Stability Testing Guideline for Dietary Supplements
Final Draft – January 2011
Provided by the NSF Stability Testing Working Group

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1.0 Introduction

The purpose of this guideline is to present recommendations for supporting the voluntary shelf-life (expiration) dating claims of dietary supplements. Federal regulations do not require the use of dietary supplement product expiration dates. However, the Federal Register GMP Preamble, June 25, 2007, Volume 72 Page, 34856 states, "the preamble to the 2003 cGMP Proposal emphasized that, if you use an expiration date on a product, you should have data to support that date (68 FR 12157 at 12204)". For the purpose of this document, these recommendations also apply to dietary supplements that voluntarily claim "best by", "use before", etc. dates in lieu of a specific "expiration date."

This document is intended to provide practical guidance on what should be considered when developing voluntary written shelf-life study protocols to establish or confirm expiration dating of dietary supplements. It is written so as to leave sufficient flexibility to address the range of situations that may be encountered due to the specific scientific considerations and characteristics of the products and container-closure systems being evaluated.

For markets outside of the United States, applicable regulations from other countries should be followed. The initial draft of this guideline was developed based on the ICH Guidelines developed for conducting Stability Testing for Pharmaceutical products.

2.0 Scope

These guidelines are intended to be applicable to dietary supplements as defined and covered by DSHEA and the United States Food and Drug Administration's Code of Federal Regulations Title 21, Part 111 [Current Good Manufacturing Practices in Manufacturing, Packaging, Labeling, or Holding Operations for Dietary Supplements]

also known as 21 CFR Part 111, but may also be applied to dietary ingredients and other components.

3.0 General Principles of Shelf-life Studies

The user of this guideline should:

a) understand that the FDA final GMP rule for dietary supplements does not require the establishment of product expiration dates, but that any expiration date placed on a product label (including claims such as, ``best if used by'') should be supported by data.

b) consider and identify issues related to the organoleptic, physical, chemical, biological and microbiological characteristics of the components under long term storage.

c) consider the impact that manufacturing, packaging, labeling, and holding/warehouse processes may have on the stability profile of products.

d) ensure that the finished products have initial release specifications that help assure compliance with long-term stability specifications and suitable overages.

e) determine the stability of the finished product in the container-closure system that will be marketed.

f) consider any historical data related to the formulation or the container-closure system that may be relevant to the stability of the product.

g) review applicable label claims related to stability aspects of the finished product to ensure that stability studies are designed to support these claims, as necessary.

h) understand the distribution (including transportation) and storage conditions that the dietary supplement will be subjected to over its shelf life and factor these conditions into the stability study as necessary.

i) consider the suitability of accelerated stability studies on new product forms, as some components may react differently at increased temperatures or humidity levels than at ambient conditions.

j) consider the potential for the formation of degradation products and set corresponding stability indicating specifications as necessary.

k) understand that while federal regulations do not require the use of product expiration dates on marketed dietary supplements, if an expiration date is used reserve samples must be maintained for one year past the shelf-life date (per 21 CFR Part 111 Sections 83 and 465). Also written records related to the product must be retained for one year past the shelf-life date (per 21CFR 111 Section 605). Therefore if a shelf life date is added to or extended for a dietary supplement, the user may need to update the period of time that reserve samples and/or written records are maintained for that dietary supplement.

4.0 Selection of Batches

Stability data should be gathered on batches that are representative of the product and container-closure system being marketed. Where possible, variables that are applicable to the normal manufacturing and distribution processes (e.g., different batches of components) should be included. If laboratory or pilot-scale batches are used initially for testing, the manufacturing process used should closely simulate full-scale production and the product should be packaged in containers of the same quality and meeting the same specification(s) as that intended for marketing. After an expiration date is

established or confirmed initially, it may be appropriate to place a new batch of product on shelf-life study under long-term conditions annually or more frequently. All batches used in stability testing should be fully traceable back to the component and packaging material lots used.

