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SUPERIOR COURT OF THE STATE OF CALIFORNIA

FOR THE CITY AND COUNTY OF SAN FRANCISCO

PEOPLE OF THE STATE OF CALIFORNIA ex rel. DANIEL E. LUNGREN, Attorney General of the State of California, Plaintiffs, v. MINNESOTA MINING AND MANUFACTURING COMPANY (3M), Defendant.	No. 992239 NOTICE OF ENTRY OF CONSENT JUDGMENT

TO ALL PARTIES AND THEIR COUNSEL OF RECORD: PLEASE TAKE NOTICE that on January 21, 1998 the Court entered the attached Consent Judgment between the People of the State of California and Minnesota Mining and Manufacturing Company (3M).

Dated: 1/26, 1998

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SUPERIOR COURT OF THE STATE OF CALIFORNIA

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PEOPLE OF THE STATE OF CALIFORNIA ex rel. DANIEL E. LUNGREN, Attorney General of the State of California,

Plaintiffs,

v.

MINNESOTA MINING AND MANUFACTURING COMPANY (3M),

Defendant.

No. 992239

CONSENT JUDGMENT

Plaintiff, the People of the State of California ("People") and defendant MINNESOTA MINING AND MANUFACTURING COMPANY ("3M" or "Settling Defendant") herein enter into this Consent Judgment as follows:

1. Introduction

1.1 On January 12, 1998 the People of the State of California, ex rel. Daniel E. Lungren, ("People") filed a Complaint for Civil Penalties and Injunctive Relief ("Complaint") in the Superior Court of the State of California, City and County of San Francisco, against 3M.

1.2 3M is a company that employs more than ten persons and offers for sale within the State of California, Titrilac Antacid containing calcium which is intended to be ingested by human beings (hereinafter "Antacid"). The term "calcium" as used in this Consent Judgment means elemental calcium when referring to an amount of calcium and means any form or salt of calcium when referring to calcium as an ingredient (active or inactive) in an Antacid. For purposes of this Consent Judgment the "date of shipment" shall be the date on which the Antacid first enters the stream of commerce.

1.3 The People's Complaint alleges that 3M, through the sale of Antacids to consumers in California, violated provisions of the Safe Drinking Water and Toxic Enforcement Act of 1986, Health and Safety Code sections 25249.5 et seq. ("Proposition 65"), and Business and Professions Code sections 17200 et seq. ("Unfair Competition Act"), by knowingly exposing persons to lead, a chemical known to the State of California to cause reproductive toxicity, without first providing a clear and reasonable warning to such individuals.

1.4 For purposes of this Consent Judgment only, the parties stipulate that this Court has jurisdiction over the allegations of violations contained in the Complaint and personal jurisdiction over 3M as to the acts alleged in the Complaint, that venue is proper in the City and County of San Francisco and that this Court has jurisdiction to enter this Consent Judgment.

1.5 The parties enter into this Consent Judgment as a settlement of certain disputed claims between the parties as alleged in the Complaint for the purpose of avoiding prolonged and costly litigation between the parties hereto. By execution of this Consent Judgment, 3M does not admit any facts or conclusions of law suggesting or demonstrating any violations of Proposition 65, the Unfair Competition Act or any other statutory, common law or equitable requirements relating to Antacids. Nothing in this Consent Judgment shall be construed as an admission by 3M of any fact, issue of law or violation of law, nor shall compliance with the Consent Judgment constitute or be construed as an admission by 3M of any fact, issue of law, or violation of law. Nothing in this Consent Judgment shall prejudice, waive or impair any right, remedy or defense 3M may have in this or any other or future legal proceedings. However, this paragraph shall not diminish or otherwise affect the obligations, responsibilities and duties of 3M under this Consent Judgment.

2. Injunctive Relief - Warning Program

2.1 Where required herein, clear and reasonable warning that use of Antacids exposes persons to lead, a chemical known to the State of California to cause birth defects or other reproductive harm, shall be provided by 3M in the manner provided in this Consent Judgment.

2.2 3M shall provide a warning, pursuant to paragraph 2.5, for each Antacid whose date of shipment is on or after January 1, 1998, unless 3M can show, pursuant to paragraph 2.10 and the testing protocol set forth in attached Exhibit A attached to this Consent Judgment that the Antacid causes a total daily exposure to lead of 0.5 micrograms or less, based on the amount of the Antacid supplying a thousand (1,000) milligrams of elemental calcium, excluding any naturally occurring lead in the Antacid as set forth in paragraph 2.3 below. For those Antacids where the recommended or maximum daily dose supplies more than 1500 milligrams of calcium, 3M shall provide a warning for each antacid whose date of shipment is one or after January 1, 1998 unless 3M can show, pursuant to paragraph 2.10 and the testing protocol set forth in attached Exhibit A, that the

recommended daily dose of the Antacid causes a total daily exposure to lead equal to or less than that set forth in paragraphs 2.3 and 2.4 below.

2.3 3M shall be entitled to exclude from the calculation of the daily lead exposure caused by a Antacid, the amount of lead per 1000 milligrams of calcium as set forth in Table 2.3 of this paragraph 2.3. Compliance with this Consent Judgment is established and no warning is required under Proposition 65 where the lead exposure caused by an amount of the Antacid supplying 1000 milligrams of calcium does not exceed the sum of: (a) 0.5 micrograms of lead per thousand milligrams of elemental calcium and (b) the amount of lead excluded on the date of shipment as "naturally occurring" pursuant to Table 2.3 of this paragraph 2.3. For purposes of this Consent Judgment, Table 2.3 of this paragraph 2.3 sets forth the amount of lead per 1000 milligrams of elemental calcium which shall be deemed to be "naturally occurring" at the "lowest level currently feasible" pursuant to Section 12501 of Title 22 of the California Code of Regulations ("CCR"). The amounts of lead and dates set forth in Table 2.3 shall apply as of the date of shipment of the Antacid.

TABLE 2.3

DATE	NATURALLY OCCURRING AMOUNT OF LEAD PER 1000 MILLIGRAMS OF CALCIUM
January 1, 1998	1.0 microgram

2.4 Even if no warning is required by paragraphs 2.2 and 2.3 above, in the event that the recommended daily dose of any Antacid, as specified on the label or in any other package material, exceeds 1500 milligrams of elemental calcium, 3M shall provide a warning pursuant to Proposition 65 if the total daily lead exposure from the Antacid, based on the recommended daily dose, exceeds 150% of the level that would require a warning pursuant to paragraphs 2.2 and 2.3 above (based on an amount of the Antacid supplying 1000 milligrams of calcium) as of the date of shipment of the Antacid. For Antacids that do not have a recommended daily dose, for purposes of this Consent Judgment, the term "recommended daily dose" shall mean two-thirds of the maximum daily dose as set forth on

the label or in any other package material.

2.5 For an Antacid that does not meet the standards set forth in paragraphs 2.2 through 2.4 above, 3M shall, at the point of manufacture, prior to shipment to California or prior to distribution within California, (1) affix to or print on the Antacid container, cap, label or unit package or (2) display at the point of sale of the Antacid, the following warning (the language in brackets in the warning below is optional):

> **WARNING**: This product contains [lead] a chemical known [to the State of California] to cause birth defects or other reproductive harm

2.6 The warning required by paragraph 2.5 above shall be prominently affixed to, printed on or displayed proximately to the point-of-sale of each Antacid with such conspicuousness, as compared with other words, statements, designs, or devices on the labeling as to render it likely to be read and understood by an ordinary individual under customary conditions of purchase or use. If the warning is displayed on the product container or labeling, the warning shall be at least the same size as the largest of any other health or safety warnings on the product container or labeling and the word "warning" shall be in all capital letters and in bold print. If printed on the labeling itself the warning shall be contained in the same section of the labeling that states other safety warnings concerning the use of the product. The Attorney General agrees to review any labeling or point of sale signs proposed to be used under this section and advise 3M as to whether he believes such labeling or point of sale signs comply with this section. The requirement for product labeling, set forth herein is imposed pursuant to the terms of this Consent Judgment and is recognized by the parties as not being the exclusive method of providing a warning under Proposition 65 and its implementing regulations for the Antacid.

2.7 In the event that the Attorney General determines that the naturally occurring levels set forth in Table 2.3 of paragraph 2.3 above are higher than the "lowest level currently feasible" as stated in 22 CCR section 12501(a)(4), he shall have the right to seek a modification of the Consent Judgment to reflect the alleged "lowest level currently feasible"

of naturally occurring lead in the Antacids. Prior to seeking such modification, the Attorney General shall provide written notice to 3M that he intends to seek the modification. The parties shall have ninety (90) days in which to confer with the Attorney General concerning the modification. If 3M and the Attorney General are unable to agree on a modification to the Consent Judgment, the Attorney General may file a motion with the Court, seeking a modification of the Consent Judgment. In any motion by the Attorney General seeking such a modification, the burden of producing evidence shall be initially upon the Attorney General to demonstrate a prima facie case that the modification sought by the Attorney General is the "lowest level currently feasible." If 3M does not agree to such modification, it retains the ultimate burden of proving that the modification sought by the Attorney General is lower than the "lowest level currently feasible." The parties hereby agree that the Consent Judgment should be modified to reflect any agreement of the parties or any determination by the Court concerning what is the "lowest level currently feasible" for lead in Antacids.

2.8 In the event that 3M determines that the naturally occurring levels set forth in Table 2.3 of paragraph 2.3 above are lower than the "lowest level currently feasible," as stated in 22 CCR section 12501(a)(4), 3M shall have the right to seek modification of the Consent Judgment to reflect the alleged "lowest level currently feasible." Prior to seeking such modification, 3M shall provide written notice to the Attorney General that it intends to seek the modification. The parties shall have ninety (90) days in which to confer concerning the modification. If the parties are unable to agree on a modification to the Consent Judgment, 3M may file a motion with the Court, seeking a modification of the Consent Judgment. In any motion by 3M seeking such modification, the burden of producing evidence and of proof shall be on 3M to prove that the modification sought by 3M is the "lowest level currently feasible." The parties hereby agree that the Consent Judgment should be modified to reflect any agreement of the parties or any determination by the Court concerning what is the "lowest level currently feasible" for lead in Antacids.

