

Takehara Kagaku Kogyo Co., Ltd.

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CERTIFICATE OF ANALYSIS

Product: Calcium Carbonate Heavy USP/FCC powder
"U-CAL SSS"

Date: July, 16, 2024

Weight: 12,000 kgs (25 kgs × 480 bags)

Lot No.: 42606

<u>Analysis</u>	<u>Specification</u>	<u>Test Results</u>
Assay (CaCO ₃)	98.0 to 100.5 %	99.1%
Identification	Pass/Fail	Passed test
Loss on drying (200°Cx4hrs)	NMT 2.0 %	0.4%
Acid-insoluble substances	NMT 0.2 %	0.03%
Limit of fluoride	NMT 0.005 %	LT 0.005 %
Arsenic	NMT 3 ppm	LT 3.0 ppm
Barium	Pass/Fail	Passed test
Lead	NMT 3.0 ppm	0.27 ppm
Iron	NMT 0.1 %	LT 0.1%
Magnesium and alkali salts	NMT 1.0 %	0.5%
Mercury	NMT 0.5 ppm	0.02 ppm
pH	8.5 to 9.5	9
Whiteness	-	95.4%
Residual solvents	Based on our knowledge of the manufacturing process and controlled handling of Calcium Carbonate Heavy USP/FCC powder, there is no potential for any solvents to be present and that the material, if tested by USP Chemical Tests (467), will meet the requirements.	

Butuh penyesua
terlampir

Date of Manufacturing: June, 26, 2024

Expiry Date: June, 25, 2027

TAKEHARA KAGAKU KOGYO CO., LTD.
Technical Department


E. Nagaki manager

STANDARISASI CERTIFICATE OF ANALYSIS BAHAN BAKU PRODUK

No	Nama Bahan	Manufaktur	Referensi pada CoA Manufaktur saat ini	Referensi pada CoA yang diusulkan	Penyesuaian Parameter				Nomor SPBB	Keterangan
					Parameter yang Dihilangkan	Parameter yang Ditambah	Parameter yang direvisi			
1	Calcium Carbonate	Takehara Kagaku Kogyo	USP	USP47/ FI VI	pH	Pemerian menyesuaikan USP 47	Identifikasi menyesuaikan USP 47	3VA00 / SPSB8 / 026.00		Penyesuaian CoA dari Manufacturer
					Whiteness	Kelarutan menyesuaikan USP 47				
					Residual Solvent	Logam berat : Tidak lebih dari 20 bpg (FI VI)	Barium menyesuaikan USP 47			

Diperiksa Oleh


15/10/24
Koordinator QC


15/10/24
Koordinator TS


14/10/24
Koordinator QA

Disetujui Oleh,


15/10/24
Manager QA

Calcium Carbonate

CaCO₃ 100.09
Carbonic acid, calcium salt (1:1);
Calcium carbonate (1:1) CAS RN®: 471-34-1.

DEFINITION

Calcium Carbonate, dried at 200° for 4 h, contains calcium equivalent to NLT 98.0% and NMT 100.5% of calcium carbonate (CaCO₃).

IDENTIFICATION

- **A. IDENTIFICATION TESTS—GENERAL, Calcium (191):** The addition of acetic acid to it produces effervescence (presence of carbonate), and the resulting solution, after boiling, meets the requirements of the tests.

ASSAY

• TITRIMETRY (541)

Sample: 200 mg of Calcium Carbonate, previously dried at 200° for 4 h

Blank: 100 mL of water and 15 mL of 1 N sodium hydroxide

Titrimetric system

(See *Titrimetry* (541).)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Indicator: 300 mg of hydroxy naphthol blue

