



GUIDELINE FOR VITAMIN A & D FORTIFICATION OF FLUID MILK

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ABSTRACT

This guideline is designed to help the processor concerning proper fortification of fluid milk products with vitamins A & D. It briefly discusses the history and need for vitamin fortification. Information is given about types of vitamin concentrates available, the problems involved in fortification, and the best methods for properly fortifying fluid milk products.

PREFACE

The first edition of this guideline was prepared by A. F. Zimmermann, consultant and former president of QC Inc., and J. W. Nisonger, now retired engineer for Embassy Dairy, the Southland Corp. Providing assistance were S. E. Barnard, The Pennsylvania State University, Les Latchford, Quality Control Director, Baxter Dairies, Ltd., Gary Senyk and Dr. W. Frank Shipe at Cornell University. The first revision represented the joint effort of Mr. Zimmermann and Professor Barnard, with the assistance of Mr. Nisonger. The second revision was prepared by Donald F. George, Vermont Department of Agriculture, Food & Markets with assistance from M. Jeffrey Bloom, Häagen-Dazs Company, Inc., and Gaylord B. Smith, Mohawk Associates.

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INTRODUCTION/HISTORY

Fluid milk products have been fortified with vitamins since the 1930's when an industry wide program for vitamin D fortification was initiated in an effort to prevent infantile rickets, a bone disease of children related to vitamin D deficiency. This practice, which was recommended by the American Medical Association's Council on Foods and Nutrition, was credited as instrumental in the near eradication of this disease in the US and elsewhere during this period (Selitzer, 1976; Stevenson, 1955). While methods including animal feed supplementation ("metabolized") and direct irradiation of the milk were initially used in an attempt to increase the vitamin D content of milk, the direct addition of vitamin concentrates proved to be most effective and became the accepted practice (Roadhouse & Henderson, 1950). As the addition of vitamin D to milk was becoming common practice, other nutrients were also being considered. This included vitamin A, which was added to milk in vitamin-mineral formulations manufactured in the 1940's.

From a regulatory perspective, vitamin fortification of milk was first addressed in the 1930's. In the 1939 edition of the Milk Ordinance and Code, *Vitamin D Milk* was defined as "milk that the vitamin D content of which has been increased by a method and in an amount approved by the health officer." Though no specific level was recommended or required, Section 6 of this document recommended monitoring levels by "bioassays of the vitamin D content as required by the health officer in a laboratory approved (by the health officer) for such examinations." Based on recommendations from the American Medical Association's Council on Foods and Nutrition, the level of "at least 400 U.S.P. units (currently International Units) per quart" was later defined for Vitamin D Milk in the 1953 edition of the Milk Ordinance and Code. This recommended level carried over into the 1965 Grade "A" Pasteurized Milk Ordinance (PMO) where the level of 400 International Units (IU) per quart was required for Vitamin D milk, though vitamin D fortification was considered optional. This document also addressed the option of fortifying milk with other vitamins and minerals under "Fortified Milk and Milk Products," though no levels were given.

As skim and lower fat milks became popular, the reduction of vitamin A levels with fat reduction became an issue since whole milk was considered a good source of vitamin A. This was addressed in the 1978 PMO under the standards of identity for lowfat and nonfat milks, which stated that in these products, "vitamin A shall be present in such quantity that each quart of the food contains not less than 2000 IU thereof within limits of Good Manufacturing Practices." Vitamin D fortification at 400 IU/qt. remained optional for all products. Acceptable levels within limits of "Good Manufacturing Practices" were generally interpreted as the range of 80% to 120% of the label claim for both vitamin A and vitamin D. Determination of vitamin levels was required at least annually "in a laboratory acceptable to the regulatory agency," though there were no established procedures to determine the competency of vitamin testing laboratories.

These general requirements for vitamin fortification of milk remained in place until the 1990's, when a number of regulatory changes were implemented. To some extent these changes were in response to the adoption of the Nutritional Labeling and Education Act (NLEA) of 1990. These changes also appeared to have been prompted by an episode involving a dairy plant where milk that was grossly over-fortified with vitamin D resulted in human illness (Jacobus et al. 1992) and also by the results of a number of survey studies suggesting that milk fortification levels were not meeting label claims (Brown et al, 1992; Holick et al, 1992; Nichols, 1991). The regulatory changes put into practice included a revision of the acceptable levels of vitamins, changes in standards of identity for fluid milk products and the implementation of a vitamin testing laboratory certification program.

On review of what was considered to be acceptable vitamin fortification levels, the Food and Drug Administration (FDA) revised the levels to 100% to 150% of the label claim from what was previously 80% to 120% to be more consistent with other regulations (Nichols, M-I-92-13). With the acceptance of the NLEA of 1990, the standards for lowfat and nonfat were eliminated, placing these fluid milk products under the "general standard" for milk, but named with defined nutrient content claims, i.e. reduced fat,

lowfat, nonfat (Federal Register, Schultz 1996). Compliance with the new guidelines was mandated as of January 1, 1998. Within these guidelines, vitamin A is required to be added to lower fat milks so that they are “nutritionally equivalent” to the reference standard of identity (milk). Though this would be based on 1200 IU per quart for milk, the fortification level of 2000 IU per quart was still recommended for these products and is routinely followed by the dairy industry. The optional addition of vitamin D remained the same.

