

5 Manual of Test & Analysis Methods for Finished & Circulating Sulfolane Solvent

Scope of Application

Incoming raw material acceptance, in-plant circulating solvent central control, finished product factory release, recovered regenerated solvent inspection

Covers complete testing protocols for industrial grade, polymer/pharmaceutical grade, lithium battery electronic grade, including instruments, reagents, operating steps, calculation formulas, pass/fail limits and abnormal troubleshooting

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1 General Test Specifications

1.1 Sampling Standards

1. Liquid samples at temperature $\geq 30^{\circ}\text{C}$ without crystallization: stainless steel or glass sampling bottles, sealed under nitrogen blanketing. For winter solidified samples, heat to 35°C and fully homogenize before sampling.
2. On-line circulating solvent sampling: cool bypass stream to below 40°C to prevent thermal oxidative degradation during sampling.
3. High-purity polymer / electronic grade sampling: operate in glove box with dew point $\leq -30^{\circ}\text{C}$, use PTFE sampling tools with full air & moisture isolation.
4. Sample retention period: 7 days for industrial grade, 30 days for pharmaceutical & electronic grade; store sealed away from light at $\leq 30^{\circ}\text{C}$.

1.2 General Instrument Requirements

- Gas Chromatography (GC): FID detector, polar capillary column; GC-MS mandatory for electronic grade products
- Moisture testing: Coulometric Karl Fischer titrator (for trace water)
- Metal ion analysis: ICP-MS (electronic grade), Atomic Absorption Spectrophotometer (AAS, industrial & polymer grade)
- Acid value test: Potentiometric titrator
- Color measurement: APHA platinum-cobalt colorimeter

2 Standard Test Protocols for 12 Core Indexes

Method 1 Determination of Sulfolane Purity (GC Internal Standard Method, GB/T 23946)

Applicability

All grades of sulfolane, dry-base purity quantification

1. GC Instrument Conditions

Column: DB-Wax 30 m × 0.25 mm × 0.25 μm; oven program: hold at 100°C for 2 min, ramp 10°C/min to 220°C, hold 8 min; inlet temperature 240°C, FID detector 260°C; carrier gas: high-purity nitrogen

2. Reagent: Tetradecane (chromatographic grade, internal standard)

3. Operating Steps

1. Accurately weigh 0.1 g sample and 0.02 g tetradecane, dilute to 10 mL with methanol and homogenize.
2. Inject 1 μL sample solution for chromatogram acquisition; calculate purity via internal standard normalization correction.

1. Calculation Formula

$$w = \frac{A_{\text{sample}} \times f_{\text{internal}} \times m_{\text{internal}}}{A_{\text{internal}} \times f_{\text{sample}} \times m_{\text{sample}}} \times 100\%$$

Where:

A_{sample} = peak area of sulfolane; A_{internal} = peak area of internal standard

f = correction factor; m = mass

2. Pass Limits

Industrial ≥99.0 wt%; Polymer/Pharmaceutical ≥99.9 wt%; Electronic ≥99.99 wt%

Method 2 Water Content Determination

Scheme A Coulometric Karl Fischer Titration (Mandatory for Polymer & Electronic Grade, Trace Water)

1. Instrument: Coulometric Karl Fischer moisture titrator with non-methanol electrolyte without side reactions

2. Operation: Melt sample at 35°C, inject 0.1–0.5 g sample via syringe, read water content directly.

3. Limits: Polymer ≤50 ppm; Electronic ≤10 ppm

Scheme B Volumetric Karl Fischer Titration (Industrial Grade Crude Measurement)

Applicable to circulating solvent with water content 0.05%–0.3%

Limit for industrial circulating solvent: ≤3000 ppm (0.3 wt%)

Method 3 Total Acid Value (Potentiometric Titration, Indicator of Solvent Degradation)

1. Reagents: Anhydrous isopropanol, 0.01 mol/L KOH standard titrant in isopropanol

2. Steps: Weigh 10 g sample, dissolve in 50 mL anhydrous isopropanol, perform potentiometric titration until potential jump endpoint.

