

Vinyltris(methylethylketoxime)silane

Process Troubleshooting Guide

Contents

Vinyltris(methylethylketoxime)silane Process Troubleshooting Guide	1
I. Basic Product Standards and Full Process Overview	3
1.1 Product Overview and Applicable Standards	3
1.2 Internal Control Quality Indicators of Finished Products and Troubleshooting Thresholds for Abnormalities	4
1.3 Core Segments of the Full Process (Mainstream Low-Temperature Solvent Oximation Route)	6
1.4 Access Control Standards for Raw and Auxiliary Materials	7
II. Special Troubleshooting for Typical Quality Defects of Finished Products	8
2.1 Yellow/Green Discoloration of Products with Deepening Color During High-Temperature Storage	8
2.2 Low VOS Main Product Content and Excessive Dimer Siloxane By-products	9
2.3 Excessive Free Methyl Ethyl Ketoxime, Retarded Curing of Adhesives and Strong Odor from High-Temperature Volatilization	9
2.4 Excessive Chloride Ions, Corrosion of Metal Substrates and Pink Spots on Adhesives	10
2.5 Excessive Moisture, Liquid Turbidity and Precipitation of White Gel Floccs During Storage	11
2.6 Liquid Delamination, Precipitation of White Ammonium Chloride Salt and Filter Blockage	11
2.7 Abnormally High Viscosity, Excessive Dimer By-products and Declined Crosslinking Activity	12

2.8 pH Deviation from the 6.8~7.5 Range and Rapid Hydrolysis Failure During Storage	12
III. In-Depth Process Fault Troubleshooting by Section	13
3.1 Raw Material Drying Pretreatment Section	13
3.2 Nitrogen-Protected Low-Temperature Oximation Section (Core Control for Purity, Color and Hydrolysis By-products)	13
3.3 Static Stratification Section (Key Control for Chloride Salt Entrainment and Chloride Ions)	13
3.4 Ammonia Neutralization and Desalination Section (Control for pH and Ammonium Chloride Precipitation)	14
3.5 Multi-Stage Precision Filtration Section (Control for Chloride Ions, Turbidity and Solid Impurities)	14
3.6 Vacuum Solvent Removal Section (Control for Free Methyl Ethyl Ketoxime, Dimer By-products and Moisture)	14
3.7 Light-Proof Nitrogen Filling and Warehousing Section	14
IV. Systematic Troubleshooting for Utility Engineering and Equipment	15
4.1 Nitrogen, Vacuum, Refrigeration, Heating and Drying Systems	15
4.2 Temperature Control, pH and Metering Systems	15
4.3 Reaction, Stratification, Filtration, Solvent Removal and Storage Tank Equipment Systems	15
4.4 Closed Light-Proof Clean Systems	15
V. Safety and Environmental Risk Troubleshooting	16
5.1 Process Safety Risks	16
5.2 Three-Waste Environmental Risks	16
5.3 Batch Quality Scrap Risks	16
VI. Standardized Five-Step Fault Troubleshooting Process	17
6.1 Full-Dimensional Abnormal Data Collection	17
6.2 Segmented Sampling for Source Tracing and Location Identification	17

6.3 Full-Coverage Inspection of Utility Engineering.....	17
6.4 Categorization and Judgment of Four Root Causes.....	17
6.5 Closed-Loop Verification of Rectification.....	18
VII. Quick Reference Table for Fault Troubleshooting.....	18
VIII. Periodic Preventive Troubleshooting Checklist.....	20
8.1 Per-Shift Inspection.....	20
8.2 Daily Inspection.....	20
8.3 Weekly Inspection.....	20
8.4 Monthly In-Depth Testing.....	21
8.5 Quarterly Shutdown Deep Maintenance.....	21
Disclaimer.....	21

As a new type of foreign trade service provider driven by technological transformation and foreign trade services, HiSiaddi has established a "1+2+3+4=1" service system and can supply original factory goods of Vinyltris(methylethylketoxime)silane from multiple well-known brands. As a research and development-oriented foreign trade enterprise, HiSiaddi sorts out key points requiring attention for process troubleshooting of Vinyltris(methylethylketoxime)silane products from a professional perspective for your reference.

If you need more professional and in-depth analysis regarding process troubleshooting or product consultation of

Vinyltris(methylethylketoxime)silane, please contact HiSiaddi customer service or visit the official website www.hisiaddi.com

I. Basic Product Standards and Full Process Overview

1.1 Product Overview and Applicable Standards

Vinyltris(methylethylketoxime)silane (abbreviated as VOS) is a ketoxime-type neutral silane crosslinker. Its molecule contains vinyl double bonds and three active methyl ethyl ketoxime groups. It is applied in one-component RTV silicone sealants, electronic potting adhesives and organosilicon coatings. It cures without corrosion to metal substrates and features dual functions of

crosslinking and adhesion promotion. The product is highly sensitive to moisture, high temperature, chloride ions and metal ions, and is prone to defects such as yellowing, turbidity and delamination, excessive free methyl ethyl ketoxime, hydrolysis and condensation to form siloxane dimers, gelation during storage, and abnormal curing speed. This guide complies with industry standards T/FSI 147—2024 and HG/T 5093, covering fault tracing, defect troubleshooting and closed-loop rectification for the full batch solvent synthesis process: raw material drying → low-temperature oximation reaction → static stratification → ammonia neutralization and desalination → filtration → vacuum solvent removal → finished product blending and light-proof filling.