2.9 The term "feasible" as used in paragraphs 2.7 and 2.8 above includes, but is

not limited to, a consideration of the following factors: availability and reliability of a supply of low-lead calcium that meets the requirements set forth in paragraphs 2.2, 2.3 and 2.4 above; cost of low-lead calcium and resulting increase in manufacturers' prices resulting from the use of the low-lead calcium; performance characteristics of low-lead calcium and of the resulting Antacid, including, but not limited to formulation, performance, safety, efficacy and stability. Nothing in this Consent Judgment shall be interpreted to require 3M to use any calcium material as an ingredient in an Antacid that would render its Antacid unlawful under state or federal law as measured by existing and/or future applicable California and federal food and drug laws and regulations. Nothing in this Consent Judgment shall be interpreted to preclude 3M from advocating, for purposes of paragraphs 2.7 and/or 2.8 that any proposed modification requiring a change in the type of raw calcium source material as an ingredient in an Antacid is not feasible as defined herein. Nothing in this Consent Judgment shall be interpreted to preclude the People from advocating, for purposes of paragraphs 2.7 and/or 2.8 that any proposed modification requiring a change in the type of raw calcium source material as an ingredient in an Antacid is feasible as defined herein.

2.10 3M shall maintain records sufficient to establish its compliance with this Consent Judgment for a period of four years following the date of shipment of any Antacid into California. Such documents shall be sufficient in detail to establish compliance with the Protocol set forth in the attached Exhibit A. Upon reasonable written notice from the Attorney General's Office, 3M must produce to the Attorney General within ten (10) business days of the receipt of the Attorney General's notice, the documents required to be maintained according to this paragraph. To the extent that such documents contain information which 3M maintains is confidential, proprietary, and/or in the nature of a trade secret (or in fact a trade secret), and upon written notice as to the asserted confidential nature of this information by 3M, the Attorney General agrees not to disclose this information to third parties (though the Attorney General may disclose this information to its attorneys and employees, including professional consultants, provided that these persons also agree to

maintain the confidentiality of the information in these documents). In addition, 3M may designate as confidential "trade secret" information as that term is defined in California Government Code section 6254.7 any data provided to the Attorney General's Office pursuant to this paragraph or any other provision of this Consent Judgment or relating to the subject matter hereof and such information shall not be released to any member of the public. Provided, however, that nothing in this provision shall prohibit the Attorney General from disclosing information and/or data designated as confidential, proprietary and/or trade secret to other government agencies as is necessary in pursuit of his enforcement authority. Furthermore, nothing in this provision shall prohibit the Attorney General from applying to the Court for a ruling determining that the information and/or data designated by 3M as confidential, proprietary and/or trade secret should not be so designated and may be freely disclosed.

3. Settlement Payments

3.1 Within thirty (30) days of execution of this Consent Judgment as full, final and complete satisfaction of all claims for civil penalties or restitution for the alleged violations up through the date of execution of this Consent Judgment as set forth in paragraph 10.1, for Antacids, 3M shall pay the sum of $5,000 to the Public Health Trust, a program of the California Public Health Foundation, to be used for research, investigation and public education projects approved by the Attorney General and relating to exposure to lead in pregnancy and/or nutritional factors related to lead exposure among children. Payment shall be made by delivery of certified funds payable to the Public Health Trust. Making these payments shall not be construed as an admission by 3M of any fact, issue of law or violation of law, nor shall compliance with the Consent Judgment constitute or be construed as an admission by 3M of any fact, issue of law, or violation of law.

4. Payment of Costs and Fees

4.1 Within thirty (30) days of execution of this Consent Judgment, 3M shall pay $10,000 as reimbursement for the costs of investigating and prosecuting this action. Payment

shall be made by delivery of certified funds payable to the Attorney General of the State of California at 2101 Webster Street, 12th Floor, Oakland, California 94612-3049 (Attn: Susan S. Fiering, Deputy Attorney General).

5. Additional Enforcement Actions; Continuing Obligations

5.1. By entering into this Consent Judgment the People do not waive any right to take further enforcement actions on any violations not covered by the Complaint. Nothing in this Consent Judgment shall be construed as diminishing 3M's continuing obligation to comply with Proposition 65 or the Unfair Competition Act in its future activities.

6. Enforcement of Consent Judgment

6.1. The People may, by motion or order to show cause before the Superior Court of San Francisco, enforce the terms and conditions contained in this Consent Judgment. In any action brought by the People to enforce this Consent Judgment the People may seek whatever fines, costs, penalties or remedies as provided by law for failure to comply with the Consent Judgment. Where said failure to comply constitutes future violations of Proposition 65 or other laws, independent of the Consent Judgment and/or those alleged in the Complaint, the People are not limited to enforcement of this Consent Judgment but may seek in another action whatever fines, costs, penalties or remedies are provided by law for failure to comply with Proposition 65 or other laws. In any such future action, the standards and protocol set forth in Section 2 above, as they may be modified from time to time pursuant to paragraphs 2.7 or 2.8 shall apply. However, the rights of 3M to defend itself and its actions in law or equity shall not be abrogated or reduced in any fashion by the terms of this paragraph.

7. Application of Consent Judgment

7.1 The Consent Judgment shall apply to, be binding upon and inure to the benefit of, the parties, their divisions, subdivisions, subsidiaries, and affiliates and the successors or assigns of each of them.

8.0 Application of Testing Standard and Protocol

8.1 The testing standard and protocol set forth in Exhibit A attached to this Consent Judgment are based on determinations concerning the nature of the laboratory test used and its relationship to actual and specific conditions of Antacid use. This Consent Judgment, including, but not limited to, this standard and protocol, is the product of negotiation and compromise and is accepted by the parties, for purposes of settling, compromising and resolving issues disputed in this action, including future compliance by 3M with Section 2 of this Consent Judgment and shall not be used for any other purpose, or in any other matter and, except for the purpose of determining future compliance with this Consent Judgment shall not constitute an adoption or employment of a method of analysis for a listed chemical in a specific medium as set forth in 22 CCR section 12901(b).

9. Authority to Stipulate to Consent Judgment

9.1 Each signatory to this Consent Judgment that he or she is fully authorized by the party he or she represents to enter into this Consent Judgment on behalf of the party represented and legally to bind that party.

10. Claims Covered

10.1 This Consent Judgment is a final and binding resolution between the People and 3M of any and all alleged violations of Proposition 65, the Business and Professions Code Sections 17200 et seq. and/or the Consumers Legal Remedies Act, Civil Code section 1750 et seq. up through the date of execution of this Consent Judgment, arising from failure to warn of exposure to lead from consumption of Titrilac Antacid that was committed by 3M or by any entity within its respective chain of distribution, including, but not limited to, distributors, wholesalers and retailers of any of 3M's Titrilac Antacid. All new Antacids hereafter introduced into the stream of commerce for distribution or sale in California shall be governed by this Consent Judgment. Nothing in this Consent Judgment shall preclude 3M from establishing that any non-calcium ingredient in a calcium containing product other than Antacids, contains naturally occurring lead at the "lowest level currently feasible" pursuant to

22 CCR section 12501.

11. Modification

11.1 This Consent Judgment may be modified from time to time by express written agreement of 3M and the Attorney General with the approval of the Court or by an order of this Court.

12. Execution in Counterparts

12.1 This Consent Judgment may be executed in counterparts, which taken together shall be deemed to constitute one and the same document.

13. Entry of Consent Judgment Required

13.1 This Consent Judgment shall be null and void, and be without any force or effect, unless entered by the Court in this matter. If the Consent Judgment is not entered by the Court, the execution of this Consent Judgment by 3M shall not be construed as an admission by 3M of any fact, issue of law or violation of law.

IT IS SO STIPULATED:

Dated: 12/30, 1997

DANIEL E. LUNGREN, Attorney General of the State of California
RODERICK E. WALSTON
Chief Assistant Attorney General
THEODORA BERGER
Assistant Attorney General
CRAIG C. THOMPSON
EDWARD G. WEIL
SUSAN S. FIERING
Deputy Attorneys General

By: Susan S. Fiering
SUSAN S. FIERING
Deputy Attorney General
Attorneys for the People of the State of California

Dated:

MINNESOTA MINING AND MANUFACTURING COMPANY (3M)

By: _____

Its: Division Vice President 3M Skin Health Division

APPROVED AS TO FORM:

Dated: November 21, 1997

LATHAM & WATKINS

By: _____

BETTY-JANE KIRWAN, Esq.
Attorneys for Minnesota Mining and Manufacturing Company (3M)

IT IS SO ORDERED:

Dated: JAN 14 1998

LUCY KELLY McCABE
Presiding Judge

JUDGE, SUPERIOR COURT
CITY AND COUNTY OF SAN FRANCISCO

EXHIBIT A

Calcium Containing Finished Product Lead Testing Protocol

Inductively Coupled Plasma Mass Spectrometry Procedure (ICP-MS)

Pb 1.0 Protocol Objective and Purpose

The purpose of this protocol is to define the procedures and methods used to analyze lead in calcium containing products. The protocol defines the following requirements: (1) method validation, (2) sample collection & retention, (3) analyses of samples, and (4) Limits. This lead testing protocol defines the procedures, limits and provides experimental confirmation that the data is reliable for the tested products. This protocol shall become effective for purposes of establishing compliance with lead level limits only after all challenges to its contents and validity have been resolved or waived.