To establish an expiration date for a new or modified product or container-closure system: It may be appropriate to establish initial shelf life dating through either long-term or accelerated stability studies. If accelerated stability studies are used initially, it is recommended that at least one of the first full-scale batches also be tested under long-term conditions. An initial expiration date may be established from the accelerated study results and modified as long-term study results become available.

To establish or confirm an expiration date for an existing product or container-closure system for which no previous stability data is available: It is recommended that the procedure described above for new products be followed. In addition, testing of reserve samples of the existing product in the same or similar container-closure system may be used to initially establish or confirm the expiration date, provided that sufficient reserve samples also are maintained for GMP retention sample purposes.

5.0 Bracketing or Matrixing

Shelf-life studies should be conducted on each unique dietary supplement formulation and container-closure system combination unless bracketing or matrixing can be applied. Studies using bracketing [testing only the extremes of certain dietary supplement / container-closure factors (such as potency or package size)] or matrixing [testing only a selected subset of all possible combinations of dietary supplement /

container-closure at any given time] should be scientifically justified. See ICH Guideline

Q1D for suggestions on how bracketing and/or matrixing are applied to shelf-life studies

of new drug substances or products.

http://www.ich.org/fileadmin/Public_Web_Site/ICH_Products/Guidelines/Quality/Q1D/Step4/Q1D_Guideline.pdf.

   NOTE: The criteria included in Q1D may be instructive, but are not required for dietary

   supplement shelf-life studies.

6.0 Types of Testing

All shelf-life testing should be undertaken using defined specifications. These

specifications should include the: (a) test parameters to be performed; (b) acceptance

criteria / values for each item tested and (c) appropriate and scientifically valid analytical

procedure to be used for each test item.

Test Parameters – The properties tested during shelf-life studies should include any

characteristics that are subject to change during storage and that could affect product

quality, safety, label claims, or efficacy. The parameters may include organoleptic

properties, dietary ingredient strengths, chemical fingerprints[1], ingredient degradation

products, microbial growth, preservative content, moisture content, disintegration,

dissolution, pH, viscosity and oxidation, and should be customized for each product.

Determination of the appropriate stability-indicating (or rate limiting) analyte(s) is

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[1] Regarding chemical fingerprints – these are often at least as important, if not more important, than the content of individual chemicals, at least in the case of complex chemical mixtures (such as many herbal and animal ingredients). Please refer to AHPA's document "Use of Marker Compounds in Manufacturing and Labeling Botanically Derived Dietary Supplements" and various European documents such as EMEA's "Guideline on Quality of Herbal Medicinal Products / Traditional Herbal Medicinal Products" (CPMP/QWP/2819/00 Rev. 1).]

dependent on a preliminary review of the components to determine which has the shortest shelf-life based on its stability characteristics.

Generally the parameters should include all quantitative label claims for components subject to degradation, where scientifically valid test methods exist for the components. Claimed components which are not subject to degradation, such as many kinds of minerals, often do not need to be tested. In highly complex products/matrices, it may be appropriate to select only the most labile components for shelf-life testing. The decision is typically based on available historical data.

Acceptance criteria / values –The acceptance criteria should be customized for each product and defined for each test parameter. Shelf-life acceptance criteria may be derived from consideration of all available stability information. It may be appropriate to have justifiable differences between the shelf-life and initial release specifications (acceptance criteria) based on the stability evaluation and the changes observed during storage. Any differences between the initial release and shelf-life acceptance criteria should be justified.

At the conclusion of a product's shelf-life, label claim ingredient levels should meet the minimum legal requirements whether natural or added. According to FDA 21 CFR 101.9 (g)(3)(i)(ii) and 101.9 (g)(4)(i)(ii), "the minimum acceptance criteria for naturally-occurring dietary ingredients is 80% and for added dietary ingredients is 100% of label claim at the end of shelf-life".

Analytical procedures – All test procedures used should be appropriate and scientifically valid for all relevant characteristics (e.g., accuracy, precision, sensitivity, etc).

