Operational Guidelines for Addition Methods of Rosemary Grapefruit Concentrate in Application Area

I. Implementation Standards

GB 2760-2024 "National Food Safety Standard for the Use of Food Additives", GB 1886.311-2021 "National Food Safety Standard - Food Additive - Grapefruit Extract", GB 14881-2013 "National Food Safety Standard - General Hygienic Specification for Food Production", EC 1223/2009 "EU Cosmetics Regulation", GB 30616-2020 "General Rules for Food Supplements".

II. Scope of Application

This guide applies to scenarios including functional foods, dietary supplements, cosmetics, Foods for Special Medical Purposes (FSMP), pet food, and natural preservative compounding. It covers the entire process of addition, operation, measurement, and quality control for food-grade/cosmetic-grade Rosemary Grapefruit Concentrate Powder (total polyphenol content ≥15%, carnosol ≥5%). It encompasses laboratory sample preparation, small-batch production, and industrial-scale continuous production modes.

III. Operational Principles

Precise measurement, uniform dispersion, adaptation to system characteristics, strict compliance with limits, and preservation of active efficacy to ensure finished product efficacy stability, sensory compliance, safety, and regulatory adherence, while balancing process characteristics and component synergy needs across different fields.

IV. Core Characteristics of Rosemary Grapefruit Concentrate Powder

The concentrate powder referred to in this guide is a composite natural functional powder (particle size 80-120 mesh, moisture ≤5.0%). Its core active components are rosemary phenolic diterpenes (carnosic acid, carnosol) and grapefruit polyphenols. Its characteristics determine key points for addition operations, requiring precise adaptation:

1. **Active Stability:** Heat resistance ≤180°C (pre-emulsification protection required above 180°C). pH tolerance range: 3.0-7.5. Strong light, high temperature, and high humidity environments can easily cause polyphenol oxidation and degradation, requiring light- and oxygen-protected operations.
2. **Solubility:** Easily soluble in purified water and 50%-70% ethanol aqueous solutions; slightly soluble in oils/fats. Direct addition of pure powder can lead to agglomeration; industrial production prioritizes preparation as a suspension/solution.
3. **Compatibility:** Synergistic effects with antioxidant components like Vitamin C, Vitamin E, and Coenzyme Q10. Compounding with citrus essential oils or mint extract can mask herbal off-notes. Avoid direct contact with strong oxidizing agents, strong acids (pH <3.0), and strong alkalis (pH >7.5).
4. **Safety:** ADI limit 0-1 mg/kg body weight (expressed as carnosic acid). Addition level ≤2.0% (cosmetics),

≤200 mg/kg (food, based on fat content). No acute toxicity or strong skin irritation.

5. **Hygroscopicity:** Slightly hygroscopic. Powder must be stored sealed. After opening, it is prone to moisture absorption and caking, affecting dispersion uniformity.

V. Core Addition Principles

1. **Compliance & Limit Principle:** Strictly adhere to standard limits for the corresponding field. Food applications must calculate dosage based on GB 2760-2024; cosmetics applications must not exceed 2.0%. When compounding, calculate total active component intake; exceeding limits is strictly prohibited.
2. **Precise Measurement Principle:** Use appropriately accurate equipment according to the addition range: high-precision balances for small batches, automatic dosing equipment for industrialization. Avoid errors leading to efficacy fluctuations or safety risks.
3. **Uniform Dispersion Principle:** Powder requires pre-mixing or pre-dissolution treatment to avoid localized high concentrations causing sensory abnormalities or skin irritation. Ensure homogeneous distribution within the system.
4. **System Compatibility Principle:** Select corresponding addition timing, method, and dissolution medium based on product state (liquid/semi-solid/solid), process (hot/cold processing), and pH value.
5. **Efficacy Preservation Principle:** Avoid scenarios involving prolonged high temperatures or strong light exposure. Liquid preparations can be nitrogen-purged for protection to maximize retention of polyphenol and phenolic diterpene activity.
6. **Compounding Synergy Principle:** When compounding with other ingredients, first dissolve/disperse the main system materials, then add the concentrate preparation. Determine the optimal compounding ratio through small-scale testing first.