The manufacturer shall be responsible for ensuring that all testing of calcium containing products, whether performed by the manufacturer's employees or by independent laboratories, is performed properly. All samples shall be obtained from either the production line or packaged product. Sufficient quantities of product shall be obtained to perform the testing in duplicate at a minimum and to maintain "retain" samples sufficient in quantity for additional investigation. Testing of a given formula of a calcium product shall be deemed to establish the lead level only for that formula of calcium product and formulas of calcium products which share all of the same ingredients (or a subset of the same ingredients but no additional ingredients) in substantially the same ratios as the tested calcium product. Test results for a lot of a calcium product showing compliance on a lot-by-lot basis shall remain valid for purposes of demonstrating compliance for that lot of the calcium product. Test results for a calcium product showing compliance on a product line basis shall remain valid for purposes of demonstrating product line compliance unless there is a material change in the product's formula, manufacturing process or ingredients. For calcium products which are to be shipped on or after July 1, 1997, the manufacturer must test such calcium products pursuant to this protocol by July 1, 1997, or as soon thereafter as is reasonably feasible. Manufacturers may rely on analytical testing which is substantially equivalent (i.e., results within 15%, validation meeting the acceptance criteria for validation of this protocol, and showing no assay bias) to this protocol to demonstrate compliance for calcium products to be shipped on or after July 1, 1997, until testing pursuant to this protocol is completed. For calcium products which are to be shipped on or after April 1, 1999, the manufacturer must test such calcium products pursuant to this protocol by April 1, 1999. In the event of disagreement between testing results produced using a method complying with this protocol and testing results produced using a method which is not substantially equivalent to this protocol, the former shall be preferred.

1.1 References

This lead testing protocol is designed to be used in combination with additional documentation included, but are not limited, to the following:

a. Instrument manuals.
b. Instrument Software manuals
c. Standard Operating Procedures
d. Calibration Standard Certifications
e. Computerized System Qualification
f. Instrument Installation Qualification
g. Instrument Operational Qualification
h. Instrument Performance Qualification
i. Analyst Training Records.
j. USP 23 Section <1225>, Validation of Compendial Methods, pp. 1982 to 1985, Category II Quantitative assays for impurities in bulk drug substances or degradation products in finished pharmaceutical products."
k. Federal Register Notice, March 1, 1995, International Conference on Harmonization (ICH), Guideline on Validation of Analytical Procedures: Definitions and Terminology.

Pb 2.0 Method Validation Requirements

As detailed in this section, the method shall be validated within any laboratory scheduled to conduct analyses of calcium containing products prior to conducting analyses intended to demonstrate compliance. Validation of the method shall be repeated when and if significant changes in the laboratory (e.g., replacement of equipment) make reliance on the prior validation inappropriate.

2.1 Accuracy

2.1.1 Definition

The accuracy of an analytical method expresses the closeness of test results obtained by that method to the true value. Accuracy may often be expressed as percent recovery by the assay of known, added amounts of analyte. Accuracy is a measure of the exactness of the analytical method.

2.1.2 Recovery Studies

The accuracy of the method should be assessed for the individual formulation tested. The recovery studies should be performed in the range of 0.05 µg/g to 3.00 µg/g (ppm) on the representative finished product sample.

A 0.5 µg/mL lead stock solution should be prepared by diluting 10 mL of 10 ppm lead standard solution with water to a total volume of 200 mL. A minimum of sixteen samples should be prepared from a composite, each having the sample weight defined in the procedure (1 gram). The first four samples are used to obtain the mean lead value (no lead addition). To the remaining samples, add appropriate volumes of the lead stock solution to cover the recovery range of 0.05 µg/g to 3.00µg/g, using a minimum of three concentrations with four samples per concentration level.

The theoretical amounts of lead in each sample is obtained by adding the average value obtained from the samples containing no spiked lead to the amount spiked in each of the three groups. The µg of lead analyzed in each sample is divided by the theoretical calculated µg of lead amount and multiplied by 100 to obtain percent recovery.

2.1.3 Accuracy Acceptance Criteria

The acceptance criteria for the spiked samples should be within **80% to 120%** recovery.

2.2 Precision & Ruggedness

2.2.1 Definitions

1. **Repeatability:** Repeatability expresses the precision under the same operation conditions over a short period of time.

2. **Intermediate Precision:** Intermediate precision expresses within-laboratory variation. Different days (inter-day precision), different analysts, different equipment, different reagents, acids, and standards, etc. (Part of a ruggedness demonstration.)

2.2.2 Precision Study (Repeatability)

Measure a prepared sample solution ten times and calculate the mean, standard deviation, and percent relative standard deviation (coefficient of variation).

2.2.3 Precision Study Acceptance Criteria

The percent relative standard deviation is less than 15%.

2.2.4 Ruggedness (Intermediate Precision) Study

Prepare a composite sample of at least 20 tablets or equivalent as defined in the method. Have two different analysts analyze six samples each from the same composite sample on different days, using different equipment (if possible), reagents, standards, and acids. Calculate the mean, standard deviation, and relative standard deviations separately for the two analysts data.

2.2.5 Ruggedness Study Acceptance Criteria

The relative standard deviations for each of the analysts are less than 25%. The mean values between the two analysts are within 25% relative.

2.3 Limit of Detection

2.3.1 Definition

The limit of detection is a parameter of limit tests. It is the lowest concentration of analyte in a sample that can be detected, but not necessarily quantitated, under the stated experimental conditions. Thus, limit tests merely substantiate that the analyte concentration is above or below a certain level. The limit of detection is usually expressed as the concentration of analyte (e.g. percentage, parts per billion, etc.) in the sample.

2.3.2 Instrumental Limit of Detection Study

Six replicate measurements of the blank solution are made and the standard deviation of the baseline noise is calculated. The standard deviation of the baseline noise is multiplied by 3 to give an estimate of the instrument signal at the limit of detection. The limit of detection is subsequently validated by the analysis of three standards which will provide peak intensities at or near the signal level calculated for the limit of detection.

2.3.3 Instrumental Limit of Detection Acceptance Criteria

The instrumental limit of detection for lead should be **0.0010 ppm (µg/mL) or below.**

2.4 Limit of Quantitation

2.4.1 Definition

Limit of quantitation is a parameter of quantitative assays for low levels of compounds in sample matrices, such as impurities in bulk drug substances and degradation products in finished pharmaceuticals. It is the lowest concentration analyte in a sample that can be determined with acceptable precision and accuracy under the stated experimental conditions. The limit of quantitation is expressed as the concentration of analyte (e.g. percent, parts per billion, etc.) in the sample.

2.4.2 Instrumental Limit of Quantitation Study

Six replicate measurements of the blank solution are made and the standard deviation of the baseline noise is calculated. The standard deviation of the baseline noise is multiplied by 10 to give an estimate of the instrument signal at the limit of quantitation. The limit of quantitation is subsequently validated by the analysis of three standards which will provide peak intensities at or near the signal calculated for the limit of quantitation.

2.4.3 Instrumental Limit of Quantitation Acceptance Criteria

The instrumental limit of quantitation for lead should be **0.003 ppm (µg/mL) or below.**

2.5 Linearity and Range

2.5.1 Definitions

1. **Linearity:** The linearity of the system is the ability to elicit test results that are directly, or by a well-defined mathematical transformation, proportional to the concentration of analyte in samples within a given range. Linearity is usually expressed in terms of the variance around the slope of the regression line calculated according to an established mathematical relationship from test results obtained by the analysis of samples with varying concentrations of analyte.

2. **Range:** The range of an analytical method is the interval between the upper and lower levels of analyte (including these levels) that have been demonstrated to be determined with precision, accuracy, and linearity using the same units as test results (e.g. percent, parts per million) obtained by the analytical method.

2.5.2 Linearity Study

Linearity check shall be performed using a minimum of eight different concentrations of a lead (Pb) standard solution and one or more internal standard solutions which will bracket the standard working range (from the limit of detection to 45.0 ppb) of the analysis. The following stock standard solutions may be used in the linearity test: Pb, Ho, Re, Sc, In, Tl, Bi and Tb. If desired, an additional linearity study may be conducted using a calcium containing solution shown to contain a lead solution concentration less than the method detection limit. A linear regression plot and equation is calculated plotting the analyte concentration against response values.

2.5.3 Linearity Acceptance Criteria

The response of the instrument is linear in the concentration range as demonstrated by a correlation coefficient (r^2) of 0.98 or better.

2.5.4 Range Data

Range is established for each of the trace lead analysis test being validated by summarizing the accuracy, Linearity, and precision data.

A result is invalid if it is above the validated range of the analytical method. Values below 0.05 µg/g should be reported as numbered estimates. An acceptable range must include all specification limits for a method and expected results which may fall outside the specification level.

2.5.5 Range Acceptance Criteria

The summarized data meets acceptance criteria defined in each section and demonstrates that samples within the concentration range of 0.05 µg/g to 3.0 µg/g (ppm) of lead can be analyzed by the analytical procedure.

Pb 3.0 ICP-MS Finished Product Sampling and Analytical Methodology

3.1 Scope

This method describes the sampling plan, procedure, data analysis, and limits to be used to analyze calcium containing dosage forms for trace lead.

Special Notes:

- All references in this protocol to the terms "purified water" or "water" shall mean ASTM Type I water.
- All glassware must be lead free and must be rinsed with 1:1 trace quality nitric acid and purified water, followed by purified water, followed by 1:1 hydrochloric acid and purified water, followed again by purified water.
- All internal standards must be prepared from the same batch and contain the same amount of internal standard reference material.
- Special precaution should be taken to avoid contamination.
- Nitric acid may be substituted for hydrochloric acid if the acceptance criteria for validation of this protocol continue to be met.
- Sample preparation shall be appropriate for the dosage form being analyzed (e.g., gums which do not lend themselves to composite sample preparation) if the acceptance criteria for validation of this protocol continue to be met.

3.2 Finished Product Sampling Plan

Special Notes:

- Sufficient sample of all products tested should be retained to permit additional testing (at least in duplicate).
- "Random selection" as used herein shall be pursuant to a scientifically and/ or regulatorily acceptable procedure.

A. For "Lot-by-Lot" compliance testing pursuant to Section 3.15, the samples used to prepare the composite shall be randomly selected from a given lot.

B. For "Product Line" compliance testing pursuant to Section 3.15, one sample shall be randomly selected from each of six different lots representative of the product to be shipped during the time period in question.

3.3 Equipment

- Inductively Coupled Plasma Mass Spectrometer
- Analytical Balance
- Class A volumetric flasks or equivalent
- Class A Pipets or equivalent
- Sample grinding equipment
- Teflon Beakers or equivalent
- Heating Apparatus: Hot plate or two stage microwave

3.4 Reagents

- Plasma Grade Lead Standards - NIST Traceable - Certified
- Purified Water
- Reference Control Sample: NIST Bone Meal 1486
- Plasma Grade Internal Standards - NIST Traceable - Certified
- Trace Analysis grade Acids (Ultrex® or equivalent): Hydrochloric and/or nitric

3.5 Preparation of Solutions

Note: Volumes may be increased proportionally

A. Blank Solution

Prepare a solution of 1% HNO_3 / 1% HCl in water to be used in diluting standards and samples.