The following list of parameters for each dietary supplement type is presented as a guide for the types of tests that should be considered for inclusion in a shelf-life study. The list presented for each product is not intended to be exhaustive, nor is it expected that every listed parameter be included in the design of a shelf-life study protocol for a particular dietary supplement. Actual parameters chosen may also be dependent on whether a long-term or accelerated study is being conducted, as some parameters may not be appropriate for accelerated studies on certain products.   Not all parameters need to be performed at all time points in a shelf-life study.

1. Tablets

   - Appearance, color, odor, taste; and/or mouthfeel

   - Chemical fingerprints

   - Assay [i.e., label claim ingredient(s)]

   - Hardness/friability

   - Disintegration or dissolution rate

   - Moisture content

   - Microbial content (e.g. Total Bacterial Count, Total Yeast & Mold count)

   - Fat oxidation

   - Ingredient degradation products

   - Loss on drying

2. Capsules (hard and soft)

   - Appearance, color, odor, taste; and/or mouthfeel

   - Chemical fingerprints

   - Assay [i.e., label claim ingredient(s)]

- Disintegration or dissolution rate

- Capsule integrity such as brittleness or leakage

- Moisture content

- Microbial content (e.g. Total Bacterial Count, Total Yeast & Mold count)

- Fat oxidation

- Ingredient degradation products

- Loss on drying

3. Liquids and semi-solids (gels)

- Appearance, color, odor, taste, and/or mouthfeel

- Chemical fingerprints

- Assay [i.e., label claim ingredient(s)]

- pH

- Viscosity and/or texture

- Moisture content and/or water activity

- Microbial content (e.g. Total Bacterial Count, Total Yeast & Mold count)

- Preservative content

- Fat oxidation

- Ingredient degradation products

- Loss on drying

4. Powders

- Appearance, color, odor, taste, and/or mouthfeel

- Chemical fingerprints

- Assay [i.e., label claim ingredient(s)]

- Moisture content and/or water activity

- Microbial content (e.g. Total Bacterial Count, Total Yeast & Mold count)

- Fat oxidation

- Ingredient degradation products

- Hygroscopicity

- Loss on drying

7.0 Container-closure Systems

Shelf-life studies should be conducted on the dietary supplement packaged in the same container-closure system used or proposed for marketing the supplement (including, as appropriate, container label and any primary packaging and on-shelf / secondary packaging that may impact the shelf-life).

If use of the exact container-closure system is not possible or practical, the container-closure system used for shelf-life studies should simulate (e.g. be similar to but not necessarily identical to) the marketed system as closely as possible.

8.0 Types of Studies / Testing to Support Shelf-life (Expiration) Dating Claims

8.1 Accelerated Studies

Accelerated shelf-life studies may provide an early indication of longer term effects at non-accelerated conditions and of the effects of short-term excursions outside the label storage conditions that may occur during shipping. However, accelerated studies should be viewed only as a short term means of justifying shelf-life claims – long-term studies should also be carried out as soon as feasible.

It is important to recognize that accelerated studies may not be appropriate for all products or container-closure systems, and should be conducted at the discretion of the user. It may be challenging to establish acceptance criteria with accelerated data only.

It is also important to note that accelerated studies may not be 100% predictive of real time dating and that variability does occur. To truly assess the stability outcome, the stability of the product must be evaluated under real time conditions. Currently there are few official resources available to provide guidance on the evaluation of stability data. The ICH Guideline Q1E – Evaluation For Stability Data was developed with the intention to apply to active pharmaceutical ingredients (API's) but the principles and concepts may be applicable to dietary supplements.

http://www.ich.org/fileadmin/Public_Web_Site/ICH_Products/Guidelines/Quality/Q1E/Step4/Q1E_Guideline.pdf.

Accelerated studies generally extend from three to six months as a means of predicting product shelf-life for up to two years. Typically, samples should be analyzed at several time points during the study to provide a means of plotting or trending the results. For products with no special storage conditions specified on the label, the following storage conditions and tolerances for accelerated studies are recommended:

40^0C +/- 2^0C and 75% relative humidity (RH) +/- 5% RH.