3. Calculation Formula

$$\text{Acid Value (mgKOH/g)} = \frac{c \times V \times 56.1}{m}$$

c = KOH concentration (mol/L); V = titrant volume (mL); m = sample mass (g)

4. Limits

Industrial ≤0.3 mgKOH/g; Polymer ≤0.1 mgKOH/g; Electronic ≤0.03 mgKOH/g

Acid value >0.5 mgKOH/g = severe solvent degradation requiring vacuum distillation regeneration

Method 4 Color (APHA Platinum-Cobalt Method)

1. Standard APHA color scale 0–500, 50 mm colorimetric tubes

2. Precondition sample at 35°C to eliminate crystallization before visual matching

Limits: Industrial ≤30 APHA; Polymer ≤10 APHA; Electronic ≤5 APHA

Method 5 2-Cyclobutene Sulfone Impurity (GC Trace Quantification)

2-cyclobutene sulfone is synthetic by-product prone to polymerization under high temperature; GC quantification detection limit 1 ppm

Limits: Industrial ≤500 ppm; Polymer ≤50 ppm; Electronic ≤1 ppb

Method 6 Metal Ion Content Analysis

6.1 Industrial & Polymer Grade: AAS Atomic Absorption (Na, K, Fe, Ca, Cu)

Sample pre-treatment: High-temperature dry ashing, dilute nitric acid constant volume for single-element testing

Limits: Single metal ≤10 ppm (industrial); Single metal ≤3 ppm (polymer)

6.2 Lithium Battery Electronic Grade: ICP-MS Multi-Element Simultaneous Analysis

Microwave digestion pre-treatment, constant volume with ultra-pure water,
ppt-level detection limit

Hard mandatory limit: Na, K, Fe, Ca ≤ 1 ppb each

Method 7 Free Sulfur & Sulfide Impurities

Spectrophotometry with lead acetate color development at 420 nm, external
standard quantification

Limits: Industrial ≤ 10 ppm; Polymer ≤ 2 ppm; Electronic ≤ 0.5 ppb

Method 8 Thermal Stability Test (SO₂ Release Test, Judge Thermal
Decomposition Tendency)

1. Seal 50 g sample in pressure vessel, hold constant at 220°C for 2 h,
absorb released SO₂
2. Titrate with standard iodine solution to calculate SO₂ release mass per
kg sample

Limits: Industrial ≤ 20 mg/kg; Electronic ≤ 1 mg/kg

Method 9 Density Measurement

Pycnometer or digital densimeter at constant 25°C; standard reference
density 1.261 g/cm³, used for feeding mass calibration

Method 10 Solidification Point Measurement

Low-temperature thermostatic bath gradient cooling, observe crystallization
temperature; standard reference value 27.4°C, used to judge purity and trace
water impurity interference

Method 11 Non-Volatile Residue

Weigh 10 g sample, vacuum bake at 220°C to constant mass, calculate
residue mass fraction

All grades limit: ≤ 0.01 wt%; high residue = excessive heavy polymer
degradation products

Method 12 Chloride Ion (Ion Chromatography IC)

Ultrasonic extraction of sample with pure water, IC quantification

Petrochemical plant raw material control threshold: Cl⁻ < 1 ppm to prevent
equipment corrosion and solvent catalytic degradation

3 Mandatory & Sampling Test Items by Product Grade

3.1 Industrial Grade (Aromatic Extraction / Gas Sweetening)

Mandatory per batch: GC purity, water content, acid value, APHA color, 2-
cyclobutene sulfone, density, solidification point

Sampling (once every 5 batches): Non-volatile residue, chloride ion, total free
sulfur, AAS total metal

3.2 Polymer / Pharmaceutical High-Purity Grade

Full test per batch mandatory: Purity, coulometric trace water, acid value, color, 2-cyclobutene sulfone, Na/Fe/Ca single metal, free sulfur, non-volatile residue

Sampling items: Thermal stability, solidification point, chloride ion

3.3 AR Analytical Reagent Grade

Full identical testing items as polymer grade plus chromatographic separation performance verification

3.4 Lithium Battery Electronic Ultra-Pure Grade

All indexes 100% tested per batch without exception: GC purity, coulometric trace water ≤ 10 ppm, acid value, APHA color, ppb-level 2-cyclobutene sulfone, ICP-MS full metal screening, free sulfur, thermal stability, non-volatile residue, chloride ion

4 Rapid Central Control Test Scheme for Circulating Solvent (On-Site Workshop, Precision Instrument Unnecessary)

1. Visual Appearance: Colorless & transparent = qualified; pale yellow = mild degradation; dark brown/black = severe degradation
2. Rapid volumetric Karl Fischer water test, result available within 10 min
3. Simplified indicator titration for acid value (isopropanol + phenolphthalein), quick judgment whether acid value exceeds 0.5 mgKOH/g
4. Filtration residue test: Vacuum filter 100 mL solvent through 5 μ m membrane; heavy black residue = excessive system degradation tar
5. Foam test: Shake 100 mL solvent for 1 min; foam persists >30 s without collapse = high tar content requiring anti-foam dosing