B. Stock Internal Standard Solution

Prepare a 10 ppm internal standard solution using one or more of the standards listed in section 2.5.2.

C. Lead Stock Solution

Prepare a 1000 ppb lead stock solution by diluting reference material in 1% HNO_3 / 1% HCl.

D. Rinse Solution Containing 1:1 Trace Quality Nitric Acid and Water

Carefully add 100 mL of nitric acid to 100 mL of water.

E. Rinse Solution Containing 1:1 Trace Quality Hydrochloric Acid and Water

Carefully add 100 mL of hydrochloric acid to 100 mL of water.

3.6 Preparation of Standards

A. Zero Level Standard Solution

Prepare a zero level standard (blank) with 1% HNO_3 / 1% HCl solution and add the internal standard solution to obtain a level of 20 µl per 10 mL.

B. Standard Solutions of Lead

Prepare standard solutions in order to bracket the concentration range of the samples. Matrix match standards and samples with 1% HNO_3 / 1% HCl solution and add the internal standard to obtain a level of 20 µl per 10 mL.

3.7 Analytical Composite Sample

Weigh a minimum of 20 tablets (or equivalent) and determine the average tablet (or equivalent) weight. Grind the tablets to a fine, uniform powder. For non-tablet dosage forms, an equivalent sample shall be prepared. Proceed as directed under "Sample Preparation Procedure."

3.8. Instrument Sample Sequence

Prepare and analyze all samples in duplicate at a minimum.

3.9 Sample Preparation Procedure

A. Accurately weigh approximately 1.0 gram, or a sample size appropriate to ensure that the result is in the validated range, of the composite sample into a 250 mL teflon beaker (or equivalent).

B. Add 8 mLs of trace quality concentrated nitric acid to the beaker (enough to wet the sample).

C. Allow the carbonate (if present) reaction to dissipate and swirl to mix or dissolve.

D. Cover with a lead-free watch glass (or equivalent).

E. Heat the sample using a hotplate or other heating technique such as a microwave digestion unit under a fume hood to aid digestion of the sample and, if necessary, reflux without boiling to dryness for a minimum of 10 to 15 minutes and for an additional time period as determined by the recovery studies if necessary to completely digest the sample. If necessary to ensure complete digestion, add an additional 5 mL of trace quality concentrated nitric acid to the sample during refluxing. The need for this additional digestion must be demonstrated during the validation studies. Remove from heat.

F. If necessary to ensure digestion of organic chemicals in the products that may interfere with the analysis, a hydrogen peroxide reaction step may be added to the procedure. In this case, the product of Step E is further heated without boiling using a ribbed lead free watch glass until the solution evaporates to approximately 5 mL. A covering solution over the bottom of the beaker must be maintained. The sample is cooled and 2 mL of purified water and 3 mL of 30% hydrogen peroxide is added. The beaker is covered with a lead free watch glass and warmed with a hot plate to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Continue to add 30% hydrogen peroxide in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 10 mL hydrogen peroxide.

G. To either the solution from E of F, depending upon whether the hydrogen peroxide reaction step was incorporated, add either 3 mL of trace quality concentrated hydrochloric acid (to the solution from E) or 5 mL of trace quality concentrated hydrochloric acid (to the solution from F). If additional heating and reflux is required, add 10 mL of purified water. Replace the watch glass, and reflux without boiling to dryness. For some products, heating and reflux will not be necessary. In that case, the solution is swirled to mix and the reaction allowed to subside.

H. Cool by adding about 50 mL of purified water.

I. Bring sample to a volume of 100 mLs with purified water.

J. Particulates that might remain in the digestate should be removed by filtration (filter through Whatman No. 41 filter paper or equivalent), centrifugation (2,000 - 3,000 rpm for 10 minutes is usually sufficient) or by allowing the sample to settle.

K. Dilute sample for ICP-MS with 1% HNO_3 / 1% HCl diluent. If the sample reading is outside the linear range, dilute to bring the sample reading within the linear range, but not below the limit of quantitation.

L. Add appropriate mLs of internal standard solution to match standards in order to obtain a level of 20 µl per 10 mL of final volume.

M. For each set of samples processed, preparation blanks should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.

3.10. Reference Control Sample Procedure

A. Accurately weigh an amount of the reference material which, after dilution, is expected to yield an amount of lead comparable to the amount of lead expected in the calcium finished product sample.

B. Proceed as directed for steps B through L of Section 3.9.

3.11. Instrument Calibration

Calibrate the instrument in order to bracket the concentration range of the prepared sample solutions. Verify instrument calibration with midrange calibration checks.

3.12. Instrument Conditions

Instrument must pass manufacturer's specifications for resolution and sensitivity. Read all isotopes for lead (206, 207, 208 amu) and report total lead as the sum of all three isotopes. Read sample solution three times and average the intensities.

3.13. Quality Control During Analysis

Initial QC Checks: Include a reagent blank, midrange calibration check, second source midrange calibration check, and spike. If all data is acceptable, the run can be accepted.

Acceptance Criteria for Initial QC Checks:	Relative Limits
Midrange Calibration Check:	94% to 106%
Second Source Midrange Calibration Check	93% to 107%
Spike	80% to 120%

Running QC Checks: There should be a blank sample prep every ten samples, spike sample every ten samples, and a midrange calibration check every ten samples. If all data is acceptable, the data from the run can be reported. If not, a laboratory investigation will need to be conducted and specific corrective action put in place.

Acceptance Criteria for Running QC Checks:	Relative Limits
Midrange Calibration Check:	94% to 106%
Spike	80% to 120%

Reference Sample: For each run, analyze either a standard reference material or a previously analyzed sample.

Acceptance Criteria for Reference Sample is ± 20% of previous or certified value.

3.14 Sample Calculations

1. Micrograms of lead per gram (ppm)

$$\mu g\ Pb/g = (C \times DF)/(g\ Sample\ wt \times 1000).$$

Where:

C	=	Concentration of lead in ppb
DF	=	Dilution Factor in mL
Sample wt	=	sample weight in grams
1000	=	Factor to convert from ppb to ppm

2. Micrograms of lead per unit dose

$$\mu g\ Pb/unit\ dose = (\mu g\ Pb/g)(g\ ave.\ unit\ dose\ wt.)$$

Where:

g ave. unit dose wt.	=	Average unit dose weight in grams
µg Pb/g	=	ppm sample
µg Pb/unit dose	=	micrograms lead per unit dose

3. Micrograms of lead per gram of elemental calcium

$$\mu g\ Pb/g\ Ca = (\mu g\ Pb \text{ per unit dose})/(g\ Ca \text{ per unit dose})$$

Where:

µg Pb per unit dose	=	micrograms of lead per unit dose
Ca per unit dose	=	grams of elemental calcium per unit dose
µg Pb/g Ca	=	micrograms of lead per gram of elemental calcium

3.15 Comparison of Analytical Data to Sample Compliance Limit

The analytical data can establish that a given calcium product meets a given compliance limit for either: (a) the product line by testing multiple lots of the calcium product, or (b) for individual lots of the product line if six separate lots are not available for analysis, if the results of the analysis of six lots does not establish product line compliance for that calcium product, or if the manufacturer elects to establish compliance on a lot-by-lot rather than product line basis.

A. Product Line Compliance

Compliance is established for a product line if the results of analyzing six samples selected pursuant to Section 3.2 produces a single-tailed 90% upper confidence limit of the mean lead concentration based on the averages of the replicate analyses, using a Student's t-test or equivalent method, which does not exceed the compliance limit. If the mean does not exceed the compliance limit but the 90% confidence limit does, analysis of additional samples selected pursuant to Section 3.2 may be performed, and compliance established for that product line, if the 90% confidence limit for the entire set of samples does not exceed the compliance limit. If an unusual result (greater than 3 standard deviations from the mean of the other five lots) is obtained for a single lot, then confirmatory testing is required to verify correctness of the initial result. In the event that the unusual result is more than 3 standard deviations from the mean of the other results after confirmatory testing, the unusual result can be disregarded. The basis for such confirmatory testing is to assure that the procedure (particularly sample preparation) was followed correctly.

If product line compliance is not established as of a given point in time, the manufacturer may undertake subsequent testing to establish product line compliance. Unless and until product line compliance is established, or as an alternative to establishing product line compliance, lot-by-lot compliance may be established by the manufacturer.

B. Lot-By-Lot Compliance

An individual lot demonstrates compliance based on analysis of the composite sample selected pursuant to Section 3.2 if the average of the analytical replicates on that lot does not exceed the compliance limit, and: (a) for the first compliance phase, no individual result may exceed the compliance limit, and (b) for the second compliance phase, no individual result may exceed 120% of the compliance limit. If an individual lot does not demonstrate compliance pursuant to the immediately

preceding sentence, analysis of additional samples selected pursuant to Section 3.2 may be performed, and compliance established for that lot, if the single-tailed 90% upper confidence limit for the entire set of samples does not exceed the compliance limit. If an unusual result (greater than 3 standard deviations from the mean of the other results) is obtained for a single result, then confirmatory testing is required to verify correctness of the initial result. In the event that the unusual result is more than 3 standard deviations from the mean of the other results after confirmatory testing, the unusual result can be disregarded.

Pb 4.0 Protocol Deviation

Document any deviations from the protocol with rational, justification, cause, corrective action, and any significance.

DECLARATION OF SERVICE BY MAIL AND/OR FACSIMILE

Case Name: PEOPLE OF THE STATE OF CALIFORNIA
ex rel. DANIEL E. LUNGREN,
Attorney General of the State of California
v.
MINNESOTA MINING AND MANUFACTURING COMPANY (3M)

Case No.: San Francisco County Superior Court, Case No. 992239

I declare:

I am employed in the Office of the Attorney General, which is the office of a member of the Bar of this Court at which member's direction this service is made. I am familiar with the business practice at the Office of the Attorney General for collection and processing of correspondence for mailing with the United States Postal Service. In accordance with that practice, correspondence placed in the internal mail collection system at the Office of the Attorney General is deposited with the United States Postal Service that same day in the ordinary course of business.