VI. General Basic Operation Specifications

(I) Measurement Equipment Requirements

1. **Small-Batch Production (≤50 kg):** Use electronic analytical balances (precision 0.001 g) or high-precision electronic scales (precision 0.01 g), suitable for sample testing and laboratory preparation.
2. **Industrial Large-Batch Production:** Use fully automatic powder dosing equipment (precision 0.1 g) or liquid dosing pumps (compatible with concentrate solutions/suspensions), equipped with online calibration function. Calibrate at least once per month to ensure measurement accuracy.
3. All measurement equipment must be used within their calibration validity period. Perform zero calibration before use. Clear residual powder and impurities from equipment surfaces to avoid affecting measurement results.

(II) Concentrate Preparation Formulation

Direct addition of pure powder easily causes agglomeration; priority is given to formulating as a preparation. For

sample testing, powder can be added directly (requires pre-mixing). Specific specifications are as follows:

1. **Preparation Concentration:** Recommended preparation of 5%-10% (w/w) aqueous solution or 5%-8% ethanol aqueous solution (ethanol concentration 50%-70%), compatible with most application systems. Excessive concentration leads to agglomeration; too low increases addition volume.
2. **Dissolution Medium:** Use purified water (complying with GB 5749-2022) for food/dietary supplements. For acidic systems, base liquid can be used directly for dissolution. Use deionized water for cosmetics; ethanol aqueous solutions can be prepared for alcohol-containing systems.
3. **Preparation Operation:** Heat dissolution medium to 40-50°C (to accelerate dissolution, avoid high-temperature oxidation). Slowly add concentrate powder proportionally with stirring at 300-500 rpm for 10-15 minutes until no visible particles remain, forming a uniform suspension/solution.
4. **Preparation Storage:** After preparation, store sealed in stainless steel tanks. Shelf life at room temperature is 72 hours; can be extended to 7 days at low temperature (5-10°C). Stir again before use to avoid settling or precipitation.

(III) Pre-Addition Preparation

1. **Raw Material Verification:** Confirm concentrate powder batch number, active component content, production date. Check powder for no moisture absorption, caking, or mold; check preparation for no cloudiness, separation, or precipitation.
2. **Equipment Cleaning:** Clean measurement scales, dissolution tanks, transfer pipelines, homogenizers, etc., with purified water 3 times to be residue-free, grease-free, and impurity-free to avoid cross-contamination.
3. **System Confirmation:** Before addition, stir product base material to a homogeneous state. Liquid base materials require removal of sediment; solid/semi-solid base materials require crushing to uniform particle size to ensure concentrate powder dispersion effect.

VII. Addition Operation Methods for Different Application Scenarios

(I) Dietary Supplement Sector

Applicability Characteristics: Covers dosage forms like capsules, tablets, solid beverages, oral liquids, etc. Requires high component uniformity and activity retention. Some require high-temperature granulation or low-temperature storage, compatible with concentrate stability characteristics.

Addition Timing & Method:

1. **Oral Capsules/Tablets:** Powder sieved through 80 mesh, then mixed with excipients like microcrystalline cellulose, lactose via geometric dilution (rotation speed 15-20 rpm, time 15-20 min). Control environment humidity ≤60% for capsule filling; tablet compression pressure 3-5 MPa.
2. **Solid Beverages/Meal Replacement Powders:** Pre-mix concentrate powder with maltodextrin, then dry-mix with main materials (sieved through 60 mesh). For instant types, co-granulation is needed: granulation inlet air 160-180°C, outlet air 80-90°C.

3. **Oral Liquid Preparations:** Add 5%-8% concentrate aqueous solution before final volume adjustment of base material. Disperse with stirring at 40-50°C (300-500 rpm). Adjust pH to 4.0-6.0. Sterilize after filtration through a 0.45 μm filter membrane.

   Reference Addition Level: Oral capsules/tablets: 100-250 mg/person/day; Solid beverages/meal replacement powders: 0.5%-1.0% (of finished product mass); Oral liquid preparations: 0.3%-0.8% (of finished product mass).