To ensure proper fortification of dairy products it became evident that enforcement of product monitoring and laboratory testing was needed. In April of 1996, in response to proposals submitted at the 1993 and 1995 National Conference on Interstate Milk Shipments (NCIMS), Laboratory Quality Assurance Branch (LQAB) of the FDA initiated a certification program for laboratories testing for vitamins in milk. Wording in Section 6 of the 1995 PMO was changed to “assays ... shall be made at least annually in a laboratory that has been certified by the FDA ...using test methods acceptable to FDA or other official methodology which gives statistically equivalent results to the FDA method.” This program was designed to bring uniformity and credibility to vitamin testing requirements in an effort to ensure that fluid milks are properly fortified. A number of testing laboratories, from industry, regulatory and academia, participate in the program. Laboratories and methods that met all the required quality assurance requirements and analysts within such laboratories that performed all required split sample analyses with acceptable results were certified and listed in the Interstate Milk Shippers (IMS) List. The program was implemented in July 1997 when all official testing for vitamins A and D was required to be performed in IMS listed certified laboratories.

NEED FOR FORTIFICATION

Vitamin D is a regulator of calcium metabolism and is involved in the absorption of calcium in the intestines and in the mineralization process required for bone growth. Lack of vitamin D in growing children causes Rickets or other bone disorders, manifested by weak bones and bone curvature that result in bowlegs, knock knees and narrow chest. In adults, vitamin D deficiencies can result in softening and weakening of bones (osteomalacia) and can hasten the development of osteoporosis commonly associated with calcium deficiencies. While people generate their own vitamin D through exposure to the sun, the level can be insufficient where sunlight is lacking. Recommended use of sunscreen also blocks the UV radiation required for this process. Although milk as it comes from the cow is a poor source of vitamin D, fortified milk is considered an excellent source, especially because of its calcium content. Other foods considered good dietary sources of vitamin D are relatively rare but do include fatty fish, eggs and liver. Vitamin D is a fat-soluble vitamin.

Vitamin A performs many functions. One is to prevent night blindness by enabling the retina of the eye to respond to dim lights. Vitamin A is also needed for normal growth, maintenance and function of epithelial cells, which include skin cells and the cells lining the respiratory, gastro-intestinal and reproductive tracts. Whole milk is a good source of vitamin A, though because it is a fat-soluble vitamin, its value is reduced in lower fat milks.

REGULATORY REQUIREMENTS

Fortification Levels:

In the US, fluid milks, which make vitamin addition claims (fortified), must be fortified as indicated on the product label and in accordance with Federal and State regulations. Federal Regulations dictate the minimum requirements while specific state regulations may vary. The current US Federal Regulations for vitamin fortification levels, as stated in Title 21 of the Code of Federal Regulations (CFR), are:

SECTION 131.110 Milk

- (a) Description.
- (b) Vitamin addition (optional). (1) If added, vitamin A shall be present in such quantity that each quart of the food contains not less than 2000 International Units (IU) thereof within limits of good manufacturing practice. (2) If added, vitamin D shall be present in such quantity that each quart of the food contains 400 IU thereof within limits of good manufacturing practice.

Title 21 Standards of Identity for lowfat (Section 131.135) and skim milk (Section 131.143) were eliminated as of January 1, 1998. Standards for “Milk” (CFR 21 131.110) remained unchanged and serve as the “general standard” or reference standard for the lower fat products. Vitamin D fortification remains optional for all milk, while milks with lowered fat values are required to be fortified with vitamin A to at least the level of “nutritional equivalence” of what is present in whole milk (1,200 IU per qt.). Currently, regulatory agencies are suggesting the dairy industry to continue fortifying lower fat milks at the level of 2000 IU vitamin A per quart, which is consistent with item (b) of the milk standard (CFR 21 131.110).

To fortify “within limits of good manufacturing practices,” the acceptable range is 100 - 150% of label claims for both vitamins A and D (Summary of FDA M-I-92-13, Nichols, 1992): See Table 1

Over-Fortification Guidance:

Vitamins A and D are considered to be toxic if consumed in excessive levels. These are fat soluble vitamins that accumulate in tissue over time and are not easily eliminated from the body. Infants under 12 months of age may be at risk to the lower levels of these vitamins while the elderly and pregnant women may also be more susceptible to excessive levels. Based on the probability of adverse effects within a reasonable margin of safety, levels considered to be of public health concern have been determined. **“Vitamin A above 6000 IU per quart and vitamin D above 800 IU per quart should be considered harmful. Fluid milk products with levels of vitamin A or D above these values may create the potential for a public health threat and further distribution should be prohibited.”**

Nutritional Label Claims and Ingredient Declarations:

For fortified milks, the ingredient declaration, fortification levels and current % Daily Values (DV) are shown in Table 2:

Vitamin D is optional for all milk while vitamin A fortification is required for all lower fat milks. The vitamin A level of whole milk (3.25% BF) without vitamin A fortification is considered to be 1200 IU/qt or 300 IU per serving (6% DV). ***For vitamin D milks, though most processors are using vitamin D₃, there are some that use vitamin D₂. It is essential that the ingredient declaration accurately list the vitamins that are used.***

Additional PMO Requirements. The 1999 PMO addresses vitamin fortification in Appendix O and in other sections throughout the document. Specific requirements that should be emphasized include:

1. All vitamin concentrates must be added prior to the pasteurization process.
2. With continuous metered vitamin addition used with continuous pasteurization systems (i.e. HTST), metering pumps should be interwired to the HTST control panel to shut down during diverted flow and product recycle modes to ensure that vitamins are added only when product is in forward flow.

- The amount of vitamin concentrates used must be recorded and cross-referenced with the amount of product fortified to ensure that the actual amount of concentrate used closely matches what is required for the total product made.

Table 1: Fortification Limits

<u>Vitamin</u>	<u>Label Claim (IU/qt.)</u>	<u>Acceptable Range</u>	<u>Level for Public Health Concerns</u>
A	2000 IU/qt.	2000 – 3000 IU/qt.	> 6000 IU/qt.
D	400 IU/qt.	400 – 600 IU/qt.	> 800 IU/qt.