On January 26, 1998, I placed the attached

NOTICE OF ENTRY OF CONSENT JUDGMENT

by a in the internal mail collection system at the Office of the Attorney General, 2101 Webster Street, 12th Floor, Oakland, California 94612-3049, for deposit in the United States Postal Service that same day in the ordinary course of business, in a sealed envelope, postage fully prepaid, addressed as follows:

B. J. Kirwan, Esq.
LATHAM & WATKINS
633 West 5th Street, Suite 4000
Los Angeles, CA 90071

I declare under penalty of perjury the foregoing is true and correct and that this declaration was executed on **January 26, 1998** at Oakland, California.

DEBRA BALDWIN

Debra Baldwin
Signature

DANIEL E. LUNGREN, Attorney General
of the State of California
RODERICK E. WALSTON
Chief Assistant Attorney General
THEODORA BERGER
Assistant Attorney General
CRAIG C. THOMPSON
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Attorneys for the People

SUPERIOR COURT OF THE STATE OF CALIFORNIA

FOR THE CITY AND COUNTY OF SAN FRANCISCO

PEOPLE OF THE STATE OF CALIFORNIA ex rel. DANIEL E. LUNGREN, Attorney General of the State of California,

Plaintiffs,

v.

MINNESOTA MINING AND MANUFACTURING COMPANY (3M),

Defendant.

No. 992239

CONSENT JUDGMENT

Plaintiff, the People of the State of California ("People") and defendant MINNESOTA MINING AND MANUFACTURING COMPANY ("3M" or "Settling Defendant") herein enter into this Consent Judgment as follows:

1. Introduction

1.1 On January 12, 1998 the People of the State of California, ex rel. Daniel E. Lungren, ("People") filed a Complaint for Civil Penalties and Injunctive Relief ("Complaint") in the Superior Court of the State of California, City and County of San Francisco, against 3M.

1.2 3M is a company that employs more than ten persons and offers for sale within the State of California, Titrilac Antacid containing calcium which is intended to be ingested by human beings (hereinafter "Antacid"). The term "calcium" as used in this Consent Judgment means elemental calcium when referring to an amount of calcium and means any form or salt of calcium when referring to calcium as an ingredient (active or inactive) in an Antacid. For purposes of this Consent Judgment the "date of shipment" shall be the date on which the Antacid first enters the stream of commerce.

1.3 The People's Complaint alleges that 3M, through the sale of Antacids to consumers in California, violated provisions of the Safe Drinking Water and Toxic Enforcement Act of 1986, Health and Safety Code sections 25249.5 et seq. ("Proposition 65"), and Business and Professions Code sections 17200 et seq. ("Unfair Competition Act"), by knowingly exposing persons to lead, a chemical known to the State of California to cause reproductive toxicity, without first providing a clear and reasonable warning to such individuals.

1.4 For purposes of this Consent Judgment only, the parties stipulate that this Court has jurisdiction over the allegations of violations contained in the Complaint and personal jurisdiction over 3M as to the acts alleged in the Complaint, that venue is proper in the City and County of San Francisco and that this Court has jurisdiction to enter this Consent Judgment.

1.5 The parties enter into this Consent Judgment as a settlement of certain disputed claims between the parties as alleged in the Complaint for the purpose of avoiding prolonged and costly litigation between the parties hereto. By execution of this Consent Judgment, 3M does not admit any facts or conclusions of law suggesting or demonstrating any violations of Proposition 65, the Unfair Competition Act or any other statutory, common law or equitable requirements relating to Antacids. Nothing in this Consent Judgment shall be construed as an admission by 3M of any fact, issue of law or violation of law, nor shall compliance with the Consent Judgment constitute or be construed as an admission by 3M of any fact, issue of law, or violation of law. Nothing in this Consent Judgment shall prejudice, waive or impair any right, remedy or defense 3M may have in this or any other or future legal proceedings. However, this paragraph shall not diminish or otherwise affect the obligations, responsibilities and duties of 3M under this Consent Judgment.

2. Injunctive Relief - Warning Program

2.1 Where required herein, clear and reasonable warning that use of Antacids exposes persons to lead, a chemical known to the State of California to cause birth defects or other reproductive harm, shall be provided by 3M in the manner provided in this Consent Judgment.

2.2 3M shall provide a warning, pursuant to paragraph 2.5, for each Antacid whose date of shipment is on or after January 1, 1998, unless 3M can show, pursuant to paragraph 2.10 and the testing protocol set forth in attached Exhibit A attached to this Consent Judgment that the Antacid causes a total daily exposure to lead of 0.5 micrograms or less, based on the amount of the Antacid supplying a thousand (1,000) milligrams of elemental calcium, excluding any naturally occurring lead in the Antacid as set forth in paragraph 2.3 below. For those Antacids where the recommended or maximum daily dose supplies more than 1500 milligrams of calcium, 3M shall provide a warning for each antacid whose date of shipment is one or after January 1, 1998 unless 3M can show, pursuant to paragraph 2.10 and the testing protocol set forth in attached Exhibit A, that the

recommended daily dose of the Antacid causes a total daily exposure to lead equal to or less than that set forth in paragraphs 2.3 and 2.4 below.

2.3 3M shall be entitled to exclude from the calculation of the daily lead exposure caused by a Antacid, the amount of lead per 1000 milligrams of calcium as set forth in Table 2.3 of this paragraph 2.3. Compliance with this Consent Judgment is established and no warning is required under Proposition 65 where the lead exposure caused by an amount of the Antacid supplying 1000 milligrams of calcium does not exceed the sum of: (a) 0.5 micrograms of lead per thousand milligrams of elemental calcium and (b) the amount of lead excluded on the date of shipment as "naturally occurring" pursuant to Table 2.3 of this paragraph 2.3. For purposes of this Consent Judgment, Table 2.3 of this paragraph 2.3 sets forth the amount of lead per 1000 milligrams of elemental calcium which shall be deemed to be "naturally occurring" at the "lowest level currently feasible" pursuant to Section 12501 of Title 22 of the California Code of Regulations ("CCR"). The amounts of lead and dates set forth in Table 2.3 shall apply as of the date of shipment of the Antacid.

TABLE 2.3

DATE	NATURALLY OCCURRING AMOUNT OF LEAD PER 1000 MILLIGRAMS OF CALCIUM
January 1, 1998	1.0 microgram

2.4 Even if no warning is required by paragraphs 2.2 and 2.3 above, in the event that the recommended daily dose of any Antacid, as specified on the label or in any other package material, exceeds 1500 milligrams of elemental calcium, 3M shall provide a warning pursuant to Proposition 65 if the total daily lead exposure from the Antacid, based on the recommended daily dose, exceeds 150% of the level that would require a warning pursuant to paragraphs 2.2 and 2.3 above (based on an amount of the Antacid supplying 1000 milligrams of calcium) as of the date of shipment of the Antacid. For Antacids that do not have a recommended daily dose, for purposes of this Consent Judgment, the term "recommended daily dose" shall mean two-thirds of the maximum daily dose as set forth on

the label or in any other package material.

2.5 For an Antacid that does not meet the standards set forth in paragraphs 2.2 through 2.4 above, 3M shall, at the point of manufacture, prior to shipment to California or prior to distribution within California, (1) affix to or print on the Antacid container, cap, label or unit package or (2) display at the point of sale of the Antacid, the following warning (the language in brackets in the warning below is optional):

> **WARNING**: This product contains [lead] a chemical known [to the State of California] to cause birth defects or other reproductive harm

2.6 The warning required by paragraph 2.5 above shall be prominently affixed to, printed on or displayed proximately to the point-of-sale of each Antacid with such conspicuousness, as compared with other words, statements, designs, or devices on the labeling as to render it likely to be read and understood by an ordinary individual under customary conditions of purchase or use. If the warning is displayed on the product container or labeling, the warning shall be at least the same size as the largest of any other health or safety warnings on the product container or labeling and the word "warning" shall be in all capital letters and in bold print. If printed on the labeling itself the warning shall be contained in the same section of the labeling that states other safety warnings concerning the use of the product. The Attorney General agrees to review any labeling or point of sale signs proposed to be used under this section and advise 3M as to whether he believes such labeling or point of sale signs comply with this section. The requirement for product labeling, set forth herein is imposed pursuant to the terms of this Consent Judgment and is recognized by the parties as not being the exclusive method of providing a warning under Proposition 65 and its implementing regulations for the Antacid.

2.7 In the event that the Attorney General determines that the naturally occurring levels set forth in Table 2.3 of paragraph 2.3 above are higher than the "lowest level currently feasible" as stated in 22 CCR section 12501(a)(4), he shall have the right to seek a modification of the Consent Judgment to reflect the alleged "lowest level currently feasible"

of naturally occurring lead in the Antacids. Prior to seeking such modification, the Attorney General shall provide written notice to 3M that he intends to seek the modification. The parties shall have ninety (90) days in which to confer with the Attorney General concerning the modification. If 3M and the Attorney General are unable to agree on a modification to the Consent Judgment, the Attorney General may file a motion with the Court, seeking a modification of the Consent Judgment. In any motion by the Attorney General seeking such a modification, the burden of producing evidence shall be initially upon the Attorney General to demonstrate a prima facie case that the modification sought by the Attorney General is the "lowest level currently feasible." If 3M does not agree to such modification, it retains the ultimate burden of proving that the modification sought by the Attorney General is lower than the "lowest level currently feasible." The parties hereby agree that the Consent Judgment should be modified to reflect any agreement of the parties or any determination by the Court concerning what is the "lowest level currently feasible" for lead in Antacids.

