government Code, or other nondiscretionary savings to local agencies.

The Executive Officer has determined that there will be no, or an insignificant, potential cost impact on private persons or businesses directly affected resulting from the proposed action.

The Executive Officer has also determined that the proposed regulatory action will not have a significant adverse economic impact on businesses, including the ability of California businesses to compete with businesses in other states, or on directly-affected private persons. In accordance with Government Code section 11346.3, the Executive Officer has determined that the proposed regulatory action will not affect the creation or elimination of jobs within the State of California, the creation of new businesses and the elimination of existing businesses in California, or the expansion of businesses currently doing business within California. A assessment of the economic impacts of the proposed amendments can be found in the ISOR.

The Board's Executive Officer has also determined, pursuant to Government Code section 11346.5(a)(3)(B), that the regulation will affect small business.

Before taking final action on the proposed regulatory action, the ARB must determine that no alternative considered by the agency would be more effective in carrying out the purpose for which the action is proposed or would be as effective and less burdensome to affected private persons or businesses than the proposed action.

SUBMITTAL OF COMMENTS

The public may present comments relating to this matter orally or in writing. To be considered by the ARB, written submissions must be addressed to and received by the Clerk of the Board, Air Resources Board, P.O. Box, 2815, Sacramento, CA 95812, or 2020 L Street, 5th Floor, Sacramento, CA 95814, no later than 12:00 noon November 20, 1996, or received by the Clerk of the Board at the hearing.

The ARB requests, but does not require, that 20 copies of any written statement be submitted and that all written statements be filed at least 10 days prior to the hearing. The ARB encourages

members of the public to bring any suggestions for modification of the proposed regulatory action to the attention of staff in advance of the hearing.

STATUTORY AUTHORITY AND HEARING PROCEDURES

This regulatory action is proposed under the authority granted to the ARB in sections 39600, 39601, 39607, 41511, and 41712 of the Health and Safety Code. This action is proposed to implement, interpret, or make specific sections 39002, 39600, 39607, 40000, 41511, and 41712 of the Health and Safety Code.

The public hearing will be conducted in accordance with the California Administrative Procedure Act, Title 2, Division 3, Part 1, Chapter 3.5 (commencing with section 11340) of the Government Code. Following the public hearing, the Board may adopt the regulatory language as originally proposed or with nonsubstantial or grammatical modifications. The ARB may also adopt the proposed regulatory language with other modifications if the modifications are sufficiently related to the originally proposed text that the public was adequately placed on notice that the regulatory language as modified could result from the proposed regulatory action. In the event that such modifications are made, the full regulatory text, with the modifications clearly indicated, will be made available to the public for written comment at least 15 days before it is adopted.

The public may request a copy of the modified regulatory text from the ARB's Public Information Office, 2020 L Street, Sacramento, California 95814, (916) 322-2990.

CALIFORNIA AIR RESOURCES BOARD



Michael P. Kenny
Executive Officer

Date: September 24, 1996

California Environmental Protection Agency



Air Resources Board

**STAFF REPORT: INITIAL STATEMENT OF REASONS
FOR PROPOSED RULEMAKING**

**NOTICE OF PUBLIC HEARING TO CONSIDER THE ADOPTION OF A NEW TEST
METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS
(VOC) IN CONSUMER PRODUCTS,**

AND

**THE ADOPTION OF AMENDMENTS TO THE TEST METHOD SECTIONS OF THE
CALIFORNIA REGULATIONS FOR REDUCING VOC EMISSIONS FROM
ANTIPERSPIRANTS AND DEODORANTS, CONSUMER PRODUCTS, AND
AEROSOL COATING PRODUCTS**

October 4, 1996

To be considered by the Air Resources Board on November 21-22, 1996.

**Air Resources Board
Board Hearing Room, Lower Level
2020 L Street
Sacramento, California**

**Air Resources Board
P.O. Box 2815
Sacramento, CA 95812**

This report has been prepared by the staff of the California Air Resources Board. Publication does not signify that the contents reflect the views and policies of the Air Resources Board, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

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TABLE OF CONTENTS

	Page
I. INTRODUCTION	1
A. BACKGROUND	1
Legal Authority	2
Comparable Federal Regulations	2
B. PUBLIC PROCESS	3
C. RECOMMENDATIONS	3
II. PROPOSED AMENDMENTS TO THE CONSUMER PRODUCTS REGULATIONS	4
A. INTRODUCTION	4
B. EXISTING TEST METHODS	4
C. PROPOSED NEW TEST METHOD 310	5
Alternate Test Methods	5
Description of Proposed Test Method 310	6
D. AMENDMENTS TO THE EXISTING TEST METHODS	8
III. AIR QUALITY, ENVIRONMENTAL, AND ECONOMIC IMPACTS	9
A. INTRODUCTION	9
B. AIR QUALITY, ENVIRONMENTAL, AND ECONOMIC IMPACTS	9
Air Quality and Environmental Impacts	9
Economic Impacts	9
C. ALTERNATIVES CONSIDERED	10
REFERENCES	11
APPENDIX A: Proposed Amendments to the Test Methods Sections of "The California Consumer Products Regulations"	12
APPENDIX B: Proposed ARB Method 310: The Determination of Volatile Organic Compounds (VOC) in Consumer Products	16

I. INTRODUCTION

The Air Resources Board (ARB or Board) staff proposes to amend the test method sections of the California Consumer Products Regulations (sections 94500-94506.5, 94507-94517, and 94520-94528, Title 17 of the California Code of Regulations (CCR)). In addition, this report proposes that the Board adopt Test Method 310, "The Determination of Volatile Organic Compounds (VOC) in Consumer Products," which will be used to determine the VOC content of consumer products.

Method 310 includes established analytical test methods based on procedures by the United States Environmental Protection Agency (U.S. EPA), American Society for Testing and Materials (ASTM), and the National Institute of Occupational Safety and Health (NIOSH). In conjunction with current test methods, eight additional procedures are proposed to be included into Test Method 310 for the purpose of determining VOC content. Method 310 is applicable to a wide variety of consumer products, providing both a calculation procedure for determining VOC content and specifying precision and accuracy for the overall method.

A. Background

Section 41712 of the California Health and Safety Code requires the ARB to adopt regulations to achieve the maximum feasible reduction in reactive organic compounds (ROCs) emitted by consumer products ("ROC" is equivalent to "VOC"). To date, the Board has adopted three regulations which establish VOC limits for various categories of consumer products. These regulations are the Regulation for Reducing VOC Emissions from Antiperspirants and Deodorants (the "antiperspirant and deodorant regulation"; sections 94500-94506.5, Title 17, CCR), the Regulation for Reducing VOC Emissions from Consumer Products (the "consumer products regulation"; sections 94507-94517, Title 17, CCR), and the Regulation for Reducing VOC Emissions from Aerosol Coating Products (the "aerosol coatings regulation"; sections 94520-94528, Title 17, CCR).

Each of these regulations contains a section specifying the applicable test methods. The test methods sections of the regulations are section 94506 (antiperspirants and deodorants), section 94515 (consumer products), and section 94526 (the aerosol coating products), Title 17, CCR. These sections incorporate a number of different test methods by reference. Some of the incorporated test methods are used to determine compliance with the applicable VOC limits, and some of the test methods are used to determine other types of compliance.