Alternative storage conditions and/or tolerances may be used, as appropriate.

Depending on the product components and container-closure system, lighting conditions during product storage should be considered as an additional storage factor.

8.2 Long-term Studies

Long-term studies should be carried out under conditions simulating the storage conditions recommended on the product label. Long-term studies should extend at least as long as the desired shelf-life claim (expiration date) for that product, although longer-term testing may be performed at the discretion of the user. It is recommended that samples be collected and analyzed at least annually throughout the shelf-life or other intervals as deemed appropriate. Testing should be conducted at established frequencies to provide a means of plotting or trending the results (e.g. 0, 3, 6, 9, 12, 18, 24 etc. months), although more frequent testing may be performed at the discretion of the user. These testing points may vary depending on the product, container-closure system and length of the shelf-life claim, as well as the user's experience with the product.

As mentioned under Accelerated Studies, there are few official resources available to provide guidance on the evaluation of stability data,but the ICH Guideline Q1E – Evaluation For Stability Data may be applicable to dietary supplements.

http://www.ich.org/fileadmin/Public_Web_Site/ICH_Products/Guidelines/Quality/Q1E/Step4/Q1E_Guideline.pdf

Recommended storage conditions and tolerances for long-term studies are shown below. For products intended for:

    a. Ambient (room temperature) storage or for which no special storage conditions are specified on the label:

        20-25^{0}C +/- 2^{0}C and 60% RH +/- 5% RH

    b. Refrigerated storage:

        5^{0}C +/- 3^{0}C

    c. Frozen storage:

-20^{0}C +/- 5^{0}C

d. Other types of storage:

alternative storage conditions and/or tolerances may be also considered, as appropriate.

Lighting conditions during product storage should be considered also.

As data are gathered, acceptable results from long-term shelf-life studies may be used to justify increasing the shelf-life claim (expiration date) for a dietary supplement. Conversely, failure to meet any of the stability-indicating (shelf-life) specifications during long-term studies should result in an out-of-specification investigation and conclusion regarding the validity of current shelf-life claims and/or the need for adjustments to the product formulation and/or the product shelf life claim.

8.3 Reserve Sample Testing

If used to confirm or establish the expiration date of a product, reserve samples should have the same formulation and component specifications, be stored under typical use conditions (as labeled) and be packaged in a similar unopened container-closure system as the marketed product. Reserve samples should only be used if sufficient reserve samples also are maintained for GMP retention sample purposes.

It is recommended that appropriate accelerated and/or long-term shelf-life studies be conducted in tandem with the reserve sample testing.

8.4 Open Package Testing

If a product label indicates that a product is to be used within a specified period of time after opening the container-closure system, an open package storage study should be considered. The study should use storage conditions (i.e. temperature, relative humidity) similar to those expected to be used by the consumer and should last for at least as long as the period stated on the label. Typically the study is performed on product opened at 0 months with appropriate testing performed at 0 months and again at 50% and 100% of the period stated on the label. It is recommended to perform this study on at least one of the same batches that is being evaluated in accelerated and/or long-term studies in order to correlate the results with data from these studies.

8.5 Excursion Testing

The purpose of this testing is to explore the possible effects of shipping conditions on the product stability. Excursion testing may also be used for:

1) providing justification for shipping requirements

2) determining the effect of temporary storage at conditions that fall outside of recommended storage conditions during normal product use

3) determining acceptable storage conditions: dependent upon product handling and shipping conditions

4) simulating thermal cycling (e.g., freeze-thaw cycling) if such conditions are anticipated.

Excursion testing may be performed on its own or before placing a product into a formal shelf-life study. It should simulate expected shipping conditions to the extent possible.