Table 2: Nutritional Label Claims and Ingredient Declarations

<u>Ingredient Declaration</u>	<u>Fortification Level (IU/qt.)</u>	<u>%DV of 1 cup (8 oz) serving</u>
Vitamin A Palmitate	2000	10%
Vitamin D ₃	400	25%
Vitamin D ₂	400	25%

- Fortified products must be tested by a FDA Certified Laboratory at least once per year.

The regulatory requirements will be further discussed and emphasized throughout this document.

CONCERNS WITH VITAMIN FORTIFICATION OF MILK

Inaccurate vitamin fortification of milk is of concern as vitamin levels below label claims prevent consumers from receiving the perceived nutritional benefits of the product, while excessive levels have the potential to be toxic. Though vitamin fortification procedures for milk are relatively simple to implement and follow, analytical surveys of market milk samples suggest that a large percentage of milks do not fall within the established compliance ranges. FDA issued the results of a survey of 406 IMS plants and 124 non-IMS plants in 1991 (M-I-91-7) when acceptable levels were plus or minus 20% of the label claims. The results indicated that approximately 20% of the milks were under-fortified while 27% of the milks tested were over-fortified for both vitamins A and D. Other studies supported these findings (Brown et al., 1992; Holick et al., 1992) More recent results of ongoing survey work of the New York State Milk Quality Improvement Program at Cornell University reflect current acceptable levels of 100% to 150% of label claim. Though improvement has been seen over recent years, results of milk samples tested from 1997 to 2000 indicated that only 51.4% of the milks fortified with vitamin A (516 samples) and 46.3% of milks fortified with vitamin D (648 samples) met the acceptable levels (Murphy et al, 2001). In most cases the milks were under-fortified, while 22% of vitamin A results and 18.7% of vitamin D results fell within 20% below the target levels of 2000 IU/qt. and 400 IU/qt., respectively, suggesting a reasonable, though insufficient effort was made in product fortification by most processors. These results also suggest that minor errors in fortification procedures can result in non-compliance. The breakdown of the results of this survey work is as follows:

	<u>Vitamin Test Range</u>	<u>% of Samples</u>
<u>Vitamin A</u>	< 2000	51.4
(516 samples)	2000 – 3000	44.5
	> 3000	4.1
<u>Vitamin D</u>	< 400	46.3
(648 samples)	400 – 600	47.7
	> 600	6.0

* Reported results are of assays performed in a FDA certified laboratory. Data from the Milk Quality Improvement Program 1997-2000.

The fact that milks are found to be incorrectly fortified has been attributed to a number of factors. Commitment of management in some cases is in question, where adequate procedures to ensure proper vitamin fortification are lacking. "Human error" on the part of employees responsible for this step during processing is often to blame. Other influential factors are related to natural levels, the method(s) of addition and the handling and storage of the vitamin concentrates and the fortified products. With the advent of the laboratory certification program and improved vitamin testing procedures, regulatory agencies are more likely to take a more rigorous approach to enforcing these standards. In this regard, it is essential that dairy processors develop a program that will ensure proper vitamin fortification levels

PROCEDURES FOR PROPER VITAMIN A & D FORTIFICATION OF FLUID MILK PRODUCTS

Vitamin Concentrates:

Several companies supply vitamin A & D concentrates to the dairy industry. The first step in developing a proper fortification program is to establish a good working relationship with a supplier that is willing to provide expert technical assistance along with reliable vitamin concentrates. Concentrates and methods of addition need to be selected and implemented based on the processing parameters that are specific to each plant. Once a supplier and method of addition is chosen and implemented, fortified milks should be tested to ensure that vitamin levels are correct.

A number of different types of vitamin concentrates are available, including oil based and water dispersible formulations. Most contain vitamin D₃ (or less often vitamin D₂) and/or vitamin A palmitate in a carrier generally consisting of a combination of any of the following: corn oil, water, polysorbate 80, propylene glycol and glycerol monooleate. Antioxidants and/or preservatives may also be added. Concentrates containing both vitamins A and D are used for lowfat milks by a majority of processors, although they may also be added separately. Vitamin D concentrates are used alone for homogenized milk. The most frequently used concentrates are formulated such that 1 ml of vitamin D concentrate fortifies 500 quarts of homogenized milk with vitamin D (400 IU per qt.) and 1 ml of vitamin A/D concentrate fortifies 100 quarts of reduced fat, lowfat milks or skim milks with both vitamins A (2000 IU per qt.) and D (400 IU per qt.). Where small batches of milk are processed, more dilute concentrates will improve measuring accuracy (i.e., 1 ml to fortify 100 quarts of homogenized milk with vitamin D). Most fluid milk processors add slightly higher levels of concentrate (i.e., 10 to 20%) to the calculated usage rate to ensure that the milks fall within the acceptable range of 100% to 150% of the label claim. Some suppliers are willing to customize formulations based on their customer's needs and more dilute concentrates are available to improve accuracy.

Mishandling and prolonged storage of vitamin concentrates can result in loss of vitamin potency. It is best to store vitamin concentrates as recommended by the manufacturer, avoiding heat and light. If precipitates or sediment are noted, bring these to the attention of the manufacturer. Generally, oil based concentrates should not be stored under refrigeration unless recommended by the manufacturer, as they may precipitate. If stored under refrigeration, viscous concentrates should be brought to room temperature and mixed gently but thoroughly, before addition. Vitamins will also lose potency with age. Excessive exposures to heat, light and air have the potential to shorten vitamin shelf-life even more so.