The current test methods used for determining the VOC content include the following:

for the antiperspirant and deodorant and consumer products regulations: U.S. EPA Method 24-24A, U.S. EPA Method 18, NIOSH Method 1400, U.S. EPA Method 8240;

for the aerosol coatings regulation: ASTM D-5325-92 and the Bay Area Air Quality Management District (BAAQMD) Method 35.

Legal Authority

This regulatory action is proposed under the authority granted to the ARB in sections 39600, 39601, 39607, 41511, and 41712 of the Health and Safety Code. Sections 39600 and 39601 authorize the ARB to adopt regulations and do such acts as may be necessary for the proper execution of the powers and duties granted to and imposed upon the ARB. Section 39607(d) authorizes the ARB to adopt test procedures to measure compliance with its nonvehicular emission standards. Section 41511 authorizes the ARB to adopt regulations requiring air pollution emission sources to take such action as the ARB determines to be reasonable to determine emissions from such sources. Finally, section 41712 authorizes the ARB to adopt regulations to achieve the maximum feasible reduction in VOCs emitted by consumer products.

Pursuant to these sections, the ARB has previously adopted a number of consumer products test methods. Test methods are an integral part of the ARB consumer products regulations and are necessary to determine and verify compliance with the regulatory standards.

Comparable Federal Regulations

The U.S. EPA has published a proposed rule, *National Volatile Organic Compound Emission Standards for Consumer Products*, which appeared in the Federal Register on April 2, 1996 (Vol. 61, No. 64, pages 14531-14543). The proposed rule specifies VOC standards for antiperspirants and deodorants and other consumer products, but not for aerosol coatings, and is similar to the ARB consumer products regulation. However, the U.S. EPA's proposed rule does not include a test method that compares with the ARB Method 310. Instead, the U.S. EPA's proposed rule relies predominately on formulation information to demonstrate compliance with the regulation.

Whenever possible, the ARB avoids unnecessary duplication and conflict with federal regulations addressing the same issues. We, nevertheless, believe that it is appropriate to include a test method in ARB consumer products regulations because the U.S. EPA proposed rule does not specify a comparable test method. Method 310 will greatly assist the ARB's enforcement determinations and will allow accurate information

to be generated when a manufacturer's formulation data are unavailable, incomplete, or inaccurate.

Method 310 also incorporates a number of U.S. EPA, NIOSH, and ASTM test methods by reference, many of which are also incorporated by reference in the Code of Federal Regulations. However, the test methods incorporated in the Code of Federal Regulations are not used for the same purpose as these test methods are used in Method 310 (i.e., to determine compliance with VOC regulatory standards for consumer products).

B. Public Process

In developing Method 310, we conducted four workshops on the proposed Method 310. To maximize public participation, the workshops were scheduled and coordinated with the meeting of the ARB Consumer Products Working Group. Workshops were held on October 25, 1995; and on January 19, June 19, and August 22, 1996. In addition, we had a number of discussions, teleconferences, and responded to written comments.

The ARB staff has discussed Method 310 in detail, specifying the procedures for determining the VOC content of a product and the manner in which data will be reported. ARB Method 310 has received a thorough review by the consumer product manufacturers, with minimal controversy. However, the consumer products industry expressed concern over how the data would be used to enforce the regulations; to address this concern, we included a description the enforcement process in the regulations. To further scientific consensus on the proposed test method, in July 1996, we initiated a round-robin study of Method 310 to obtain interlaboratory precision and accuracy data. We plan to have the results of the interlaboratory analysis available in October 1996.

C. Recommendations

We recommend that the Board adopt the proposed amendments to the CCR as described in Appendix A and adopt proposed Test Method 310 in Appendix B.

II. PROPOSED NEW TEST METHOD AND AMENDMENTS TO THE TEST METHOD SECTIONS OF THE CONSUMER PRODUCTS REGULATIONS

A. Introduction

In this Chapter, we provide a plain English discussion of the proposed Method 310 and the amendments to the test methods sections of the antiperspirant and deodorant, consumer products, and aerosol coatings regulations. This discussion is intended to satisfy the requirements of Government Code 11346.2(a)(1), which requires that a noncontrolling "plain English" summary of the regulation be made available to the public.

B. Existing Test Methods

The ARB currently uses several established test methods to determine the VOC, acetone, and halogenated hydrocarbons content in consumer products in California. The existing test methods include ASTM, U.S. EPA, NIOSH, and BAAQMD procedures, which are referenced in Sections 94506, 94515, and 94526, of Title 17, CCR.

However, the current test methods have several limitations. First, they do not specify a guide to integrate the various procedures. Without a specific guide, the regulations may be more difficult to enforce because the user must interpret how to implement the procedures which could lead to results that are inconsistent and possibly inaccurate. Second, the current methods do not include a procedure for determining ammonia. For ammonia containing products, the current regulation will overestimate the VOC content if the test to determine ammonia content is not included. Third, the existing methods do not include a procedure to collect the propellant from aerosol products. In the existing test methods, the ASTM methods are essentially quality control procedures for determining the pressure of a container and cannot be applied to collecting a propellant for analysis.

Finally, the existing methods do not contain procedures to document the overall precision and accuracy of the various procedures when applied to consumer products. These procedures are necessary to determine the variability of the method. The variability of the method is important, because it is ARB policy to take enforcement action based on test results only if the test results are outside the variability range of the test method. For example, suppose the VOC standard for a particular product is 60 percent, the measured VOC content is 62 percent, and the variability of the test method is ± 2 percent. In this situation, the ARB would not use the test results to take enforcement action for violation of the 60 percent VOC standard. If instead the measured VOC content is 63 percent, however, the ARB would then take appropriate enforcement action.

C. Proposed New Test Method 310

We proposed that the Board adopt Test Method 310 for the antiperspirant regulation, consumer product regulation, and aerosol coating regulation (Appendix B contains the text of the regulation). While Method 310 contains the same procedures referenced in the current regulations, it includes the following eight additional procedures:

- 1) ASTM D1426 for determining ammonia by selective ion electrode;
- 2) EPA Method 300.7 for determining the ammonia content by ion chromatography;
- 3) ASTM D2887 for quantifying the percent of volatile compounds with greater than 12 carbon atoms by gas chromatography with a flame ionization detector (compounds having greater than 12 atoms are defined as low vapor pressure compounds and are exempted by the regulations);
- 4) ASTM D859 for the analyses of polymethylsiloxanes by colorimetry (this is a category of exempt VOCs);
- 5) ASTM E1131-86 for determining the composition of volatile compounds by varying the temperature using a thermogravimetric device to produce a boiling point profile of the sample;
- 6) ASTM D3074-72 with modifications that allow for the collection of a propellant sample for analysis from metal aerosol metal containers (contained in Appendix A);
- 7) ASTM D3063-79 with modifications that allow for the collection of a propellant sample from a glass aerosol bottle for analysis (contained in Appendix A); and
- 8) ASTM D3064-89 defines aerosol product standard terminology to use in modified ASTM D3074-72 and ASTM D3063-79.

The additional procedures expand Method 310's capability to determine the volatile content of a consumer product, increasing the applicability and flexibility in determining the VOC content from the various consumer product categories. Test Method 310 corrects the limitations in the existing method by defining the steps to determine the percent VOC content, specifying a procedure for determining the ammonium content, establishing a procedure for collecting aerosol propellants, and defining the overall accuracy and precision of the method^a. These overall changes will improve the quality of the data, ensuring confidence in the results from Method 310. The proposed regulation removes BAAQMD Method 35 from the Aerosol Coatings Regulations as Method 310 encompasses the procedures referenced therein.

