



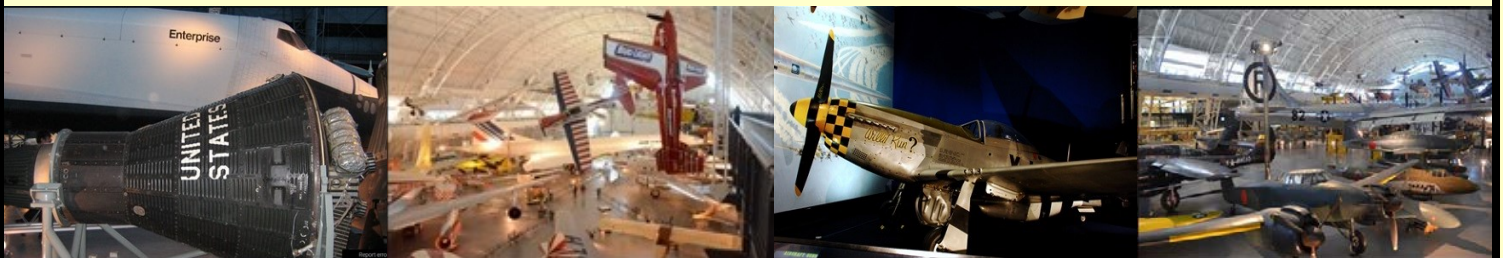
The Scientific Association Dedicated to Analytical Excellence®

AOAC INTERNATIONAL

**Stakeholder Panel on
Infant Formula and Adult Nutritionals (SPIFAN)**

**EXPERT REVIEW PANEL
(NUTRIENTS)**

Web/Teleconference



Wednesday, April 26, 2017

AOAC INTERNATIONAL
2275 Research Blvd., Suite 300
Rockville, MD, 20850
UNITED STATES
dboyd@aoac.org
301.924.7077 x126

AOAC INTERNATIONAL BYLAWS

As Amended September 26, 2010

ARTICLE I Name

The name by which this Association shall be known is "AOAC INTERNATIONAL" (hereinafter referred to as the "Association").¹

ARTICLE II Purpose

The primary purpose of the Association is to promote methods validation and quality measurements in the analytical sciences.

ARTICLE III Membership

Section 1. Types of Membership

There shall be three (3) types of membership in the Association: Individual Members, Sustaining Member Organizations, and Organizational Affiliates.

A. Individual Members

There shall be four (4) categories of Individual Members in the Association: Members, Retired Members, Student Members, and Honorary Members.

B. Sustaining Member Organizations

There shall be one (1) category of Sustaining Member Organizations.

C. Organizational Affiliate

There shall be one (1) category of Organizational Affiliate.

Section 2. Qualifications for Membership

A. Individual Members

[1] Members

Qualifications for Members shall be a degree in science, or equivalent as approved by the Board of Directors, and interest in supporting and furthering the purpose and goals of the Association. Such scientists shall be eligible for membership provided they are engaged, or have been engaged, directly or indirectly, in a field relevant to the purpose of the Association.

[2] Retired Members

¹ AOAC INTERNATIONAL was incorporated in the District of Columbia on January 20, 1932, as the Association of Official Agricultural Chemists. On November 10, 1965, the name of the corporation was changed to the Association of Official Analytical Chemists, and on September 12, 1991, the current name was adopted.

A current Member who is no longer actively engaged, directly or indirectly, in a field relevant to the purpose of the Association but who has served the Association as a Member for at least ten (10) years shall be eligible for Retired Member status upon written request and payment of the annual Retired Member dues. Any special benefits accorded Retired Members shall be determined by the Executive Director.

[3] Student Members

Any full-time student working toward an undergraduate or graduate degree in the areas of chemistry, microbiology, food science or other related science shall be eligible for Student Membership in AOAC INTERNATIONAL.

[4] Honorary Members

Honorary Members shall be persons recognized for their substantial contribution toward the achievement of the objectives of the Association. They shall be nominated by the Board of Directors and may be elected by a two-thirds vote of the Individual Members voting.

B. Sustaining Member Organizations

A Sustaining Member Organization shall be any agency of a local, state, provincial, national, or international government; a university, college, or academic department; or any firm, business, or organization with an interest in supporting and furthering the purpose of the Association. Every Sustaining Member Organization must have a designated representative(s). All such Sustaining Member Organization representatives must meet the qualifications for Members and become Individual Members with all the rights and privileges thereof.

C. Organizational Affiliate

An Organizational Affiliate Organization shall be any agency of a local, state, provincial, national, or international government; a university, college, or academic department; or any firm, business, or organization with an interest in supporting and furthering the purpose of the Association. Every Organizational Affiliate must have a designated representative(s). All such Organizational Affiliate representatives must meet the qualifications for Members and become Individual Members with all the rights and privileges thereof.

Section 3. Application for Membership

Applications or requests for membership shall be submitted to the Association's headquarters office. Membership shall become effective upon approval of the application or request, payment of any required membership dues, entry on the membership rolls, and assignment of a member number.

Section 4. Expulsion

The Board of Directors, at any duly called meeting of the Board, by a two-thirds vote of those holding office, may terminate the membership of any member who in its judgment has violated the Bylaws or has been guilty of conduct detrimental to the best interests of the Association. Any member convicted of a felony is subject to immediate expulsion from the Association. Expulsion of a member by the Board of Directors shall be final and shall cancel all rights, interest, or privileges of such member in the services or resources of the Association. Any member, for whom expulsion is proposed, for reasons other than conviction of a felony, shall be entitled to not less than 60 days advance notice of the charges, the date upon which a hearing will be scheduled, and the right to present evidence in defense. The date and place of any such hearing, if held other than at the headquarters or annual meeting site of the Association, must be reasonable with respect to the location of any individual so charged.

Section 5. Dues, Membership Year, and Waivers

- A. Annual dues for membership in the Association shall be fixed by the Board of Directors, subject to approval by the majority of the Individual Members voting by ballot by any of the following means (whichever is deemed appropriate by the Board at the time): mail, telephone call, telegram, cablegram, electronic mail or other means of electronic or telephonic transmission.
- B. Honorary Members of the Association shall be exempt from payment of dues and annual meeting registration fees.
- C. The membership year and the delinquency date shall be determined by the Board of Directors.
- D. The authority to grant waivers of membership dues rests with Executive Director.
- E. Student Member dues shall be one-third of regular Member dues, rounded up to the nearest \$5.00 increment.

Section 6. Members in Good Standing; Rights and Privileges

All Individual Members who maintain their membership by payment of dues as required under these Bylaws and who otherwise qualify shall be considered in good standing and entitled to full privileges of membership.

ARTICLE IV Officers

Section 1. Elected Officers

The elected officers of the Association shall be Individual Members and shall consist of a President, President-Elect, Secretary, Treasurer, and Immediate Past President.

A. President

The President shall be the principal elected officer of the Association, shall preside at meetings of the Association and of the Board of Directors and of the Executive Committee, and shall be a member ex-officio, with right to vote, of all committees except the Nominating Committee. He or she shall also, at the annual meeting of the Association and at such other times as he or she shall deem proper, communicate to the Association or the Board of Directors such matters and make such suggestions as may in his or her opinion tend to promote the welfare and further the purpose of the Association and shall perform such other

duties as are necessarily incident to the office of President or as may be prescribed by the Board of Directors.

B. President-Elect

In the absence of the President, or in the event of the President's inability or refusal to act, the President-Elect shall perform the duties of the President, and, when so acting, shall have all the powers of and be subject to all the restrictions upon the President. The President-Elect shall perform such other duties as from time to time may be assigned to him or her by the President or by the Board of Directors.

C. Secretary

The Secretary shall give notice of all meetings of the Association, keep a record of all proceedings, attest documents, and, in general, perform such other duties as are usual of the office of Secretary and such other duties as may be assigned by the President or by the Board of Directors.

D. Treasurer

The Treasurer shall be responsible for the funds and securities of the Association; serve as financial officer of the organization and as Chairperson of the Finance Committee; manage the Board of Director's review of and action related to the Board of Director's financial responsibilities; serve as the chief Board liaison in overseeing and reviewing the annual audit, and in general, perform such other duties as are usual of the office of Treasurer and such other duties as may be assigned by the President or by the Board of Directors.

E. Immediate Past President

The Immediate Past President shall serve as advisor to the President and Directors and perform such other duties as may be assigned from time to time by the President or by the Board of Directors.

Section 2. Appointed Officers

The appointed officers shall include the Executive Director and such other appointed officers as may be designated by the Board of Directors from time to time.

A. Executive Director

The day-to-day administration and management of the Association's offices shall be vested in a salaried manager employed or appointed by, and directly responsible to, the Board of Directors. This manager shall have the title of Executive Director with responsibility for the management and direction of all operations, programs, activities, and affairs of the Association, as approved or delegated by the Board of Directors. The Executive Director shall have direct responsibility for employment and termination of employment and the determination of compensation for staff members within the budgetary framework determined by the Board of Directors. The Executive Director functions as the chief operating officer of the Association within the guidelines established by the policies and procedures of the Board of Directors and, as necessary, with the concurrence of the President. The Executive Director shall have such other duties as may be prescribed by the Board.

B. Other Appointed Officers

Other appointed officers shall have such duties as may be prescribed by the Board.

ARTICLE V
Nominations, Elections, Terms, and Appointments to the Board of Directors

Section 1. Nominating Committee

The Nominating Committee shall annually recommend to the Board of Directors a slate of Individual Members as potential nominees for the elected positions where vacancies will occur. The Nominating Committee shall consist of five (5) members who shall be three (3) immediate Past Presidents, as available, and two (2) Individual Members-at-Large of the Association. If three Past Presidents are not available to serve, other Individual Members-at-Large shall be appointed by the President to the extent necessary to form the five (5)-member committee.

Section 2. Elections and Terms of Office

The President-Elect, the Secretary, Treasurer, and the Directors of the Board of Directors shall be elected by a majority of Individual Members voting, from a slate of nominees recommended annually by the Board of Directors.

Terms of office for all Officers and Directors shall begin with the adjournment of the annual meeting following their election and shall end with the adjournment of the annual meeting occurring nearest the expiration of their term. The six (6) Directors shall be elected to staggered three-year terms with two Directors elected to full three-year terms each year, but not to more than two (2), consecutive, three-year terms. Appointment or election to fill an unexpired term shall not affect the eligibility of a person to subsequently be elected to two (2) full terms. The Secretary shall be elected to a one-year term and may be re-elected to successive one-year terms. The Treasurer shall be elected for a one-year term and may be re-elected to successive one-year terms. The President-Elect shall be elected to a one-year term; whereupon the current President-Elect shall become President and the current President shall become the Immediate Past President, each serving a one-year term.

Section 3. Appointments

Directors-at-Large are appointed by the Board in accordance with Article VI, Section 2. Directors-at-Large are appointed for one (1) year terms, renewable at the discretion of the elected Board.

ARTICLE VI
Board of Directors

Section 1. Composition

The Board of Directors shall consist of eleven (11) elected members to include the President, President-Elect, Secretary, Treasurer, Immediate Past President, six (6) Directors, and up to three (3) appointed Directors-at-Large, all of whom shall be Individual Members of the Association. The elected Board shall reflect the makeup of the Association membership and shall not be dominated by any single interest.

Section 2. Powers and Duties

The Board of Directors shall provide supervision, control, and direction of the affairs of the Association, shall determine the Association's policies or changes therein within the limits of the Bylaws, shall actively prosecute

its purpose, and shall have discretion in the disbursement of its funds. It may adopt such rules and procedures for the conduct of its business as shall be deemed advisable, and may, in the execution of the powers granted, appoint such agents as it may consider necessary. The Board of Directors may appoint up to three (3) Directors-at-Large, if, in their opinion, such appointments advance the purpose of the Association. Directors-at-Large shall be accorded the same voting privileges as elected Directors.

Section 3. Meetings

Except that the Board shall have a regular meeting at the time and place of the annual meeting, the Board shall meet, in person or via telephone conference call, upon call of the President at such times and places as he or she may designate within the policies adopted by the Board, and shall be called to meet upon demand of a majority of its members. Notice of all meetings of the Board of Directors shall be sent by any of the following means (whichever is deemed appropriate by the President at the time): mail, telephone call, telegram, cablegram, electronic mail or other means of electronic or telephonic transmission to each member of the Board at his or her last recorded address or number at least fourteen (14) days in advance of in-person meetings or forty-eight (48) hours in advance of conference call meetings.

Section 4. Quorum

A quorum for any meeting of the Board is six (6) Board members elected in accordance with Article V (1). Any less number may: (1) set a time to adjourn, (2) adjourn, (3) recess, or (4) take measures to obtain a quorum.

Section 5. Absence

Any member of the Board of Directors unable to attend a meeting of the Board shall notify the President and state the reason for his or her absence. If a member of the Board is absent from two (2) consecutive meetings, he or she may be removed by a two-thirds vote of the Board Members then in office.

Section 6. Compensation

Members of the Board of Directors, as such, shall not receive any compensation for their services as Board members, but the Board may, by resolution under policies it may adopt, authorize reimbursement of expenses incurred in the performance of members' duties. Such authorization may prescribe conditions and procedures for approval and payment of such expenses. Nothing herein shall preclude a Board member from serving the Association in any other capacity and receiving compensation for such services, if compensation is customarily paid for such services.

Section 7. Resignation or Removal

Any member of the Board may resign at any time by giving written notice to the President, Secretary, Treasurer, or to the Board of Directors. Such resignation shall take effect at the time specified therein, or, if no time is specified, at the time of acceptance thereof as determined by the President or the Board.

Any member of the Board may be removed by a three-fourths vote of the Board members then in office and present at any regular or special meeting of the Board.

Section 8. Vacancies: Members of the Board

If a vacancy should occur in the membership of the elected Board of Directors, any Past President may be appointed by action of the remaining members of the Board to temporarily fill such vacancy until the next

regularly scheduled election. At the next regularly scheduled election nominations will be presented to fill the vacancy for the unexpired portion of the term remaining.

Section 9. Vacancies: President and Other Officers

If the office of the President shall become vacant, the President-Elect shall thereupon become President of the Association for the unexpired term, followed by his or her duly elected term. In the event the office of President becomes vacant at a time when the office of President-Elect is also vacant, the Presidency shall be filled for the remainder of the term by the action of the Board of Directors. If any other officer position shall become vacant, the office may be filled for the remainder of the term by action of the Board.

**ARTICLE VII
Committees**

Section 1. Committee Formation

The Board of Directors shall form and adopt terms of reference for such standing or special boards, committees, subcommittees, task forces, or task groups as may be required by these Bylaws or as the Board may determine necessary to carry out the affairs of the Association.

Section 2. Committee Appointments

Subject to the requirements of these Bylaws and the specific terms of reference adopted by the Board, the President shall make the appointments to fill the vacancies occurring in the Association's standing or special boards, committees, subcommittees, task forces, or task groups.

**ARTICLE VIII
Official Methods of Analysis**

The Board of Directors (BoD) is empowered to develop written policies and procedures for the study, adoption, and change in status of the Official Methods of Analysis of AOAC INTERNATIONAL. Implementation of the policies and procedures shall be delegated to an Official Methods Board (OMB).

Section 1. Composition of the Official Methods Board

The Official Methods Board shall consist of a chair and a vice chair, and members who are recommended by the chair. The chair, vice chair and members are appointed by the President of AOAC INTERNATIONAL. The OMB shall be composed of members representing a balance of government, industry, and academia as appropriate to the scope of the group and shall not be dominated by any single interest.

Section 2. Purpose of the Official Methods Board

The OMB shall serve the Association in a scientific and advisory capacity on methods and the process of their adoption. The OMB shall be responsible for implementation of procedures adopted by the BoD, according to the principles in section 3 below.

Section 3. Principles of the Official Methods Program

- A. Adequate records of technical data, discussions, and decisions on the study, adoption, and change of status of Official Methods of Analysis shall be maintained for a reasonable time.
- B. Timely notice of proposed method studies, adoption, or change in status shall be published in an Association publication that is circulated to the members.
- C. Opportunity shall be provided for materially interested parties to submit input during method study and adoption procedures and to submit comments on the adoption, use of, or change in status of specific methods.
- D. Methods submitted to the OMB for inclusion in the OMA shall be thoroughly studied, scientifically reviewed, and available in published form prior to adoption as Final Action by the OMB.
- E. The OMB shall adopt methods as Final Action.

**ARTICLE IX
Meetings**

Section 1. Annual Meeting

The annual business meeting of the Association shall be held at the time and place decided by the Board of Directors. A special meeting of the entire Association may be called by the Board of Directors; announcement thereof shall be made at least thirty (30) days prior to the time of said meeting.

Section 2. Quorum

One hundred Individual Members who are present in person or by proxy and entitled to vote shall constitute a quorum at any meeting of the Association which is duly called pursuant to the provisions of these Bylaws.

**ARTICLE X
Voting**

Section 1. Voting by Ballot

By direction of the Board of Directors, unless otherwise required by these Bylaws or conducted under alternative procedures established under these Bylaws, voting on any matter, including the election of officers and directors, the election of Honorary Members, amendment of the Bylaws, and the approval of dues, may be conducted by ballot of the voting membership by any of the following means (whichever is deemed appropriate at the time): mail, telephone call, telegram, cablegram, electronic mail or other means of electronic or telephonic transmission, and the question(s) thus presented shall be determined according to the votes received, provided in each case votes of at least five (5) percent of the voting membership shall be received. Any and all action taken in pursuance of a vote by any of the means indicated above (whichever the Board deemed appropriate at the time)

in each case shall be binding upon the Association in the same manner as would be action taken at a duly called meeting and shall become effective, unless otherwise provided for in these Bylaws or otherwise stated in the ballot, on the day following certification of the vote.

Section 2. Voting by Proxy

At any duly called meeting of Individual Members, a member-of-record, as determined thirty (30) days prior to any meeting and who is entitled to vote, may vote by proxy executed in writing by the Individual Member or his or her duly authorized attorney-in-fact. No proxy shall be valid for more than eleven (11) months after the date of its execution unless otherwise provided in the proxy.

**ARTICLE XI
Earnings and Assets**

Section 1. Non-Profit Status

A. Regardless of any provision of the Bylaws which may be construed otherwise:

[1] No part of the net earnings of the Association shall under any circumstances inure to the benefit of any member or individual.

[2] The Association shall not be operated for a private profit.

B. On lawful dissolution of the Association and after settlement of all just obligations of the Association, the Board of Directors shall distribute all remaining assets of the Association to one (1) or more organizations selected by the Board of Directors which have been held exempt from Federal Income Tax as organizations described in section 501(c)(3) of the Internal Revenue Code of 1954.

Section 2. Political Activities

A. No substantial part of the Association's activities shall consist of carrying on propaganda or otherwise attempting to influence local, state, or national legislation. All activities of the Association shall be determined by the Board of Directors.

B. The Association shall not participate or intervene in any manner in any campaign on behalf of any candidate for a political office.

**ARTICLE XII
Sections**

Section 1. Sections

The Board of Directors shall set geographic limits and grant authority to groups of Individual Members of the Association residing or working in the same geographical areas for the establishment of Sections.

Section 2. Purpose of Sections

The purpose of Sections shall be to promote and further the purpose of the Association.

Section 3. Membership in Sections

Individuals interested in the purpose of the Section shall be eligible for Section membership. Only Individual Members of the Association shall be eligible for election to the Executive Committee of the Section.

Section 4. Bylaws of Sections

Subject to approval of the Board of Directors, each Section shall adopt, for its own governance, bylaws not inconsistent with these Bylaws.

Section 5. Dissolution of Sections

When any Section shall cease to function as a Section for a period of more than one year, or if its membership shall be less than ten (10) Individual Members of the Association for a period of one (1) year, the Board of Directors may terminate the existence of such Section.

Section 6. Actions of Sections

No act of a Section or its members shall be considered an act of the Association unless expressly authorized, ratified, or affirmed by the Board of Directors.

**ARTICLE XIII
Technical Divisions**

Section 1. Purpose

Technical Divisions shall represent communities of interest within the Association which have the purpose of furthering the purpose of the Association through the development of the analytical sciences either in a commodity-based or scientific discipline-based field. Their activities shall not duplicate the organizational structure nor conflict with the policies or procedures for the adoption of official methods of analysis by the Association.

Section 2. Creation, Combination, Discontinuance, or Change

Technical Divisions may be created, existing Technical Divisions may be combined or discontinued, or the name of a Technical Division may be changed under policies and procedures adopted by the Board of Directors. Each Technical Division shall adopt bylaws not inconsistent with these Bylaws. The jurisdiction of each Technical Division shall be described in its bylaws. No act of any Technical Division or its members shall be considered an act of the Association unless expressly authorized, ratified, or affirmed by the Board of Directors.

**ARTICLE XIV
Indemnification**

The Association shall have the power to pay, by indemnity, reimbursement, or otherwise, to or for the use of any person designated by resolution of the Board of Directors who was or is a party or is threatened to be made a party to any threatened, pending, or completed action, suit, or proceeding, whether civil, criminal, administrative, or investigative (other than an action by or on behalf of the Association), by reason of the fact he or she is or was a director, officer, committee member, employee or agent of the Association, or was serving as such for another at the request of the Association, against expenses (including legal, accounting, witness and other), judgments, fines, and amounts paid in settlement so long as such person was not found by a court of competent jurisdiction to have been willfully negligent of the interests of the Association or such person had reasonable cause to believe that his or her conduct was lawful.

ARTICLE XV
Parliamentary Authority

The rules contained in the current edition of *Robert's Rules of Order Newly Revised* shall govern the Association in all cases in which they are applicable and in which they are not inconsistent with these Bylaws or any special rules of order the Association may adopt.

ARTICLE XVI
Amendments to the Bylaws

These Bylaws may be amended, repealed, or altered, in whole or in part, by a three-fourths vote: (a) of the Individual Members at any annual business or duly called special meeting of the Association, provided notice of any amendment proposed for consideration shall be sent by any of the following means (whichever may be deemed appropriate at the time): mail, telephone call, telegram, cablegram, electronic mail or other means of electronic or telephonic transmission to the last recorded address or number of each Individual Member at least thirty (30) days prior to the date of the meeting; or (b) by approval of the Individual Members through ballot sent by any means indicated above in accordance with the provisions of Article X, Voting.

All proposed amendments of these Bylaws shall be presented in writing to the Board of Directors. The Board shall present the proposals to the Association membership, with recommendations. All amendments to the Bylaws, unless otherwise stated, will become effective at the adjournment of the meeting where action is taken or on the day following the certification of a vote by mail ballot.

AOAC INTERNATIONAL
ANTITRUST POLICY
STATEMENT AND GUIDELINES

Introduction

It is the policy of AOAC INTERNATIONAL (AOAC) and its members to comply strictly with all laws applicable to AOAC activities. Because AOAC activities frequently involve cooperative undertakings and meetings where competitors may be present, it is important to emphasize the on-going commitment of our members and the Association to full compliance with national and other antitrust laws. This statement is a reminder of that commitment and should be used as a general guide for AOAC and related individual activities and meetings.

Responsibility for Antitrust Compliance

The Association's structure is fashioned and its programs are carried out in conformance with antitrust standards. However, an equal responsibility for antitrust compliance __ which includes avoidance of even an appearance of improper activity __ belongs to the individual. Even the appearance of improper activity must be avoided because the courts have taken the position that actual proof of misconduct is not required under the law. All that is required is whether misconduct can be inferred from the individual's activities.

Employers and AOAC depend on individual good judgment to avoid all discussions and activities which may involve improper subject matter and improper procedures. AOAC staff members work conscientiously to avoid subject matter or discussion which may have unintended implications, and counsel for the Association can provide guidance with regard to these matters. It is important for the individual to realize, however, that the competitive significance of a particular conduct or communication probably is evident only to the individual who is directly involved in such matters.

Antitrust Guidelines

In general, the U.S. antitrust laws seek to preserve a free, competitive economy and trade in the United States and in commerce with foreign countries. Laws in other countries have similar objectives. Competitors (including individuals) may not restrain competition among themselves with reference to the price, quality, or distribution of their products, and they may not act in concert to restrict the competitive capabilities or opportunities of competitors, suppliers, or customers.

Although the Justice Department and Federal Trade Commission generally enforce the U.S. antitrust laws, private parties can bring their own lawsuits.

Penalties for violating the U.S. and other antitrust laws are severe: corporations are subject to heavy fines and injunctive decrees, and may have to pay substantial damage judgments to injured competitors, suppliers, or customers. Individuals are subject to criminal prosecution, and will be punished by fines and imprisonment.

Under current U.S. federal sentencing guidelines, individuals found guilty of bid rigging, price fixing, or market allocation must be sent to jail for at least 4 to 10 months and must pay substantial minimum fines.

Since the individual has an important responsibility in ensuring antitrust compliance in AOAC activities, everyone should read and heed the following guidelines.

1. Don't make any effort to bring about or prevent the standardization of any method or product for the purpose or intent of preventing the manufacture or sale of any method or product not conforming to a specified standard.
2. Don't discuss with competitors your own or the competitors' prices, or anything that might affect prices such as costs, discounts, terms of sale, distribution, volume of production, profit margins, territories, or customers.
3. Don't make announcements or statements at AOAC functions, outside leased exhibit space, about your own prices or those of competitors.
4. Don't disclose to others at meetings or otherwise any competitively sensitive information.
5. Don't attempt to use the Association to restrict the economic activities of any firm or any individual.
6. Don't stay at a meeting where any such price or anti_competitive talk occurs.
7. Do conduct all AOAC business meetings in accordance with AOAC rules. These rules require that an AOAC staff member be present or available, the meeting be conducted by a knowledgeable chair, the agenda be followed, and minutes be kept.
8. Do confer with counsel before raising any topic or making any statement with competitive ramifications.
9. Do send copies of meeting minutes and all AOAC_related correspondence to the staff member involved in the activity.
10. Do alert the AOAC staff to any inaccuracies in proposed or existing methods and statements issued, or to be issued, by AOAC and to any conduct not in conformance with these guidelines.

Conclusion

Compliance with these guidelines involves not only avoidance of antitrust violations, but avoidance of any behavior which might be so construed. Bear in mind, however, that the above antitrust laws are stated in general terms, and that this statement is not a summary of applicable laws. It is intended only to highlight and emphasize the principal antitrust standards which are relevant to AOAC programs. You must, therefore, seek the guidance of either AOAC counsel or your own counsel if antitrust questions arise.

* * * * *

Adopted by the AOAC Board of Directors: September 24, 1989
Revised: March 11, 1991
Revised October 1996

AOAC INTERNATIONAL
POLICY ON THE USE OF THE
ASSOCIATION NAME, INITIALS,
IDENTIFYING INSIGNIA, LETTERHEAD, AND BUSINESS CARDS

Introduction

The following policy and guidelines for the use of the name, initials, and other identifying insignia of AOAC INTERNATIONAL have been developed in order to protect the reputation, image, legal integrity and property of the Association.

The name of the Association, as stated in its bylaws, is "AOAC INTERNATIONAL". The Association is also known by its initials, AOAC, and by its logo, illustrated below, which incorporates the Association name and a representation of a microscope, book, and flask. The AOAC logo is owned by the Association and is registered with the U.S. Patent and Trademark Office.



The full Association insignia, illustrated below, is comprised of the logo and the tagline, "The Scientific Association Dedicated to Analytical Excellence," shown below. The typeface used is Largo. The AOAC tagline is owned by the Association and is registered with the U.S. Patent and Trademark office.



The Scientific Association Dedicated to Analytical Excellence®

Policy

Policy on the use of the Association's name and logo is established by the AOAC Board of Directors as follows:

“The Board approves and encourages reference to the Association by name, either as AOAC INTERNATIONAL or as AOAC; or reference to our registered trademark, AOAC®, in appropriate settings to describe our programs, products, etc., in scientific literature and other instances so long as the reference is fair, accurate, complete and truthful and does not indicate or imply unauthorized endorsement of any kind.

The insignia (logo) of AOAC INTERNATIONAL is a registered trade and service mark and shall not be reproduced or used by any person or organization other than the Association, its elected and appointed officers, sections, or committees, without the prior written permission of the Association. Those authorized to use the AOAC INTERNATIONAL insignia shall use it only for the purposes for which permission has been specifically granted.

The name and insignia of the Association shall not be used by any person or organization in any way which indicates, tends to indicate, or implies AOAC official endorsement of any product, service, program, company, organization, event or person, endorsement of which, has not been authorized by the Association, or which suggests that membership in the Association is available to any organization.”

The Executive Director, in accordance with the above stated policy, is authorized to process, approve, fix rules, and make available materials containing the Association name and insignia.

It should be noted that neither the Association's name nor its insignia nor part of its insignia may be incorporated into any personal, company, organization, or any other stationery other than that of the Association; nor may any statement be included in the printed portion of such stationery which states or implies that an individual, company, or other organization is a Member of the Association.

Instructions

1. Reproduction or use of the Association name or insignia requires prior approval by the Executive Director or his designate.
2. Association insignia should not be altered in any manner without approval of the Executive Director or his designate, except to be enlarged or reduced in their entirety.
3. Artwork for reproducing the Association name or insignia, including those incorporating approved alterations, will be provided on request to those authorized to use them (make such requests to the AOAC Marketing Department). Examples of the types of alterations that would be approved are inclusion of a section name in or the addition of an officer's name and address to the letterhead insignia.

4. When the Association name is used without other text as a heading, it should, when possible, be set in the Largo typeface.
5. Although other colors may be used, AOAC blue, PMS 287, is the preferred color when printing the AOAC insignia, especially in formal and official documents. It is, of course, often necessary and acceptable to reproduce the insignia in black.
6. Do not print one part of the logo or insignia in one color and other parts in another color.
7. The letterhead of AOAC INTERNATIONAL shall not be used by any person or organization other than the Association, its elected and appointed officers, staff, sections, or committees; except by special permission.