Endpoint detection: Visual, change to distinct blue

Analysis: Transfer the *Sample* to a 250-mL beaker. Moisten thoroughly with a few mL of water, and add, dropwise, sufficient 3 N hydrochloric acid to dissolve. Add 100 mL of water, 15 mL of 1 N sodium hydroxide, and 300 mg of hydroxy naphthol blue. Titrate with the *Titrant*. Calculate the percentage of calcium carbonate (CaCO₃) in the *Sample* taken:

$$\text{Result} = [(V - B) \times M \times F \times 100] / W$$

V = *Sample* titrant volume (mL)
B = *Blank* titrant volume (mL)
M = titrant molarity (mmol/mL)
F = equivalency factor, 100.09 mg/mmol
W = weight of the *Sample* (mg)

Acceptance criteria: 98.0%–100.5% on the dried basis

IMPURITIES

• ACID-INSOLUBLE SUBSTANCES

Sample: 5.0 g

Analysis: Mix the *Sample* with 10 mL of water, and add hydrochloric acid, dropwise, with agitation, until it ceases to cause effervescence, then add water to make the mixture measure 200 mL, and filter. Wash the insoluble residue with water until the last washing shows no chloride, and ignite and weigh the residue.

Acceptance criteria: NMT 0.2%; the weight of the residue does not exceed 10 mg.

Change to read:

- **ARSENIC (211), Procedures, Procedure 1** (CN 1-Jun-2023)

Sample solution: Slowly dissolve 1.0 g in 15 mL of hydrochloric acid, and dilute with water to 55 mL.

Analysis: Omit the addition of 20 mL of 7 N sulfuric acid specified in *Arсенic* (211), *Procedures, Procedure 1* (CN 1-Jun-2023).

Acceptance criteria: NMT 3 ppm

- **BARIUM:** A platinum wire, dipped in the filtrate obtained in the test for *Acid-Insoluble Substances* and held in a nonluminous flame, does not impart a green color.

Change to read:

- **IRON (241), Procedures, Procedure 1** (CN 1-Jun-2023)

Sample solution: 40 mg in 5 mL of 2 N hydrochloric acid. Transfer to a beaker with the aid of water, and dilute with water to 10 mL.

Standard solution: Transfer 4.0 mL of the *Standard Iron Solution*, prepared as directed in *Iron* (241), *Procedures, Procedure 1* (CN 1-Jun-2023), to a beaker, and dilute with water to 10 mL.

Instrumental conditions

(See *Ultraviolet-Visible Spectroscopy* (857).)

Analytical wavelength: 530 nm

Blank: Water

Analysis: Separately to the *Sample solution* and *Standard solution* add 2 mL of citric acid solution (1 in 5) and 2 drops of thioglycolic acid, adjust with ammonia TS to a pH of 9.5 ± 0.1, dilute with water to 20 mL, and allow to stand for 5 min. Dilute with water to 50 mL. Concomitantly determine the absorbances of the solutions from the *Sample solution* and the *Standard solution*.

Acceptance criteria: NMT 0.1%; the absorbance of the solution from the *Sample solution* does not exceed that of the *Standard solution*.

Change to read:

- **LEAD (251), Procedures, Procedure 1** (CN 1-Jun-2023)

Sample solution: 1.0 g in 5 mL of water

Analysis: To the *Sample solution* slowly add 8 mL of 3 N hydrochloric acid, evaporate on a steam bath to dryness, and dissolve the residue in 5 mL of water.

Acceptance criteria: NMT 3 ppm

• LIMIT OF FLUORIDE

[NOTE—Prepare and store all solutions in plastic containers.]

Solution A: 294 mg/mL of sodium citrate dihydrate in water
Sample: 2.0 g

Standard stock solution: 1.11 mg/mL of USP Sodium Fluoride RS in water

Standard solution: Combine 20.0 mL of the *Standard stock solution* with 50.0 mL of *Solution A*, and dilute with water to 100.0 mL. [NOTE—Each mL of this solution contains 100 µg of fluoride ion]

Electrode system: Use a fluoride-specific ion-indicating electrode and a silver–silver chloride reference electrode connected to a pH meter capable of measuring potentials with a minimum reproducibility of ±0.2 mV (see *pH* (791)).