2.8 In the event that 3M determines that the naturally occurring levels set forth in Table 2.3 of paragraph 2.3 above are lower than the "lowest level currently feasible," as stated in 22 CCR section 12501(a)(4), 3M shall have the right to seek modification of the Consent Judgment to reflect the alleged "lowest level currently feasible." Prior to seeking such modification, 3M shall provide written notice to the Attorney General that it intends to seek the modification. The parties shall have ninety (90) days in which to confer concerning the modification. If the parties are unable to agree on a modification to the Consent Judgment, 3M may file a motion with the Court, seeking a modification of the Consent Judgment. In any motion by 3M seeking such modification, the burden of producing evidence and of proof shall be on 3M to prove that the modification sought by 3M is the "lowest level currently feasible." The parties hereby agree that the Consent Judgment should be modified to reflect any agreement of the parties or any determination by the Court concerning what is the "lowest level currently feasible" for lead in Antacids.

2.9 The term "feasible" as used in paragraphs 2.7 and 2.8 above includes, but is

not limited to, a consideration of the following factors: availability and reliability of a supply of low-lead calcium that meets the requirements set forth in paragraphs 2.2, 2.3 and 2.4 above; cost of low-lead calcium and resulting increase in manufacturers' prices resulting from the use of the low-lead calcium; performance characteristics of low-lead calcium and of the resulting Antacid, including, but not limited to formulation, performance, safety, efficacy and stability. Nothing in this Consent Judgment shall be interpreted to require 3M to use any calcium material as an ingredient in an Antacid that would render its Antacid unlawful under state or federal law as measured by existing and/or future applicable California and federal food and drug laws and regulations. Nothing in this Consent Judgment shall be interpreted to preclude 3M from advocating, for purposes of paragraphs 2.7 and/or 2.8 that any proposed modification requiring a change in the type of raw calcium source material as an ingredient in an Antacid is not feasible as defined herein. Nothing in this Consent Judgment shall be interpreted to preclude the People from advocating, for purposes of paragraphs 2.7 and/or 2.8 that any proposed modification requiring a change in the type of raw calcium source material as an ingredient in an Antacid is feasible as defined herein.

2.10 3M shall maintain records sufficient to establish its compliance with this Consent Judgment for a period of four years following the date of shipment of any Antacid into California. Such documents shall be sufficient in detail to establish compliance with the Protocol set forth in the attached Exhibit A. Upon reasonable written notice from the Attorney General's Office, 3M must produce to the Attorney General within ten (10) business days of the receipt of the Attorney General's notice, the documents required to be maintained according to this paragraph. To the extent that such documents contain information which 3M maintains is confidential, proprietary, and/or in the nature of a trade secret (or in fact a trade secret), and upon written notice as to the asserted confidential nature of this information by 3M, the Attorney General agrees not to disclose this information to third parties (though the Attorney General may disclose this information to its attorneys and employees, including professional consultants, provided that these persons also agree to

maintain the confidentiality of the information in these documents). In addition, 3M may designate as confidential "trade secret" information as that term is defined in California Government Code section 6254.7 any data provided to the Attorney General's Office pursuant to this paragraph or any other provision of this Consent Judgment or relating to the subject matter hereof and such information shall not be released to any member of the public. Provided, however, that nothing in this provision shall prohibit the Attorney General from disclosing information and/or data designated as confidential, proprietary and/or trade secret to other government agencies as is necessary in pursuit of his enforcement authority. Furthermore, nothing in this provision shall prohibit the Attorney General from applying to the Court for a ruling determining that the information and/or data designated by 3M as confidential, proprietary and/or trade secret should not be so designated and may be freely disclosed.

3. Settlement Payments

3.1 Within thirty (30) days of execution of this Consent Judgment as full, final and complete satisfaction of all claims for civil penalties or restitution for the alleged violations up through the date of execution of this Consent Judgment as set forth in paragraph 10.1, for Antacids, 3M shall pay the sum of $5,000 to the Public Health Trust, a program of the California Public Health Foundation, to be used for research, investigation and public education projects approved by the Attorney General and relating to exposure to lead in pregnancy and/or nutritional factors related to lead exposure among children. Payment shall be made by delivery of certified funds payable to the Public Health Trust. Making these payments shall not be construed as an admission by 3M of any fact, issue of law or violation of law, nor shall compliance with the Consent Judgment constitute or be construed as an admission by 3M of any fact, issue of law, or violation of law.

4. Payment of Costs and Fees

4.1 Within thirty (30) days of execution of this Consent Judgment, 3M shall pay $10,000 as reimbursement for the costs of investigating and prosecuting this action. Payment

shall be made by delivery of certified funds payable to the Attorney General of the State of California at 2101 Webster Street, 12th Floor, Oakland, California 94612-3049 (Attn: Susan S. Fiering, Deputy Attorney General).

5. Additional Enforcement Actions; Continuing Obligations

5.1. By entering into this Consent Judgment the People do not waive any right to take further enforcement actions on any violations not covered by the Complaint. Nothing in this Consent Judgment shall be construed as diminishing 3M's continuing obligation to comply with Proposition 65 or the Unfair Competition Act in its future activities.

6. Enforcement of Consent Judgment

6.1. The People may, by motion or order to show cause before the Superior Court of San Francisco, enforce the terms and conditions contained in this Consent Judgment. In any action brought by the People to enforce this Consent Judgment the People may seek whatever fines, costs, penalties or remedies as provided by law for failure to comply with the Consent Judgment. Where said failure to comply constitutes future violations of Proposition 65 or other laws, independent of the Consent Judgment and/or those alleged in the Complaint, the People are not limited to enforcement of this Consent Judgment but may seek in another action whatever fines, costs, penalties or remedies are provided by law for failure to comply with Proposition 65 or other laws. In any such future action, the standards and protocol set forth in Section 2 above, as they may be modified from time to time pursuant to paragraphs 2.7 or 2.8 shall apply. However, the rights of 3M to defend itself and its actions in law or equity shall not be abrogated or reduced in any fashion by the terms of this paragraph.

7. Application of Consent Judgment

7.1 The Consent Judgment shall apply to, be binding upon and inure to the benefit of, the parties, their divisions, subdivisions, subsidiaries, and affiliates and the successors or assigns of each of them.

8.0 Application of Testing Standard and Protocol

8.1 The testing standard and protocol set forth in Exhibit A attached to this Consent Judgment are based on determinations concerning the nature of the laboratory test used and its relationship to actual and specific conditions of Antacid use. This Consent Judgment, including, but not limited to, this standard and protocol, is the product of negotiation and compromise and is accepted by the parties, for purposes of settling, compromising and resolving issues disputed in this action, including future compliance by 3M with Section 2 of this Consent Judgment and shall not be used for any other purpose, or in any other matter and, except for the purpose of determining future compliance with this Consent Judgment shall not constitute an adoption or employment of a method of analysis for a listed chemical in a specific medium as set forth in 22 CCR section 12901(b).

9. Authority to Stipulate to Consent Judgment

9.1 Each signatory to this Consent Judgment that he or she is fully authorized by the party he or she represents to enter into this Consent Judgment on behalf of the party represented and legally to bind that party.

10. Claims Covered

10.1 This Consent Judgment is a final and binding resolution between the People and 3M of any and all alleged violations of Proposition 65, the Business and Professions Code Sections 17200 et seq. and/or the Consumers Legal Remedies Act, Civil Code section 1750 et seq. up through the date of execution of this Consent Judgment, arising from failure to warn of exposure to lead from consumption of Titrilac Antacid that was committed by 3M or by any entity within its respective chain of distribution, including, but not limited to, distributors, wholesalers and retailers of any of 3M's Titrilac Antacid. All new Antacids hereafter introduced into the stream of commerce for distribution or sale in California shall be governed by this Consent Judgment. Nothing in this Consent Judgment shall preclude 3M from establishing that any non-calcium ingredient in a calcium containing product other than Antacids, contains naturally occurring lead at the "lowest level currently feasible" pursuant to

22 CCR section 12501.

11. Modification

11.1 This Consent Judgment may be modified from time to time by express written agreement of 3M and the Attorney General with the approval of the Court or by an order of this Court.

12. Execution in Counterparts

12.1 This Consent Judgment may be executed in counterparts, which taken together shall be deemed to constitute one and the same document.

13. Entry of Consent Judgment Required

13.1 This Consent Judgment shall be null and void, and be without any force or effect, unless entered by the Court in this matter. If the Consent Judgment is not entered by the Court, the execution of this Consent Judgment by 3M shall not be construed as an admission by 3M of any fact, issue of law or violation of law.

IT IS SO STIPULATED:

Dated: 12/30, 1997

DANIEL E. LUNGREN, Attorney General of the State of California
RODERICK E. WALSTON
Chief Assistant Attorney General
THEODORA BERGER
Assistant Attorney General
CRAIG C. THOMPSON
EDWARD G. WEIL
SUSAN S. FIERING
Deputy Attorneys General

By: Susan S. Fiering
SUSAN S. FIERING
Deputy Attorney General
Attorneys for the People of the State of California

Dated:

MINNESOTA MINING AND MANUFACTURING COMPANY (3M)

By: _____

Its: Division Vice President 3M Skin Health Division

APPROVED AS TO FORM:

Dated: November 21, 1997

LATHAM & WATKINS

By: _____

BETTY-JANE KIRWAN, Esq.
Attorneys for Minnesota Mining and Manufacturing Company (3M)

IT IS SO ORDERED:

Dated: JAN 14 1998

LUCY KELLY McCABE
Presiding Judge

_____

JUDGE, SUPERIOR COURT
CITY AND COUNTY OF SAN FRANCISCO

EXHIBIT A

Calcium Containing Finished Product Lead Testing Protocol

Inductively Coupled Plasma Mass Spectrometry Procedure (ICP-MS)

Pb 1.0 Protocol Objective and Purpose

The purpose of this protocol is to define the procedures and methods used to analyze lead in calcium containing products. The protocol defines the following requirements: (1) method validation, (2) sample collection & retention, (3) analyses of samples, and (4) Limits. This lead testing protocol defines the procedures, limits and provides experimental confirmation that the data is reliable for the tested products. This protocol shall become effective for purposes of establishing compliance with lead level limits only after all challenges to its contents and validity have been resolved or waived.