5 Test Result Classification & Non-Conforming Disposal Flow

5.1 Non-Conformity Classification

1. Minor non-conformity (conditional acceptance only for industrial grade): Slightly excessive color or water content, recoverable via distillation/dehydration; high-purity & electronic grades reject absolutely without concession
2. Critical non-conformity (full rejection)
 - Industrial grade: Purity <99.0 wt%, acid value >0.5 mgKOH/g, 2-cyclobutene sulfone >800 ppm
 - Polymer grade: Water >80 ppm, single metal >5 ppm
 - Electronic grade: Water >15 ppm, any single metal >5 ppb

5.2 Standard Disposal Workflow

1. Incoming raw material non-conformity: Isolate storage area, issue full non-conformance COA, return to supplier

2. In-plant circulating solvent non-conformity: Cut off from circulation loop for off-line vacuum distillation regeneration; only re-enter system after full requalification test
3. Finished product non-conformity: Collect separately for re-distillation, prohibited from blending into qualified finished product tanks

6 Laboratory Safety Operation Specifications

1. Sulfolane decomposes above 230°C to release toxic SO₂ gas; all high-temperature ovens and GC equipment must operate inside fume hoods
2. All high-purity electronic grade samples require nitrogen blanketing to isolate moisture & air during testing
3. Classify waste liquid separately: Metal-containing digestion waste liquid and sulfolane organic waste liquid stored in independent containers for centralized hazardous waste disposal
4. Prevent mixing sulfolane with strong oxidants (potassium permanganate, hydrogen peroxide, concentrated nitric acid) inside lab; exothermic decomposition risk exists
5. Operators must wear nitrile gloves and chemical splash goggles; flush skin contact area with large volume of running water for minimum 15 minutes

7 Wall-Mounted Quick Reference Test Card

1. Purity: GC FID internal standard dry-base quantification
2. Trace water: Coulometric Karl Fischer (polymer & electronic grade exclusive)
3. Acid value: Isopropanol-KOH potentiometric titration to judge solvent degradation
4. Metal testing: AAS for industrial grade, ICP-MS for electronic grade
5. Rapid degradation screening: Color + acid value + filtration residue three combined tests
6. Critical red lines: Acid value >0.5, excessive water, over-limit metal = unqualified

6 Industry COA Test Standard Manual for Sulfolane (Chinese Domestic & International Dual Specification System)

Preface

Certificate of Analysis (COA) acts as the sole quality certificate for import/export, incoming acceptance and factory release. Domestic testing is governed by GB/T 23946-2009 Industrial Sulfolane together with general chemical national standards. International testing is divided into five systems:

Petroleum Refining ASTM/UOP, Pharmaceutical ICH, Lithium/Electronics SEMI, EU REACH and US TSCA. COA index limits and test protocols differ drastically across downstream industries.

CAS No.: 126-33-0; Common English Name: Sulfolane / Tetramethylene sulfone

Part 1 Domestic Chinese Test Specifications for COA Issuance (Domestic Production & Domestic Sales / Raw Material Incoming Inspection)

1.1 Core National Standard GB/T 23946-2009 Industrial Sulfolane

Two grades defined: Premium Grade & First Grade; this standard must be marked on all domestic factory COAs for aromatic extraction and gas sweetening.

表格

Test Item	Premium Grade (Main Export & Large Refineries)	First Grade (Small Unit Internal Circulation)	Supporting Domestic National Test Standard
Sulfolane mass fraction (dry GC)	≥99.5 wt%	≥99.0 wt%	GB/T 23946 GC internal standard, DB-Wax capillary FID
Water mass fraction	≤0.05 wt% (500 ppm)	≤0.30 wt% (3000 ppm)	GB/T 6283 Volumetric Karl Fischer
APHA Color	≤10	≤30	GB/T 3143 Platinum- Cobalt Colorimetry for Liquids
Total Acid Value (mgKOH/g)	≤0.1	≤0.3	GB/T 12008.4 Isopropanol potentiometric titration
2-Cyclobutene sulfone impurity ppm)	≤0.05 wt% (500 ppm)	≤0.10 wt%	GC normalized trace impurity test
Non-volatile residue	≤0.01 wt%	≤0.02 wt%	GB/T 2397 Vacuum constant mass evaporation
Density (30°C, g/cm³)	1.260~1.268	1.258~1.270	GB/T 4472 Digital densimeter