To ensure that the vitamin concentrates are of proper potency, rotate stocks and order only what will be used in a short period of time, generally within a three-month period. Water dispersible concentrates generally have a shorter shelf life and should be used more rapidly. Some vitamin suppliers have minimum

orders which small processors may not be able to use within the practical storage life of the concentrate. If this is the case, it is suggested that small processors share minimum orders or buy smaller amounts from larger processors that have a rapid turnaround of their concentrates. Using concentrates that require higher levels of addition per unit volume of milk will speed the usage rate so that minimum orders will be depleted more rapidly. Regardless, vitamin concentrates should be used or replaced well before their expiration date and/or when routine testing of milk indicates that potency is lost.

Methods and Location of Addition:

Both manual (batch) addition and continuous addition with metering pumps have been used to fortify fluid milk products. Regulations require that vitamin concentrates must be added to milk prior to pasteurization. Vitamin fortification has been accomplished by the addition of vitamins at many different points in the processing system including addition to the raw tanker before unloading, to the raw silo tank, to the pasteurizing vat (when vat pasteurization is used), to the HTST balance tank, or directly into a milk line prior to pasteurization. The type of vitamin concentrate selected for use will depend on the point and method of addition. Oil based vitamin concentrates are required to be added after the products have been standardized. Though generally not recommended, water dispersible vitamin concentrates can be added prior to separation, which may be more advantageous in some situations.

It is recommended that vitamins be added after separation/standardization and before homogenization. Homogenization will help disperse and stabilize the vitamins. Vitamins A and D are fat-soluble and have the potential to become more concentrated in the fat portion of the milk. Water dispersible vitamin concentrates are available which minimize this problem, allowing addition before separation/standardization. Vitamin D is added directly to the incoming raw milk (i.e., tank truck) in some situations. This is not the preferred method of addition and processors who use this procedure should perform confirmatory assays to ensure proper fortification levels of each product.

Fortified "rework" milk must not be re-fortified during reprocessing. If fortified rework milk represents a significant portion of a product, then this should not be re-fortified or should be only fortified to the extent that sufficient vitamin concentrate is added for the unfortified portion. For more information on product rework, see Dairy Practices Council (DPC) Guideline # 63.

Manual (Batch) Addition:

Manual addition is routinely used for batch pasteurized milk products or for small batches of product destined for High-Temperature Short-Time (HTST) pasteurization. Although metered addition is recommended for nearly all HTST processing systems, manual addition is still used for all products in some HTST operations.

The batch procedure requires accurate measurement of the volume of milk to be fortified, accurate measurement and addition of the required amount of vitamin concentrate and proper mixing. Vitamins should be added to prestandardized milk in batching tanks where it can be properly mixed before pasteurization. Concentrates should be measured in the proper amount using calibrated non-breakable (plastic) graduated cylinders (do not use medicine cups or kitchen measuring devices) of appropriate size (i.e., if 6 ml are needed, use a 10 ml cylinder), or plastic calibrated pipettes for smaller batches. After addition, any residual vitamin concentrate left in the cylinder or pipette should be rinsed with and into the milk to be fortified to ensure that all the concentrate is added. It may be advantageous to premix the vitamin concentrate with a small volume of milk before addition to the batch tank. In small batches the degree of error is large so extreme care in measurement should be taken.

Manual addition of vitamin concentrates to the balance tank during HTST pasteurization is used in some cases. This is not a recommended procedure. If this procedure is necessary, the entire amount of milk

processed and corresponding amount of vitamin concentrate should be accurately calculated. All the milk processed for that batch should be in the pasteurized storage tank and mixed thoroughly before that milk is allowed to be packaged. Adding the required amount of vitamin concentrate in increments may help ensure its distribution.

Daily records should be kept. With batch fortification, the amount of each type of milk processed, the type and amount of vitamin concentrate added to each batch, the mixing time before pasteurization, and any other relevant information should be recorded.

Metered Addition:

Metering pumps allow continuous addition of vitamin concentrate based on the flow rate of the HTST or other continuous flow systems. Metered addition at a point after standardization and before pasteurization is required when in-line fat standardization is used. A recommended point for injection of the vitamin is just prior to the homogenizer, which in most cases is a point of low pressure. The product pressure at the injection site should be determined to ensure that it does not exceed the capabilities of the metering pump. Negative pressures at the point of injection can cause excessive volumes of vitamin concentrate to be drawn into the milk unless a positive displacement pump is used. A check valve must also be installed at the point of injection to prevent contamination of the vitamin lines with milk. Another injection site often used is the balance tank. Preferably the vitamin concentrate should be injected into the milk line coming out of the balance tank, just before the milk pump. Dripping concentrate into the top of the balance tank is not recommended as this could result in inadequate mixing of concentrate floating on the milk surface. The basic requirements for metered addition of vitamins are shown in Figure 1 and include the following:

1. Sanitary Metering Positive Displacement Pumps (with required wiring, switches); - separate pumps for each concentrate, each heating system, calibrated.
2. Tubing, Food Grade, Generally 1/4-inch I.D. max., Peristaltic if Required; - from reservoir to pump, from pump to injection point, shielded.
3. Check Valves, Quick Release, Cleanable (connectors and adapters); - for point of injection, one for each concentrate line.
4. Calibrated Vitamin Reservoirs, Shielded.

Vitamin supply companies are generally able to supply or assist in obtaining the required items for metered addition. Separate delivery systems, including pumps, tubing, check valves and reservoirs, should be used for each vitamin concentrate metered into the same HTST system. All metering pumps and components should conform to the requirements of the *PMO* for equipment employed in sanitary applications and/or meet the 3-A Standards. Once a metering system is in place it needs to be calibrated. Standard Operating Procedures, including record keeping and routine maintenance, need to be developed.