(II) Functional Food Sector

Applicability Characteristics: Covers categories like meat, bakery, fruit/vegetable juice, condiments, etc., involving high-temperature processing, high-salt/acidic systems. Concentrate can inhibit lipid oxidation and browning, compatible with pH 3.0-7.5 and processes ≤180°C.

Addition Timing & Method:

1. **Meat & Aquatic Products:** Add concentrate solution to marinade (0-4°C), soak for 4-6 hours. For meat mince/fish surimi, add powder directly, stir at 200-300 rpm for 5-8 minutes. Process and store at low temperature promptly after addition.
2. **Bakery Products:** Pre-mix powder with flour and fat before dough mixing. Baking temperature ≤180°C. For temperatures above 180°C, pre-emulsify with oil/fat. Control fermentation temperature 28-32°C, humidity 70%-75%.
3. **Fruit/Vegetable Juice & Herbal Tea:** Add after juice filtration (≤30°C), adjust pH 3.0-4.0, centrifuge (8000-10000 rpm) or ultrafilter. For herbal tea, add after cooling below 60°C.
4. **Condiments:** For solid forms: dry-mix stepwise with raw materials (sieved through 40 mesh). For liquid forms: pre-dissolve in warm water, then add during final formulation stage. Control salinity ≤15%, pH 4.5-7.0. Let stand for 24 hours to remove sediment.

   Reference Addition Level: Meat/aquatic products: ≤150 mg/kg (based on product fat content); Bakery products: ≤200 mg/kg (based on product fat content); Fruit/vegetable juice/herbal tea: 0.2%-0.5% (of finished product mass); Condiments: ≤50 mg/kg (based on product fat content).

(III) Cosmetics Sector

Applicability Characteristics: Covers anti-aging, sun protection, basic care formulations. Must be compatible with skin physiological pH 5.0-6.5, avoiding irritation. Concentrate provides antioxidant and soothing effects, synergizing with sunscreens.

Addition Timing & Method:

1. **Anti-Aging Creams/Serums:** Add concentrate suspension when emulsion system cools to 45-50°C. Homogenize at 8000-10000 rpm for 3-5 minutes. Adjust pH to 5.5-6.5. Store protected from light.
2. **Auxiliary Sun Protection Products:** Disperse separately from sunscreens in water/oil phases before emulsification (60-70°C). Cool below 30°C before adding heat-sensitive components. Perform skin irritation and photostability tests after addition.

3. **Basic Care Toners/Lotions:** For toners: dissolve directly (stir 500-800 rpm for 15 min), filter through 0.22 μm membrane. For lotions: add during aqueous phase preparation (40-50°C), homogenize for 2-3 minutes after emulsification.

   Reference Addition Level: Anti-aging creams/serums: 1.0%-2.0% (of finished product mass); Auxiliary sun protection products: 0.8%-1.5% (of finished product mass); Basic care toners/lotions: 0.5%-1.0% (of finished product mass).

(IV) Other Fields

Foods for Special Medical Purposes (FSMP)

Addition Timing & Method: For powders: use geometric dilution dry-mixing. For liquid preparations: dissolve and disperse at low temperature (40-50°C). Control osmotic pressure 280-320 mOsm/kg, pH 4.5-7.0.

Reference Addition Level: Adjust according to formula. Daily intake ≤1 mg/kg body weight (expressed as carnosic acid).

Pet Food & Natural Preservative Compounding

Addition Timing & Method: For solid pet food: dry-mix before extrusion expansion (≤180°C). For wet food: dissolve before adding to meat mince. When compounding with ε-polylysine, natamycin: mix after dissolution.

Reference Addition Level: Adjust according to pet breed/preservation target. Determine optimal dosage through testing.

VIII. Specialized Operation for Compounding Antioxidant Components

1. **Compounding Ratio:** Compound with Vitamin C and Vitamin E at 1:2:1 (concentrate powder : VC : VE); with Coenzyme Q10 at 3:1 to synergistically improve antioxidant efficiency. Compound with citrus essential oil or mint extract at 10:1 to mask herbal off-notes.
2. **Addition Sequence:** First dissolve/disperse main antioxidant components like VC, VE into the system and mix uniformly, then add the concentrate preparation. Avoid main components adsorbing concentrate powder leading to uneven dispersion.
3. **Adaptation Adjustment:** In acidic systems (pH 3.0-4.0), appropriately increase the concentrate powder proportion (without exceeding limits) to offset slight activity inhibition by acidity. Perform compatibility tests in advance for high-salt systems.