1.2 Internal Control Quality Indicators of Finished Products and Troubleshooting Thresholds for Abnormalities

Test Item	High-Grade Electronic Premium Grade	General Sealant Industrial Qualified Product	Threshold Triggering Troubleshooting
Appearance	Colorless transparent low-viscosity liquid, free of turbidity, white salt floc precipitates, delamination and pungent amine odor	Light yellow transparent liquid without a large amount of solid impurities	Dark brown/tan color, milky haze, white ammonium chloride precipitates, pungent methyl ethyl ketoxime sour odor, two-phase delamination
Main Product VOS Content ≥ 98.5 (GC, %)		97.0~98.4	<97.0: insufficient oximation conversion, accumulation of dimer by-products, solvent residue diluting active components
Free Methyl Ethyl Ketoxime (%)	≤ 0.30	0.31~0.80	>0.80: unbalanced oximation ratio, failed solvent removal process, high-temperature

Test Item	High-Grade Electronic Premium Grade	General Sealant Industrial Qualified Product	Threshold Triggering Troubleshooting
Dimer Siloxane By-product (%)	≤0.40	0.41~1.00	volatilization of adhesive and retarded curing >1.00: hydrolysis due to water in the system, excessive condensation during high-temperature solvent removal, sharply reduced crosslinking efficiency >30: failed neutralization, desalination and filtration, long-term corrosion of metal substrates and yellowing/powdering of adhesives
Chloride Ion Cl ⁻ (ppm)	≤10	11~30	>300: continuous hydrolysis during storage, formation of gel flocs and caking at the bottom of filling drums
Moisture (ppm)	≤150	151~300	>25: nitro impurities in raw materials, iron ion- catalyzed oxidation, thermal degradation at high temperature generating chromophores
Color (Hazen)	≤10	11~25	Low viscosity indicates high solvent residue; high viscosity indicates hydrolytic crosslinking to form high-molecular siloxane
Viscosity (25°C, mPa·s)	3.0~4.2	2.5~4.8	

Test Item	High-Grade Electronic Premium Grade	General Sealant Industrial Qualified Product	Threshold Triggering Troubleshooting
pH (Diluted in Anhydrous Ethanol)	6.8~7.5	6.5~7.8	<6.5: acidity accelerates hydrolysis and metal corrosion; >7.8: alkalinity accelerates vinyl polymerization and yellowing
Thermal Storage Stability (60°C/72h)	Color increase ≤5 Hazen, no precipitate, free oxime increase ≤0.15%	Color increase ≤12 Hazen, no gel precipitation	Obvious tan discoloration, delamination with precipitated white salt/gel, doubled free oxime and delayed curing failure

1.3 Core Segments of the Full Process (Mainstream Low-Temperature Solvent Oximation Route)

1. Raw Material Drying Pretreatment Section: Vacuum rectification of methyl ethyl ketoxime to remove water and nitro impurities; airtight moisture-proof storage of vinyltrichlorosilane; negative pressure deep drying of 120# solvent oil/petroleum ether, strictly controlling water content of all three materials below 100 ppm, with filtration to remove suspended solid impurities.
2. Low-Temperature Oximation Reaction Section: Nitrogen purging of the reactor to eliminate oxygen, cooling and temperature control ≤35°C; premixing of methyl ethyl ketoxime and solvent, uniform dropwise addition of vinyltrichlorosilane with molar ratio 6.05~6.1:1, full-process temperature control ≤40°C, closed-loop capture of released HCl to suppress vinyl polymerization and hydrolysis side reactions.
3. Static Stratification Section: Constant-temperature standing for 2h after reaction completion; the system separates into an upper organic phase (crude VOS + solvent + excess methyl ethyl ketoxime) and a lower hydrochloric methyl ethyl ketoxime salt slurry; sufficient stratification to avoid salt entrainment into the organic phase.
4. Ammonia Neutralization and Desalination Section: Dry ammonia gas is introduced into the organic phase at low temperature to neutralize

residual HCl, with pH controlled at 7~8 to generate crystalline ammonium chloride solids; constant-temperature equilibration for 4h to allow full precipitation of salt particles.