Metering Pumps. Two types of metering pumps are generally used. Piston positive displacement pumps have been used extensively for vitamin addition by the dairy industry. With piston pumps, delivery rates are most often set with a micrometer, allowing accurate and reproducible amounts of vitamins to be added. These pumps have proven to be dependable and require little maintenance, although routine cleaning is required. Peristaltic (tubing) positive displacement pumps are becoming more popular, offering precise control and ease of cleaning (only the tube is in contact with the vitamin concentrates). The peristaltic tubing of these pumps needs to be replaced on a routine basis (i.e., every 1-2 months or more often if needed). Peristaltic pumps with microprocessor controls are currently in use by many dairy plants. These allow for easy adjustments of settings as well as for digital measurement of the volume of concentrate delivered. Regardless of the type of pump used, it must be capable of working with the pressures of the

milk flow at the site of injection. Metering pumps should be wired with on/off switches so that the appropriate pump can be easily turned on for the appropriate product. Some in-line standardization systems can be wired to automatically turn on the appropriate pump (i.e., A/D concentrate) for the appropriate product (i.e., lower fat milks). **All pumps must be wired into the control panel so that vitamin is metered only when the system is in forward flow and not when recirculating.**

Tubing. Tubing should be food grade and should have relatively narrow inner diameter ($\leq 1/4$ inch). Additional connecting pieces (i.e., reducers, clamps, etc.) may be needed to connect the tubing to the pumps, check valves and reservoirs. The system should be set up near the point of injection to minimize the length of tubing both into and away from the pump. Tubing and reservoirs should be shielded from light. Tubing should be routinely inspected for air bubbles, cracks and other signs of wear and replaced when necessary, before leaks occur. For peristaltic pumps, pump head tubing should be sized and selected according to the manufacturers recommendations. It should be of food grade material. This tubing needs to be replaced on a more frequent, routine basis (1 to 2 months, or sooner if needed).

Check Valves. Check valves should be stainless steel, of sanitary design and with a quick release for easy tube attachment. They should be set into a pipe cap with one check valve for each vitamin delivery system. Set-ups with “T”-adapters to connect tubing from two pump systems to one check valve are not recommended. If the injection site is in a “T” pipe, this should be as short as possible to ensure mixing in the product flow. Other considerations for the injection site and design should include whether it is accessible and cleanable.

Calibrated Vitamin Concentrate Reservoirs. Vitamin reservoirs are on the suction side of the pumps with the tubing either fully submerged or connected to a port or tubing adapter at the bottom of the reservoir. Concentrate levels of the reservoirs should be recorded at the start and finish of each product processed. Large plastic graduated cylinders (i.e. 1000 ml) or calibrated bottles have been used. Alternatively, usage levels can be checked with a calibrated measuring stick held to the outside of the bottle or by calculating the weight of the concentrate used (weight at start minus weight at end) and converting it to volume (mls) based on the concentrate density. Vitamin reservoirs should also be shielded from light and environmental contaminants (i.e. water spray). Reservoirs should be filled only with what is needed for a days processing, but enough so that it will not run out.

Calibration of Metering Systems. Delivery rates of metering pumps are based on the flow rate of the processing system and the concentration of the vitamin. It is important that delivery rates are accurately determined and routinely checked as small errors in calculations and/or measurements can have a major effect on the final concentration in the finished product. Once a metering system is fully installed, the pump should be calibrated using the entire delivery system (tubing, check valves, reservoir, etc.). Regardless of whether the system has microprocessor controls or manual settings, **all calibrations should be checked using a certified graduate cylinder and a stop watch**

Vitamin Pump Delivery Rate Calculations:

$\text{Pump Delivery Rate (ml/hr)} = \frac{\text{HTST Flow Rate (gal/hr)} \times 4 \text{ (qt./gal)}}{\text{Quarts Fortified per ml of Concentrate (qt./ml)}} = \text{qt./hr.}$

Example: If HTST System Flow Rate = 3,000 Gal/hr

Pump 1. Vitamin A & D conc: 1 ml fortifies 100 qts.

$$\text{Delivery Rate} = \frac{3,000 \text{ gal/hr} \times 4 \text{ qts/gal}}{100 \text{ qt./ml}} = \frac{12,000 \text{ qts/hr}}{100 \text{ qt./ml}} = 120 \text{ ml/hr or } 2 \text{ ml/min}$$

$$+ 10\% * = \frac{12,000 \text{ qt./hr} \times 1.1}{100 \text{ qt./ml}} = 120 \text{ ml/hr} \times 1.1 = 132 \text{ ml/hr or } 2.2 \text{ ml/min}$$

Pump Calibration (run time=volume): 30 min = 60 ml, 60 min = 120 ml, 90 min = 180ml

Pump 2. Vitamin D conc: 1 ml fortifies 500 qts.

$$\text{Delivery Rate} = \frac{3,000 \text{ gal/hr} \times 4 \text{ qts/gal}}{500 \text{ qt./ml}} = \frac{12,000 \text{ qts/hr}}{500 \text{ qt./ml}} = 24 \text{ ml/hr or } 0.4 \text{ ml/min}$$

$$+ 10\% * = \frac{12,000 \text{ qt./hr} \times 1.1}{500 \text{ qt./ml}} = 24 \text{ ml/hr} \times 1.1 = 26.4 \text{ ml/hr or } 0.44 \text{ ml/min}$$

Pump Calibration (run time=volume): 30 min = 12 ml, 60 min = 24 ml, 120 min = 48 ml

*Concentrates are often added at a rate slightly above (i.e., 10-20%) what is recommended.