IX. Key Quality Control Points for Addition Operations

1. **Measurement Review:** Implement a dual-check system: "Operator initial measurement, Quality Inspector review." Record addition amount and equipment number to ensure deviation ≤±2%.
2. **Dispersion Check:** Randomly sample different parts of the system after addition. Liquid: no particle separation/stratification; Solid: no localized caking. Content uniformity RSD ≤3.0%.
3. **Activity Testing:** Test total polyphenols and carnosol content in each finished product batch using HPLC

method to ensure active ingredient retention rate ≥90% (≥85% for thermally processed products).

4. **Stability Check:** Observe finished product retention samples. Food: ambient storage for 3 months; Cosmetics: light-protected storage for 6 months. Confirm no oxidative discoloration or efficacy attenuation.
5. **Compliance & Traceability:** Retain third-party test reports for raw materials and addition records to ensure compliance with standards for the corresponding field. Record retention period ≥ product shelf life + 6 months.

X. Safety Operations & Equipment Maintenance Requirements

(I) Operator Safety Procedures

1. Wear dust masks and disposable gloves when handling powder. Maintain ventilation in the operation area to avoid powder inhalation.
2. Avoid contact of powder and preparations with eyes. In case of accidental contact, rinse immediately with copious amounts of purified water and seek medical attention if necessary.
3. Operators must be trained and certified, familiar with equipment operation and emergency handling methods, and aware of incompatibilities between concentrate powder and other ingredients.

(II) Equipment Maintenance & Cleaning

1. **Measurement Equipment:** Wipe with a dry cloth after daily use. Calibrate precision monthly. Send for external verification annually. Avoid residual powder moisture absorption and caking.
2. **Dissolution Tanks, Transfer Pipelines:** Clean with purified water 3 times after each production batch. Drain pipeline liquid accumulation. Disinfect periodically to prevent cross-contamination.
3. **Homogenizers, Powder Dosing Equipment:** Regularly inspect homogenizer heads and feed screws. Keep dry and clean to prevent powder residue causing equipment failure.

(III) Raw Material Storage & Retrieval

1. Store powder sealed in a cool, dry place (≤25°C, relative humidity <65%), protected from light and high temperature. Use within 72 hours after opening; reseal and label remaining portion.
2. Prepare concentrate preparations for immediate use. Store unused portions sealed at low temperature (5-10°C), use within 7 days. Stir thoroughly before use.
3. Store raw materials, semi-finished products, and finished products in separate areas with clear labeling to avoid mixing and contamination. Keep raw materials away from strong oxidizing agents, strong acids, and strong alkalis.

XI. Handling of Abnormal Situations

1. **Measurement Error:** If addition amount is excessive, dilute with base liquid (liquid systems) or add supplemental excipients and mix (solid systems). If addition amount is insufficient, supplement with concentrate preparation according to the deficit value. Continue production only after small-sample testing

passes.

2. **Uneven Dispersion:** For liquid systems, increase stirring rate (500-800 rpm) for 15-20 minutes. For solid systems, re-crush, sieve, and mix. Add a small amount of solubilizer if necessary.
3. **Raw Material Quality Issue:** If powder moisture absorption/caking or preparation cloudiness/sedimentation is found, immediately stop use and replace with qualified raw material. Isolate materials already added. Continue use only after testing confirms compliance.
4. **Equipment Failure:** In case of dosing or homogenizer equipment failure, stop production immediately for repair. Use backup equipment. Store materials from the failure period separately; reprocess only after testing confirms no quality issues.
5. **Component Oxidation:** If system discoloration or off-odor occurs, investigate causes such as high temperature or strong light. Immediately implement light protection and cooling measures. Add a small amount of antioxidant as remediation. Decide whether to continue production after efficacy assessment.