Alternate Test Methods

To provide additional flexibility to the consumer product industry, Method 310 allows the ARB Executive Officer to approve alternate test methods as long as the alternate methods are found to accurately determine the concentration of VOC or constituents in

consumer product.

Description of Proposed Test Method 310

Figure 1 describes how Method 310 will be implemented (Table 1 references the appropriate ASTM, U. S. EPA, or NIOSH procedures for these determinations). The ARB Executive Officer¹ collects consumer product samples throughout the state for analysis. Throughout the sample collection and analytical process the Executive Officer shall maintain the proper chain of custody. If the samples are not aerosol products, the analyses consist of a minimum of four tests to determine the VOC content. These tests include the determination of total volatiles, ammonia, water, and exempt compounds (including acetone/alcohol). Non-aerosol products may be solid, semi-solid, or liquid. For aerosol products, the propellant is vented, and a small portion is collected in a tedlar bag for analysis to quantify the amount of exempt compounds. The amount of propellant remaining after subtracting the amount of exempt compounds is considered to be VOC. The VOC content of the liquid portion of an aerosol product is then determined in same manner as a non-aerosol product.

The proposed test method is based on calculating the total volatile content of a product and then subtracting the water, acetone, and other substances which are exempt under the regulations. For the liquid portion of an aerosol and all non-aerosol products, an aliquot of the sample is weighed and then heated for an hour at 110 degrees Celsius. Through heating the volatile content is driven off. The difference in weight between the sample before and after heating represents the total volatile material, which includes ammonia, water and exempt compounds. The ammonia, water, and exempt compounds are determined using the methods referenced in Table 1. As noted above, these substances are subtracted from the total volatile material, and the remainder is the VOC content of the sample.

Once the tests are completed, the Executive Officer will use the appropriate formula as specified in Method 310, section 4.0, to make an initial determination of whether the product meets the applicable VOC standards specified in ARB regulations. If initial results show that the product does not meet the applicable VOC standards, the Executive Officer may perform additional testing to confirm the initial results.

If the retest confirms that the product does not meet the applicable VOC standards, the Executive Officer will request the product manufacturer or responsible party to supply product formulation data. Information submitted to the ARB Executive Officer may be claimed as confidential: such information will be handled in accordance with the confidentiality procedures specified in Title 17, CCR, sections 91000 to 91022.

¹. The term ARB Executive Officer means the Executive Officer of the Air Resources Board or his or her authorized representative.

If the information supplied by the manufacturer or responsible party shows that the product does not meet the applicable VOC standards, then the Executive Officer will take appropriate enforcement action. If there exists a discrepancy that cannot be resolved between the results of Method 310 and the supplied formulation data or if the formulation data is not supplied, then the results of Method 310 shall take precedence. The results of Method 310 shall then determine if the product is in compliance with the applicable VOC standards, and may be used to establish a violation of ARB regulations.

Figure 1: Flow Chart for Analysis via Method 310

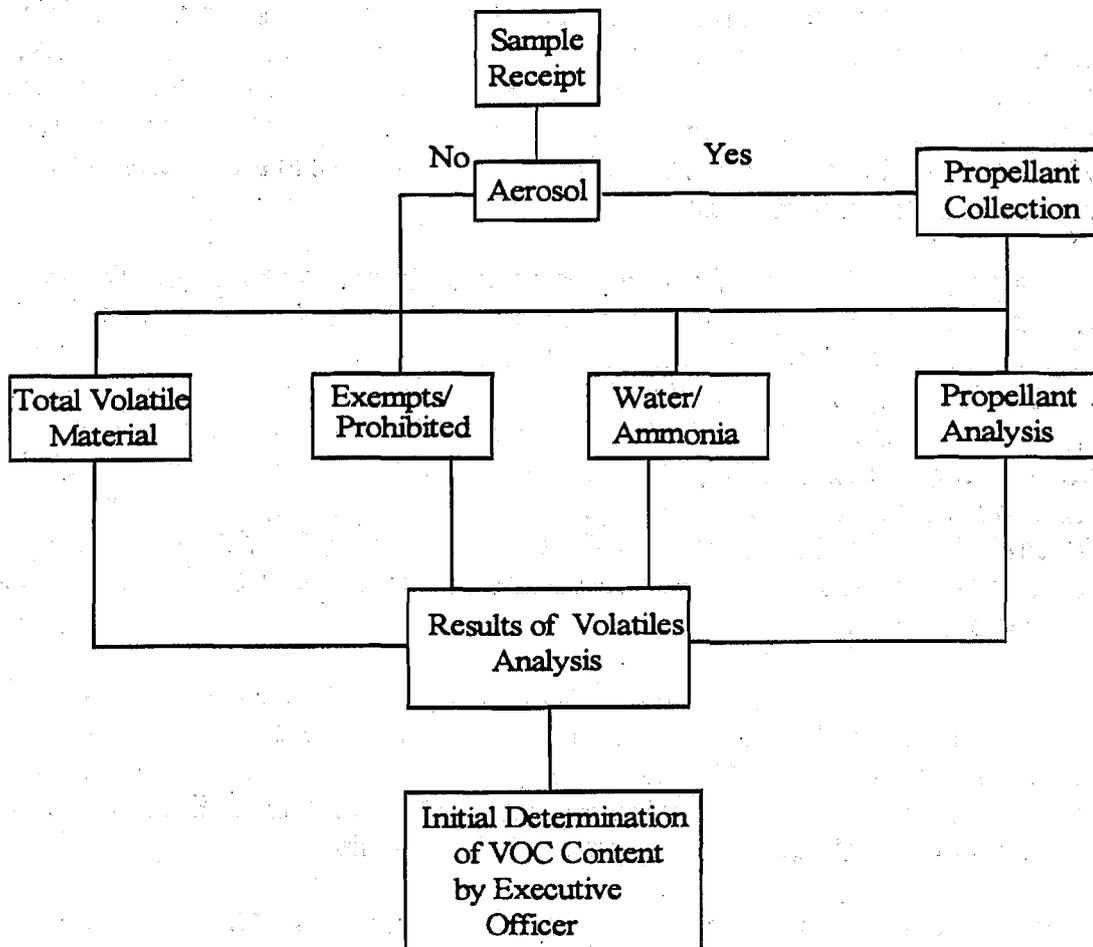


Table 1: Method 310 Analyses	
Analysis to be Performed	Reference Method
Total Volatile Material (gravimetric)	U.S. EPA Method 24/24A; ASTM 2369-87; ASTM E1131-86**
Water Content	ASTM D4017-88; ASTM D3792-86
Ammonium Content	ASTM D1426-93**; U.S. EPA Method 300.7**
Acetone/Alcohols	NIOSH 1400
Aerosol Propellants	U.S. EPA Method 18; ASTM D3074-72 (as modified-metal aerosol container)**; ASTM D3063-79 (as modified-glass aerosol container)**
Exempts/Prohibited	U.S. EPA Method 8240; U.S. EPA Method 18; ASTM D2887-93**; ASTM D859-88**, ASTM E1131-86**
** Methods not currently in the regulations.	

D. Amendments to the Existing Test Methods

As noted above, we are proposing that the Board amend the test method sections of the antiperspirant regulation, the consumer products regulation, and the aerosol coatings regulation. The amendments incorporate the proposed test method 310, replacing the sections of the current test methods for determining VOC content with test method 310.