Correspondence of AOAC official business should be conducted using AOAC letterhead. However, those authorized to use AOAC letterhead shall use it for official AOAC business only.

Copies of all correspondence using AOAC letterhead or conducting AOAC official business, whether on AOAC letterhead or not, must be sent to the appropriate office at AOAC headquarters.

8. AOAC INTERNATIONAL business cards shall not be used by any person or organization other than the Association, its staff, and elected officials, except by special permission.

Those authorized to use AOAC business cards shall use them for official AOAC business only and shall not represent themselves as having authority to bind the Association beyond that authorized.

Sanctions

1. Upon learning of any violation of the above policy, the Executive Director or a designate will notify the individual or organization that they are in violation of AOAC policy and will ask them to refrain from further misuse of the AOAC name or insignia.
2. If the misuse is by an Individual Member or Sustaining Member of the Association, and the misuse continues after notification, the Board of Directors will take appropriate action.
3. If continued misuse is by a nonmember of the Association or if a member continues misuse in spite of notification and Board action, ultimately, the Association will take legal action to protect its property, legal integrity, reputation, and image.

* * * * *



The Scientific Association Dedicated to Analytical Excellence®

AOAC INTERNATIONAL
POLICY AND PROCEDURES ON
VOLUNTEER CONFLICT OF INTEREST

Statement of Policy

While it is not the intention of AOAC INTERNATIONAL (AOAC) to restrict the personal, professional, or proprietary activities of AOAC members nor to preclude or restrict participation in Association affairs solely by reason of such activities, it is the sense of AOAC that conflicts of interest or even the appearance of conflicts of interest on the part of AOAC volunteers should be avoided. Where this is not possible or practical under the circumstances, there shall be written disclosure by the volunteers of actual or potential conflicts of interest in order to ensure the credibility and integrity of AOAC. Such written disclosure shall be made to any individual or group within the Association which is reviewing a recommendation which the volunteer had a part in formulating and in which the volunteer has a material interest causing an actual or potential conflict of interest.

AOAC requires disclosure of actual or potential conflicts of interest as a condition of active participation in the business of the Association. The burden of disclosure of conflicts of interest or the appearance of conflicts of interest falls upon the volunteer.

A disclosed conflict of interest will not in itself bar an AOAC member from participation in Association activities, but a three-fourths majority of the AOAC group reviewing the issue presenting the conflict must concur by secret ballot that the volunteer's continued participation is necessary and will not unreasonably jeopardize the integrity of the decision-making process.

Employees of AOAC are governed by the provision of the AOAC policy on conflict of interest by staff. If that policy is in disagreement with or mute on matters covered by this policy, the provisions of this policy shall prevail and apply to staff as well.

Illustrations of Conflicts of Interest

1. A volunteer who is serving as a committee member or referee engaged in the evaluation of a method or device; who is also an employee of or receiving a fee from the firm which is manufacturing or distributing the method or device or is an employee of or receiving a fee from a competing firm.
2. A volunteer who is requested to evaluate a proposed method or a related collaborative study in which data are presented that appear detrimental (or favorable) to a product distributed or a position supported by the volunteer's employer.
3. A referee who is conducting a study and evaluating the results of an instrument, a kit, or a piece of equipment which will be provided gratis by the manufacturer or distributor to one or more of the participating laboratories, including his or her own laboratory, at the conclusion of the study.

4. Sponsorship of a collaborative study by an interest (which may include the referee) which stands to profit from the results; such sponsorship usually involving the privilege granted by the investigator to permit the sponsor to review and comment upon the results prior to AOAC evaluation.
5. A volunteer asked to review a manuscript submitted for publication when the manuscript contains information which is critical of a proprietary or other interest of the reviewer.

The foregoing are intended as illustrative and should not be interpreted to be all-inclusive examples of conflicts of interest AOAC volunteers may find themselves involved in.

Do's and Don'ts

Do avoid the appearance as well as the fact of a conflict of interest.

Do make written disclosure of any material interest which may constitute a conflict of interest or the appearance of a conflict of interest.

Do not accept payment or gifts for services rendered as a volunteer of the Association without disclosing such payment or gifts.

Do not vote on any issue before an AOAC decision-making body where you have the appearance of or an actual conflict of interest regarding the recommendation or decision before that body.

Do not participate in an AOAC decision-making body without written disclosure of actual or potential conflicts of interest in the issues before that body.

Do not accept a position of responsibility as an AOAC volunteer, without disclosure, where the discharge of the accepted responsibility will be or may appear to be influenced by proprietary or other conflicting interests.

Procedures

Each volunteer elected or appointed to an AOAC position of responsibility shall be sent, at the time of election or appointment, a copy of this policy and shall be advised of the requirement to adhere to the provisions herein as a condition for active participation in the business of the Association. Each volunteer, at the time of his or her election or appointment, shall indicate, in writing, on a form provided for this purpose by AOAC, that he or she has read and accepts this policy.

Each year, at the spring meeting of the AOAC Board of Directors, the Executive Director shall submit a report certifying the requirements of this policy have been met; including the names and positions of any elected or appointed volunteers who have not at that time indicated in writing that they have accepted the policy.

Anyone with knowledge of specific instances in which the provisions of this policy have not been complied with shall report these instances to the Board of Directors, via the Office of the Executive Director, as soon as discovered.

* * * * *

Adopted: March 2, 1989
Revised: March 28, 1990
Revised: October 1996



AOAC INTERNATIONAL

Stakeholder Panel for Infant Formula and Adult Nutritionals (SPIFAN)

EXPERT REVIEW PANEL (ERP) – NUTRIENTS DRAFT MEETING AGENDA Web/Teleconference

Wednesday, April 26, 2017
Meeting Start Time: 10:00AM (Eastern US)

SPIFAN Nutrients Chair: Darryl Sullivan
(Covance Laboratories)

I. WELCOME & INTRODUCTION (Sullivan – 10:00AM-10:05AM)

Darryl Sullivan (Covance) will call the meeting to order. Participants will review the AOAC policy documents and draft meeting agenda.

II. REVIEW OF METHODS BY EXPERT REVIEW PANEL (ERP) FOR FINAL ACTION *OFFICIAL METHOD*SM STATUS* (ERP – 10:05AM)

The ERP will review for recommendation to the Official Methods Board (OMB) regarding Final Action *Official Method*SM status consideration.

1. **Chloride – Chair: Christopher Blake (Nestlé)**

❖ 2016.03 Chlor-02/04 Nestlé/CAIQ (Combined)

2. **Minerals & Trace Elements – Chair: Eric Poitevin (Nestlé)**

❖ 2011.14 MTE-01/03 Nestlé/FrieslandCampina (Combined)

III. NEXT STEPS/FEEDBACK FROM NUTRIENTS ERP (Sullivan)

Darryl Sullivan will discuss next steps including feedback/comments from the ERP.

Collaborative Study report: AOAC 2011.14 | ISO/CD 15151 | IDF 229 Infant formula, Adult nutrition Milk and milk products — Determination of calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium and zinc contents - Inductive coupled plasma atomic emission spectrometric method (ICP-AES)

January 2017

Hans Cruijssen, FrieslandCampina, The Netherlands
Eric Poitevin, Nestle, Switzerland

Summary

An Inductive coupled plasma atomic emission spectrometric method to determine calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium and zinc contents in Infant formula, Adult nutrition Milk and milk products using wet digestion by microwave destruction was evaluated in an international collaborative trial. The study involved 14 participants from 11 countries. The method was tested on a total of 25 products: Infant formulas, Adult nutrition products, Placebo formulas milk, milk powder, whey powder, whey protein concentrate (powder), butter and processed cheese. A certified reference material NIST 1849a was included in the study.

For most infant formula and adult nutrition products the repeatability and reproducibility are within limits set in the Minerals and trace elements SMPR (1) Acceptable reproducibility was also demonstrated with HorRat values for the method ranging from 0.4-2 with few exceptions.

With the exception of butter the dairy matrices the repeatability relative standard deviation RSD_r for the minerals (Ca, Mg, P, K, Na, Zn) and reproducibility relative standard deviation RSD_R gave acceptable results. The repeatability relative standard deviation RSD_r for the trace elements (Fe, Cu, Mn) and reproducibility relative standard deviation RSD_R were higher compared to the other minerals for the dairy samples explained by the low concentrations for these elements in the dairy products.

Table of Contents

Summary.....	1
Table of Contents.....	2
Collaborators.....	3
Study Design.....	4
Analytical Method.....	4
Method Performance	5
Safety Considerations.....	51
Comments from Collaborators	51
Conclusions.....	52
Acknowledgements	52
References.....	52

Collaborators

Eurofins LZV, Graauw, The Netherlands	Mrs. Saskia van Goethem & Mr.Hans vander Moolen
Eurofins Suzhou, China	Mrs. Fancy Li
Fonterra, Waitoa, New Zealand	Mr. Marc Connolly & Mrs. Bharathi Sadipiralla
FrieslandCampina LQS, Leeuwarden, The Netherlands	Mrs. Margriet Postma
Guangzhou Quality Supervision and Testing Institute, China	Guo Xindong & Li Shan
Laboratorio Tecnológico del Uruguay LATU, Uruguay	Mrs Q.F. Raquel Huertas & Mrs Q. F. Marina Torres
Megmilk Snow Brand Co.LTD, Japan	Mr. Totsuka Shinichi; Mr. Yoshihiro Ikeuchi & Mr. Mariko Nagatoshi
Meiji Co. LTD, Japan	Mr. Shigeki Kimura
MUVA, Germany	Mr. Ingo Piccon, Mrs. Ute Braun
Neutron S.P.A., Italy	Mrs. Andrea Rizzo & Mrs. Lorena Giuliani
Nestle, NQAC, São Paulo, Brasil	Mr. Fernando Silva and Mr. Sergio Almeida
Nestle NQAC, Dublin, USA	Mr. John Kittleson and Mr Greg Jaudzems
Nestle, NQAC Shah Alam, Malaysia	Soo Hui
Wyeth Nutrition , Askeaton, Ireland	Mr. Ger Larkin & Mrs Adrienne McMahon

Method Performance

Part 1 of the method evaluation within participating laboratories involved the calibration parameters the LOQ and a practice sample. The NIST 1849a CRM was selected for this purpose because it was readily available in most laboratories, since the expected values are known, participant could get instant feedback on the method was working appropriate. It provides additional confidence that no bias in the operation of the method amongst all participants. Two participating laboratories showed bias for the NIST 1849a and were not qualified. All other 12 laboratories provided acceptable data for the practice sample. All collaborators returned acceptable calibration parameters based on linear regression correlation coefficients or quadratic regression (r^2 0.9942-1.0000).

Laboratory 2 was not capable to measure P. Laboratories having problems with Fe, Cu, Mn, at low range in placebos and dairy products were excluded from statistical treatment (negative values). All remaining data were statistically analyses using the AOAC protocol for overall mean, intra-laboratory repeatability (Sr), repeatability relative standard deviation (RSDr), inter-laboratory reproducibility (SR), reproducibility relative standard deviation (RSDR) and HorRat. Cochran ($p=0.025$, 1 tail) and Grubbs (single and double), $p=0.025$, 2-tail) tests were utilized to determine outliers. In some instances statistical outliers were identified but deemed reasonable to do so, these were retained in the data set for calculation of precision of method.

Results of the laboratories showed no bias with the certified values from NIST (also measured with ICP-AES)

Table 2: Interlaboratory study results for NIST 1849a with certified values obtained by ICP-AES.

Element	Mean (mg/kg)	Certified value (mg/kg)		
Calcium (Ca)	5302	5253	±	51
Copper (Cu)	19.47	19.78	±	0.26
Iron (Fe)	174.0	175.6	±	2.9
Magnesium (Mg)	1634	1648	±	36
Manganese (Mn)	48.87	49.59	±	0.97
Phosphorus (P)	4009	3990	±	140
Potassium (K)	9319	9220	±	110
Sodium (Na)	4269	4265	±	83
Zinc (Zn)	152.6	151	±	5.6

Summary of the summary tables is shown in the next three tables for infant formula and adult nutrition product, for the Placebo samples and for the dairy samples.

SPIFAN sample kit

Table 3: Interlaboratory study results for infant formula and adult nutrition products obtained by ICP-AES.

Element	Range SMPR	Range concentration (mg/100g RTF)	RSDr (%)	RSDR (%)
Calcium (Ca)*	20-1280	35-99	3.9	8.7
Copper (Cu)	0.001-1.2	0.029-0.239	3.9	9.3
Iron (Fe)	0.01-20	0.54-2.4	3.6	9.5
Magnesium (Mg)	3-110	4.3-39	2.2	7.4
Manganese (Mn)	0.001-1	0.0059-0.42	3.3	12.2
Phosphorus (P)	15-800	23-95	2.6	7.5
Potassium (K)	10-2000	44-222	2.3	6.0
Sodium (Na)	10-850	15-140	2.8	7.7
Zinc (Zn)	0.1-18	0.34-2.6	3.0	7.6

* Adult RTF High Fat not included

Acceptable reproducibility was also demonstrated with HorRat values for the method ranging from 0.33- 2.05 (recommended range 0.5-2.0).

SPIFAN sample kit PLACEBOS

Table 4: Interlaboratory study results for placebos infant formula and adult nutrition products obtained by ICP-AES.

Element	Range SMPR	Range concentration (mg/100g RTF)	RSDr (%)	RSDR (%)
Calcium (Ca)	20-1280	7.4-63	2.3	7.9
Copper (Cu)	0.001-1.2	0.0036-0.011	19.1	56.4
Iron (Fe)	0.01-20	0.018-0.49	8.6	18.0
Magnesium (Mg)	3-110	1.7-5.1	1.8	10.3
Manganese (Mn)	0.001-1	0.0008-0.0083	7.7	24.7
Phosphorus (P)	15-800	24-71	1.9	5.4
Potassium (K)	10-2000	7.6-177	2.9	6.5
Sodium (Na)	10-850	5.0-138	2.7	7.9
Zinc (Zn)	0.1-18	0.0065-0.46	8.8	18.5

The placebos were not fortified with Fe, Cu, Mn and Zn. The same is found for the dairy products. The precision for these samples is poor for Fe, Cu, Mn. But this is to be expected given the Cu, Fe and Mn concentration are near or below the method quantification limit.

IDF sample kit

Table 5: Interlaboratory study results for dairy samples obtained by ICP-AES.

Element	Range concentration (mg/100g RTF)	RSDr (%)	RSDR (%)
Calcium (Ca)	105-709	2.8	5.3
Copper (Cu)	0.0058-0.12	9.4	55.0
Iron (Fe)	0.016-0.66	17.4	35.7
Magnesium (Mg)	9.2-202	2.3	7.2
Manganese (Mn)	0.0021-0.014	15.0	37.3
Phosphorus (P)	86-991	2.2	7.1
Potassium (K)	99-1868	1.6	8.8
Sodium (Na)	33-972	2.5	6.4
Zinc (Zn)	0.13-2.4	3.6	11.4
* Butter not included, because of low concentrations			

Acceptable reproducibility was also demonstrated with HorRat values for the method ranging from 0.4- 2.1 (recommended range 0.5-2.0) for most of elements, except for Cu, Fe and Mn and for the product butter.

Table 06: Reproducibility data for Infant Adult Nutritional Formula SRM1849a sample.

Sample ID: Infant Adult Nutritional Formula SRM1849a																		
Analyte Laboratory	Ca (mg/100g)		Cu (mg/100g)		Fe (mg/100g)		K (mg/100g)		Mg (mg/100g)		Mn (mg/100g)		Na (mg/100g)		P (mg/100g)		Zn (mg/100g)	
	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	520	527	1.95	1.94	17.4	17.2	937	971	161	163	5.00	4.93	424	437	414	399	16.1	15.6
2	567	583	2.23	2.12	19.0	18.6	977	1022	168	173	5.29	5.13	433	450	-	-	15.7	14.7
3	521	525	1.96	1.97	17.3	17.5	926	927	166	166	4.80	4.83	426	426	391	397	14.8	15.0
4	554	552	1.77	1.94	17.0	17.2	967	946	158	158	4.93	5.14	431	430	384	418	14.1	15.0
5	525	524	1.77	1.82	17.3	17.6	930	941	157	160	4.79	4.84	404	422	396	405	15.3	15.8
6	473 ^e	522 ^e	1.94	1.95	16.1	16.6	786 ^e	942 ^e	150	159	4.64	4.75	357 ^c	442 ^c	353	387	13.1	14.8
7	569	554	1.98	1.90	17.9	17.4	972	936	172	165	4.55	4.26	442	426	441	430	16.6	16.1
8	502	505	1.96	1.94	16.4	16.4	921	925	165	164	4.72	4.75	437	439	425	414	15.5	15.4
9	527	503	2.00	1.91	17.2	16.9	907	898	166	163	5.04	4.93	421	408	396	388	15.0	14.5
10	523	521	1.86	1.88	17.2	17.1	912	920	157	158	4.95	4.95	414	417	392	392	14.3	14.4
11	531	550	1.95	2.04	19.0	18.4	896	952	167	174	5.07	5.08	426	428	398	399	16.5	15.2
12	524	524	1.98	1.99	17.4	17.5	928	929	166	166	4.96	4.98	428	423	399	402	15.3	15.2

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 07: Reproducibility data for Adult Powder Low Fat sample.

Sample ID: Adult Powder Low Fat																		
Analyte Laboratory	Ca (mg/100g)		Cu (mg/100g)		Fe (mg/100g)		K (mg/100g)		Mg (mg/100g)		Mn (mg/100g)		Na (mg/100g)		P (mg/100g)		Zn (mg/100g)	
	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	30.0	29.8	0.062	0.061	0.558	0.562	37.9	38.8	11.7	11.6	0.142	0.142	22.1	22.6	27.0	27.2	0.673	0.677
2	35.3	35.1	0.081	0.080	0.713	0.709	45.6	45.5	13.3	13.1	0.175	0.176	24.9	24.7	-	-	0.761	0.750
3	33.6	33.7	0.071	0.071	0.648	0.653	43.6	43.9	13.2	13.3	0.159	0.159	23.6	23.9	29.3	29.2	0.731	0.738
4	37.5	36.3	0.065	0.065	0.658	0.642	47.2	45.5	13.2	12.9	0.172 ^c	0.084 ^c	25.8	25.0	29.6	29.5	0.712	0.713
5	33.0	33.4	0.065	0.072	0.545	0.574	42.9	44.4	12.5	12.5	0.149	0.150	23.4	23.4	28.4	28.6	0.723	0.737
6	33.5	33.4	0.071	0.072	0.628	0.626	44.0	44.1	12.8	12.6	0.156	0.156	23.9	23.4	29.6	29.2	0.708	0.726
7	36.4	34.9	0.067	0.063	0.646	0.618	44.6	42.9	13.3	12.7	0.141	0.141	24.8	23.9	32.5	30.9	0.795	0.757
8	32.3 ^e	39.2 ^e	0.066	0.064	0.583	0.556	43.1	41.9	13.3	12.6	0.145 ^e	0.150 ^e	23.4	22.9	31.1	29.9	0.732	0.681
9	34.3	34.0	0.074	0.073	0.522	0.506	43.4	43.1	12.7	12.6	0.160	0.159	24.6	23.9	27.0	26.7	0.728	0.717
10	34.4	34.2	0.070	0.068	0.650	0.644	44.0	43.5	12.9	12.8	0.164	0.162	24.4	24.1	29.4	29.2	0.706	0.702
11	39.2	39.0	0.084	0.088	0.806 ^c	0.988 ^c	49.0	49.7	15.8 ^d	15.8 ^d	0.205 ^b	0.204 ^b	30.8 ^d	29.5 ^d	34.7	33.8	0.949 ^d	0.929 ^d
12	33.9	33.3	0.072	0.071	0.624	0.667	43.6	44.6	13.4	13.5	0.162	0.165	25.1	24.7	30.3	31.1	0.799	0.817

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 08: Reproducibility data for Adult RTF High Fat sample.

Sample ID: Adult RTF High Fat																		
Analyte Laboratory	Ca (mg/100g)		Cu (mg/100g)		Fe (mg/100g)		K (mg/100g)		Mg (mg/100g)		Mn (mg/100g)		Na (mg/100g)		P (mg/100g)		Zn (mg/100g)	
	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	43.8	43.4	0.237	0.240	2.13	2.51	233	226	38.9	39.0	0.310	0.316	143	138	82.3	83.2	2.58	2.62
2	56.4	46.0	0.270	0.255	2.32	2.08	250 ^e	229 ^e	42.9 ^e	40.7 ^e	0.377	0.323	152	142	-	-	2.52	2.49
3	62.5	61.7	0.242	0.247	2.72	2.75	219	217	39.7	40.1	0.405	0.404	136	135	91.3	92.0	2.61	2.57
4	56.7	59.3	0.232	0.235	2.25	2.26	219	224	37.5	37.8	0.368	0.378	136	139	92.0	94.5	2.54	2.59
5	109.3 ^c	216.5 ^c	0.222	0.219	2.59	3.19	228	224	38.7	38.8	0.563 ^c	0.992 ^c	135	131	118.0 ^c	162.2 ^c	3.14 ^c	4.05 ^c
6	84.5	95.2	0.273	0.281	2.62	3.11	231	224	39.6	38.8	0.460	0.502	149	142	101.0	104.0	2.87	2.94
7	68.1	66.8	0.223	0.217	2.26	2.29	213	209	38.1	37.3	0.355	0.353	133	131	96.9	94.9	2.71	2.65
8	39.4	43.8	0.217	0.220	2.00	2.16	218	217	38.5	38.8	0.273	0.279	145	151	86.2	86.8	2.49	2.43
9	40.8	38.2	0.234	0.227	2.14	2.11	208	209	39.3	39.1	0.305	0.287	131	133	79.8	76.4	2.37	2.32
10	47.7	55.6	0.232	0.230	2.32	2.47	220	220	37.9	37.9	0.338	0.373	138	137	84.3	87.7	2.45	2.50
11	42.0	42.0	0.250	0.250	2.40	2.53	228	228	43.3 ^d	43.7 ^d	0.306	0.307	150	150	84.2	84.2	2.59	2.60
12	72.1	55.9	0.247	0.241	2.63	2.59	218	220	40.3	40.4	0.453	0.377	137	137	97.9	90.0	3.02 ^c	2.81 ^c

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 09: Reproducibility data for Adult RTF High Fat PLACEBO sample.

Sample ID: Adult RTF High Fat PLACEBO																		
Analyte Laboratory	Ca (mg/100g)		Cu (mg/100g)		Fe (mg/100g)		K (mg/100g)		Mg (mg/100g)		Mn (mg/100g)		Na (mg/100g)		P (mg/100g)		Zn (mg/100g)	
	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	14.3	14.2	0.009	0.009	0.365	0.514	181	181	2.02	2.00	0.008	0.008	138	138	73.2	71.9	0.487	0.480
2	14.1	13.9	0.014	0.015	0.437	0.496	182	181	1.51	1.50	0.020 ^b	0.021 ^b	140	140	-	-	0.455	0.463
3	14.2	14.2	0.008	0.008	0.741	0.736	178	179	2.12	2.08	0.009	0.009	139	139	69.1	69.4	0.459	0.446
4	15.2	15.5	0.008	0.007	0.474	0.362	188	193	1.99	2.08	0.008	0.008	144	148	70.5	71.0	0.419	0.427
5	12.1 ^e	13.3 ^e	0.007	0.008	2.79 ^c	0.32 ^c	179	191	1.48	1.64	0.006	0.004	133	137	68.2	70.9	0.365 ^c	0.494 ^c
6	14.4	13.9	0.012	0.014	0.410	0.416	157	168	1.89	1.86	0.008	0.008	112	128	71.3	68.7	0.457 ^e	0.519 ^e
7	15.0	14.7	0.003	0.004	0.424	0.249	181	177	2.01	1.99	0.007	0.007	140	138	73.0	71.3	0.461	0.450
8	15.2	15.4	0.009	0.014	0.290	0.305	174	171	1.79	1.64	0.009	0.011	147	138	74.9	70.8	0.447	0.447
9	14.5	14.4	0.011	0.012	0.408	0.187	166	167	0.43 ^b	0.43 ^b	0.009	0.008	129	127	70.9	68.6	0.451	0.439
10	14.5	14.3	0.008	0.007	0.442	0.467	174	172	1.96	1.92	0.009	0.009	138	137	69.4	68.6	0.423	0.416
11	15.3	14.9	0.002	0.007	0.534	0.553	178	178	2.26	2.26	0.008	0.008	149	147	72.6	71.9	0.501	0.496
12	15.0	15.5	0.008	0.011	0.581	0.592	180	176	2.71	2.70	0.011	0.011	139	146	71.4	69.6	0.720 ^b	0.851 ^b

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 10: Reproducibility data for Adult RTF High Protein sample.

Sample ID: Adult RTF High Protein																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	83.3	95.8	0.181	0.181	1.86	2.01	154	153	32.5	32.8	0.37	0.41	103	102	87.1	93.9	2.14	2.25
2	106.7	99.6	0.203	0.187	2.06	2.02	164 ^e	153 ^e	35.3 ^e	32.5 ^e	0.45	0.43	109 ^e	102 ^e	-	-	2.12	2.07
3	101.8	100.9	0.195	0.191	2.39 ^d	2.43 ^d	149	149	34.4	33.6	0.45	0.45	99	101	97.7	96.7	2.24	2.29
4	105.4	89.9	0.177	0.181	2.11	1.89	149	150	31.9	31.6	0.45	0.39	100	101	102.3	92.9	2.25	2.08
5	125.4	109.9	0.175	0.169	2.26	2.02	152	156	31.2	31.7	0.49	0.45	97	97	106.1	100.6	2.54	2.41
6	106.0	106.0	0.211	0.227	1.92	1.97	158 ^e	151 ^e	32.5	32.2	0.43	0.44	119 ^d	119 ^d	94.3	95.0	2.26	2.26
7	94.2	96.9	0.172	0.167	2.08	1.82	143	142	31.7	31.5	0.35	0.36	96	96	95.1	96.4	2.18	2.21
8	93.2	115.5	0.167	0.170	1.79	2.13	147	147	32.4	31.9	0.39	0.43	101	104	93.2	100.8	2.17	2.22
9	84.0	81.8	0.167	0.169	1.91	1.87	144	144	32.5	32.4	0.38	0.36	99	97	84.9	82.5	2.00	1.95
10	95.5	99.8	0.171	0.176	1.93	2.09	147	147	31.8	31.7	0.42	0.44	100	99	92.2	93.9	2.08	2.12
11	93.5	91.9	0.191	0.201	2.17	2.03	155	155	35.6	35.6	0.42	0.41	106	105	90.4	90.3	2.12	2.13
12	96.9	102.5	0.185	0.199	2.12	2.22	151	149	34.9	33.8	0.44	0.48	103	100	100.5	100.3	2.66	2.56

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 11: Reproducibility data for Adult RTF High Protein PLACEBO sample.

Sample ID: Adult RTF High Protein PLACEBO																		
Analyte Laboratory	Ca (mg/100g)		Cu (mg/100g)		Fe (mg/100g)		K (mg/100g)		Mg (mg/100g)		Mn (mg/100g)		Na (mg/100g)		P (mg/100g)		Zn (mg/100g)	
	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	7.40	7.35	0.010	0.010	0.444	0.433	104	100	1.77	1.75	0.006	0.006	108	106	41.7	42.4	0.312	0.315
2	8.33	8.23	0.024	0.023	0.510	0.531	104	104	1.02 ^b	0.99 ^b	0.020 ^b	0.020 ^b	110	109	-	-	0.289	0.283
3	7.22	7.30	0.009	0.01	0.590	0.595	96	97	1.79	1.78	0.007	0.007	102	102	41.4	41.3	0.294	0.298
4	7.55	7.81	0.007	0.007	0.443	0.463	98	101	1.71	1.75	0.007	0.007	102	105	43.7	42.7	0.292	0.286
5	5.79	5.67	0.016	0.016	0.278	0.409	108	103	1.42	1.43	0.003 ^b	0.004 ^b	95	95	38.9	39.1	0.249	0.245
6	7.53	7.56	0.015	0.015	0.502	0.592	96	98	1.61	1.61	0.006	0.007	94	94	41.6	42.0	0.362 ^e	0.328 ^e
7	7.68	7.59	0.006	0.005	0.507	0.526	95	96	1.76	1.76	0.005	0.006	99	99	42.0	42.2	0.294	0.307
8	9.09	9.41	0.010	0.011	0.205 ^b	0.008 ^b	93	96	1.48	1.54	0.007	0.007	105	105	41.0	42.1	0.282	0.270
9	5.05	5.59	0.005	0.007	0.214 ^c	0.531 ^c	95	95	-1.47 ^a	-1.33 ^a	-0.00 ^a	0.00 ^a	97	99	37.4	37.9	0.269	0.282
10	7.16	7.26	0.008	0.008	0.463	0.432	94	96	1.68	1.70	0.006	0.006	99	101	40.8	41.2	0.269	0.274
11	7.32	7.32	0.005	0.007	0.526	0.523	103	102	1.86	1.86	-0.00 ^a	-0.00 ^a	114	112	42.4	42.2	0.281	0.280
12	8.90	8.50	0.013	0.013	0.535	0.469	92	93	2.49 ^b	2.41 ^b	0.009 ^b	0.008 ^b	103	103	38.7 ^e	41.7 ^e	0.641 ^c	0.539 ^c

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 12: Reproducibility data for Child Milk Powder sample.

