2 Full Technical Guide for Lithium Battery & Electronic Grade Sulfolane Purification

Target Clients

Lithium battery electrolyte manufacturers in Japan & South Korea, high-voltage battery material enterprises in Europe & America, semiconductor wet chemical procurement teams, global new energy traders

Document Position

Downloadable for supplier factory audit, raw material bidding, process benchmarking and annual procurement budgeting; compliant with 2026 mass production standards, aligned with SEMI EL-II/EL-III electronic solvent standards

Core Coverage

Two-stage high-vacuum continuous distillation, ppb-level full-process trace metal control, closed-circuit deep dehydration, lithium battery cycle stability test data, clean mass production system, factory acceptance specifications

Catalog

1 Baseline Technical Indicators for Electronic Grade Sulfolane

表格

Test Item	**Industrial Refining Grade**	**High Purity Grade (99.9%)**	**Lithium Battery/Semiconductor Electronic Grade (99.99%)**	**Test Method**
Sulfolane Purity (GC)	≥99.50 wt%	≥99.90 wt%	≥99.99 wt%	GC-FID

Test Item	Industrial Refining Grade	High Purity Grade (99.9%)	Lithium Battery/Semiconductor Electronic Grade (99.99%)	Test Method
Water Content	≤50 ppm	≤20 ppm	≤10 ppm	Karl Fischer Coulometry
Color (APHA)	≤10	≤5	≤3	Platinum-Cobalt Colorimetry
Free Acidity (H_2SO_4)	≤0.001%	≤0.0005 %	≤0.0002%	Acid-Base Titration
Single Metal Ion (Na/K/Ca/Fe/Cu/Al/Mg)	≤10 ppb	≤5 ppb	≤1 ppb	High-Resolution ICP-MS
Total Heavy Metal Ion	≤30 ppb	≤12 ppb	≤5 ppb	ICP-MS
Low-Boiling Impurities (Butadiene, Mercaptan)	≤100 ppm	≤30 ppm	≤5 ppm	GC-MS
High-Boiling Polymer Tar	≤50 ppm	≤15 ppm	≤3 ppm	High-Temperature Residue Test
Peroxide	≤10 ppm	≤3 ppm	≤0.5 ppm	Iodometric Titration
Chloride Ion (Cl^-)	≤5 ppm	≤1 ppm	≤0.1 ppm	Ion Chromatography (IC)
Particles (≥0.2μm)	No Control	≤50 pcs/mL	≤10 pcs/mL	Liquid Particle Counter

Key Restrictions for Lithium Batteries

Metal ions, trace water and acidic impurities damage SEI film, accelerate electrolyte decomposition and cause rapid capacity decay of high-voltage cells. 4.4V/4.8V high-nickel ternary and 5V LNMO batteries in Japan & South Korea mandate electronic grade sulfolane, mixing with industrial grade prohibited.

2 Core Two-Stage High-Vacuum Continuous Distillation Purification Process

2.1 Process Principle

Sulfolane atmospheric boiling point: 285°C; obvious thermal decomposition above 230°C to generate SO_2 and butadiene. Two-stage low-temperature vacuum distillation is adopted with maximum kettle temperature ≤180°C to separate light impurities, heavy tar and metal complexes via volatility difference to reach 99.99% ultra-high purity. Single set capacity: 500–3000 tons/year.

2.2 Complete Two-Tower Process

Pre-Treatment Unit Before Distillation

Crude industrial sulfolane → Strong base anion exchange resin column → 1 μm PTFE precision filter → Nitrogen-blanketed raw material buffer tank

Function: Pre-remove sulfonate, sulfate and trace metal salt to reduce coking and metal precipitation load of distillation tower.

Stage 1: Light Removal Vacuum Distillation Tower

1. Operating conditions: Vacuum 3–5 kPa, kettle temp 155–165°C, reflux ratio 8:1
2. Separation target: Remove water, butadiene, mercaptan, alkane and aldehyde light impurities
3. Overhead discharge: Light waste solvent; tower bottom crude sulfolane sent to heavy removal tower

Stage 2: Heavy Removal Vacuum Distillation Tower (Core Finishing Section)

1. Operating conditions: Vacuum 2–3 kPa, kettle temp 165–175°C, reflux ratio 12:1 (high reflux ratio for electronic grade to boost purity)
2. Separation logic: Metal salt and polymer tar remain in tower bottom; high-purity sulfolane extracted from overhead vapor
3. Two-stage gradient low-temperature condenser (cooling water + ethylene glycol) to eliminate entrainment and metal contamination
4. Side draw of high-purity finished product into ultra-clean intermediate tank; tower bottom residue sent to regeneration system regularly

Electronic Grade Exclusive Vacuum System

Oil-free dry screw vacuum pump matched with low-temperature cold trap replaces water ring pump to avoid solvent hydrolysis, vacuum oil back-contamination and air oxygen ingress.