Standard response line: Transfer 50.0 mL of *Solution A* and 4.0 mL of hydrochloric acid to a beaker, and add water to make 100 mL. Add a plastic-coated stirring bar, insert the electrodes into the solution, stir for 15 min, and read the potential, in mV. Continue stirring, and at 5-min intervals add 100, 100, 300, and 500 µL of the *Standard solution*, reading the potential 5 min after each addition. Plot the logarithms of the cumulative fluoride ion concentrations (0.1, 0.2, 0.5, and 1.0 µg/mL) versus potential, in mV.

Analysis: Transfer the *Sample* to a beaker containing a plastic-coated stirring bar, add 20 mL of water and 4.0 mL of hydrochloric acid, and stir until dissolved. Add 50.0 mL of *Solution A* and sufficient water to make 100 mL of test solution. Rinse and dry the electrodes, insert them into the *Sample solution*, stir for 5 min, and read the potential, in mV. From the measured potential and the *Standard response line*, determine the concentration, *C*, in µg/mL, of fluoride ion in the *Sample solution*. Calculate the content of fluoride in the specimen taken:

$$\text{Result} = (V \times C) / W$$

V = volume of the *Sample solution* (mL)

C = concentration of fluoride in the *Sample solution* ($\mu\text{g/mL}$)
 W = weight of *Sample* (g)

Acceptance criteria: NMT 50 ppm

• **LIMIT OF MAGNESIUM AND ALKALI SALTS**

Sample: 1.0 g

Analysis: Mix the *Sample* with 35 mL of water. Carefully add 3 mL of hydrochloric acid, heat the solution, and boil for 1 min. Rapidly add 40 mL of oxalic acid TS, and stir vigorously until precipitation is well-established. Add immediately to the warm mixture 2 drops of methyl red TS and then 6 N ammonium hydroxide, dropwise, until the mixture is just alkaline. Cool to room temperature, transfer to a 100-mL graduated cylinder, dilute with water to 100 mL, mix, and allow to stand for 4 h or overnight. Filter, and to 50 mL of the clear filtrate in a platinum dish add 0.5 mL of sulfuric acid, and evaporate the mixture on a steam bath to a small volume. Carefully heat over a free flame to dryness, and continue heating to complete decomposition and volatilization of ammonium salts. Finally, ignite the residue to constant weight.

Acceptance criteria: NMT 1.0%; the weight of the residue is NMT 5 mg.

Change to read:

- **MERCURY** <261>, *Procedures, Procedure 2*▲ (CN 1-Jun-2023)
Mercury stock solution and Standard mercury solution:
Proceed as directed in *Mercury* <261>.

Standard solution: Proceed as directed in *Mercury* <261>, except use 3 mL of hydrochloric acid instead of 3 mL of sulfuric acid.

Sample stock solution: 4.0 g in a 100-mL beaker, and cautiously dissolve in 14 mL of 6 N hydrochloric acid

Sample solution: Proceed as directed in *Mercury* <261> using the *Sample stock solution*, except use 3 mL of hydrochloric acid instead of 3 mL of sulfuric acid.

Analysis

Samples: *Standard solution* and *Sample solution*
Proceed as directed in *Mercury* <261>.

Acceptance criteria: NMT 0.5 ppm

SPECIFIC TESTS

- **LOSS ON DRYING** <731>: Dry a sample at 200° for 4 h: it loses NMT 2.0% of its weight.

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **USP REFERENCE STANDARDS** <11>
USP Sodium Fluoride RS

Butabarbital Sodium: White powder, having a bitter taste. Freely soluble in water and in alcohol; practically insoluble in absolute ether.

Butalbital: White, crystalline, odorless powder, having a slightly bitter taste. Is stable in air. Its saturated solution is acid to litmus. Freely soluble in alcohol, in ether, and in chloroform; soluble in boiling water, and in solutions of fixed alkalies and alkali carbonates; slightly soluble in cold water.