9.0 Shelf-life Study Protocol (see addendum A for example)

There should be a written protocol that describes the shelf-life study program used to assess the stability characteristics of dietary supplements. The results of the stability testing should be used in determining appropriate storage conditions and shelf-life. The protocol should be defined prior to study commencement (e.g., in the design of the shelf-life study or in applicable standard operating procedures, etc). This protocol should include the following:

1. name, strength and package quantity of each product included in the study
2. type and size of each container closure system included
3. each formulation included
4. each batch code included
5. sample size and test intervals (e.g. frequency of sample testing) for each attribute
6. storage conditions for samples retained for testing
7. test parameters
8. acceptance criteria / values
9. explanation of bracketing and/or matrixing, if used
10. use of reliable, meaningful, specific and preferably validated test methods
11. recognition that the dietary supplement should be tested in the same type of container-closure system as that in which the dietary supplement is/will be marketed
12. method of data evaluation, including any statistical analysis used to establish product shelf life

The sample storage facility's heating, ventilation and air conditioning system should be capable of controlling the storage conditions within the ranges defined in the stability protocol developed for the shelf-life study. The actual temperature, humidity and light levels (when applicable) should be monitored during shelf-life storage. Short-term spikes due to opening of doors of the storage facility are generally accepted as unavoidable. The effect of excursions due to equipment failure should be addressed and reported if judged to affect stability results. Excursions that exceed the defined tolerances for more than 24 hours should be described in the study report and their effect assessed.

Addendum A is presented as an example of a shelf-life study protocol and is not meant to be all inclusive.

10.0 Shelf-life Evaluation / Study Report (see Addendum B for example)

A systematic approach should be utilized in the presentation and evaluation of the stability information, including applicable statistical treatments.

The evaluation should include all tests that were defined for the study. Any failure to meet defined stability-indicating (shelf-life) specifications or data trends should be investigated and conclusions should be documented regarding potential impact on shelf-life claims and/or the need to modify the product formulation or manufacturing process.

It is recommended that test results, investigations and conclusions be documented in a written study report.

Addendum B is presented as an example of a shelf-life study report and is not meant to be all inclusive.

11.0 Statements / Labeling

While federal regulations do not require that storage conditions be included on dietary supplement labels, it is recommended that product storage conditions be established and included on the product labeling. Any statement should be consistent with the conditions used in the stability evaluation of the product.

The storage conditions (e.g. temperature, light, humidity) indicated should be readily understood by consumers. Examples of storage temperature language are as follows:

- "Store in a cool, dry place."

- "Keep at room temperature, tightly closed."

- "Store at room temperature away from direct sunlight."

- "Refrigerate after opening."

- "Avoid excessive heat and moisture."

12.0 Process or Product Changes

Shelf-life studies determine the stability over time of a defined dietary supplement product produced in a given manufacturing facility, from a defined set of components, under a defined manufacturing and packaging process, storage conditions, and packaged in a defined container-closure system. Any time there are significant changes to any of these conditions, Quality Control personnel should determine whether (and if so, what type of) additional shelf-life studies should be undertaken to confirm the shelf-life claims of the marketed product . These determinations should be conducted even if

there are no changes to the label claims and should be documented and conclusions justified.

13.0 Glossary

The following definitions are provided to facilitate interpretation of the guideline.

Accelerated studies means shelf-life studies designed to increase the rate of chemical degradation and/or physical change of a dietary supplement by using exaggerated storage conditions as part of a shelf-life study protocol. Data from these studies, in addition to long-term shelf-life studies, may give an indication of longer term product changes at non-accelerated conditions and may assist in the evaluation of the effect of short-term excursions outside the label storage conditions such as might occur during shipping. However, results from accelerated testing studies are not always predictive of the chemical, organoleptic or physical changes that occur during long-term testing or actual product distribution and storage.

Batch means a specific quantity of a dietary supplement that is uniform, that is intended to meet specifications for identity, purity, strength, and composition, and that is produced during a specified time period according to a single manufacturing record during the same cycle of manufacture.