2.3 Ultra-Low Metal Precipitation Material Selection for Distillation Equipment

All material contact parts forbidden carbon steel or common 304 stainless steel to avoid Fe/Cr/Ni dissolution

1. Tower, reboiler, condenser & pipeline: Electropolished 316L stainless steel (EP Ra≤0.8μm)

2. Tower structured packing: PTFE / Hastelloy C22 without carbon steel support
3. Valves & pumps: PTFE-lined diaphragm pump, high-purity PTFE ball valve
4. Gaskets & seals: FFKM perfluoroelastomer, nitrile rubber forbidden

2.4 Critical Process Control Thresholds

1. Maximum kettle temperature strictly capped at 175°C; reduce vacuum & heating steam immediately if over limit
2. Full nitrogen micro-positive pressure blanketing, dissolved oxygen <0.1 ppm to prevent solvent oxidation
3. Reflux ratio fluctuation controlled within ±0.5 to avoid impurity penetration into finished product
4. Continuous 1% feed volume discharge of tower bottom residue daily to prevent metal salt enrichment

3 Full-Process Trace Metal Ion Control System (Single Metal ≤1 ppb)

Metal ions precipitate on graphite anode, pierce SEI film and cause self-discharge & thermal runaway. Full-chain control covers raw material, distillation, transportation, storage and filling.

3.1 Raw Material Pre-Treatment Metal Removal

Two-stage composite ion exchange bed:

1. Cation resin: Remove Na^+, K^+, Ca^{2+}, Fe^{3+}, Al^{3+}
2. Strong base anion resin: Remove sulfate, sulfonate and metal complex anion

   Space velocity 2–4 h^{-1}, two towers standby; resin regenerated with dilute alkali every 7 days, total metal reduced to ≤5 ppb before distillation.

3.2 Anti-Metal Dissolution Control During Distillation

1. Electropolished inner wall, citric acid passivation maintenance regularly
2. All flanges, fasteners made of 316L/Hastelloy without carbon steel contact
3. Continuous small flow discharge of tower bottom residue to avoid long-term high-temperature enrichment of metal salt

3.3 Transportation & Storage Metal Control

1. Full EP 316L pipeline without dead zone or welding burr
2. PTFE-lined stainless finished tank, nitrogen blanketing & passivated inner wall
3. Series filtration: 5μm→1μm→0.2μm PTFE filter to intercept metal particles washed from pipeline

3.4 Zero-Contamination Filling Control

1. ISO 7 clean filling workshop, air particle ≤352 pcs/m³ (0.5μm)
2. PTFE-lined IBC/steel drums, pre-rinsed with high-purity sulfolane 3 times before filling
3. Full nitrogen protection during filling without air contact; ICP-MS spot check before and after each batch filling

3.5 Real-Time On-Line Monitoring

On-line ICP rapid test for distillation side draw and tank outlet every 2 hours; switch unqualified fraction to recovery tank automatically if any metal >1 ppb.

4 Closed-Circuit Deep Dehydration Process (Finished Water Content Stabilized ≤10 ppm)

Sulfolane fully miscible with water; trace water reacts with lithium salt in high-voltage batteries to generate HF, corrode aluminum collector and consume lithium salt continuously. Crude industrial sulfolane water content: 300–800 ppm, three-stage coupled dehydration to reach standard.

4.1 Primary Dehydration: Light Removal Distillation

Most free water separated in overhead phase separation, feed water reduced to ≤50 ppm.

4.2 Secondary Deep Dehydration: Molecular Sieve Adsorption

3Å/4Å molecular sieve tower for continuous switching operation at 40–50°C, space velocity 1.5 h^{-1}; molecular sieve activated at 200°C under nitrogen cooling, regenerated via 180°C nitrogen purge with 3-year service life. Single stage reduces water to 12–18 ppm.

4.3 Tertiary Precision Dehydration: Pervaporation Membrane Coupling

Organic pervaporation membrane selectively traps water molecules, final finished water content stabilized at 5–10 ppm to meet high-standard electrolyte requirements of Japan & South Korea.

4.4 Full-Process Anti-Water Ingress Control

1. All tanks equipped with molecular sieve drying cylinder on breathing valve under continuous dry nitrogen micro-positive pressure
2. Complete isolation of cooling water & ethylene glycol circulation to prevent heat exchanger leakage
3. Vacuum drying + nitrogen replacement for IBC drums before filling, internal dew point ≤-60°C

5 Complete Lithium Battery Cycle Stability Comparative Test Data

Unified Test Conditions

Cell system: 4.45V NCM811 ternary / graphite pouch cell; electrolyte formula: 1.2M $LiPF_6$ EC:EMC=3:7, 5% sulfolane additive; charge/discharge rate

0.5C/0.5C; test temperature 25°C & 60°C, 1000 cycles, record capacity retention, initial coulomb efficiency and interfacial impedance growth.