Butamben: White, crystalline powder. Is odorless and tasteless. Soluble in dilute acids, in alcohol, in chloroform, in ether, and in fixed oils; very slightly soluble in water. Is slowly hydrolyzed when boiled with water.

Butane: Colorless, flammable gas (boiling temperature is about -0.5°). One volume of water dissolves 0.15 volume, and 1 volume of alcohol dissolves 18 volumes at 17° and 770 mm; 1 volume of ether or chloroform at 17° dissolves 25 or 30 volumes, respectively. Vapor pressure at 21° is about 1620 mm of mercury (17 psig). *NF category:* Propellant.

Butoconazole Nitrate: White to off-white, crystalline powder. Melts at about 160° . Sparingly soluble in methanol; slightly soluble in acetonitrile, in acetone, in dichloromethane, and in tetrahydrofuran; very slightly soluble in ethyl acetate; practically insoluble in water.

Butorphanol Tartrate: White powder. Its solutions are slightly acidic. Melts between 217° and 219° , with decomposition. Soluble in dilute acids; sparingly soluble in water; slightly soluble in methanol; insoluble in alcohol, in chloroform, in ethyl acetate, in ethyl ether, and in hexane.

Butyl Alcohol: Clear, colorless, mobile liquid, having a characteristic, penetrating vinous odor. Soluble in water. Miscible with alcohol, with ether, and with many other organic solvents. *NF category:* Solvent.

Butylene Glycol: Clear, colorless, hygroscopic, viscous liquid. It is miscible with water, with acetone, and with ether in all proportions; immiscible with fixed oils. It dissolves most essential oils and synthetic flavoring substances. *NF category:* Solvent; wetting and/or solubilizing agent.

Butyl Palmitostearate: Colorless or pale yellowish waxy solid at temperatures below 17° – 24° or colorless or pale yellowish liquid at temperatures of 17° – 24° or above. Soluble in acetone, in alcohol, in ether, in mineral oils, and in vegetable oils; practically insoluble in water and in propylene glycol. *NF category:* Emulsifying and/or solubilizing agent; flavors and fragrance; plasticizer.

Butyl Stearate: Colorless or pale yellowish waxy solid at temperatures below 19° – 24° or colorless or pale yellowish liquid at temperatures of 19° – 24° or above. Soluble in acetone, in alcohol, in ether, in mineral oils, and in vegetable oils; practically insoluble in water and in propylene glycol. *NF category:* Emulsifying and/or solubilizing agent; flavors and fragrance; plasticizer.

Butylated Hydroxyanisole: White or slightly yellow, waxy solid, having a faint, characteristic odor. Freely soluble in alcohol, in propylene glycol, in chloroform, and in ether; insoluble in water. *NF category:* Antioxidant.

Butylated Hydroxytoluene: White, crystalline solid, having a faint, characteristic odor. Freely soluble in alcohol, in chloroform, and in ether; insoluble in water and in propylene glycol. *NF category:* Antioxidant.

Butylparaben: Small, colorless crystals or white powder. Freely soluble in acetone, in alcohol, in ether, and in propylene glycol; very slightly soluble in water and in glycerin. *NF category:* Antimicrobial preservative.

Cabergoline: White or almost white, crystalline or amorphous powder. Freely soluble in alcohol (96%); slightly soluble in 0.1 M hydrochloric acid; very slightly soluble in hexane; practically insoluble in water.

Caffeine: White powder or white, glistening needles, usually matted together. Is odorless and has a bitter taste. Its

solutions are neutral to litmus. The hydrate is efflorescent in air. Freely soluble in chloroform; sparingly soluble in water and in alcohol; slightly soluble in ether.

Calamine: Pink, odorless, practically tasteless, fine powder. Soluble in mineral acids; insoluble in water.