For pump calibrations, start the pump, making sure all air is purged from the lines. When concentrate is flowing at a uniform rate, place the check-valve end to drip into the graduated cylinder and start the stopwatch. Measure the volume after the desired time (i.e., 30 min to 2 hr) or alternatively stop timing when a desired volume is reached (50 to 100 ml). When first setting a vitamin pump short times or small volumes can be used until the delivery rate is fine tuned. Before operation, the delivery rate should be checked with an extended run time, generally with final volumes of 50 to 100 ml of concentrate. After a pump system is put into operation an approved analytical laboratory should confirm the vitamin levels in the milk. Delivery rates should be checked at least bi-weekly with a graduated cylinder and a stop watch, even when microprocessor pumps with volume readouts are used.

Operating Procedures. Most HTST vitamin-metering systems are set up with two pumps; one for vitamin A concentrate and the other for vitamin A or A/D concentrates. Daily operation requires: 1) ensuring that the concentrate reservoirs are full and levels recorded, 2) ensuring that the appropriate pump is turned on promptly at the start for the appropriate product being processed, 3) ensuring that pumps are switched promptly at appropriate times (i.e., D only for Homogenized to A/D for lower fat) and/or turned off promptly at appropriate times and 4) ensuring that the system is functioning properly. Keeping accurate records, as described in detail in the next section, will help ensure proper operation and fortification of milks.

Maintenance Procedures. Maintenance procedures required for metered addition include daily inspection of tubing for leaks and air bubbles, daily inspection of check valves and pump components, weekly cleaning of vitamin reservoir (discard residual concentrate), bi-monthly cleaning of pump heads (piston pumps), monthly or bi-monthly replacement of peristaltic tubing (peristaltic pumps), and bi-weekly calibration checks. These are suggested time frames, which may vary depending on the needs and use of individual plants. Regardless, a schedule for routine maintenance should be developed and should include cleaning procedures that prevent the metering system from becoming a source of microbial contamination.

RECORD KEEPING

Section 6 of the 1999 PMO states that “all facilities fortifying products with vitamins must keep volume control records. These volume control records must cross reference the form and amount of vitamin D, vitamin A and/or vitamin A & D used with the amount of products produced and indicate a percent of expected use, plus or minus.”

Inaccuracies in vitamin fortification most often occur due to personnel error or neglect. Keeping and reviewing accurate daily records will help ensure that errors do not occur. In many cases vitamin addition is forgotten or the amount of concentrate needed is miscalculated, especially with manual addition. In pump delivery systems, the operator may not switch on the proper vitamin pumps at the proper time, the vitamin pumps may run during divert flow resulting in excess vitamin or the vitamin reservoirs may not be properly filled and may empty during processing (digital pump read-outs will continue to register vitamin flow). Sources of error may also occur due to the use of mishandled or old concentrates, malfunctioning delivery systems or the use of rework milk (see DPC # Guideline 63). Standard operating procedures that include keeping accurate records should be developed that help prevent fortification errors. Having operators write down times for each step helps ensure that vitamins are added accordingly. A sample record keeping form is shown in Figure 2. Following are items that should be recorded:

1. *Total volume of each milk type processed* (i.e., homo., 2%, 1%, skim). In batch systems this should be determined for each batch. For HTST or other continuous flow systems, this should be based on product flow rate or other means of measurement (metering system). If volume is determined by product flow, the time processing begins and the time processing stops for each product should be recorded to determine the total processing time and calculated volume. These times should be indicated on the HTST recording chart. With a two pump system, one pump for vitamin D for homogenized and one pump for vitamin A & D for lower fat milks, the volumes of the lower fat milks can, in many cases, be combined (e.g., 4.5 hrs for lower fat milks at 3000 gal/hr = 13,500 gal or 54,000 qt.).

2. *Type and total volume of vitamin concentrate added for each product.* For manual or batch addition this is easily determined from the measuring device used. For metering systems, this should be based on readings from calibrated reservoirs although it can also be determined from vitamin pump flow rates or the pump's digital read-out. If based on flow rate, record each time the vitamin pump is turned on (when milk is in forward flow) and each time the pump is turned off to determine the total running time. This time should match the product run time in forward flow. Calculate the volume used based on the pump flow rate (e.g., 4.5 hours at 132 ml/hr = 594 ml A/D concentrate). Pumps with digital read-outs of delivery volumes will continue cumulating even if the reservoirs are empty. Calculated or digital reads of concentrate volumes should be confirmed with calibrated vitamin reservoirs, recording the starting volume (or weight) and ending volume (or weight) of each concentrate used. Many of these items (i.e., when a specific pump is turned on) can be recorded on the HTST chart.

3. *Cross reference milk volume with concentrate volume and determine the percent of actual use.* Determine the amount of vitamin concentrate required to fortify the milk that was run (i.e., 54,000 qt. when 1 ml of A/D concentrate fortifies 100 qt. requires 540 ml concentrate) and determine what percentage was actually used (e.g., if 594 ml concentrate was used $594/540 \times 100 = 110\%$)

Daily records should be reviewed to make sure that the actual vitamin concentrate used matches the theoretical amount needed for the amount of milk processed. All daily records should be filed and kept available for regulatory review. Daily records can be transferred to a cumulative record sheet (See Figure 2).

Other information that should be recorded includes ordering, lot numbers and rotation of concentrates and the routine maintenance schedule and procedures for metering systems.