Sample ID: Child Milk Powder																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	42.0	41.8	0.097	0.097	1.045	1.041	60.9	63.0	8.23	8.24	0.148	0.147	16.5	17.1	40.5	40.4	0.679	0.674
2	51.4	58.2	0.122	0.138	1.25 ^e	1.43 ^e	74.1	84.8	9.46	10.73	0.182 ^e	0.207 ^e	18.5 ^e	21.0 ^e	-	-	0.743 ^e	0.846 ^e
3	47.4	47.5	0.113	0.113	1.240	1.230	68.4	68.7	9.52	9.54	0.167	0.166	17.9	17.9	43.9	44.0	0.730	0.734
4	49.8	49.0	0.105	0.102	1.160	1.140	70.3	69.6	8.92	8.86	0.175	0.176	18.2	18.2	45.6	43.7	0.733	0.707
5	48.5	50.0	0.111	0.112	1.177	1.186	70.9	72.6	8.98	9.08	0.160	0.162	18.0	18.5	44.4	44.8	0.771	0.761
6	38.8	46.8	0.113	0.110	1.03 ^e	1.15 ^e	53.4	66.9	8.20	9.18	0.150	0.161	13.3 ^c	18.3 ^c	35.9 ^e	43.5 ^e	0.533 ^c	0.788 ^c
7	51.5	49.9	0.107	0.105	1.211	1.179	69.8	69.6	9.77	9.29	0.147	0.144	18.4	18.3	48.9	47.4	0.787	0.773
8	57.5	60.8	0.106	0.107	1.133	1.157	68.6	70.4	9.20	9.36	0.163	0.169	17.8	18.9	43.7	44.3	0.709	0.719
9	47.9	48.0	0.116	0.111	1.068	1.027	67.2	67.1	8.47	8.43	0.164	0.159	18.2	17.9	41.1	40.1	0.717	0.699
10	48.4	48.5	0.106	0.108	1.198	1.207	68.5	69.6	9.04	9.16	0.169	0.170	17.9	18.1	44.0	44.2	0.700	0.713
11	56.5	54.4	0.136	0.120	1.61 ^b	1.49 ^b	78.5	78.2	11.35	11.36	0.211 ^b	0.210 ^b	21.7	21.0	50.7	51.4	0.926 ^b	0.938 ^b
12	47.0	47.1	0.111	0.111	1.182	1.202	70.2	70.3	9.77	9.91	0.171	0.172	18.6	18.5	47.4	47.8	0.790	0.820

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 13: Reproducibility data for Child Milk Powder PLACEBO sample.

Sample ID: Child Milk Powder PLACEBO																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	30.7	30.5	0.005	0.005	0.032 ^d	0.031 ^d	6.64	6.54	1.73	1.73	0.003	0.003	4.23	4.29	22.3	21.7	0.147	0.141
2	35.9	35.8	0.008	0.008	0.088 ^b	0.080 ^b	7.43	7.53	1.69	1.67	0.010 ^b	0.010 ^b	4.78	4.81	-	-	0.152	0.149
3	34.4	34.2	0.006	0.01	0.046	0.047	7.46	7.47	1.97	1.98	0.004	0.004	4.71	4.71	24.0	24.2	0.155	0.156
4	34.9	35.5	0.004	0.005	0.045	0.046	7.76	7.85	1.79	1.82	0.003	0.004	4.74	4.83	22.7	23.4	0.141	0.151
5	33.8	32.3	0.007	0.006	-0.06 ^a	-0.06 ^a	6.85	6.35	1.60	1.49	0.001 ^b	0.000 ^b	4.52	4.26	22.9	21.7	0.131	0.119
6	30.8	33.4	0.009	0.008	0.047	0.048	6.76	7.62	1.74	1.82	0.003	0.003	4.03 ^e	5.05 ^e	20.9	22.8	0.319 ^c	0.148 ^c
7	35.6	34.9	0.003	0.003	0.047	0.043	7.57	7.49	1.87	1.85	0.004	0.003	4.66	4.61	25.5	25.2	0.170	0.159
8	31.8	34.7	0.015 ^b	0.014 ^b	<0,1 ^a	<0,1 ^a	11.78 ^c	9.74 ^c	1.68	1.69	0.004	0.004	4.95 ^e	4.50 ^e	24.6	25.2	0.162	0.157
9	34.1	34.0	0.008	0.009	-0.10 ^a	-0.10 ^a	9.01	9.10	0.36 ^b	0.33 ^b	0.004	0.004	5.75	5.76	21.9	21.6	0.170	0.170
10	35.0	34.5	0.006	0.006	0.05	0.05	7.87	7.72	1.89	1.86	0.004	0.004	4.81	4.74	24.5	24.1	0.159	0.155
11	40.9 ^d	40.6 ^d	0.008	0.005	0.146 ^c	0.062 ^c	8.94	8.88	2.46	2.45	0.003	0.003	6.42	6.39	27.5	27.6	0.191	0.194
12	33.2	34.3	0.008	0.007	0.070 ^c	0.121 ^c	8.15	7.00	2.05	2.10	0.004 ^c	0.005 ^c	5.74	5.59	25.0	24.6	0.204	0.219

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 14: Reproducibility data for Illuma Child Powder sample.

Sample ID: Illuma Child Powder																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	33.5	32.7	0.041	0.040	0.637	0.634	48.6	48.5	4.96	4.89	0.008	0.008	18.5	18.5	21.1	20.7	0.525	0.515
2	41.8	40.9	0.051	0.053	0.900	0.809	59.3	58.7	5.58	5.46	0.018 ^b	0.018 ^b	21.1	20.8	-	-	0.579	0.595
3	37.8	37.9	0.046	0.046	0.735	0.730	55.1	55.4	5.67	5.75	0.01	0.01	20.2	20.2	22.4	22.6	0.564	0.560
4	38.9	40.0	0.044	0.044	0.694	0.710	55.6	57.4	5.27	5.43	0.009	0.010	20.2	20.8	22.9	23.2	0.546	0.554
5	36.2	37.8	0.041	0.053	0.609 ^c	0.857 ^c	54.3	56.7	4.95	5.22	0.005 ^c	0.008 ^c	18.8 ^e	19.9 ^e	20.9	22.1	0.531	0.562
6	37.6	34.0	0.046	0.047	0.708	0.665	53.8 ^e	47.2 ^e	5.33	5.04	0.009	0.008	20.1 ^e	16.8 ^e	22.7	20.3	0.542 ^e	0.463 ^e
7	39.9	38.9	0.041	0.041	0.721	0.711	55.5	55.0	5.44	5.35	0.009	0.008	20.4	20.2	24.5	23.3	0.597	0.590
8	36.2 ^c	44.7 ^c	0.044	0.044	0.663	0.645	54.9	55.1	5.40	5.30	0.010	0.010	19.5	19.5	23.4	23.4	0.548	0.530
9	37.9	37.7	0.048	0.048	0.589	0.582	54.0	53.6	4.32 ^b	4.28 ^b	0.009	0.009	20.4	20.4	19.4	19.2	0.550	0.546
10	37.9	38.1	0.044	0.044	0.725	0.729	54.2	54.7	5.34	5.39	0.009	0.009	19.8	20.0	22.4	22.5	0.536	0.542
11	44.6	43.3	0.059	0.048	0.922	0.987	62.0	60.5	6.80 ^b	6.60 ^b	0.010	0.010	25.0 ^d	25.1 ^d	26.8	25.7	0.732 ^b	0.717 ^b
12	37.3	36.8	0.045	0.045	0.708	0.718	55.9	55.5	5.87	5.81	0.010	0.010	20.8	20.3	23.3	23.7	0.619	0.589

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 15: Reproducibility data for Infant Elemental Powder sample.

Sample ID: Infant Elemental Powder																		
Analyte Laboratory	Ca (mg/100g)		Cu (mg/100g)		Fe (mg/100g)		K (mg/100g)		Mg (mg/100g)		Mn (mg/100g)		Na (mg/100g)		P (mg/100g)		Zn (mg/100g)	
	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	58.5	58.4	0.073	0.071	1.03	1.02	76.5	77.7	4.67	4.63	0.046	0.046	24.7	25.0	46.4	46.3	0.68	0.67
2	15.1 ^c	78.1 ^c	0.009 ^c	0.099 ^c	0.34 ^c	1.35 ^c	62.9 ^c	98.9 ^c	3.11 ^c	5.68 ^c	0.027 ^c	0.068 ^c	19.7 ^c	29.8 ^c	-	-	0.23 ^c	0.80 ^c
3	67.4	67.5	0.084	0.084	1.19	1.20	84.9	84.6	5.47	5.43	0.052	0.052	26.3	26.5	50.3	50.6	0.73	0.73
4	63.8 ^e	75.2 ^e	0.074	0.083	1.01 ^e	1.22 ^e	76.4 ^c	92.3 ^c	4.63 ^e	5.42 ^e	0.056	0.054	23.7 ^e	28.6 ^e	49.9	55.4	0.70	0.77
5	66.2	67.5	0.076	0.085	1.11	1.14	85.5	87.9	4.78	4.94	0.046	0.048	25.5	26.4	50.1	51.1	0.98 ^c	0.73 ^c
6	66.0	63.6	0.082	0.082	1.16	1.14	83.4	81.9	5.09	4.99	0.050	0.050	26.7	25.1	49.3	48.7	0.73	0.68
7	71.2	68.5	0.079	0.076	1.19	1.14	85.9	83.6	5.33	5.14	0.046	0.045	27.1	26.4	55.7	53.7	0.78	0.75
8	78.8	80.9	0.074	0.079	1.07	1.13	82.4	84.8	5.01	5.11	0.052	0.053	26.0	26.3	51.2	51.9	0.71	0.72
9	66.8	67.4	0.085	0.087	1.08	1.08	83.2	83.3	3.94	3.99	0.052	0.052	26.7	27.0	49.3	49.4	0.73	0.73
10	68.1	68.0	0.080	0.080	1.19	1.19	85.0	84.4	5.16	5.13	0.052	0.052	26.6	26.5	51.0	51.1	0.71	0.71
11	81.1	78.8	0.098	0.094	1.50 ^b	1.64 ^b	97.4	94.3	6.53	6.35	0.065	0.064	33.6 ^d	31.8 ^d	60.6	58.4	0.94 ^d	0.92 ^d
12	66.1	65.4	0.086	0.085	1.13	1.20	85.0	86.8	5.52	5.63	0.053	0.055	27.4	26.9	52.4	55.7	0.79	0.81

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 16: Reproducibility data for Infant Elemental Powder PLACEBO sample.

Sample ID: Infant Elemental Powder PLACEBO																		
Analyte Laboratory	Ca (mg/100g)		Cu (mg/100g)		Fe (mg/100g)		K (mg/100g)		Mg (mg/100g)		Mn (mg/100g)		Na (mg/100g)		P (mg/100g)		Zn (mg/100g)	
	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	55.4	55.0	0.000	0.001	0.025	0.024	74.4	73.1	4.73	4.67	0.003	0.003	23.2	22.9	43.1	42.3	0.003	0.004
2	67.4	68.6	0.010	0.011	0.086 ^b	0.080 ^b	95.7	97.0	5.08	5.09	0.012 ^b	0.012 ^b	28.6	29.0	-	-	-0.00 ^a	-0.00 ^a
3	62.0	62.4	0.000	0.000	0.029	0.030	84.8	85.0	5.25	5.29	0.004	0.004	26.6	26.4	47.7	47.4	0.003	0.003
4	66.5	64.2	0.000	0.000	0.029	0.027	88.8	86.0	5.15	5.05	0.004	0.004	27.8	27.1	51.7	50.9	0.011	0.005
5	60.5	61.3	0.006	0.007	-0.05 ^a	-0.05 ^a	87.5	89.1	4.74	4.82	0.002	0.002	25.6	26.0	46.6	47.0	-0.02 ^a	-0.02 ^a
6	61.5	60.7	0.003	0.006	0.033	0.031	81.4	81.0	4.99	4.92	0.004	0.004	26.0	26.5	48.8	47.7	0.032 ^c	0.061 ^c
7	59.8	64.1	-0.00 ^a	-0.00 ^a	0.023	0.024	77.8	81.4	4.74	4.95	0.004	0.003	24.6	25.8	46.7	49.1	-0.00 ^a	-0.01 ^a
8	63.4	64.7	0.004	0.006	<0,1 ^a	<0,1 ^a	87.7	90.7	5.14	5.21	0.005	0.005	26.5	27.4	48.3	48.1	0.007	0.007
9	59.9	59.2	-0.00 ^a	-0.00 ^a	-0.13 ^a	-0.13 ^a	83.6	84.0	3.58 ^b	3.57 ^b	-0.00 ^a	-0.00 ^a	26.4	26.2	44.0	44.0	-	0.001 ^a
10	61.8	65.0	-	0.00 ^a	0.029	0.029	84.7	88.7	5.09	5.31	0.00	0.00	26.7	28.0	47.2	49.8	0.012	0.010
11	76.7	75.2	-0.00 ^a	-0.00 ^a	0.01 ^a	-0.00 ^a	103.1	97.9	6.41 ^b	6.24 ^b	-0.00 ^a	-0.00 ^a	32.4	31.5	55.3	54.5	-0.02 ^a	-0.02 ^a
12	60.1	61.4	0.00	0.00	0.095 ^b	0.053 ^b	85.9	85.6	5.61	5.51	0.005 ^c	0.004 ^c	27.1	27.6	51.9	50.0	0.109 ^c	0.052 ^c

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 17: Reproducibility data for Infant PH Powder Milk sample.

Sample ID: Infant PH Powder Milk																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	40.0	40.7	0.051	0.051	0.89	0.90	61.4	64.3	3.94	4.09	0.011	0.012	15.9	16.4	22.9	23.6	0.495	0.508
2	48.1 ^c	2.5 ^c	0.066 ^c	0.006 ^c	1.02 ^c	0.05 ^c	73.4 ^c	16.6 ^c	4.11 ^a	<LOQ ^a	0.022 ^c	0.006 ^c	17.9 ^c	3.6 ^c	-	-	0.550 ^c	0.022 ^c
3	45.0	44.8	0.057	0.057	1.03	1.01	66.8	66.5	4.51	4.49	0.019 ^c	0.013 ^c	17.0	16.7	25.4	24.8	0.534	0.524
4	47.7	48.7	0.054	0.055	1.01	1.03	69.2	69.9	4.31	4.36	0.014	0.014	17.7	17.9	27.0	27.6	0.534	0.542
5	46.4	46.4	0.056	0.057	0.97	0.98	72.1	71.6	4.11	4.18	0.011	0.011	17.4	17.6	24.9	25.2	0.555	0.554
6	43.6	44.2	0.058	0.061	0.98	0.99	61.3	63.6	4.09	4.14	0.013	0.013	17.1	17.9	25.2	25.8	0.631 ^e	0.545 ^e
7	46.3	45.8	0.050	0.050	1.00	0.98	65.4	64.9	4.25	4.26	0.012	0.011	16.9	16.7	26.5	26.5	0.554	0.544
8	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
9	42.6	42.4	0.051	0.051	0.88	0.87	65.7	65.3	2.74 ^b	2.76 ^b	0.008	0.008	16.6	16.8	22.1	22.1	0.506	0.500
10	44.9	45.4	0.054	0.055	1.03	1.04	66.4	67.1	4.29	4.35	0.013	0.013	17.0	17.2	25.5	25.7	0.526	0.532
11	54.4	54.8	0.063	0.065	1.27	1.22	77.2	77.4	5.30 ^b	5.28 ^b	0.006 ^e	0.008 ^e	21.3 ^d	20.9 ^d	29.3	28.9	0.617	0.611
12	45.0	44.3	0.061	0.059	1.05	1.04	67.4	67.1	4.73	4.64	0.014	0.014	17.7	17.5	26.6	26.4	0.638 ^e	0.587 ^e

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 18: Reproducibility data for Infant PH Powder Soy sample.

Sample ID: Infant PH Powder Soy																		
Analyte Laboratory	Ca (mg/100g)		Cu (mg/100g)		Fe (mg/100g)		K (mg/100g)		Mg (mg/100g)		Mn (mg/100g)		Na (mg/100g)		P (mg/100g)		Zn (mg/100g)	
	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	61.0	60.4	0.057	0.056	1.12	1.11	70.7	73.1	5.97	5.87	0.022	0.022	24.4	25.5	38.1	38.0	0.531	0.530
2	74.2	74.8	0.071	0.071	1.36	1.37	84.1	80.9	6.68	6.65	0.035 ^b	0.034 ^b	27.5	26.8	-	-	0.602	0.595
3	67.9	68.0	0.063	0.063	1.27	1.26	77.7	77.5	6.74	6.80	0.024	0.024	26.4	26.2	40.6	40.9	0.563	0.566
4	72.7 ^e	62.8 ^e	0.064	0.058	1.22	1.14	77.9 ^e	70.2 ^e	6.99 ^c	5.61 ^c	0.026	0.026	26.4	23.4	41.2	41.4	0.566	0.550
5	67.3	69.1	0.056	0.066	1.16	1.24	79.5	82.7	6.09	6.32	0.018 ^c	0.022 ^c	24.8	26.1	39.1	41.0	0.543	0.575
6	68.4	66.9	0.064	0.063	1.25	1.23	77.8	75.7	6.50	6.46	0.024	0.024	27.1	25.6	41.6	40.9	0.562	0.544
7	70.5	72.4	0.056	0.058	1.23	1.28	76.8	79.3	6.44	6.63	0.022	0.022	26.2	27.0	42.6	43.6	0.575	0.595
8	67.0 ^c	81.7 ^c	0.062	0.055	1.18	1.15	77.1	77.1	6.46	6.22	0.024	0.025	25.3	25.4	39.8	40.7	0.549	0.538
9	68.6	68.1	0.065	0.064	1.13	1.11	76.8	75.9	5.49	5.53	0.024	0.023	26.9	26.2	37.9	37.3	0.556	0.549
10	69.5	69.3	0.060	0.060	1.27	1.27	78.3	78.2	6.50	6.48	0.024	0.024	26.5	26.5	40.8	40.6	0.552	0.544
11	82.1	81.6	0.067	0.078	1.60 ^b	1.64 ^b	87.8	87.1	8.07	8.08	0.029	0.030	32.3 ^d	31.9 ^d	47.9	48.3	0.718 ^d	0.725 ^d
12	65.9	66.8	0.064	0.065	1.35	1.25	78.2	79.0	6.92	6.98	0.027	0.025	26.6	27.1	43.5	43.2	0.671 ^d	0.622 ^d

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 19: Reproducibility data for Infant Powder fos_gos sample.

Sample ID: Infant Powder fos_gos																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	32.1	32.1	0.043	0.041	0.499	0.476	43.3	42.7	4.21	4.17	0.011	0.011	14.6	14.5	21.4	21.2	0.581	0.549
2	38.7	38.3	0.055	0.055	0.629	0.616	50.7	49.9	4.55	4.58	0.022 ^c	0.029 ^c	16.2	16.0	-	-	0.653	0.638
3	36.5	36.2	0.054	0.051	0.627	0.575	47.9	47.5	4.81	4.75	0.013	0.01	15.7	15.6	23.3	23.2	0.674	0.627
4	35.8	38.0	0.042	0.046	0.504	0.532	46.2	49.0	4.22 ^e	4.67 ^e	0.012	0.013	15.1	16.0	22.8	23.7	0.570	0.600
5	35.0	35.5	0.041	0.044	0.432	0.448	49.3	47.2	4.14	4.21	0.008	0.009	14.7	14.9	21.6	22.3	0.594	0.596
6	36.2	35.9	0.050	0.048	0.538	0.535	46.6	45.9	4.49	4.47	0.012	0.012	16.1	15.5	23.4	23.4	0.592	0.581
7	38.4	38.4	0.043	0.043	0.556	0.560	48.5	48.6	4.60	4.61	0.011	0.011	15.9	16.0	25.3	25.3	0.658	0.657
8	34.9	35.7	0.046	0.045	0.488	0.484	48.2	48.3	4.50	4.51	0.013	0.013	15.4	15.4	24.3	24.5	0.606	0.605
9	33.7	36.3	0.045	0.049	0.411	0.412	47.0	47.3	3.00 ^b	3.40 ^b	0.007 ^c	0.012 ^c	15.1	15.9	20.1	20.2	0.587	0.602
10	37.1	37.1	0.047	0.046	0.563	0.560	48.3	47.9	4.60	4.57	0.012	0.012	15.8	15.7	23.5	23.5	0.594	0.589
11	42.7	43.7	0.049	0.055	0.698	0.699	54.3	55.2	5.73 ^b	5.84 ^b	0.014	0.014	18.7 ^d	19.9 ^d	27.2	27.8	0.790 ^b	0.794 ^b
12	35.7	36.3	0.049	0.047	0.606	0.590	49.8	49.9	4.99	5.07	0.014	0.013	16.0	16.6	25.1	25.0	0.684 ^b	0.737 ^b

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 20: Reproducibility data for Infant Powder Milk sample.

Sample ID: Infant Powder Milk																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	50.9	51.0	0.052	0.055	1.10	1.13	70.0	69.4	4.57	4.66	0.005	0.005	13.8	14.2	29.0	30.5	0.503	0.534
2	59.8	64.8	0.069	0.070	1.34	1.40	81.8	84.1	5.01	5.27	0.015 ^b	0.015 ^b	15.8	16.2	-	-	0.591	0.602
3	57.2	57.4	0.061	0.061	1.27	1.28	75.7	75.4	5.27	5.24	0.006	0.006	15.3	15.2	32.3	32.4	0.575	0.568
4	61.2	59.1	0.053	0.055	1.26	1.25	78.3	78.5	5.06	5.04	0.006	0.006	15.8	15.7	30.7	32.0	0.522	0.533
5	57.4	54.2	0.056	0.053	1.20	1.16	75.9	73.9	4.71	4.52	0.003	0.002	14.6	14.1	31.6	30.5	0.558	0.531
6	57.3	49.4	0.061	0.063	1.25	1.14	74.0 ^e	61.5 ^e	4.96	4.58	0.006	0.005	16.1 ^c	12.6 ^c	32.6 ^e	28.0 ^e	0.555	0.456
7	59.0	60.7	0.055	0.056	1.28	1.25	75.4	76.3	5.04	5.06	0.005	0.005	15.2	15.4	34.3	34.4	0.587	0.599
8	56.3	57.6	0.062	0.060	1.23	1.22	75.5	76.4	5.11	5.04	0.008	0.008	14.9	15.0	32.6	32.2	0.569	0.560
9	55.1	57.4	0.053 ^c	0.063 ^c	1.11	1.17	74.2	74.3	3.59 ^b	3.92 ^b	-0.00 ^a	0.006 ^a	14.0 ^e	15.7 ^e	28.2	30.0	0.503	0.560
10	58.9	58.5	0.060	0.060	1.32	1.30	76.4	75.6	5.08	5.03	0.006	0.006	15.3	15.2	33.4	32.9	0.563	0.552
11	67.3	68.2	0.077	0.079	1.59 ^b	1.63 ^b	84.5	85.6	6.16 ^b	6.33 ^b	0.006	0.006	19.2 ^d	19.3 ^d	37.1	38.3	0.703 ^b	0.730 ^b
12	55.3	56.8	0.059	0.064	1.39	1.26	75.9	76.9	5.41	5.46	0.008	0.007	15.6	15.9	34.6	34.0	0.707 ^b	0.644 ^b

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 21: Reproducibility data for Infant Powder Soy sample.

Sample ID: Infant Powder Soy																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	64.2	64.5	0.061	0.059	1.14	1.11	75.0	74.3	7.20	7.15	0.031	0.031	23.3	22.1	45.8	43.3	0.779	0.747
2	75.4	74.3	0.076	0.072	1.35	1.36	87.5	84.5	8.10	8.08	0.044 ^b	0.043 ^b	25.5	25.0	-	-	0.851	0.824
3	73.2	72.7	0.068	0.068	1.29	1.29	80.7	81.0	8.28	8.18	0.035	0.035	24.5	24.1	48.8	48.9	0.835	0.828
4	84.3 ^e	73.3 ^e	0.073	0.062	1.43	1.26	95.2 ^e	85.4 ^e	8.95 ^e	7.92 ^e	0.081 ^c	0.036 ^c	28.6	25.8	56.0	47.4	0.932	0.800
5	73.0	69.6	0.070	0.059	1.27	1.14	84.2	79.2	7.77	7.41	0.032 ^e	0.029 ^e	25.0	23.2	49.2	45.7	0.867	0.791
6	70.5	67.4	0.070	0.072	1.24	1.21	78.7	76.1	7.94	7.72	0.034	0.033	25.4	22.1	47.9	46.0	0.805	0.760
7	75.5	79.6	0.064	0.066	1.31	1.33	82.2	84.1	8.05	8.28	0.033	0.033	25.1	25.7	51.6	54.6	0.894	0.903
8	85.2	80.1	0.065	0.062	1.22	1.18	81.5	82.0	7.96	7.86	0.036	0.035	24.4	23.9	48.7	48.7	0.810	0.808
9	73.3	71.7	0.071	0.071	1.18	1.19	79.7	79.4	7.09	7.10	0.035	0.036	25.0	24.7	47.4	46.9	0.831	0.836
10	74.4	74.6	0.066	0.066	1.31	1.29	82.3	81.8	8.02	7.97	0.035	0.035	25.0	24.8	49.4	49.2	0.816	0.808
11	82.2	83.9	0.075	0.079	1.76 ^b	1.64 ^b	92.3	93.7	9.75 ^d	9.95 ^d	0.042 ^b	0.043 ^b	29.4 ^d	30.6 ^d	54.4	57.8	1.046 ^d	1.082 ^d
12	71.5	71.8	0.071	0.071	1.25	1.28	81.3	82.9	8.34	8.54	0.036	0.037	24.9	25.6	50.8	52.4	0.867	0.934

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 22: Reproducibility data for Infant RTF Milk sample.

Sample ID: Infant RTF Milk																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	55.8	53.3	0.047	0.048	1.34	1.35	79.4	78.2	6.36	6.34	0.012	0.012	17.9	17.4	29.8	30.1	0.739	0.737
2	55.4	44.3	0.064 ^b	0.064 ^b	1.47	1.49	92.3	92.3	6.06	6.08	0.027 ^b	0.027 ^b	19.0	19.2	-	-	0.692	0.692
3	50.0	50.4	0.045	0.046	1.39	1.40	80.9	80.7	6.37	6.37	0.012	0.012	17.7	17.9	28.7	29.1	0.712	0.729
4	53.9	48.5	0.046	0.045	1.31	1.34	80.5	81.7	6.17	5.98	0.012	0.012	17.8	18.1	30.9	30.8	0.724	0.706
5	48.0	47.3	0.050	0.048	1.26	1.27	85.2	85.0	5.71	5.76	0.011 ^c	0.009 ^c	17.5	17.7	27.6	27.7	0.713	0.715
6	50.3	41.5	0.053	0.055	1.33	1.31	75.6	74.1	5.99	5.91	0.012	0.011	20.1	19.4	29.8	28.4	0.743	0.700
7	43.7	39.6	0.040	0.039	1.29	1.29	77.6	77.5	5.96	6.02	0.010	0.010	17.2	17.2	29.7	29.3	0.722	0.697
8	52.1	53.8	0.045	0.046	1.32	1.33	80.0	81.3	6.15	6.21	0.014	0.014	17.2	17.6	30.6	30.9	0.698	0.705
9	49.0	47.6	0.040	0.040	1.13	1.13	79.6	79.0	3.57 ^b	3.56 ^b	0.004 ^b	0.004 ^b	17.0	17.1	24.6	24.5	0.666	0.662
10	37.3	36.8	0.043	0.043	1.44 ^e	1.33 ^e	79.2	78.3	6.01	5.95	0.011	0.010	17.6	17.4	27.7	27.8	0.651	0.649
11	57.5	58.6	0.046 ^e	0.043 ^e	1.51	1.47	86.7	84.3	6.96	6.75	0.004 ^b	0.003 ^b	18.3	18.5	30.7	30.0	0.739	0.733
12	43.6	55.9	0.049	0.048	1.47	1.51	81.2	83.2	6.83 ^e	7.60 ^e	0.014	0.015	21.3 ^e	24.4 ^e	29.6 ^e	32.0 ^e	0.895 ^c	1.206 ^c

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 23: Reproducibility data for Infant RTF PLACEBO sample.