III. AIR QUALITY, ENVIRONMENTAL, AND ECONOMIC IMPACTS

A. Introduction

In this Chapter, we discuss the air quality, environmental, and economic concerns, as well as the alternatives to the proposed test method and amendments.

B. Air Quality, Environmental, and Economic Impacts

Air Quality and Environmental Impacts

Both the California Environmental Quality Act (CEQA) and Board policy require the ARB to consider the potential environmental impacts of proposed regulations. The proposed Method 310 is fundamentally a mechanism for determining compliance with the VOC regulatory standards. The adoption of Method 310 will not result in any air quality impacts because the VOC standards for consumer products will remain the same. Also, the Executive Officer has not identified any adverse non-air quality environmental impacts that would result from the proposed regulatory action. The Executive Officer has therefore concluded that proposed regulatory action will not have any significant adverse impacts on the environment.

Economic Impacts

In developing this regulatory proposal, the Executive Officer evaluated the potential economic impacts on private persons and businesses. State law (Government Code sections 11346.3 and 11346.5) requires such an economic analysis. Government Code section 11346.3(a) requires that in proposing to adopt or amend an administrative regulation, state agencies must assess the potential for adverse economic impact on California business enterprises and individuals. The assessment must include the impact of the proposal on the ability of California businesses to compete with businesses in other states. In addition, Government Code section 11346(b) requires state agencies to assess the potential impact of proposed regulations on the creation or elimination of jobs in California, the creation of new businesses and the elimination of existing businesses in California, or the expansion of businesses currently doing business within California. Government Code section 11346.5(a)(9) also requires that the potential cost impact be considered on private persons or businesses directly affected by the proposed regulation.

The effect of the proposed regulatory action will be to replace some of the existing consumer products test methods with the new Method 310. Method 310 will primarily be used by the Executive Officer to test samples of consumer products to determine if they comply with the applicable VOC standards. Manufacturers are already required to comply with these standards, which will not be changed by the proposed action. The proposed action will impose no additional requirements on any person to do any act or to refrain from

doing any act. No costs will be imposed on the regulated community.

The Executive Officer has therefore determined that the proposed regulatory action will not have a significant adverse economic impact on the ability of California businesses to compete with businesses in other states or on directly-affected private persons. In accordance with Government Code section 11346.3, the Executive Officer has determined that the proposed regulatory action will not affect the creation or elimination of jobs within the State of California, the creation of new businesses and the elimination of existing business in California, or the expansion of businesses currently doing business within California.

The Board's Executive Officer has also determined, pursuant to Government Code section 11346.5(a)(3)(B), that the regulation may affect small business. This determination was made because California regulations provide that a proposed regulation "affects small business" if a small business "derives a benefit ... or incurs a detriment from the enforcement of the regulation" (see Title 1, California Code of Regulations, section 4(a)). This definition is so broadly worded that it is possible to conceive of some scenario in which a small business might "derive a benefit or incur a detriment" from the proposed regulatory action.

C. Alternatives Considered

In developing test method 310 and the proposed amendments to the consumer products regulations, the staff considered the following three alternatives:

1. Retain the current regulations: This alternative provides the ability to analyze consumer products, but it is limited in scope as there is no common test method to integrate the various procedures to determine the VOC standards as defined in the regulations. In addition, as noted in Chapter II, this alternative has several additional limitations.
2. Eliminate the existing consumer products test procedures: This alternative would make the consumer products regulations difficult if not impossible to enforce. As a result, the anticipated emission reductions required in the California State Implementation Plan may not be achieved. Any shortfall will have to be made up by further emission reductions from stationary and mobile sources.
3. Adopt the proposed Method 310 and the proposed amendments: Method 310, along with the amendments, integrates the reference methods by including a calculation procedure to determine VOC for aerosol and non-aerosol consumer product, revises existing methods, adds additional test methods to expand applicability, elaborates on precision and accuracy of the

method, and allows for maximum ability to measure the components of consumer products. Overall, the proposed method 310 and amendments provide an enforcement mechanism and flexibility in the choice of procedure for analysis, without reducing the quality of the test results.

REFERENCES

- a. Memorandum from William V. Loscutoff, Chief, Monitoring and Laboratory Division to James Morgester, Chief, Compliance Division: Precision and Accuracy of Consumer Product Test Method 310, January 3, 1996.

APPENDIX: A

**Proposed Amendments to the Test Methods Sections of the
Consumer Products Regulations**

Proposed Amendments to the Test Methods Sections of the Antiperspirant and Deodorant Regulation, the Consumer Products Regulation, and the Aerosol Coatings Regulation

[Note: The proposed amendments are shown below in underline to indicate additions and ~~strikeout~~ to show deletions.]

Amend Section 94506(a), Title 17, California Code of Regulations, to read as follows:

94506. Test Methods

- (a) Testing to determine the volatile organic compound content of an antiperspirant or deodorant, or to determine compliance with the requirements of this article, shall be performed using ~~one or more of the following analytical methods which are incorporated by reference herein: (1) Method 24-24A, Part 60, Title 40, Code of Federal Regulations, Appendix A, July 1, 1988; (2) Method 18, Federal Register 48, no. 202, October 18, 1983; (3) Method 1400, (NIOSH Manual of Analytical Methods, Volume 1, February 1984; or (4) Environmental Protection Agency Method 8240, "GC/MS Method for Volatile Organics," September 1986~~ Air Resources Board Method 310, Determination of Volatile Organic Compounds (VOC) in Consumer Products, adopted [date of adoption], which is incorporated herein by reference. Alternative methods which are shown to accurately determine the concentration of VOCs in a subject product or its emissions may be used upon approval of the Executive Officer.

Amend Section 94515(a), Title 17, California Code of Regulations, to read as follows:

94515. Test Methods

- (a) Testing to determine compliance with the requirements of this article, shall be performed using ~~one or more of the following analytical methods which are incorporated by reference herein: (1) Method 24-24A, Part 60, Title 40, Code of Federal Regulations, Appendix A, July 1, 1988; (2) Method 18, Federal Register 48, no. 202, October 18, 1983; (3) Method 1400, (NIOSH Manual of Analytical Methods, Volume 1, February 1984; or (4) Environmental Protection Agency Method 8240, "GC/MS Method for Volatile Organics," September 1986~~ Air Resources Board Method 310, Determination of Volatile Organic Compounds (VOC) in Consumer Products, adopted [date of adoption], which is incorporated herein by reference. Alternative methods which are shown to accurately determine the concentration of VOCs in a subject product or its emissions may be used upon approval of the Executive Officer.