5. Multi-Stage Precision Filtration Section: Pressure filtration via organic solvent-resistant anti-corrosion filter elements to completely remove ammonium chloride crystals and trace metal salt colloids, reducing chloride ions and ash content.
 6. Vacuum Solvent Removal Section: Low-temperature high-vacuum separation and recovery of solvents and excess free methyl ethyl ketoxime, gradient heating to avoid hydrolytic condensation at high temperature, with precise control of volatile content indicators.
 7. Stabilization Blending Section: Trace neutral buffer additives to fine-tune pH, compounding low-moisture anti-oxidation stabilizers to improve hydrolysis resistance and yellowing resistance during storage.
 8. Light-Proof Nitrogen Filling and Warehousing Section: 316L/HDPE light-proof storage tanks sealed under positive nitrogen pressure, moisture-proof sealed packaging to isolate water vapor, ultraviolet light and air oxidation.
- 1.4 Access Control Standards for Raw and Auxiliary Materials
1. Vinyltrichlorosilane: Industrial refined grade, colorless and transparent, moisture ≤ 80 ppm, free of free chlorine impurities and heavy metal catalytic ions.
 2. Methyl Ethyl Ketoxime: Rectified and purified grade, moisture ≤ 100 ppm, nitroalkane impurities < 5 ppm, clear color without yellowing; nitro impurities are the core inducer of green/yellow discoloration of finished products.
 3. Solvent Oil (120#/Petroleum Ether): Boiling range 60~90°C, moisture < 100 ppm, low aromatic hydrocarbon and sulfur-free impurities to prevent catalyst poisoning and color generation.
 4. Dry Liquid Ammonia: High-purity anhydrous ammonia free of water vapor and iron impurities; neutralization does not introduce extra metal ions.
 5. Anti-Oxidation Buffer Stabilizer: Neutral organic oxime additives that do not interfere with crosslinking activity, providing long-term inhibition of hydrolysis and oxidative discoloration.
 6. Equipment Materials: Reactors, separators, filters and distillation equipment shall be lined with enamel or passivated 316L stainless steel; direct contact of materials with carbon steel or copper pipelines is

strictly prohibited to prevent iron/copper ion complex-induced yellowing and catalyzed hydrolysis.

II. Special Troubleshooting for Typical Quality Defects of Finished Products

2.1 Yellow/Green Discoloration of Products with Deepening Color During High-Temperature Storage

2.1.1 Root Causes of Failure

Excessive nitroalkane impurities in methyl ethyl ketoxime raw materials generate conjugated colored intermediates; water in raw materials produces acidic substances upon hydrolysis, forming color complexes with trace iron ions; oximation temperature exceeding 45°C triggers vinyl oxidation and thermal degradation of methyl ethyl ketoxime to form chromophores; insufficient nitrogen purging leaves oxygen in the reactor to oxidize oxime structures; incomplete neutralization and filtration allow ammonium chloride and chloride ions to coexist with metal ions, catalyzing chain oxidation; excessively high temperature in the solvent removal kettle causes thermal degradation of materials over prolonged residence time; light-transmitting storage tanks lead to continuous photooxidation and deepening yellowing; corrosion of carbon steel equipment releases iron ions that synergize with chloride ions to produce discoloration.

2.1.2 Troubleshooting Points

Detection of nitro impurities, moisture and color of methyl ethyl ketoxime after rectification; oxygen content in the reactor after nitrogen purging before feeding; peak temperature during full oximation and solvent removal processes; pH at neutralization endpoint, chloride/iron ion content of filtrate after filtration; vacuum degree and material residence time during vacuum solvent removal; light-proof sealing of storage tanks and ambient warehousing temperature.

2.1.3 Rectification Measures

Mandatory secondary rectification of incoming methyl ethyl ketoxime to remove nitro impurities; reject raw materials with yellow discoloration or excessive nitro content; three times nitrogen purging per batch with oxygen content <10 ppm before feeding; hard temperature limits of ≤40°C for oximation and low-temperature solvent removal to eliminate thermal degradation; precise control of neutralization pH at 7~8 with two-stage precision filtration to fully remove ammonium chloride salts; quarterly passivation and anti-corrosion treatment of all equipment to isolate iron/copper ion leaching; full-process light-proof nitrogen-sealed storage of finished products with constant warehousing temperature ≤30°C; re-refining of

yellowed batches via low-temperature removal of light components and refiltration with added stabilizers.

2.2 Low VOS Main Product Content and Excessive Dimer Siloxane By-products

2.2.1 Root Causes of Failure

Insufficient molar ratio of methyl ethyl ketoxime to vinyltrichlorosilane leads to incomplete oximation; excessive moisture in raw materials causes premature hydrolysis of Si-Cl bonds to form silanols, which undergo intermolecular condensation into dimers; excessively fast dropwise addition during oximation causes local enrichment of trichlorosilane and sharp surge of hydrolysis side reactions; insufficient vacuum and high temperature during solvent removal trigger hydrolytic crosslinking of VOS molecules at high heat; excessive entrainment of salt slurry from the stratification section into the organic phase leads to salt impurities interfering with purification; repeated recycling of recovered solvents accumulates hydrolysis by-products that continuously dilute active components.

2.2.2 Troubleshooting Points

Monthly calibration records of raw material metering pumps and oximation feeding molar ratio; central control moisture data of the three incoming raw materials; dropwise flow rate and reactor temperature curve during oximation; static standing time during stratification and salt entrainment volume in the organic phase; vacuum degree and kettle temperature operating parameters during solvent removal; recycling batches and by-product content of recovered solvents.