Sample ID: Infant RTF PLACEBO																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	29.7	29.7	0.002	0.001	0.012 ^a	-0.00 ^a	61.0	60.4	3.48	3.49	0.000	0.000	12.3	12.2	26.3	26.1	0.093	0.092
2	31.3	30.5	0.005	0.005	0.086 ^b	0.079 ^b	64.9	63.8	3.10	3.03	0.012 ^b	0.012 ^b	12.3	11.9	-	-	0.100	0.092
3	30.3	30.2	0.002	0.002	0.018	0.019	62.0	61.9	3.53	3.57	0.001	0.001	11.9	12.0	25.0	25.1	0.092	0.092
4	31.2	31.6	0.001	0.000	0.019	0.018	63.2	63.5	3.32	3.35	0.001	0.001	12.3	12.4	24.2	24.1	0.082	0.081
5	29.4 ^e	26.9 ^e	0.001	0.002	-0.16 ^a	-0.16 ^a	66.0	57.6	2.98	2.74	-0.00 ^a	-0.00 ^a	11.5 ^e	10.1 ^e	23.4	22.1	0.021 ^b	0.026 ^b
6	29.9	29.2	0.006	0.004	0.019	0.024	57.4	55.9	3.28	3.22	0.001	0.001	13.6	13.0	25.1	24.5	0.113	0.103
7	31.0	31.6	-0.00 ^a	-0.00 ^a	0.010 ^c	0.061 ^c	61.4	62.2	3.39	3.45	0.001	0.001	11.8	12.0	27.0	27.3	0.090 ^c	0.109 ^c
8	30.6	31.1	0.010	0.007	<0,1 ^a	<0,1 ^a	60.7	58.9	3.28	3.29	0.002	0.002	11.7	11.2	26.5	26.9	0.089	0.091
9	30.0	30.0	0.005	0.005	-0.13 ^a	-0.12 ^a	59.9	60.0	2.03 ^b	2.05 ^b	0.001 ^c	0.002 ^c	12.5	12.7	23.7	23.2	0.105	0.107
10	29.3	29.5	0.002	0.002	0.020	0.016	61.2	61.6	3.34	3.38	0.001	0.001	11.7	11.6	25.1	25.0	0.088	0.089
11	31.3	31.2	0.002 ^a	-0.00 ^a	0.012	0.016	62.0	61.5	3.73	3.74	0.000	0.000	13.5	13.3	25.9	26.0	0.093	0.095
12	30.7	31.9	0.007	0.004	0.342 ^c	0.122 ^c	58.6	67.5	4.23 ^b	4.39 ^b	0.004 ^c	0.002 ^c	15.8 ^b	17.8 ^b	25.8	26.5	0.389 ^c	0.453 ^c

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 24: Reproducibility data for Toddler Powder sample.

Sample ID: Toddler Powder																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	56.8	56.6	0.027	0.025	0.732	0.728	124	122	5.40	5.39	0.079	0.078	26.5	26.1	45.1	44.6	0.310	0.309
2	63.5 ^e	69.9 ^e	0.033	0.036	0.879 ^c	1.076 ^c	152	144	5.97	6.10	0.108	0.100	30.5	29.3	-	-	0.344 ^c	0.480 ^c
3	65.7	65.9	0.029	0.028	0.834	0.829	135	135	6.29	6.24	0.087	0.087	28.5	28.6	48.0	47.9	0.331	0.324
4	71.3	68.9	0.025	0.027	0.849	0.821	150	145	6.18	5.99	0.095	0.090	30.5	30.0	48.3	50.5	0.312	0.327
5	61.2	63.2	0.025	0.025	0.698	0.717	132	137	5.33	5.44	0.075	0.077	26.5	27.2	45.8	46.6	0.290	0.292
6	66.0	66.1	0.030	0.031	0.823	0.809	123	123	5.95	5.94	0.086	0.086	32.3	32.5	48.5	48.7	0.342	0.327
7	69.4	71.0	0.024	0.024	0.821	0.822	138	137	6.12	6.07	0.077	0.078	29.1	29.0	52.4	52.4	0.334	0.338
8	63.0	62.3	0.031	0.028	0.756	0.716	130	128	5.94	5.83	0.080	0.078	27.6	27.1	47.5	46.7	0.321	0.314
9	64.9	64.6	0.030	0.031	0.691	0.708	132	132	4.85	4.89	0.085	0.086	28.3	29.0	46.1	47.1	0.329	0.334
10	67.4	66.4	0.028	0.027	0.848	0.827	141	136	6.03	5.92	0.091	0.087	29.0	28.5	49.7	48.3	0.330	0.319
11	75.9	74.0	0.035	0.030	1.023	1.056	151	150	7.22	7.15	0.108	0.107	33.8	34.5	55.6	55.5	0.402	0.404
12	64.1	64.4	0.030	0.030	0.863	0.807	137	138	6.42	6.39	0.090	0.088	28.8	29.1	52.2	51.9	0.403	0.371

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 25: Reproducibility analysis for calcium.

Sample	Precision data for calcium																			
	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g	8 ^h	9 ⁱ	10 ^j	11 ^k	12 ^l	13 ^m	14 ⁿ	15 ^o	16 ^p	17 ^q	18 ^r	19 ^s	
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	14	14	14	14	14	14	14	14	14	14	13	14	14	14	14	14	14	14	14
Number of non-compliant laboratories	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2
Number of outliers (laboratories)	0	0	1	0	0	0	0	0	1	1	0	1	1	0	0	0	0	0	0	0
Number of laboratories after eliminating outliers	12	12	11	12	12	12	12	12	11	11	12	10	11	12	12	12	12	12	12	12
Number of accepted results	24	24	22	24	24	24	24	24	22	22	24	20	22	24	24	24	24	24	24	24
Overall mean of all data (grand mean)	530	34.6	55.5	14.5	99	7.44	34.4	49.5	38.2	68.9	63.2	45.7	69.5	36.7	58.0	74.4	48.9	30.3	65.9	
Repeatability standard deviation S _r	12.7	1.48	5.1	0.3	7.2	0.167	0.91	2.33	0.98	2.7	1.34	0.4	2.22	0.774	2.18	2.84	4.14	0.63	1.57	
Reproducibility standard deviation S _R	24.7	2.49	15.4	0.8	10.2	1.102	2.57	5.27	2.93	6.5	5.18	3.8	5.52	2.72	4.59	5.81	6.32	1.08	4.73	
Repeatability relative standard deviation RSD _r	2.40	4.29	9.3	2.2	7.3	2.25	2.64	4.70	2.56	3.9	2.13	0.9	3.20	2.11	3.76	3.81	8.46	2.08	2.38	
Reproducibility relative standard deviation RSD _R	4.65	7.20	27.8	5.3	10.3	14.8	7.47	10.6	7.7	9.5	8.20	8.4	7.9	7.42	7.92	7.8	12.9	3.56	7.17	
HORRAT value	1.06	1.09	4.50	0.70	1.82	1.77	1.12	1.69	1.17	1.58	1.35	1.32	1.33	1.13	1.29	1.32	2.05	0.53	1.19	
Repeatability limit in SMPR	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

^a SRM NIST 1849a, ^b Adult Powder Low Fat, ^c Adult RTF High Fat, ^d Adult RTF High Fat Placebo, ^e Adult RTF High Protein, ^f Adult RTF High Protein Placebo, ^g Child Milk Powder Placebo, ^h Child Milk Powder, ⁱ Illuma Child Powder, ^j Infant Elemental Powder, ^k Infant Elemental Powder Placebo, ^l Infant PH Powder Milk, ^m Infant PH Powder Soy, ⁿ Infant Powder fos-gos, ^o Infant Powder Milk, ^p Infant Powder Soy, ^q Infant RTF Milk, ^r Infant RTF Placebo, ^s Toddler Powder

NOTE: The results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight). SRM NIST 1849a is expressed in mg/100g product.

Table 26: Reproducibility analysis for copper.

Sample	Precision data for copper																			
	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g	8 ^h	9 ⁱ	10 ^j	11 ^k	12 ^l	13 ^m	14 ⁿ	15 ^o	16 ^p	17 ^q	18 ^r	19 ^s	
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14
Number of non-compliant laboratories	2	2	2	2	2	2	2	2	2	2	6	3	2	2	2	2	2	2	2	2
Number of outliers (laboratories)	0	0	0	0	0	0	1	0	0	1	0	1	0	0	1	0	1	2	0	0
Number of laboratories after eliminating outliers	12	12	12	12	12	12	11	12	12	11	8	10	12	12	11	12	11	10	12	12
Number of accepted results	24	24	24	24	24	24	22	24	24	22	16	20	24	24	22	24	22	20	24	24
Overall mean of all data (grand mean)	1.95	0.071	0.239	0.009	0.184	0.011	0.006	0.111	0.046	0.082	0.004	0.056	0.063	0.047	0.061	0.068	0.046	0.004	0.029	0.029
Repeatability standard deviation S _r	0.053	0.002	0.004	0.002	0.006	0.001	0.001	0.005	0.003	0.003	0.001	0.001	0.004	0.002	0.002	0.004	0.001	0.001	0.002	0.002
Reproducibility standard deviation S _R	0.099	0.007	0.018	0.004	0.016	0.005	0.002	0.010	0.005	0.007	0.004	0.005	0.006	0.004	0.008	0.006	0.004	0.003	0.003	0.003
Repeatability relative standard deviation RSD _r	2.71	2.57	1.82	20	3.37	6	10.37	4.21	7.52	3.84	25.9	1.73	5.82	4.07	2.71	5.16	2.34	32.9	5.25	5.25
Reproducibility relative standard deviation RSD _R	5.09	9.97	7.51	39	8.7	50	27.4	9.0	10.1	8.2	95	8.1	9.1	9.18	12.30	8.12	9.1	71	11.3	11.3
HORRAT value	0.50	0.59	0.54	1.71	0.60	2.23	1.13	0.57	0.56	0.49	3.66	0.47	0.53	0.51	0.71	0.48	0.51	2.68	0.59	0.59
Repeatability limit in SMPR	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	16	10	10	10	10	10	10	16	10	10	10	10	10	10	16	10	10

^a SRM NIST 1849a, ^b Adult Powder Low Fat, ^c Adult RTF High Fat, ^d Adult RTF High Fat Placebo, ^e Adult RTF High Protein, ^f Adult RTF High Protein Placebo, ^g Child Milk Powder Placebo, ^h Child Milk Powder, ⁱ Illuma Child Powder, ^j Infant Elemental Powder, ^k Infant Elemental Powder Placebo, ^l Infant PH Powder Milk, ^m Infant PH Powder Soy, ⁿ Infant Powder fos-gos, ^o Infant Powder Milk, ^p Infant Powder Soy, ^q Infant RTF Milk, ^r Infant RTF Placebo, ^s Toddler Powder

NOTE: The results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight). SRM NIST 1849a is expressed in mg/100g product.

Table 27: Reproducibility analysis for iron.

Sample	Precision data for iron																			
	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g	8 ^h	9 ⁱ	10 ^j	11 ^k	12 ^l	13 ^m	14 ⁿ	15 ^o	16 ^p	17 ^q	18 ^r	19 ^s	
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14
Number of non-compliant laboratories	2	2	2	2	2	2	5	2	2	2	6	3	2	2	2	2	2	6	2	
Number of outliers (laboratories)	0	1	0	1	0	2	3	1	1	2	2	1	1	0	1	1	0	3	1	
Number of laboratories after eliminating outliers	12	11	12	11	12	10	6	11	11	10	6	10	11	12	11	11	12	5	11	
Number of accepted results	24	22	24	22	24	20	12	22	22	20	12	20	22	24	22	22	24	10	22	
Overall mean of all data (grand mean)	17.4	0.615	2.43	0.454	2.05	0.489	0.044	1.17	0.724	1.132	0.028	1.008	1.23	0.543	1.24	1.26	1.35	0.018	0.808	
Repeatability standard deviation S_r	0.227	0.015	0.190	0.074	0.126	0.040	0.001	0.048	0.026	0.053	0.001	0.015	0.035	0.014	0.042	0.048	0.027	0.002	0.019	
Reproducibility standard deviation S_R	0.755	0.058	0.312	0.141	0.169	0.075	0.006	0.093	0.104	0.066	0.003	0.101	0.081	0.082	0.084	0.081	0.107	0.003	0.096	
Repeatability relative standard deviation RSD_r	1.30	2.41	7.81	16	6.14	8.2	3.3	4.11	3.61	4.7	2.836	1.45	2.88	2.58	3.42	3.81	2.0	12	2.38	
Reproducibility relative standard deviation RSD_R	4.34	9.44	12.8	31	8.26	15.4	15	8.0	14.3	5.8	11.791	9.97	6.6	15.0	6.81	6.48	7.9	17	11.9	
HORRAT value	0.59	0.78	1.30	2.44	0.81	1.22	0.81	0.72	1.21	0.52	0.608	0.88	0.60	1.21	0.62	0.59	0.73	0.82	1.02	
Repeatability limit in SMPR	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	
Reproducibility limit in SMPR	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	

^a SRM NIST 1849a, ^b Adult Powder Low Fat, ^c Adult RTF High Fat, ^d Adult RTF High Fat Placebo, ^e Adult RTF High Protein, ^f Adult RTF High Protein Placebo, ^g Child Milk Powder Placebo, ^h Child Milk Powder, ⁱ Illuma Child Powder, ^j Infant Elemental Powder, ^k Infant Elemental Powder Placebo, ^l Infant PH Powder Milk, ^m Infant PH Powder Soy, ⁿ Infant Powder fos-gos, ^o Infant Powder Milk, ^p Infant Powder Soy, ^q Infant RTF Milk, ^r Infant RTF Placebo, ^s Toddler Powder

NOTE: The results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight). SRM NIST 1849a is expressed in mg/100g product.

Table 28: Reproducibility analysis for potassium.

Sample	Precision data for potassium																			
	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g	8 ^h	9 ⁱ	10 ^j	11 ^k	12 ^l	13 ^m	14 ⁿ	15 ^o	16 ^p	17 ^q	18 ^r	19 ^s	
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14
Number of non-compliant laboratories	2	2	2	2	2	2	2	2	2	2	2	3	2	2	2	2	2	2	2	2
Number of outliers (laboratories)	0	0	0	0	0	0	1	0	0	2	0	1	0	0	0	0	0	0	0	0
Number of laboratories after eliminating outliers	12	12	12	12	12	12	11	12	12	10	12	10	12	12	12	12	12	12	12	12
Number of accepted results	24	24	24	24	24	24	22	24	24	20	24	20	24	24	24	24	24	24	24	24
Overall mean of all data (grand mean)	932	44.0	222	177	150	98	7.64	69.7	55.1	84.9	86.4	67.5	78.3	48.3	76.1	82.7	81.4	61.4	136	
Repeatability standard deviation S _r	36.9	0.73	4.97	4	2.91	1.60	0.329	3.59	1.53	1.3	1.82	0.9	2.04	0.783	2.66	2.46	0.859	2.56	2.41	
Reproducibility standard deviation S _R	42.1	2.60	9.3	8	5.27	4.47	0.82	6.06	3.49	4.73	7.20	4.5	4.26	2.79	5.09	5.38	4.59	2.70	9.2	
Repeatability relative standard deviation RSD _r	3.96	1.65	2.24	2.2	1.93	1.62	4.31	5.16	2.79	1.5	2.10	1.3	2.61	1.62	3.50	2.98	1.055	4.18	1.77	
Reproducibility relative standard deviation RSD _R	4.52	5.90	4.18	4.6	3.50	4.54	10.7	8.7	6.33	5.6	8.33	6.6	5.44	5.78	6.7	6.5	5.63	4.39	6.7	
HORRAT value	1.12	0.92	0.83	0.89	0.66	0.80	1.28	1.46	1.02	0.96	1.44	1.11	0.93	0.92	1.14	1.12	0.97	0.72	1.25	
Repeatability limit in SMPR	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

^a SRM NIST 1849a, ^b Adult Powder Low Fat, ^c Adult RTF High Fat, ^d Adult RTF High Fat Placebo, ^e Adult RTF High Protein, ^f Adult RTF High Protein Placebo, ^g Child Milk Powder Placebo, ^h Child Milk Powder, ⁱ Illuma Child Powder, ^j Infant Elemental Powder, ^k Infant Elemental Powder Placebo, ^l Infant PH Powder Milk, ^m Infant PH Powder Soy, ⁿ Infant Powder fos-gos, ^o Infant Powder Milk, ^p Infant Powder Soy, ^q Infant RTF Milk, ^r Infant RTF Placebo, ^s Toddler Powder

NOTE: The results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight). SRM NIST 1849a is expressed in mg/100g product.

Table 29: Reproducibility analysis for magnesium.

Sample	Precision data for magnesium																			
	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g	8 ^h	9 ⁱ	10 ^j	11 ^k	12 ^l	13 ^m	14 ⁿ	15 ^o	16 ^p	17 ^q	18 ^r	19 ^s	
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14
Number of non-compliant laboratories	2	2	2	2	2	3	2	2	2	2	2	4	2	2	2	2	2	2	2	2
Number of outliers (laboratories)	0	0	0	1	0	2	1	0	2	1	2	2	1	2	2	0	1	2	0	0
Number of laboratories after eliminating outliers	12	12	12	11	12	9	11	12	10	11	10	8	11	10	10	12	11	10	12	12
Number of accepted results	24	24	24	22	24	18	22	24	20	22	20	16	22	20	20	24	22	20	24	24
Overall mean of all data (grand mean)	163	13.1	39.5	1.97	32.8	1.68	1.86	9.34	5.37	5.13	5.07	4.30	6.54	4.54	5.01	8.07	6.25	3.33	5.96	
Repeatability standard deviation S _r	2.95	0.218	0.515	0.05	0.67	0.019	0.033	0.347	0.107	0.19	0.080	0.052	0.087	0.105	0.115	0.240	0.177	0.062	0.064	
Reproducibility standard deviation S _R	5.68	1.001	1.80	0.33	1.40	0.141	0.247	0.875	0.283	0.623	0.264	0.221	0.651	0.263	0.277	0.732	0.453	0.249	0.580	
Repeatability relative standard deviation RSD _r	1.81	1.67	1.30	2.8	2.03	1.1	1.75	3.72	1.99	3.6	1.58	1.20	1.34	2.32	2.30	2.98	2.84	1.86	1.07	
Reproducibility relative standard deviation RSD _R	3.48	7.65	4.56	16.9	4.26	8.4	13.29	9.37	5.27	12.1	5.22	5.14	9.95	5.80	5.54	9.07	7.24	7.5	9.74	
HORRAT value	0.66	1.00	0.70	1.66	0.64	0.80	1.29	1.16	0.60	1.37	0.59	0.57	1.17	0.64	0.62	1.10	0.84	0.79	1.13	
Repeatability limit in SMPR	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

^a SRM NIST 1849a, ^b Adult Powder Low Fat, ^c Adult RTF High Fat, ^d Adult RTF High Fat Placebo, ^e Adult RTF High Protein, ^f Adult RTF High Protein Placebo, ^g Child Milk Powder Placebo, ^h Child Milk Powder, ⁱ Illuma Child Powder, ^j Infant Elemental Powder, ^k Infant Elemental Powder Placebo, ^l Infant PH Powder Milk, ^m Infant PH Powder Soy, ⁿ Infant Powder fos-gos, ^o Infant Powder Milk, ^p Infant Powder Soy, ^q Infant RTF Milk, ^r Infant RTF Placebo, ^s Toddler Powder

NOTE: The results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight). SRM NIST 1849a is expressed in mg/100g product.

Table 30: Reproducibility analysis for manganese.

Sample	Precision data for manganese																			
	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g	8 ^h	9 ⁱ	10 ^j	11 ^k	12 ^l	13 ^m	14 ⁿ	15 ^o	16 ^p	17 ^q	18 ^r	19 ^s	
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14
Number of non-compliant laboratories	2	2	2	2	2	4	2	2	2	2	4	3	2	2	3	2	2	3	2	2
Number of outliers (laboratories)	0	2	1	1	0	3	3	1	2	1	2	2	2	2	1	3	4	3	3	0
Number of laboratories after eliminating outliers	12	10	11	11	12	7	9	11	10	11	8	9	10	10	10	9	8	8	8	12
Number of accepted results	24	20	22	22	24	14	18	22	20	22	16	18	20	20	20	18	16	16	16	24
Overall mean of all data (grand mean)	4.89	0.156	0.357	0.008	0.420	0.006	0.004	0.165	0.009	0.052	0.004	0.012	0.025	0.012	0.006	0.034	0.012	0.001	0.088	
Repeatability standard deviation S _r	0.088	0.001	0.023	0.001	0.023	0.000	0.000	0.006	0.000	0.001	0.000	0.000	0.001	0.000	0.000	0.001	0.000	0.000	0.002	
Reproducibility standard deviation S _R	0.221	0.011	0.062	0.002	0.039	0.000	0.000	0.014	0.001	0.005	0.001	0.002	0.002	0.002	0.001	0.002	0.002	0.001	0.010	
Repeatability relative standard deviation RSD _r	1.81	0.9	6.5	8.5	5.36	2.80	4.9	3.65	2.34	1.6	4.63	3.20	2.1	1.5	6.30	2.9	3.45	18	2.48	
Reproducibility relative standard deviation RSD _R	4.51	6.9	17.5	18.5	9.2	7.3	11.5	8.7	8.4	10.3	21.6	20.7	9.1	13.0	23.7	6.3	13.0	65	11.44	
HORRAT value	0.51	0.46	1.32	0.80	0.72	0.30	0.43	0.58	0.37	0.58	0.82	0.94	0.46	0.59	0.97	0.33	0.59	1.95	0.70	
Repeatability limit in SMPR	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	16	10	10	10	10	10	10	10	16	10	10	16	10	10

^a SRM NIST 1849a, ^b Adult Powder Low Fat, ^c Adult RTF High Fat, ^d Adult RTF High Fat Placebo, ^e Adult RTF High Protein, ^f Adult RTF High Protein Placebo, ^g Child Milk Powder Placebo, ^h Child Milk Powder, ⁱ Illuma Child Powder, ^j Infant Elemental Powder, ^k Infant Elemental Powder Placebo, ^l Infant PH Powder Milk, ^m Infant PH Powder Soy, ⁿ Infant Powder fos-gos, ^o Infant Powder Milk, ^p Infant Powder Soy, ^q Infant RTF Milk, ^r Infant RTF Placebo, ^s Toddler Powder

NOTE: The results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight). SRM NIST 1849a is expressed in mg/100g product.

Table 31: Reproducibility analysis for sodium.

Sample	Precision data for sodium																			
	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g	8 ^h	9 ⁱ	10 ^j	11 ^k	12 ^l	13 ^m	14 ⁿ	15 ^o	16 ^p	17 ^q	18 ^r	19 ^s	
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14
Number of non-compliant laboratories	2	2	2	2	2	2	2	2	2	2	2	3	2	2	2	2	2	2	2	2
Number of outliers (laboratories)	1	0	0	0	0	0	0	1	0	1	0	1	0	0	1	0	0	1	0	0
Number of laboratories after eliminating outliers	11	12	12	12	12	12	12	11	12	11	12	10	12	12	11	12	12	11	12	12
Number of accepted results	22	24	24	24	24	24	24	22	24	22	24	20	24	24	22	24	24	22	24	24
Overall mean of all data (grand mean)	427	24.5	140	138	102	102	4.96	18.5	20.3	26.9	26.9	17.5	26.6	15.9	15.5	25.2	18.4	12.2	29.3	29.3
Repeatability standard deviation S _r	7.6	0.452	3.09	4	1.77	0.93	0.238	0.64	0.729	1.20	0.495	0.27	0.82	0.384	0.407	1.05	0.664	0.358	0.408	0.408
Reproducibility standard deviation S _R	10.9	1.99	6.75	8	6.22	5.60	0.662	1.26	1.76	2.21	2.15	1.39	1.99	1.24	1.42	2.04	1.71	0.80	2.25	2.25
Repeatability relative standard deviation RSD _r	1.78	1.84	2.21	3.1	1.73	0.90	4.80	3.45	3.59	4.5	1.84	1.5	3.10	2.42	2.62	4.18	3.61	2.95	1.40	1.40
Reproducibility relative standard deviation RSD _R	2.54	8.12	4.84	5.9	6.08	5.48	13.4	6.8	8.66	8.2	7.98	8.0	7.48	7.77	9.13	8.10	9.32	6.6	7.68	7.68
HORRAT value	0.56	1.16	0.90	1.10	1.08	0.97	1.50	0.93	1.20	1.19	1.16	1.08	1.08	1.04	1.22	1.16	1.28	0.85	1.13	1.13
Repeatability limit in SMPR	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

^a SRM NIST 1849a, ^b Adult Powder Low Fat, ^c Adult RTF High Fat, ^d Adult RTF High Fat Placebo, ^e Adult RTF High Protein, ^f Adult RTF High Protein Placebo, ^g Child Milk Powder Placebo, ^h Child Milk Powder, ⁱ Illuma Child Powder, ^j Infant Elemental Powder, ^k Infant Elemental Powder Placebo, ^l Infant PH Powder Milk, ^m Infant PH Powder Soy, ⁿ Infant Powder fos-gos, ^o Infant Powder Milk, ^p Infant Powder Soy, ^q Infant RTF Milk, ^r Infant RTF Placebo, ^s Toddler Powder

NOTE: The results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight). SRM NIST 1849a is expressed in mg/100g product.

Table 32: Reproducibility analysis for phosphorus.

Sample	Precision data for phosphorus																			
	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g	8 ^h	9 ⁱ	10 ^j	11 ^k	12 ^l	13 ^m	14 ⁿ	15 ^o	16 ^p	17 ^q	18 ^r	19 ^s	
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14
Number of non-compliant laboratories	3	3	3	3	3	3	3	3	3	3	3	4	3	3	3	3	3	3	3	3
Number of outliers (laboratories)	0	0	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Number of laboratories after eliminating outliers	11	11	10	11	11	11	11	11	11	11	11	10	11	11	11	11	11	11	11	11
Number of accepted results	22	22	20	22	22	22	22	22	22	22	22	20	22	22	22	22	22	22	22	22
Overall mean of all data (grand mean)	401	29.7	89.5	70.8	94.9	41.1	23.8	44.4	22.6	51.8	48.3	25.6	41.3	23.6	32.3	49.6	29.1	25.2	49.1	
Repeatability standard deviation S _r	11.8	0.498	2.3	1.4	3.28	0.74	0.55	1.73	0.69	1.54	0.95	0.310	0.56	0.272	1.18	2.34	0.635	0.37	0.65	
Reproducibility standard deviation S _R	18.5	2.11	7.4	1.8	5.85	1.68	1.86	3.68	1.88	3.64	3.42	1.98	2.89	2.04	2.61	3.67	1.95	1.39	3.18	
Repeatability relative standard deviation RSD _r	2.94	1.68	2.6	2.0	3.46	1.81	2.32	3.89	3.04	2.98	1.96	1.21	1.36	1.16	3.66	4.72	2.18	1.49	1.33	
Reproducibility relative standard deviation RSD _R	4.62	7.1	8.3	2.5	6.17	4.08	7.8	8.3	8.3	7.0	7.08	7.76	7.0	8.7	8.1	7.4	6.69	5.5	6.5	
HORRAT value	1.01	1.04	1.44	0.42	1.08	0.63	1.11	1.29	1.18	1.13	1.12	1.12	1.08	1.23	1.20	1.18	0.98	0.79	1.03	
Repeatability limit in SMPR	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

^a SRM NIST 1849a, ^b Adult Powder Low Fat, ^c Adult RTF High Fat, ^d Adult RTF High Fat Placebo, ^e Adult RTF High Protein, ^f Adult RTF High Protein Placebo, ^g Child Milk Powder Placebo, ^h Child Milk Powder, ⁱ Illuma Child Powder, ^j Infant Elemental Powder, ^k Infant Elemental Powder Placebo, ^l Infant PH Powder Milk, ^m Infant PH Powder Soy, ⁿ Infant Powder fos-gos, ^o Infant Powder Milk, ^p Infant Powder Soy, ^q Infant RTF Milk, ^r Infant RTF Placebo, ^s Toddler Powder

NOTE: The results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight). SRM NIST 1849a is expressed in mg/100g product.

Table 33: Reproducibility analysis for zinc.

Sample	Precision data for zinc																			
	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g	8 ^h	9 ⁱ	10 ^j	11 ^k	12 ^l	13 ^m	14 ⁿ	15 ^o	16 ^p	17 ^q	18 ^r	19 ^s	
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14	14
Number of non-compliant laboratories	2	2	2	2	2	2	2	2	2	2	7	3	2	2	2	2	2	2	2	2
Number of outliers (laboratories)	0	0	1	1	0	1	1	2	1	2	2	1	0	2	2	0	1	3	1	1
Number of laboratories after eliminating outliers	12	12	11	11	12	11	11	10	11	10	5	10	12	10	10	12	11	9	11	11
Number of accepted results	24	24	20	20	24	22	22	20	22	20	10	20	24	20	20	24	22	18	22	22
Overall mean of all data (grand mean)	15.2	0.748	2.57	0.46	2.22	0.289	0.161	0.740	0.554	0.750	0.007	0.552	0.580	0.608	0.551	0.852	0.706	0.094	0.335	
Repeatability standard deviation S _r	0.559	0.015	0.034	0.015	0.058	0.009	0.006	0.026	0.020	0.020	0.002	0.023	0.014	0.015	0.028	0.037	0.012	0.003	0.009	
Reproducibility standard deviation S _R	0.80	0.071	0.151	0.028	0.178	0.026	0.024	0.047	0.034	0.075	0.003	0.044	0.055	0.034	0.037	0.083	0.029	0.009	0.033	
Repeatability relative standard deviation RSD _r	3.69	2.05	1.32	3.3	2.61	3.09	3.5	3.45	3.64	2.6	30.6	4.16	2.41	2.53	5.03	4.35	1.71	3.3	2.7	
Reproducibility relative standard deviation RSD _R	5.26	9.4	5.9	6.2	7.99	9.0	14.9	6.3	6.1	10.0	54	7.98	9.5	5.6	6.6	9.7	4.11	9.0	9.9	
HORRAT value	0.70	0.80	0.60	0.49	0.80	0.66	1.00	0.54	0.50	0.84	2.22	0.64	0.77	0.46	0.54	0.84	0.34	0.56	0.75	
Repeatability limit in SMPR	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

^a SRM NIST 1849a, ^b Adult Powder Low Fat, ^c Adult RTF High Fat, ^d Adult RTF High Fat Placebo, ^e Adult RTF High Protein, ^f Adult RTF High Protein Placebo, ^g Child Milk Powder Placebo, ^h Child Milk Powder, ⁱ Illuma Child Powder, ^j Infant Elemental Powder, ^k Infant Elemental Powder Placebo, ^l Infant PH Powder Milk, ^m Infant PH Powder Soy, ⁿ Infant Powder fos-gos, ^o Infant Powder Milk, ^p Infant Powder Soy, ^q Infant RTF Milk, ^r Infant RTF Placebo, ^s Toddler Powder

NOTE: The results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight). SRM NIST 1849a is expressed in mg/100g product.