Bracketing means the design of a shelf-life study such that only samples on the extremes of certain design factors (e.g., strength, package size) are tested at all time points as in a full design. The design assumes that the stability of any intermediate levels is represented by the stability of the extremes tested. Where a range of strengths is to be tested, bracketing is applicable if the strengths are identical or very closely

related in composition (e.g., for a tablet range made with different compression weights of a similar basic granulation, or a capsule range made by filling different plug fill weights of the same basic composition into different size capsule shells). Bracketing may be applied to different container sizes, different fill weights in the same dose form and container-closure system, and to other circumstances as appropriate.

Component means any substance intended for use in the manufacture of a dietary supplement, including those that may not appear in the finished batch of the dietary supplement. Components include dietary ingredients (as described in Section 201 (ff) of the Federal Food Drug & Cosmetic Act – the Act), other ingredients and processing aids.

Container-closure system means the sum of packaging elements that together contain and protect the dietary supplement. This includes primary packaging (e.g. bottle and cap, desiccants, oxygen-absorbers, and cap liners that can come into contact with the product) and secondary packaging (e.g. label, and corrugated cardboard) if the latter are intended to provide additional protection to the dietary supplement.

Excursion testing means testing that is conducted to explore the possible effects of shipping conditions on the product stability.

Expiration date means the date placed on a dietary supplement container designating the time point prior to which a batch of the product should remain within the established shelf-life specifications, if stored under the conditions defined on the product container. The expiration date may also be called the shelf-life date, best-before date, best-by date, use-by date, or any similar description.

Long-term studies means stability studies conducted under the recommended storage condition for the shelf-life proposed (or established) on the label of a dietary supplement

as part of a shelf-life study protocol. Also known as real-time testing. Typically long-term means ambient condition but it could also be refrigerated or frozen conditions.

Matrixing means the design of a shelf-life study such that a selected subset of the total number of possible samples for all factor combinations is tested at a specified time point. At subsequent time points, other subsets of samples for all factor combinations are tested. The design assumes that the stability of each subset of samples tested represents the stability of all samples at a given time point. The differences in the samples for the same dietary supplement should be identified as, for example, covering different batches, different strengths, different sizes of the same container-closure system, and, possibly in some cases, different container-closure systems.

Pilot-scale batch means a batch of a dietary supplement manufactured and packaged by a smaller scale procedure and/or equipment fully representative of and simulating a full-scale (e.g. commercially marketed) production batch.

Product quality means that the dietary supplement consistently meets the established specifications for identity, purity, strength, and composition, and limits on contaminants, and has been manufactured, packaged, labeled, and held under conditions to prevent adulteration under Section 402(a)(1), (a)(2), (a)(3), and (a)(4) of the Act. Companies are free to assign other relevant parameters to quality such as organoleptic and any other characterizing product aspects.

Release specification means the combination of organoleptic, physical, chemical, biological, and/or microbiological tests and acceptance criteria that determine the suitability of a dietary supplement batch at the time of its release.

Reserve sample means a representative sample of a product and container-closure system that is held for a designated period of time after manufacturing and packaging.

Scientifically valid*:* means an analytical method that is based on scientific data or results published in, for example, scientific journals, references, text books, or proprietary research and is fit for its intended use.

Shelf-life (also referred to as expiration date or best-by period) means the time period during which a dietary supplement should remain within the established shelf-life specifications**,** provided that it is stored under the conditions defined on the product's packaging.

Shelf-life studies means long-term and accelerated studies undertaken on dietary supplement batches according to a defined stability protocol or procedure to establish or confirm the shelf-life of a dietary supplement.

Shelf-life study protocol means a detailed defined plan used to generate and analyze stability data in support of the shelf-life (expiration date) of a product in a given container-closure system. It should include product information, packaging information, time points and conditions employed, and methodology used to generate stability data.

Storage conditions and tolerances means the acceptable range of temperature, relative humidity and possibly lighting parameters of storage facilities for samples used for formal shelf-life studies.

User refers to the user of this document.