2.2.3 Rectification Plan

Full calibration of all raw material metering and conveying equipment monthly, locking the standard oxime-silane molar ratio of 6.05~6.1:1; two-stage vacuum deep drying of raw materials with feeding only after moisture compliance; segmented slow uniform dropwise addition of trichlorosilane with enhanced cooling and temperature control simultaneously; extended static stratification time to reduce salt phase entrainment; optimized gradient low-temperature vacuum process for solvent removal to suppress thermal condensation; limit solvent recycling times and regular distillation to remove accumulated dimer impurities; re-purification of batches with substandard purity via low-temperature re-oximation and secondary solvent removal.

2.3 Excessive Free Methyl Ethyl Ketoxime, Retarded Curing of Adhesives and Strong Odor from High-Temperature Volatilization

Core Root Causes: Inaccurate matching of theoretical feeding volume with excessive methyl ethyl ketoxime; insufficient holding reaction time during oximation leading to incomplete conversion of chlorosilane and residual free oxime; insufficient negative pressure of solvent removal unit and premature judgment of solvent removal endpoint preventing full separation of light methyl ethyl ketoxime; excessively low solvent removal temperature sharply reducing oxime distillation rate; insufficient static stratification after neutralization mixing excess oxime dissolved in the salt phase into finished products; damaged tank sealing allowing water vapor from air to induce reversible hydrolysis and continuous release of methyl ethyl ketoxime.

Rectification Measures: Precise calculation of theoretical feeding volume with strict control of methyl ethyl ketoxime excess ratio; standardized holding reaction time for oximation, with GC central control monitoring of residual chlorosilane compliance before discharging materials; overhaul vacuum units to boost negative pressure and extend holding time for light component removal; lock the minimum temperature lower limit for solvent removal to guarantee oxime evaporation and separation efficiency; strict separation of salt slurry in the stratification process to eliminate entrainment; nitrogen positive-pressure sealed storage of finished products to block regenerated free oxime from hydrolysis; re-refining of batches with excessive free oxime via secondary low-temperature vacuum light component removal.

2.4 Excessive Chloride Ions, Corrosion of Metal Substrates and Pink Spots on Adhesives

Core Root Causes: Insufficient ammonia dosing during neutralization leaving residual unneutralized HCl unsalted; incomplete stratification leading to massive entrainment of hydrochloric methyl ethyl ketoxime salt into crude organic products; insufficient precision or damage of filter elements allowing penetration of microcrystalline ammonium chloride into finished products; insufficient static equilibration time after neutralization resulting in tiny salt particles difficult to settle and filter; long-term cyclic recycling of recovered mother liquor with continuous accumulation of chloride salts; rusting of equipment pipelines introducing chloride impurities into materials.

Rectification Measures: Real-time online pH monitoring during neutralization to stabilize the neutral range of 7~8; extended static settling time after stratification and neutralization to increase salt crystal particle size; replace high-precision organic phase-resistant anti-corrosion filter elements and monitor filter pressure difference per shift; regular complete replacement of recovered mother liquor to remove accumulated chloride salt impurities;

passivation and rust removal of all equipment to eliminate mixing of chloride corrosion products; re-purification of materials with excessive chloride ions via temperature-boosted salt dissolution and multi-stage pressure filtration for desalination.

2.5 Excessive Moisture, Liquid Turbidity and Precipitation of White Gel Flocs During Storage

Core Root Causes: Insufficient negative pressure for vacuum drying of methyl ethyl ketoxime, solvent and silane raw materials leading to excessive intrinsic moisture; water-laden nitrogen used for reactor purging introducing water vapor; high ambient humidity during stratification and neutralization with open transfer absorbing atmospheric moisture; when system moisture exceeds 300 ppm, VOS oxime groups hydrolyze to form silanols, which condense into insoluble siloxane gel; trace bound water cannot be removed during solvent removal; aging sealing gaskets of finished product tanks cause air leakage and cumulative moisture absorption over long-term storage.

Rectification Measures: Independent vacuum deep drying of the three raw materials with feeding only after moisture compliance; purging with dry high-purity nitrogen and full-process closed negative-pressure conveying, prohibiting open intermediate transfer; boosting vacuum gradient water removal during solvent removal to eliminate trace bound water; regular replacement of tank sealing gaskets and continuous micro positive nitrogen pressure storage; re-drying of gel-containing materials at low temperature and pressure filtration to remove gel impurities.

2.6 Liquid Delamination, Precipitation of White Ammonium Chloride Salt and Filter Blockage

Core Root Causes: Low temperature during neutralization increasing ammonium chloride solubility and preventing full crystallization; insufficient static standing time during stratification leading to incomplete solid-liquid separation; uneven stirring during neutralization causing local acid-base imbalance and ultra-fine salt particles difficult to settle; rapid temperature drop after solvent removal triggering saturated precipitation of ammonium chloride dissolved in the system; long-term storage temperature of finished products below 15°C inducing low-temperature crystallization and precipitation of salts; filter elements clogged with salt particles not replaced in a timely manner leading to continuous accumulation inside finished products.

Rectification Measures: Constant temperature control of 30~38°C during neutralization to promote salt crystal growth and settling; extended static settling time after stratification and neutralization; optimized stirring speed to

eliminate local acid-base dead zones in the reactor; gradient slow cooling after solvent removal to avoid sudden cooling-induced salt precipitation; constant warehousing temperature $\geq 18^{\circ}\text{C}$ for finished products to prevent low-temperature salt precipitation; temperature-boosted dissolution of stratified precipitated materials and multi-stage precision filtration for salt removal.