Table 34: Reproducibility data for Butter sample.

Sample ID:	Butter																	
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	18.4	19.6	0.000	0.000	-0.00 ^a	0.034 ^a	31.2	40.0	1.99	2.30	-0.00 ^a	-0.00 ^a	231	249	25.0	26.8	0.062	0.070
2	46.8 ^c	13.9 ^c	0.069	0.058	0.297 ^b	0.257 ^b	76.6 ^c	22.8 ^c	0.17 ^a	-3.39 ^a	0.069 ^b	0.067 ^b	582 ^c	186 ^c	-	-	0.109 ^c	0.020 ^c
3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4	19.7	20.4	-0.01 ^a	-0.01 ^a	0.014	0.013	36.3	38.4	2.10	2.22	0.001	0.001	236	246	27.5	28.1	0.076	0.060
5	13.5 ^b	12.6 ^b	0.028	0.029	-0.48 ^a	-0.57 ^a	45.4	53.8	1.27	1.15	-0.00 ^a	-0.01 ^a	229	229	22.7	22.3	-0.13 ^a	-0.18 ^a
6	20.5	20.8	0.027	0.004	0.071	0.047	37.2	37.3	1.99	1.96	0.000	0.002	259	281	25.8	25.6	0.459 ^b	0.377 ^b
7	22.3	22.3	-0.01 ^a	-0.01 ^a	-0.03 ^a	-0.01 ^a	37.5	38.1	2.71	2.71	-0.00 ^a	-0.00 ^a	259	261	29.8	30.0	-0.02 ^a	-0.01 ^a
8	30.9 ^c	27.4 ^c	0.038	0.026	<0,1 ^a	<0,1 ^a	56.9	54.1	1.37	0.79	0.016 ^c	0.062 ^c	234	240	23.6 ^e	20.4 ^e	0.135	0.103
9	9.5 ^b	8.8 ^b	-0.02 ^a	-0.03 ^a	-1.25 ^a	-1.30 ^a	52.4	54.0	-13.8 ^a	-14.4 ^a	-0.03 ^a	-0.03 ^a	238	235	4.22 ^b	3.58 ^b	0.024	0.031
10	19.1	19.8	-	-	0.051	0.004	33.4	35.5	2.1	2.2	-	-	226	240	25.22	25.91	0.060	0.037
11	17.1	17.1	-0.01 ^a	0.01 ^a	-0.12 ^a	-0.12 ^a	33.4	34.8	1.69	1.72	-0.03 ^a	-0.04 ^a	251	251	23.7	23.6	-0.02 ^a	-0.02 ^a
12	19.5	19.8	0.001	0.001	0.022	0.021	32.5	32.6	2.30	2.37	0.001	0.001	233	235	24.6	25.4	0.072	0.066

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 35: Reproducibility data for Cheese sample.

Sample ID: Cheese																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	488	490	0.051	0.048	0.156	0.156	96.3	100.7	20.7	20.5	0.013	0.013	988	1018	854	863	2.51	2.52
2	529	522	0.119	0.117	0.427 ^b	0.469 ^b	92.8	91.9	16.7	17.0	0.084 ^b	0.088 ^b	1050	1050	-	-	2.23	2.29
3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4	504	504	0.047	0.050	0.173	0.180	97.8	98.0	19.7	20.3	0.016	0.015	1010	1013	859	865	2.42	2.36
5	498	496	0.068	0.082	-0.26 ^a	-0.40 ^a	109.9	109.9	19.4	18.8	0.007	0.003	962	948	817	802	2.40	2.33
6	474	473	0.067 ^c	0.137 ^c	0.195	0.244	109.0	111.0	18.2	18.4	0.013	0.016	779 ^d	732 ^d	727	717	2.61	2.44
7	500	517	0.029	0.031	0.141	0.13	91.6	94.7	20.5	21.4	0.010	0.011	958	975	863	894	2.39	2.41
8	468 ^a	-	0.064 ^a	-	<0,1 ^a	-	131 ^a	-	19.8 ^a	-	0.018 ^a	-	1047 ^a	-	868 ^a	-	2.36 ^a	-
9	457	463	0.025	0.017	-1.14 ^a	-1.10 ^a	107.0	106.0	5.0 ^b	5.2 ^b	-0.02 ^a	-0.02 ^a	942	942	758	783	2.17	2.26
10	495	485	0.042	0.044	0.192	0.175	94.4	93.8	20.2	20.0	0.014	0.012	1003	992	858	824	2.42	2.39
11	501	491	0.032	0.028	0.114 ^c	0.386 ^c	90.7	90.6	20.3	20.4	-0.02 ^a	-0.02 ^a	1020	1036	787	809	2.25	2.33
12	515	487	0.072	0.074	0.606 ^b	0.769 ^b	95.8	92.0	23.7	21.9	0.024	0.021	1011	1005	923	889	3.55 ^b	3.09 ^b

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 36: Reproducibility data for Wheypowder sample.

Sample ID: Wheypowder																		
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	702	700	0.040	0.039	0.311	0.286	1895	1953	205	205	0.005	0.005	932	965	1034	1021	0.230	0.228
2	689e	728e	0.078	0.082	0.564	0.506	1850	1891	198	206	0.070 ^b	0.069 ^b	898	928	-	-	0.116	0.117
3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4	505 ^b	496 ^b	0.018	0.030	0.143	0.557	2448	2457	116 ^b	119 ^b	0.004	0.015	628 ^b	625 ^b	643b	646b	0.142	0.134
5	711	715	0.070	0.069	-0.37 ^a	-0.37 ^a	1833	1808	194	196	-0.00 ^a	-0.00 ^a	889	911	956	968	0.012	0.00
6	764	749	0.066	0.071	0.338	0.328	1439	1419	185	185	0.008	0.008	1045 ^d	1036 ^d	893	881	0.722 ^b	0.336 ^b
7	725	729	-0.00 ^a	-0.00 ^a	0.268	0.27	1807	1764	199	198	0.005	0.004	899	875	1043	1015	0.070	0.069
8	682	674	0.05	0.05	0.059	0.083	1562	1542	208	209	0.011	0.012	1019 ^e	851 ^e	1033	1030	0.208	0.209
9	464 ^b	463 ^b	0.005 ^a	-0.00 ^a	-1.17 ^a	-1.19 ^a	2270	2270	113 ^b	114 ^b	-0.03 ^a	-0.03 ^a	601 ^b	605 ^b	622 ^b	625 ^b	0.073	0.080
10	700	704	0.035	0.032	0.31	0.32	1834	1839	199	200	0.01	0.01	911	916	984	988	0.243	0.256
11	697	699	0.015	0.008	0.226	0.174	1804	1743	221	211	-0.02 ^a	-0.02 ^a	948	919	968	931	0.068	0.044
12	691	693	0.062	0.069	0.517	0.839	1831	1830	207	208	0.01	0.02	923	930	1054	1052	0.501 ^b	0.875 ^b

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 37: Reproducibility data for Whole Milk sample.

Sample ID:	Whole Milk																	
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	113	112	0.006	0.005	0.012	0.017	162	157	10.65	10.44	0.002	0.002	37.5	36.5	96.0	94.5	0.405	0.402
2	124	121	0.016	0.017	0.082 ^b	0.074 ^b	177	175	10.46	10.21	0.018 ^c	0.016 ^c	38.6	38.3	-	-	0.362	0.356
3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4	120	121	0.004	0.005	0.018	0.016	161	164	10.23	10.38	0.003	0.003	37.2	37.9	99.4	102.1	0.389 ^c	0.399 ^c
5	112	114	0.011 ^c	0.013 ^c	-0.12 ^a	-0.11 ^a	163	163	9.82	9.84	-0.00 ^a	0.003 ^a	35.9	35.7	92.2	93.4	0.356	0.356
6	112	111	0.009 ^c	0.013 ^c	0.024	0.023	139	135	10.00	9.96	0.002	0.002	36.6	35.9	94.4	94.3	0.403	0.388
7	118	105	-0.00 ^a	-0.00 ^a	0.008	0.009	155 ^e	140 ^e	10.28 ^e	9.30 ^e	0.002	0.002	36.0 ^e	32.5 ^e	99.4 ^e	87.9 ^e	0.390	0.336
8	130 ^e	130 ^e	0.006	0.006	<0,1 ^a	<0,1 ^a	153	153	10.24	10.18	0.003	0.003	35.1	34.7	96.9	96.1	0.367	0.366
9	110	109	-0.00 ^a	0.026 ^a	-0.25 ^a	-0.24 ^a	153	152	8.16	8.18	-0.00 ^a	-0.00 ^a	35.4	35.0	90.3	86.9	0.362	0.375
10	113	114	0.004	0.004	0.015	0.022	156	156	10.41	10.57	0.002	0.002	36.0	36.0	90.5	92.1	0.377	0.388
11	120	121	0.000	0.001	-0.00 ^a	-0.02 ^a	158	160	11.26	11.27	-0.00 ^a	-0.00 ^a	37.3	38.0	96.3	96.5	0.359	0.364
12	113	113	0.012	0.012	0.132 ^c	0.100 ^c	156	155	11.22	11.76	0.005 ^b	0.005 ^b	39.9	41.9	96.9	96.5	0.562	0.773

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 38: Reproducibility data for WMP sample.

Sample ID:	WMP																	
Analyte Laboratory	Ca (mg/100g)		Cu (mg/100g)		Fe (mg/100g)		K (mg/100g)		Mg (mg/100g)		Mn (mg/100g)		Na (mg/100g)		P (mg/100g)		Zn (mg/100g)	
	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	94	93	0.004	0.004	0.024	0.021	119	115	8.54	8.54	0.002	0.002	29.9	29.0	78.9	78.1	0.340	0.335
2	110	112	0.011	0.012	0.056	0.066	141	142	9.48	9.45	0.009 ^c	0.011 ^c	34.2	34.4	-	-	0.323	0.338
3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4	109 ^c	82 ^c	0.004	0.003	0.027	0.020	138	137	9.40	8.12	0.002 ^c	0.001 ^c	34.9	34.7	88.6 ^c	70.6 ^c	0.362	0.240
5	104	107	0.010	0.011	-0.05 ^a	-0.05 ^a	132	133	8.78	9.02	0.001	0.001	31.9	33.3	85.3	87.6	0.343	0.343
6	101	99	0.008	0.012	0.034	0.028	116	117	9.22	8.68	0.002	0.002	37.0	36.8	89.1 ^e	80.6 ^e	0.371	0.399
7	106	104	0.000	0.000	0.022	0.019	126	126	8.98	8.95	0.002	0.002	32.1	31.9	87.2	85.9	0.352 ^e	0.344 ^e
8	115 ^e	101 ^e	0.005	0.008	<0,1 ^a	0.019 ^a	126	130	8.95	9.74	0.004	0.004	31.5	33.0	83.3	86.8	0.327	0.354
9	99	96	0.002	0.002	-0.14 ^a	-0.13 ^a	126	123	8.21	7.98	-0.00 ^a	-0.00 ^a	31.8	31.4	80.7	80.4	0.336	0.321
10	108	106	0.003	0.003	0.026	0.026	133	129	9.77	9.48	0.002	0.002	33.6	32.7	86.3	84.3	0.367	0.360
11	124	123	0.000 ^a	-0.00 ^a	-0.00 ^a	-0.00 ^a	149	147	11.3 ^d	11.2 ^d	-0.00 ^a	-0.00 ^a	38.3	37.2	97.0	96.4	0.375	0.367
12	101	101	0.007	0.01	0.038	0.060	131	131	9.70	9.75	0.003	0.003	33.4	33.4	87.9	88.9	0.408 ^c	0.425 ^c

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 39: Reproducibility data for WPC sample.

Sample ID:	WPC																	
Analyte	Ca		Cu		Fe		K		Mg		Mn		Na		P		Zn	
	(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)		(mg/100g)	
Laboratory	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2	Rep-1	Rep-2
1	667	676	0.115	0.113	0.625	0.629	800	806	73.5	74.5	0.007	0.006	259	259	493	497	0.882	0.895
2	723	732	0.155	0.170	0.854	0.882	861	876	76.2	75.3	0.347 ^c	0.074 ^c	267	271	-	-	0.694	0.731
3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4	709	706	0.118	0.115	0.645	0.641	819	814	73.1	72.2	0.009	0.009	263	261	534	537	0.869	0.898
5	681	699	0.146	0.144	-0.04 ^a	0.023 ^a	835	810	71.0	70.7	-0.00 ^a	-0.00 ^a	255	258	500	500	0.619	0.644
6	669	650	0.153	0.137	0.682	0.724	653 ^d	620 ^d	71.6	70.4	0.008	0.011	306 ^{d+e}	281 ^{d+e}	494	473	1.09 ^c	0.961 ^c
7	697	684	0.084	0.082	0.637	0.578	787	777	72.8	72.0	0.006	0.005	253	250	518	510	0.737	0.732
8	820 ^e	667 ^e	0.112	0.121	0.409 ^c	0.539 ^c	804	791	75.6	76.6	0.012	0.015	257	259	527	521	0.825	0.832
9	648	644	0.105	0.105	-0.57 ^a	-0.64 ^a	761	765	65.8	65.6	-0.02 ^a	-0.03 ^a	243	245	458	483	0.819	0.773
10	681	677	0.109	0.109	0.658	0.665	795	792	72.4	74.2	0.009	0.006	255	254	492	481	0.864	0.866
11	701	696	0.085	0.111	0.523	0.521	795	781	79.6	77.4	-0.03 ^a	-0.04 ^a	264	262	502	484	0.725	0.776
12	664	664	0.153	0.145	0.738 ^c	1.227 ^c	799	800	77.0	77.5	0.011 ^c	0.022 ^c	260	260	510	513	1.28 ^b	1.35 ^b

^a Eliminated from data before statistical analysis (e.g. negative values).

^b Identified as Grubbs outlier; results removed from data set for statistical analysis.

^c Identified as Cochran outlier; results removed from data set for statistical analysis.

^d Identified as Grubbs outlier; results kept in data set for statistical analysis.

^e Identified as Cochran outlier; results kept in data set for statistical analysis.

NOTE: results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 40: Reproducibility analysis for calcium.

Precision data for calcium							
Sample	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	13	13	13	13	13	13
Number of non-compliant laboratories	2	2	2	2	2	2	2
Number of outliers (laboratories)	0	0	1	2	0	4	0
Number of laboratories after eliminating outliers	12	11	10	9	11	7	11
Number of accepted results	24	22	20	18	22	14	20
Overall mean of all data (grand mean)	530	116	105	709	689	19.8	494
Repeatability standard deviation S_r	12.7	2.89	3.45	10.3	33.5	0.42	8.37
Reproducibility standard deviation S_R	24.7	6.77	8.61	23.7	37.8	1.62	19.2
Repeatability relative standard deviation RSD_r	2.40	2.48	3.28	1.45	4.86	2.1	1.69
Reproducibility relative standard deviation RSD_R	4.65	5.82	8.19	3.3	5.49	8.2	3.88
HORRAT value	1.06	1.05	1.46	0.79	1.30	1.14	0.87
Repeatability limit in SMPR	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10

^a SRM 1949a, ^b Whole Milk, ^c WMP, ^d Wheypowder, ^e WPC, ^f Butter, ^g Cheese

NOTE: The results are expressed as mg/100 g. WMP results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 41: Reproducibility analysis for copper.

Precision data for copper							
Sample	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	13	13	13	13	13	13
Number of non-compliant laboratories	2	4	3	4	2	7	3
Number of outliers (laboratories)	0	2	0	0	0	0	1
Number of laboratories after eliminating outliers	12	7	10	9	11	6	9
Number of accepted results	24	14	20	18	22	12	18
Overall mean of all data (grand mean)	1.95	0.007	0.006	0.049	0.122	0.023	0.054
Repeatability standard deviation S_r	0.053	0.000	0.001	0.004	0.008		0.004
Reproducibility standard deviation S_R	0.099	0.005	0.004	0.024	0.025		0.030
Repeatability relative standard deviation RSD_r	2.71	4.1	20.7	8.40	6.24		7.6
Reproducibility relative standard deviation RSD_R	5.09	78.2	71.3	48.5	20.7		56.1
HORRAT value	0.50	3.27	2.91	2.72	1.33		3.20
Repeatability limit in SMPR	5	5	5	5	5		5
Reproducibility limit in SMPR	10	10	16	10	10		10

^a SRM 1949a, ^b Whole Milk, ^c WMP, ^d Wheypowder, ^e WPC, ^f Butter, ^g Cheese

NOTE: The results are expressed as mg/100 g. WMP results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 42: Reproducibility analysis for iron.

Precision data for iron							
Sample	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	13	13	13	13	13	13
Number of non-compliant laboratories	2	6	6	4	4	8	5
Number of outliers (laboratories)	0	2	0	0	2	1	3
Number of laboratories after eliminating outliers	12	5	7	9	7	4	5
Number of accepted results	24	10	14	18	14	8	10
Overall mean of all data (grand mean)	17.4	0.016	0.033	0.338	0.662	0.030	0.174
Repeatability standard deviation S_r	0.23	0.00	0.01	0.13	0.021		0.02
Reproducibility standard deviation S_R	0.75	0.01	0.02	0.20	0.11		0.03
Repeatability relative standard deviation RSD_r	1.30	16	21	37.0	3.13		10
Reproducibility relative standard deviation RSD_R	4.34	35	49	59	16.3		20
HORRAT value	0.59	1.69	2.58	4.40	1.35		1.34
Repeatability limit in SMPR	5	5	5	5	5		5
Reproducibility limit in SMPR	10	10	10	10	10		10

^a SRM 1949a, ^b Whole Milk, ^c WMP, ^d Wheypowder, ^e WPC, ^f Butter, ^g Cheese

NOTE: The results are expressed as mg/100 g. WMP results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1 by weight).

Table 43: Reproducibility analysis for potassium.

Precision data for potassium							
Sample	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	13	13	13	13	13	13
Number of non-compliant laboratories	2	2	2	2	2	2	3
Number of outliers (laboratories)	0	0	0	0	0	1	0
Number of laboratories after eliminating outliers	12	11	11	11	11	10	10
Number of accepted results	24	22	22	22	22	20	20
Overall mean of all data (grand mean)	932	157	130	1868	788	40.7	98.7
Repeatability standard deviation S_r	36.9	3.64	1.63	23.5	10.6	2.9	1.59
Reproducibility standard deviation S_R	42.1	10.2	9.73	287	57.6	8.8	7.5
Repeatability relative standard deviation RSD_r	3.96	2.32	1.25	1.26	1.34	7.2	1.61
Reproducibility relative standard deviation RSD_R	4.52	6.49	7.46	15.4	7.30	21.6	7.6
HORRAT value	1.12	1.23	1.37	4.22	1.76	3.34	1.34
Repeatability limit in SMPR	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10

^a SRM 1949a, ^b Whole Milk, ^c WMP, ^d Wheypowder, ^e WPC, ^f Butter, ^g Cheese

NOTE: The results are expressed as mg/100 g. WMP results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 44: Reproducibility analysis for magnesium.

Precision data for magnesium							
Sample	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	13	13	13	13	13	13
Number of non-compliant laboratories	2	2	2	2	2	4	3
Number of outliers (laboratories)	0	0	0	2	0	0	1
Number of laboratories after eliminating outliers	12	11	11	9	11	9	9
Number of accepted results	24	22	22	18	22	18	18
Overall mean of all data (grand mean)	163	10.2	9.24	202	73.4	1.94	19.9
Repeatability standard deviation S_r	2.95	0.3	0.352	3.13	0.815	0.164	0.511
Reproducibility standard deviation S_R	5.68	0.9	0.873	9.1	3.61	0.537	1.72
Repeatability relative standard deviation RSD_r	1.81	2	3.81	1.55	1.11	8.5	2.57
Reproducibility relative standard deviation RSD_R	3.48	9	9.44	4.5	4.92	27.7	8.7
HORRAT value	0.66	1.08	1.17	0.88	0.83	2.71	1.20
Repeatability limit in SMPR	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10

^a SRM 1949a, ^b Whole Milk, ^c WMP, ^d Wheypowder, ^e WPC, ^f Butter, ^g Cheese

NOTE: The results are expressed as mg/100 g. WMP results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Table 45: Reproducibility analysis for manganese.

Precision data for manganese							
Sample	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	13	13	13	13	13	13
Number of non-compliant laboratories	2	4	4	5	5	8	5
Number of outliers (laboratories)	0	3	2	1	2	2	1
Number of laboratories after eliminating outliers	12	6	7	7	6	3	7
Number of accepted results	24	12	14	14	12	6	14
Overall mean of all data (grand mean)	4.89	0.002	0.002	0.009	0.009	0.0010	0.013
Repeatability standard deviation S_r	0.088		0.000	0.004			0.002
Reproducibility standard deviation S_R	0.221		0.001	0.005			0.005
Repeatability relative standard deviation RSD_r	1.81		3.3	38			11.531
Reproducibility relative standard deviation RSD_R	4.51		42.5	50			39.447
HORRAT value	0.51		1.49	2.20			1.824
Repeatability limit in SMPR	5		5	5			5
Reproducibility limit in SMPR	10		16	16			16

^a SRM 1949a, ^b Whole Milk, ^c WMP, ^d Wheypowder, ^e WPC, ^f Butter, ^g Cheese

NOTE: The results are expressed as mg/100 g. WMP results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1 by weight).

Table 46: Reproducibility analysis for sodium.

Precision data for sodium							
Sample	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	13	13	13	13	13	13
Number of non-compliant laboratories	2	2	2	2	2	2	3
Number of outliers (laboratories)	0	0	0	2	0	1	0
Number of laboratories after eliminating outliers	12	11	11	9	11	10	10
Number of accepted results	22	22	22	18	22	20	20
Overall mean of all data (grand mean)	427	36.7	33.5	933	261	243	972
Repeatability standard deviation S_r	7.6	0.93	0.575	42.3	5.50	7.7	14.1
Reproducibility standard deviation S_R	10.9	1.99	2.42	54	13.2	14.1	83.4
Repeatability relative standard deviation RSD_r	1.78	2.54	1.72	4.54	2.11	3.2	1.46
Reproducibility relative standard deviation RSD_R	2.54	5.43	7.24	5.7	5.04	5.8	8.59
HORRAT value	0.56	0.83	1.09	1.42	1.03	1.17	2.14
Repeatability limit in SMPR	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10

^a SRM 1949a, ^b Whole Milk, ^c WMP, ^d Wheypowder, ^e WPC, ^f Butter, ^g Cheese

NOTE: The results are expressed as mg/100 g. WMP results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1 by weight).

Table 47: Reproducibility analysis for phosphorus.

Precision data for phosphorus							
Sample	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	13	12	12	12	12	12	12
Number of non-compliant laboratories	2	2	2	2	2	2	3
Number of outliers (laboratories)	0	0	1	2	0	1	0
Number of laboratories after eliminating outliers	11	10	9	8	10	9	9
Number of accepted results	22	20	18	16	20	18	18
Overall mean of all data (grand mean)	401	94.6	85.8	991	501	25.4	827
Repeatability standard deviation S_r	11.8	2.82	2.33	12.9	9.01	0.92	16.5
Reproducibility standard deviation S_R	18.5	3.89	5.38	56	20.9	2.56	59.0
Repeatability relative standard deviation RSD_r	2.94	2.98	2.72	1.30	1.80	3.63	1.99
Reproducibility relative standard deviation RSD_R	4.62	4.11	6.27	5.7	4.17	10.1	7.13
HORRAT value	1.01	0.72	1.08	1.42	0.94	1.45	1.73
Repeatability limit in SMPR	5	5	5	5	5	5	5
Reproducibility limit in SMPR	10	10	10	10	10	10	10

^a SRM 1949a, ^b Whole Milk, ^c WMP, ^d Wheypowder, ^e WPC, ^f Butter, ^g Cheese

NOTE: The results are expressed as mg/100 g. WMP results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1 by weight).

Table 48: Reproducibility analysis for zinc.

Precision data for zinc							
Sample	1 ^a	2 ^b	3 ^c	4 ^d	5 ^e	6 ^f	7 ^g
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016
Number of laboratories	14	13	13	13	13	13	13
Number of non-compliant laboratories	2	2	2	2	2	5	3
Number of outliers (laboratories)	0	2	1	2	2	2	1
Number of laboratories after eliminating outliers	12	9	10	9	9	6	9
Number of accepted results	24	20	20	18	18	12	18
Overall mean of all data (grand mean)	15.2	0.375	0.357	0.127	0.788	0.066	2.4
Repeatability standard deviation S_r	0.559	0.013	0.011	0.008	0.021		0.1
Reproducibility standard deviation S_R	0.80	0.020	0.029	0.086	0.089		0.1
Repeatability relative standard deviation RSD_r	3.69	3.57	3.04	6.1	2.63		2.4
Reproducibility relative standard deviation RSD_R	5.26	5.27	8.17	67.2	11.3		4.8
HORRAT value	0.70	0.40	0.62	4.36	0.96		0.48
Repeatability limit in SMPR	5	5	5	5	5		5
Reproducibility limit in SMPR	10	10	10	10	10		10

^a SRM 1949a, ^b Whole Milk, ^c WMP, ^d Wheypowder, ^e WPC, ^f Butter, ^g Cheese

NOTE: The results are expressed as mg/100 g. WMP results are expressed as mg/100g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water); liquid concentrates diluted 1:1by weight).

Safety Considerations

There are no major hazards beyond those typical found in chemistry laboratories. Use appropriate safety equipment when handling nitric acid. Refer to MSD sheets for detailed safety instructions for each chemical used.

Comments from Collaborators

Lab 1: The concentration of working standards are not the same as mentioned in protocol or spreadsheet. Adult RTF High Protein samples contained lumps, weighted sample after homogenizing thoroughly. WMP created inhomogeneous solution.

Lab 2: We work gravimetrically. We didn't report the K 404.72 line because quality control requirements were not achieved. An alternative digestion procedure is used by us:

Step	Outlet Pressure (W)	Time (min)	Final Temperature (°C)
1	1500	10	120
2	1500	15	180
3	1500	15	180

Lab 3: For the milk: place the milk at 20°C or 40°C and then mix thoroughly, accurately weigh about 2.5000 g samples in MDC vessel. For the infant formula: accurately weigh about 25.00 g powder and dissolve them into 200.00g water and then mix by vibrating, accurately weigh about 4.000g prepared test sample in MDC vessel. Add 5.0ml HNO₃ into MDC, predigest 10 mins at room temperature, then close MDC vessels, ramp temperature from ambient to 200°C in 15 mins and hold at 200°C for 25 mins.

Lab 5: We used an on line internal standard method and In-house optimized calibration curve.

Lab 7: For calcium a blank from CsCl₂ is used for LOQ determination. We adapted the sample intake to our digestion system.

Lab 8: The Preparation of samples and analysis was done on the same day. It seems to be first wave line results are more reliable than second wave line results. Wave line Ca 779 was not able to select due to limitation of the instrument. Yttrium was added to the sample as internal standard.

Lab 9: We modified volume of test solutions (25ml →50ml). We modified concentration of calibration standard solutions. The CCV is calibration standard 3.

Lab 10: Adaption of slurry sample digestion has been made: weigh about 2 gram slurry, add four 4 mL HNO₃ and 1 mL H₂O₂ to complete microwave digestion.

Conclusions

A Collaborative study of AOAC 2011.14 ISO/CD 15151 | IDF 229an ICP-AES method for the analysis of minerals and trace elements was undertaken. The method was applied to a number of different infant formula, adult nutrition and dairy samples and demonstrated acceptable precision for infant formula and adult nutrition and dairy samples (except for butter). The method showed lesser precision for Fe, Cu, and Mn in the placebo infant formulas and dairy products tested in this study due to low concentrations.

Acknowledgements

We are grateful to the collaborators and their colleagues for the participation in this study. Covance (USA) and MUVA (Germany) are gratefully acknowledged for the supply of infant formulas and dairy products as part of this study. Special thanks to Stefan de Boer and Ilse Voortman from FrieslandCampina for their great contribution on compiling the data, statistical treatment and reporting the results of this study.

References

- 1) AOAC SMPR 2014.004 Standard method performance requirements for minerals and trace elements in infant formula and adult/pediatric nutritional formula
- 2) Noel, L, Carl, M; Vastel C, Guerin, T.; International Dairy Journal 18(2008) 899-904.
- 3) EN 16943:2015 Foodstuffs-determination of elements and their chemical species- determination of minerals by ICP-OES.

MLT REPORT

Report Title:	Multi Laboratory Test – AOAC First Action Method 2016.03 Chloride in Infant Formula and Adult/Pediatric Nutritionals.
Date of Issue:	January 23, 2017

Report Information

Name/s of Author/s:	G. Jaudzems – NQAC-Dublin
Keywords:	Laboratory/Validation/Chloride/Infant Formula

Distribution:

A. Hugget, CO-QM	
E. Konings, NRC/FSQ	L. Buratti, NQAC AOA
G. Daix, NRC/FSQ	E. Vanegas, NQAC AMS
K. Kraehenbuehl, NRC/AS	C. Brouard, NQAC EUR
R. Stadler, NRC/FSQ	F. Robert, NQAC-Dublin
A. McMahon, Wyeth Nutrition, Askeaton	M-H. Le Breton, NQAC-Dublin
J.-D. Fournier, NN	SPIFAN Expert Review Panel
A. Ayres, CO-QM	
	Minerals and metals group

Summary

Business and Technical Impact

The objective of the project is to establish international consensus methods for infant formula and adult/pediatric nutritionals.