Amend Section 94526, Title 17, California Code of Regulations, to read as follows:

94526. Test Methods

Compliance with the requirements of this article shall be determined by using the following test methods, which are incorporated by reference herein. Alternative test methods which are shown to accurately determine the VOC content, exempt compound content, metal content, specular gloss, or acid content may also be used after approval in writing by the Executive Officer:

- (a) VOC Content. The VOC content of solvent-based all aerosol coating products subject to the provisions of this article shall be determined by the procedures set forth in ~~Bay Area Air Quality Management District Manual of Procedures, Volume III, Laboratory Procedures, Method 35, "Determination of Volatile Organic Compounds (VOC) in Solvent Based Aerosol Paints", as amended January 19, 1994. For water-containing aerosol coating products subject to the provisions of this article, the VOC content shall be determined by ASTM D-5325-92, "Standard Test Method for Determination of Weight Percent Volatile Content of Water-Borne Aerosol Paints", November 15, 1992. "Air Resources Board Method 310, Determination of Volatile Organic Compounds (VOC) in Consumer Products." adopted [date of adoption].~~
- (b) Exempt Compounds. Compounds exempt from the definition of VOC shall be analyzed according to the test methods listed below:
 - (1) ~~the content of 1,1-difluoroethane exempt compound content of all aerosol coating products shall be determined by BAAQMD Method 35, "Determination of Volatile Organic Compounds (VOC) in Solvent Based Aerosol Paints", as amended January 19, 1994. Other exempt compounds which are used as propellants in aerosol coatings may also be quantified using this method with prior written approval of the Executive Officer. "Air Resources Board Method 310, Determination of Volatile Organic Compounds (VOC) in Consumer Products." adopted [date of adoption].~~
 - (2) ~~the content of dichloromethane (methylene chloride) and 1,1,1-trichloroethane shall be determined by ARB Method 432, California Code of Regulations, Title 17, Section 94144, after removal of the propellant following the procedure in ASTM Method D-5325-92, "Standard Test Method for Determination of Weight Percent Volatile Content of Water-Borne Aerosol Paints", November 15, 1992. Other exempt compounds (except for propellants) in aerosol coatings may also be quantified using this method with prior written approval of the Executive Officer, and~~

- (23) the following classes of compounds will be analyzed as exempt compounds only if manufacturers specify which individual compounds are used in the product formulations and identify the test methods, which, prior to such analysis, have been approved by the Executive Officer of the ARB, and can be used to quantify the amounts of each exempt compound: cyclic, branched, or linear, completely fluorinated alkanes; cyclic, branched, or linear, completely fluorinated ethers with no unsaturations; cyclic, branched, or linear, completely fluorinated tertiary amines with no unsaturations; and sulfur-containing perfluorocarbons with no unsaturations and with sulfur bonds only to carbon and fluorine.
- (c) Metal Content. The metal content of metallic aerosol coating products shall be determined by South Coast Air Quality Management District Test Method 311 (SCAQMD "Laboratory Methods of Analysis for Enforcement Samples" manual), June 1, 1991, after removal of the propellant following the procedure in ASTM Method D-5325-92, "Standard Test Method for Determination of Weight Percent Volatile Content of Water-Borne Aerosol Paints", November 15, 1992.
- (d) Specular Gloss. Specular gloss of flat and nonflat coatings shall be determined by ASTM Method D-523-89, March 31, 1989.
- (e) Acid Content. The acid content of rust converters shall be determined by ASTM Method D-1613-91, "Standard Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products, May 15, 1991, after removal of the propellant following the procedure in ASTM Method D-5325-92, "Standard Test Method for Determination of Weight Percent Volatile Content of Water-Borne Aerosol Paints", November 15, 1992.
- (f) Lacquers. Lacquer aerosol coating products shall be identified according to the procedures specified in ASTM Method D-5043-90, "Standard Test Methods for Field Identification of Coatings," April 27, 1990.

NOTE: Authority cited: Sections 39600, 39601, 39607, 41511, and 41712, Health and Safety Code. Reference: Sections 39002, 39600, 39607, 40000, 41511, and 41712, Health and Safety Code

APPENDIX: B

**PROPOSED ARB METHOD 310:
Determination of Volatile Organic Compounds (VOC) in Consumer
Products**



Air Resources Board

PROPOSED

METHOD 310

**DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOC) IN
CONSUMER PRODUCTS**

Adopted: [date of adoption]

DISCLAIMER: Mention of any trade name or commercial product in Method 310 does not constitute endorsement or recommendation of this product by the Air Resources Board.

DRAFT METHOD 310
DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOC) IN CONSUMER PRODUCTS

1.0 APPLICABILITY

- 1.1 This method (Method 310) applies to the determination of the percent by weight of volatile organic compounds (VOC) in consumer products, antiperspirant and deodorant products, and aerosol coatings products, as defined in Title 17, California Code of Regulations, Sections 94500 *et seq.*
- 1.2 Method 310 determines the total volatile material in a product and the presence of any compounds prohibited by ARB regulations ("prohibited compounds"). Components of the product that do not meet the definition of a VOC or are exempted by ARB regulations for a specific product category ("exempt compounds") are subtracted from the total volatile material to determine the final VOC content for the product.
- 1.3 Method 310 does not apply to the determination of the composition or concentration of fragrance components in products.
- 1.4 The term "Executive Officer" as used in this document means the Executive Officer of the Air Resources Board or his or her authorized representative.

2.0 TEST METHODS

Method 310 incorporates by reference the following American Society for Testing and Materials (ASTM), National Institute for Occupational Safety and Health (NIOSH), and United States Environmental Protection Agency (U.S. EPA) analytical test methods:

- 2.1 ASTM D2369-87: Standard Test Method for Volatile Content of Coatings (June 10, 1987).
- 2.2 ASTM D1426-93: Standard Test Methods for Ammonia Nitrogen in Water (September 15, 1993).
- 2.3 ASTM D4017-88: Standard Test Method for Water in Paints and Paint Materials by the Karl Fischer Titration Method (October 31, 1988).

- 2.4 ASTM D3792-86: Standard Test Method for Water Content of Water-Reducible Paints by Direct Injection Into a Gas Chromatograph (November 28, 1986).
- 2.5 ATSM D2887-93: Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography (determination of hydrocarbon content with greater than 12 carbons). (August 15, 1993).
- 2.6 ASTM D859-88: Standard Test Method for Silica in Water (determination of polymethylsiloxanes after digestion) (August 19, 1988).
- 2.7 ASTM E1131-86: Standard Test Method for Compositional Analysis by Thermogravimetry (September 26, 1986).
- 2.8 ASTM D3074-72: (Reapproved 1988) Standard Test Methods for Pressure in Metal Aerosol Containers (Approved July 28, 1972 and reapproved in 1988) with the modifications found in Appendix A.
- 2.9 ASTM D3063-79: (Reapproved 1984) Standard Test Methods for Pressure in Glass Aerosol Bottles (April 27, 1979 and reapproved in 1984) with the modifications found in Appendix A.
- 2.10 ASTM D3064-89: Standard Terminology Relating to Aerosol Products (November 24, 1989).
- 2.11 NIOSH: Method 1400 Alcohols I (analysis of acetone and ethanol by gas chromatography). NIOSH Manual of Analytical Methods, Volume 1 (February 1984).
- 2.12 U.S. EPA Method 8240, September 1986 revision 0, Gas Chromatography/Mass Spectrometry for Volatile Organics (analysis of exempt and/or prohibited compounds in the product by headspace/gas chromatography/mass spectrometry), Test Methods for Evaluating Solid Waste, Volume 1B: Laboratory Manual Physical Chemical Methods, SW-846, November 1986.
- 2.13 U.S. EPA Reference Method 24, Determination of Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids of Surface Coatings: 40 Code of Federal Regulations (CFR) Part 60, Appendix A, as it existed on July 1, 1994.

- 2.14 U.S. EPA Reference Method 24A, Determination of Volatile Matter Content and Density of Printing Inks and Related Coatings: 40 CFR Part 60, Appendix A, as it existed on July 1, 1994.
- 2.15 U.S. EPA Reference Methods 18, Measurement of Gaseous Organic Compound Emissions by Gas Chromatography: 40 CFR Part 60, Appendix A, as it existed on July 1, 1994.
- 2.16 U.S. EPA Method 300.7, March, 1986. Dissolved sodium, ammonium, potassium, and calcium in wet deposition by chemically suppressed ion chromatography.