2.7 Abnormally High Viscosity, Excessive Dimer By-products and Declined Crosslinking Activity

Core Root Causes: Excessive moisture in the system triggering intermolecular hydrolytic condensation to form high-molecular dimeric/polymeric siloxane; temperature exceeding upper limits during oximation and solvent removal accelerating siloxane crosslinking side reactions; nitro impurities in methyl ethyl ketoxime catalyzing molecular oxidative polymerization; recovered solvents enriched with hydrolysis by-products cycled back into the reaction system; failure to filter immediately after neutralization allowing salt particles to catalyze continuous condensation.

Rectification Measures: Strict full-process control of raw material and system moisture to block hydrolysis at the source; rigid locking of upper temperature limits for each section to eliminate high-temperature polymerization; secondary rectification of methyl ethyl ketoxime raw materials to remove nitro chromogenic catalytic impurities; regular replacement of recycled solvents to eliminate accumulated by-products; immediate pressure filtration upon completion of neutralization to reduce salt-phase catalytic condensation time; separation of heavy dimer impurities via secondary molecular distillation for materials with excessive viscosity.

2.8 pH Deviation from the 6.8~7.5 Range and Rapid Hydrolysis Failure During Storage

Core Root Causes: Insufficient ammonia dosing during neutralization leaving trace residual HCl rendering the system acidic; excessive ammonia dosing making finished products alkaline and inducing oxidative yellowing of vinyl double bonds; oxidation of methyl ethyl ketoxime raw materials generating trace organic acids; thermal decomposition of a small amount of oxime groups during high-temperature solvent removal releasing acidic small molecules; insufficient stabilizer dosing or uneven stirring leading to local absence of buffer components; long-term storage with oxygen absorption and continuous carboxylic acid generation lowering pH.

Rectification Measures: Closed-loop pH control during neutralization with trace ammonia supplementation for precise adjustment to the neutral range; pre-rectification of methyl ethyl ketoxime raw materials to remove oxidized

acidic impurities; low-temperature solvent removal process to reduce thermal decomposition of oxime groups; standardized blending ratio of buffer stabilizers with extended homogenization stirring time for blending; light-proof nitrogen-sealed low-temperature storage of finished products to slow oxidative acid generation; low-temperature trace neutralization and homogenization blending for batches with abnormal pH.

III. In-Depth Process Fault Troubleshooting by Section

3.1 Raw Material Drying Pretreatment Section

Hidden Hazards: Methyl ethyl ketoxime contains excessive nitro and moisture introducing discoloration and hydrolysis triggers; insufficient vacuum drying of solvent oil and vinyltrichlorosilane leading to excessive total system moisture; insufficient precision of raw material filtration allowing suspended solids and rust impurities to enter the reactor; open storage of raw material tanks causing moisture absorption and oxidative degradation of components; uncalibrated metering pumps leading to inherent imbalance of oxime-silane ratio.

Process Red Line: All three main raw materials must undergo vacuum drying, precision filtration and full compliance testing of moisture, color and nitro indicators before feeding; raw materials with excessive moisture, yellow discoloration or nitro impurities shall be rejected directly.

3.2 Nitrogen-Protected Low-Temperature Oximation Section (Core Control for Purity, Color and Hydrolysis By-products)

Hidden Hazards: Insufficient nitrogen purging leaving residual oxygen in the reactor to oxidize oxime and vinyl groups generating color; uncontrolled dropwise addition rate of vinyltrichlorosilane causing local enrichment and rapid hydrolysis to form dimers; cooling system failure leading to reactor temperature over 40°C triggering vinyl polymerization and thermal degradation of oxime; excessive moisture in raw materials simultaneously inducing hydrolytic condensation side reactions; stirring dead zones causing uneven local material reaction and accumulation of salts and by-products.

3.3 Static Stratification Section (Key Control for Chloride Salt Entrainment and Chloride Ions)

Hidden Hazards: Excessively low constant temperature during stratification narrowing the density difference between organic phase and salt slurry leading to incomplete stratification; insufficient static standing time leaving micro salt particles suspended in crude organic products; excessively fast stirring shutdown emulsifying the system and preventing stratification; long-term uncleared salt accumulation at the bottom of the separator causing cross-batch entrainment of massive hydrochloric methyl ethyl ketoxime salt;

water vapor from ambient air entering the separator triggering premature turbidity and hydrolysis of crude products.

3.4 Ammonia Neutralization and Desalination Section (Control for pH and Ammonium Chloride Precipitation)

Hidden Hazards: Water-laden dry ammonia introducing water vapor and raising system moisture; metering deviation of ammonia dosing leading to over-acidic or over-alkaline pH; low temperature during neutralization generating tiny ammonium chloride crystals difficult to settle; insufficient stirring during neutralization causing large local acid-base concentration differences; insufficient static equilibration time after neutralization allowing incompletely precipitated salts to enter the filtration section.