The manufacturer shall be responsible for ensuring that all testing of calcium containing products, whether performed by the manufacturer's employees or by independent laboratories, is performed properly. All samples shall be obtained from either the production line or packaged product. Sufficient quantities of product shall be obtained to perform the testing in duplicate at a minimum and to maintain "retain" samples sufficient in quantity for additional investigation. Testing of a given formula of a calcium product shall be deemed to establish the lead level only for that formula of calcium product and formulas of calcium products which share all of the same ingredients (or a subset of the same ingredients but no additional ingredients) in substantially the same ratios as the tested calcium product. Test results for a lot of a calcium product showing compliance on a lot-by-lot basis shall remain valid for purposes of demonstrating compliance for that lot of the calcium product. Test results for a calcium product showing compliance on a product line basis shall remain valid for purposes of demonstrating product line compliance unless there is a material change in the product's formula, manufacturing process or ingredients. For calcium products which are to be shipped on or after July 1, 1997, the manufacturer must test such calcium products pursuant to this protocol by July 1, 1997, or as soon thereafter as is reasonably feasible. Manufacturers may rely on analytical testing which is substantially equivalent (i.e., results within 15%, validation meeting the acceptance criteria for validation of this protocol, and showing no assay bias) to this protocol to demonstrate compliance for calcium products to be shipped on or after July 1, 1997, until testing pursuant to this protocol is completed. For calcium products which are to be shipped on or after April 1, 1999, the manufacturer must test such calcium products pursuant to this protocol by April 1, 1999. In the event of disagreement between testing results produced using a method complying with this protocol and testing results produced using a method which is not substantially equivalent to this protocol, the former shall be preferred.

1.1 References

This lead testing protocol is designed to be used in combination with additional documentation included, but are not limited, to the following:

a. Instrument manuals.
b. Instrument Software manuals
c. Standard Operating Procedures
d. Calibration Standard Certifications
e. Computerized System Qualification
f. Instrument Installation Qualification
g. Instrument Operational Qualification
h. Instrument Performance Qualification
i. Analyst Training Records.
j. USP 23 Section <1225>, Validation of Compendial Methods, pp. 1982 to 1985, Category II Quantitative assays for impurities in bulk drug substances or degradation products in finished pharmaceutical products."
k. Federal Register Notice, March 1, 1995, International Conference on Harmonization (ICH), Guideline on Validation of Analytical Procedures: Definitions and Terminology.

Pb 2.0 Method Validation Requirements

As detailed in this section, the method shall be validated within any laboratory scheduled to conduct analyses of calcium containing products prior to conducting analyses intended to demonstrate compliance. Validation of the method shall be repeated when and if significant changes in the laboratory (e.g., replacement of equipment) make reliance on the prior validation inappropriate.

2.1 Accuracy

2.1.1 Definition

The accuracy of an analytical method expresses the closeness of test results obtained by that method to the true value. Accuracy may often be expressed as percent recovery by the assay of known, added amounts of analyte. Accuracy is a measure of the exactness of the analytical method.

2.1.2 Recovery Studies

The accuracy of the method should be assessed for the individual formulation tested. The recovery studies should be performed in the range of 0.05 µg/g to 3.00 µg/g (ppm) on the representative finished product sample.

A 0.5 µg/mL lead stock solution should be prepared by diluting 10 mL of 10 ppm lead standard solution with water to a total volume of 200 mL. A minimum of sixteen samples should be prepared from a composite, each having the sample weight defined in the procedure (1 gram). The first four samples are used to obtain the mean lead value (no lead addition). To the remaining samples, add appropriate volumes of the lead stock solution to cover the recovery range of 0.05 µg/g to 3.00µg/g, using a minimum of three concentrations with four samples per concentration level.

The theoretical amounts of lead in each sample is obtained by adding the average value obtained from the samples containing no spiked lead to the amount spiked in each of the three groups. The µg of lead analyzed in each sample is divided by the theoretical calculated µg of lead amount and multiplied by 100 to obtain percent recovery.

2.1.3 Accuracy Acceptance Criteria

The acceptance criteria for the spiked samples should be within **80% to 120%** recovery.

2.2 Precision & Ruggedness

2.2.1 Definitions

1. **Repeatability:** Repeatability expresses the precision under the same operation conditions over a short period of time.

2. **Intermediate Precision:** Intermediate precision expresses within-laboratory variation. Different days (inter-day precision), different analysts, different equipment, different reagents, acids, and standards, etc. (Part of a ruggedness demonstration.)

2.2.2 Precision Study (Repeatability)

Measure a prepared sample solution ten times and calculate the mean, standard deviation, and percent relative standard deviation (coefficient of variation).

2.2.3 Precision Study Acceptance Criteria

The percent relative standard deviation is less than 15%.

2.2.4 Ruggedness (Intermediate Precision) Study

Prepare a composite sample of at least 20 tablets or equivalent as defined in the method. Have two different analysts analyze six samples each from the same composite sample on different days, using different equipment (if possible), reagents, standards, and acids. Calculate the mean, standard deviation, and relative standard deviations separately for the two analysts data.

2.2.5 Ruggedness Study Acceptance Criteria

The relative standard deviations for each of the analysts are less than 25%. The mean values between the two analysts are within 25% relative.

2.3 Limit of Detection

2.3.1 Definition

The limit of detection is a parameter of limit tests. It is the lowest concentration of analyte in a sample that can be detected, but not necessarily quantitated, under the stated experimental conditions. Thus, limit tests merely substantiate that the analyte concentration is above or below a certain level. The limit of detection is usually expressed as the concentration of analyte (e.g. percentage, parts per billion, etc.) in the sample.

2.3.2 Instrumental Limit of Detection Study

Six replicate measurements of the blank solution are made and the standard deviation of the baseline noise is calculated. The standard deviation of the baseline noise is multiplied by 3 to give an estimate of the instrument signal at the limit of detection. The limit of detection is subsequently validated by the analysis of three standards which will provide peak intensities at or near the signal level calculated for the limit of detection.

2.3.3 Instrumental Limit of Detection Acceptance Criteria

The instrumental limit of detection for lead should be **0.0010 ppm (µg/mL) or below.**

2.4 Limit of Quantitation

2.4.1 Definition

Limit of quantitation is a parameter of quantitative assays for low levels of compounds in sample matrices, such as impurities in bulk drug substances and degradation products in finished pharmaceuticals. It is the lowest concentration analyte in a sample that can be determined with acceptable precision and accuracy under the stated experimental conditions. The limit of quantitation is expressed as the concentration of analyte (e.g. percent, parts per billion, etc.) in the sample.

2.4.2 Instrumental Limit of Quantitation Study

Six replicate measurements of the blank solution are made and the standard deviation of the baseline noise is calculated. The standard deviation of the baseline noise is multiplied by 10 to give an estimate of the instrument signal at the limit of quantitation. The limit of quantitation is subsequently validated by the analysis of three standards which will provide peak intensities at or near the signal calculated for the limit of quantitation.

2.4.3 Instrumental Limit of Quantitation Acceptance Criteria

The instrumental limit of quantitation for lead should be **0.003 ppm (µg/mL) or below.**

2.5 Linearity and Range

2.5.1 Definitions

1. **Linearity:** The linearity of the system is the ability to elicit test results that are directly, or by a well-defined mathematical transformation, proportional to the concentration of analyte in samples within a given range. Linearity is usually expressed in terms of the variance around the slope of the regression line calculated according to an established mathematical relationship from test results obtained by the analysis of samples with varying concentrations of analyte.

2. **Range:** The range of an analytical method is the interval between the upper and lower levels of analyte (including these levels) that have been demonstrated to be determined with precision, accuracy, and linearity using the same units as test results (e.g. percent, parts per million) obtained by the analytical method.

2.5.2 Linearity Study

Linearity check shall be performed using a minimum of eight different concentrations of a lead (Pb) standard solution and one or more internal standard solutions which will bracket the standard working range (from the limit of detection to 45.0 ppb) of the analysis. The following stock standard solutions may be used in the linearity test: Pb, Ho, Re, Sc, In, Tl, Bi and Tb. If desired, an additional linearity study may be conducted using a calcium containing solution shown to contain a lead solution concentration less than the method detection limit. A linear regression plot and equation is calculated plotting the analyte concentration against response values.

2.5.3 Linearity Acceptance Criteria

The response of the instrument is linear in the concentration range as demonstrated by a correlation coefficient (r^2) of 0.98 or better.

2.5.4 Range Data

Range is established for each of the trace lead analysis test being validated by summarizing the accuracy, Linearity, and precision data.

A result is invalid if it is above the validated range of the analytical method. Values below 0.05 µg/g should be reported as numbered estimates. An acceptable range must include all specification limits for a method and expected results which may fall outside the specification level.

2.5.5 Range Acceptance Criteria

The summarized data meets acceptance criteria defined in each section and demonstrates that samples within the concentration range of 0.05 µg/g to 3.0 µg/g (ppm) of lead can be analyzed by the analytical procedure.

Pb 3.0 ICP-MS Finished Product Sampling and Analytical Methodology

3.1 Scope

This method describes the sampling plan, procedure, data analysis, and limits to be used to analyze calcium containing dosage forms for trace lead.

Special Notes:

- All references in this protocol to the terms "purified water" or "water" shall mean ASTM Type I water.
- All glassware must be lead free and must be rinsed with 1:1 trace quality nitric acid and purified water, followed by purified water, followed by 1:1 hydrochloric acid and purified water, followed again by purified water.
- All internal standards must be prepared from the same batch and contain the same amount of internal standard reference material.
- Special precaution should be taken to avoid contamination.
- Nitric acid may be substituted for hydrochloric acid if the acceptance criteria for validation of this protocol continue to be met.
- Sample preparation shall be appropriate for the dosage form being analyzed (e.g., gums which do not lend themselves to composite sample preparation) if the acceptance criteria for validation of this protocol continue to be met.

3.2 Finished Product Sampling Plan

Special Notes:

- Sufficient sample of all products tested should be retained to permit additional testing (at least in duplicate).
- "Random selection" as used herein shall be pursuant to a scientifically and/ or regulatorily acceptable procedure.

A. For "Lot-by-Lot" compliance testing pursuant to Section 3.15, the samples used to prepare the composite shall be randomly selected from a given lot.

B. For "Product Line" compliance testing pursuant to Section 3.15, one sample shall be randomly selected from each of six different lots representative of the product to be shipped during the time period in question.