3.0 TESTING PROCEDURE

- 3.1 The testing begins when the Executive Officer selects a consumer product sample for analysis by Method 310. The Executive Officer will maintain sample chain of custody throughout the selection and analytical process.
- 3.2. **Initial Testing of Aerosol Products.** If the sample is an aerosol product, the aerosol propellant is separated from the liquid portion of the product by using ASTM D3074-72 (as modified in Appendix A for metal aerosol container) or ASTM D3063-79 (as modified in Appendix A for glass aerosol container). The propellant portion is analyzed for exempt or prohibited compounds by using U.S. EPA Method 18. The remaining liquid portion of the product is then analyzed as specified in section 3.3.
- 3.3 **Initial Testing of Non-Aerosol Products and the Liquid Portion of Aerosol Products.** The liquid, solid, or gel product sample is analyzed to determine the total volatile material present in the sample and to determine the presence of any exempt or prohibited compounds. This analysis is conducted by performing the following tests:²
 - 3.3.1 Gravimetric analysis of samples to determine the weight percent of total volatile material, using U.S. EPA Method 24/24A, ASTM E1131-86, ASTM D2369-87..
 - 3.3.2 Determination of sample water content. For determination of water content either ASTM D4017-88 or ASTM D3792-86 may be used.
 - 3.3.3 Determination of ammonium content using ASTM D1426-93 or U.S. EPA

²

Alternate test methods may be used, as provided in section 6.0

Method 300.7.

3.3.4 Determination of ketones and alcohol content using NIOSH 1400.

3.3.5 Analysis of exempt and prohibited compounds, if present (U.S. EPA Method 18, U.S. EPA Method 8240, ASTM D2887-93, ASTM D859-88, NIOSH 1400, ASTM E1131-86).

3.4 **Prohibited Compounds.** If the sample is found to contain compounds prohibited by ARB regulations (i.e., ozone-depleting compounds) at concentrations equal to or exceeding 0.1 percent by weight, the Executive Officer will reanalyze the sample for confirmation.

3.5 **Initial Determination of VOC Content.** The Executive Officer will determine the VOC content pursuant to sections 3.2 and 3.3. Only those components with concentrations equal to or greater than 0.1 percent by weight will be reported.

3.5.1 Using the appropriate formula specified in section 4.0, the Executive Officer will make an initial determination of whether the product meets the applicable VOC standards specified in ARB regulations. If initial results show that the product does not meet the applicable VOC standards, the Executive Officer may perform additional testing to confirm the initial results.

3.5.2 If the results obtained under section 3.5.1 show that the product does not meet the applicable VOC standards, the Executive Officer will request the product manufacturer or responsible party to supply product formulation data. The manufacturer or responsible party shall supply the requested information. Information submitted to the ARB Executive Officer may be claimed as confidential; such information will be handled in accordance with the confidentiality procedures specified in Title 17, California Code of Regulations, sections 91000 to 91022.

3.5.3 If the information supplied by the manufacturer or responsible party shows that the product does not meet the applicable VOC standards, then the Executive Officer will take appropriate enforcement action.

3.5.4 If the manufacturer or responsible party fails to provide formulation data as specified in section 3.5.2, the initial determination of VOC content under this section 3.5 shall determine if the product is in compliance with the applicable VOC standards. This determination may be used to establish a violation of ARB regulations.

3.6 **Final Determination of VOC Content.** If a product's compliance status is not satisfactorily resolved under section 3.5, the Executive Officer will conduct further analyses and testing as necessary to verify the formulation data.

3.6.1 If the accuracy of the supplied formulation data is verified and the product sample is determined to meet the applicable VOC standards, then no enforcement action for violation of the VOC standards will be taken.

3.6.2 If the Executive Officer is unable to verify the accuracy of the supplied formulation data, then the Executive Officer will take appropriate enforcement action.

3.6.4 If there exists a discrepancy that cannot be resolved between the results of Method 310 and the supplied formulation data, then the results of Method 310 shall take precedence over the supplied formulation data. The results of Method 310 shall then determine if the product is in compliance with the applicable VOC standards, and may be used to establish a violation of ARB regulations.

4.0 CALCULATION OF VOC CONTENT

4.1 *Aerosol Products*

For aerosol products, the percent VOC content shall be calculated using the following equation:

$$\text{PERCENT VOC} = \frac{\text{WL(TV-A-H-EL)} + \text{WP} - \text{EP}}{\text{WL} + \text{WP}} \times 100\%$$

Where³:

WL = weight (gm) of liquid product excluding container and packaging

TV = weight fraction of non-propellant total volatile material (U.S. EPA 24/24A, ASTM D2369-87, ASTM E1131-86)

³ Alternate test methods, as provided in 6.0, or appropriate approved methods from section 2.0 may be used.

- A = weight fraction of ammonia (as NH₄) in liquid (ASTM D1426-93) or U.S. EPA Method 300.7
- H = weight fraction of water in liquid (ASTM D3792-86 or ASTM D4017-88)
- EL = weight fraction of exempt compounds in liquid (U.S. EPA Method 8240, U.S. EPA Method 18, ASTM D2887-93, ASTM D859-88, NIOSH 1400, ASTM E1131-86)
- WP = weight (gm) of propellant (ASTM D3074-72 [as modified and include ASTM D3064-89] or ASTM D3063-79 [as modified and include ASTM D3064-89])
- EP = weight (gm) of exempt compounds in propellant (U.S. EPA Method 18)

4.2 Non-Aerosol Products

For non-aerosol products, the percent VOC content shall be calculated using the following equation:

$$\text{PERCENT VOC} = (TV-A-H-EL) \times 100\%$$

5.0 METHOD PRECISION AND ACCURACY

The precision of Method 310 was evaluated using seven representative products with known volatile organic compound (VOC) contents ranging from 6.2 to 81.2 percent VOC by weight. Each sample was divided into six portions, and each portion was separately analyzed to determine the VOC content. Based on the results of this analysis, the 95 percent confidence interval for Method 310 is 2.00 percent.

6.0 ALTERNATE TEST METHODS

Alternative test methods which are shown to accurately determine the concentration of VOCs or constituent components in antiperspirant/deodorants, consumer products, or aerosol coating products (or their emissions) may be used upon written approval of the Executive Officer.

Appendix A
ASTM D3074-72 and D3063-79 MODIFICATIONS

1.0 SUMMARY OF MODIFICATIONS

This procedure modifies ASTM D3074-72 and D3063-79 to allow collection of the propellant for analysis and density measurement for metal aerosol containers and glass aerosol containers, respectively. These modified procedures also retain the aerosol standard terminology listed in ASTM D3064-89. The aerosol product container is pierced and the propellant is bled into an evacuated manifold. After the manifold reaches atmospheric pressure, approximately 1 liter of the propellant is collected in a clean, evacuated Tedlar bag. For density measurement the propellant is collected into an evacuated 250 ml glass dilution bulb that has been weighed to the nearest 0.1 mg. After filling, the dilution bulb is re-weighed to determine the density of the propellant. Alternately, density may be determined using a Density/Specific Gravity Meter. The Tedlar bag with the propellant aliquot is taken to the laboratory for analysis.