14.0 References

International Conference on Harmonization (ICH) – Guidance for Industry: Q1A(R2)

Stability Testing of New Drug Substances and Products

http://www.fda.gov/downloads/RegulatoryInformation/Guidances/ucm128204.pdf

ICH Guideline Q1D – Bracketing and Matrixing Designs for Stability Testing of New Drug

Substances and Products

http://www.ich.org/fileadmin/Public_Web_Site/ICH_Products/Guidelines/Quality/Q1D/Step4/Q1D_Guideline.pdf.

ICH Guideline Q1E – Evaluation of Stability Data

http://www.ich.org/fileadmin/Public_Web_Site/ICH_Products/Guidelines/Quality/Q1E/Step4/Q1E_Guideline.pdf

US Code of Federal Regulations Title 21, Part 111 [Current Good Manufacturing

Practices in Manufacturing, Packaging, Labeling, or Holding Operations for Dietary

Supplements]

http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?CFRPart=111%26showFR=1

American Herbal Products Association. Use of Marker Compounds in Manufacturing and

Labeling Botanically Derived Dietary Supplements. Silver Spring, MD: American Herbal

Products Association; 2001.

http://www.ahpa.org/Default.aspx?tabid=68#gdncmanufsalebotextr

Stability Testing of Dietary Supplements – January 2011

EMEA - "Guideline on Quality of Herbal Medicinal Products / Traditional Herbal

Medicinal Products" (CPMP/QWP/2819/00 Rev. 1)

http://www.ema.europa.eu/pdfs/human/qwp/281900en.pdf

15.0 Appendix

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TITLE: STABILITY TESTING PROTOCOL - ACCELERATED TIME STUDY	
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FILEPATH:	

	NAME & POSITION	SIGNATURE	DATE
REVISED BY:			
REVIEWED BY:			
APPROVED BY:			

1. PURPOSE

Example

2. REQUIREMENTS

3. DEFINITIONS

This is an example template of what a study protocol might look like. This example is not intended to be specifically or closely adhered to, and users should use it only as a guide in developing their own protocol. The values provided are fictitious and are intended solely to provide users with an example of how a study report might appear.

4. RESPONSIBILITY

5. OPERATION PROCEDURE - ACCELERATED STABILITY STUDIES

6. RECORDS

7. REFERENCES

8. CHANGE HISTORY

This is an example template of what a study protocol might look like. This example is not intended to be specifically or closely adhered to, and users should use it only as a guide in developing their own protocol. The values provided are fictitious and are intended solely to provide users with an example of how a study report might appear.

Addendum B - Example Shelf-life Study Report

STABILITY STUDY (REPORT & CHART) - REAL TIME

Product name :				Manufactured date :	
Bulk Code #				Shelf-Life Date:	
Bulk Lot #				Container :	
Finished Product				Closure:	
Finish Good Lot #				Storage conditions :	

Real Time Study At 3, 6, 9, 12, 18, 24 and 36 Months -----

Test Dates:	Intervals:	Appearance	Moisture Content	Friability	Hardness	Nutrient A	Nutrient B	Nutrient C	Nutrient D	Nutrient E
Label Claim					23 kp	5,000 IU	22.5 mcg	250 mcg	1.125 mg	150 mcg
	Months			NMT						
Range			0-5%	0.80%	20 - 26 kp	100-150 %	100-150 %	100-150 %	100-150 %	100-150 %
	0	PASS	2.30%	0.10%	24 kp	148	126	119	111	131
	3	PASS	2.30%	0.10%	24 kp	142	114	117	113	101
	6	PASS	2.30%	0.10%	24 kp	146	125	116	112	110
	9	PASS	2.20%	0.10%	24 kp	148	109	135	109	109
	12	PASS	3.80%	0.10%	24 kp	143	110	119	108	108
	18	PASS	2.30%	0.10%	24 kp	144	117	105	110	115
	24	PASS	1.70%	0.10%	24 kp	146	129	101	112	112
	36									

Comments

	Updated by :
	Stability Co-ordinator
	Approved by :
	Q. A. Supervisor

Completed Stability Report & Reviewed By: ----- --- Stability Co-ordinator   Date:

Nutrient A

Nurtrient B

Nutrient C

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