Calcipotriene: White or almost white crystalline powder. Freely soluble in alcohol; slightly soluble in methylene chloride; practically insoluble in water.

Calcitriol: White or almost white crystals. Freely soluble in alcohol; soluble in ether and in fatty oils; practically insoluble in water. It is sensitive to air, heat, and light.

Calcium Acetate: White, odorless or almost odorless, hygroscopic, crystalline powder. When heated to above 160° , it decomposes to calcium carbonate and acetone. Freely soluble in water; slightly soluble in methanol; practically insoluble in acetone, in dehydrated alcohol, and in benzene. *NF category:* Antimicrobial preservative; sequestering agent.

Calcium Ascorbate: White to slightly yellow, practically odorless powder. Freely soluble in water (approximately 50 g per 100 mL); slightly soluble in alcohol; insoluble in ether.

Calcium Carbonate: Fine, white, odorless, tasteless, microcrystalline powder. Is stable in air. Practically insoluble in water. Its solubility in water is increased by the presence of any ammonium salt or of carbon dioxide. The presence of any alkali hydroxide reduces its solubility. Insoluble in alcohol. Dissolves with effervescence in 1 N acetic acid, in 3 N hydrochloric acid, and in 2 N nitric acid. *NF category:* Diluent; pH modifier (acidifying agent/alkalizing agent/buffering agent); coating agent; wet binder.

Calcium Chloride: White, hard, odorless fragments or granules. Is deliquescent. Very soluble in boiling water; freely soluble in water, in alcohol, and in boiling alcohol. *NF category:* Desiccant; antimicrobial preservative.

Calcium Citrate: White, odorless, crystalline powder. Freely soluble in diluted 3 N hydrochloric acid and in diluted 2 N nitric acid; slightly soluble in water; insoluble in alcohol.

Calcium Gluceptate: White to faintly yellow, amorphous powder. Is stable in air, but the hydrous forms may lose part of their water of hydration on standing. Freely soluble in water; insoluble in alcohol and in many other organic solvents.

Calcium Gluconate: White, crystalline, odorless, tasteless granules or powder. Is stable in air. Its solutions are neutral to litmus. Freely soluble in boiling water; sparingly (and slowly) soluble in water; insoluble in alcohol.

Calcium Hydroxide: White powder. Has an alkaline, slightly bitter taste. Soluble in glycerin and in syrup; slightly soluble in water; very slightly soluble in boiling water; insoluble in alcohol. *NF category:* pH modifier (acidifying agent/alkalizing agent/buffering agent).

Calcium Hydroxide Solution: Clear, colorless liquid having an alkaline taste. Is alkaline to litmus.

Calcium Lactate: White, practically odorless granules or powder. The pentahydrate is somewhat efflorescent and at 120° becomes anhydrous. The pentahydrate is soluble in water; it is practically insoluble in alcohol. *NF category:* pH modifier (acidifying agent/alkalizing agent/buffering agent); antimicrobial preservative; wet binder.

Calcium Levulinate: White, crystalline or amorphous, powder, having a faint odor suggestive of burnt sugar. Has a bitter, salty taste. Freely soluble in water; slightly soluble in alcohol; insoluble in ether and in chloroform.

Calcium Pantothenate: Slightly hygroscopic, white powder. Is odorless and has a bitter taste. Freely soluble in water; soluble in glycerin; practically insoluble in alcohol, in chloroform, and in ether.

Racemic Calcium Pantothenate: White, slightly hygroscopic powder, having a faint, characteristic odor, and a bitter taste. Is stable in air. Its solutions are neutral or alkaline to litmus. Is optically inactive. Freely soluble in water; soluble

hidroksinaftol P. Titrasi dengan *dinatrium edetat* 0,05 M LV sampai titik akhir berwarna biru.

Tiap mL *dinatrium edetat* 0,05 M setara dengan 3,705 Ca (OH)₂

Wadah dan penyimpanan Dalam wadah tertutup rapat.