A new path to achieve AOAC Official Method status was approved in 2011. Under this path, methods can be granted Official First Action status by vetted Expert Review Panels and remain on First Action for about 2 years. During this period, Multi Laboratory Testing takes places and/or method performance data are collected before Final Action status can be recommended by the Expert Review Panel to the Official Methods Board. Once Final Action status is granted, the method will be presented to CCMAS (Comité du Codex sur les méthodes d'analyse et d'échantillonnage), to become Codex Type II method (dispute resolution); which will ensure the international recognition of results provided by Nestlé laboratories.

In addition, during this study matrix expansion data was collected for milk, whey, cheese, and butter.

Summary, Continued

Objective

Objective of the Project: To establish international consensus methods for infant formula and adult/pediatric nutritionals.

Objective of the Work: To validate the current method (AOAC First Action 2016.03) by determining its precision figures (repeatability and reproducibility) through a multi-laboratory testing (MLT). This is mandatory to finalize the work in progress and go forward to obtain Final Action status.

Methodology and Trials

The MLT was carried out on nineteen SPIFAN matrixes representing most of the products in the scope of the project (Infant Formula and Adult Nutritionals made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch and amino acids, with and without intact protein). In addition six IDF matrixes covering butter, milk, and whey were included. Twenty four laboratories accepted to participate, from which seventeen received samples and reported valid data.

Results and Conclusions

The precision results (repeatability and reproducibility) obtained during the MLT showed that AOAC Official method 2016.03 is in accordance to requirements established by SMPR 2014.015, and thus is fit for purpose for the analysis of chloride in infant formula and adult/pediatric nutritionals. The method should therefore be recommended for AOAC Final Action Official Method status.

Actions

Results will be presented for consideration by the Expert Review Panel on March 2017 to obtain the Final Action status.

Signature/s of all Authors

	Complete name	Signature	Date
Author :	Greg Jaudzems		1-23-17

Detailed Report

Table of Contents

Summary	1
STUDY DESCRIPTION.....	4
Study Information.....	4
Materials, Method and Laboratories	5
Forms	6
INSTRUCTIONS TO PARTICIPANTS.....	7
Part 1: Method set up and qualification of participants	7
Part 2: Multi-Laboratory Test Participation	8
RESULTS.....	9
Part 1. Method set up and qualification of participants	9
Part 2. Multi Laboratory Test.....	9
CONCLUSIONS.....	11
ANNEXES.....	12
List of Annexes	12
Annex A: List of Materials	13
Annex B: Method	14
Annex C: List of participants	28
Annex D: Cover letters to participants.....	29
Annex E: Receipt Forms	30
Annex F: Reporting templates.....	31
Annex G: Raw data MLT samples.....	33

STUDY DESCRIPTION

Study Information

Study Title Chloride in Milk, Milk Powder, Whey Powder, Infant Formula and Adult Nutritionals Potentiometric titration.
AOAC First Action 2016.03: Multi-Laboratory Study.
This method is a combination of AOAC 2015.07 and AOAC 2015.08 in collaboration with CIAQ.

Study Objective The objective of the study was to validate the current method (AOAC First Action 2016.03) by determining its precision figures (repeatability and reproducibility) through a multi-laboratory testing (MLT). This is mandatory to finalize the work in progress and go forward to obtain Final Action status.

Study Directors Greg Jaudzems
Nestlé Quality Assurance Center
6625 Eiterman Road
Dublin, OH 43017
USA
Phone (direct): +1 614.525.5357
E-mail: greg.jaudzems@us.nestle.com
Author: AOAC 2015.08

Fengxia Zhang
Chinese Academy of Inspection and
Quarantine Comprehensive Test Center
Beijing, 100123
China
Author: AOAC 2015.07

Study Monitor Liaison with SPIFAN/AOAC (Official Methods Board): TDB

Study Description The study was divided in two parts: method set up and qualification of participants (part 1) and multi-laboratory test participation (part 2).

Part 1: method set up and qualification of participants

Each participating laboratory was asked to analyze two practice samples using the method provided in the protocol and report the results to the Study Director within the required timelines (6 weeks after receipt). Results within a range of expected levels qualified the laboratory for the second part of the study.

Part 2: Multi-Laboratory Test participation

Each qualified laboratory received fifty (25 blind duplicates) samples to be analyzed using the method provided in the protocol. The laboratory was asked to analyze them on two different days. Each sample was assigned to either day 1 or day 2. The results were submitted to the Study Director for evaluation.

Study Information, Continued

Statistical evaluation

After data collection, outliers were detected using Cochran and Grubbs tests. The number and coded identity of statistical outlier laboratories are included in this report.

Average chloride concentrations, standard deviations of repeatability (S_r) and relative standard deviations of repeatability (RSD_r) were estimated from blind duplicates in MLT samples. The duplicates were assigned to be analyzed on the same day.

Standard deviations of reproducibility (S_R), relative standard deviations of reproducibility (RSD_R), and HORRAT values ($RSD_R/\text{predicted } RSD_R$) were also estimated.

Details on statistical analysis can be found in the "Appendix D: Guidelines for Collaborative Study Procedures To Validate Characteristics of a Method of Analysis" of the Official Methods of Analysis of AOAC INTERNATIONAL, 18th Edition (2005).

Materials, Method and Laboratories

Matrices

The study took place using SPIFAN matrices, which represent most of the products in the scope of the project (Infant Formula and Adult Nutritionals made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch and amino acids, with and without intact protein). In addition, IDF matrixes were added to this MLT in planning to be able to expand the scope to include dairy. All samples were codified.

The complete list of samples can be found in Annex A.

Homogeneity Tests

SPIFAN matrixes:

Homogeneity tests were performed by analyzing several compounds (i.e. calcium, manganese, zinc, pyridoxine and tocopheryl acetate). The testing was performed by Covance Laboratories. The report is available upon request at AOAC.

IDF matrixes:

Homogeneity tests were performed by analyzing several compounds (i.e. calcium, manganese, zinc, fat and chloride). The testing was performed by MUVA kempten. The report is available upon request from MUVA.

Method

The full method description is given in Annex B.

Participating laboratories

Twenty three laboratories tentatively agreed to participate in the study. The complete list can be found in Annex C. Random codes were attributed to the laboratories for identification.

Abbreviations **SPIFAN:** Stakeholder Panel on Infant Formula and Adult Nutritionals
IDF: International Dairy Federation
MLT: Multi-Laboratory Testing
S_r: Standard deviation of repeatability
RSD_r: Relative standard deviation of repeatability
S_R: Standard deviation of reproducibility
RSD_R: Relative standard deviation of reproducibility

Forms

Cover Letters Accompanying cover letters were addressed to participants together with practice and MLT samples, they can be found in Annex D.

Sample Receipt Forms The practice and study materials receipt forms can be found in Annex E.

Reporting Templates Electronic templates were provided to the participants for data reporting (see Annex F). Moreover raw data (i.e. standardization and titer volumes) were required. The option to use an additional protein precipitation step required in China was documented on the data reporting form.

INSTRUCTIONS TO PARTICIPANTS

Part 1: Method set up and qualification of participants

Practice Samples Receipt

All participant laboratories received two practice samples. The “SPIFAN Practice Samples Receipt Form” (see Annex E) was to be filled and sent back to the Study Director.

Before undertaking the analysis, laboratories were instructed to keep the samples at room temperature, away from direct sunlight. Once open, the containers were to be kept at 4 °C in tightly closed containers for the duration of the study.

Analysis

The laboratories set up the method described in Annex B. It was highlighted to closely follow the method described in this document, since small adaptations might have been included with respect to the previously published method (AOAC 2016.03) for SPIFAN matrixes, ISO 5943 for processed cheese, and ISO 15648 for salted butter.

The laboratories were asked to analyze each of the two practice samples in duplicate (two extractions from each reconstituted sample). Any deviation, such as necessity to substitute reagents, apparatus or instruments, was to be recorded and reported.

Timeline

The results on the two practice samples were to be sent to the Study Director within 6 weeks from sample reception.

Reporting

An electronic template was provided for data reporting (see Annex F).

Participating laboratories were required to report Silver nitrate standard concentrations, and volumes. Furthermore, detailed information on the different masses and volumes used during sample preparation as indicated in the method were requested.

Laboratories were asked to report final results in mg chloride/100 g in both “reconstituted powder” and as received for powder samples or “as is” for liquid products, with 3 significant figures.

Laboratory Qualification

After review by the Study Director, results within a range of expected levels identified the laboratories which had the capability to run the analysis successfully. They were therefore qualified for the second part of the study. The laboratories received notification within 2 weeks from data reporting.

Part 2: Multi-Laboratory Test Participation

MLT Samples Receipt All qualified laboratories were provided feedback to go forward with the MLT. The “SPIFAN and IDF/ISO MLT Samples Receipt Form” (Annex E) was to be filled and sent back to the Study Director.

Analysis The laboratories were asked to analyze all the samples (single determination from each reconstituted sample) on two days (25 samples per day). Each sample was assigned to either day 1 or day 2 (see Annex A).

The samples were analyzed singly and individual values reported. It was of critical importance to analyze the samples on their assigned day. It was also highlighted to the participants that this was a study of the method, not the laboratory; the method set up in the first part of the study should be closely followed. Any deviation was to be recorded and reported.

All powdered samples were required to be analyzed on a reconstituted basis, using 25 grams of material and 200 grams of water, as stated in the method, with the exception of MLT Sample 26 (KGSZ273) and MLT Sample 29 (LTCT316). These two samples were to be reconstituted by dissolving 10 grams of powder in 90 grams of water (see Annex A for reconstitution rates).

Special notes on IDF samples:

Method matrix addition samples – See note in method section.

- 1) For processed cheese ISO 5943 to be used: Weigh 2-5 g of test sample into the vessel (for processed cheese 2.5 g). Add 30 ml of water at about 55°C. Suspend the test portion using the blender. Rinse the blender with approximately 10 ml of (demi) water, collecting the rinsings in the vessel. Add 2 ml to 3 ml of the nitric acid (4 mol/l). titrate (same method AOAC 2016.03 can be used)
- 2) For salted butter ISO 15648 to be used: Weigh 2-4 g of test sample into the vessel (for salted butter 2.5 g). Add 100 ml of boiling water and heat to boiling to dissolve the test portion. Cool the obtained suspension to below 55°C. Titrate (same method AOAC 2016.03 can be used). In the case of a difficult titration, the addition of 2 ml to 3 ml of the nitric acid (4 mol/l) to the contents of the vessel before titration is allowed. (By testing this in the collaborative study (in ANNEX of ISO 15648) it was shown that the same level of precision was attained.

If there were any questions, the laboratories were instructed to directly address them to the Study Director.

Timeline The results on the MLT samples were to be provided within 6 weeks from sample reception.

Reporting An electronic template was provided for data reporting (see Annex E). Participating laboratories were required to report AgNO₃ standardization data, as well as volumes of AgNO₃ used for titration of samples. Furthermore, detailed information on the different masses and volumes used during sample preparation as indicated in the method were requested. Laboratories that requires the additional protein precipitation steps [7.2-7.4] were asked to indicate which samples utilized these steps.

Laboratories were asked to report final results in mg chloride /100 g of sample “as received”, with three decimal points. This accounted for reconstitution factors.

RESULTS

Part 1. Method set up and qualification of participants

Participation Twenty three laboratories initially agreed to participate to the MLT. Five laboratories dropped out during Part 1 due to issues related to availability of resources or samples (customs). One laboratory was disqualified due to performance during Part 1.

Method set up All seventeen laboratories managed to easily set up the method as described in the protocol. Four laboratories used the extra protein precipitation steps as described in the method.

During the method set up, suitability of the standards and instrumentation were determined per the method. One laboratory required some assistance in obtaining silver nitrate at the correct concentration.

Practice samples results Four of the laboratories did not manage to receive the IDF samples due to Customs restrictions. One lab was asked late in 2016 to participate only in the IDF samples as customs was not an issue due to location.

After data compilation, average and standard deviation of repeatability and reproducibility were calculated. One laboratory reporting data above (average $\pm 2 * S_R$) was flagged as possible outlier and informed about it. Nevertheless, it was accepted to continue since the results, although questionable, were still within ($\pm 3 * S_{D_R}$). This laboratory was later dropped from the study as the final results were all high biased close to $3 * S_{D_R}$.

Full set of data is shown in Annex G.

Part 2. Multi Laboratory Test

Participation to MLT The remaining 17 laboratories reported valid data.

Results

Summarized results of the full MLT set of samples can be found in the table below. Full set of data is given in Annex G.

Requirements (SMPR 2014.015)			≤ 2 %	≤ 4 %	
Sample	n	Mean	RSD _r (%)	RSD _R (%)	HorRat (%)
Adult Nutritional Powder Low Fat	16	385.5	0.58	0.58	3.46
Adult Nutritional RTF High Fat	10	162.6	1.05	2.54	0.48
Adult Nutritional RTF High Protein	16	154.2	1.94	3.01	0.57
SRM 1849a	16	701.1	0.72	2.44	0.58
Toddler Formula Powder Milk Based	16	478.6	0.87	3.76	0.84
Infant Formula RTF Milk Based	15	42.4	0.94	1.21	0.19
Infant Formula Powder Soy Based	14	510.3	1.12	2.01	0.45
Infant Formula Powder Milk Based	15	413.7	1.76	3.49	0.76
Infant Formula Powder FOS/GOS Based	15	330.4	1.12	2.54	0.54
Infant Formula Powder Part Hyd Soy Based	15	386.1	0.73	4.08	0.88
Infant Formula Powder Part Hyd Milk Based	14	380.2	0.61	2.27	0.49
Infant Formula Powder Milk Based	13	357.3	1.00	3.67	0.78
Child Formula Powder Milk Based	16	443.6	0.44	3.56	0.79
Infant Elemental Powder	15	375.6	0.40	2.06	0.44
Adult Nutritional RTF High Fat – Placebo	8	35.41	3.14*	7.21*	1.09
Adult Nutritional RTF High Protein – Placebo	11	40.20	1.12	3.89	0.60
Child Formula Powder Milk Based – Placebo	11	32.4	12.4*	20.8*	3.11
Infant Formula RTF Milk Based – Placebo	16	22.5	1.49	3.50	0.49
Infant Elemental Powder – Placebo	16	358.9	0.52	2.24	0.48
IDF – Butter	12	375.5	1.68	3.47	0.75
IDF- Cheese	12	562.2	1.5	2.8	0.64
IDF - Whey Protein Concentrate	10	169.5	1.28	3.85	0.74
IDF – Whole Milk Powder	13	711.6	0.56	2.57	0.61
IDF – Whole Milk	11	95.3	1.37	2.72	0.48
IDF – Whey Powder	9	281.3	0.67	3.57	0.74

n = number of laboratories (after outliers removal)
RSD_r : relative standard deviation of repeatability
RSD_R : relative standard deviation of reproducibility

*Two samples showed much higher variability of results than the rest (Adult Nutritional RTF High Fat – Placebo and Child Formula Powder Milk Based – Placebo).

Ready-to-Feed products are much more sensitive to suffer from degradation during storage, being most likely the reason why the RTF Formula showed much higher than expected repeatability and reproducibility values. It was noted by some participants that this liquid product was no longer homogeneous.

The Child Formula Powder – Placebo may have had handling errors.

In addition, no significant differences could be observed between laboratories using the extra protein precipitation steps listed as optional from the combination of AOAC 2015.07 with 2015.08. See summary at the end of this report.

In general, the precision results (repeatability and reproducibility) are well within the limits stated in the SMPR. Repeatability ranged from 0.4 to 1.9 %, in accordance with data obtained during SLV¹, with reported RSD_r from 0.03 - 1.6 %. Meanwhile reproducibility ranged from 0.6 to 4.0 %. Finally the HorRat values are all below 1, from 0.2 to 0.9 %. Except for the two samples already discussed above.

Two of the IDF samples had reference values available to expand the accuracy of the method into milk matrixes. Butter had recoveries ranging from 96-108 %, and for Processed cheese recoveries ranged from 97-106 %.

CONCLUSIONS

Conclusions

The precision results (repeatability and reproducibility) obtained during the MLT showed that AOAC Official method 2016.03 comply with the requirements set in the corresponding SMPR 2014.015, and thus is fit for purpose for the analysis of chloride in infant formula and adult/pediatric nutritionals.

It is proposed to include the optional extra protein precipitation (regional requirement in China) conditions in the final method to increase method applicability in all laboratories. It has been shown that the results are not different provided that extra dilutions are respected.

Two samples (Adult Nutritional RTF High Fat – Placebo and Child Formula Powder Milk Based – Placebo) were suspected to have suffered from spoilage during sample storage, and or had handling errors. It is recommended to disregard them from the study, but not exclude these matrixes from approval of the method.

The Study Director recommends that this method should be proposed for AOAC Final Action Official Method status. In addition, the data for the six milk matrixes show that the method is also fit for purpose.

Acknowledgments

The Study Director sincerely thanks all the participating laboratories and their staff for the interest in the method and their valuable contribution to this study

ANNEXES

List of Annexes

Annex A: List of Materials
Annex B: Method
Annex C: List of participating laboratories
Annex D: Cover letters to participants
Annex E: Practice and MLT samples receipt forms
Annex F: Reporting templates
Annex G: Raw data MLT samples

Annex A: List of Materials

Practice Samples

Sample	Code	Reconstitution rate
Practice Sample A	CULF358	25 g + 200 g water
Practice Sample B	TJHR217	25 g + 200 g water

MLT Samples

MLT Samples Day1: run samples in this order and make sure all are analyzed by the end of Day 1

Sample	Code	Reconstitution rate
MLT Sample 1	DYLB360	not applicable
MLT Sample 2	KDOX966	25 g + 200 g water
MLT Sample 3	TJHR217	25 g + 200 g water
MLT Sample 4	GBZC169	25 g + 200 g water
MLT Sample 5	XKIP216	not applicable
MLT Sample 6	DOMY545	not applicable
MLT Sample 7	ATAN351	25 g + 200 g water
MLT Sample 8	ZMQM883	not applicable
MLT Sample 9	NSRB999	25 g + 200 g water
MLT Sample 10	MYHK654	25 g + 200 g water
MLT Sample 11	BFAO941	25 g + 200 g water
MLT Sample 12	OACN211	25 g + 200 g water
MLT Sample 13	JSDT587	25 g + 200 g water
MLT Sample 14	CULF358	25 g + 200 g water
MLT Sample 15	EFXN778	25 g + 200 g water
MLT Sample 16	HYJU890	not applicable
MLT Sample 17	SWUO667	25 g + 200 g water
MLT Sample 18	FPTE312	not applicable
MLT Sample 19	IDF 12	not applicable
MLT Sample 20	IDF 23	25 g + 200 g water
MLT Sample 21	IDF 34	25 g + 200 g water
MLT Sample 22	IDF 42	not applicable
MLT Sample 23	IDF 94	25 g + 200 g water
MLT Sample 24	IDF 123	25 g + 200 g water

MLT Samples Day2: run samples in this order and make sure all are analyzed by the end of Day 2

Sample	Code	Reconstitution rate
MLT Sample 25	DRWO880	25 g + 200 g water
MLT Sample 26	KGSZ273	25 g + 200 g water
MLT Sample 27	PZGP859	25 g + 200 g water
MLT Sample 28	IQVG111	not applicable
MLT Sample 29	LTCT316	25 g + 200 g water
MLT Sample 30	RQXQ518	25 g + 200 g water
MLT Sample 31	WKHN288	25 g + 200 g water
MLT Sample 32	XJDD334	not applicable
MLT Sample 33	GVPE615	25 g + 200 g water
MLT Sample 34	ARJT349	25 g + 200 g water
MLT Sample 35	UOPM297	25 g + 200 g water
MLT Sample 36	URTF231	25 g + 200 g water
MLT Sample 37	BYJK962	not applicable
MLT Sample 38	LYNY751	25 g + 200 g water
MLT Sample 39	SAEQ748	25 g + 200 g water
MLT Sample 40	YXBH789	25 g + 200 g water
MLT Sample 41	ECHL425	25 g + 200 g water
MLT Sample 42	YMZB323	not applicable
MLT Sample 43	TJMN542	not applicable
MLT Sample 44	VVOL664	not applicable
MLT Sample 45	IDF 65	25 g + 200 g water
MLT Sample 46	IDF 86*	not applicable
MLT Sample 47	IDF 51**	not applicable
MLT Sample 48	IDF 106*	not applicable
MLT Sample 49	IDF 115	25 g + 200 g water
MLT Sample 50	IDF 71**	not applicable

Annex B: Method

ISO/TC 34/SC 5 N

Date: 2016-01-27

ISO/WD

ISO/TC 34/SC 5/WG

Secretariat: NEN

Milk, milk products, infant formula and adult nutritionals — Determination of chloride — Potentiometric titration method

Élément introductif — Élément central — Élément complémentaire

Warning

This document is not an ISO International Standard. It is distributed for review and comment. It is subject to change without notice and may not be referred to as an International Standard.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

Copyright notice

This ISO document is a working draft or committee draft and is copyright-protected by ISO. While the reproduction of working drafts or committee drafts in any form for use by participants in the ISO standards development process is permitted without prior permission from ISO, neither this document nor any extract from it may be reproduced, stored or transmitted in any form for any other purpose without prior written permission from ISO.

Requests for permission to reproduce this document for the purpose of selling it should be addressed as shown below or to ISO's member body in the country of the requester:

[Indicate the full address, telephone number, fax number, telex number, and electronic mail address, as appropriate, of the Copyright Manager of the ISO member body responsible for the secretariat of the TC or SC within the framework of which the working document has been prepared.]

Reproduction for sales purposes may be subject to royalty payments or a licensing agreement.

Violators may be prosecuted.

Contents

1	Scope	18
2	Principle	18
3	Reagents	19
4	Preparation of solutions	19
5	Apparatus	20
6	Sample preparation	21
7	Extraction	21
8	Instrument operating conditions	22
9	System suitability test	23
10	Calculations	23
11	Precision	24
12	Test report	24
Annex A (informative)		25
Annex B (informative)	Error! Bookmark not defined.	

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO was prepared by Technical Committee ISO/TC 34, *Food analysis*, Subcommittee SC 5, *Milk and milk products*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

Milk, milk products, infant formula and adult nutritionals — Determination of chloride — Potentiometric titration method

1 Scope

This International Standard specifies a method for the determination of chloride in milk, milk powder, whey powder, infant formula and adult nutritionals by potentiometry, with an analytical range of 0,35 mg to 1 060 mg chloride/100 g reconstituted product, or ready-to-feed liquids).

2 Principle

Reconstitute powder samples by dissolving 25 g powder sample in 200 g warm water (40 ° C), while ready-to-feed products (RTF) are used as-is. Precipitate proteins by adding precipitation reagents I and II and centrifuge. Acidify the supernatant with nitric acid solution. Titrate chloride ions against standardized silver nitrate solution, 0,1 mol/l, potentiometrically using a silver electrode to detect the end point.

3 Reagents

3.1 Water, purified, greater than 18 MΩ (EMD Millipore²) Corp., Billerica, MA, USA, or equivalent).

3.2 Sodium chloride (NaCl), purity ≥ 99,5 %, certified reference material for titrimetry, certified by BAM, according to ISO 17025—Sigma Aldrich #71387¹⁾ or equivalent.

3.3 Silver Nitrate (AgNO₃), meets analytical specification of Ph. Eur., BP, USP, assay 99,8 % -100,5 %, Sigma-Aldrich 10220¹⁾, or equivalent.

3.4 Potassium ferrocyanide trihydrate (K₄Fe(CN)₆ · 3H₂O), puriss. p.a., ACS reagent, reag. ISO, reag. Ph. Eur., ≥ 99%, Sigma-Aldrich # 31524¹⁾ or equivalent.

3.5 Zinc acetate dihydrate Zn(CH₃COO)₂ · 2H₂O, ACS reagent puriss p.a., ≥ 99,0 %. Sigma Aldrich # 96459¹⁾ or equivalent.

3.6 Nitric acid (HNO₃), minimum 65 % p.a., Merck #100452¹⁾, or equivalent.

3.7 Standardized AgNO₃ solution, 0,1 mol/l Titripur® Reag. Ph Eur,Reag. USP, # 1.09081.1000 or EM3214-1, or ready-to-use standardized titrant prepared according to GB/T 601¹⁾, or equivalent.

3.8 Sodium chloride (NaCl) standardized solution, 0,100 mol/l $\pm 0.5\%$ | U-CTS1)Alfa Aesar¹⁾, # 35616, (Ward Hill, MA, USA), or equivalent.

3.9 Glacial acetic acid, 100 %, anhydrous for analysis EMSURE® ACS, ISO, Reag. Ph Eur MERCK, # 100063¹⁾ or equivalent.

3.10 Potassium nitrate, (KNO₃), for analysis EMSURE® ISO,Reag. Ph EurMERCK, # 105063¹⁾ or equivalent.

3.11 Acetone, for cleaning of the electrode. Honeywell, # 010-4, (Muskegon, MI, USA) ¹⁾ or equivalent.

3.12 Dimethylpolysiloxane, defoaming agent. Sigma-Aldrich, #DMPS2C¹⁾ or equivalent.

4 Preparation of solutions

4.1 Standardized AgNO₃ solution, 0,1 mol/l.

If ready-to-use AgNO₃ standard solution (3.3) is not available, then weigh 16,9890 g ± 0,0005 g AgNO₃ previously dried for 2 h at 120 ± 2°C. Dissolve in water and make up to the mark in a 1 000 ml volumetric flask. Store in a brown reagent bottle.

² This is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by either ISO or IDF of the product named. Equivalent products may be used if they can be shown to lead to the same results.

After preparation, check the titer by titration of 5,0 ml with exactly 0,1 mol/l NaCl solution. For either commercial or in-house solution verify the titer on a regular basis. The standardized AgNO₃ solution shall be protected from light, and can be stored for up to 2 months.

4.2 Sodium chloride solution, 0,1 mol/l.

If ready-to-use NaCl standard solution is not available, weigh 5,8440 g ± 0,0005 g NaCl (3.2), previously dried for 2 h at 110 ± 2°C. Dissolve in water and make up to the mark in a 1000 ml volumetric flask.

This solution is stable for up to 1 month.

4.3 Precipitating solution (Carrez) I.

Weigh 106 g potassium ferrocyanide trihydrate, (3.4) dissolve in an appropriate amount of water and transfer into a 1000 ml volumetric flask. Make up to the mark using water. Mix well.

4.4 Precipitating solution (Carrez) II.

Weigh 220 g zinc acetate dihydrate (3.5) and transfer into a 1000 ml volumetric flask. Dissolve with an appropriate amount of water, and add 30 ml glacial acetic acid. Make up to the mark using water. Mix well.

4.5 Nitric acid solution.

With care, add 100 ml concentrated nitric acid (3.6) to 300 ml water. Mix well.

4.6 Wash solution.

According to autosampler/titrator manufacturer's instructions. (e.g. Acetone, Nitric acid solution (4.5), or other).

4.7 AgNO₃ solution, 0,025 mol/l (optional).

Into a 1 000 ml volumetric flask, pipet 250 ml AgNO₃ solution, 0,1 mol/l (3.3 or 4.1). Make up to the mark with water. Prepare freshly before use. Then check the titer by titration of 25 ml against 0,025 mol/l NaCl solution.

4.8 NaCl solution, 0,025 mol/l (optional).

Into a 100 ml volumetric flask pipet 25 ml NaCl solution, 0,1 mol/l (4.2). Make up to the mark with water. Prepare freshly before use.

4.9 KNO₃ solution, 1 mol/l.

Weigh 10,11 g potassium nitrate (3.10) into a 100 ml volumetric flask. Add about 80 ml water and place it in an ultrasonic cleaner (5.12) to dissolve with ultrasound and heating until dissolved thoroughly. Cool down to room temperature and make up to the mark with water. Filter using a 0,45 membrane disposable syringe before use.

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Analytical balance, precision [J-CTS2]0,1 mg.

5.2 Centrifuge, table-top with rotor for 50 ml conical tubes, capable of operating at $\geq 12\ 000\ g$.

5.3 Centrifuge tubes, 50 ml, conical, polypropylene.

5.4 Pipettes, 1 ml, 10 ml, 20 ml, 50 ml and 100 ml, Class A glass volumetric or automatic (Eppendorf or equivalent).

5.5 One-mark volumetric flasks, 50 ml, 100 ml, 500 ml, and 1 000 ml glass, Class A.

Graduated cylinders, 25 ml, 100 ml and 500 ml, glass.

5.6 Autosampler Beaker, e.g. 120 ml, depending on the titrator used.

5.7 pH-meter or mV-meter, with a scale covering $\pm 700\ mV$, and burette, 20 ml or 25 ml.

5.8 Automatic titrator, (autosampler, motorized piston burette, with remote-control dispensing and filling).

Mettler T50, Roundo Tower autosampler, MettlerLabX 3.1 software or Metrohm 862 Compact Titrosampler, 800 Dosino, 10 ml Exchange Unit, or equivalent). Alternatively a semi-automated (e.g. MetrohmTitrado 905/907, with MetrohmTiamo™ software or equivalent) or manual titrator (using a burette with accuracy of 0,01 ml) may be used.

5.9 Combined ring silver electrode, e.g. Mettler DM 141 or DMi145-SC, Metrohm Ag Titrode 6.0430.100 or equivalent, alternatively a silver electrode with reference electrode.