2.0 LIMITATIONS

Nitrogen analysis: Nitrogen may be used as a component of the propellant system. Ambient air is 78 percent nitrogen and may be present as a contaminate in the system prior to sample collection. This is eliminated by completely evacuating the propellant collection system and sweeping out any connecting lines to the Tedlar bag with product before starting sample collection. This procedure will eliminate or reduce nitrogen contamination to less than 0.1% by weight of the sample and the analysis of the propellant gas will be unaffected.

3.0 APPARATUS AND MATERIALS

3.1 Propellant Collection System⁴: See Figure 1. The system was built from 1/4" stainless steel and Teflon tubing. The vacuum pump is of bellows diaphragm design.

3.2 Tedlar Bags, 1 liter, equipped with slip valve and septum

3.3 Density Measurement

The metal piercing adapter is available from Mid-West Screw Products, Inc., 3523 North Kenton Ave., Chicago, IL 60641. Interim Part Number: 8013A-3/4 Longer SS. The gasket is available from Alltech Associate 2051 Waukegan road, Deerfield, IL 60015, part number 80-16. The glass aerosol adapter is available from Modern Machine Ship, Inc. P.O. Box 826, 123 N. Hazel Street, Danville, IL 61832.

- 3.3.1 250 ml gas dilution bulb
- 3.3.2 or, an Density/Specific gravity meter meeting the following minimum specifications:

- 3.3.2(a). Measurement Method: Natural Oscillation Type

- 3.3.2(b). Range: 0 - 3g/cm³

- 3.3.2(c). Measurement Temperature Range: 4°C ~ 70°C.

- 3.3.2(d). Temperature Accuracy: +/- 0.02°C (10° C ~ 30 C) and +/- 0.05°C (4°C ~70°C).

- 3.3.2(e). Temperature Control Accuracy: +/- 0.01°C.

- 3.3.2(f). Measurement Time: 1- 4 minutes.

- 3.4 Gas tight syringe, 100 µl

- 3.5 Balance, capable of accurately weighing to 0.1 mg

- 3.6 Can Piercing Platform. See Figure 2 (metal cans) and Figure 3 (glass containers).

- 3.7 Platform Shaker, equivalent to Thermolyne M49125

4.0 PROCEDURE

4.1 Propellant Collection for Metal Aerosol Containers

- 4.1.1 Turn on vacuum pump, close valves and evacuate the system (see Figure 1).

- 4.1.2 Remove the valve actuator on the aerosol can and weigh can to the nearest 0.01 g. Invert the can into cork holding ring on the piercing apparatus, center and snug against the gasket. (Figure 2)

- 4.1.3 Connect Tedlar bag to output 2, evacuate bag and seal. Connect 250 ml glass dilution bulb to output 1, evacuate bulb and seal.

- 4.1.4 Slowly raise the hydraulic jack until the can is pierced. Record the pressure of the can.

- 4.1.5 Vent the can until the pressure is at about 25 psi. Collect the propellant in the Tedlar bag.
 - 4.1.6 After the propellant is collected, close and remove the Tedlar bag and vent the remainder of the propellant.
 - 4.1.7 Weigh the evacuated 250 ml bulb to the nearest 0.1 mg. Use gloves while handling the bulb. Connect the bulb to the tedlar bag and open to fill the bulb. Close the valves and re-weigh the dilution bulb, record the weight gain and calculate the propellant density in gm/l.
 - 4.1.8 After the flow ceases from the can, it is removed from the assembly and allowed to vent overnight. The can may be placed on a platform shaker to vent the remainder of the propellant.
 - 4.1.9 Reweigh can to the nearest 0.01 gm and record weight loss (total gms propellant). The can may now be opened for analysis of the liquid product.
- 4.2 Propellant Collection for Glass Aerosol Containers
- 4.2.1 Turn on vacuum pump, close valves and evacuate the system (see Figure 1).
 - 4.2.2 Connect Tedlar bag to output 2, evacuate bag and seal. Connect 250 ml glass dilution bulb to output 1, evacuate bulb and seal.
 - 4.2.3 The gauge assembly is prepressurized in order to minimize product expulsion and system contamination.
 - 4.2.4 Remove actuator from valve of the aerosol glass container, and weigh container to the nearest 0.01gm.
 - 4.2.5 With container in an inverted position place the valve onto the tapered adaptor. Bring the top plate down to the flat of the container and tighten the nuts. A cork ring may be required to stabilize the container.
 - 4.2.6 Record pressure of container and vent until the pressure is approximately one-half of recorded pressure. Collect propellant sample into the tedlar bag.
 - 4.2.7 After the propellant is collected, close and remove the tedlar bag and vent the remainder of the propellant.

- 4.2.8 Weigh the evacuated 250 ml bulb to the nearest 0.1 mg. Use gloves while handling the bulb. Connect the bulb to the Tedlar bag and open to fill the bulb. Close the valves and re-weigh the dilution bulb, record the weight gain and calculate the propellant density in gm/l.
- 4.2.9 Continue to vent container on the platform assembly overnight.
- 4.2.10 Remove container from platform and loosen valve assembly, do not remove valve assembly at this time.
- 4.2.11 Placed container on a platform shaker to vent the remainder of the propellant.
- 4.2.12 Reweigh container and valve assembly to the nearest 0.01 gm and record weight loss (total gms propellant). The container may now be opened for analysis of the liquid product.

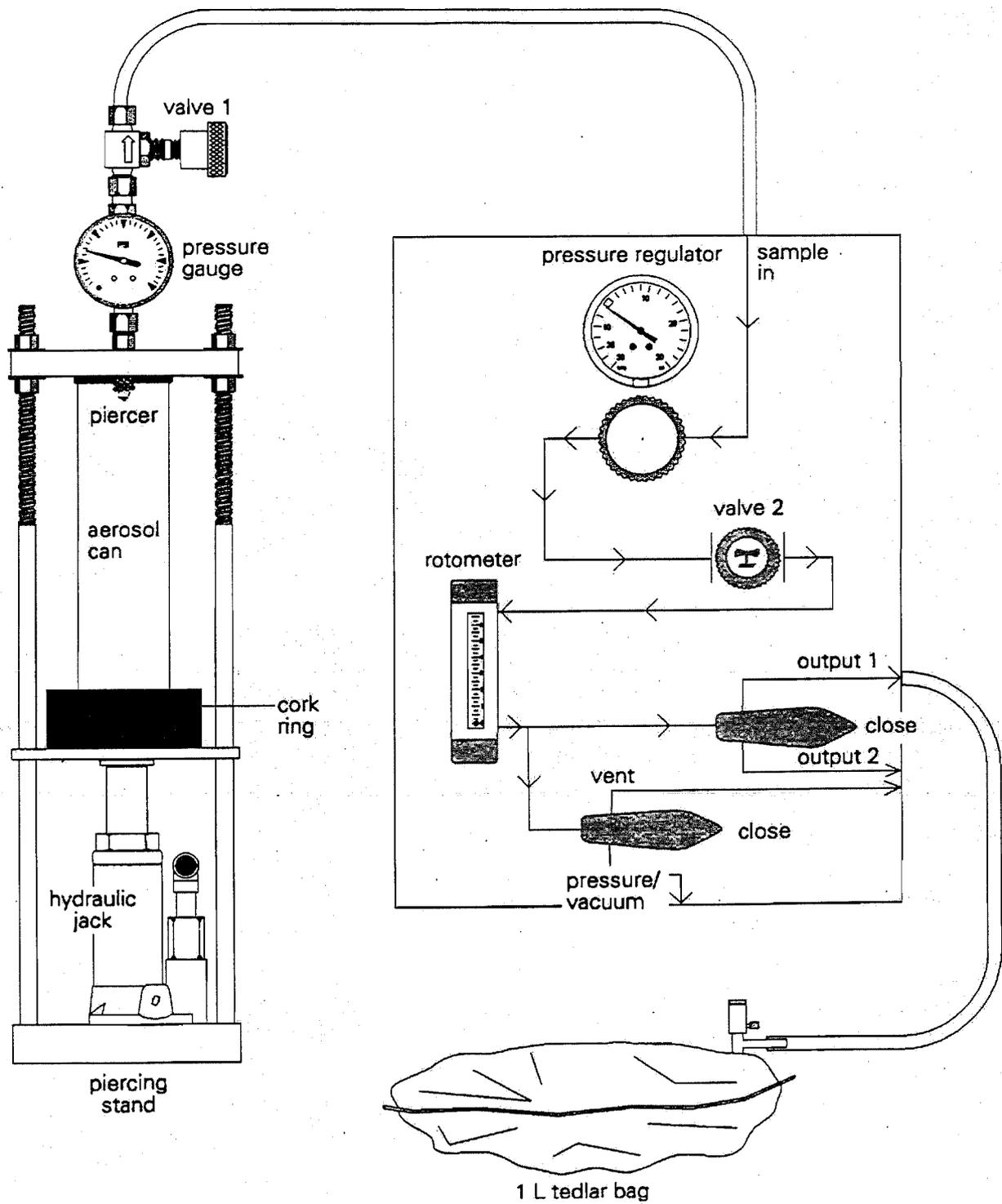
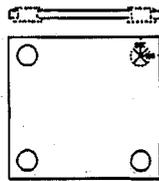
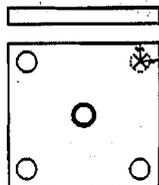