LARUTAN TOPIKAL KALSIMUM HIDROKSIDA

Calcium Hydroxide Topical Solution

Larutan Topikal Kalsium Hidroksida adalah larutan yang mengandung tidak kurang dari 140 mg Ca(OH)₂ per 100 mL. Larutan topikal kalsium hidroksida dibuat dengan cara menambahkan 3 g *kalsium hidroksida P* pada 1000 mL air dingin, kocok kuat dan berulang kali selama 1 jam. Biarkan kelebihan kalsium hidroksida mengendap. Gunakan beningan.

[Catatan Kelarutan kalsium hidroksida bervariasi tergantung suhu larutan disimpan, lebih kurang 170 mg per 100 mL pada suhu 15° dan berkurang pada suhu yang lebih tinggi. Kadar ditetapkan pada suhu 25°.]

Bagian yang tidak larut tidak dapat digunakan lagi untuk membuat larutan topikal kalsium hidroksida.

Identifikasi

A. Menyerap karbon dioksida dari udara: terbentuk lapisan kalsium karbonat pada permukaan cairan.

B. Jika dipanaskan menjadi keruh, karena terjadi pemisahan kalsium hidroksida.

C. Menunjukkan reaksi *Kalsium* cara A dan B seperti tertera pada *Uji Identifikasi Umum* <291>.

Alkali dan garam karbonat Jenuhkan sejumlah larutan dengan karbon dioksida dan dididihkan: larutan bereaksi netral.

Penetapan kadar Pipet 100 mL larutan ke dalam labu Erlenmeyer yang sesuai, tambahkan 50 mL air, 15 mL *natrium hidroksida 1 N* dan 300 mg *biru hidroksi naftol P* dan titrasi dengan *dinatrium edetat* 0,05 M LV hingga titik akhir berwarna biru.

Tiap mL *dinatrium edetat* 0,05 M setara dengan 3,705 Ca(OH)₂

Wadah dan penyimpanan Dalam wadah tertutup rapat, terisi penuh pada suhu tidak lebih dari 25°.

KALSIMUM KARBONAT

Calcium Carbonate

Kalsium karbonat (1:1) [471-34-1]
CaCO₃

BM 100,09

Kalsium Karbonat yang telah dikeringkan pada suhu 200° selama 4 jam mengandung kalsium setara tidak kurang dari 98,0% dan tidak lebih dari 100,5% CaCO₃.

Pemerian Serbuk halus mikro hablur, putih; tidak berbau; tidak berasa; stabil di udara.

Kelarutan Praktis tidak larut dalam air; kelarutan dalam air meningkat dengan adanya sedikit garam amonium atau karbon dioksida; adanya alkali hidroksida menurunkan kelarutan; tidak larut dalam etanol; larut dalam *asam asetat 1 N*, *asam hidroklorida 3 N* dan *asam nitrat 2 N* dengan membentuk gelembung gas.

Identifikasi Pada sejumlah zat tambahkan *asam asetat P* terbentuk gelembung gas, setelah dididihkan larutan menunjukkan reaksi *Kalsium* cara A, B seperti tertera pada *Uji Identifikasi Umum* <291>.

Susut pengeringan <1121> Tidak lebih dari 2,0%; lakukan pengeringan pada suhu 200° selama 4 jam.

Senyawa tidak larut asam Tidak lebih dari 0,2%; lakukan penetapan sebagai berikut: Campur 5,0 g zat dengan 10 mL air, tambahkan *asam hidroklorida P* tetes demi tetes sambil dikocok, sampai tidak terbentuk gelembung gas, kemudian tambahkan air sampai 200 mL dan saring. Cuci residu dengan air hingga air cucian terakhir tidak mengandung klorida dan pijarkan. Bobot residu tidak lebih dari 10 mg.

Fluorida Tidak lebih dari 0,005% [Catatan Siapkan dan simpan semua larutan dalam wadah plastik.]