5.10 Magnetic stirrer, Heidolph MR 3000, or Metrohm 804Ti Stand with 802 Rod Stirrer, or equivalent.

5.11 Water bath, Labotech DWM 16 or equivalent.

5.12 Ultrasonic cleaner, Tianjin Auto Science AS2060B or equivalent.

5.13 Disposable syringe, 3 ml, with handspike and 0,45 μm disposable syringe filter.

6 Sample preparation

Mix well to ensure that sample is homogeneous. Reconstitute powder samples by dissolving 25 g powder sample in 200 g [J-CTS3] warm water (40 °C).

7 Extraction

7.1 General

Optionally, based on regional requirements [J-CTS4] for additional protein precipitation than that accomplished by addition of nitric acid solution perform 7.2 to 7.4. Otherwise begin with 7.5.

7.2 Weigh an appropriate aliquot of ready-to-feed (RTF) or reconstituted powder (e.g. 25 g) (accurate to 0,1 mg) into a 50 ml centrifuge tube. For

samples with a high chloride content, weigh a smaller test portion e.g. 5 g of reconstituted or RTF product.

7.3 Transfer 2,5 ml precipitating solution I (4.3) and 2,5 ml precipitating solution II (4.4) into the tube. Complete to 50 ml with water. Mix well. If foam impacts the constant volume, one or two drops of defoaming agent (3.12) shall be used.

7.4 Centrifuge at 12 000 *g* for 5 min at 4°C. Then equilibrate to room temperature.

7.5 Accurately transfer either 10 ml supernatant from steps 7.2 to 7.4 or weigh an appropriate aliquot of RTF or reconstituted powder (e.g. 25 g accurate to 0,1 mg). For samples with a high chloride content, weigh a smaller test portion e.g. 5 g of reconstituted or RTF product.

Into a 120 ml sample beaker or autosampler cup. Add 5 ml nitric acid solution (4.5), and 50 ml of water before titration. Add a magnetic stirring rod (if the titrator does not have a build-in rod stirrer). Place the autosampler cup or beaker onto a magnetic stirrer and stir until dissolved or finely suspended.

7.6 The pH of the test solution shall be below 1,5. In case of any doubts, check by means of a pH-meter and, if necessary, add a little more nitric acid solution (4.5).

8 Instrument operating conditions

8.1 Check and maintenance of the combined silver electrode

Rinse electrode with deionized water and wipe before use. Renew the electrolyte 1 mol/l KNO₃ (4.9) periodically per manufacturer's recommendations. If fat sticks to the electrodes during a series of analyses, then eliminate it by briefly immersing the electrode in acetone. The silver electrode shall be stored in 1 mol/l KNO₃ (4.9) or per manufacturer's recommendations after appropriate cleaning.

NOTE: In place of the combined silver electrode, separate silver and reference electrodes may also be used.

8.2 Titration

Connect the combined silver electrode to the titration apparatus, according to the manufacturer's indications. Ensure that the titration vessels are correctly placed on the autosampler and there are enough reagents: both nitric acid solution (if added automatically) (4.5) and 0,1 mol/l AgNO₃ (3.7 or [J-CTS] 4.1). If no autosampler is available, then place the sample solutions manually under the titration equipment.

Put the wash solution (4.6) in the washing position if an auto sampler used. Ensure that the volume of wash solution is adequate.

Under continuous stirring and without touching the electrode, titrate the sample solution automatically with 0,1 mol/l standardized silver nitrate solution (3.7 or [J-CTS] 4.1), up to the end potential. The consumption of 0,1 mol/l of silver nitrate solution should be recorded automatically and can be read from the titrator software, or documented in the titrator operating records. For manual titration, using a burette add 0,1 mol/l standardized silver nitrate solution, until the end potential has nearly been met. Then continue to titrate slowly until the end point is met as observed by the two small (about 0,05 ml) silver nitrate solution.

Special case: determination of very low amounts of chloride

When determining low chloride concentrations, like desalted whey powder, for greater precision it is preferable to use a standardized 0,025 mol/l AgNO₃ (4.7) solution for the titration.

8.3 Blank test: determination of reagent background content of chloride

Perform a blank test using reagents substituting water (3.1) for the sample portion. The titrant consumption of the blank test obtained at the endpoint shall be less than 0,05 ml for using the 0,1 mol/l standardized silver nitrate and 0,2 ml when using the 0,025 mol/l standardized silver nitrate. Otherwise check the reagents and water involved in the procedures, and perform the blank test again until the criteria is achieved.

9 System suitability test

Prior to use, transfer 5 ml of NaCl solution (3.8) or (4.2) into a 120 ml sample beaker. If 0,025 mol/l AgNO₃ titrant is required then use 1 ml NaCl solution. Add 5 ml nitric acid solution (4.5) and 50 ml water. Place the washing solution (4.6) in the washing position of the auto sampler. Titrate using an automatic, semi-automatic or manual titrator. Repeat in triplicate.

Calculate concentration of the silver nitrate solution according to Clause 10. The difference between the calculated concentration and the certified value should be within 0,5 %. If outside the acceptance value, check the experimental procedures and titration system. If issue is not resolved, then use fresh standardized silver nitrate. If fresh standardized silver nitrate does not provide an acceptable result, replace the electrolyte of the electrode and check the operating condition of the dosing unit.

10 Calculations

Calculate the concentration of silver nitrate, c_{snc} , in mol/l, for system suitability verification and report to 4 decimal places.

If using 4.2:

$$c_{snc} = \frac{m_1}{5,844 \times V_1 \times 10} \quad (1)$$

where

- m_1 = mass of sodium chloride in 5 ml or 1 ml of (4.2) standard solution (in mg);
- V_1 = volume of 0,1 mol/l or 0,025 mol/l AgNO₃ consumed at titration endpoint (in ml);
- 5,844 = sodium chloride mass in µg corresponding to 1 ml of 0,1 mol/l AgNO₃;
- 10 = mass conversion from titer to the concentration of titrant.

Or if using a purchased standard grade 0,1 mol/l NaCl (3.8):

$$c_{snc} = \frac{0,1 \times V_3}{V_1} \quad (2)$$

where

- V_3 = volume of purchased standard grade 0,10 mol/l sodium chloride added (in ml);
- V_1 = volume of 0,1 mol/l or 0,025 mol/l AgNO₃ consumed at titration endpoint (in ml).

Calculate the chloride mass fraction, w_{cl} , in mg/100 g the sample and report to 3 significant digits:

$$w_{cl} = \frac{35,45 \times c \times (V_2 - V_0) \times f \times 100}{m} \quad (3)$$

where

- m = the sample mass (in g);
- c = the certified concentration of silver nitrate titrant (0,1000 mol/l or standardized concentration (Formula 1));
- V_2 = the volume of AgNO₃ consumed at titration endpoint (in ml);

- V_0 = the volume of AgNO_3 consumed at titration endpoint for blank (8.3)(in ml);
- f = the dilution factor for preparation of reconstituted powder, RTF or concentrate. For samples requiring the protein precipitation step (7.2 to 7.4) and additional factor (e.g. for 25 g sample $f = 2$) is needed;
- 35,45 = chloride mass in μg corresponding to 1 ml of 1 mol/l AgNO_3 ;
- 100 = the mass conversion to mg/100 g.

11 Precision

11.1 Repeatability

Under the repeatability analysis condition, the absolute difference between two independent test results shall not exceed 2 % of the arithmetic mean.

To be completed.

11.2 Reproducibility

To be completed.

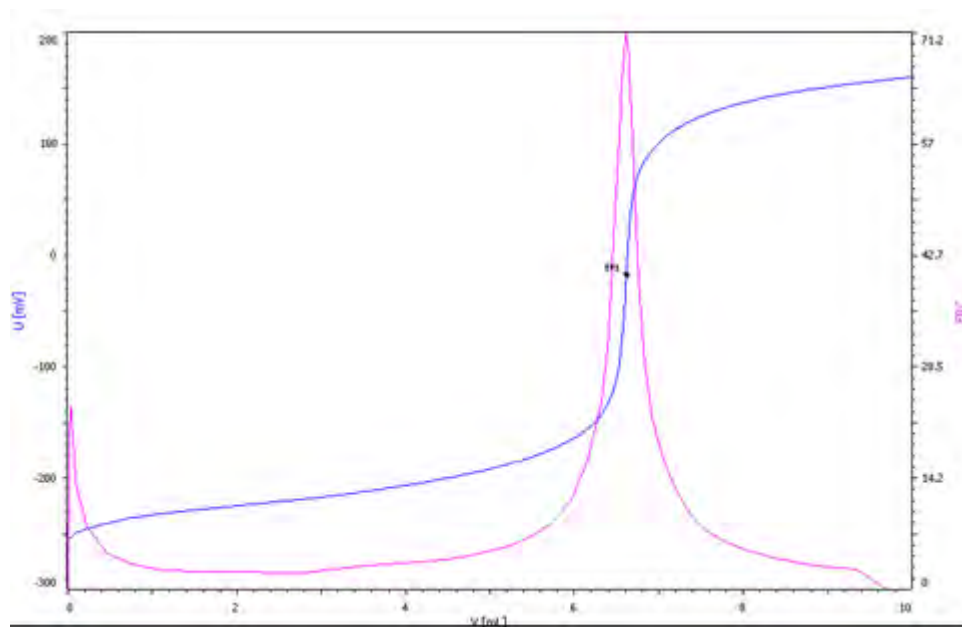
12 Test report

The test report shall at least include the following information:

all information necessary for the identification of the sample (type of sample, origin and designation of the sample); a reference to this International Standard; the date and type of sampling procedure (if known); the date of receipt; the date of test; the test results and the units in which they have been expressed; any operations not specified in the method or regarded as optional, which might have affected the results.

(informative)

Examples of titration end point determination



Key

U(mV) the voltage of Ag electrode detected during titration
V(ml) the volume of consumption of the standardized AgNO_3 titrant during titration

ERC the first derivative of the titration curve drawn by voltage of electrode versus volume of titrant consumption

Figure A.1 — Automatic titration endpoint recognition using dynamic titration (DET U) mode on Methohm Titrodo 905

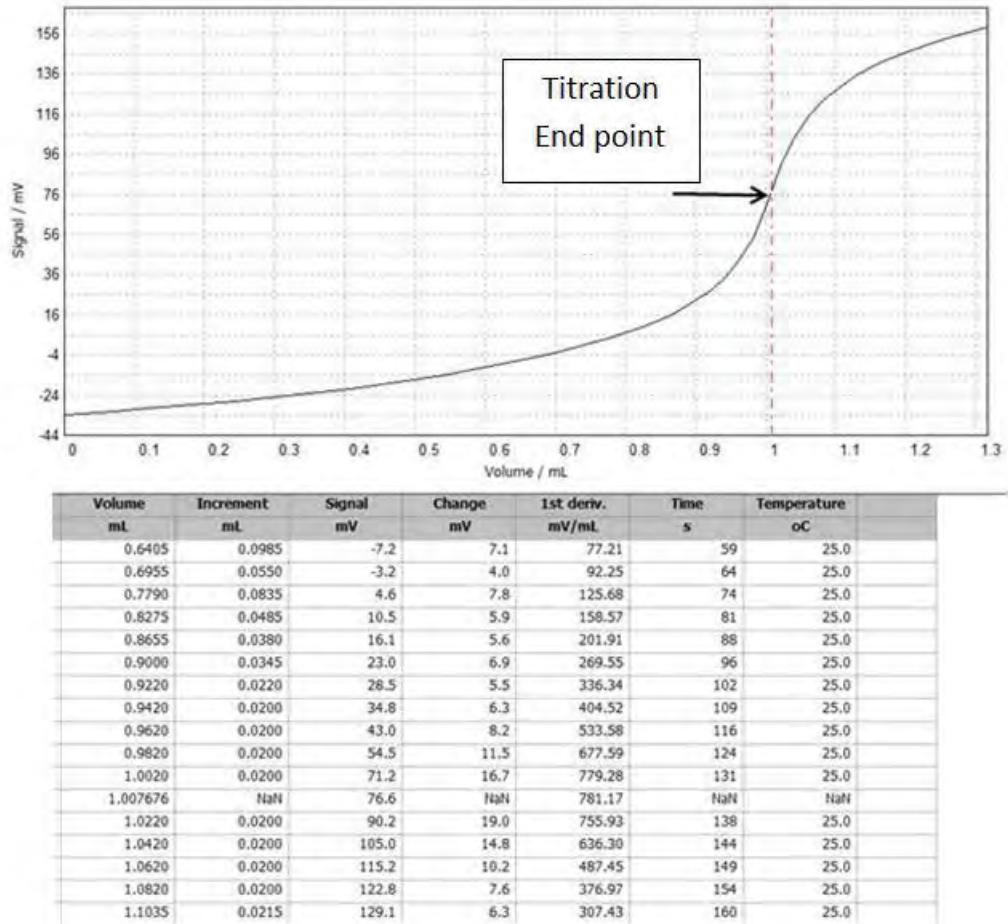


Figure A.2 — Example of a titration curve from Mettler autotitrator

Bibliography

- [1] AOAC SMPR 2014.015
- [2] AOAC Official Method 2015.07. Chloride in infant formula and adult/pediatric nutritional formula: Potentiometric titration method, 1st action 2015
- [3] AOAC Official Method 2015.08. Chloride in infant formula and adult/pediatric formula: Potentiometry, 1st action 2015
- [4] GB/T 601-2002 Chemical reagent – Preparation of standard volumetric solutions
- [5] AOAC Official Method 2016.03^[J-CTS8]. Chloride in infant formula and adult/pediatric nutritional formula: Potentiometric titration method, 1st action 2016

Annex C: List of participants

Laboratory	Country
NQAC Shah Alam Nestlé Malaysia Jalan Playar 15/1 P.O.Box 7010, Shah Alam – Selangor 40700	Malaysia
Nestle Quality Assurance Center - São Paulo Av. Guido Caloi, 1935 - 2º andar - Bloco A - Piso 3 Sao Paulo, 05802-140	Brazil
ASKEATON, Wyeth Nutrition Co. Limerick, Askeaton	Ireland
FrieslandCampina LQS FrieslandCampina Laboratory and quality services P. Stuyvesantweg 1, Leeuwarden 8937 AC	Netherlands
Nestle Quality Assurance Center – Dublin, OH Nestle 6625 Eitermand Rd Dublin, OH 43017	USA
Covance NCFs Singapore The Synergy #05-13, 609917	Singapore
Mead Johnson Nutrition 2400 W. Lloyd Expressway Evansville, IN 47721	USA
Eurofins CLF Specialised Nutrition Testing Services GmbH Professor-Wagner-Str. 11 D-61381 Friedrichsdorf, Germany	Germany
Eurofins Nutrition Analysis Center 2200 Rittenhouse Street, Ste 150 ; Des Moines IA	USA
Ausnutria Hyproca Runderweg 6, 8219 PK Lelystad, The Netherlands	Netherlands
Qlip Oostzeestraat 2a 7202 CM Zutphen	Netherlands
Eurofins NZ Laboratory Services Ltd. 35 O’Rorke Road Penrose, Auckland 1061	New Zealand
Guangzhou Quality Supervision and Testing Institute(GQT)	China
Shandong Technical Center of Inspection&Quarantine 70 Qutangxia Road, Qingdao, ShanDong province, China,Post Code: 266001	China
Analysis Center Laboratory of Inner Mongolia Mengniu Dairy (Group) Co., Ltd.	China
Technical Center For Animal Plant And Food Inspection And Quarantine in Shanghai Entry-Exit Inspection and Quarantine Bureau	China
Inner Mongolia Yili Industrial Group Co.,Ltd.	China

Annex D: Cover letters to participants

Cover letter:

- Chloride in Milk, Milk Powder, Whey Powder, Infant Formula and Adult Nutritionals Potentiometric titration method: Multi Laboratory Test to validate the combined First Action method AOAC 2016.03 (combined AOAC Official Method 2015.07 and 2015.08).

Greg Jaudzems greg.jaudzems@us.nestle.com
Ms. Wan Xin wanxin@caiqtest.com

Ms. Tian Yan tianyan@caiqtest.com

Dear participant,

Thank you for your participation in one or more of the three above multi-laboratory testing (MLT) studies. These studies are a collaborative effort between AOAC INTERNATIONAL and ISO/IDF (International Organization for Standardization/International Dairy Federation) The objective of the study is to validate each method by determining its precision figures (repeatability and reproducibility) with MLT.

The repeatability and reproducibility attained for the methods will be compared against the AOAC SMPR to ascertain whether this method is suitable for its intended use as a best-in-class global dispute resolution method.

In addition to the AOAC SPIFAN matrixes, this study will also include IDF Dairy matrixes with the plan to be able to expand the scope of the combined method based on collected data. IDF Dairy matrixes will be sent separately from the AOAC SPIFAN matrixes.

We would appreciate it if you could ensure that the contents of the parcel has not been damaged during transit, and then complete the enclosed Samples Receipt Form and send to the Study director above per the method you are participating in via email. **IDF powder samples must be kept at room temperature, away from direct sunlight. Once open, the containers must be kept in tightly closed containers away from direct sunlight for the duration of the study. The IDF liquid milk and cheese need to be stored at 6 °C ± 2 °C upon arrival and for the duration of the study. The IDF butter needs to be stored at -20 °C upon arrival and for the duration of the study.** AOAC SPIFAN matrixes will have a separate Receipt Form.

Please do not start to test the enclosed samples until directed to do so by your Study Director (they are not test samples); then closely follow the protocol instructions sent to you electronically for analysis of the samples and data reporting. If you have any questions, do not hesitate to contact us for assistance.

Your participation in this Multi-Laboratory Testing study is greatly appreciated.

Yours sincerely,

Greg Jaudzems

Continued on next page

Annex E: Receipt Forms

Samples Receipt Form

Chloride AOAC 2016.03 MLT Samples Receipt Form

Participant: «Contact_person» _____
 «Laboratory» _____
 «Address» _____
 «City» «Country» _____

Please ensure the items listed below have been received undamaged, insert date of receipt, and circle the relevant statement:

Contents of parcel

	Code
Practice Sample A	CULF358
Practice Sample B	TJHR217

		Code
Day 1	MLT Sample 1	DYLB360
	MLT Sample 2	KD08966
	MLT Sample 3	TJHR217
	MLT Sample 4	GBZC169
	MLT Sample 5	8KIP216
	MLT Sample 6	DOMY545
	MLT Sample 7	ATAN351
	MLT Sample 8	ZMQM883
	MLT Sample 9	NSRB999
	MLT Sample 10	MYHK654
	MLT Sample 11	BFA0941
	MLT Sample 12	OACN211
	MLT Sample 13	JSDT587
	MLT Sample 14	CULF358
	MLT Sample 15	EFXN778
	MLT Sample 16	HYJU890
	MLT Sample 17	SWU0667
	MLT Sample 18	FPT2312
	MLT Sample 19	IDF 12
	MLT Sample 20	IDF 23
	MLT Sample 21	IDF 34
	MLT Sample 22	IDF 42
	MLT Sample 23	IDF 94
	MLT Sample 24	IDF 123

		Code
Day 2	MLT Sample 25	DRW0880
	MLT Sample 26	KGS2273
	MLT Sample 27	P2GP859
	MLT Sample 28	IQVG111
	MLT Sample 29	LTCT316
	MLT Sample 30	RQW0518
	MLT Sample 31	WKHN288
	MLT Sample 32	8JDD334
	MLT Sample 33	GVPE615
	MLT Sample 34	ARJT349
	MLT Sample 35	UOPM297
	MLT Sample 36	URTF231
	MLT Sample 37	BYJK962
	MLT Sample 38	LYNY751
	MLT Sample 39	SAEQ748
	MLT Sample 40	Y8BH789
	MLT Sample 41	ECHL425
	MLT Sample 42	YH2B323
	MLT Sample 43	TJMN542
	MLT Sample 44	VVOL664
	MLT Sample 45	IDF 65
	MLT Sample 46	IDF 86
	MLT Sample 47	IDF 51
	MLT Sample 48	IDF 106
	MLT Sample 49	IDF 115
	MLT Sample 50	IDF 71

Date of Receipt _____

All items have been received undamaged Yes / No

Items are missing/damaged Yes / No

List missing/damaged items needing replacement, if a whole series is not needed:

I require an additional series of samples Yes / No

Please fax or email the completed form to:

Greg Jaudzems

Fax: +001 480 379 6639

greg.jaudzems@us.nestle.com

Continued on next page

Annex F: Reporting templates

AgNO₃ Standardization

Chloride AOAC 2016.03 - MLT Chloride in Infant Formula and Adult Nutritionals Potentiometric titration method.			
Name:			
Compagny/Institution:			
Country:			
Wt. NaCl (g) of 4.2 =			
Wt. NaCl (mg) in 5mL of 4.2 = m_1 =			
AgNO ₃ standardization using in-house made 0.100 NaCl			
	mL ~0.1 M AgNO ₃ (V_1)	AgNO ₃ concentration	Final AgNO ₃ to use for [10,3] calculations (c)
Standardization 1			Average mol/l of AgNO ₃ =
Standardization 2			
Standardization 3			
Standardization 4			
AgNO ₃ standardization using purchased certified 0.1000 NaCl			
	mL ~0.1 mol/l AgNO ₃	mol/l AgNO ₃	Final AgNO ₃ to use for [10,3] calculations (c)
Standardization 1			Average mol/l of AgNO ₃ =
Standardization 2			% difference from cert. value =
Standardization 3			
Standardization 4			

Practice samples

Chloride in Infant Formula and Adult Nutritionals Potentiometric titration method. AOAC 2016.03 MLT											
Name:											
Laboratory:											
Country:											
										Certified or standardized concentration of AgNO ₃ 0.1000 M	
Code	Sample reconstitution		Titration	protein precipitation step [7.2-7.4]		f		Volume AgNO ₃		Practice samples results	
	Sample (g)	Water (g)	Mass (g)	if not required enter "1"		sample	duplicate	sample	duplicate	Chloride (as reconstituted)	Chloride (as received)
	m1	m2	m	sample	duplicate						
Practice Sample A	CULF358			1	1						
Practice Sample B	TJHR217			1	1						

Annex F: Reporting templates, Continued

Chloride 200C 2016.03 - MLT Chloride in Infant Formula and Adult Nutritional Polymerization Titration method.																			
Name										Coulter or standard concentration of K_2CrO_7									
Reference										0.1000 M									
Sample																			
Day	MIT Range	Code	Range concentration		Titrant	garden quantity along (litre)		Final volume used											
			Range 1	Range 2		Initial	Final	Range 1	Range 2	Range 3									
			ml	ml		ml	ml	ml	ml	ml									
Day 1	MIT Range 1	848258	not applicable	not applicable			1.0												
MIT Range 2	888266						1.0												
MIT Range 3	7788217						1.0												
MIT Range 4	888269						1.0												
MIT Range 5	882316	not applicable	not applicable				1.0												
MIT Range 6	888268	not applicable	not applicable				1.0												
MIT Range 7	8788281						1.0												
MIT Range 8	888268	not applicable	not applicable				1.0												
MIT Range 9	888269						1.0												
MIT Range 10	888269						1.0												
MIT Range 11	888269						1.0												
MIT Range 12	888269						1.0												
MIT Range 13	888269						1.0												
MIT Range 14	888269						1.0												
MIT Range 15	888269						1.0												
MIT Range 16	888269	not applicable	not applicable				1.0												
MIT Range 17	888269						1.0												
MIT Range 18	888269	not applicable	not applicable				1.0												
MIT Range 19	888269	not applicable	not applicable				1.0												
MIT Range 20	888269						1.0												
MIT Range 21	888269						1.0												
MIT Range 22	888269	not applicable	not applicable				1.0												
MIT Range 23	888269						1.0												
MIT Range 24	888269						1.0												
MIT Range 25	888269						1.0												
MIT Range 26	888269						1.0												
MIT Range 27	888269						1.0												
MIT Range 28	888269	not applicable	not applicable				1.0												
MIT Range 29	888269						1.0												
MIT Range 30	888269						1.0												
MIT Range 31	888269						1.0												
MIT Range 32	888269	not applicable	not applicable				1.0												
MIT Range 33	888269						1.0												
MIT Range 34	888269						1.0												
MIT Range 35	888269						1.0												
MIT Range 36	888269	not applicable	not applicable				1.0												
MIT Range 37	888269	not applicable	not applicable				1.0												
MIT Range 38	888269	not applicable	not applicable				1.0												
MIT Range 39	888269	not applicable	not applicable				1.0												
MIT Range 40	888269						1.0												
MIT Range 41	888269						1.0												
MIT Range 42	888269	not applicable	not applicable				1.0												
MIT Range 43	888269	not applicable	not applicable				1.0												
MIT Range 44	888269	not applicable	not applicable				1.0												
MIT Range 45	888269	not applicable	not applicable				1.0												
MIT Range 46	888269	not applicable	not applicable				1.0												
MIT Range 47	888269	not applicable	not applicable				1.0												
MIT Range 48	888269	not applicable	not applicable				1.0												
MIT Range 49	888269						1.0												
MIT Range 50	888269	not applicable	not applicable				1.0												

* Not applicable to the first 2 of the points.
 * Not applicable to the first 2 of the points.

Annex G: Raw data MLT samples

All results for both SPIFAN and IDF samples. Results in orange were omitted from the statistics.

Adult Nutritional Powder Low Fat		
Lab	LYNY751	PZGP859
1	386.25	385.07
2	387.91	386.32
3	383.56	388.76
4	388.51	386.66
5	396.67	393.25
6	382.55	385.32
7	381.93	380.74
8	386.68	389.36
9	383.10	382.53
10	385.96	385.31
11	383.72	383.95
12	416.29	408.48
13	380.19	380.18
14	345.39	343.88
15	390.06	393.01
16	394.61	389.48
Total number of laboratories	p	16.00
Total number of replicates	Sum(n(L))	32.00
Overall mean of all data (grand mean)	XBARBAR	385.49
Repeatability standard deviation	s(r)	2.25
Reproducibility standard deviation	s(R)	13.34
Repeatability relative standard deviation	RSD(r)	0.58
Reproducibility relative standard deviation	RSD(R)	3.46
HORRAT value		0.75

Adult Nutritional RTF High Fat (liq.)		
Lab	DYLB360	ZMQM883
1	163.31	163.91
2	170.69	167.93
3	162.26	165.36
4	156.64	158.64
5	167.23	167.26
6	105.80	102.71
7	155.51	158.13
8	158.59	125.02
9	65.49	73.26
10	141.95	
11	162.01	165.16
12	157.35	161.12
13	162.73	160.65
14	164.03	163.10
15	107.56	120.14
16	128.30	51.15
Total number of laboratories	p	10.00
Total number of replicates	Sum(n(L))	20.00
Overall mean of all data (grand mean)	XBARBAR	162.65
Repeatability standard deviation	s(r)	1.70
Reproducibility standard deviation	s(R)	4.14
Repeatability relative standard deviation	RSD(r)	1.05
Reproducibility relative standard deviation	RSD(R)	2.54
HORRAT value		0.48

Adult nutritional ready to feed – high fat placebo		
Lab	IQVG111	VVOL664
1	36.96	36.87
2	41.30	39.24
3	98.32	93.68
4	31.09	31.43
5	37.29	34.25
6	23.35	22.11
7	38.46	39.06
8	33.97	30.80
9	28.32	33.94
10		82.57
11	33.98	32.38
12	39.66	44.65
13	36.04	36.01
14	36.22	38.30
15	33.19	34.96
16	32.19	23.15
Total number of laboratories	p	8.00
Total number of replicates	Sum(n(L))	16.00
Overall mean of all data (grand mean)	XBARBAR	35.41
Repeatability standard deviation	s(r)	1.11
Reproducibility standard deviation	s(R)	2.55
Repeatability relative standard deviation	RSD(r)	3.14
Reproducibility relative standard deviation	RSD(R)	7.21
HORRAT value		1.09

Adult Nutritional RTF High Protein (liq.)		
Lab	DOMY545	FPTE312
1	152.58	154.97
2	162.54	159.15
3	151.17	152.91
4	152.55	152.77
5	158.69	157.11
6	152.96	152.57
7	153.46	155.41
8	155.77	155.39
9	152.59	152.05
10	147.54	152.23
11	155.80	155.64
12	160.54	163.56
13	153.34	153.66
14	154.03	154.25
15	155.26	155.37
16	151.71	136.65
Total number of laboratories	p	16
Total number of replicates	Sum(n(L))	32
Overall mean of all data (grand mean)	XBARBAR	154.19
Repeatability standard deviation	s(r)	2.99
Reproducibility standard deviation	s(R)	4.65
Repeatability relative standard deviation	RSD(r)	1.94
Reproducibility relative standard deviation	RSD(R)	3.01
HORRAT value		0.57

Adult nutritional ready to feed – high protein placebo

Lab	YMZB323	BYJK962
1	40.43	36.86
2	38.81	37.52
3	94.75	99.75
4	37.75	37.77
5	40.68	40.08
6	30.11	29.85
7	42.37	41.89
8	39.97	39.99
9	40.19	39.61
10	38.95	38.08
11	40.84	40.65
12	49.95	47.61
13	41.69	40.82
14	41.17	40.99
15	42.04	42.57
16	41.73	49.90

Total number of laboratories	p	11.00
Total number of replicates	Sum(n(L))	22.00
Overall mean of all data (grand mean)	XBARBAR	40.20
Repeatability standard deviation	s(r)	0.45
Reproducibility standard deviation	s(R)	1.56
Repeatability relative standard deviation	RSD(r)	1.12
Reproducibility relative standard deviation	RSD(R)	3.89
HORRAT value		0.60

Toddler Formula Powder Milk-Based

Lab	EFXN778	BFAO941
1	470.91	486.86
2	475.83	470.29
3	480.80	493.48
4	471.99	470.81
5	489.55	491.87
6	477.12	480.13
7	475.77	475.66
8	479.53	480.51
9	470.22	474.63
10	483.47	480.42
11	474.61	477.15
12	515.86	512.39
13	476.08	474.73
14	423.49	426.57
15	486.57	490.19
16	491.39	486.69

Total number of laboratories	p	16.00
Total number of replicates	Sum(n(L))	32.00
Overall mean of all data (grand mean)	XBARBAR	478.61
Repeatability standard deviation	s(r)	4.17
Reproducibility standard deviation	s(R)	17.97
Repeatability relative standard deviation	RSD(r)	0.87
Reproducibility relative standard deviation	RSD(R)	3.76
HORRAT value		0.84

Infant Formula RTF Milk Based (liq.)