<p>OTHER CONSIDERATIONS REGARDING LEVELS AND STABILITY OF VITAMINS A AND D IN MILK</p>

Natural Levels of both vitamins A and D are associated with the fat phase of the milk. Milk and milk products containing a large proportion of fat are relatively good dietary sources of vitamin A, but as is the case with other natural foods, the vitamin D content of unfortified milk is quite low. Removal of fat will result in the loss of vitamin A, which is the basis behind required fortification of lower fat products.

Natural levels of both vitamins A and D in milk will vary from herd to herd and will depend on nutritional and lactation status of the cows, exposure to sunlight and/or the time of the year. Natural levels of vitamin A have been reported to range from 400 IU in the winter to 1500 IU in the summer. Vitamin D has been reported to range from 5 IU in the winter to 40 IU in the summer. These values were reported when pasture feeding was more common. However, present day feeding practices that limit pasture feeding of cows along with the commingling of milk supplies gives a more stable value throughout the year. Standardized homogenized milk has been shown to contain from 800 to 1,500 IU vitamin A, though values outside this range occur. The general standard for 3.25% fat milk assumes 1200 IU per quart or 6% DV per 8 oz serving. Current estimates for the contribution of natural vitamin A in milk are based on 300-400 IU/qt per percent (1%) of fat. Thus the approximate natural levels in 2%, 1% and skim milks are 600-800, 300-400 and 0 IU/qt. respectively. The contribution of natural vitamin D to milk is insignificant.

Most commercial concentrates are prepared to fortify milks with 2,000 IU per quart vitamin A and/or 400 IU per quart vitamin D. If concentrates are of proper potency and are used as directed, vitamin A levels should fall within the acceptable range (2,000 - 3,000 IU per qt.) regardless of the contribution of natural levels. The expected vitamin A levels of 2 %, 1 % and skim would be approximately 2,600, 2,300 and 2,000, respectively.

Fortified "rework" milk must not be refortified during reprocessing. If fortified rework milk represents a significant portion of a product, then this should not be re-fortified or should be only fortified to the extent that sufficient vitamin concentrate is added for the unfortified portion.

Stability of vitamins in milk depends on the vitamin and milk storage conditions, especially exposure to light. Vitamin D is stable in homogenized whole milk and is not affected by pasteurization or other processing procedures. Generally, no significant loss of vitamin D will occur within expected shelf-life periods though vitamin D has been shown to deteriorate with the growth of certain spoilage microorganisms. Vitamin A may deteriorate gradually under normal storage conditions of milk. When milk is exposed to sunlight or fluorescent light, especially in transparent containers, vitamin A can be rapidly destroyed (Shipe et al, 1984). This photo destruction of vitamin A is dependent on the intensity and wave length of light, length of exposure (time) and the milk source. The use of amber or brown glass bottles, pigmented plastic containers formulated with specific light barriers and paper cartons has been shown to retard this destruction. Paper, especially with limited print or ink, is not exempt from light degradation, though it is generally more protective than the transparent plastics. Use of gold shields over fluorescent tubes has also been shown to help reduce losses by modifying the wavelength of the light.

There is some question as to the influence of microbial growth on the levels of vitamins in milk. Recent work suggests that vitamins can deteriorate due to microbial activity such that vitamin analyses of aged milk samples will not reflect the actual level of fortification. All testing should be done on fresh samples.

VITAMIN TESTING IN FLUID MILK

Section 6 of the 1999 PMO states that “assays ... shall be made at least annually in a laboratory which has been accredited by the U. S. Food and Drug Administration and which is acceptable to the regulatory agency, using test methods acceptable to FDA and other official methodologies which gives statistically equivalent results to the FDA methods.” Some states may require more frequent testing. Currently HPLC methodologies are the testing procedures of choice for vitamins A & D in milk. FDA is involved in the standardization of testing procedures among laboratories and has implemented a certification program for vitamin testing laboratories. Current listings of certified laboratories can be found in the IMS Quarterly Report. Samples for vitamin testing should be sent to an approved laboratory within a few days of processing, kept cold ($\leq 4^{\circ}\text{C}$) ($\leq 39.2^{\circ}\text{F}$) without freezing and protected from light. Once received at the laboratory, analyses should begin as soon as possible. With the laboratory certification program in place, vitamin fortification levels should be more actively enforced. Certification, at the present time, is limited only to white milk.

SUMMARY

Accurate fortification of milks with vitamins A and D is essential to ensure that consumers are receiving the perceived nutritional benefits of milk products and that no milks are distributed that present a public health concern. Vitamin fortification procedures as outlined in this document and summarized in Appendix A, if followed, should result in a fluid milk supply that is in compliance with nutritional labeling and regulatory fortification standards.