Larutan dapar, *Larutan baku* dan *Sistem elektroda* Lakukan seperti tertera pada uji *Fluorida* dalam *Kalsium Fosfat Dibasa Anhidrat*.

Garis respons baku Lakukan seperti tertera pada uji *Fluorida* dalam *Kalsium Fosfat Dibasa Anhidrat*, gunakan 4,0 mL *asam hidroklorida P* sebagai pengganti 2,0 mL *asam hidroklorida P*.

Prosedur Lakukan seperti tertera pada uji *Fluorida* dalam *Kalsium Fosfat Dibasa Anhidrat*, gunakan 4,0 mL *asam hidroklorida P*.

Arsen <321> *Metode I* Tidak lebih dari 3 bpj; lakukan penetapan menggunakan *Larutan uji* yang dibuat sebagai berikut: Larutkan sedikit demi sedikit 1,0 g zat dalam 15 mL *asam hidroklorida P* dan encerkan dengan air hingga 55 mL. Larutan memenuhi *Uji Batas Arsen*, tanpa penambahan 20 mL *asam sulfat 7 N*, seperti tertera pada *Prosedur*.

Barium Celupkan kawat platina ke dalam filtrat yang diperoleh dari uji *Senyawa tidak larut asam*, bakar: nyala tidak berwarna hijau.

Timbal <401> Tidak lebih dari 3 bpj; lakukan penetapan menggunakan larutan uji yang dibuat sebagai berikut: Campur 1,0 g zat dengan 5 mL air, tambahkan sedikit demi sedikit 8 mL *asam hidroklorida 3 N*, uapkan di atas tangas uap hingga kering, larutkan residu dalam 5 mL air.

Besi Tidak lebih dari 0,1%; lakukan penetapan sebagai berikut: Buat *Larutan uji* dengan melarutkan 40 mg zat dalam 5 mL *asam hidroklorida 2 N*, masukkan ke dalam gelas piala dan encerkan dengan air hingga 10 mL. Buat *Larutan baku* menggunakan 4,0 mL *Larutan baku besi* seperti tertera pada *Uji batas besi* <331> dalam gelas piala dan encerkan dengan air hingga 10 mL. Pada masing-masing gelas piala tambahkan 2 mL larutan *asam sitrat P* (1 dalam 5) dan 2 tetes *asam tioglikolat P*, atur pH hingga $9,5 \pm 0,1$ dengan penambahan *amonium hidrokksida 6 N*, encerkan dengan air hingga 20 mL, campur, diamkan selama 5 menit. Encerkan dengan air hingga 50 mL. Ukur serapan *Larutan uji* dan *Larutan baku* pada panjang gelombang serapan maksimum lebih kurang 530 nm menggunakan air sebagai blangko: serapan *Larutan uji* tidak lebih dari *Larutan baku*.

Raksa <381> *Metode IIA* Tidak lebih dari 0,5 bpj; lakukan penetapan menggunakan larutan yang dibuat sebagai berikut: Pindahkan 4,0 g zat ke dalam gelas piala 100 mL, larutkan hati-hati dalam 14 mL *asam hidroklorida 6 N*. Gunakan 3 mL *asam hidroklorida 6 N* sebagai pengganti 3 mL *asam sulfat P* pada pembuatan *Larutan baku* dan *Larutan uji*.

Logam berat <371> Tidak lebih dari 20 bpj; lakukan penetapan menggunakan larutan yang dibuat sebagai berikut: Campur 1,0 g zat dalam 5 mL air, tambahkan sedikit demi sedikit 8 mL *asam hidroklorida 3 N* dan uapkan di atas tangas uap hingga kering. Larutkan residu dalam 20 mL air, saring dan tambahkan air pada filtrat hingga 25 mL.