Lab	XKIP216	HYJU890
1	42.06	42.69
2	43.66	41.99
3	94.84	99.28
4	42.10	42.22
5	42.75	42.79
6	41.90	41.96
7	41.78	41.86
8	42.28	42.16
9	41.26	41.37
10	42.09	42.18
11	41.03	42.08
12	45.14	45.59
13	41.81	42.19
14	41.86	41.83
15	43.10	42.95
16	42.67	42.62

Total number of laboratories	p	15
Total number of replicates	Sum(n(L))	30
Overall mean of all data (grand mean)	XBARBAR	42.40
Repeatability standard deviation	s(r)	0.40
Reproducibility standard deviation	s(R)	0.99
Repeatability relative standard deviation	RSD(r)	0.94
Reproducibility relative standard deviation	RSD(R)	2.34
HORRAT value		0.36

Infant Formula Powder Soy Based

Lab	TJHR217	OACN211
1	501.01	507.10
2	525.42	516.52
3	498.04	524.53
4	507.78	507.19
5	516.52	518.37
6	505.70	506.16
7	503.84	504.06
8	508.50	507.65
9	497.93	499.74
10	409.25	506.83
11	503.83	503.25
12	530.45	538.07
13	500.94	500.72
14	436.23	503.87
15	516.86	515.43
16	508.55	513.10

Total number of laboratories	p	14
Total number of replicates	Sum(n(L))	28
Overall mean of all data (grand mean)	XBARBAR	510.26
Repeatability standard deviation	s(r)	5.69
Reproducibility standard deviation	s(R)	10.24
Repeatability relative standard deviation	RSD(r)	1.12
Reproducibility relative standard deviation	RSD(R)	2.01
HORRAT value		0.45

Infant Formula Powder Milk Based

Lab	CULF358	GBZC169
1	407.92	415.97
2	402.60	404.55
3	421.27	409.17
4	404.98	408.86
5	417.37	416.50
6	407.97	406.92
7	405.95	405.71
8	410.15	409.71
9	401.03	402.16
10	409.47	409.47
11	405.97	407.00
12	445.84	464.35
13	404.77	405.31
14	330.72	363.97
15	414.27	440.22
16	413.72	432.16

Total number of laboratories	p	15
Total number of replicates	Sum(n(L))	30
Overall mean of all data (grand mean)	XBARBAR	413.71
Repeatability standard deviation	s(r)	7.28
Reproducibility standard deviation	s(R)	14.45
Repeatability relative standard deviation	RSD(r)	1.76
Reproducibility relative standard deviation	RSD(R)	3.49
HORRAT value		0.76

Infant Formula Powder FOS/GOS Based

Lab	WKHN288	URTF231
1	336.36	325.69
2	329.51	324.40
3	332.26	318.47
4	325.94	320.87
5	335.20	336.88
6	327.43	328.39
7	325.64	327.40
8	331.26	329.92
9	324.05	325.28
10	327.90	329.14
11	326.04	325.91
12	358.55	353.14
13	325.26	325.18
14	293.67	394.11
15	332.24	335.65
16	334.08	332.98

Total number of laboratories	p	15
Total number of replicates	Sum(n(L))	30
Overall mean of all data (grand mean)	XBARBAR	330.37
Repeatability standard deviation	s(r)	3.70
Reproducibility standard deviation	s(R)	8.40
Repeatability relative standard deviation	RSD(r)	1.12
Reproducibility relative standard deviation	RSD(R)	2.54
HORRAT value		0.54

Infant Formula Powder Partially Hydrolyzed Soy Based

Lab	SWUO667	MYHK654
1	384.34	395.03
2	391.95	392.28
3	390.17	391.08
4	387.74	385.88
5	393.92	395.87
6	383.76	382.43
7	380.68	381.54
8	385.33	386.07
9	379.06	376.15
10	382.63	383.84
11	381.44	383.05
12	417.15	418.94
13	378.98	379.34
14	340.64	347.08
15	407.10	399.49
16	383.14	132.46

Total number of laboratories	p	15
Total number of replicates	Sum(n(L))	30
Overall mean of all data (grand mean)	XBARBAR	386.10
Repeatability standard deviation	s(r)	2.83
Reproducibility standard deviation	s(R)	15.77
Repeatability relative standard deviation	RSD(r)	0.73
Reproducibility relative standard deviation	RSD(R)	4.08
HORRAT value		0.88

Infant Formula Powder Partially Hydrolyzed Milk Based

Lab	KDOX966	ATAN351
1	384.52	386.45
2	392.43	392.64
3	382.22	385.47
4	376.98	380.89
5	382.32	383.20
6	375.71	375.62
7	374.60	373.61
8	376.65	375.79
9	371.58	369.11
10	375.61	376.37
11	374.00	417.45
12	393.84	402.15
13	370.86	370.54
14	371.89	370.30
15	390.73	384.22
16	280.34	379.96

Total number of laboratories	p	14
Total number of replicates	Sum(n(L))	28
Overall mean of all data (grand mean)	XBARBAR	380.22
Repeatability standard deviation	s(r)	2.34
Reproducibility standard deviation	s(R)	8.63
Repeatability relative standard deviation	RSD(r)	0.61
Reproducibility relative standard deviation	RSD(R)	2.27
HORRAT value		0.49

Infant Formula RTF, Milk Based (liq.) PLACEBO

Lab	TJMN542	XJDD334
1	22.57	23.47
2	22.38	21.97
3	23.44	22.00
4	22.43	22.52
5	22.47	22.53
6	22.16	21.93
7	21.90	22.18
8	22.21	22.35
9	21.88	21.90
10	22.34	21.83
11	21.95	22.00
12	24.53	24.54
13	22.05	21.80
14	22.09	22.03
15	23.91	23.85
16	22.02	22.15

Total number of laboratories	p	16.00
Total number of replicates	Sum(n(L))	32.00
Overall mean of all data (grand mean)	XBARBAR	22.48
Repeatability standard deviation	s(r)	0.33
Reproducibility standard deviation	s(R)	0.79
Repeatability relative standard deviation	RSD(r)	1.49
Reproducibility relative standard deviation	RSD(R)	3.50
HORRAT value		0.49

Infant Formula Powder Milk Based

Lab	JSDT587	NSRB999
1	363.09	364.96
2	361.11	360.12
3	368.43	361.66
4	357.30	359.79
5	366.56	367.73
6	357.90	359.40
7	354.18	357.11
8	360.64	360.09
9	471.79	476.13
10	360.50	359.06
11	356.40	357.15
12	510.02	505.38
13	355.16	356.56
14	308.39	324.36
15	490.10	487.76
16	365.89	365.48

Total number of laboratories	p	13.00
Total number of replicates	Sum(n(L))	26.00
Overall mean of all data (grand mean)	XBARBAR	357.27
Repeatability standard deviation	s(r)	3.56
Reproducibility standard deviation	s(R)	13.10
Repeatability relative standard deviation	RSD(r)	1.00
Reproducibility relative standard deviation	RSD(R)	3.67
HORRAT value		0.78

Child formula powder – milk-based

Lab	RQXQ518	GVPE615
1	439.04	437.77
2	441.29	448.32
3	442.32	442.91
4	435.77	437.91
5	452.74	453.54
6	444.88	442.87
7	435.71	438.29
8	447.04	448.06
9	444.46	442.87
10	446.14	447.87
11	441.76	442.52
12	475.80	472.78
13	440.75	440.28
14	395.93	397.62
15	462.68	459.76
16	450.81	445.53

Total number of laboratories	p	16.00
Total number of replicates	Sum(n(L))	32.00
Overall mean of all data (grand mean)	XBARBAR	443.63
Repeatability standard deviation	s(r)	1.96
Reproducibility standard deviation	s(R)	15.80
Repeatability relative standard deviation	RSD(r)	0.44
Reproducibility relative standard deviation	RSD(R)	3.56
HORRAT value		0.79

SRM 1849a

Lab	KGSZ273	LTCT316
1	697.62	693.35
2	715.80	706.81
3	736.73	748.59
4	694.07	688.54
5	704.76	706.44
6	693.23	696.90
7	696.55	694.88
8	676.90	694.77
9	692.68	692.24
10	685.88	695.90
11	687.18	687.19
12	736.99	736.42
13	687.75	688.02
14	695.72	688.33
15	705.69	709.07
16	697.10	702.45

Total number of laboratories	p	16.00
Total number of replicates	Sum(n(L))	32.00
Overall mean of all data (grand mean)	XBARBAR	701.08
Repeatability standard deviation	s(r)	5.01
Reproducibility standard deviation	s(R)	17.14
Repeatability relative standard deviation	RSD(r)	0.72
Reproducibility relative standard deviation	RSD(R)	2.44
HORRAT value		0.58

Infant elemental powder placebo

Lab	ARJT349	SAEQ748
1	369.61	364.11
2	368.05	365.69
3	366.09	363.13
4	360.00	360.03
5	361.34	363.55
6	354.09	355.34
7	353.17	352.11
8	354.51	354.16
9	352.24	351.69
10	353.37	354.27
11	352.69	353.18
12	377.11	383.63
13	349.77	350.91
14	353.18	353.24
15	362.35	360.57
16	354.54	358.13

Total number of laboratories	p	16.00
Total number of replicates	Sum(n(L))	32.00
Overall mean of all data (grand mean)	XBARBAR	358.93
Repeatability standard deviation	s(r)	1.88
Reproducibility standard deviation	s(R)	8.03
Repeatability relative standard deviation	RSD(r)	0.52
Reproducibility relative standard deviation	RSD(R)	2.24
HORRAT value		0.48

Infant Elemental Powder

Lab	UOPM297	ECHL425
1	383.06	387.06
2	384.18	382.42
3	382.07	377.58
4	378.65	378.60
5	379.50	377.76
6	371.79	370.09
7	368.40	369.51
8	371.88	369.74
9	368.49	367.62
10	370.44	370.62
11	368.80	368.69
12	394.06	393.72
13	365.75	368.94
14	318.73	334.04
15	377.96	376.66
16	373.47	371.15

Total number of laboratories	p	16
Total number of replicates	Sum(n(L))	32
Overall mean of all data (grand mean)	XBARBAR	372.54
Repeatability standard deviation	s(r)	3.08
Reproducibility standard deviation	s(R)	14.52
Repeatability relative standard deviation	RSD(r)	0.83
Reproducibility relative standard deviation	RSD(R)	3.90
HORRAT value		0.84

Child formula powder – milk-based placebo

Lab	YXBH789	DRWO880
1	29.40	31.91
2	19.54	20.56
3	30.75	66.77
4	25.09	25.04
5	36.14	36.72
6	37.80	35.45
7	35.47	34.91
8	130.73	126.45
9	37.08	34.26
10	54.15	27.23
11	34.85	21.15
12	74.11	288.80
13	35.92	34.22
14	28.48	35.27
15	63.05	62.72
16	46.59	36.90

Total number of laboratories	p	11
Total number of replicates	Sum(n(L))	22
Overall mean of all data (grand mean)	XBARBAR	32.40
Repeatability standard deviation	s(r)	4.00
Reproducibility standard deviation	s(R)	6.75
Repeatability relative standard deviation	RSD(r)	12.35
Reproducibility relative standard deviation	RSD(R)	20.84
HORRAT value		3.11

IDF samples

Butter		
Lab	IDF 51**	IDF 71**
1		
2		
3		
4		
5	376.68	375.80
6	406.92	403.94
7	363.72	368.53
8	360.44	370.78
9	364.42	369.26
10	371.31	378.02
11	365.12	364.13
12	391.69	393.21
13	377.14	360.18
14	374.29	382.31
15	368.72	384.94
16	19.22	29.03
17	375.87	364.50
Total number of laboratories	p	12
Total number of replicates	Sum(n(L))	24
Overall mean of all data (grand mean)	XBARBAR	375.50
Repeatability standard deviation	s(r)	6.31
Reproducibility standard deviation	s(R)	13.03
Repeatability relative standard deviation	RSD(r)	1.68
Reproducibility relative standard deviation	RSD(R)	3.47
HORRAT value		0.75

Processed cheese		
Lab	IDF 106*	IDF 86*
1		
2		
3		
4		
5	563.87	555.39
6	568.91	571.36
7	541.13	540.22
8	554.96	560.68
9	547.67	561.02
10	533.74	555.76
11	562.23	561.41
12	587.67	577.30
13	549.69	572.47
14	590.45	589.27
15	566.80	579.23
16	69.89	38.93
17	557.43	545.02
Total number of laboratories	p	12
Total number of replicates	Sum(n(L))	24
Overall mean of all data (grand mean)	XBARBAR	562.24
Repeatability standard deviation	s(r)	8.45
Reproducibility standard deviation	s(R)	15.74
Repeatability relative standard deviation	RSD(r)	1.50
Reproducibility relative standard deviation	RSD(R)	2.80
HORRAT value		0.64

Whey protein concentrate

Lab	IDF 34	IDF 94
1		
2		
3		
4		
5	169.94	170.82
6	164.12	167.80
7	166.48	164.63
8	167.21	170.30
9	163.98	156.73
10	174.12	171.05
11	163.52	163.57
12	216.98	210.99
13	163.81	166.12
14	171.01	175.80
15	183.77	185.84
16	147.91	162.93
17	167.71	162.69
Total number of laboratories	p	10
Total number of replicates	Sum(n(L))	20
Overall mean of all data (grand mean)	XBARBAR	169.5159481
Repeatability standard deviation	s(r)	2.173421903
Reproducibility standard deviation	s(R)	6.534000456
Repeatability relative standard deviation	RSD(r)	1.282134175
Reproducibility relative standard deviation	RSD(R)	3.854504859
HORRAT value		0.737793444

Whole milk powder

Lab	IDF 23	IDF 123
1		
2		
3		
4		
5	712.56	719.99
6	711.98	712.20
7	714.92	714.10
8	705.27	704.79
9	694.81	702.11
10	709.42	712.77
11	702.28	696.09
12	768.06	752.91
13	696.29	697.04
14	713.88	712.48
15	733.17	729.46
16	691.92	690.72
17	701.81	699.88
Total number of laboratories	p	13
Total number of replicates	Sum(n(L))	26
Overall mean of all data (grand mean)	XBARBAR	711.57
Repeatability standard deviation	s(r)	3.97
Reproducibility standard deviation	s(R)	18.32
Repeatability relative standard deviation	RSD(r)	0.56
Reproducibility relative standard deviation	RSD(R)	2.57
HORRAT value		0.61

Whole Milk, 1.0 g as is

Lab	IDF 12	IDF 42
1		
2		
3		
4		
5	95.79	94.31
6	93.24	93.57
7	113.46	95.20
8	94.86	94.02
9	93.62	93.16
10	94.95	95.19
11	95.01	95.03
12	99.53	101.52
13	94.18	94.75
14		
15	97.79	97.74
16	97.77	96.66
17	94.386	89.05

Total number of laboratories	p	11
Total number of replicates	Sum(n(L))	22
Overall mean of all data (grand mean)	XBARBAR	95.28
Repeatability standard deviation	s(r)	1.30
Reproducibility standard deviation	s(R)	2.59
Repeatability relative standard deviation	RSD(r)	1.37
Reproducibility relative standard deviation	RSD(R)	2.72
HORRAT value		0.48

Whey powder

Lab	IDF 65	IDF 115
1		
2		
3		
4		
5	285.73	283.26
6	280.15	277.38
7	1533.24	1528.70
8	278.01	276.85
9	1561.30	1459.20
10	277.72	278.77
11	1510.67	1448.95
12	307.35	303.13
13	270.25	274.02
14	275.32	274.29
15	287.31	283.30
16	273.20	622.36
17	275.66	275.08

Total number of laboratories	p	9
Total number of replicates	Sum(n(L))	18
Overall mean of all data (grand mean)	XBARBAR	281.31
Repeatability standard deviation	s(r)	1.91
Reproducibility standard deviation	s(R)	10.04
Repeatability relative standard deviation	RSD(r)	0.68
Reproducibility relative standard deviation	RSD(R)	3.57
HORRAT value		0.74

Summary of instruments and extra protein precipitation steps used by lab

	Lab 01	Lab 02	Lab 03	Lab 04	Lab 5
Country:	China	China	China	China	the Netherlands
Auto titrator manufacturer	Metrohm	888 Titrande ,800 Dosino	Metrohm	Metrohm	Metrohm
Auto titrator model	809 Tirando	tiamo	862	848 Titrino plus	716 Titrino
Software	tiamo	version 2.5		DET_U	Tiamo
Software version	1.3				1.2
protein precipitation step [7.2-7.4]	Yes	Yes	Yes	Yes	No

	Lab 6	Lab 7	Lab 8	Lab 9	Lab 10
Country:	Singapore	US	Germany	USA	New Zealand
Auto titrator manufacturer	Mettler Toledo	Mettler Toldedo	Mettler Toledo	Mettler	Metrohm
Auto titrator model	T70	DL53	T50	G20 Rondolino Auto	916 Ti- Touch
Software	Mettler Toledo T70	LabX		LabX 2014	
Software version	2.0.0			6.0.0 Build 494	
protein precipitation step [7.2-7.4]	No	No	No	No	No

	Lab 11	Lab 12	Lab 13	Lab 14	Lab 15
Country:	USA	the Netherlands	Malaysia	Brazil	the Netherlands
Auto titrator manufacturer	Mettler Toledo	Mettler Toledo	Mettler Toledo	Mettler Toledo	Mettler Toledo
Auto titrator model	T50	T70	G20	T50	T70
Software	LabX 2016	LabX	LabX 2016		
Software version	7.00 build 754				
protein precipitation step [7.2-7.4]	No	No	No	No	No

	Lab 16	Lab 17
Country:	Ireland	the Netherlands
Auto titrator manufacturer	Metrohm	Not provided
Auto titrator model	855	
Software	Tiamo	
Software version	2	
protein precipitation step [7.2-7.4]	No	No

All results for both SPIFAN and IDF samples. Results in orange were omitted from the statistics.

Adult Nutritional Powder Low Fat		
Lab	LWY751	RZGR859
1	386.25	385.07
2	387.91	386.32
3	383.56	388.76
4	388.51	386.66
5	396.67	393.25
6	382.55	385.32
7	381.93	380.74
8	386.68	389.36
9	383.10	382.53
10	385.96	385.31
11	383.72	383.95
12	416.29	408.48
13	380.19	380.18
14	345.39	343.88
15	390.06	393.01
16	394.61	389.48
17	394.61	389.48
Total number of laboratories	p	16.00
Total number of replicates	Sum(n/L)	32.00
Overall mean of all data (grand mean)	XBARBAR	385.49
Repeatability standard deviation	s(r)	2.25
Repeatability standard deviation	S(r)	13.34
Repeatability relative standard deviation	RSD(R)	0.58
Repeatability relative standard deviation	RSD(R)	3.46
HORRAT value		0.75

Adult Nutritional RTF High Fat (Liq.)		
Lab	DYL8360	ZMQQ883
1	163.31	163.91
2	170.69	167.93
3	162.26	165.36
4	156.64	158.64
5	167.23	167.26
6	158.35	157.71
7	155.51	158.13
8	158.59	155.02
9	65.49	73.26
10	141.95	141.95
11	162.91	165.16
12	157.35	161.12
13	162.73	160.65
14	164.03	163.10
15	107.56	120.14
16	128.30	131.15
17	128.30	131.15
Total number of laboratories	p	10.00
Total number of replicates	Sum(n/L)	20.00
Overall mean of all data (grand mean)	XBARBAR	162.65
Repeatability standard deviation	s(r)	1.70
Repeatability standard deviation	S(r)	4.14
Repeatability relative standard deviation	RSD(R)	1.05
Repeatability relative standard deviation	RSD(R)	2.54
HORRAT value		0.48

Adult nutritional ready to feed – high fat placebo		
Lab	IGV161	WV0654
1	36.96	36.87
2	41.30	39.24
3	38.32	39.08
4	31.09	31.43
5	37.29	34.25
6	33.35	35.25
7	38.46	39.06
8	33.97	30.80
9	28.32	33.94
10	11	11
11	33.98	32.38
12	39.66	44.03
13	36.04	36.01
14	36.22	38.30
15	33.19	34.96
16	32.19	31.15
17	32.19	31.15
Total number of laboratories	p	8.00
Total number of replicates	Sum(n/L)	16.00
Overall mean of all data (grand mean)	XBARBAR	35.41
Repeatability standard deviation	s(r)	2.99
Repeatability standard deviation	S(r)	1.55
Repeatability relative standard deviation	RSD(R)	3.14
Repeatability relative standard deviation	RSD(R)	7.21
HORRAT value		1.09

Adult Nutritional RTF High Protein (Liq.)		
Lab	DQW545	PPT6112
1	152.58	154.97
2	162.54	159.15
3	151.17	152.91
4	152.55	152.77
5	158.69	157.11
6	152.96	152.57
7	153.46	155.41
8	155.77	155.39
9	152.59	152.05
10	147.54	152.23
11	155.80	155.64
12	160.54	163.56
13	153.34	153.66
14	154.03	154.25
15	155.26	155.37
16	151.71	136.65
17	151.71	136.65
Total number of laboratories	p	16
Total number of replicates	Sum(n/L)	32
Overall mean of all data (grand mean)	XBARBAR	154.19
Repeatability standard deviation	s(r)	2.99
Repeatability standard deviation	S(r)	4.65
Repeatability relative standard deviation	RSD(R)	1.94
Repeatability relative standard deviation	RSD(R)	3.81
HORRAT value		0.57

Adult nutritional ready to feed – high protein placebo		
Lab	YKQ123	BVW962
1	40.43	36.86
2	38.81	37.52
3	94.75	99.75
4	37.75	37.77
5	40.68	40.08
6	31.11	33.25
7	42.37	41.89
8	39.97	39.99
9	40.19	39.61
10	38.95	38.08
11	40.84	40.65
12	515.86	471.61
13	41.69	40.82
14	41.17	40.99
15	42.04	42.57
16	47.43	49.90
17	47.43	49.90
Total number of laboratories	p	11.00
Total number of replicates	Sum(n/L)	22.00
Overall mean of all data (grand mean)	XBARBAR	478.61
Repeatability standard deviation	s(r)	0.45
Repeatability standard deviation	S(r)	1.56
Repeatability relative standard deviation	RSD(R)	0.62
Repeatability relative standard deviation	RSD(R)	3.89
HORRAT value		0.84

Toddler Formula Powder Milk-Based		
Lab	EFXN778	BFA0941
1	470.91	486.86
2	475.83	470.29
3	480.80	493.48
4	471.99	470.81
5	489.55	491.87
6	477.13	489.13
7	475.77	475.66
8	479.53	480.51
9	470.22	474.63
10	483.47	480.42
11	474.61	477.15
12	515.86	512.39
13	476.08	474.73
14	423.49	426.57
15	486.57	490.19
16	491.39	486.69
17	491.39	486.69
Total number of laboratories	p	16.00
Total number of replicates	Sum(n/L)	32.00
Overall mean of all data (grand mean)	XBARBAR	478.61
Repeatability standard deviation	s(r)	4.17
Repeatability standard deviation	S(r)	17.97
Repeatability relative standard deviation	RSD(R)	0.87
Repeatability relative standard deviation	RSD(R)	3.76
HORRAT value		0.84

Child formula powder – milk-based placebo		
Lab	YKH789	DRWC880
1	29.40	31.91
2	19.54	20.56
3	30.76	66.77
4	25.09	25.04
5	36.14	36.72
6	37.80	35.45
7	35.47	34.91
8	130.73	126.45
9	470.22	474.63
10	54.15	27.23
11	34.85	21.15
12	14.11	288.40
13	35.92	34.22
14	28.48	35.27
15	63.05	62.72
16	46.59	36.90
17	46.59	36.90
Total number of laboratories	p	11
Total number of replicates	Sum(n/L)	22
Overall mean of all data (grand mean)	XBARBAR	32.40
Repeatability standard deviation	s(r)	4.00
Repeatability standard deviation	S(r)	6.75
Repeatability relative standard deviation	RSD(R)	12.44
Repeatability relative standard deviation	RSD(R)	20.84
HORRAT value		3.11

Infant Formula RTF Milk Based (Liq.)		
Lab	XKP216	HVJL890
1	42.06	42.07
2	43.66	41.99
3	94.84	99.28
4	42.10	42.22
5	42.75	42.79
6	41.90	41.96
7	41.78	41.96
8	42.28	42.16
9	41.26	41.37
10	42.09	42.18
11	41.03	42.08
12	45.14	45.69
13	41.81	42.19
14	41.86	41.83
15	43.10	42.95
16	42.67	42.62
17	42.67	42.62
Total number of laboratories	p	15
Total number of replicates	Sum(n/L)	30
Overall mean of all data (grand mean)	XBARBAR	42.40
Repeatability standard deviation	s(r)	0.40
Repeatability standard deviation	S(r)	0.99
Repeatability relative standard deviation	RSD(R)	0.94
Repeatability relative standard deviation	RSD(R)	3.49
HORRAT value		0.36

Infant Formula Powder Soy Based		
Lab	TJHR217	OACN211
1	501.97	502.15
2	525.42	516.52
3	498.04	524.53
4	507.78	507.19
5	516.52	518.37
6	505.70	506.16
7	503.84	504.06
8	508.50	507.65
9	497.93	499.74
10	409.25	506.83
11	503.83	503.25
12	45.14	45.69
13	500.94	500.72
14	436.23	503.87
15	516.86	515.43
16	508.55	513.10
17	508.55	513.10
Total number of laboratories	p	14
Total number of replicates	Sum(n/L)	28
Overall mean of all data (grand mean)	XBARBAR	510.26
Repeatability standard deviation	s(r)	5.69
Repeatability standard deviation	S(r)	10.24
Repeatability relative standard deviation	RSD(R)	1.12
Repeatability relative standard deviation	RSD(R)	2.01
HORRAT value		0.45

Infant Formula Powder Milk Based		
Lab	CULF358	GBZC169
1	507.02	511.97
2	402.60	404.55
3	421.27	409.17
4	404.98	408.86
5	417.37	416.50
6	407.87	406.92
7	405.95	405.71
8	410.15	409.71
9	401.03	402.16
10	409.47	409.47
11	405.97	407.00
12	445.84	444.36
13	404.77	405.31
14	330.72	363.97
15	332.24	440.22
16	413.72	432.16
17	413.72	432.16
Total number of laboratories	p	15
Total number of replicates	Sum(n/L)	30
Overall mean of all data (grand mean)	XBARBAR	413.71
Repeatability standard deviation	s(r)	7.28
Repeatability standard deviation	S(r)	14.45
Repeatability relative standard deviation	RSD(R)	1.76
Repeatability relative standard deviation	RSD(R)	3.49
HORRAT value		0.76

Infant Formula Powder FOS/GOS Based		
Lab	WJHN288	LRTF231
1	415.97	411.91
2	329.51	324.40
3	332.26	318.47
4	325.94	320.87
5	335.20	336.88
6	327.43	328.39
7	325.54	327.40
8	331.26	329.92
9	324.05	325.28
10	327.90	329.14
11	326.04	325.91
12	445.84	444.36
13	325.26	325.18
14	293.67	304.11
15	332.24	440.22
16	334.08	332.98
17	334.08	332.98
Total number of laboratories	p	15
Total number of replicates	Sum(n/L)	30
Overall mean of all data (grand mean)	XBARBAR	330.37
Repeatability standard deviation	s(r)	3.70
Repeatability standard deviation	S(r)	8.40
Repeatability relative standard deviation	RSD(R)	1.12
Repeatability relative standard deviation	RSD(R)	2.64
HORRAT value		0.54

Infant Formula Powder Partially Hydrolyzed Soy Based		
Lab	SWU067	MYHK654
1	394.34	393.93
2	391.95	392.28
3	390.17	391.08
4	387.74	385.88
5	393.92	395.87
6	383.76	382.43
7	380.68	381.54
8	385.33	386.07
9	379.06	376.15
10	382.63	383.84
11	381.44	383.05
12	445.84	444.36
13	378.98	379.34
14	340.64	347.08
15	407.10	399.49
16	383.14	132.46
17	383.14	132.46
Total number of laboratories	p	15
Total number of replicates	Sum(n/L)	30
Overall mean of all data (grand mean)	XBARBAR	386.10
Repeatability standard deviation	s(r)	2.83
Repeatability standard deviation	S(r)	15.77
Repeatability relative standard deviation	RSD(R)	0.73
Repeatability relative standard deviation	RSD(R)	3.84
HORRAT value		0.88

Infant Formula Powder Partially Hydrolyzed Milk Based		
Lab	KDOX966	ATAN351
1	334.52	339.13
2	392.43	392.64
3	382.22	385.47
4	376.98	380.89
5	382.32	383.20
6	375.71	375.62
7	374.60	373.61
8	376.65	375.79
9	371.58	369.11
10	375.61	376.37
11	374.00	371.45
12	393.84	402.17
13	370.86	370.54
14	371.89	370.30
15	390.73	384.22