3.5 Multi-Stage Precision Filtration Section (Control for Chloride Ions, Turbidity and Solid Impurities)

Hidden Hazards: Damaged or oversized organic phase filter elements allowing penetration of micro ammonium chloride particles into finished products; low filtration temperature inducing low-temperature salt precipitation and filter element blockage; insufficient filtration stages failing to intercept ultra-fine colloidal salts; long-term unchanged filter elements with excessive pressure difference allowing impurities to break through the filter layer; acid-alkali resistance failure of filter media leaching organic impurities and aggravating color turbidity.

3.6 Vacuum Solvent Removal Section (Control for Free Methyl Ethyl Ketoxime, Dimer By-products and Moisture)

Hidden Hazards: Negative pressure leakage or insufficient pumping capacity of vacuum units failing to remove solvents and free oxime; excessively high kettle temperature during solvent removal triggering hydrolytic condensation of VOS to form dimer siloxane; premature judgment of solvent removal endpoint leaving excessive light oxime residues; air leakage of the solvent removal system introducing air to oxidize materials and cause yellowing; long-term accumulation of heavy dimer impurities at the kettle bottom causing cross-batch contamination of fresh materials.

3.7 Light-Proof Nitrogen Filling and Warehousing Section

Hidden Hazards: Incomplete nitrogen purging of storage tanks leading to long-term oxygen absorption and oxidative discoloration; high humidity in filling workshops causing rapid moisture absorption and hydrolysis of finished products; light-transmitting packaging barrels and aging leaking sealing gaskets accelerating photooxidation under ultraviolet light; large diurnal temperature fluctuations in warehousing triggering repeated temperature rise

and fall leading to low-temperature salt precipitation and delamination; mixed tank storage of different reaction batches causing overall indicator fluctuations from component differences.

IV. Systematic Troubleshooting for Utility Engineering and Equipment

4.1 Nitrogen, Vacuum, Refrigeration, Heating and Drying Systems

Key Troubleshooting Points: Excessive moisture and oxygen in nitrogen failing to isolate water vapor and oxidation; leakage of nitrogen pipelines causing continuous air inhalation into reactors; negative pressure leakage or insufficient pumping capacity of vacuum units rendering raw material drying and solvent removal ineffective; unstable temperature control of refrigeration cooling systems failing to maintain the low-temperature oximation range; scaling of heating coils inducing local overheating triggering material hydrolysis, polymerization and yellowing; unstable temperature of raw material drying vacuum units leading to inadequate water removal.

4.2 Temperature Control, pH and Metering Systems

Key Troubleshooting Points: Drift of temperature measuring instruments for oximation, neutralization and solvent removal causing over-temperature polymerization or incomplete low-temperature reaction; salt fouling failure of online pH electrodes leading to distorted acid-base monitoring; uncalibrated metering pumps for vinyltrichlorosilane, methyl ethyl ketoxime and ammonia causing ratio imbalance; failure of pressure transmitters leading to unstable nitrogen and vacuum pressure; out-of-control heating gradient program disrupting dropwise reaction rhythm.

4.3 Reaction, Stratification, Filtration, Solvent Removal and Storage Tank Equipment Systems

Key Troubleshooting Points: Insufficient stirring speed of reactors and accumulated materials at bottom dead zones undergoing hydrolysis to form dimer impurities; peeling passivation layer of 316L stainless steel/enamel lining leading to iron ion leaching and complex-induced discoloration; aging, blocked or damaged organic phase filter elements at all levels allowing penetration of micro salt particles; accumulation of heavy dimer by-products on the inner wall of solvent removal towers causing continuous cross-contamination of fresh materials; accumulated ammonium chloride salt and gel impurities at tank bottoms leading to substandard batch indicators after material mixing; accumulated aged hydrolyzed materials at pipeline elbow dead zones.

4.4 Closed Light-Proof Clean Systems

Key Troubleshooting Points: Failed sealing of equipment flanges, manholes and storage tanks allowing air and water vapor intrusion to induce hydrolysis and oxidation; lack of light-proof protection in filling workshops accelerating photooxidation of oxime groups under ultraviolet light; excessively high ambient workshop humidity causing continuous moisture absorption during finished product transfer; mixing of dust and rust foreign matter into filled finished products; absence of constant-temperature light-proof protection for warehousing with dual degradation of materials from light and low temperature.

V. Safety and Environmental Risk Troubleshooting

5.1 Process Safety Risks

Vinyltrichlorosilane releases corrosive HCl gas with violent exothermic reaction upon contact with water; leakage splashes with water vapor causing burns; methyl ethyl ketoxime and solvent oil are flammable liquids with VOC explosion hazards from organic vapor; strong exothermic oximation reaction with risk of thermal runaway and reactor overflow upon cooling failure, posing explosion risks; ammonia is corrosive and toxic gas with respiratory irritation risks upon leakage; improper nitrogen purging of closed reactors creates confined space asphyxiation hazards; finished organic liquids are flammable with fire risks upon storage and transportation leakage.