3.3 Equipment

- Inductively Coupled Plasma Mass Spectrometer
- Analytical Balance
- Class A volumetric flasks or equivalent
- Class A Pipets or equivalent
- Sample grinding equipment
- Teflon Beakers or equivalent
- Heating Apparatus: Hot plate or two stage microwave

3.4 Reagents

- Plasma Grade Lead Standards - NIST Traceable - Certified
- Purified Water
- Reference Control Sample: NIST Bone Meal 1486
- Plasma Grade Internal Standards - NIST Traceable - Certified
- Trace Analysis grade Acids (Ultrex® or equivalent): Hydrochloric and/or nitric

3.5 Preparation of Solutions

Note: Volumes may be increased proportionally

A. Blank Solution

Prepare a solution of 1% HNO_3 / 1% HCl in water to be used in diluting standards and samples.

B. Stock Internal Standard Solution

Prepare a 10 ppm internal standard solution using one or more of the standards listed in section 2.5.2.

C. Lead Stock Solution

Prepare a 1000 ppb lead stock solution by diluting reference material in 1% HNO_3 / 1% HCl.

D. Rinse Solution Containing 1:1 Trace Quality Nitric Acid and Water

Carefully add 100 mL of nitric acid to 100 mL of water.

E. Rinse Solution Containing 1:1 Trace Quality Hydrochloric Acid and Water

Carefully add 100 mL of hydrochloric acid to 100 mL of water.

3.6 Preparation of Standards

A. Zero Level Standard Solution

Prepare a zero level standard (blank) with 1% HNO_3 / 1% HCl solution and add the internal standard solution to obtain a level of 20 µl per 10 mL.

B. Standard Solutions of Lead

Prepare standard solutions in order to bracket the concentration range of the samples. Matrix match standards and samples with 1% HNO_3 / 1% HCl solution and add the internal standard to obtain a level of 20 µl per 10 mL.

3.7 Analytical Composite Sample

Weigh a minimum of 20 tablets (or equivalent) and determine the average tablet (or equivalent) weight. Grind the tablets to a fine, uniform powder. For non-tablet dosage forms, an equivalent sample shall be prepared. Proceed as directed under "Sample Preparation Procedure."

3.8. Instrument Sample Sequence

Prepare and analyze all samples in duplicate at a minimum.

3.9 Sample Preparation Procedure

A. Accurately weigh approximately 1.0 gram, or a sample size appropriate to ensure that the result is in the validated range, of the composite sample into a 250 mL teflon beaker (or equivalent).

B. Add 8 mLs of trace quality concentrated nitric acid to the beaker (enough to wet the sample).

C. Allow the carbonate (if present) reaction to dissipate and swirl to mix or dissolve.

D. Cover with a lead-free watch glass (or equivalent).

E. Heat the sample using a hotplate or other heating technique such as a microwave digestion unit under a fume hood to aid digestion of the sample and, if necessary, reflux without boiling to dryness for a minimum of 10 to 15 minutes and for an additional time period as determined by the recovery studies if necessary to completely digest the sample. If necessary to ensure complete digestion, add an additional 5 mL of trace quality concentrated nitric acid to the sample during refluxing. The need for this additional digestion must be demonstrated during the validation studies. Remove from heat.

F. If necessary to ensure digestion of organic chemicals in the products that may interfere with the analysis, a hydrogen peroxide reaction step may be added to the procedure. In this case, the product of Step E is further heated without boiling using a ribbed lead free watch glass until the solution evaporates to approximately 5 mL. A covering solution over the bottom of the beaker must be maintained. The sample is cooled and 2 mL of purified water and 3 mL of 30% hydrogen peroxide is added. The beaker is covered with a lead free watch glass and warmed with a hot plate to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Continue to add 30% hydrogen peroxide in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 10 mL hydrogen peroxide.

G. To either the solution from E of F, depending upon whether the hydrogen peroxide reaction step was incorporated, add either 3 mL of trace quality concentrated hydrochloric acid (to the solution from E) or 5 mL of trace quality concentrated hydrochloric acid (to the solution from F). If additional heating and reflux is required, add 10 mL of purified water. Replace the watch glass, and reflux without boiling to dryness. For some products, heating and reflux will not be necessary. In that case, the solution is swirled to mix and the reaction allowed to subside.

H. Cool by adding about 50 mL of purified water.

I. Bring sample to a volume of 100 mLs with purified water.

J. Particulates that might remain in the digestate should be removed by filtration (filter through Whatman No. 41 filter paper or equivalent), centrifugation (2,000 - 3,000 rpm for 10 minutes is usually sufficient) or by allowing the sample to settle.

K. Dilute sample for ICP-MS with 1% HNO_3 / 1% HCl diluent. If the sample reading is outside the linear range, dilute to bring the sample reading within the linear range, but not below the limit of quantitation.

L. Add appropriate mLs of internal standard solution to match standards in order to obtain a level of 20 µl per 10 mL of final volume.

M. For each set of samples processed, preparation blanks should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.

3.10. Reference Control Sample Procedure

A. Accurately weigh an amount of the reference material which, after dilution, is expected to yield an amount of lead comparable to the amount of lead expected in the calcium finished product sample.

B. Proceed as directed for steps B through L of Section 3.9.

3.11. Instrument Calibration

Calibrate the instrument in order to bracket the concentration range of the prepared sample solutions. Verify instrument calibration with midrange calibration checks.

3.12. Instrument Conditions

Instrument must pass manufacturer's specifications for resolution and sensitivity. Read all isotopes for lead (206, 207, 208 amu) and report total lead as the sum of all three isotopes. Read sample solution three times and average the intensities.

3.13. Quality Control During Analysis

Initial QC Checks: Include a reagent blank, midrange calibration check, second source midrange calibration check, and spike. If all data is acceptable, the run can be accepted.

Acceptance Criteria for Initial QC Checks:	Relative Limits
Midrange Calibration Check:	94% to 106%
Second Source Midrange Calibration Check	93% to 107%
Spike	80% to 120%

Running QC Checks: There should be a blank sample prep every ten samples, spike sample every ten samples, and a midrange calibration check every ten samples. If all data is acceptable, the data from the run can be reported. If not, a laboratory investigation will need to be conducted and specific corrective action put in place.

Acceptance Criteria for Running QC Checks:	Relative Limits
Midrange Calibration Check:	94% to 106%
Spike	80% to 120%

Reference Sample: For each run, analyze either a standard reference material or a previously analyzed sample.

Acceptance Criteria for Reference Sample is ± 20% of previous or certified value.

3.14 Sample Calculations

1. **Micrograms of lead per gram (ppm)**

$$\mu g\ Pb/g = (C \times DF)/(g\ Sample\ wt \times 1000).$$

Where:

C	=	Concentration of lead in ppb
DF	=	Dilution Factor in mL
Sample wt	=	sample weight in grams
1000	=	Factor to convert from ppb to ppm

2. Micrograms of lead per unit dose

$$\mu g\ Pb/unit\ dose = (\mu g\ Pb/g)(g\ ave.\ unit\ dose\ wt.)$$

Where:

g ave. unit dose wt.	=	Average unit dose weight in grams
µg Pb/g	=	ppm sample
µg Pb/unit dose	=	micrograms lead per unit dose

3. Micrograms of lead per gram of elemental calcium

$$\mu g\ Pb/g\ Ca = (\mu g\ Pb\ per\ unit\ dose)/(g\ Ca\ per\ unit\ dose)$$

Where:

µg Pb per unit dose	=	micrograms of lead per unit dose
Ca per unit dose	=	grams of elemental calcium per unit dose
µg Pb/g Ca	=	micrograms of lead per gram of elemental calcium

3.15 Comparison of Analytical Data to Sample Compliance Limit

The analytical data can establish that a given calcium product meets a given compliance limit for either: (a) the product line by testing multiple lots of the calcium product, or (b) for individual lots of the product line if six separate lots are not available for analysis, if the results of the analysis of six lots does not establish product line compliance for that calcium product, or if the manufacturer elects to establish compliance on a lot-by-lot rather than product line basis.

A. Product Line Compliance

Compliance is established for a product line if the results of analyzing six samples selected pursuant to Section 3.2 produces a single-tailed 90% upper confidence limit of the mean lead concentration based on the averages of the replicate analyses, using a Student's t-test or equivalent method, which does not exceed the compliance limit. If the mean does not exceed the compliance limit but the 90% confidence limit does, analysis of additional samples selected pursuant to Section 3.2 may be performed, and compliance established for that product line, if the 90% confidence limit for the entire set of samples does not exceed the compliance limit. If an unusual result (greater than 3 standard deviations from the mean of the other five lots) is obtained for a single lot, then confirmatory testing is required to verify correctness of the initial result. In the event that the unusual result is more than 3 standard deviations from the mean of the other results after confirmatory testing, the unusual result can be disregarded. The basis for such confirmatory testing is to assure that the procedure (particularly sample preparation) was followed correctly.

If product line compliance is not established as of a given point in time, the manufacturer may undertake subsequent testing to establish product line compliance. Unless and until product line compliance is established, or as an alternative to establishing product line compliance, lot-by-lot compliance may be established by the manufacturer.

B. Lot-By-Lot Compliance

An individual lot demonstrates compliance based on analysis of the composite sample selected pursuant to Section 3.2 if the average of the analytical replicates on that lot does not exceed the compliance limit, and: (a) for the first compliance phase, no individual result may exceed the compliance limit, and (b) for the second compliance phase, no individual result may exceed 120% of the compliance limit. If an individual lot does not demonstrate compliance pursuant to the immediately

preceding sentence, analysis of additional samples selected pursuant to Section 3.2 may be performed, and compliance established for that lot, if the single-tailed 90% upper confidence limit for the entire set of samples does not exceed the compliance limit. If an unusual result (greater than 3 standard deviations from the mean of the other results) is obtained for a single result, then confirmatory testing is required to verify correctness of the initial result. In the event that the unusual result is more than 3 standard deviations from the mean of the other results after confirmatory testing, the unusual result can be disregarded.

Pb 4.0 Protocol Deviation

Document any deviations from the protocol with rational, justification, cause, corrective action, and any significance.