Magnesium dan garam alkali Tidak lebih dari 1,0%; lakukan penetapan sebagai berikut: Campur 1,0 g zat dengan 35 mL air, tambahkan hati-hati 3 mL *asam hidroklorida P*, panaskan larutan dan dididihkan selama 1 menit. Segera tambahkan 40 mL *asam oksalat LP* dan aduk kuat sampai terbentuk endapan. Segera tambahkan pada campuran hangat 2 tetes larutan *merah metil LP* dan tetes demi tetes *amonium hidrokksida 6 N* sampai campuran tepat basa, dinginkan hingga suhu ruang. Masukkan ke dalam gelas ukur, encerkan dengan air hingga 100 mL dan diamkan selama 4 jam atau semalam. Saring, masukkan 50 mL filtrat ke dalam cawan platina, tambahkan 0,5 mL *asam sulfat P*, uapkan di atas tangas uap sampai hampir kering. Panaskan perlahan-lahan di atas api bebas sampai kering, lanjutkan pemanasan sampai garam-garam amonium terurai dan menguap. Pijarkan residu sampai bobot tetap. Bobot residu tidak lebih dari 5 mg.

Penetapan kadar Timbang saksama lebih kurang 200 mg zat kering pada suhu 200° selama 4 jam. Masukkan ke dalam gelas piala 250 mL, basahkan dengan beberapa mL air, tambahkan tetes demi tetes *asam hidroklorida 3 N* secukupnya hingga larut sempurna. Tambahkan 100 mL air, 15 mL *natrium hidrokksida 1 N* dan 300 mg *biru hidrokksinaftol P*. Titrasi dengan *dinatrium edetat 0,05 M LV* sampai titik akhir berwarna biru.

Tiap mL dinatrium edetat 0,05 M setara dengan 5,004 mg CaCO_3

Wadah dan penyimpanan Dalam wadah tertutup baik.

SUSPENSI ORAL KALSIMUM KARBONAT Calcium Carbonate Oral Suspension

Suspensi Oral Kalsium Karbonat mengandung kalsium karbonat, CaCO_3 , tidak kurang dari 90,0% dan tidak lebih dari 110,0%, dari jumlah yang tertera pada etiket.

Baku pembanding *Natrium Fluorida BPFI*; tidak boleh dikeringkan. Simpan dalam wadah tertutup rapat.

Identifikasi Pada sejumlah zat tambahkan *asam asetat P*, terbentuk gelembung gas, setelah dididihkan larutan menunjukkan reaksi *Kalsium* seperti tertera pada *Uji Identifikasi Umum* <291>.

pH <1071> Antara 7,5 dan 8,7.

Arsen <321> *Metode I* Tidak lebih dari 3 bpj dihitung terhadap kalsium karbonat yang tertera pada etiket; lakukan penetapan menggunakan *Larutan uji* yang dibuat sebagai berikut: larutkan sedikit demi sedikit suspensi oral setara dengan 1,0 g kalsium karbonat dalam 15 mL *asam hidroklorida P* dan encerkan dengan air hingga 55 mL. Larutan memenuhi *Uji Batas Arsen*, tanpa penambahan 20 mL *asam sulfat 7 N*, seperti tertera pada *Prosedur*.

Timbal <401> Tidak lebih dari 3 bpj dihitung terhadap kalsium karbonat yang tertera pada etiket; lakukan penetapan menggunakan *Larutan uji* yang dibuat sebagai berikut: campur sejumlah suspensi oral setara dengan 1,0 g kalsium karbonat dengan 5 mL air, tambahkan sedikit demi sedikit 8 mL *asam hidroklorida 3 N*, uapkan di atas tangas uap hingga kering, larutkan residu dalam 5 mL air.

Logam berat <371> Tidak lebih dari 20 bpj dihitung terhadap kalsium karbonat yang tertera pada etiket; lakukan penetapan menggunakan *Larutan uji* yang dibuat sebagai berikut: campur sejumlah suspensi oral setara dengan 1,0 g kalsium karbonat dengan 5 mL air, tambahkan sedikit demi sedikit 8 mL *asam*