5.2 Three-Waste Environmental Risks

Process cleaning wastewater contains siloxane, methyl ethyl ketoxime and ammonium chloride with high COD and salinity, requiring flocculation precipitation and neutralization pretreatment before compliance discharge; waste organic filter elements, salt filter residues and heavy gel residues are hazardous solid wastes; organic waste gas from solvent removal containing solvent and methyl ethyl ketoxime volatiles requires condensation and activated carbon adsorption devices to control VOC emissions; hazardous waste collection and separate disposal of chlorine-containing organic waste liquids are mandatory, with direct discharge prohibited.

5.3 Batch Quality Scrap Risks

Failure of raw material drying water removal and nitro impurity elimination, deviation of oximation ratio and temperature control, incomplete stratification neutralization and desalination, failed filtration dechlorination, drifting solvent removal parameters, and lack of light-proof moisture-proof storage control will directly lead to whole batches with insufficient purity, yellow turbidity, excessive chloride ions, strong odor from free oxime, hydrolysis and gelation during storage, resulting in curing failure of downstream electronic adhesives

and sealants, corrosion and discoloration of metal substrates, and mass scrap and rework of end products.

VI. Standardized Five-Step Fault Troubleshooting Process

6.1 Full-Dimensional Abnormal Data Collection

Record finished product appearance and color, VOS main content, free methyl ethyl ketoxime, dimer content, chloride ions, moisture, viscosity, pH and 60°C thermal storage stability; collect full-process parameters including moisture of dried raw materials, oximation temperature and pressure, ammonia neutralization dosage, static stratification time, vacuum temperature for solvent removal and filtration pressure difference during filling and warehousing; check ledgers of metering calibration, filter element replacement, equipment passivation and nitrogen leakage inspection.

6.2 Segmented Sampling for Source Tracing and Location Identification

Sampling points: dried raw materials → oximation reaction intermediates → stratified crude organic products → post-neutralization materials → post-solvent removal semi-finished products → blended finished products after filtration; sequentially test moisture, chloride ions, color, free oxime and dimer content to accurately locate the section where faults occur.

6.3 Full-Coverage Inspection of Utility Engineering

Verify purity and moisture of dry nitrogen, negative pressure of vacuum units, temperature control precision of heating and refrigeration systems and calibration of metering equipment; inspect operating conditions of oximation, stratification, neutralization, filtration and solvent removal equipment; check ambient temperature, humidity, light-proof sealing of filling workshops and constant-temperature light-proof storage environmental parameters.

6.4 Categorization and Judgment of Four Root Causes

1. Deviated Process Parameters: Oximation temperature, oxime-silane feeding ratio, neutralization pH, vacuum temperature for solvent removal and raw material drying moisture exceeding standard ranges;
2. Failed Equipment: Drifted metering pumps, damaged filter elements, vacuum leakage, drifted temperature measuring instruments, corroded stainless steel with iron leaching;
3. Defective Raw and Auxiliary Materials: Methyl ethyl ketoxime with excessive nitro/moisture, water-laden raw silane, water/oxygen-containing nitrogen;
4. Material Degradation and Side Reactions: Hydrolysis induced by moisture generating dimers, high-temperature polymerization, nitro/iron

ion-catalyzed yellowing, low-temperature salt precipitation and stratification, acid-base imbalance accelerating hydrolysis.

6.5 Closed-Loop Verification of Rectification

Isolate abnormal batches urgently, lock process red lines including anhydrous raw material pretreatment, low-temperature oximation, sufficient stratification and neutralization, multi-stage desalination filtration, low-temperature solvent removal and nitrogen-sealed light-proof storage; short-term measures: replace damaged consumables, calibrate metering instruments, passivate equipment to remove iron, optimize oximation cooling and dropwise addition rate; long-term solidified systems: full inspection of incoming raw materials for moisture/nitro content, monthly metering calibration, regular filter element replacement and quarterly equipment passivation system; resume mass production only after three consecutive batches meet all indicators with no yellowing or stratification after 72h thermal storage at 60°C and compliant chloride ion and free oxime content.

VII. Quick Reference Table for Fault Troubleshooting

Abnormal Phenomenon	Core Root Cause	Priority Troubleshooting Section
Yellow/green discoloration of products with deepening color during high-temperature storage	Excessive nitro impurities in methyl ethyl ketoxime, water in raw materials, over-temperature during oximation and solvent removal, iron/chloride ion-catalyzed oxidation, light-transmitting storage tanks	Raw material drying and rectification, low-temperature oximation, neutralization desalination and filtration, light-proof airtight storage
Low VOS main content and excessive dimer siloxane	Insufficient oxime-silane ratio, hydrolysis due to water in the system, excessively fast dropwise addition during oximation, thermal condensation during high-temperature solvent removal, salt phase entrainment of by-products	Raw material metering, oximation reaction, stratification separation, vacuum solvent removal