5.1 Three Sample Gradients

- Sample A: Industrial grade sulfolane (metal 12–30 ppb, water 48 ppm, purity 99.52%)
- Sample B: High-purity grade (metal 4–8 ppb, water 18 ppm, purity 99.91%)
- Sample C: Lithium electronic grade (single metal ≤1 ppb, water 7 ppm, purity 99.99%)

5.2 25°C Room Temperature Cycle Capacity Retention

表格

Cycle Number	Sample A Retention	Sample B Retention	Sample C Retention
100	92.7%	95.4%	97.8%
300	83.1%	89.2%	94.5%
500	72.4%	83.6%	91.2%
800	58.3%	74.1%	86.7%
1000	47.6%	66.3%	82.4%

5.3 60°C High-Temperature Accelerated Aging (1000 Cycles)

- Sample A: 31.2% retention, obvious cell swelling, turbid electrolyte, massive metal deposition on anode
- Sample B: 52.8% retention, interfacial impedance increased by 126%
- Sample C: 76.9% retention, impedance only +38%, uniform & dense SEI film without metal deposition

5.4 Initial Coulomb Efficiency Comparison

- Sample A: 84.2% (massive irreversible side reaction induced by water & metal)
- Sample B: 87.5%
- Sample C: 91.6% (admission threshold ≥90% for 4.4V high-voltage batteries in Japan & South Korea)

5.5 Impurity Failure Mechanism Summary

1. Metal ion >5 ppb: Continuous deposition on graphite anode during cycling, piercing SEI film, self-discharge rise, cycle life shortened by 35%–50%
2. Water >20 ppm: Hydrolysis of LiPF6 to HF, aluminum foil corrosion, transition metal dissolution, doubled attenuation under high temperature

3. Mercaptan & peroxide: Destroy electrolyte stability, cause electrolyte discoloration and cell gas swelling

6 Clean Mass Production Supporting System (Mandatory for Client Factory Audit)

6.1 Clean Workshop Classification

1. Distillation main area: ISO 8 clean room
2. Ion exchange & membrane dehydration area: ISO 7
3. Precision filtration & automatic filling area: ISO 6 clean room

6.2 Closed-Circuit Nitrogen Supply System

Full plant high-purity nitrogen station (≥99.999%, dew point ≤-70°C); full nitrogen blanketing for all tanks, towers and pipelines to isolate oxygen & moisture.

6.3 Three-Waste Closed-Circuit Recovery

Distillation bottom residue and ion exchange regeneration waste liquid sent to residual distillation unit with sulfolane recovery rate ≥90%; residue incinerated as hazardous waste without direct discharge, complying with EU CBAM and Japanese/Korean environmental standards.

7 Full-Scope Factory Inspection & Compliance Delivery Document List

Basic Documents Attached to Each Shipment

1. Chinese & English TDS dedicated for electronic grade sulfolane
2. Batch full-index COA with CNAS lab seal
3. 16-Chapter GHS English SDS (REACH/TSCA compliant)
4. Distillation PFD & clean workshop qualification certificate

Additional Documents for Japan & South Korea Lithium Clients

1. Bilingual Japanese SDS, K-REACH registration copy, raw ICP-MS metal spectrum
2. ISO clean room certificate & nitrogen purity test report

Additional Documents for European & American Electrolyte/Semiconductor Clients

1. REACH registration, OR authorization, DoC compliance statement, RoHS halogen-free report
2. SEMI EL-II certification, GLP toxicology assessment
3. Electropolishing & passivation test certificate for all process equipment

8 Procurement Selection & Supply Chain Optimization Suggestions

1. 4.4V ternary & 5V LNMO high-voltage batteries: Electronic grade mandatory (single metal ≤1 ppb, water ≤10 ppm); high-purity grade substitution forbidden, long-term hidden cost of cell scrapping far exceeds raw material premium

2. Low-voltage LFP batteries: High-purity grade acceptable (metal ≤5 ppb) to balance procurement cost
3. Long-term fixed-price contract for annual procurement ≥500 tons: Stable distillation load, consistent batch quality, unit cost cut by 8%–12%
4. Inventory management: PTFE-lined IBC drums, storage temperature 20–30°C under nitrogen blanketing, shelf life 24 months, FIFO, quarterly re-test of water & metal content
5. Supply chain risk avoidance: Prioritize suppliers with self-owned two-stage vacuum distillation & ISO7 clean filling; small factories with only crude distillation fail to stabilize ppb-level metal control and are unsuitable for long-term new energy procurement

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