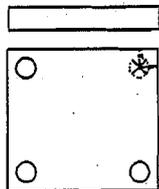
FIGURE 1



6" X 6" X 3/16" Steel Jack Plate
Center Holes 5/8" from edge
Drill 4 perimeter holes to allow
for a 1/2" bushing that works
with the smooth portion of the 1/2" rods
Tack weld the lift portion of the
hydraulic jack to the center of the plate
(weld while jack is fully extended as to
not damage it)



6" X 6" X 1/2" Aluminum Piercing Plate
Center holes 5/8" from edge
Drill 4 perimeter hole with 9/16" bit
Drill center holes with 7/16" bit
Tap center using 1/2 X 20 NF tap
Sample piercer is included to ensure
drill bit and tap size as center hole is
crucial to apparatus)



6" X 6" X 3/4" Aluminum Base Plate
Center holes 5/8" from edge
Drill 4 perimeter holes with 23/32" bit
Tap 4 perimeter holes with 1/2 X 13

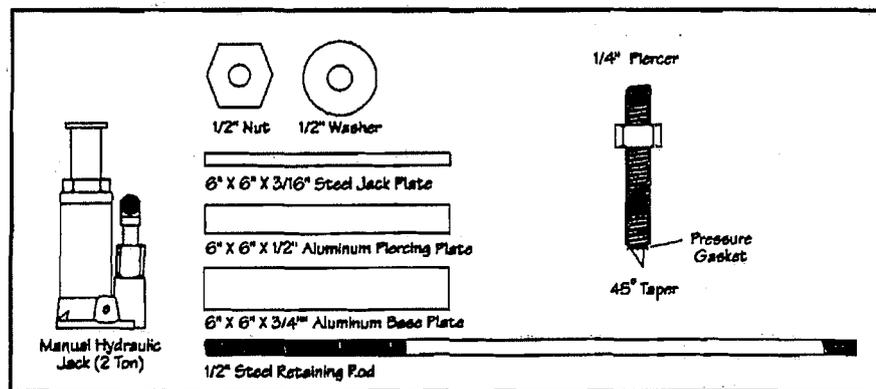
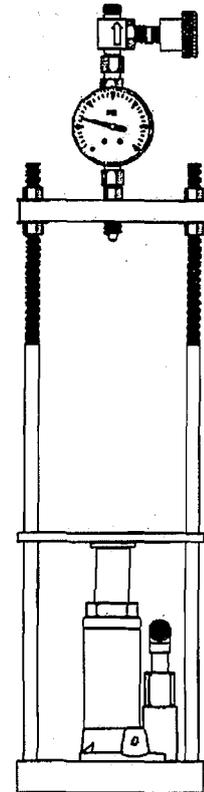


Figure 2

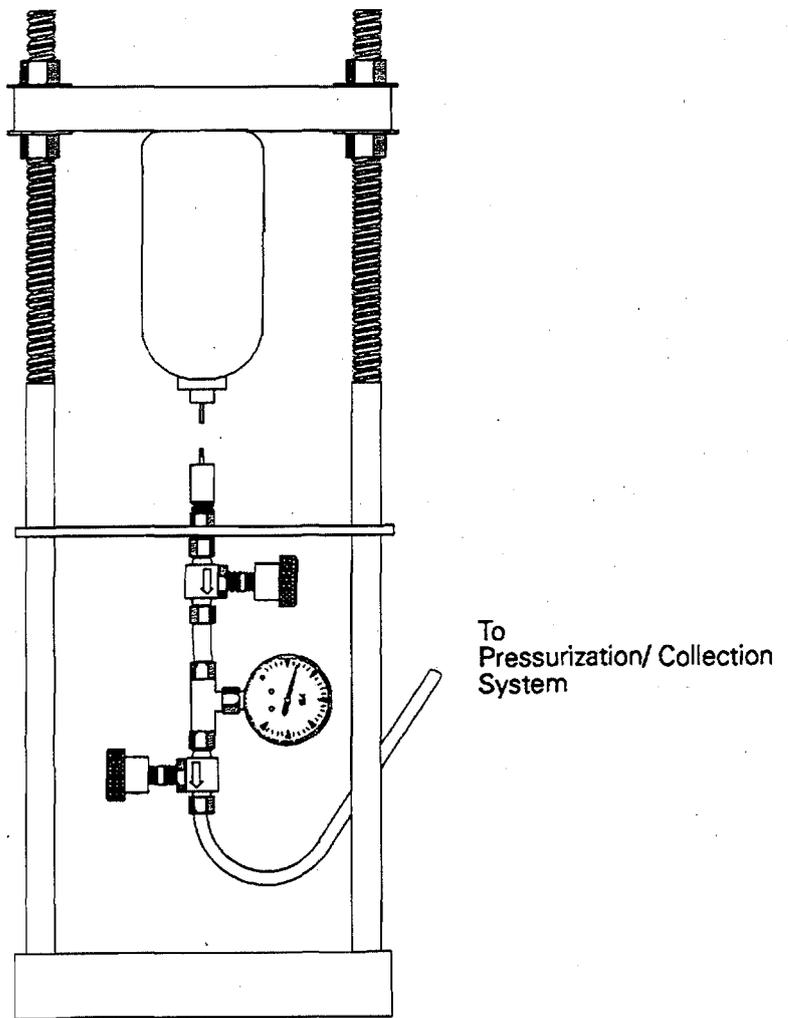


Figure 3