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FIGURE 1: METERING PUMP SYSTEM FOR FLUID MILK FORTIFICATION

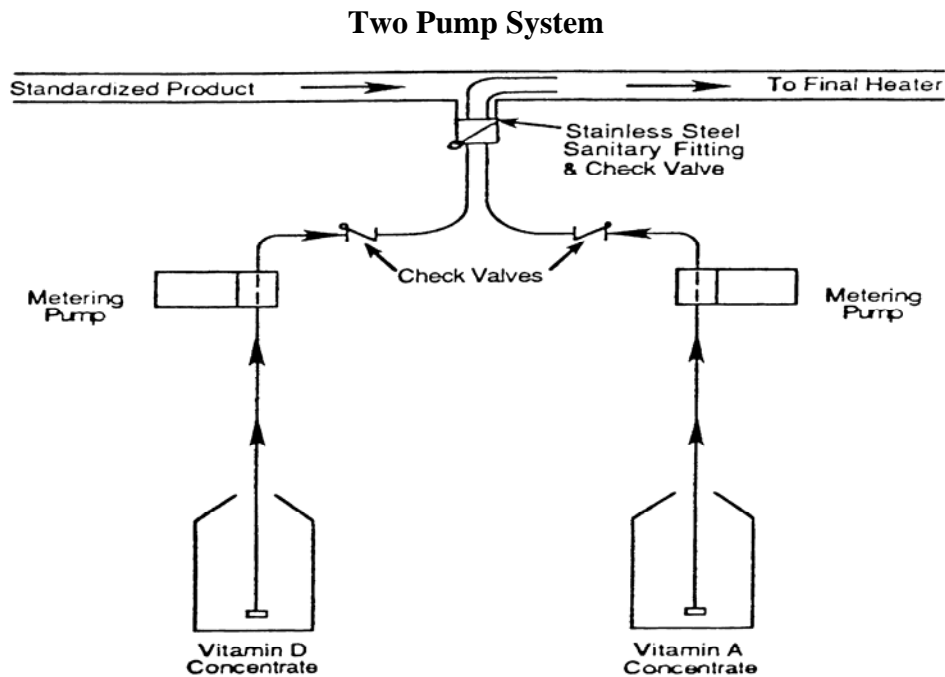


Figure 1 details a vitamin fortification installation using two pumps and two vitamin concentrate sources. This enables changing from different vitamin concentrates via activating either or both pumps.

Recommendations:

1. Use sanitary check valve(s) to separate milk lines from vitamin concentrates.
2. Keep all milk contact surfaces of sanitary design, easily cleanable, and available for inspection.

Summary Guideline-Fact Sheet:

APPENDIX A - VITAMIN A & D FORTIFICATION OF FLUID MILK

Vitamin fortification of fluid milk should be done in a manner that results in levels that are accurate and agreeable to product label claims as determined by routine testing. Following are general guidelines that should help maintain correct vitamin fortification levels in your fluid milk products.

1. **Work with your vitamin supply company** to develop a fortification program that will work best for your operation. Vitamin companies should be able to provide expert technical support in this regard.
2. **Accurately determine the amount of vitamin concentrate needed** according to the manufacturers recommendations. Appendix O of the PMO (1999 Rev.) states that "fluid dairy products are allowed, not less than 100% and not more than 150% of the required values or label claims to be in compliance with good manufacturing practices." Typical label claims and allowances are:

	<u>Acceptable Range</u>	
Homogenized Vitamin D Milk	(Vit. D-label claim: 400 IU/qt)	Vit. D: 400 - 600 IU/qt
Fortified Lower fat milks	(Vit. D-label claim: 400 IU/qt)	Vit. D: 400 - 600 IU/qt
	(Vit. A-label claim: 2000 IU/qt)	Vit. A: 2000 - 3000 IU/qt

Currently, fortification with vitamin D is optional in all products while fortification with vitamin A is required for lower fat products (i.e., 2%, 1% & skim). **All vitamin addition must be done before the milk is pasteurized.** Though natural vitamin D in milk is insignificant, natural vitamin A levels associated with the butterfat should be considered. Approximately 300 IU/qt are contributed per percent of fat (i.e., Skim - 0 IU/qt; 1% - 300 IU/qt; 2% - 600 IU/qt). Generally, concentrates formulated to provide 2000 IU/qt, when used as directed, will fortify all lower fat products to levels within the acceptable ranges (see above).

3. **Batch fortification** requires accurate measurement of milk volume, accurate measurement of the vitamin concentrate required for that amount of milk, and sufficient mixing time. Appropriate sized graduated cylinders should be used for measuring concentrates while any residual concentrate left in the cylinder should be rinsed out with milk into the batch to be fortified. Ideally, add the appropriate vitamin concentrate after the milk is standardized and allow sufficient mixing before pasteurization. If concentrates must be added manually at the balance tank, add in increments during processing and be sure that the amount of milk in the pasteurized storage tank agrees with the amount of concentrate used and that the product is thoroughly mixed before packaging.
4. **Metered fortification** using vitamin metering pumps can accurately deliver the required amount of concentrate during product flow. Metering pumps commonly used are positive pressure piston or peristaltic type pumps, some of which provide digital readouts of flow rates and amounts used. Pumps are calibrated to continuously feed the correct amount of concentrate to the milk based on the product flow rate. Metering pumps should be connected to the control panel so that they are turned off during divert flow. The best point of metered addition is after standardization prior to homogenization and pasteurization. The exit line of the balance tank is also used. Check valves to prevent backflow are required at the point of injection. Pump calibration and maintenance, including replacement of tubing, should be done routinely. Vitamin reservoirs should be calibrated so the volume of concentrate used can be recorded daily.
5. **Keep daily records** of the amount of vitamin concentrate used and match this to the volume of milk fortified (theoretical use). A check sheet for the operator should be in place and checked daily by a supervisor. With batch addition, the check sheet should include the volume of each milk type, the amount of concentrate added and the mixing time. With a pump system, the check sheet should include the starting time/concentrate volume for each milk product, the time vitamin pumps are started or switched (i.e., from D to A & D), and the volumes of concentrate used. Pumps providing digital readouts of the concentrate volume used should be confirmed with calibrated reservoirs, as these will read even if the reservoir is empty. Check sheets will help prevent "human errors".
6. **Vitamin concentrate storage, avoiding heat and light**, should be according to the manufacturers recommendations. Order only what you will use in a short period of time (generally within 3 months), rotate stock and use opened bottles as quickly as possible. Pump reservoirs and tubing should also be protected from light and maintained so that concentrate remaining after processing is at a minimum or is to be cleaned and replaced with fresh concentrate before the next day.