Abnormal Phenomenon	Core Root Cause	Priority Troubleshooting Section
Excessive free methyl ethyl ketoxime, retarded curing of adhesives and strong odor	Excessive feeding of methyl ethyl ketoxime, incomplete conversion during oximation, insufficient vacuum for solvent removal, regenerated oxime from hydrolysis during storage	Oximation holding reaction, vacuum light component removal, nitrogen-sealed storage
Excessive chloride ions, metal corrosion and pink spots on adhesives	Insufficient ammonia for neutralization, salt entrainment during stratification, damaged filter elements, cumulative chloride salts in recycled mother liquor	Static stratification, ammonia neutralization, multi-stage precision filtration, replacement of recycled mother liquor
Excessive moisture, liquid turbidity and precipitated gel flocs	Substandard raw material drying, water-laden nitrogen, moisture absorption during open workshop transfer, air leakage of storage tank seals	Raw material vacuum drying, full-process nitrogen protection and closed conveying, nitrogen-sealed storage tanks
Liquid delamination and precipitation of white ammonium chloride salt	Low neutralization temperature preventing sufficient salt precipitation, insufficient static stratification time, sudden cooling during solvent removal, low-temperature crystallization during storage	Constant-temperature salt precipitation during neutralization, stratification section, gradient cooling after solvent removal, constant-temperature warehousing
Excessively high viscosity and declined crosslinking activity	Hydrolytic polymerization induced by moisture, dimer generation at high temperature, nitro impurity-catalyzed oxidation,	Deep water removal of raw materials, temperature control during oximation and solvent removal, rectification of methyl ethyl ketoxime to

Abnormal Phenomenon	Core Root Cause	Priority Troubleshooting Section
pH deviation from 6.8~7.5 and rapid hydrolysis failure during storage	accumulation of by-products in recycled solvents	remove impurities, regular replacement of solvents
	Metering deviation of neutralization ammonia, acid generation from oxidized raw materials, thermal decomposition of oxime groups during solvent removal, insufficient stabilizer, long-term oxygen absorption and oxidation	Fine-tuning section of ammonia neutralization, raw material pretreatment, finished product stabilizer blending, light-proof oxygen-isolated storage

VIII. Periodic Preventive Troubleshooting Checklist

8.1 Per-Shift Inspection

Appearance clarity of finished products, presence of salt precipitates, gel or delamination; preliminary screening of moisture and color of three dried raw materials; oxygen content after nitrogen purging in reactors; real-time oximation temperature, silane dropwise addition rate and cooling operating conditions; stratification state after static separation; neutralization pH and salt precipitation effect; vacuum and kettle temperature operating parameters during solvent removal; filtration pressure difference and clarity of filtrate; nitrogen positive pressure of filling storage tanks, workshop temperature, humidity and light-proof inspection.

8.2 Daily Inspection

Sampling inspection of operating parameters for dry nitrogen and vacuum units; rapid central control testing of finished product main content, moisture, pH, color and free oxime; simple recheck of oximation conversion rate; observation of pressure difference across all organic filter elements; real-time monitoring of viscosity and GC components at solvent removal endpoint; inspection of light-proof sealing and constant-temperature state of finished product storage tanks.

8.3 Weekly Inspection

Full-line leakage verification of nitrogen and vacuum negative pressure systems; cleaning of salt scale and dimer accumulated at the bottom of reactors, separators and solvent removal kettles; integrity inspection of all filter elements and replacement of blocked elements; simple passivation

inspection of stainless steel/enamel equipment inner walls; testing of chloride ion and dimer impurity content in recovered solvents, replacement upon reaching upper limit; discharge of water and solid impurities at the bottom of raw material storage tanks.

8.4 Monthly In-Depth Testing

Full calibration of full-line temperature measuring instruments, pH electrodes and metering pumps; full-indicator testing of raw materials and finished products with special re-inspection of chloride ions, free methyl ethyl ketoxime and thermal stability; deep purification of utility systems including dry nitrogen, vacuum and refrigeration heating; non-destructive testing of metal passivation layers on reactors, solvent removal kettles and storage tanks; verification of 60°C accelerated thermal storage stability of finished products.

8.5 Quarterly Shutdown Deep Maintenance

Complete cleaning of salt deposits, dimers and colored impurities from all raw material drying, oximation, stratification, neutralization, filtration and solvent removal equipment; maintenance of stirring, vacuum, heat exchange and conveying transmission components; complete replacement of all sealing gaskets and organic filter elements; comprehensive verification of oximation explosion protection, nitrogen oxygen isolation, vacuum negative pressure and light-proof clean systems; screening of stock methyl ethyl ketoxime, vinyltrichlorosilane and solvents to reject deteriorated raw materials with excessive moisture, nitro impurities or oxidation.

For more professional and in-depth analysis regarding process troubleshooting or product consultation of

Vinyltris(methylethylketoxime)silane, please contact HiSiaddi customer service or visit the official website www.hisiaddi.com

Disclaimer

All technical processes and compliance information related to Vinyltris(methylethylketoxime)silane covered in this document, including but not limited to product basic parameters, application guidelines, addition ratios, production processes, mass production optimization, fault troubleshooting, material compatibility, product selection, testing methods, national and international regulations and market access, are for industry popular science and technical exchange reference only. They do not constitute professional engineering design, production operation specifications, third-party test reports, product quality warranties, chemical safety operation manuals, import and export compliance legal opinions, nor form contractual commitments for sales, technical services or commissioned processing. There is no guarantee

that the content is fully applicable to your production, procurement and export scenarios.